MIL-HDBK-407

31 JANUARY 1972

MILITARY STANDARDIZATION HANDBOOK

CONTAMINATION CONTROL TECHNOLOGY

PRECISION CLEANING METHODS AND PROCEDURES

FSC 3694

MIL-HDBK-407
Contamination Control Technology
Precision Cleaning Methods and Procedures
31 January 1972

1. This standardization handbook was developed by the US Army Missile Command in accordance with established procedure.

2. This publication was approved on 31 January 1972 for printing and inclusion in the military standardization handbook series.

3. This document provides fundamental information on cleaning methods and procedures for use in precision cleaning of materials. The handbook is not intended to be referenced in its entirety for mandatory use in procurement specifications except for informational purposes. Specific materials may be specified. No criteria herein is intended to supersede any specification requirements.

4. Every effort has been made to reflect the latest information available. It is the intent to review this handbook periodically to insure its completeness and currency. Users requests for changes and inclusions in this handbook should be sent to:

Commanding General
US Army Missile Command
Standardization Division
AMSMI-RCS
Redstone Arsenal, Alabama 35809

FORWARD

The purpose of this document was to establish general data for operational and performance characteristics in handbook form in accordance with Defense Standardization Manual 4120.3-M for use in contamination control technology.

The information in this handbook was an accumulation of existing documents, Governmental, Technical Society, and DOD contractor data which after evaluation was considered pertinent to the state-of-the-art.

Recommended cleaning methods and procedures included are those used by military services and industry in gross and precision cleaning of materials, parts, and assemblies being processed or assembled under controlled environmental conditions. The most feasible sequence of application for each method or procedure in order to obtain maximum results during the precision cleaning cycles of categorized types of sensitive mechanisms (or components) was considered in conjunction with the requirements of Mil-Std-1246A.

SAFETY WARNING

Adequate safety precautions should be taken when handling any chemical (solvent, cleaning material, etc.). Inhalation or direct contact with the skin should be avoided. Appropriate protective clothing (suits, gloves, masks, etc.) should be used at all times, while handling chemicals, or when working around them. Some chemicals present fire hazards, others vary in toxicity, and others are asphyxiants.

The Threshold Limit Values (TLV) or Maximum Allowable Concentration (MAC) is listed for most chemicals encountered in the clean room environment. These are recommended values only, and should be used as a guide, as some individuals are more sensitive to certain chemicals than others.

When combinations of chemicals are used together or in proximity to each other the effects of the different hazards should be considered as additive. That is the sum of the following fractions.

C_1, C_2, and C_n = Concentrations of the constituents

T_1, T_2, and T_n = TLV

$$\frac{C_1}{T_1} + \frac{C_2}{T_2} + \cdots\cdots \frac{C_n}{T_n}$$

Example. Assume that the air contains 15 ppm carbontetrachloride (TLV=25), 100 ppm trichloroethylene (TLV=200), and 200 ppm methyl ethyl ketone (TLV=250).

$$\frac{15}{25} + \frac{100}{200} + \frac{200}{250} = \frac{475}{250} = 1.9$$

Thus the accumulated threshold limit is exceeded.

Note. More detailed information may be obtained from the Governmental Industrial Hygienists, 1014 Broadway, Cincinnati, Ohio 45202 or the Handbook of Toxicology, Vol. 1, Acute Toxicities, prepared by The National Academy of Sciences, National Research Council.

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SECTION I

INTRODUCTION

1.1 Objective.

1.1.1 Purpose. The purpose of this program is to delineate requirements to that extent necessary to establish general data for operational and performance characteristics essential to developing an adequate document support structure suitable for use in contamination control technology.

1.1.2 Contaminant. Relative to contamination control technology a contaminant is any material, substance, or energy which is unwanted or adversely effects the contaminee. More specifically defined, contamination may be classified into four general categories:

(a) Particulate: dust, chips, fibers, etc.

(b) Chemical: gases, liquids, oily films, etc.

(c) Biological: bacteria, virus, fungi, spores, etc.

(d) Energy or changes of state: light, radiation, magnetic fields, etc.

1.1.2.1 Effects. The effects of contaminants include such things as increased electrical contact resistance; micro-organism transmission of infection; malfunction or failure to function of systems, devices, or missions. Means of controlling these types of problems are discussed in the following sections.

1.1.3 Reason. Due to advancements in technology, the need for contamination control has increased considerably. The development of extremely sophisticated electronic, electro-mechanical, electro-optical, and hydraulic equipment has created a need for contamination control to microscopic measurements.

1.2 Scope.

1.2.1 Contamination control. For our purposes, contamination control will be defined as the planning, organization, and implementation of all activities required to determine, achieve, and maintain a required cleanliness level in, on, or around the contaminee. The sources and physical attributes of contaminants most commonly encountered and the most recent analytical techniques and means of control are discussed in the following sections.

1.2.2 Precision cleaning. As pertains to this handbook, precision cleaning is defined as final or fine cleaning accomplished in a controlled environment to remove minute quantities of contaminants to better than visual standards.

1.3 Major problem areas. Three major problem areas exist in contamination control technology. First, attitudes of personnel and management; second, training of clean room personnel; third, compatibility of cleaning solutions to materials being processed.

1.3.1 Attitudes of personnel and management. Clean room operations require stringent enforcement of regulations and therefore require constant alertness. Because self-motivation varies greatly among personnel, the clean room supervisor has a demanding task. The management personnel must spend considerably more time watching workers for inadvertent infractions than they would in usual working situations. Development of conscientious attitudes is essential to the success of a clean room operation.

1.3.2 Training of clean room personnel. Contamination control technology has developed into a science in which exactness is critical. Error, in some instances, can cause loss of life or loss of millions of dollars. Therefore, control of the human factor to eliminate or lessen error probability is of paramount concern.

It appears that the information presently available for training programs is deficient in promoting motivation and is void of principles of learning behavior that have proven effective in areas where retention of information is imperative.

1.3.3 Cleaning solution to material compatibility. There appears to be insufficient information available to determine the degree of chemical corrosion or etching that may occur on various metals during cleaning processes such as pickling or passivating. For example, if a metal with a number sixty-four finish is placed in solution "A" for ten minutes at 140°F, to what degree will the finish be affected? Flared tubing and metal sealing surfaces present definite cause for concern in this area. Another compatibility consideration is solution effects on plastics of discrete components mounted on circuit boards.

1.4 Responsibility. To assure that a given product will function reliably, contamination control must be considered along with all other requirements during each stage of product development. Ideally, the product designer should design his product to tolerate the contamination environment under which it will be manufactured and actually used. Therefore, primary responsibility for contamination control lies with the design engineer. Next, the assurance of continuing reliability of a product depends on the ability of contamination control technologists to maintain a product at the required level of cleanliness to insure function reliability.

1.5 References.

1. Clean Room Technology, James W. Useller, Lewis Research Center, Cleveland, Ohio, NASA SP-5074, 1969.

2. Contamination Analysis and Control, James L. Dwyer, Reinhold Publishing Corporation, 1966.

3. Contamination Control Handbook, Sandia Laboratories, NASA SP-5076, 1969.

4. Contamination Control, Hospital Sanitation - Past, Present, and Future, Jan W. Leemhorst, Vol. IX No. 9 & 10, Sep. - Oct., 1970.

5. Contamination Control Principles, Sandia Corporation, NASA SP-5045, 1967.

6. Corrosion Prevention/Deterioration Control in Electronic Components and Assemblies, R. H. Sparling, U. S. Army Missile Command, CR 6-347-958-001, DDC-AD815470.

7. Spacecraft Sterilization Training Manual, Elizabeth A. Quinn, Hayes International Corporation, NASA CR-61253, 1969.

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SECTION 2

GLOSSARY OF TERMS

2.1 Definitions.

A. Absolute rated filter. A filter that theoretically and essentially retains all particles whose smallest dimension is equal to or greater than the absolute rating.

Absorption. Penetration of a substance into the body of another; to take in and incorporate; assimilate. Adhesion of the molecules of a gas liquid, or dissolved substance to a surface.

Acceptable environmental range test. A test to determine the range of the environmental conditions in which an equipment has a reliability at least as high as required.

Acceptable quality level. A nominal value expressed in terms of percent defective or defects per hundred units, whichever is applicable, specified for a given group of defects of a product.

Acceptable reliability level. A nominal value expressed in terms of percent failure per thousand operating hours specified for acceptance of parts or equipment. It is a measure of reliability which will be accepted, some preassigned percentage of the time, by a reliability sampling plan.

Acceptance inspection. Examination and testing, to determine conformance of supplies or services to certain specified requirements which serve as a basis for acceptance.

Accessory. A part, subassembly or assembly designed for use in conjunction with or to supplement another assembly, unit, or set. An accessory contributes to the effectiveness of the assembly or set thereof without extending or varying the basic function.

Acid. A substance whose molecules ionize in water solutions to give off hydrogen ions; a substance which registers less than 7 on the pH scale.

Acid cleaning. The removal of oil, dirt, and soil additions to the scale and rust by the use of acids or acid compounds containing detergents.

Aclar. A fluorohalocarbon used in packaging. Excellent moisture vapor barrier; particles are LOX-compatible.

Acoustics. The science of sound, its production, transmission, and effects.

Adcad. Auto-radiographic detection of contamination by adsorption/desorption.

Adsorption. Adhesion of the molecules of a gas, liquid, or dissolved substance to a surface; the taking up of one substance at the surface of another.

Aerobic microorganisms (or obligate aerobic). Microorganisms that grow only in the presence of free or atmospheric oxygen.

Aerosol. A suspension of microscopic solid or liquid particles in air or gas.

Agglomeration. The combining, joining, clumping, or clustering of two or more particles or droplets by any means.

Airborne. Suspended or carried in a gas or airstream.

Airborne particulate matter. Particulate matter suspended in ambient atmosphere.

Air-cleanliness class. Each class of air cleanliness is determined by the particle count per unit volume, based on tabulation of particles 0.5 micron and larger or 5.0 microns and larger. Examples of standard air-cleanliness classes are described and illustrated in Section 4, Paragraph 5.1.

Air conditioning. Air conditioning is the control and conditioning of the air in the clean room to maintain specified standards of temperature, humidity, airborne particulate contamination, and pressurization. This is accomplished by cooling and heating equipment, humidifiers, dehumidifiers, air filters, and allied controls.

Air contamination. General ranges from 40,000 particles per cubic foot in rural areas to 1.5 million particles per cubic foot in metropolitan areas. Particle sizes ranging from 0.5 microns to 600 microns.

Airlock. A chamber with doors functioning to maintain pressure during entry to and exit from an enclosed area. It is designed to provide an air barrier for the controlled environment area by preventing the entry of contaminated air.

Air shower. A chamber with interlocked doors and equipped with an exhaust system, having numerous air nozzles arranged in a predetermined pattern, for the purpose of forcibly blowing loose particles, fibers, dust, and other particulate matter from the person and garments.

Aliphatic hydrocarbon. An organic compound having an open chain structure; as a class, petroleum-type solvents.

Alkali. Any base or hydroxide that is soluble in water and can neutralize acids; a substance that registers more than 7 on the pH scale.

Ambient condition. Environmental conditions such as pressure, temperature, humidity, etc., which are normal for one specific location.

Anaerobic microorganisms (or obligate anaerobic). Microoragnisms that grow in an environment with no free oxygen.

Antiseptic. A substance that has sufficient antimicrobial activity to interfere with the development of infection but which is non toxic to living tissue.

Aqueous. Aqueous solutions are water base solutions. They are used in cleaning to remove many salts, acids, sugars, and syrup deposits; have little or no solvent effect on oils and greases.

Article. A unit of hardware, or any portion thereof, required by the contract.

Artificial environment. The state or conditions produced in a controlled test space which generates a simulated natural environment as an input to the equipment undergoing test.

Assembly. An assembly is defined as a unit consisting of two or more replaceable parts (e.g. filter element, filter housing and fittings are part of a filter assembly) having a common mounting and which is within its physical make-up, capable of performing a definite function. (Examples: filter assembly, regulator assembly, and valve assembly).

Atmosphere. The body of air surrounding earth. Also the body of gases surrounding or comprising any planet or other celestial body.

Autotrophic microorganisms. Microorganisms capable of growth in the presence of only simple salts and carbon dioxide.

B.

Bactercide. A substance capable of killing bacteria.

Bioclean facility. Any enclosed area (room or work station) characterized by having its own filtered air or gas supply where there is control over viable and non viable particulates in air with temperature, humidity, and pressure control as required to maintain standards for the product.

Blocking. The physical phenomenon of a material sticking or adhering to itself in the presence of heat; an undesirable characteristic for packaging films at temperatures under 200° F.

Buffer activity. The means by which a solution resists a change in the degree of acidity or alkalinity.

Built-in contamination. That which exists within a system due to the processes used in manufacturing lines, components,and fluid.

C.

Cavitation. Sound waves create an alternating *expansion and* compression of the solution the wave is passing through (detergent, solvent, etc.) and causes a vibration in it. This vibrating action has the ability to physically "rupture" a liquid forming numerous bubbles on the surface of the object being cleaned. This is called cavitation. As the compression part of the wave cycle commences these bubbles burst with considerable energy, physically removing adhering particles or other contamination.

Checkout fluid. A fluid other than operational fluid for use in evaluating performance of a system; sometimes called a test fluid.

Chemical cleaning. The term shall indicate pickling, passivating, descaling, deoxidizing and other cleaning processes where surface *conversion or preparation* is the prime objective.

Chemotroph. An organism whose primary energy source is chemical.

Clean. That contaminant level just below that which affects the operation or reliability of the part, *component, system, or environment.*

Cleanable. Capable of being cleaned to specified levels without detrimental effect.

Clean-down capability. The time required for a clean room to recover and return to its normal level or class of air cleanliness after introduction or generation of any unusual quantity of contamination.

Cleaning. Act of removing unwanted substances.

Cleaning agent. Any compound or substance which promotes the removal of soils through mechanical or chemical action.

Cleaning criteria or standards. The quantitative expression of a specified cleanliness level.

Cleaning procedures. A detailed, stepwise method for achieving a cleaning requirement.

Clean item. A part, component, or system which is wholly cleaned or has significant surfaces which have been cleaned and verified to a specified level of cleanliness.

Cleanliness level. An established maximum allowable distribution of contamination of a given size and quantity in a stipulated area or volume.

Clean packaging. The application of clean preservation and packaging measures and material to maintain the cleanliness of a clean item during handling, storage, or shipment.

Clean room. A clean room is an enclosed area employing control over the particulate matter in air with temperature, humidity, and pressure control as required. To meet the requirements of a "clean room", the area must meet the particulate count as specified in Fed-Std-209a, Section 4, Paragraph 4.2.2.

Clean water. Color-free water containing no visible suspended particles.

Clean work station. A work bench or similar working enclosure characterized by having its own filtered air supply. The filters must be capable of providing the required air-cleanliness level.

Climatic environments. Environments consisting of ambient atmospheric conditions such as degree of vacuum, radiation, and temperature.

Clone of cells. The aggregate of asexually produced cellular progeny.

Cold boil. Cavitation which is not dependent upon heat for its bubbling action.

Combustible contaminants. Flowable solvents, cleaning solutions, oils, paints, preservatives, wood, or other materials that are present in a component or system.

Complete blocking. Blocking which occurs when individual contamination particles are large enough to plug a clearance.

Component. A series of two or more parts, sub-assemblies, assemblies, or any combination thereof, which in turn becomes a piece of functional equipment or assembly.

Container. Any receptacle or flexible covering capable of completely enveloping an item or items.

Contaminant. Any material, substance, or energy which is unwanted or adversely affects the contaminee.

Contaminant level. A quantitative expression for the size, distribution, shape, quantity and physical properties of one type of particulate matter in a fluid.

Contaminant-sensitive part. A part whose function may deteriorate with the presence of material(s) other than those for which it was designed. The foreign material may be liquid, gaseous, or solid in nature, with size, number, or character harmful to the operation of the device.

Contaminant transmission rating. The contaminant transmission rating is the maximum particle size that is permitted as a result of the extrusion of particulate through the filter media under the rated flow and maximum working pressure of the filter being tested.

Contaminate. The act of introducing any contaminant; to make impure or unclean; to pollute, defile, sully, taint, or soil.

Contamination. The presence of one or more contaminants in or on the contaminee.

Contamination control. The planning, organization, and implementation of all activities needed to determine, achieve, and maintain a required cleanliness level in, on, or around the contaminee.

Contamination level. A quantitative expression for the size, distribution, shape, quantity and lumped physical properties of a mixture of contaminants in a fluid. This is the general case.

Contamination tolerance level. The contamination level which cannot be exceeded and still allow components to have a specified performance, reliability, and life expectance. Contaminant tolerance level would refer only to the influence of one type of contaminant.

Contaminee. That which is or can be contaminated. Contaminees may be products, materials, devices, people, gases, or surfaces.

Controlled area. Any enclosure which has a degree of control of contaminants in air, gases, and fluids (may include temperature, humidity, and pressure) and which will not qualify as a clean room.

Controlled environment facility. A specified working area that has the primary objective of controlling one or more physical, chemical, or biological variables.

Controlled work area. An area where a high degree of cleanliness is maintained by the enforcement of strict controls on personnel access, operations, and facility maintenance as opposed to a clean room where the total environment is controlled by high efficiency air filters, humidity and temperature controls, and the enforcement of more strict personnel and clothing controls.

Conventional clean room. A clean room in which the airflow patterns are random.

Coolant. A heat transfer fluid.

Corrosion. A specific type of deterioration resulting in damage or impairment of metals or metallic parts as the result of attack by moisture, air, acid, alkali, chemicals, or electrochemical action. Although mechanical stress is a factor in certain types of corrosion, damage, or breakage as the result of purely mechanical load or shock is not included. (Mil-Std-1250 (MI), 31 March 67.

Crossflow clean room. A clean room in which the air enters through an entire wall with filters and is exhausted through the opposite wall; air travel within the room is predominantly horizontal. (See Horizontal Laminar Airflow Clean Room).

Cryogenics. The science of low temperature phenomena.

Curie point. The temperature at which the ultrasonic piezoelectric effect can no longer be produced.

Cycle. One beat of a vibrating body. One complete sound or electromagnetic wave.

D.

Decontamination. The process of removing unwanted matter; the reduction of contamination to an acceptable level.

Deionization. The removal of mineral ions from water.

Deep-bed filter. Common form of high-efficiency filter for low-pressure use in ventilation systems.

Deflocculating agent. Chemical materials which have the ability to break up a solid mass into small particles and to disperse them through a liquid medium.

Deflocculation. A mechanism of a deflocculating agent which enables the neutralization of those forces of surface tension responsible for the attraction between solid particles.

Demineralize. Removal of suspended soluble minerals such as calcium carbonate from water or other liquids.

Demineralized water. Water having a total solids content of not more than 20 parts per million (ppm) by weight suspended and dissolved solids, and pH of 5.5 to 9.0. The water shall be filtered through a 10 micron nominal rated filter with an absolute rating of 40 microns.

Descaling. Removal of rust, scale, welding, flux,smut, discoloration (corrosion), and light soil.

Design packaging. The enclosure of a unitized assembly of components, or parts in a protective container, covering,or coating which is an integral part of the item and will enhance the function of the item; usually involves the use of sealed cans, encapsulating compounds, etc.

Deviation. A specific authorization, granted before the fact, to depart from a particular requirement or specification or related documents.

Dew point. Dew point is used to indicate dryness (or wetness) of a gas and is defined as the temperature at which water vapor in a gas becomes saturated and condenses out of the gas.

Diatomaceous earth. A light, friable, siliceous material derived chiefly from diatoms (minute planktonic algae); chiefly used for filters.

Direct test. Any surface cleanliness test in which the parameter measured is the amount of soil remaining on a surface. (See Indirect Test.)

Disinfectant. An antimicrobial substance that destroys all (vegetative) microorganisms; applied to inanimate material; may be toxic.

Distillation. The phase changes from liquid to vapor to liquid; a process for removing impurities from liquids.

DOP (Dioctyl Phthalate) test. DOP aerosol or "smoke" is introduced upstream of the filter at a concentration of 80 to 100 micrograms per liter and the downstream side of the filter is scanned with an aerosol photometer sensitive to 0.3 micron particles.

Droplet nuclei. Airborne particles that originate as droplets, but have dried and left behind mucous, salt, microorganisms, and other material that may have been associated with the droplet. Because of their small size, they frequently remain suspended in the air for long periods of time.

Dry steam. Steam containing no water in suspension.

Dust preventive clothing. This clothing is specially designed for wear by all personnel who enter a clean room. Special particle/lint-free clothing such as coveralls, caps, gloves, finger cots, boots and shoes are primarily manufactured from synthetic fabrics and materials. Their purpose is to minimize contamination of clean rooms by particulate matter from an individual's skin, hair, clothing, and shoes such as dust, lint, dandruff, skin flakes, etc.

E.

Electrostatic. Pertaining to the phenomena due to attractions and repulsions of electrical charges.

Electrostriction. The use of an electric charge to change the dimensions in certain crystals; the principle used in some ultrasonic transducers.

Emission factor. A statistical average of the rate at which contaminants are emitted from any given source.

Emulsification. A mixture of liquids that do not dissolve in each other; colloidal mixture. In an emulsion, one of the liquids contain minute droplets of the other, which are evenly distributed throughout.

Emulsion. A liquid in which other particles or liquids are suspended; a characteristic of some cleaning agents in holding and carrying away soils.

Entrapment. The act of securing and holding, as dirt is entrapped in a filter.

Environment. The total of all factors which might influence or cause contamination of a contaminee. The primary factors of environment are the forms of air, gas, liquid, solid, or surfaces.

Environmental contamination. Contamination that enters the system through breathers and seals.

Environmental control. Environmental control is the positive control of atmospheric conditions within a specific area. It is a collective term to identify the control of all factors of the environment of the clean room or clean work area. These factors include air temperature, humidity, air-borne particle control, pressurization, illumination, and injurious vapor control.

Environmental control system. This system maintains suit and cabin environment, provides thermal control for electronic equipment and is used to service the portable life support system.

Environmental design standards. Those values which are mandatory in the design of an environmentally controlled area or device.

Environmental operational standards. Those standards which are mandatory in the operation of an environmentally controlled area or device.

Environmentally adjacent. A condition in which packaging materials are not in direct contact with clean or significant surfaces but are exposed to these surfaces.

Environmental package. A clean material closure, wrap, or container over the intimate package that is sealed to provide an additional contamination barrier to protect the intimate package and its contents from environmental elements.

Environmental test chamber. A vessel in which space environments and atmospheric conditions can be simulated.

F.

Face velocity. A measure of lineal airflow at approximately 12 inches from the down-stream face of a filter.

Facultative. Ability to live or thrive under more than one set of conditions, i.e., a microorganism that can live in the presence or absence of oxygen.

Fiber. A particle whose length is at least ten times its width.

Filter. A device using a medium to selectively separate certain substances from a given environment.

Filtered tap water. Tap water which is filtered to meet the same total solids and filtration requirements as demineralized water.

Filter rating. The capability of a media to restrict passage of matter as established by testing techniques such as pore size, bubble point, and DOP. Filter ratings for fluid filters include both nominal and absolute ratings. The absolute rating represents the particulate size that the filter will retain with 100 percent efficiency. The nominal rating represents the particulate size retained at a minimum efficiency of 80 percent.

Filtration. The process of removing contaminants from a gas or liquid by passing them through a porous media.

First air. The air which issues directly from the HEPA filter.

First work location. The work location that is first in the path of the airstream.

Fixture. A device for holding a work piece in a fixed position while fabrication and assembly processes are performed.

Fluid contaminant. Liquid, gaseous, or particulate matter suspended or dissolved in a fluid.

Flush. Rinsing a component, system, etc., using a liquid as the rinsing medium.

Flux. A substance, as borax or rosin, used to help metals fuse together (as in soldering).

Free radical. A chemical species that has an unpaired electron; species having an odd number of electrons.

Frequency. The number of vibrations or cycles per second.

Fume hood. A work station that has a specially provided exhaust system for control of vapor contamination from volatile liquids; usually provided with a sink and disposal facility.

G.

Garments. Coveralls, coats, and smocks.

Gas. A state of matter in which the molecules are practically unrestricted by cohesive forces. A gas has neither shape nor volume, and in form is neither liquid nor solid.

Gas cylinder safety relief device. A device installed in a gas cylinder or container to prevent rupture of a cylinder by overpressures as a result of certain conditions of exposure. Such a device includes a frangible disc, fusible plug, or relief valve.

Gas pressure regulator. A device that may be connected to the cylinder valve outlet to regulate the gas pressure delivered to a system.

Gas supply system inspection. The periodic testing which is made to determine that excessive contamination has not been introduced into a previously qualified gas supply system.

Gas supply system qualification. A series of tests which are performed to demonstrate that the gas distribution system is capable of meeting the established requirements and to assure delivery of gases within specified cleanliness limits to the gas utilization points.

Generated contamination. That which is generated within the system components as a result of such actions as wear, cavitation, and erosion.

Genus. A group marked by common characteristics or by one common characteristic; a category of biological classification between the family and the species.

Germ. A microorganism, usually a bacterium, which can produce disease.

Germicide. A substance capable of killing germs.

GOX. Gaseous oxygen.

Gravimetric level. The quantity of contamination by weight per standard specification.

Gross cleaning. Preliminary or rough cleaning to remove scale, rust, metal chips, shop dirt, etc.; this cleaning is done in a normal work area to visual inspection standards.

Gross contaminants. Large amount of corrosion products, metal chips, scale, rust, weld, slag, or particulate matter present on a component or in a system.

Gas measurement (standard cubic foot of gas, scf). Amount of gas occupying one cubic foot of space at one standard atmosphere pressure (760 mm-Hg) at zero degrees.

H.

Hertz (hz). A unit of frequency in the centimeter-gram-second system, equivalent to one cycle per second.

Heterotroph. Microorganisms that require preformed organic compounds for their carbon source.

High efficiency particulate air filter (HEPA). Mil-T-51068A specifies filters with minimum efficiency of 99.97 percent determine by the homogeneous DOP method at airflows of 20 and 100 percent of the rated flow capacity of the filter. It is referred to as the HEPA filter.

Horizontal laminar airflow clean room. A room equipped with one entire vertical wall of HEPA filters through which the air passes at a predetermined speed to an exhaust wall directly opposite the HEPA filter wall. The entire body of air moves horizontally across the room along essentially parallel flow lines at uniform velocity.

Hydrocarbon. A chemically identifiable compound of carbon and hydrogen.

Hydrophilic soil. Soil which exhibits an affinity for water or is miscible with water.

Hydrophobic soil. Soil which exhibits a lack of affinity for or miscibility with water; nonwetting.

Hygroscopic. The ability to readily absorb and retain moisture.

Hyphae. The "threads" that make up the mycelium (filamentous structures) of a fungus.

I.

Implosion. A bursting inward; the opposite of an explosion. When the cavitations in an energized solution collapse, implosion occurs.

Incrustation. Severe scaling.

Indirect test. Any surface cleanliness test in which the parameter measured is the amount of soil removed from a surface. (See direct test.)

Intermediate packaging. The application or use of a closure, wrap, covering, or container over the clean package to provide general physical protection to preserve the integrity of the clean package.

Internal surfaces. Internal surfaces are defined as those surfaces of assemblies, subsystems, and ground support equipment that contact service medium (gases) during use.

Intimate cushioning. A clean material used to protect additional packaging materials from puncture or damage caused by the clean item.

Intimate package. A clean material closure, wrap, or container that will have intimate contact with or is environmentally adjacent to the clean item or surface and is sealed to provide a barrier to external contaminants and environments.

Iodophore. Organic compounds of iodine in which iodine is loosely combined with some surface active agent; nonstaining, nonirritating, and practically odorless.

K.

Kauri-butanol value. A numeric designation indicating the solvency power of a cleaning agent. The kauri-butanol value for benzene is 100.

Kilocycle (kc). 1000 cycles.

Kilohertz (kHz). 1000 Hertz or 1000 cycles per second.

L.

Laminar airflow. Airflow in which the entire body of air within a confined area moves with uniform velocity along parallel flow lines.

Laminar airflow clean work station. A work station in which the laminar airflow characteristics predominate throughout the entire air space.

Laminar airflow room. A room in which the laminar airflow characteristics predominate throughout the entire air space.

Latent heat. The calories of heat required to change one gram of liquid to vapor at the boiling point.

Lethal. An agent capable of causing death.

Light scattering. A technique for detecting, counting, and sizing fluid-borne particulate matter passing through a high intensity light beam; the distorted light beams are converted to electrical impulses by a photomultiplier tube and registered on appropriate counters and tapes.

Limited linting. The term limited linting applies to towels, garments, and other fabric materials which have been tested and proven to exhibit limited shedding or linting characteristics.

Liquid. A state of matter in which the molecules are relatively free to change their positions with respect to each other but restricted by cohesive forces so as to maintain a relatively fixed volume.

Localized clean operations. Operations conducted under locally maintained environment provided by tenting and conditioned air source.

LOX. Liquid oxygen.

M.

Magnetostriction. Applying an alternating magnetic field in the direction of a metal rod's axis to change its length. This is the principle used in the lower cycle(20 to 25 khz) ultrasonic transducers.

Materials cleaning room. A materials cleaning room is a room immediately adjacent to the clean room, equipped with vacuum lines, ultrasonic cleaners, and other mechanical devices for cleaning parts, tools, and materials immediately prior to their entry into the clean room.

Maximum allowable concentration (MAC). Now referred to as TLV.

Maximum bubble point. The initial bubble point of a filter that successfully passes the absolute filter rating requirements using the test method of ARP 901.

Mechanism. The physical or chemical means or process by which a natural action, reaction, or result is achieved.

Membrane filter. Cellulose plastic porous membrane material with controlled pore sizes ranging from 5 microns downward to approximately 8 millimicrons, composed of 15 to 20 percent solid material and 80 to 85 percent void.

Mesh. The number of openings per linear inch of sieve surface. A 50 mesh sieve has 50 openings per linear inch, each 300 microns across.

Metabolism. The total chemical changes by which the nutritional and functional activities of an organism are maintained. (Subdivisions: anabolism - systhesis; catabolism - degradation.)

Mg. Milligram.

mg/l. Milligrams per liter.

Microbe. An organism of microscopic or submicroscopic size, generally including viruses, rickettsiae, bacteria, algae, yeasts, and molds.

Micron. A unit of measurement equal to one-millionth of a meter or approximately 0.001 inch).

Microorganism. An organism of microscopic size, limited to bacteria, fungi, including molds, yeast, algae, and protozoa.

Migration. Act of changing locations; moving from place to place, as fine dust is moved by air currents or agitation.

ml. Milileter.

Molecular attraction. The adhesive forces exerted between the surface molecules of two bodies in contact.

Monitor. To systematically check or test for the purpose of controlling the concentration of a specific ingredient or the execution of a process. May include qualitative or quantitative measurements, or both.

Moisture-resistant material. A material which will not readily absorb moisture when subjected to conditions of high humidity for extended periods of time.

N.

Nonaqueous. Derived basically from petroleum, coal, and wood (organic base). Possesses the ability to dissolve oils, greases, many resins, rubber, bitumens, paraffin, waxes, and plastics.

Noncritical surface. A surface of a fluid system which does not directly contact the system fluid.

Nonlaminar flow clean room. A room characterized by nonuniform airflow patterns and velocities.

Nonlaminar flow clean work station. A work station characterized by non-uniform air patterns and velocities.

Nonvolatile residue (NVR). Soluble or suspended material and insoluble particulate matter remaining after controlled evaporation of a filtered volatile liquid, usually measured in grams; filtration is normally through a 0.45 or 0.8 micron membrane filter.

Nylon. A polyamide with high abrasion resistance, but relatively poor moisture barrier characteristics.

O.

Operational guidelines. Operational guidelines provide information to be considered in the operation of an environmentally controlled area or device. They are not mandatory except as noted. Operational guidelines reflect the current state of the art at the time.

Operational fluid. Fluid used within a fluid system or fluid subsystem as the operational media.

Operational periods. That period of time when an environmental test chamber is classified as a controlled work area. Such periods will normally commence with cleaning and inspection or inspection to the requirements operational readiness and continue to completion of test.

Operational readiness. All systems are prepared and test may proceed.

Organic. Designating any chemical compound containing carbon.

Orifice. A fixed restriction in a fluid passage which established the rate of fluid flow.

Oxide. A binary compound of oxygen with some other element or with a radical.

Oxidizer. A substance that supports the combustion reaction of a fuel.

P.

Packaging. The application or use of appropriate closures, wrappings, cushioning, containers, and complete identification, up to, but not including the shipping container and associated packing.

Packaging film. Any film or sheet material used as a packaging wrap or container; usually limited to thicknesses less than 10 mils.

Packing. The application or use of exterior shipping containers with the packages therein, together with necessary blocking, bracing, cushioning, weather proofing, and exterior strapping of the shipping container to protect and preserve the integrity of the packaged item during shipment or extended storage.

Part. A part is defined as one piece or two or more pieces joined together which are not normally subject to disassembly without destruction of designed use.

Particle. A piece of matter with observable length, width, and thickness; usually measured in microns. A particle is measured by its greatest dimension.

Particle counters. Automatic electronic devices designed to electronically separate, size, and count individual particles.

Particle size. Particle size is expressed as the apparent maximum linear dimension or diameter of the particle. The linear dimension is implied unless otherwise specified.

Particle size distribution. The cumulative frequency of occurrence of each contaminant particle size in a fluid.

Particulate matter. The general term applied to matter of miniature size, with observable length, width, and thickness and contrasted to nonparticulate matter without definite dimension. Particles and fibers less than 10,000 microns in length.

Personnel cleaning chamber. A chamber equipped with shoe cleaning devices, vacuum lines, and other devices to remove dirt, dust, and lint from clothing prior to entrance into clean rooms.

pH. Potential of Hydrogen; a symbol for the logarithm of the reciprocal of the hydrogen ion concentrations, expressed in gram atoms per liter of a solution; used to indicate acidity or alkalinity. The pH scale indicates for a value above 7, alkalinity; for 7, neutrality; for a value below 7, acidity.

Pickling. Process in which strong hydrofluoric muriatic, or nitric acid, or both is used to remove scale and rust or oxides from metals.

Plenum. An enclosed space in which the air pressure is greater than that of the adjoining outside area.

Polyethylene. A thermoplastic polymer capable of being produced in thin sheets; exhibits good moisture-vapor barrier qualities but has a high sloughing tendancy.

Precision cleaning. Final or fine cleaning accomplished in a controlled environment to remove minute quantities of contaminants to better than visual standards.

Precision packaging. Packaging or protection to preserve precision cleanliness for a specified period and condition.

Precleaning. That cleaning which is accomplished outside of a clean area for the purpose of removing contaminants such as rust, oxidation, grease, oil, heavy scale, or soil deposits to control the amount of contaminants brought into the clean room.

Pressure drop. The pressure differential resulting from forcing a gas or liquid through a restricting media.

Projected diameter. The diameter of a circle having the same area as the projected image of the particle when viewed through a microscope in the direction perpendicular to the plane of greatest stability.

Purge. To flow an inert gas or system media through a system (or line, tank, etc.) for the purpose of ridding the system of a residual fluid or for providing a positive flow of gas from some opening in the system.

Q.

Quality control. A process or test oriented operation for obtaining or manufacturing a uniform product within specified limits.

R.

Random flow clean room. Air enters the room through diffusers located on or near the ceiling and is exhausted through openings near the floor; air within this type room follows a random pattern.

Range. The difference between the maximum and the minimum of a set of various values.

Reagent. A substance used to produce a characteristic reaction in chemical analysis.

RH. Relative humidity.

Rinse test. A test to determine cleanliness by entrainment or by solution of soluble materials with a suitable rinsing liquid; the liquid is sloshed or agitated over the critical surfaces of the component to ensure entrainment of particles.

Rough cleaning. Pre-cleaning.

S.

Saponification. The process whereby the fatty acids in animal and vegetable oils unite chemically with certain alkaline substances to form soap.

SCFM. Standard cubic feet per minute.

Significant surface. The surface of a part, component, or system which contacts the operating medium of a system and requires a specified level of cleanliness which must be maintained for proper operation.

Silt. Particulate matter settled from fluid generally in particle size ranging less than 5.0 microns.

Silting. An accumulation of very minute particles which are usually not counted. These particles, if in sufficient quantities, can cause a haze. This haze can cause partial or complete obscuring of grid lines on test filter membrane when viewed visually or under 40 - power (maximum) magnification.

Size dimension. A specified value of a diameter, width, length, or other geometrical characteristic directly related to the size of an object.

Slough. To release particles of the base material as a result of erosion or abrasion.

Smut. The accumulation of noticeable amounts of nonadherent reaction products on chemically treated metal surfaces, usually resulting from cleaning or etching.

Solid. A state of matter in which the relative motion of molecules is restricted; the molecules tend to retain a fixed position relative to each other.

Solvent. That solution or constituent of a solution which exhibits the capability to dissolve other substances through chemical action.

Sonic. Sound wave energy within range of human hearing as applicable to contamination control . (See Cavitation.) It is used as a part of an overall cleaning process or degreasing operation.

Sonochemistry. Acceleration of chemical effects by the use of ultrasonics.

Species. One kind of organism; the subdivision of a genus.

Specification. A detailed description of the characteristics of a product and of the criteria which must be used to determine whether the product is in conformity with the description.

Specific heat. The thermal capacity of a liquid as a ratio of that of water at 15° C.

Standard. A uniform method of defining basic parameters for processes, product, or measurements.

Standard blocking. Blocking which occurs when contaminating particles in fluid streamlines adjacent to the walls of a clearance adhere to the walls.

Substrate. Substances which undergo chemical reactions due to the catalytic action of enzymes.

Surface cleanliness tests. Visual; tissue paper on white cloth; water break; automizer; contact angle; radio tracer; evaporative rate analysis; indium adhesion; ellipsometry.

Surface tension. A phenomenon of molecular attraction between the molecules of a liquid which tends to contract the exposed surface to the smallest possible area; expressed as dynes per centimeter.

T.

Tapwater. Water from a domestic system.

Test. Examination, investigation, and evaluation of inherent properties, functionability, environmental reaction, variances, and reliability of any product, system, sub-system, vehicle, equipment assembly, part, material, and process.

Test data. Results of a test in the form of numbers, charts, tapes, etc.

Test fluid. A measured volume of fluid which is evaluated by analytical methods to determine contaminant content; also applicable to fluids used for other test purposes.

TLV. Threshold limit value; a guideline between safe and probably dangerous concentrations having adverse effects on personnel as recommended by the American Conference of Governmental Industrial Hygienists. Formerly referred to as (MAC); is generally expressed as parts per million (ppm) by volume or milligrams per cubic meter (mg/m^3) of air contaminants and represents concentration to which personnel may be exposed 8 hours per day for normal working lifetime without adverse health effects.

Total filterable solids. Filterable residue or particulate matter in a liquid or gaseous medium which is retained on a filter or sieve when the medium is passed through a filter or sieve. Total insoluble particulate matter smaller than a specified particle size.

Total solids. The residue from a known volume of liquid which has been evaporated to dryness in an oven.

Toxic. Poisonous.

Transducer. A device that converts energy from one form to another. An ultrasonic transducer changes high-frequency electrical energy into high-frequency sound waves.

Tunnel-flow clean room. A room where the incoming air enters through an entire wall of filters and is exhausted through an opposite open area. Air travel within the enclosure is basically laminar.

U.

Ubiquitous. Present everywhere or in many places.

Ultrasonics. The physical science of those acoustic waves that oscillate in the approximate range of 18 to 80 kHz, beyond range of human hearing. For use in contamination control see Cavitation. Ultrasonic cleaning is not a complete and separate method. It is used as a part of an overall cleaning process or degreasing operation.

V.

Vapor. The gaseous form of any substance which is usually a liquid or a solid.

Vapor degreasing. The process of cleaning metals with non-flammable solvents.

Vertical laminar airflow room. A room equipped with a ceiling of HEPA filters with a grated or perforated floor for the exhausting of air issuing from the ceiling filters; the airflow is vertical and moves within the walled area along essentially parallel lines at uniform velocity.

Viable. Capable of living; growing, or developing; metabolizing.

Viscous. Plastic coating materials.

Visibly clean. Freedom from surface particulate matter approximately 50 microns or larger and from all films other than known innocuous films.

Visual cleanliness. The degree of freedom from contaminants that may be detected by the unaided eye; special lighting effects, ultraviolet light, wipe test, water break test, and similar means may be used as techniques to determine visual cleanliness.

W.

Water break check. One means of quickly checking effectiveness of the alkaline detergent cleaning process. After the part has undergone the rinse following any alkaline detergent cleaning, the water should remain in a clean sheet-like manner over the observed outer surface for at least 30 seconds to 1 minute after the part has been removed from the rinse. As an indication of apparent absence of hydrocarbons on the surface, there should be no bubbles, understreaking, or obvious breaks in the watery covering during the observation period.

Wave length. The distance between two undulations of a wave train.

Wetting. Wetting action brings the detergent or cleaner in closer contact with surface and oil. (Water has less wetting power than ammonia, acetone, benzene, and most oils. Waters wetting power can be greatly increased by chemical treatments.)

White room. A room designed to be free of dust and other contaminants but not controlled to the same level as a clean room.

Work station. A work bench or similar working enclosure that has its own filtered air supply.

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Section 3

Abbreviations

3.1 Abbreviations defined.

1. A.C.S. American Chemical Society.
2. ADCAD. Auto-radiographic detection of contamination by adsorption/desorption.
3. amp/ft^2. Amperes per square feet.
4. ARP. Aerospace recommended practice.
5. asf. Amperes per square feet.
6. ASTM. American Society for Testing and Materials.
7. Be'. Baume'.
8. Btu/lb. British thermal unit per pound.
9. °C. Degrees Centigrade.
10. CEF. Controlled environmental facility.
11. cf. Cubic feet.
12. cfm. Cubic feet per minute.
13. cm. Centimeter.
14. c.p. Chemically pure.
15. DC. Direct current.
16. DOP Test. Dioctyl phthalate test.
17. DOT. Department of Transportation.
18. dynes/cm. Dynes per centimeter.
19. emf. Electromotive force.
20. °F. Degrees fahrenheit.

21. FCC. Federal Communication Commission.

22. Fed -Spec. Federal specification.

23. fpm. Feet per minute.

24. g. Gram.

25. GOX. Gaseous oxygen.

26. HEPA. High efficiency particulate air filter.

27. HLF. Horizontal laminar airflow.

28. Hz. Hertz.

29. kc. Kilocycle.

30. kHz. Kilohertz.

31. KSC. Kennedy Space Flight Center.

32. kw. Kilowatt.

33. LOX. Liquid oxygen.

34. MAC. Maximum allowable concentration. Now referred to as TLV.

35. mg. Milligram.

36. mg/l. Milligram per liter.

37. Mil-Hbk. Military handbook

38. Mil-Spec. Military Specification.

39. mm. Millimeter.

40. MPN. Most probable number.

41. MSFC. Marshall Space Flight Center.

42. ohms/cm. Ohms per centimeter.

43. NASA. National Aeronautics and Space Administration.

44. NLA. Nonlaminar airflow.

45. NVR. Nonvolatile residue.

46. ohms/cm. Ohms per centimeter.
47. pH. Potential of Hydrogen.
48. ppm. Parts per million.
49. PR cleaning. Periodic reverse cleaning.
50. psi. Pounds per square inch.
51. psig. Pounds per square inch gauge.
52. PVC. Polyvinal chloride.
53. QA. Quality assurance.
54. QC. Quality control.
55. RH. Relative humidity.
56. R.M.S. Root-mean-square.
57. SAC. Strategic Air Command.
58. TLV. Threshold limit value.
59. µ. Micron.
60. mho/cm. Microhos per centimeter.
61. v. Volts.
62. VLF. Vertical laminar airflow.
63. V-W-T-R. Vapor water transmission rate.
64. WG. Water gage.
65. W/in^2. Watts per square inch.
66. wt. Weight.

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SECTION 4

HISTORY OF CONTAMINATION CONTROL TECHNOLOGY

4.1 First application. Although the first application of the most basic considerations for contamination control can be traced back to 1865 when Sir Joseph Lister realized and scientifically demonstrated the effect of antiseptics in reducing operative deaths, credit for refinement and addition of new principles in acquiring very high levels of contamination control must go to recent industrial efforts.

4.1.1 Wartime influence. The establishment of clean room policies became a necessity with the advent of World War II and the ensuing mechanical wartime innovations. Two of the wartime implements requiring high levels of cleanliness were the Norden bombsight and the first navigational gyroscopes. Particles in the range of 20 to 50 microns would cause the bombsight and the gyroscope to malfunction. Further degrees of refinement in equipment created the need for greater control of ambient conditions. In 1956 the government acquired an inertial guidance system containing two floating gyros. The suspension system was highly dependent on contaminant free fluid. It was observed that contamination one-half the size of cigarette smoke particles could compromise the system, indicating the degree of control necessary in the development of this system.

4.1.2 Earliest clean rooms. The earliest clean rooms in the United States were Air Force facilities. At one time, Olmstead Air Force Base, in Pennsylvania maintained the largest clean room facility. This Air Force base was deactivated and the clean room operation moved to Wright-Patterson Air Force Base in Ohio. The Strategic Air Command (SAC) has several clean room units that are used for photographic reproduction. North Island Naval Air Station in California houses a Naval clean room facility. Other institutions that have been influential in the development of clean room facilities are the National Bureau of Standards, the U. S. Public Health Service and the Atomic Energy Commission in collaboration with the Sandia Corporation in Sandia, New Mexico. Because of the highly sophisticated equipment used by the National Aeronautics and Space Administration (NASA), the space industry has been one of the primary users of clean room technology.

4.1.3 Advancement by industry. Major developments in the area of industrial contamination control, in specific air filtration techniques, occurred during the late 1950's and early 1960's. The improved degree of technique development occurred as a result of manufacturing advances and development of extremely sophisticated electronic, electro-mechanical, electro-optical, and hydraulic equipment. These devices demanded cleanliness levels to be such that the presence of microscopic particles could result in malfunction or failure to function of an entire system, device, or mission.

4.1.4 Implications of technological advancements. As technological advancements ensued, intricate machinery requiring higher degrees of cleanliness put further demands on contamination control. The transition to transistors and integrated circuits, which required hundreds of circuits placed on a single chip, developed the need for higher levels of cleanliness control than had been previously needed. Development of photographic techniques have also created a need for precision controlled environments. Manufacturing and processing of photographic film has long required a dust free environment; however, new methods of photographic enlargements have required an even more stringent environmental control. Dust particles 5 to 10 microns in size can cause spotting when film is enlarged.

4.2 Cleanliness standards. The development of a cleanliness standard was the direct outcome of the various cleanliness levels required by industry. This standard established classes of clean rooms that were defined by the number of particles within a given area.

4.2.1 Technical Order 00-25-203. The first written standards for clean room facilities were established by the help of the government. In 1961 the Technical Order 00-25-203, "Standard Functional Criteria for the Design and Operation of Clean Rooms and Clean Work Stations" was published. This report was redefined and reissued in July 1963.

4.2.2 Federal Standard 209. In April of 1963, the Atomic Energy Commission held a meeting in Sandia, New Mexico, for the purpose of reviewing the existing clean room standards. As a result of this conference, a committee was formed to write a standard for clean room operations. The Federal Standard 209, the present basis for clean room facilities, was the product of the committee and was published by the end of 1963. This standard defined and established three classes of clean rooms; Class 100,000, Class 10,000, and Class 100. Class 100,000 corresponds to the clean room defined in Technical Order 00-25-203 previously mentioned. Federal Standard 209 was revised in 1966. The revised edition, Federal Standard 209a, became effective on August 16, 1966.

4.2.2.1 Air cleanliness classes. In general, precision mechanical devices which have clearances in a range from 100 to 1000 millionths inch are affected by particle sizes from 2.5 to 25 microns. These items will require contamination control both during manufacture and overhauls. Clearances less than 100 millionths inch (2.5 μ) will require the use of higher quality clean rooms (probably Class 100) or a clean work station capable of Class 100 control. The need for several levels of clean work areas has been recognized, and Federal Standard 209 established three classes of air cleanliness. The levels of these classes are based on statistical distribution for average particle sizes. They depend on particle concentrations with a maximum allowable number of particles per unit volume of air of 0.5 micron and larger, or 5.0 microns and larger. For statistical purposes, the particle counts are taken during normal work activity periods and at locations where the air approaches the work area. The clean room classes defined in Federal Standard 209a are:

Table I.

Federal Standard 209a - Air cleanliness classes

Maximum number of particles per cubic foot 0.5 micron and larger (per liter)*	Class English System (metric system)*	Maximum number of particles per cubic foot 5.0 microns and larger (per liter)*
100 (3.5)*	100 (3.5)*	Counts below 10 (0.35) particles per cubic foot (liter) are unreliable except when a large number of samplings is taken.
10,000 (350)*	10,000 (350)*	65 (2.3)*
100,000 (3500)*	100,000 (3500)*	700 (25)*

*Metric System

a) Class 100 (3.5 liter). Particle count not to exceed a total of 100 particles per cubic foot. (3.5 particles per liter) of a size 0.5 micron and larger.

b) Class 10,000 (350 liter). Particle count not to exceed a total of 10,000 particles per cubic foot (350 particles per liter) of a size 0.5 micron and larger of 65 particles per cubic foot (2.3 particles per liter) of a size 5.0 microns and larger.

c) Class 100,000 (3500 liter). Particle count not to exceed a total of 100,000 particles per cubic foot (3500 particles per liter) of a size 0.5 micron and larger or 700 particles per cubic foot (25 particles per liter) of a size 5.0 microns and larger.

4.2.3 Mil-Std-1246A. In 1962 a military standard was developed - Mil-Std-1246(MI); on 18 August 1967 this standard became Mil-Std-1246A. The purpose of the document was to establish requirements and guidelines to enable the achievement of the degree of cleanliness essential to meet product reliability or quality specifications. The following Table shows the classification of cleanliness levels as established by Mil-Std 1246A.

Table II.

Mil-Std-1246A - Classification of cleanliness levels

Cleanliness level **	Range Particle Size-Micron		Quantity of Particulates	*Non Volatile Residue	
	Surfaces, Components, parts, Assemblies	Fluids (Liquids and gases)		Residue Level	Quantity NVR
10	5-10	Same as Surfaces	Less than 2	A	Less than 1.0 mg
	Greater than 10		Less than 1		
25	5-10	Same Surfaces	12	B	1.0 mg to 2.0 mg
	10-25		3		
	Greater than 25		Less than 1		
50	5-15	Same as Surfaces	60	C	2.0 mg to 3.0 mg
	15-25		12		
	25-50		3		
	Greater than 50		Less than 1		
100	15 and smaller	5 and smaller	No limit	D	3.0 mg to 4.0 mg
	-	5-15	600		
	15-25	15-25	150		
	25-50	Same as Surfaces	30		
	50-100		3		
	Greater than 100		Less than 1		
200	25 and smaller	5 and smaller	No limit	E	4.0 mg to 5.0 mg
	-	5-15	10,000		
	-	15-25	2,000		
	25-50	25-50	500		
	50-100	Same as Surfaces	40		
	100-200		3		
	Greater than 200		Less than 1		
300	25 and smaller	5 and smaller	No limit	F	5.0 mg to 7.0 mg
	-	5-15	60,000		
	-	15-25	12,000		
	-	25-50	3,000		
	50-100	50-100	250		
	100-200	Same as Surfaces	20		
	200-300		2		
	Greater than 300		Less than 1		

Clean-liness level **	Range Particle Size-Micron		Quantity of Particu-lates	* Non Volatile Residue	
	Surfaces, Compo-nents, parts, Assemblies	Fluids (Liquids and gases)		Residue Level	Quantity NVR
500	50 and smaller	15 and smaller	No limit	G	7.0 mg to 10.0 mg
	-	15-25	150,000		
	-	25-50	30,000		
	50-100	50-100	2,500		
	100-200	Same as Surfaces	200		
	200-300		25		
	300-500		2		
	Greater than 500		Less than 1		
750	100 and smaller	50 and smaller	No limit	H	10.0 mg to 15.0 mg
	-	50-100	15,000		
	100-200	100-200	1,500		
	200-300	Same as Surfaces	150		
	300-500		15		
	500-750		3		
	Greater than 750		Less than 1		
1000	100 and smaller	50 and smaller	No limit	J	15.0 mg to 25.0 mg
	-	50-100	125,000		
	100-200	100-200	0,000		
	200-300	Same as Surfaces	1,000		
	300-500		100		
	500-750		20		
	750-1000		2		
	Greater than 1000		Less than 1		

*Note: NVR (Non Volatile Residue) applies only if specified. NVR level is designated by letter following the particulate cleanliness level designation. Example: cleanliness level 100 refers to particulates only, at level 100; cleanliness level 100E refers to particulate level 100 and NVR level E.

**Note: The quantity (number) of the smallest particulates of significance (or interest) determines the cleanliness level.

Where the quantity of larger particulates (larger than those determining the level) must be less than the quantity specified in this table, then the cleanliness level shall be defined in a manner similar to the following example: cleanliness level 100 except 50 to 100 not over 1.

Two levels may be used when necessary to designate particles and fibres separately. Example: cleanliness level 100 particles, 1000 fibres.

4.2.4 NAS 1638. In 1964, the Aerospace Industries Association of America introduced the NAS 1638 cleanliness requirements of parts used in hydraulic systems.

4.3 References.

1. Clean Room and Work Station Requirements, Controlled Environment, Federal Standard No. 209a, August 16, 1966.

2. Clean Room Technology, James W. Useller, Lewis Research Center, NASA SP-5074, 1969.

3. Contamination Control Principles, Sandia Corporation, NASA SP-5045, 1967.

4. Grumman Aerospace Corporation.

5. Hospital Sanitation - Past, Present, and Future, Contamination Control, Jon W. Leemhorst, Vol. IX No. 8 & 10, Sept. - Oct, 1970.

6. Department of the Navy, Naval Weapons Center, China Lake, California.

7. Particle Size Measurement, ASTM Special Tech. Publ. No. 234, Philadelphia, Pa., 1958.

8. Product Cleanliness Levels and Contamination Control Program, Military Standard 1246A, August 18, 1967.

9. Technical Order Standards and Guidelines for the Design and Operation of Clean Rooms and Clean Work Stations, T.O. 00-25-203.

10. The Starting Point for Contamination Control, Whitby, K. T., Am. Assoc. Cont. Control, Boston, May, 1963.

SECTION 5

CONTAMINANTS DEFINED

5.1 General definition. By definition, to contaminate is to make impure, unclean, or corrupt by contact; to pollute.

5.1.1 Pertaining to contamination control technology. Relative to the art of contamination control, a contaminant is regarded as "Any material, substance, or energy that is unwanted or adversely affects the contaminee."

5.1.1.1 Categories of contamination. Contamination may be classified in four general categories:

(a) Particulate: dust, chips, fibers, etc.

(b) Chemical: gases, liquids, oily films, etc.

(c) Biological: bacteria, virus, fungi, spores, etc.

(d) Energy or changes of state: light, magnetic fields, radiation, heat, etc.

5.1.2 Earth atmosphere. The entire earth atmosphere is composed of suspended particles. These particles may be either natural or man-made. Natural particulate matter are of two classifications: (1) organic, (2) inorganic. Organic matter is that which is derived from living things and inorganic matter is that which is derived from inanimate materials.

5.1.3 Everyday environmental contamination. Generally speaking, the extent of everyday environmental contamination varies with location and season. Rural areas, under normal atmospheric conditions, will contain up to 40,000 particles per cubic foot. In metropolitan areas the contamination count is many times greater. Counts up to 1.5 million particles per cubic foot (larger than 0.5 μ) are normal. The general industrial atmosphere has concentrations up to one million particles above 1.0 micron with a normal range of distribution up to 600 microns.

5.1.4 Contaminant sizes. Contaminants may be so large in size that they are readily visible; however, the reverse is also true. In some instances, contaminants are sub-microscopic in size defying identification by present day methods. Frequently, the presence of contaminants is indirectly discovered when a product malfunctions. A frequent product impairment is increased electrical contact resistance of components contaminated by an invisible film.

5.1.5 Sources and forms. Very frequently, contaminants are labeled such because of their location. For instance, light is usually desirable; however, in the film developing process it is a contaminant. A great number of materials are regarded as contaminants in specific cases. (See Table III.) Contaminants noted in the various forms are normally found in that form but may be found in a minor condition in other forms.

Table III.

Contaminant sources and forms

Sources and Related Contaminants	Contaminant Form or State					
	Particulate	Gaseous	Liquid	Film	Radiation	Microbial
Atmosphere:						
Condensates			x	x		
Airborne particulate	x					x
Gas		x				
Clothing:						
Lint and fibers	x					
Industrial Processes:						
Chemical films				x		
Chemical vapors		x		x		
Chips and burrs	x					
Combustion gases		x				
Cleaning solvents			x			
Coolants and oils			x			
Flue dust	x					
Flux particles	x					
Flux vapors		x	x	x		
Lubricants			x	x		
Machining: heat, sound, vibration					x	
Oxides				x		
Plating baths			x	x		
Sand, etc.	x					
Smoke fumes	x	x				
Solder/weld splatter	x					
Soldering, heat					x	
Solvent residue				x		
Sublimated flux		x				
Welding: light, heat					x	

(continued)

Sources and Related Contaminants	Contaminant Form or State					
	Particulate	Gaseous	Liquid	Film	Radiation	Microbial
People:						
Bacteria and virus	x					x
Body vapors		x				
Cosmetics	x					
Epidermal scale	x					x
Hair	x					
Skin Oils			x			x
Rickettsiae	x					x
Tobacco smoke	x					
Plants:						
Fungi	x					x
Pollen	x					x
Product:						
Corrosion	x	x				
Material shedding	x					
Outgassing		x		x		
Sublimated materials from flux, plastics, etc.		x		x		
Wear particles	x					
Radioactive Materials:						
Alpha particles	x				x	
Beta particles	x				x	
Electrons	x				x	
Gamma rays					x	
Neutrons	x				x	
Soil:						
Bacteria	x					x
Fungi	x					x
Protozoa	x					x
Sun:						
Electromagnetic					x	
Ultraviolet					x	
Visible light					x	
X-ray					x	
Transportation:						
Exhaust fumes	x	x				
Wear particles: tires, wheels, paint, etc.	x					
Wear particles: road, rail	x					

(continued)

Sources and Related Contaminants	Particulate	Gaseous	Liquid	Film	Radiation	Microbial
Construction:						
Dust	x					
Fumes	x	x				
Sawdust	x					

5.2 Origin of contaminants. Contamination is the product of activity; activity is the result of expended energy and may be mechanical, chemical, electrical, thermal, or radiological in nature. Every activity of man generates or contributes to the generation of contamination. In the U.S. it is estimated that man, either directly or indirectly, unleashes approximately 50 million tons of particles in the atmosphere yearly.

5.2.1 Control of atmospheric contamination. Attempts are presently being made to try to control the man-made generation of airborne particles which are in most cases contaminants in the atmosphere. Industrial attempts encompass the use of devices on automobiles, the use of centrifugal collectors, electrostatic precipitators, incinerators, and charcoal filters.

5.2.2 Control of clean room contamination. Within the closed environment of clean rooms, the method most generally applied to control airborne particles is non-woven filters. In some cases charcoal filters, gas scrubbers, and incinerators are used in conjunction with non-woven filters.

Approximately 80 per cent of airborne particulate matter present in a controlled clean room situation is introduced by personnel. The particles are either shed from the bodies or clothing of the individuals. The amount of particles shed is relative to the degree of motion, the physical condition of the individual, and the type and material of the outer garments.

5.3 Transportation of contaminants. In order to establish means of control for contamination, it is necessary to recognize (1) the sources of contamination, (2) means in which they are commonly transported. If contaminants were immobile their restrictions would be more easily accomplished. Contaminants are usually transported in fluids, on solids, by physical contact, or by waves.

5.3.1 Fluids. If contaminants are transported by fluids, they are either suspended, absorbed, or dissolved in the liquid.

5.3.2 Solids. If contaminants are transported by solids they are either entrapped, adsorbed, or absorbed as the solid is moved.

5.3.3 Physical contact carriers. Contaminants moved by physical contact carriers migrate by means of attaching to operator's physical being, tools, fixtures, etc.

5.3.4 Waves. Generally, if migration is by waves such as heat, light, sound, etc., the propagation mechanism is an inherent characteristic of the contaminating energy.

Table IV.

Typical sources of particles

Activity	Particle Size (In Microns)
1. Rubbing ordinary painted surface	90
2. Sliding metal surfaces (nonlubricated)	75
3. Crumbling or folding paper	65
4. Rubbing an epoxy painted surface	40
5. Seating screws	30
6. Belt drive	30
7. Writing with ball point pen on ordinary paper	20
8. Handling passivated metals such as fastening materials	10
9. Vinyl fitting abraded by a wrench	8
10. Rubbing the skin	4

Table V.

Increase of contamination levels by personnel

Activity	Times increase over ambient levels (particles, 0.2 to 50μ)
Personnel movement:	
Gathering together 4 to 5 people at one location	1.5 to 3
Normal walking	1.2 to 2
Sitting quietly	1 to 1.2
Laminar flow work station with hands inside	1.01
Laminar flow work station-no activity	None
Personnel protective clothing (synthetic fibers):	
Brushing sleeve of uniform	1.5 to 3
Stamping on floor without shoe covering	10 to 50
Stamping on floor with shoe covering	1.5 to 3
Removing handkerchief from pocket	3 to 10
Personnel per se:	
Normal breath	None
Breath of smoker up to 20 min. after smoking	2 to 5
Sneezing	5 to 20
Rubbing skin on hands and face	1 to 2

Table VI.

Sources of industrial contamination

Source	Particle Size Range (In Microns)
Combustion products:	
Power generating plants	0.5 to 50
Refineries	0.5 to 50
Commercial transportation (including private autos)	0.1 to 10
Heating plants	0.1 to 1200
Exhaust from chemical processing plants	2 to 10
Construction:	
Erection of new buildings	1 to 50
Demolition of buildings	1 to 100
Construction and repair of streets and roads	1 to 100
General:	
Mining and quarries	1 to 500
Cement plants, foundries, steel mills	0.5 to 1000
Domestic coal smoke	0.01 to 5

5.4 Contamination retention. Contamination retention usually occurs through the attachment of particles, liquids, and gases to surfaces. The type and strength of the forces exerted to hold the contamination to a surface are important considerations in determining the most appropriate method of removal.

5.4.1 Common mechanisms of retention.

(a) Gravity. The amount of contamination retained by gravitational forces depends on the angle of the surface from horizontal.

(b) Electrostatic charges. Such charges not only hold contamination, but also attract it from the surrounding environment. If possible the polar characteristics of a cleaning agent should match that of the soil to be removed.

(c) Molecular attraction. Involves adhesion and absorption and the effects of Van der Wall's forces. It is directly affected by particle size, shape, composition, and contact area as well as the surface roughness.

(d) Viscous surface coatings. A common cause of contamination retention particularly where machining, lubricating, or skin oils are encountered.

(e) Physical entrapment. Should be a definite consideration for rough surface finishes or porous surfaces and materials.

5.5 References.

1. Chemical Engineers Handbook, J. H. Perry (Ed), McGraw-Hill Book Co., Inc., N.Y., 1963.

2. Clean Room and Work Station Requirements, Controlled Environment, Federal Standard 209a, August 16, 1966.

3. Clean Room Technology, James W. Useller, Lewis Research Center, Cleveland, Ohio, NASA SP-5074, 1969.

4. Contamination Analysis and Control, James L. Dwyer, Reinhold Publishing Corp., N.Y., 1966.

5. Contamination Control Handbook, Sandia Laboratories, NASA SP-5076, 1969.

6. Contamination Control Principles, Sandia Corporation, NASA SP-5045, 1967.

7. Fluid Filters, Media and Contamination, H. L. Wheeler, Jr., Bendix Filter Division, Bendix Aviation Corporation, Madison Heights, Mich., 1960.

8. Procedure for the Determination of Particulate Contamination of Air in Dust Controlled Spaces by the Particle Counting and Particle Size Measurement, J. Air Pollution Control Assoc., 13, 9, 402, Sept., 1963.

9. Product Cleanliness Levels and Contamination Control Program, Military Standard 1246A, Dec. 19, 1962.

10. Recommended Standard Method for Determination of Fine Particulate Matter in the Atmosphere by Particle Counting and Particle Size Measurement, J. Air Pollution Control Assoc., 13, 4, 402, Sept., 1963.

11. Tentative Methods for Sizing and Counting Airborne Particulate Contamination in Clean Rooms and Other Dust Controlled Areas Designed for Electronic and Similar Applications, ASTM F25-63T.

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SECTION 6

CONTAMINATION CONTROL THROUGH PRODUCT DESIGN

6.1 Effect of product design on contamination control. To assure that a given product will function reliably, contamination control must be considered along with all other requirements during each stage of product development.

Ideally, the product designer should design his product to tolerate the contamination environment under which it will be manufactured and actually used. All too often, contamination control procedures are not developed until after a product failure has taken place. It is difficult to foresee every potential contaminant in every case; but many costly rejects caused by contamination might have been prevented if contamination control had been a factor in product design. At this stage, features can be incorporated in the design to:

(a) Minimize or eliminate sources of contaminant generation.

(b) Render the product least susceptible to contamination.

(c) Facilitate contamination removal and monitoring.

6.2 Methods of contamination control in product design. The designer should evaluate the materials and the product size and function which might influence compromises between materials, the product, and contamination controls.

6.2.1 Anticipate effects of contaminants. To assure reliability at a reasonable cost, the designer must anticipate the potential effects of various kinds of contaminants on the life and function of the product. This requires careful consideration of the sources of contamination, the sizes, types, and quantities of contamination which will originate from these sources, and their effect on the product.

6.2.2 Control of manufacturing environment. The need for highly reliable products has caused industry to turn to the precise control of manufacturing environments. This need has been amplified by the wider use of new and more sensitive materials and alloys, the trend toward microminiaturization, and greater complexity of hardware leading to increased costs and time involved.

6.2.3 Other considerations. In order to create a truly effective design, the product designer must be aware of more than just the function and reliability requirements; he must also be aware of the following factors of product life:

(a) Cleanliness requirements.

(b) Manufacturing and processing environments.

(c) Manufacturing processes.

(d) Test procedures and equipment.

(e) Storage and use conditions.

6.3 Specific steps to control contamination. In taking specific steps to control contamination, the designer should:

(a) Consider all possible sources, types, and amounts of contamination which the product may encounter.

(b) Be aware of the different control techniques available.

(c) Specify requirements and features which will help achieve and maintain the needed cleanliness level.

6.4 Checklist for contamination control measures. The following list was developed as a sample guide to assist in determining whether all reasonable contamination control measures have been considered.

(a) Identify contaminant-sensitive parts, assemblies, surfaces, parts, areas, etc., that require some level of cleanliness.

(b) Define the level of cleanliness required.

(c) Define the contaminants that cannot be tolerated.

(d) Define purity requirements for basic materials.

(e) Define sequences of assembly to facilitate controls.

(f) Define cleaning methods to be employed.

(g) Define cleanliness levels required for assembly and process areas and facilities.

(h) Define purity requirements of materials used in production and cleaning processes.

(i) Define methods for measuring cleanliness levels of controlled areas and facilities.

(j) Define methods to be employed for excluding contaminants from clean items and assemblies.

(k) Define tests and measurements to verify effectiveness of control techniques.

(l) Define methods for servicing or maintenance operations that assure continuity of the required cleanliness level.

6.5 Factors which may affect contamination of a product. There are various factors which may affect contamination of a product. Outlined, as follows, is a list of considerations, techniques, and specific methods for control that should be deliberated by designers.

6.5.1 Considerations.

(a) Establish cleanliness level required of contaminee.

(b) Determine quantity and type of contamination tolerable.

(c) Ascertain what contaminants effect the product.

(d) Determine the origin of the contaminant.

(e) Establish the means by which the contaminants travel.

(f) Ascertain ways in which contaminants effect the contaminee.

(g) Determine the best method of contaminant removal.

(h) Establish the most suitable and efficient cleaning method for the part.

(i) Determine which solutions are not compatible with materials.

6.5.2 Techniques for control.

(a) Eliminate the contaminant and its source.

(b) Exclude the contaminant from the contaminee.

(c) Reduce the size and volume of the contaminant in the critical environment.

(d) Minimize the affect of the contaminant.

(e) Remove contaminant generating materials.

6.5.3 Specific methods.

(a) Material. Select compatible, noncorrosive, low contaminant-generating materials.

(b) Configuration. Design to facilitate cleaning, flushing, and monitoring; eliminate blind holes and dirt traps; isolate sensitive areas.

(c) Dimensions. Specify maximum tolerances allowable and suitable surface finishes.

(d) Processes. Specify manufacturing and cleaning processes and process controls to be compatible with cleanliness levels required.

(e) Monitoring. Specify best available monitoring techniques for each type of contaminant encountered.

(f) Storage and use. Specify the type and method of packaging needed to protect the product, i.e., moisture-proof, nonshedding; sealable, strong, etc. Maintenance instructions may be included.

The ensuing tables list factors affecting contamination in products and should be used as references.

Table VII.

Factors affecting contamination in products

General Areas of Consideration		Characteristics for Design Considerations
Materials	Purity	Electrical Mechanical Method of analysis
	Generation of contaminants	Corrosion Outgassing Wear products Shedding Flaking
	Workability	Burrs Flashing Galling Finish

(continued)

General Areas of Consideration		Characteristics for Design Considerations
	Compatibility with other materials	Corrosion Galvanic action Galling Wear Lubricants Preservatives Reaction to cleaning
Configuration	Parts	Accessibility to surfaces Blind holes Capillary traps
	Assemblies	Accessibility to sensitive areas Mating of materials Generation of contaminants Protection of parts
Dimensioning	Parts	Tolerance limits Contaminant limits
	Assemblies	Mating surfaces Contaminant limits
Fabrication (forming and finishing	Casting	Sand and cores Residues Blind holes Capillary traps Entrapped gases
	Molding	Flash Mold release residue Mold wear products
	Forming, drawing, extrusion	Burrs Lubricants Release compounds Particles Scales
	Forging	Scale
	Machining	Blind holes Burrs and chips Sharp edges Capillary traps Resultant dusts from dry and wet honing

(continued)

General Areas of Consideration		Characteristics for Design Considerations
		Coolants Cutting oils Dry and wet honing and resultant dusts
	Chemical milling	Etch residue
	Plating	Scale Flaking Residue
	Heat treat	Scale Silica Liquids
	Cleaning	Effect on material Drying Residue Temperature effect
	Polishing	Oils Polishing compounds Chemical residue Dust Oxides
	Tools, equipment, and personnel	Dirt Oils Wear products Personnel contaminants Airborne contaminants
Assembly processes	Product flow and assembly sequence	Migration and transfer of contaminants from: Mating assemblies Tools, jigs, fixtures Work surfaces Personnel Environment
	Rivets	Galvanic action Swaging fragments
	Nuts, bolts, screws	Shedding Burrs Chips Abrasives

(continued)

General Areas of Consideration		Characteristics for Design Considerations
	Welding, brazing, and soldering	Metal fragments Solder fragments Flux residue Fumes Oxides
	Gaskets	Flaking Shedding Lubricants
	Encapsulation, sealing	Outgassing Shedding Flaking Thermic reaction
	Cleaning	Compatibility with all materials Entrapment Gas generation Residue
	Lubricants	Excess Migration Reaction to materials
	Marking	Etch residue Flaking Shedding
	Tools, jigs, fixtures	Flaking Shedding Oils Abrasive products
	Personnel environment	Personnel contaminants Airborne contaminants
Test and inspection	Equipment, gages, and fixtures	Flaking Shedding Oil Dirt Abrasion products
	Chemical tests	Dyes Residues
	Operations	Burrs Chips

(continued)

General Areas of Consideration		Characteristics for Design Considerations
		Plating scale Released fluids and gases Wear products Personnel contaminants
Storage	Packaging	Proper selection of: Containers Wrapping materials Cushioning materials Desiccants Barriers
	Environments	Temperature Humidity Pressure Shock Vibration
Use	Operation- Continuous Intermittent Self-destruct	Wear pressures Lubrication Contaminant generation Radiation
	Servicing	Introduction of or exposure to contaminants

6.6 Performance levels. An absolute measurement or definition of the degree of cleanliness required for a specific case is frequently impracticable or impossible. The alternative is to establish minimum product performance levels. Performance levels can be developed for:

(a) Production processes.

(b) Appropriate levels of assembly of product.

(c) Controlled areas and facilities.

(d) Cleaning methods.

(e) Finished product.

(f) Other activities where indication of a cleanliness level is required.

When performance levels are established in lieu of cleanliness levels, they must be commensurate with the performance requirements of the product as the state-of-the-art permits at the moment. Control methods and monitoring techniques must be employed to maintain uniform and consistent operations and to assure consistent levels of performance at all times.

6.7 References.

1. Clean Room Technology, James W. Useller, Lewis Research Center, NASA SP-5074, 1969.

2. Contamination Control Handbook, Sandia Laboratories, NASA SP-5076, 1969.

3. Grumman Aerospace Corporation.

4. Sterilization and Quarantine Parameters for Consideration During the Design of Planetary Vehicles, Charles W. Craven, Joseph J. McDade, and Jay O. Light, Proceedings of the National Conference on Spacecraft Sterilization Technology, NASA SP-108, November 16, 1965.

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SECTION 7

CORROSION

7.1 Corrosion defined. Corrosion is the destructive attack of a metal by chemical or electrochemical reaction with its environment. Deterioration of a metal by physical causes such as galling or wear is called erosion. Chemical attack when accompanied with physical deterioration is defined as corrosion-erosion, or fretting corrosion.

7.1.1 Chemical corrosion. Direct chemical corrosion is limited to unusual conditions involving highly corrosive environments or high temperature or both. However, most of the phenomena involving corrosion of metals is electrochemical in nature.

7.1.2 Electrochemical corrosion. Electrochemical corrosion is the anodic and cathodic reaction with an electrolyte normally provided in the environment to which the metal is exposed. Anodic reaction takes place when the metal dissolves in the electrolyte in the form of positively charged ions. Cathodic reaction occurs when positively charged hydrogen ions plate out as atomic hydrogen on the cathodic surface. The electrons released by the anodic reaction flow through the metallic circuit to the cathode to neutralize an exactly equivalent number of hydrogen ions. The hydrogen film will eventually cover and polarize the cathodic surface, stopping the flow of electrons. The positive metal ions released near the surface of the anode combine with negative hydroxide ions from the electrolyte (water) to form a neutral metal hydroxide which may coat the anodic surface stopping the ion release. Thus, under favorable conditions, the electrochemical reaction will stifle itself at the cathode or anode or both.

7.2 Effects of environment on metals. Metal corrosion is the reaction of metal with the environment. The rate and type of corrosion that occurs are affected by the many variations of these two factors which include:

7.2.1 Metal properties.

(a) Structure and orientation.

(b) Composition including alloy components and impurities.

(c) Surface condition and finish.

(d) Metal stresses, applied and internal.

7.2.2 Environment.

(a) Temperature.

(b) Atmospheric: humidity, dust, and gases.

(c) Liquids.

(d) Soils.

(e) Contact with dissimilar metals (galvanic).

(f) Stray electrical current.

7.3 Mil-Std-1250 (MI) definition. According to Mil-Std-1250(MI) corrosion is defined as a specific type of deterioration resulting in damage or impairment of metals or metallic parts as the result of attack by moisture, air, acid, alkali, chemicals, or electrochemical action. Although mechanical stress is a factor in certain types of corrosion, damage or breakage as the result of purely mechanical load or shock is not included.

7.4 Corrosion forms. There are several forms of metal corrosion, intergranular, crevice, stress-corrosion-cracking, hydrogen embrittlement, and galvanic corrosion each of which is discussed briefly.

7.4.1 Intergranular corrosion. Because metals are not completely homogeneous, some areas, often the grain boundaries, are more susceptible to corrosion than others. In aluminum alloys, the grain boundaries are anodic to the grain centers. In a damp corrosive environment, pitting may occur in a preferential pattern, clearly outlining the grain boundaries. Brasses with more than 15 per cent are subject to a form of intergranular corrosion called "dezincification" in which the zinc is removed, leaving only a porous copper part.

7.4.2 Crevice corrosion. This kind of attack occurs in crevices or at threaded fasteners or under washers where there is a lack of oxygen; it is also called "oxygen starvation" or "concentration cell" corrosion or differential aeration. It is usually found on corrosion-resistant steel, iconel, nickel, aluminum, and other metals which may be either "passive" (protected by a thin oxide film) or "active." The passive film does not persist in the presence of electrolyte, and the absence of oxygen; thus, the metal in the joint where oxygen cannot penetrate will become "active" and therefore anodic to the rest of the part. Residues from soldering flux can cause crevice corrosion.

7.4.3 Stress corrosion. When materials are under load or stress, it takes only a mild corrosion agent to cause failure. There is a three-way relationship between the corrosent (which may be only water), the stress level, and the time before failure or cracking occurs.

7.4.4 Hydrogen Embrittlement. Closely associated with stress corrosion is hydrogen embrittlement, primarily a problem in high strength iron base and nickel base alloys and titanium. When these high strength (or highly stressed) metals are acid cleaned, pickled, electroplated, welded or otherwise exposed to nascent hydrogen, they may pick up enough hydrogen to cause them to crack spontaneously, in what is known as a delayed failure or fracture.

7.4.5 Galvanic corrosion. A frequent problem resulting in the failure or malfunction of many components is that of galvanic corrosion resulting from the contact of dissimilar metals. This occurs when two metals of different electromotive potential are placed in close contact in the presence of an electrolyte. This arrangement results in a flow of current from the anode to the cathode with the liberation of hydrogen at the cathode and the formation of alkali.

7.5 Other contributers to corrosion.

7.5.1 Porous castings. Any surface which is porous offers an aid to corrosion since contaminants and moisture can collect in the openings and cause corrosion of not only the part but the surrounding parts also.

7.5.2 Fungi and Bacteria. Any condition which holds moisture can promote the growth of microorganisms. The metabolic by-products of their growth are acids which can etch and corrode metals and cause the separation of layers of materials.

7.6 Corrosion prevention. In each of the corrosion situations mentioned previously, there are specific steps which can be taken to either prevent or lessen the possibility of the corrosion taking place.

7.6.1 Intergranular corrosion. Selection of the proper grade and heat treatment condition of the metal and the provision of protective coatings where possible will lessen the possibility of intergranular corrosion.

7.6.2 Crevice corrosion. Since the presence of water or other electrolyte is a key factor here, the sealing of joints will prevent this form of attack.

7.6.3 Stress corrosion. In order to prevent stress corrosion:

(a) Choose metals which are not susceptible.

(b) Reduce stress, both operating and residual.

(c) Protect metal from environment.

(a) Sources of stress. The residual stress which causes stress corrosion originates in the fabrication of the part, not in service. Heat treatment, especially rapid quenching, welding, grinding, joggling, swagging, bending and severe forming, excessive tightening of fasteners, pressfits and tapered bolts, all produce high levels of stress in parts.

(b) The favorable environment. Many environments are favorable to the stress corrosion of specific metals and alloys. Table VIII gives a breakdown of these environments.

Table VIII.

Favorable environments for stress-corrosion-cracking

Steel	Alkalies, nitrates, hydrogen cyanide, hydrogen sulfide, anhydrous liquid ammonia, sodium chloride solutions, marine atmospheres.
Chromium Stainless Steels (over 12% Cr)	Halides, hydrogen sulfide, steam.
Austenitic Stainless Steel (18% Cr, 8% Ni, 300 series)	Chlorides, caustic.
PH Stainless Steel	Chloride Solutions, marine atmosphere.
Copper Alloys	Ammonia, mercurous nitrate, steam.
Gold Alloys	Iron chlorides, potassium cyanide.
Magnesium Alloys	Sodium chloride/potassium chromate, fluorides, moisture.
Aluminum Alloys	Sodium chloride solutions, tropical environments, water.
Nickel Alloys	Caustic, hydrofluorosilicic acid.
Monel, Iconel	Hydrogen fluoride vapors.
Titanium Alloys	Red fuming nitric acid, hydrogen chloride, dry molten chloride, salts chlorinated hydrocarbons, methyl alcohol, certain N_2O_4 compositions.

7.6.4 Hydrogen embrittlement. Whenever possible, avoid acid cleaning, pickling, and electroplating. The designer should select materials and processes which minimize the danger of embrittlement.

(a) Choose organic films or vacuum deposited films.

(b) If plating is necessary, use alkaline baths or other processes designed for low hydrogen pick up; use cadmium instead of zinc or chromium; shot peen before plating; stress relieve before plating and immediately afterward.

(c) Use materials not subject to hydrogen embrittlement. Use 300 series corrosion resistant steels; use oxygen-free or deoxidized copper.

7.6.5 Galvanic corrosion. Anything which reduces the potential difference between metals involved or lessens the contact of the metals reduces the chance of galvanic corrosion. One may group metals by their compatibility (Table IX) or by their electromotive potential (Table X). Table IX lists groups of metals which are considered similar to one another and may be coupled with others in the same group but not with those in other groups. Table X lists acceptable couples based on the electromotive potential. Note also, Table XI which offers problems, solutions, and examples for the prevention of galvanic corrosion.

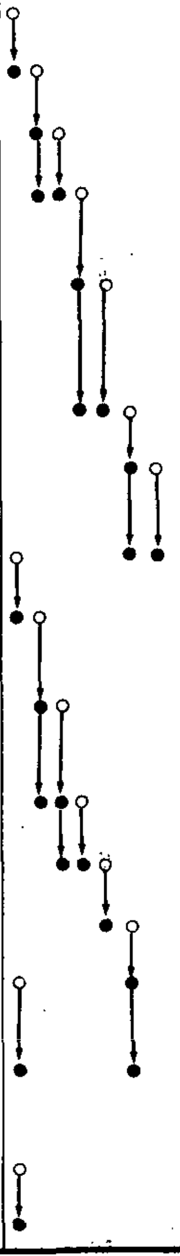
Table IX.

Compatible metal groups

Group 1	Group 2	Group 3	Group 4
Magnesium & alloys	Aluminum & alloys	Zinc	Copper & alloys
Aluminum 5056, 5052, 5356, 6061, 6063	Zinc	Cadmium	Nickel & alloys
	Cadmium	Steel	Chromium
Tin	Tin	Lead	Stainless Steel
	Stainless Steel	Tin	Gold
	Tin-Lead	Stainless Steel	Silver
	Solder	Nickel & Alloys	
		Tin-Lead	
		Solder	

Table X.

Galvanic couples

Group	Metallurgical Category	EMF (volt)	Permissible Couples *
1	Gold, gold-platinum, platinum	0.15	
2	Rhodium, graphite	0.05	
3	Silver	0	
4	Nickel, monel, high nickel-copper alloys, titanium	-0.15	
5	Copper, low brass or bronze, silver solder, nickel-chromium, austenitic (type 300) stainless steels	-0.20	
6	Commercial yellow brass and bronze	-0.25	
7	High brass and bronze; naval brass, muntz metal	-0.30	
8	18% chromium type steels	-0.35	
9	Chromium, tin, 12% chromium type steels	-0.45	
10	Tin-plate; tin-lead solders; terne-plate	-0.50	
11	Lead; high lead alloys	-0.55	
12	Aluminum, wrought, 1000 series	-0.60	
13	Iron, low alloy steels, armco iron	-0.70	
14	Aluminum, 3000, 6000 and 7000 series Aluminum silicone castings	-0.75	
15	Aluminum castings (other than silicone); cadmium	-0.80	
16	Hot dip zinc, galvanized steel	-1.05	
17	Zinc	-1.10	

(continued)

Group	Metalurgical Category	EMF (volt)	Permissible Couples*
18	Magnesium		

*Key   0 = cathodic   ● = anodic

Table XI.

Preventing galvanic corrosion

Problem	Solution	Example
Dissimilar Metals	Select permissible couples.	Use nickel, not naval brass, next to silver.
Dissimilar Metals	Plate with compatible metal to reduce potential difference.	Tin coat steel and bronze used together.
Dissimilar Metals	Keep affected area of less noble metal (anode) as large as possible.	Stainless steel hardware in aluminum may be satisfactory because of large area of aluminum.
Dissimilar Metals	Apply corrosion inhibitors such as zinc chromate paste.	Assemble dissimilar metal hardware with zinc chromate.
Contact	Interpose inert barrier or gasket to prevent the contact.	Vinyl tape, cadmium plated washer, rubber gasket.
Contact	Paint the cathode, or both of the metals.	
Electrolyte	Avoid design which allows moisture to be entrapped.	
Electrolyte	Use desiccant.	
Electrolyte	Seal joint with organic insulator, alkali resistant.	
Electrolyte	Seal faces of the metals against contact with the electrolyte.	Primer, paint or sealant.

(continued)

Problem	Solution
General	Use cadmium in preference to zinc plating. Use tin or nickel plated hardware. Avoid the use of magnesium.

7.6.6 Fungi. The damaging effects of fungi and bacteria can be limited or eliminated through several precautions.

(a) Choose materials which are funginert. (See Table XII)

(b) Keep equipment dry and use fungicides.

Table XII.

Funginert materials

Funginert in all Conditions	Funginert in Some Grades
Acrylonitrile-vinyl chloride copolymer	Acetal resins
Asbestos	Cellulose acetate
Ceramics	Cellulose acetate butyrate
Chlorinated polyether	Epoxy glass fiber laminates
Glass	Epoxy resin
Metals	Melamine-formaldehyde
Mica	Natural rubbers
Plastic laminates: Silicone glass fiber Phenolic nylon fiber Diallyl phthalate & polyethylene terephthalate	Phenol-formaldehyde Poly methyl methacrylate Poly vinyl chloride
Polyacrylonitrile	Poly vinyl chloride acetate
Polyamide	Poly vinyl fluoride
Polycarbonate	Plastic laminates using:

(continued)

Funginert in all Conditions	Funginert in Some Grades
Polyethylene	Cotton filler Linen filler Paper filler Wood-flour filler
Polymonochlorotrifluoroethylene	Synthetic rubbers
Polypropylene	Urea-Formaldehyde
Polystyrene	
Polytetrafluoroethylene	
Polyethylene terephthalate	
Poly vinylidene chloride	
Silicone resin	

7.6.7 Metallic coatings. Frequently, the desiger may consider the use of coatings for the prevention or limitation of corrosion. Table XIII offers a guideline for the selection of metallic coatings for corrosion prevention.

Table XIII.

Selection of coatings for minimum corrosion

Purpose	Recommended	Not Recommended
Contact with aluminum or magnesium	Cadmium or tin	Chromium, copper, silver, gold
Prepaint coating	Cadmium or tin	Chromium, copper, nickel, gold, silver
Tarnish prevention	Rhodium over silver	
	Gold over silver, copper or nickel	
	Nickel between copper and silver	
Marine exposure	Heavy gold 0.00030 inch minimum	
Solderability	Tin, gold or tin-lead	Nickel, chromium, rhodium

(continued)

Purpose	Recommended	Not recommended
Storage	Gold, rhodium, or reflowed heavy tin	Cadmium, silver, copper
Wear	Chromium, nickel, rhodium or hard gold	Cadmium, tin
Easy etching	Cadmium, nickel (in rhodium, silver, ferric chloride only), indium, tin.	Tin-lead, gold

Note: Many special cases exist. It is sound practice to consult materials, contamination control, and processing specialists, as well as manufacturers representatives before selecting coatings.

7.6.8 Material choices. The designer has the option of choosing materials which can limit the dangers of corrosion and lessen the choice of product failure. Table XIV offers a partial listing of materials which cannot or should not be combined in a product.

Table XIV.

Incompatible materials

Do Not Use		
Copper/manganese	with	Rubber
Cyano-acrylate sealants	with	Cellulosics, methacrylate, polycarbonate, styrene vinyl
Diester oils	with	Neoprene/plastics
Hydrocarbons/ketones	with	Acrylics, cellulosics, vinyl-chloride
Ketones, esters, alcohol	with	Vinyl butyral
Paper	with	Copper or silver
Silicone oils and greases	with	Metals to be coated

7.7 Methods of corrosion removal. Special cleaning methods for specific materials have been developed that are quite effective; however, the metals so cleaned should be well rinsed in running water to remove all traces of the solution and then dried. The solutions for cleaning specific metals are shown in Table XV.

Table XV.

Special cleaning methods to remove corrosion products

Metal	Aqueous Solution	Temp. (°C)	Operation
Aluminum	Nitric acid 75%	25	2 to 3 min. immersion, then light scrubbing with bristle brush and rinse
Copper and nickel alloys	Hydrochloric acid 18% or sulphuric acid 10%	25	2 to 3 min. immersion, then scrub with bristle brush and rinse
	Ammonium citrate 10%	25	Cathodic treatment with cathode current density at 1 amp/in.^{2} time dependent on corrosion products. Rinse.
Iron and steels (except stainless steels)	Sodium cyanide 10%	20	Cathodic treatment for 20 min. with cathode current density at 1.5 amp/in.^{2} Rinse.
	Sodium hydroxide 20% with 200 grams per liter zinc dust	Boiling	5 min. immersion and rinse
Stainless steels	Nitric acid 10%	60	Immersion followed by rinse. Time dependent on corrosion product. (Contamination with chlorides may attack base metal.)
	Acetic acid 1%	Boiling	10 min. immersion, then scrub lightly and rinse.
Lead alloys	Ammonium acetate 25%	90-100	Immersion, then scrub lightly and rinse. Time dependent on corrosion product.
Magnesium alloys	Chromic anhydride 15% with silver chromate 1%	Boiling	15 min. immersion, then rinse. (Contamination with chlorides and sulfates may attack base metal.)

(continued)

Metal	Aqueous Solution	Temp. (°C)	Operation
Tin alloys	Trisodium phosphate 15%	Boiling	10 min. immersion, then scrub lightly with bristle brush and rinse.
Zinc alloys	Ammonium chloride 10% Solution 1	60-80	5 min. immersion in solution 1, then scrub lightly with bristle brush & rinse. Followed by 20 sec. immersion in solution 2, and rinse. (Contamination with chlorides and sulfates may attack base metal.)
Zinc alloys	Chromic acid 5% with silver nitrate 1% Solution 2	Boiling	5 min. immersion in solution 1, then scrub lightly with bristle brush & rinse. Followed by 20 sec. immersion in solution 2, then rinse. (Contamination with chlorides and sulfates may attack base metal.)
All the above metals	Sulphuric acid 5% with 0.5 gram per liter of one of the following: quinoline ethiodide, diorthotolyl thiourea, betanaphthol	75	Cathodic treatment for 3 to 5 min. with cathode current density at 1.3 amp/in. ^{2}. then rinse. (Carbon anode preferred.)

7.8 Corrosion guide. The selection of materials suitable for a given environment should be one of the first considerations in product design. Certain metals and metal alloys are more resistant to some corrosive environments than others. A group of these metals together with a listing of corrosive environments that may be encountered are shown in the ensuing table with relative resistance to the corrosive environments for each metal. This is a general guide only and specific conditions may require further investigation and analysis for selection of the best material.

7.8.1 Reference notes for corrosion guide Table XVI.

Note: Corrosion resistance of metals designated by: E-Exellent, G-Good, F-Fair, NG-No Good.

1. Subject to dezincification and stress corrosion, especially at elevated temperature and with concentrated solutions.
2. May be used with cold dilute acid.
3. Alloys containing up to 60 per cent copper acceptable; high copper alloys not acceptable.
4. Temperature assumed to be below that at which gas cracks and liberates nascent nitrogen.
5. Subject to stress corrosion with low concentrations.
6. Apparently resistant to dry gas at ordinary temperatures; attacked rapidly by moist gas and by hot gas.
7. Not recommended for use with beverage grade.
8. Chemicals used for treating in manufacture assumed to be absent.
9. Temperature assumed to be no higher than that normally encountered in compression, storage, and distribution.
10. Useful at elevated temperatures.
11. Not recommended for use with carbonated beverages.
12. Water assumed to be absent.
13. Not recommended for use with edible grades.
14. Only with dilute or unaerated solutions.
15. Subject to stress corrosion by moist gas, and to severe general corrosion by saturated aqueous solution.
16. Subject to stress corrosion.
17. None of these materials recommended for use with nickel-plating solutions.
18. Higher chromium alloys (over 18%) preferred.
19. Not recommended for temperature over 100°C (212°F).
20. Alloys with less than 18% Cr not recommended for temperatures over 100°C (212°F). Others not recommended for temperatures over 200°C (390°F).

21. Up to 50°C (140°F).

22. Up to 90°C (200°F).

23. At room temperature.

24. Not recommended for temperatures over 200°C (390°F).

25. Nonferrous alloys preferred when unaerated and at temperatures above normal. Stainless steel best when aerated and at normal to moderate temperatures.

26. With cold acid only.

27. In the absence of exposure to moist air.

28. Crude produce may contain acids which corrode these materials.

29. Some of these ratings may not apply when handling light-colored products at elevated temperatures (200°C) (390°F).

30. Good with water containing no oxidizing salts; fair with water containing oxidizing salts.

31. Excellent with water containing oxidizing salts; not good with water containing oxidizing salts.

32. Subject to dezincification with hot and/or aerated waters.

33. Subject to pitting attack.

34. Copper may act as a catalyst for undesirable reactions.

35. Free sulphuric acid absent.

Table XVI.

Corrosion guide

Environments	Brass & naval bronze	Silicon Bronze	Monel Metal	Stainless Types 410, 416,& 430	Stainless Types 302, 303, 304, & 305	Stainless Type 316	Copper	Aluminum
Acetate solvents, crude	F	G	G	G	E	E	G	E
Acetate solvents, pure	E	E	E	E	E	E	E	E
Acetic acid, crude	F[1]	G	G	NG[2]	G	E	G	G
Acetic acid (pure)	F[1]	G	G	NG[2]	G	E	G	E
Acetic acid vapors	NG	G	F	NG	G	E	G	G
Acetic Anhydride	NG	G	G	NG	G	E	G	E
Acetone	E	E	E	E	E	E	E	E
Acetylene	3	NG	G	E	E	E	NG	E
Alcohols	G	E	E	E	E	E	E	G
Aluminum sulfate	F[1]	G	G	NG	F	G	G	F
Alums	F[1]	G	G	NG	F	G	G	E
Ammonia gas[4]	NG[5,6]	6	6	E	E	E	6	E
Ammonium chloride	F[1]	G	E	F	F	E	G	NG
Ammonium hydroxide	NG	NG	F	E	E	E	NG	G
Ammonium nitrate	NG	F	F	E	E	E	F	E
Ammonium phosphate (Ammoniacal)	NG	NG	G	E	E	E	NG	NG
Ammonium phosphate (neutral)	F	F	G	G	E	E	F	F

(continued)

Corrosion guide

Environments	Brass & naval bronze	Silicon Bronze	Monel Metal	Stainless Types 410, 416,& 430	Stainless Types 302, 303, 304,& 305	Stainless Type 316	Copper	Aluminum
Ammonium phosphate (acid)	F[1]	F	G	F	G	E	F	F
Ammonium sulfate	F[1]	F	G	G	E	E	F	G[35]
Asphalt	G	E	E	G	E	E	E	E
Beer	G	G	F	7	E	E	G	F
Beet sugar liquors	G	E	E	G	E	E	E	E
Benzene or benzol[8]	E	E	E	E	E	E	E	E
Benzine[8]	E	E	E	E	E	E	E	E
Borax	G	G	E	E	E	E	G	G
Boric acid	F[1]	G	E	F	G	E	G	E
Butane, butylene, Butadiene[9]	E[34]	E[34]	E	E[10]	E[10]	E[10]	E[34]	E
Calcium bisulfite	NG	G	NG	NG	G	E	G	NG
Calcium hypochlorite	F	F	F	NG	F	G	F	NG
Cane sugar liquors	G	E	E	G	E	E	E	E
Carbon dioxide (dry)	E	E	E	E	E	E	E	E
Carbon dioxide (wet) & (aqueous solution)	F[11]	G[11]	G[11]	E[11]	E	E	G[11]	E
Carbon disulfide	F	NG	F	G	E	E	NG	E
Carbon tetrachloride[12]	E	E	E	E	E	E	E	G

(continued)

Corrosion guide

Environments	Brass & naval bronze	Silicon Bronze	Monel Metal	Stainless Types 410, 416,& 430	Stainless Types 302, 303, 304, & 305	Stainless Type 316	Copper	Aluminum
Chlorine (dry)	G	G	E	G	G	G	G	NG
Chlorine (wet)	NG	F	F	NG	NG	F	F	NG
Chromic acid	NG	NG	F	F	G	E	NG	NG
Citric acid	F[1]	G	G	F	G	E	G	G
Coke oven gas	F	F	G	E	E	E	F	G
Copper sulfate	NG	F	F	E	E	E	F	NG
Core oils	E	E	E	E	E	E	E	E
Cottonseed oil	E	E	E	E	E	E	E	E
Creosote	F	G	E	E	E	E	G	G
Ethers	E	E	E	E	E	E	E	E
Ethylene glycol	G	E	E	E	E	E	E	G
Ferric chloride	NG	NG	NG	NG	NG	NG	NG	NG
Ferric sulfate	NG	F	F	E	E	E	F	G
Formaldehyde	G	G	E	E	E	E	G	G
Ferric acid	F[1]	G	G	NG[2]	G	E	G	NG
Freon	E	E	E	E	E	E	E	G
Furfural	G	G	E	E	E	E	G	E
Gasoline (sour)	F	NG	NG	F	E	E	NG	NG

(continued)

Corrosion guide

Environments	Brass & naval bronze	Silicon Bronze	Monel Metal	Stainless Types 410, 416, & 430	Stainless Types 302, 303, 304,& 305	Stainless Type 316	Copper	Aluminum
Gasoline (refined)	E	E	E	E	E	E	E	E
Gelatin	F[13]	E[13]	E	F[13]	E	E	E[13]	E
Glucose	E	E	E	E	E	E	E	E
Glue	F	E	E	E	E	E	E	F
Glycerine or glycerol	G	E	E	E	E	E	E	E
Hydrochloric acid	NG	F[14]	F[14]	NG	NG	NG	F[14]	NG
Hydrocyanic acid (Hydrogen cyanide)	NG	NG	G	F	E	E	NG	E
Hydrofluoric acid	NG	F	E	NG	NG	NG	F	NG
Hydrogen fluoride	F	G	E	F	G	G	G	NG
Hydrogen[9]	E	E	E	E	E	E	E	E
Hydrogen peroxide	NG	F	G	E	E	E	F	G
Hydrogen sulfide (dry)	F[6]	NG[6]	F[6]	G	E	E	NG[6]	E
Hydrogen sulfide (wet & aqueous solution)	F	NG	F	F[15]	G	E	NG	E
Lacquers and lacquer solvents	F	E	E	E	E	E	E	E
Lime-sulfer	NG	F	G	G	E	E	F	NG
Magnesium chloride	F	G	E	F	G	E	G	NG

(continued)

Corrosion guide

Environments	Brass & naval bronze	Silicon Bronze	Monel Metal	Stainless Types 410, 416,& 430	Stainless Types 302, 303, 304,& 305	Stainless Type 316	Copper	Aluminum
Magnesium hydroxide	G	E	E	E	E	E	F	F
Magnesium sulfate	G	E	E	E	E	E	E	G
Mercuric chloride	NG	NG	NG	NG	NG	F[16]	NG	NG
Mercury	NG	NG	G	E	E	E	NG	NG
Milk	F	F	F	G	E	E	F	E
Molasses	G	E	E	G	E	E	E	E
Natural gas	G	E	E	E	E	E	E	E
Nickel chloride[17]	NG	F	G	NG	F	G	F	NG
Nickel sulfate[17]	F	G	E	F	G	E	G	NG
Nitric acid	NG	NG	NG	G[18]	G	G	NG	F
Oleic acid	F[19]	G[24]	F	G[20]	G[20]	E	G[24]	E
Oxalic acid	F[1]	G	E	F	G	E	G	NG
Oxygen[9]	E	E	E	E	E	E	E	E
Palmitic acid	F[19]	G[24]	E	G[20]	G[20]	E	G[24]	E
Petroleum oils (sour)	F	NG	NG	F	E	E	NG	NG
Petroleum oils (refined)	E	E	E	E	E	E	E	E
Phosphoric acid 25%	NG	G[21]	G[22]	NG	F[23]	E	G[21]	NG

(continued)

Corrosion guide

Environments	Brass & naval bronze	Silicon Bronze	Monel Metal	Stainless Types 410, 416,& 430	Stainless Types 302, 303, 304,& 305	Stainless Type 316	Copper	Aluminum
Phosphoric acid 25% 50%	NG	G[21]	G[22]	NG	NG	G	G[21]	NG
Phosphoric acid 50% 85%	NG	G[21]	G[22]	NG	NG	G	G[21]	E
Picric acid	NG	NG	NG	G	E	E	NG	F
Potassium chloride	F	G	E	F	G	E	G	NG
Potassium hydroxide	NG	F	E	E	E	E	F	NG
Potassium sulfate	G	E	E	E	E	E	E	E
Propane[9]	E	E	E	E[10]	E[10]	E[10]	E	E
Rosin (dark)	G	G	E	E	E	E	G	E
Rosin (light)	NG	NG	G	E	E	E	NG	G
Shellac	G	E	E	E	E	E	E	E
Soda ash (sodium carbonate)	G	G	E	E	E	E	E	NG
Sodium bicarbonate	E	E	E	E	E	E	E	G
Sodium bisulfate	F[1]	G	E	NG	NG	E	G	F
Sodium chloride	F	G	E	F	G	E	G	G
Sodium cyanide	NG	NG	G	E	E	E	NG	NG
Sodium hydroxide	NG	F	E	E	E	E	F	NG
Sodium hypochlorite	NG	F	F	NG	F	G	F	NG

(continued)

Corrosion guide

Environments	Brass & naval bronze	Silicon Bronze	Monel Metal	Stainless Types 410, 416, & 430	Stainless Types 302, 303, 304, & 305	Stainless Type 316	Copper	Aluminum
Sodium metaphosphate	F	G	E	G	E	E	G	F
Sodium nitrate	F	G	E	E	E	E	G	E
Sodium perborate	F	G	E	E	E	E	G	F
Sodium peroxide	F	G	E	E	E	E	G	F
Sodium phosphate (alkaline)	F	G	E	E	E	E	G	NG
Sodium phosphate (neutral)	G	E	E	E	E	E	E	NG
Sodium phosphate (acid)	F[1]	G	E	NG	G	E	G	NG
Sodium silicate	F	G	E	E	E	E	G	G
Sodium sulfate	G	E	E	E	E	E	E	E
Sodium sulfide	NG	NG	G	E	E	E	NG	NG
Sodium thiosulfate (hypo)	NG	NG	G	E	E	E	NG	E
Sludge acid	NG	G	G	NG	NG	F	G	NG
Stearic acid	F[19]	G[24]	F	G[20]	G[20]	E	G[24]	E
Sulfate liquors	NG	NG	G	E	E	E	NG	NG
Sulfur	F	F	F	E	E	E	F	E
Sulfur chloride	NG	NG	G	NG	F	G	NG	NG
Sulfur dioxide (dry)[9]	F	E	E	E	E	E	E	G

(continued)

Corrosion guide

Environments	Brass & naval bronze	Silicon Bronze	Monel Metal	Stainless Types 410, 416,& 430	Stainless Types 302, 303, 304,& 305	Stainless Type 316	Copper	Aluminum
Sulfur dioxide (wet)	NG	G	NG	NG	G	E	G	F
Sulfuric acid 10%	NG	G[25]	G[25]	NG	NG	G[25]	G	NG
Sulfuric acid 10% 75%	NG	F	G	NG	NG	NG	F	NG
Sulfuric acid 75% 95%	NG	F[26]	F[26]	F[27]	F[27]	G[27]	F[26]	NG
Sulfuric acid 95%	NG	G	NG	G	G	G	NG	F
Sulfurous acid	NG	G	NG	NG	F	G	G	NG
Tar	G	E	E	G	E	E	E	E
Tartaric acid	F[1]	G	G	F	G	E	G	G
Toluene or toluol[8]	E	E	E	E	E	E	E	E
Trichloroethylene[12]	E	E	E	E	E	E	E	E
Turpentine	F[28]	E	E	G[28]	E	E	E	E
Varnish	G	G	E	E	E	E	G	E
Vegetable oils[29]	G	G	E	E	E	E	G	E
Vinegar[25]	NG	G	G	F	G	E	G	E
Water (acid mine water)	NG	30	30	31	31	31	30	F
Water (fresh)	F[32]	G	E	E	E	E	G	E
Water (salt)	F[32]	G	E	F[33]	G[33]	G[33]	G	G

(continued)

Corrosion guide

Environments	Brass & naval bronze	Silicon Bronze	Monel Metal	Stainless Types 410, 416,& 430	Stainless Types 302, 303, 304,& 305	Stainless Type 316	Copper	Aluminum
Whiskey	G	G	G	F	E	E	G	F
Wines	G	G	G	F	E	E	G	F
Xylene or xylol[8]	E	E	E	E	E	E	E	E
Zinc Chloride	NG	G	E	NG	NG	G	G	NG
Zinc sulfate	F	G	E	F	G	E	G	G

Source: Harper, H.M. Co., Corrosion Guide, Tech. Bul. No. 101

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SECTION 8

CLEAN ROOMS AND WORK STATIONS

8.1 Clean room training program. In employing clean room controls, the main prerequisite to operating the facilities to specific clean room levels and the ability to maintain the standards at a constant level is directly related to the proper indoctrination of personnel in direct contact with the facilities. Failure to emphatically relate the necessary procedures involved in regard to all phases of personnel cleanliness can result in the introduction of contaminants by personnel, thus, negating all scientific efforts engaged to enable a facility to reach a particular clean room control standard.

8.1.1 Presentation of training program. The training program should be introduced and presented by an individual knowledgeable in contamination control procedures. It has been found that the best results have been demonstrated when the instructions are given by an individual outside the regular clean room staff. As a rule, supervisors or existing company personnel are not as effective in commanding respect for the many regulations involved in a clean room operation.

8.1.2 Basic curriculum. Indoctrination must include a thorough acquaintance with clean room rules, regulations, and procedures. Explanation of the reasons for the stringent regulations must be given in order for workers to be able to grasp the significance of adherence to clean room rituals. This aspect is extremely important and, unfortunately, is often overlooked. Workers are likely to be more self motivated and abide by clean room constraints if they have a clear understanding of (1) causes of contamination (2) control techniques.

8.1.2.1 Simulated clean room. It is advisable that both individual and team training be conducted in a simulated clean room. This procedure permits smooth transfer into the actual clean room with a minimum of errors. It also insures high probability of maintaining the cleanliness level.

8.1.2.2 Management training. Training should be extended not only to the immediate level of supervision of the clean room operators but to the next higher level. Thus, the special problems that must be dealt with in clean room operations will be recognized and appreciation of the difficulties encountered in meeting these problems will ensue.

8.1.3 Specific program for all personnel involved in contamination control technology. A prescribed course of instruction should be provided for all personnel whose activities may bring them in contact with contamination sensitive articles. The minimum controlling factors that should be considered in a training program of this nature are as listed.

(a) Definition of terms associated with contamination control in order to provide a common language and improve communication and understanding.

(b) Presentation of the need for contamination control and the consequence of contamination.

(c) Discussion of the origins and types of contamination, including internal sources created by manufacturing or handling activities, external sources present in prevailing environment, and personnel-created contaminants.

(d) Presentation of the devices and techniques used to achieve and control cleanliness including applications and limitations.

(e) Discussion of tamper-proof seals on clean closures.

(f) Demonstration of methods of measuring and verifying contamination levels.

(g) Presentation of the high orders of cleanliness capable of being achieved and necessary for precision cleaning.

(h) Reasons for design of clean rooms and clean work stations.

(i) Personnel control and occupancy within controlled environment.

(j) Considerations in the selection of furniture, fixtures, and tools that are used in clean rooms.

(k) Reasons for design criteria of special garments.

(l) Maintenance and janitorial considerations for clean rooms.

8.1.4 Specialized courses. Specialized courses, in addition to the contamination control program prescribed for all personnel, should be offered to those individuals whose activities would be specific in nature. The specialized courses would be detailed instruction rather than general. A sample list of courses offered is as follows:

(a) Final cleaning.

(b) Precision clean packaging.

(c) Testing.

(d) Ground support equipment.

(e) Controlled environmental facility (CEF) housekeeping.

(f) Sampling.

(g) CEF cleanliness inspections.

8.1.5 Testing. On completion of each course of instruction, the trainee would be tested with basic questions on each element of instruction. A grade that indicates retention and understanding of the subject must be obtained to qualify for working in contamination sensitive activities. Provision should be made to award a certificate indicating completion of the prescribed training. This certificate is prepared for each successful trainee by the quality control organization. Periodic refresher sessions would be held with the clean room staff to review the procedures currently in use and to consider revisions. As new personnel are added to the clean room, special training sessions should be held.

8.2 Types of clean rooms.

8.2.1 Conventional flow clean room (non-laminar airflow). The conventional flow clean room, or non-laminar airflow clean room, is the oldest type of clean room. It is an air tight structure in which filtered air is introduced at a predetermined rate, uses standard, but filtered air handling and distribution systems. Air usually enters from the ceiling and exits near the floor. This clean room is characterized by non-uniform air patterns and velocities.

The normal volume of air moved in this type of toom is approximately 15 to 20 exchanges per hour. Velocities greater than that required to attain approximately 20 changes of air per hour may cause reentrainment of particulate matter which has fallen out within the room. Usually a High-Efficiency Particulate Air (HEPA) filter is used at air entrance ducts. To satisfactorily control exit velocities and still maintain desired differential room pressures, the air exhaust ports must be properly balanced with the entrance air system. This arrangement optimizes the random airflow patterns of conventional clean rooms and significantly improves their operational characteristics. Normally, conventional clean rooms will obtain a class 100,000 level with relative ease. Good janitorial practices must, however, be imposed in non-laminar flow rooms regardless of the level at which the room is being maintained. Achievement of a prescribed class is dependent on the janitorial service removing contamination brought in by personnel and generated by operations being performed. Strict garmenting procedures must also be observed.

The size of the enclosure usually dictates the number and capacity of ducts. Some installations have exhaust ports in the upper periphery of the walls or in the ceiling. This type of arrangement is not desirable because it is more conducive to air turbulence. Design of the structure and materials used in the room have a direct relationship to the amount of control possible. Smooth edges and elimination of shelves and crevices help greatly in minimizing areas in which particles can become lodged.

8.2.1.1 Advantages. There are several advantages to the non-laminar flow clean rooms. In this type of facility the production line work flow patterns are easily laid out and are not critical; the design is flexible and several areas can utilize one air handling system; filters and air handling equipment are normally designed for ease of maintenance; construction and operation costs are the least expensive of all clean rooms.

8.2.1.2 Disadvantages. The non-laminar airflow clean room has several limitations. The primary limitation is the degree of cleanliness that the room can achieve. Another disadvantage is the stringent regulations that must be followed in order to achieve the cleanliness level. Non-uniform air pattern allows for multiple opportunities for particulate matter to settle on work objects. In order for these particles to be removed a janitor should vacuum constantly during working hours. This activity may be lessened if the occupancy factor is low relative to the room area. The self-clean-down capabilities of the non-laminar type facility is quite low. Time required for expulsion of contaminates generated within the room becomes greater as the rate of generation is increased. It becomes obvious that in order to try to maintain a clean room, internally generated contamination must be kept to a minimum. This can be accomplished by four means (1) air showers, (2) rigorous garmenting rules, (3) constant janitorial service, (4) proper training of personnel.

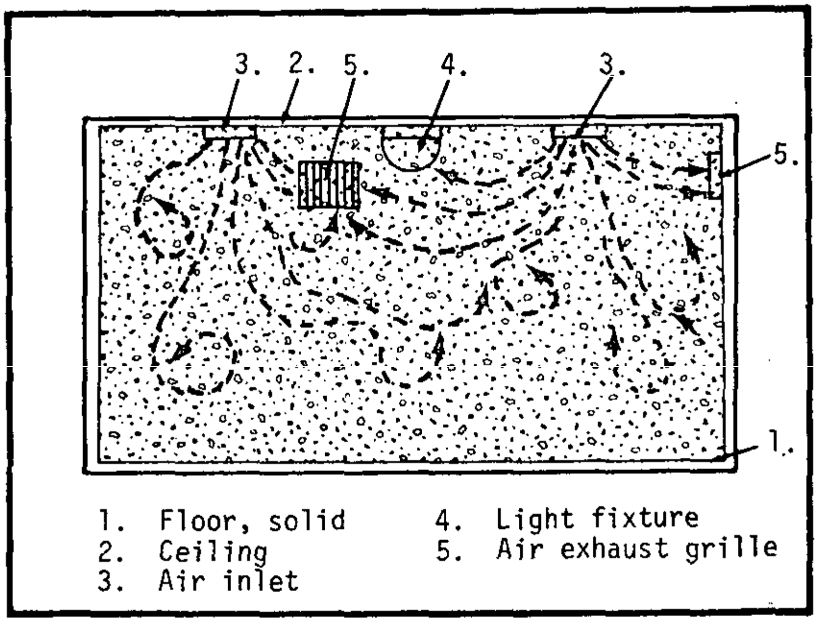

Figure 1. Random airflow pattern

8.2.2 Laminar airflow clean rooms. The laminar airflow clean room is one in which laminar airflow characteristics predominate throughout the entire air space. The airflow is in one direction, uniform in velocity, and parallel elements of flow remain parallel in one plane. Flow can be wall to wall (horizontal) or ceiling to floor (vertical). It is recommended that vertical laminar airflow rooms not be opeated below 65 linear feet per minute with normal air velocity being 90 ± 5 feet per minute. Horizontal facilities will require velocities from 100 feet per minute for installations up to 25 feet in length to speeds ranging from 125 to 140 feet per minute for rooms of greater length.

8.2.2.1 Advantages. There are definite advantages in laminar airflow designed clean rooms. The primary advantage is the amount of control that this type of design enables. Controlling the velocity and direction of the airstream is an imperative factor. Since the airstream is directional with minimal currents, particulates that become airborne will be kept in the airstream and forced out of the area. Constant janitorial service is not necessary in the laminar flow room because of the high efficiency of contamination removal of the airstream. Theoretically, the air after being forced through the HEPA filters will move directly to the exhaust grill across the room in a straight line. The airstream makes only a single pass through the clean room before being returned to the filters for recirculation. This reduces the problem of deposition and resuspension of light particulate matter. A classification of 100 is attainable and a higher degree of cleanliness is possible if stringent personnel regulations are enforced. Laminar flow units introduce greater volumes of air than conventional units; consequently, there is greater control of ambient conditions. In many cases, except in microbial or sterile conditions, air showers, double door airlocks, and large dressing rooms are not necessary. Unless high degrees of cleanliness are required, garmenting can be limited to smocks and head coverings. The laminar flow room can be secure for several days; it should be started at least an hour before use. In general, the laminar flow room has a rapid recovery from contamination conditions.

8.2.2.2 Disadvantages. There are a few disadvantages to installing laminar flow clean rooms. The main disadvantage is cost. Large fans, motors, and ducts are required for the needed higher air velocity. The velocity of air is difficult to maintain with personnel movement and furniture. More room is required than in a conventional flow room in order to accommodate the plenums. Because of the room that must be allocated for plenums, clean room space will be sacrificed. Another factor to consider is the importance of filter functioning. If one single filter module fails to function, the clean room must be shut down and reconditioned.

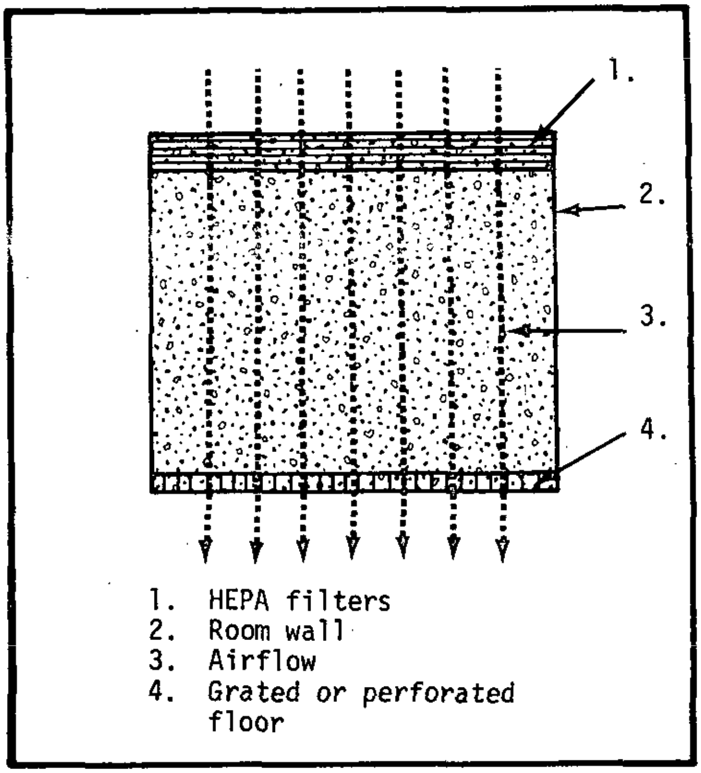

Figure 2. Basic laminar airflow concept

8.2.3 Vertical laminar airflow rooms (VLF). Vertical laminar airflow rooms utilize the basic laminar airflow principle in which the entire body of air within a confined area is directional, moving with uniform velocity along parallel lines. In the VLF room the air moves from ceiling to floor. The entire ceiling is comprised of HEPA filters and the entire floor is a grated perforated exhaust system.

8.2.3.1 Advantages. There are distinct advantages in this type of facility. The main advantage is the vertical flow of air in the system works with gravity and is able to eliminate heavy particles. These particles would normally fall out to the floor surface in non-laminar and cross-laminar flow rooms and would have to be removed by vacuum. In the vertical flow room, particles are merely exhausted through the grated floor. The vertical flow room can easily maintain a class 100 level. It can be operated within a wide range of air velocities; consequently, the system rapidly removes generated or introduced contaminants. There is little opportunity for cross contamination from personnel because the air flow is from ceiling to floor. Since the opportunity for cross contamination is minimal, the affects of personnel on critical operations is minimal. The overall cost of janitorial services, in maintaining Class 100, is substantially reduced in the vertical flow room. If expense is computed on a long term basis, this type of facility would probably be the least expensive to operate.

8.2.3.2 Disadvantages. The VLF room has a few disadvantages. From a construction viewpoint, this type of facility is difficult to build. The ceiling is composed entirely of the supporting framework and verified leak-free HEPA filters. The filter seals around the periphery of each HEPA filter should be verified as leak free. Any openings on the filters, for instance, accommodations for light fixtures, must also be leak free. In other words, very stringent construction principles must be followed. In some cases, operating difficulties arise because small objects are easily dropped through the grill work of the floor. Another disadvantage of the VLF is the expansion limitation. Once a room has been constructed, it is very difficult to enlarge and still maintain good flow characteristics. Both the upper plenums (behind HEPA filters) and lower plenums (under the grated floor) should be of sufficient depth to promote the proper distribution of air; they should be designed for future needs. Very shallow plenums may induce "dead" spots within the plenum and will create low velocity areas within the room directly below the spot. This will reduce the efficiency of the room and can cause cross contamination, a condition that is not supposed to occur in a VLF room.

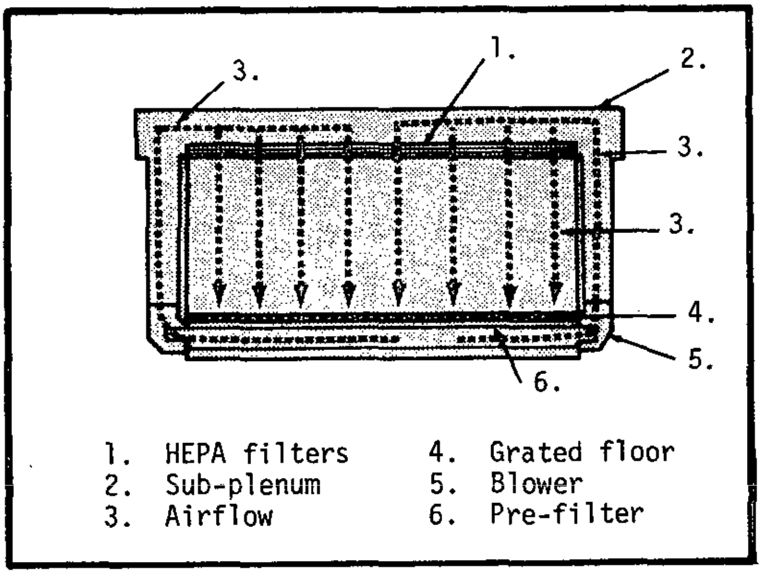

Figure 3. Vertical laminar airflow

8.2.4 Horizontal laminar airflow clean room (HLF). The HLF room is equipped with one entire vertical wall of HEPA filters and an opposite wall of exhaust louvers. The air passes at a predetermined speed and moves horizontally across the room along essentially parallel flow lines at uniform velocities. HLF rooms differ from VLF flow rooms, basically, in the direction of airflow. Both the supply and exhaust walls are backed by plenums for dispersing and balancing the introduced air. These rooms cannot be classified as a designated class. They are zoned rooms with the positions directly in front of filters being class 100; the remaining positions will vary from that to a figure equal to the amount of contamination created by the operation being performed. In order to establish the classifications in different areas of the room, particle counts will be necessary downstream from each operation.

8.2.4.1 Advantages. There are several advantages of the HLF facility. Usually, the HLF room will cost somewhat less than a comparative VLF facility. This variation in cost increases as the length of the room becomes greater. Generally, the narrow side of the room is where the HEPA filters are installed. Occasionally, product requirements are such that to increase effectiveness HEPA filters are put on the longer wall thereby equalizing cost of HLF and VLF rooms. In HLF rooms plenums are located behind the exhaust wall and behind the filter wall. Consequently, room size is not restricted by ceiling height. A room need only be as large as required to accommodate operators and equipment. The variable of a set standard of cubic feet per employee for ventilation purposes is not a factor in either VLF or HLF rooms because of air change rate. HLF rooms have rapid clean-down capabilities. Except in unusual cases (microbial or sterile conditions), air showers, double door airlocks, and large dressing rooms are not an absolute necessity. In most cases, garment requirements are minimal. Unless work is done closer than 30 inches to floor, only smocks and head coverings are necessary.

8.2.4.2 Disadvantages. There are several apparent disadvantages in HLF rooms. The HLF systems require higher air velocities than VLF facilities. If the HEPA filters are located on the narrow wall of a rectangular room, which is generally the case, the air has to travel a greater distance than ceiling to floor as in VLF rooms. The higher velocity insures that particulate matter becoming airborne will remain in the airstream until it is exhausted. Minimum velocity for a short room would be approximately 100 feet per minute and increased to 140 feet per minute for larger rooms or those with a high number of obstructions. Obstructions above work level become imperative factors when air travel is horizontal. Placement of work stations and equipment is extremely important. The generation of contaminants from one station will directly affect the operation immediately downstream from the first operation, and so on, in the case of multiple operations. It is suggested to stagger work stations whenever possible. It is also recommended to place high contaminant producing operations at the exhaust end of the room with less contaminant producing work stations nearer the HEPA filter bank. This action will minimize the affect of cross contamination. Cross contamination is a constant consideration in horizontal flow; consequently, stricter garmenting controls must be exercised in HLF rooms compared to VLF rooms. Another obvious limiting factor of HLF facilities is a single pass of air moving through a room will not assure the removal of heavy particles. These particles tend to fall out within the room and must be removed by other means. Also, particles generated during work operations normally fall out within the room because they are too heavy to be affected by the air stream. Once contaminants become attached to surfaces, the moving laminar air stream will not normally reentrain them. Vacuuming services are required to maintain established cleanliness levels. The frequency that this service is performed is relative to the type of operations being performed in the facility.

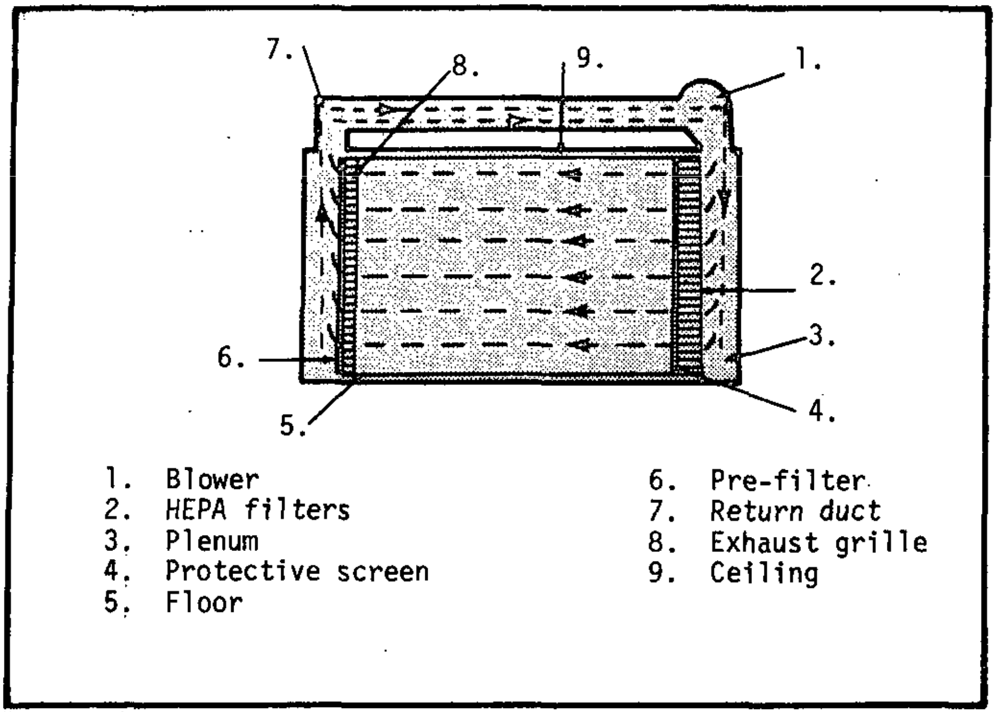

Figure 4. Horizontal laminar airflow

8.2.5 Horizontal laminar airflow (HLF) tunnel. One of the most successful outgrowths of the HLF room is the HLF open end tunnel. In the HLF tunnel the air moves horizontally across the room, along essentially parallel flow lines, at a uniform velocity. The air usually passes through HEPA filters; however, instead of the air being expelled through an exhaust wall across from the HEPA filters, as in HLF rooms, the air is expelled through an open end. HLF tunnels are usually erected within another building; whereas, HLF rooms are completely independent structures. Air exhausted from a tunnel is expelled into the surrounding area; whereas, in an HLF room approximately 75% of the air exhausted is recirculated. There is no positive pressure maintained in a tunnel as in HLF rooms. Air velocity is depended on to overcome infiltration of contaminants into a tunnel. Temperature and humidity within a tunnel is limited to that which is established within the surrounding area. Walls and ceilings of a tunnel are usually merely sheets of transparent plastic and, therefore, can utilize the lighting of the structure in which it is located. It is normal for an installation to have an air velocity of from 100 to 130 feet per minute depending on the size of the tunnel and the type of work being performed. The air cleanliness level that may be expected will be class 10,000 if cautions are observed. As in an HLF room, HLF tunnels are zoned with the positions directly in front of the filters being class 100; the remaining positions varying from that to a figure equal to the amount of contamination created by the operation being performed.

8.2.5.1 Advantages. There are several advantages offered by this type of facility. HLF tunnels offer rapid cleandown ability. They are the lowest cost per square foot facilities available. Although the installation is not truly portable, the cost of moving to another location is minimal. The unit is simple to alter and inexpensive to maintain.

8.2.5.2 Disadvantages. There are several apparent disadvantages in HLF tunnels. The HLF systems require higher air velocities than VLF facilities because of the greater distance that the air has to travel. The higher velocity insures that particulate matter becoming airborne will remain in the airstream until exhausted. Minimum velocity for this type of unit would range usually from 100 feet per minute to about 130 feet per minute. The determination of velocity is dependent on the length of the tunnel and the type of work performed. Placement of work stations and equipment is extremely important because the generation of contaminants from one station will directly affect the operation immediately downstream from the preceding operation. It is suggested to stagger work stations whenever possible. It is also recommended to place high contaminant producing operations at the open end of the tunnel with less contaminant producing operations at the HEPA filter bank. This action will minimize the affect of cross contamination. Cross contamination is a constant consideration in horizontal flow; consequently, stricter garmenting controls must be exercised in HLF tunnels compared to VLF rooms. Another obvious limiting factor of the tunnel is a single pass of air moving through the area will not assure the removal of heavy particles. These particles tend to fall out within the area and must be removed by other means. Also, particles generated during work operations normally fall out within the tunnel because they are too heavy to be affected by the air stream. Once contaminants become attached to surfaces the moving laminar air stream will not reentrain them. Vacuuming services are required to maintain cleanliness levels established. The frequency that this service is performed is relative to the type of operations being performed in the tunnel. In the HLF tunnel there is no control for humidity or temperature and because of the open end, constant observance of air velocity is necessary to overcome contaminant infiltration upstream into the tunnel.

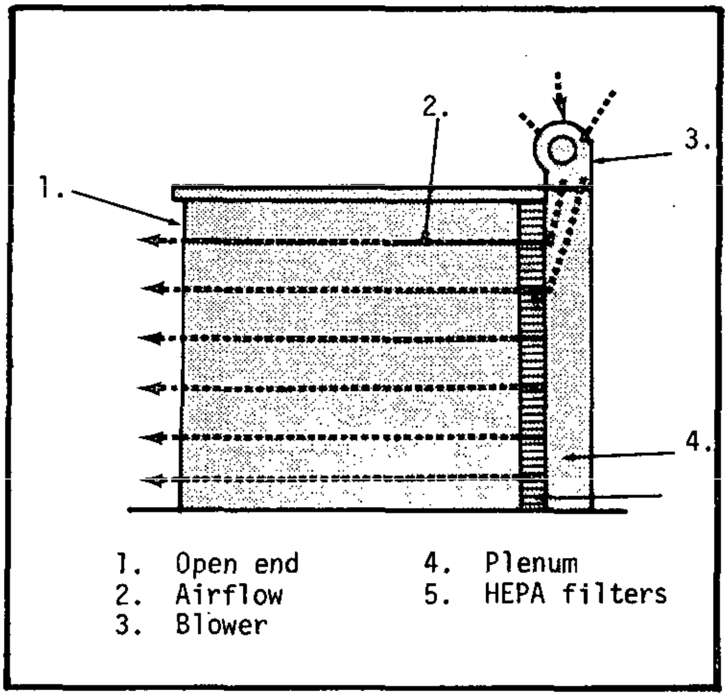

Figure 5. Horizontal laminar airflow tunnel

8.3 Work stations.

8.3.1 Vertical laminar airflow portable curtain. The portable curtain unit was developed to provide a clean room facility that was capable of the highest possible cleanliness standard and that was easily moved to cumbersome structures. This type of unit is not restricted to any particular size, length or height. The method used to extract contaminants is based on the laminar flow principle in which the entire body of air within a confined area is directional, moving with uniform velocity, along essentially parallel lines. The direction of the air is vertical, having first been forced through overhead HEPA filters, and then expelled under the curtains. Air velocity is determined by need.

8.3.1.1 Advantages. There are several desirable features in this unit. Fundamentally, the portable unit is a vertical flow room capable of achieving class 100 classification. Instead, however, of being a rigid structure the side walls are replaced by weighted plastic curtains, allowing for entrance accessibility on all sides. The corner braces are on casters and there is no permanent floor. The unit is equipped with HEPA filters and blowers overhead, as in a VLF room. It is a self contained unit except for electric power. The facility is a satisfactory piece of equipment to use in the open and with care may even be used when a moderate breeze (10-20 miles per hour) is blowing. Vertical laminar airflow portable curtains will perform to the same standards as VLF rooms if care is exercised to avoid disturbing the floor dirt. Another desirable attribute of the portable unit is the rapid clean-down capability.

8.3.1.2 Disadvantages. There are few disadvantages to this facility. The main disadvantage is that the floor must be disturbed as little as possible in order to inhibit the rise of ground surface contaminants. It is also recommended that the work level not be less than 18 inches above the bottom of the curtains. If the unit is not in constant use and has frequent shut-down periods of more than a minute, it should have the opportunity to purge itself for approximately five minutes before it is to be used. If inactive periods extend themselves beyond four hours, the clean-down time should be thirty minutes accompanied by thorough vacuuming of the interior.

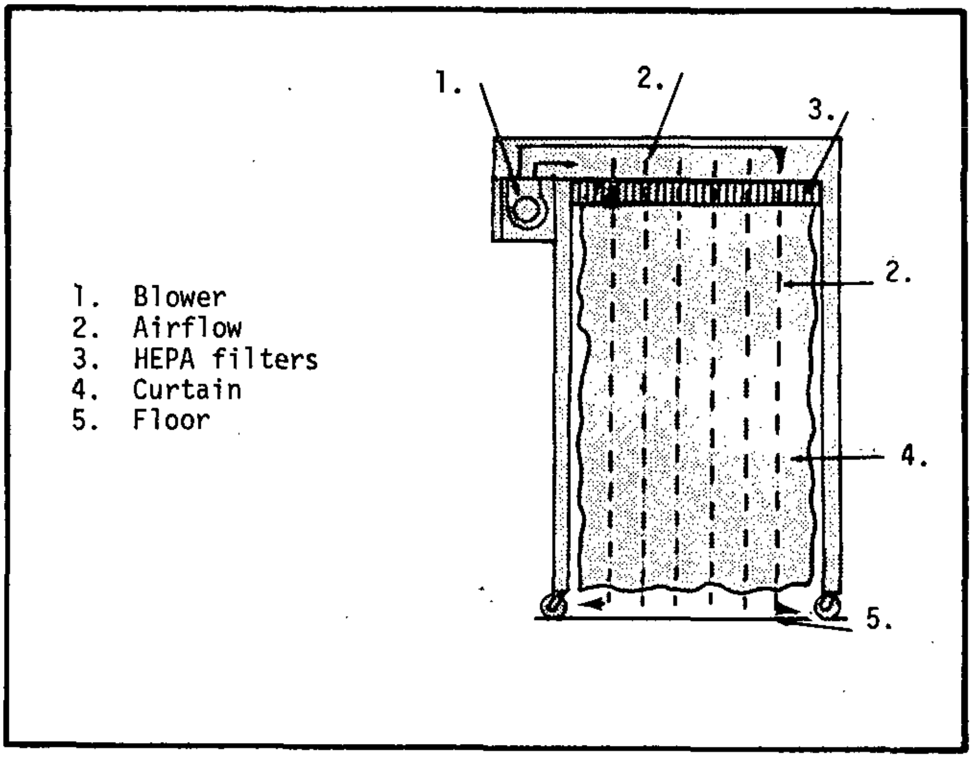

Figure 6. Portable vertical airflow curtain unit

8.3.2 Vertical laminar airflow work bench. The vertical laminar airflow work bench is an adaptation of the VLF room concept. Originally, the laminar airflow work bench operated with a horizontal filtered airflow; the VLF work bench was a sophistication of that unit. Vertical laminar airflow benches incorporate the principle of vertical laminar flow. This involves the flow of air through overhead filters moving in a vertical direction, within a specified area, along essentially parallel lines, at uniform velocities, and expelled through a grated or perforated floor.

The VLF work benches generally used have an open area of from 30 per cent to 60 per cent, utilizing a perforated metal work surface which acts as the exhaust. The amount of space allocated to the work area ranges from six to twelve feet and is usually 22 to 30 inches deep. Air velocities range from 65 feet per minute to about 145 feet per minute.

8.3.2.1 Advantages. VLF work benches definitely are valuable in contamination control operations. There are several advantages to the work benches, although their work space is limited. The units have swift clean-down capability and are able to operate within class 100 in a localized zone. The open front affords easy accessibility and the size allows portability. In working with this type of equipment there are minimal personnel restrictions. It is possible to construct a class 100 assembly line operation by placing a series of benches side-by-side by removing end panels and properly sealing to avert leaks. The benches do not have to be within a clean room of any type. Considering all, this type of set-up is an inexpensive means of providing a class 100 work facility.

8.3.2.2 Disadvantages. There are some disadvantages to the VLF work bench. The size of the work area is the main limiting factor. Another very important point to consider is the critical affect of maintaining the operating station at a constant velocity. Entrance of air to the work station must pass through the filter and any leaks around the filters or ruptures in the elements would contaminate the work area. Because of the open front, velocity measurement must be made at regular intervals to insure the proper dissipation of contaminants. Filters are an integral part of this type of system; therefore, frequent servicing of the elements will insure an efficient operation.

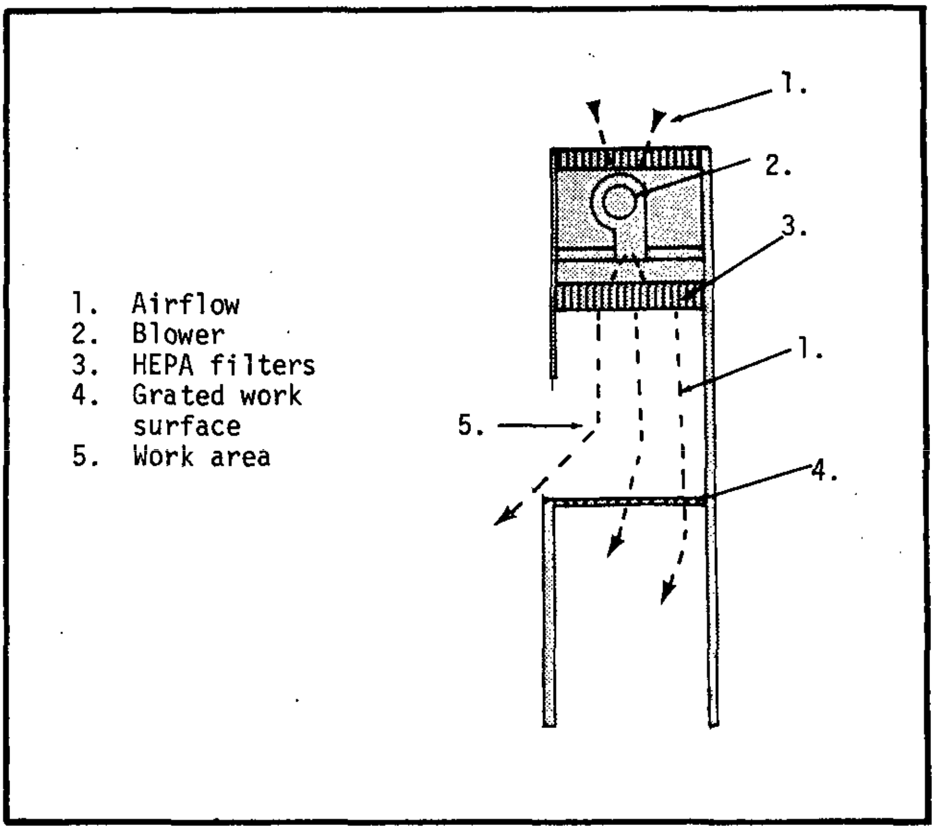

Figure 7. Typical vertical laminar airflow work bench

8.3.3 Horizontal laminar airflow work bench. The horizontal laminar airflow work bench provides a localized clean zone for bench type operations. There are many variations of this type of station. The basic operating principle of this work bench is that of horizontal laminar flow. The air passes through HEPA filters and moves horizontally across a working surface, along essentially parallel flow lines, at uniform velocities. The efficiency of a bench is influenced by good engineering practice and understanding of airflow principles.

8.3.3.1 Advantages. There are specific advantages of the HLF work bench. HLF benches have the capability of providing class 100 cleanliness levels within the confines of the work area when the air velocity is kept constant; the determination of velocity is dependent on the location of the bench. The benches have a swift clean-down capability. The open front affords easy accessibility and the size allows for portability.

It is possible to set up a class 100 assembly line operation by placing a series of benches side-by-side by removing the end panels and properly sealing to avert leaks. The benches do not have to be in a clean room of any type. If an existing clean room is inadequate for the needs of specific operations, it is feasible to upgrade such an area by the use of an HLF work station. There are minimal personnel garmenting restrictions for these benches.

8.3.3.2 Disadvantages. There are certain limitations to employing HLF work benches. The entrance of air to the work bench must pass through the filter. Any leaks around the filter or cracks in the elements would contaminate the work area. Frequent monitoring of pressure drop and air velocity through the filters is imperative to maintain a high level of cleanliness. Air across the work area must have a constant velocity; the determination of velocity is dependent on location of the bench and degree of cleanliness necessary. The air stream should be maintained up to 1 inch from the contaminant surfaces and the forward edge of the bench. Measurement of velocity is obtained by testing from back to front. Differences will occur in velocity from side to side because of variations in porosity of filter elements. Constant observance and testing for contaminant level must continue through operation period. Operators arms will cause some turbulence and this turbulence may pull in contaminants from outside the unit. One manufacturer minimized this effect by placing suction units along the outer edge of stations. These vacuum slots provide an air sink at a lower pressure than the moving air stream. This straightens the airflow after it passes over an object. Turbulence and backwash behind the object still exist but to a lesser degree. The barrier created by the moving air stream and the air sink reduces entering contamination except that which is introduced by the operator. (See Figure 8.)

8.3.4 Balanced laminar airflow hood. The balance laminar airflow hood was developed and refined to provide an open access area that had the capabilities of controlling fumes and retained the characteristics and advantages of laminar airflow.

The basic principle which this hood follows is the suction and expulsion of air: one motor/blower forces air through HEPA filters; another motor/fan causes a suction which draws air from the work area and expels it through a duct system. The use of a damper located in the exhaust duct assures a balance which compensates for any variation in motor speeds. The balance that is maintained makes the open front end possible without permitting outside air to be siphoned into work area or allowing toxic fumes or odors to escape.

8.3.4.1 Limitations. Entrance and withdrawal of large work objects would upset the balance but not enough to cause spill-out. The hood should be adjusted to compensate for the entrance and exit of worker's arms. When dealing with toxic fumes, it is usually the practice to adjust the damper to draw in a slight amount of room air as an extra precaution against fume leakage.

8.3.4.2 Functions. A unit of this type may be capable of controlling:

(a) Noxious fumes.

(b) Airborne particulates.

(c) Temperature.

(d) Humidity.

(See Figure 9.)

8.3.5 Non-laminar airflow work bench. The non-laminar airflow work bench is characterized by non-uniform air patterns and velocities. Although this bench has very limited uses, it does provide a very economical clean area and is satisfactory for certain requirements. For ideal operation, these work benches should be used within a controlled environment. Under these conditions, a degree higher cleanliness level than the surrounding environment may normally be obtained. Class 100,000 is the usual working condition offered by this work station; however, under ideally controlled conditions Class 10,000 may be achieved.

8.3.6 Fume hoods. A fume hood is an enclosure ventilated by a duct to the outside environment with air movement caused by a fan or blower. The hood can be equipped with lights, sliding frame, sinks, and added means to improve airflow. The primary purpose of hoods is to provide a means to rid the atmosphere of odoriferous and toxic materials in forms of fumes, gases, vapors, and particulate dispersions. They are designed to protect personnel working with hazardous materials. The hoods are engineered to confine spills, splashes, reduce cross contamination, and remove heat and humidity from laboratory activities.

8.3.6.1 Standard types.

(a) Conventional. Usually a rectangular compartment vented through openings at top and bottom of rear wall, includes light and may or may not include movable sash.

(b) Streamlined entrance hood. Smooth streamline entrance to reduce face velocity. This type of hood has no corner posts, offsets, or irregular configurations thus promotes a uniform flow of air. Velocities as low as 50 feet per minute have proven to be adequate in low hazard operations. Velocities up to 125 feet per minute have been necessary to prevent spill-out of heavier-than-air gases.

(c) Bypass hood. Provides an air by-pass feature when the sash is in a closed position preventing a "starved" condition for blowers.

(d) Balanced or compensating hood. Designed to induce untempered outdoor air into the hood entrance area, mix it with tempered room air, and pass it into hood opening. This type is usually used in air conditioned rooms as it puts less strain on air conditioning systems.

8.3.6.2 Requirements for operation. There are several observations necessary to maintain performance of hoods:

(a) Do not locate near doorways, open windows, exhaust grills, or in areas that have constant movement. Such movements can cause air disturbances thus causing spill-out of hazardous fumes and materials.

(b) Do not block face of access openings.

(c) Install apparatus centrally under hood in order to achieve maximum airflow.

(d) Completely inform personnel in operating techniques.

(e) Periodically inspect. Retest performance capabilities whenever equipment is moved, structure in which housed is changed, or if there is a change in air conditioning system.

(f) A special filtering or washing apparatus may be required to eliminate emissions of large quanties of toxic, pathogenic, or poisonous materials.

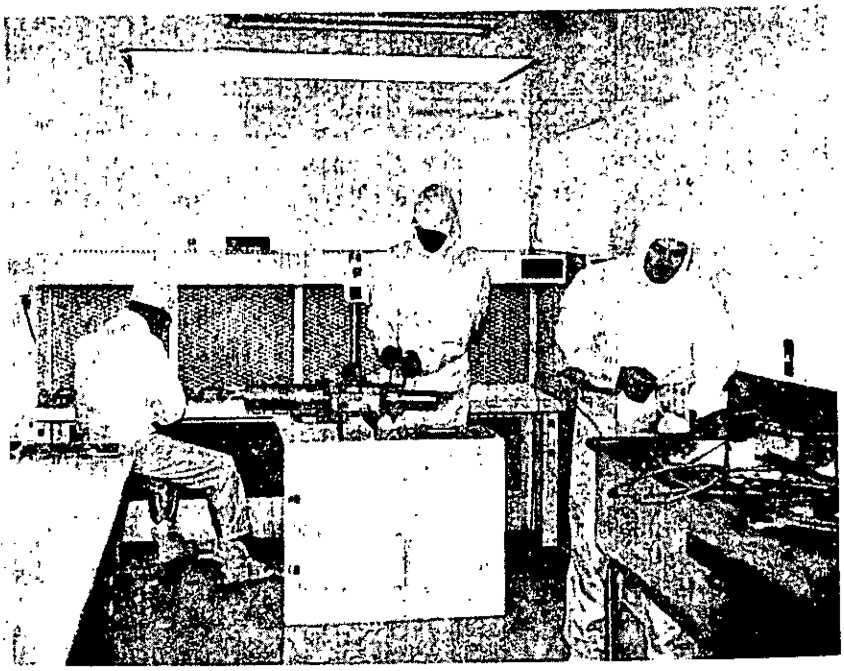

Figure 8. Horizontal laminar flow work bench
Photograph courtesy of Technical Micronics Control, Inc., Madison, Alabama.

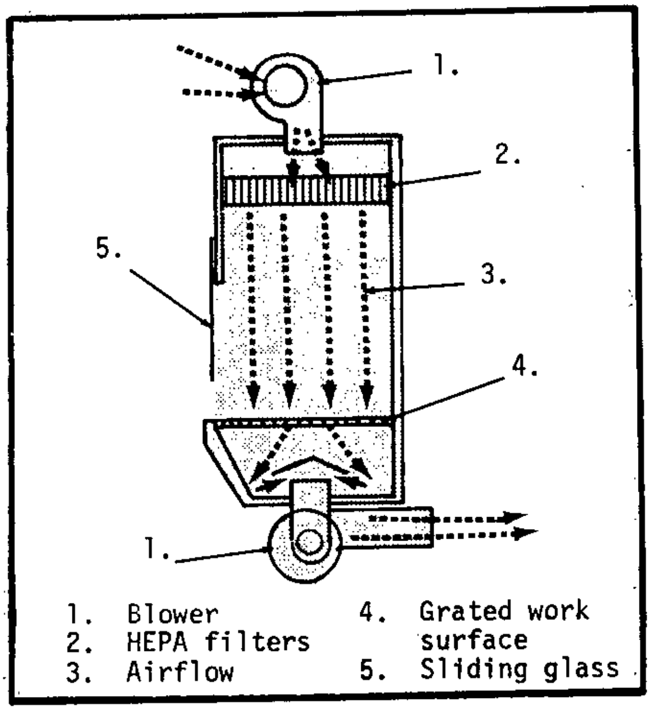

Figure 9. Balanced laminar airflow hood

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SECTION 9

CLEAN ROOM CONSTRUCTION FEATURES AND EQUIPMENT

9.1 Clean room construction features. Regardless of the class, type, or size of the clean room, it is of utmost importance in contamination control operations to construct a facility that restricts the passage of air into or out of a room except through designated plenums or ducts. Consequently, any entrances into the clean room (doors, pass-throughs) must be capable of being airtight during cleaning operations.

A positive pressure is desirable for all clean rooms. Air leakage into the room would signify a lack of positive pressure. The seepage of air out of the room, if in sufficient volume, could induce undesirable eddy currents and upset the desired airflow pattern within the room. Minor air seepage from the room will not usually be harmful. However, if the room is not operated on a continuous schedule these openings could permit contaminants to infiltrate the room during nonoperational hours.

9.1.1 Location. The clean room should be located and mounted so as to be free from as much vibration as is feasible, whether induced by adjacent manufacturing activities or by its own airsupply system. High noise levels are capable of having a detrimental effect on a structure. Sound waves will produce vibration and friction between construction members, which in turn will cause particulate generation.

9.1.2 Construction materials and design features. Although normal construction materials are subject to some degree of expansion and contraction when temperature and humidity varies, the average clean room should not undergo such broad variations as to create an air-leakage problem. It is a factor for consideration, however, when stipulating the materials which will be used in the construction of any clean room.

9.1.2.1 Wood. Specifying wood construction entails the responsibility to ascertain that reasonable age will not cause drying and shrinkage to the extent that openings and air leakage will occur.

9.1.2.2 Dry-wall construction. The use of dry-wall construction for a laminar airflow facility has been demonstrated to be satisfactory if the joints are taped, and a nonshedding or nonchalking surface sealing agent is properly applied. The more exotic and costly stainless-steel interior surfaces will preclude the necessity for wall replacement if the facility is to be long-lived, and will also reduce cleaning, maintenance, and repainting costs for wood or dry-wall construction. Thus, selection of the materials for construction of a clean room must be analyzed in the light of anticipated use, and initial cost versus maintanance cost.

9.1.2.3 Walls and ceilings. Walls and ceilings (nonfilter areas) should be free from ledges and irregularities, and have the following characteristics:

(a) Low-shedding - a sealed, glossy surface is favored.

(b) Easy to clean - washing or vacuuming.

(c) Sealed joints - at corners and between panels.

9.1.2.4 Floors. Floors (excepting perforated or grated) should be covered with a material that is able to withstand traffic wear without undue shedding, can be easily cleaned, and is free from cracks or openings into which particulate matter might lodge.

Floors in vertical laminar airflow facilities may be either perforated or grated metal. Stainless steel is preferred because it wears well, is low-shedding, and resists flaking oxide. This type floor should be constructed in sections, so that they may be raised to permit cleaning of the lower plenum.

9.1.2.5 Plenums and ducting. Provision for ready access to all plenums for cleaning purposes is very important. Periodic cleaning of all closed areas will greatly increase the productive life of the HEPA filters.

It is desirable that ducting be designed and constructed to provide the capability for dismantling quickly for cleaning. The use of accordian, corrugated or other uneven surfaces should not be used for ducting. Plasticized fabric should not be used for ducting material. The interior surfaces of whatever ducting is employed should be as smooth as possible and as free of ridges or ledges as is economically feasible. The attempt to reduce obstructions, however, must not overshadow a need for each duct to be airtight within each section, and at all joints.

The sharper the bend in a duct, the greater will be the noise and heat developed, and this is especially true when the duct size is at the minimum required. It is recommended that bends have a sufficient radius and that the duct size be adequate to permit free flow of the air.

9.1.2.6 Air inlets. Air inlets to controlled environmental facilities should be so located as to ingest clean air without toxic or asphixiating gases. Activities within CEF's should vent or exhaust toxic or asphixiating gases to external atmosphere away from inlets.

9.1.2.7 Airlocks. Airlocks are those double-door entryway facilities required for most nonlaminar airflow rooms which are equipped with interacting locks, and have forced ventilation to provide continuous flushing of the air space within the airlock. Airlocks are not required for laminar airflow facilities.

9.1.2.8 Doors. Doors should be self-sealing when closed, should be equipped with exterior mounted pneumatic closers, and be of an adequate size to accommodate the amount of traffic anticipated. If the traffic through the entrance is to be above normal, and cannot be reduced, consideration should be given to a controlled entrance and exit by providing one door for entrance to the room, and another exclusively for exit.

Pass-throughs should be provided if frequent movement of material will occur into and from the room. Their size will depend on the demands of the product to be handled, and only those which are equipped with an interlocking mechanism would be used.

Unless cleaning of product is to be accomplished within the room, no uncleaned material should be permitted in the room. In any event, the external area into which the pass-through leads must also be at least a controlled area.

9.1.2.9 Anterooms. The extent to which anterooms are provided and equipped depends upon (1) the established and maintained class of air cleanliness of the room, and (2) the planned room occupancy. (The garmenting will have been established by the class of room cleanliness.)

The ultimate would be to provide a HEPA-filtered, contaminant-controlled dressing room, clothing lockers, shoe cleaners, and special washing and warm air drying facilities. It then becomes necessary to determine how much one may retreat from this sophisticated approach to accommodate the needs of the specific room being considered.

Most nonlaminar airflow facilities require the ultimate approach, as described above. Very few full vertical laminar airflow facilities will require anything approaching the control of the described procedure, but each case must be studied and appraised on its own merits. No generalization is deemed practical.

9.2 Clean room furniture. The furnishings and equipment for a clean room should be constructed of materials which are subject to minimal chipping, flaking, oxidizing or other deterioration. Paint should be hard and nonflaking or nonchalking. Any furnishing likely to be subjected to abrasion or bumping should be constructed of either stainless steel or a nonfracture-type plastic. Other than clean packaging films, soft or pliable rubbers and plastics should be excluded.

9.2.1 Chairs or stools. Chairs and stools should not have arm rests, and should be low enough for the workers to rest their feet on the floor.

The feet of the chair or stool should be equipped with stainless-steel or heavy chrome-plated glides; coasters should be prohibited. Any movable parts, whether requiring lubrication or not, should be fully and completely enclosed to preclude the releasing of generated particles, oil droplets, or chips.

9.2.2 Benches and work surfaces. For general applications, a stainless-steel or laminated plastic work surface is desirable. Legs of benches should be constructed of materials which will resist abrading, chipping, flaking or oxidizing, and in addition should be designed to provide a solid nonvibrating work surface. The bench height from the floor should be specifically stipulated at a point which will permit the seated workers to function without undue strain or fatigue, with both feet firmly planted on the floor. Benches should not have foot rests nor bracing, which may be used for this purpose. A 1/4 to 1/2-inch bevel along the front edge of the bench will be beneficial in that it will reduce the opportunity of chipping which may occur if square edges are present.

The benches should be specified as without drawers, but they may have flat metal or laminated plastic trays which pull out from under the work surface.

9.3 Equipment. Very often a specific piece of equipment can be modified so as to place the particulate generating portion of the machine on the outside of the clean room, with just the controls and gages being mounted within the clean room proper. The probability of a slight increase in cost will be more than offset by the reduction in heat and contaminants generated within the room. Easier maintenance is also a factor.

If modification of the equipment is not feasible, then all motors, belts, chain drives, and friction drives must be enclosed to prevent the particulate matter generated from being introduced into the room.

Bulky test, calibrating, and aligning equipment used in the rooms should not be situated facing either the inlet or exhaust filter bank, but rather should be turned 90 degrees to permit the airstream to wash the product when using a horizontal airflow room. The position of the equipment is less critical in a vertical facility. The presence of this type of equipment in a nonlaminar airflow room usually results in the room being "out-of-control" most of the time, due to its low clean-down capability.

9.4 References.

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SECTION 10

AIR FILTERS FOR CONTAMINATION CONTROL FACILITIES

10.1 Characteristics of filters used in clean rooms.

10.1.1 Method of particle entrapment. Air-supply systems normally utilize a filter media of porous fiber material. The fibers may be viscous coated or a dry type. The deposition or entrapment of particulate matter is by centrifugal, electrostatic, and diffusional forces employed in various degrees and combinations. Sieving or straining action is not a major action in this type filter except for large particles on and near the surface of the filter media. The air traversing the narrow winding passage within the porous filter bed is dispersed into many small streams that bring the entrained particulates in contact with filter fibers to which they adhere.

10.1.2 Airflow. Filters differ in the degree to which they impede the flow of air. It may be assumed, as a rough guide, that for equal airflow rates the most effective filters for small particles will be associated with the highest airflow resistances. Filter resistance naturally increases as the filter ages (in use), because the accumulating particulates trapped by the filter progressively impede the flow of air. Eventually, airflow resistance increases up to the maximum capacity of the blower and after this, the total airflow decreases below the minimum required for the facility. When this occurs, the filter must be replaced, or cleansed if possible. The filter resistance is a direct measure of the power required to draw air through it. The airflow resistance of a filter element is a measure of the pressure drop or pressure difference between the input and output sides of the filter. The pressure drop is usually expressed in terms of inches of water as water gage (WG).

10.1.3 Service life. Since filters must be replaced or renewed when their resistance exceeds the capabilities of the air-moving system, service life will diminish by one-half, all other factors remaining the same. Large particles generally cause less filter resistance increase than an equal weight of smaller particles deposited on the filter. This is attributed to closer packing of small particles with a decrease in the size of the air passages. Other factors, such as particle shape, will also affect particle packing characteristics and subsequent resistance increase.

10.1.4 Resistance increase. The thickness and porosity of the dust deposit that builds up on the surface are largely responsible for the resistance increase characteristics of paper and fabric filters. On the other hand, deep bed filters, composed of loosely packed fibers, have large storage capacity between the fibers and, therefore, filter resistance increase is modified by the fiber diameter and fiber arrangement.

Fine diameter flexible fibers compress when the filter resistance exceeds the mechanical strength of the fibers, whereas the beds of large-diameter, stiff fibers remain rigid even when plugged with dirt. When the buildup of dirt is excessive, there is a tendency for dirt to blow through a rigid structure.

10.1.5 Efficiency rating. The efficiency rating of air filters is a measure of particulate matter in the air collected by the filter media. This measure is expressed as a percentage factor based on the method employed in testing the efficiency. The three standard tests commonly used are:

(a) Weight test (synthetic dust). This test compares the weight of a test dust which passes through the test filter with the weight of the dust introduced into the airstream just ahead of the filter. This test is insensitive to fine particles and is not intended for testing high-efficiency-type (HEPA) filters.

(b) Discoloration test (National Bureau of Standards). This test is known as dust stop, photometric or blackness test of atmospheric dust. This test compares the degree of discoloration produced on two filter papers, one through which cleaned air is drawn and the other, uncleaned air. This test is insensitive to fine particles and is not intended for testing high-efficiency-type (HEPA) filters.

(c) DOP test. A diotylphthalate (DOP) smoke is introduced on the upstream side of the filter and the downstream side is measured with an aerosol photometer to determine the total amount of smoke which passes the filter media. The DOP smoke consists of a homogeneous mixture of DOP vapor and clean air in a volume ratio of 1:4. The concentration is adjusted to approximately 40 grains per thousand cubic feet and the particle size to 0.3 micron. A HEPA filter should not permit more than 0.03 per cent of the 0.3 micron size smoke to penetrate through the filter media for a 99.97 per cent efficiency rating.

The differing factors in the test methods do not permit direct comparison of efficiency ratings. The following tabulation lists some typical efficiency ratings for each test and demonstrates the contrast in these rating factors.

Table XVII.

Typical efficiency ratings

Weight Test (%)	Discoloration Test (%)	DOP Test (%)
*	*	99.97
*	90-95	80-85
99	80-85	50-60
96	30-35	20-30
76	8-12	2-5

*Test not practical for more accurate reading.

10.2 Types of air filters. Air filters may be classified into four general categories.

10.2.1 Industrial filters. Designed to handle high dust loadings at low airflow rates. This category normally uses a cloth media formed in bags or envelopes with the fabric either natural or synthetic fibers. This type is used primarily in controlling emissions at the source.

10.2.2 Ventilation filters. These filters are applicable to the handling of medium to low dust loadings at high velocities. These may be the self-cleaning type unit, or may be composed of multiple units ranging in face area upwards from 24 by 24 inches, with depths from 2 to 24 inches. This type of filter may be disposable, or may be manually cleanable. Filtration velocities up to 500 fpm are not uncommon, and airflow resistance seldom goes above 0.25 inch WG, and is often less than 0.1 inch WG.

The filter media in this category includes porous beds of mineral and vegetable fibers, crimped wire mesh, and twisted metal tapes. The dust-catching elements are generally coated with a sticky oil to retain the collected dust particles. These filters are only effective for gross contaminants, and are only to be relied upon to remove about 25 per cent of the atmospheric dust, except in areas of very high concentration.

10.2.3 Intermediate efficiency filters. Designed to handle low-density dust loadings at filtration rates in the range of 30 to 40 cfm per square foot of filter media face area. Higher airflow capacity is achieved by pleating or formation of deep pockets. The majority of filters of this type utilize randomly pocketed batts, or blankets of synthetic resin-bonded glass fibers, ranging in thickness from 1/8 to 1/2 inch.

By varying the diameter of the glass fibers, the thickness of the batt, and the density of the media, wide ranges of dust collecting and holding efficiencies and airflow resistances are possible. The lower efficiency of this type is approximately 35 per cent of atmospheric dust by the discoloration test method, with an initial airflow resistance of about 0.2 inch WG. The highest efficiency normally attainable is about 95 per cent, using the same discoloration test, with an initial resistance of 0.45 inch WG.

10.2.4 High-efficiency particulate air (HEPA) filters. These assemblies use a dry fiber filter media in thin porous sheet form consisting of ultra-fine fibers usually less than 1 micron in diameter. The filter media is normally pleated or fan-folded to form deep pockets in the assembly, to achieve greater airflow capacity.

This type has a minimum efficiency of 99.97 per cent for 0.3-micron particles, as determined by the DOP test method, at airflows of 20 and 100 per cent of the rated flow capacity of the filter. Further description is found in high-efficiency particulate air (HEPA) filters, Paragraph 10.4.

10.3 Selecting air filters. Selecting a proper air filter requires an analysis of the requirements for the particular application. The final selection should resolve answers to the questions posed during the analysis, of which the following are but a few and are typical of the factors to be considered.

10.3.1 Factors to consider.

(a) What is the nature of the air to be filtered (i.e., unfiltered industrial or nonindustrial air direct from outside?)

(b) What percentage will be recirculated?

(c) What is the maximum dirt concentration of that air?

(d) What is the average particulate size? Weight? Mass?

(e) Does the air contain corrosive substances?

(f) Does the air possess any unusual characteristics? (Included would be abnormally high, or low, temperature and humidity.)

(g) How clean must the air be when it issues from the filter face into the work zone?

(h) What is the pressure drop across the filter? (This information will aid in determining the size of the motor and fan, and the power costs for operation.)

(i) What is the required face velocity?

(j) What funding is required for:

(1) acquisition of the completed facility?

(2) annual maintenance costs?

(3) operating costs?

10.3.2 Interrelated factors. Many of the above factors are closely interrelated, and a decision affecting any one of the elements may have a direct bearing on one or more of the others. As an example, capacity and pressure are completely inseparable; efficiency and operating costs also bear on the same considerations.

10.3.3 Procurement specification. Following the determination of which filter is required, the next step is the preparation of a complete and meaningful filter specification for procurement. It must be sufficiently detailed to assure receiving the exact filter needed, and must be the criteria for receiving inspectors to use as a basis for acceptance.

10.3.4 Prefiltration. High-efficiency filters are more costly than those having lesser efficiency ratings, and for this reason, it is normal practice to install some form of prefiltration ahead of the more costly and uncleanable filters to increase their service life. The selection of prefilters is a compromise of prefilter efficiency and cost as related to the replacement cost of the final filter. The contention that a more efficient prefilter will improve the efficiency of the final filter is only partially true. The less efficient filter is also less effective for small particles; therefore, the volume of small particles which it will pass will not be materially diminished by increasing the efficiency and cost.

10.4 Composition of high-efficiency particulate air (HEPA) filters. The HEPA filter uses a media of dry ultrafine fibers (usually less than 1 micron in diameter), which may be 100 per cent glass fiber or a combination of glass and asbestos fibers. This media is formed in a thin porous sheet which is pleated or fan-folded to form pockets, with separators interleaved between the folds to prevent its collapse and to render the maximum area for air filtering. The separators may be made from kraft paper, aluminum, or plastic stock. The media/separator configuration is assembled in a rigid frame. The media surfaces and edges adjacent to the interior sides of the frame are sealed and bonded to the frame with adhesive. The filter frame may be made from:

(a) Plain resin-glued plywood.

(b) Fire retardant-type plywood.

(c) Metal, either steel or aluminum, with hard nonflaking or non-scaling finish.

The depth of the pockets or folds in the media and the size of the frame determine a filter media area and the airflow capacity of the filter assembly. A standard size filter assembly, 24 x 24 x 5-7/8 inches, will provide a minimum airflow capacity of 500 cubic feet per minute (cfm).

10.4.1 Efficiency rating. A HEPA media has a minimum efficiency rating of 99.97 per cent for 0.3-micron particles, as determined by the DOP test method at airflows of 20 and 100 per cent of the rated flow capacity of the filter assembly. This is the only type filter which will provide a supply of air sufficiently free of particulate contaminants to warrant its use for clean rooms and enclosures required to meet the cleanliness levels specified in Federal Standard 209a, Paragraph 5.1. (See Section 4.)

10.4.2 Fragility of HEPA filter. The HEPA filter media is critically fragile. It demands great care in handling during monitoring, installing, removing, and repairing. These filters cannot be cleansed of accumulated contaminants; they must be replaced. The outer face in a filter bank installation should always be protected by a protective screen, as a guard against accidental puncturing of the media. This guard may be hardware cloth, stainless-steel open mesh, or expanded metal lath. The finish of the protective material should inhibit scaling, flaking, or corrosion which may be introduced into the work area. When filter units are shipped or stored for any reason, both of the exposed filter faces should be protected with rigid covers, sealed to the filter frame.

10.4.3 Gasket/fitting. The mounting surface of the frame must be flat, within 1/16 inch, and must be made to accommodate a gasket. The gasket material is usually closed-cell, sponge neoprene, and shall be fitted to the mounting surface of the frame so as to preclude any gaps which might permit air leakage around the gasket when it is compressed. This compression, for the purpose of sealing the opening against the passage of air, will cause the gasket to take a "set" after an indeterminate time; this is sufficient reason to require a replacement gasket whenever the filter is removed for any reason.

10.4.4 Effect of changes in air pressure. The upstream side of the filter is within the air-supply plenum and is, therefore, under air pressure whenever the facility is operational. When the equipment is turned off, the pressure against the filter is released, and the media/separator assembly also relaxes, causing some minor movement in the media. When the equipment is again turned on, the pressure against the back of the filter is reestablished, and some minor media movement again takes place. This movement will quite probably cause some of the fibers to fracture and become loose, and these fragments will be picked up by the airstream passing through the filter. Therefore, it should be standard practice to run any equipment that has been turned off a minimum of 30 minutes before cleansing the filter face guard, the bench tops, or any other surfaces which might be a fallout point for these fibers.

10.4.5 HEPA filters for clean rooms. HEPA filters that will meet the exacting requirements for use in clean rooms and enclosures are available if specifications adequately define the requirements and conditions for use. The manufacturer will leak-test the individual filters by the DOP test method, scanning 100 per cent of the face area, when specified. This 100 per cent scanning test normally costs more, but it provides the user with greater assurance of receiving acceptable filters.

10.5 Air-supply conditioning.

10.5.1 Purpose. The purpose of conditioning the air supply for industrial buildings and facilities is to promote the health, comfort, efficiency and safety of the personnel, and to maintain an environment favorable to the operations, processes, materials, and equipment. To provide these conditions requires the employment of methods to control temperature, humidity, distribution, ventilation, and contamination.

10.5.2 Factors influencing air condition design. Human comfort is influenced principally by temperature, humidity, and velocity of air in the work area. Control of these factors within effective ranges for normal requirements are within the capabilities of a well-designed and engineered air-conditioning system. A system of this type should provide for the exhausting of odorous and toxic fumes and for the introduction of fresh air into the air-supply system. Some control of contaminants may be included, depending on the volume of contaminants in the unfiltered air and those generated within the plant and recirculated throughout the plant area. Air washers, filters, or electric precipitators or combinations of these units are normally adequate to reduce the airborne contaminants to tolerance levels acceptable to the health and safety of personnel.

10.5.3 Special considerations. Special consideration must be given when environmental conditions required for certain operations, processes, material, or equipment are more critical than normally provided. The more critical environmental controls can be provided by establishing zones or areas in which special control methods are employed. Control facilities for the environmentally controlled areas may be auxiliary equipment to the regular system or a completely separate system. Facilities may be required to control:

(a) Temperature, for close-tolerance machining, cold room storage, etc.

(b) Humidity, for minimizing rust and corrosion, eliminating static electricity, etc.

(c) Contaminants, such as aerosols, gases, and vapors.

10.5.4 Control of airborne contaminants. The control or reduction of airborne contaminants in a specially controlled area involves any one or combination of the following contaminant types:

(a) Particles of solid or liquid substances (aerosols).

(b) Vapors of substances which are solid or liquid in their concentrated form and gases not normally found in natural atmospheric air.

(c) Water vapor and gases other than oxygen, nitrogen, and argon in amounts exceeding that normal for natural air are also considered a contaminant in the area of control.

10.5.4.1 Removal of particulate matter. Particulate matter is removed by one or a series of mechanical filters or devices such as:

(a) Filters (fibrous, cloth or viscous).

(b) Impingement filters (wet or dry).

(c) Electrostatic precipitators.

(d) Scrubbers.

(e) Adsorption (within limits of adsorbent).

10.5.4.2 Control of gases and vapors. Gases and vapors are removed by absorption, adsorption, and chemical reaction (catalysis). A typical air-purification system may include the following types of equipment:

(a) Ventilation-type dust filters for reducing high and medium density dust loadings of the air.

(b) Intermediate efficiency-type filters for high velocity particulate filtration of low density dust loading.

(c) Activated charcoal filters remove or reduce concentrations of vapors, fumes, and odors.

The series of filters is followed by the air-tempering equipment for control of temperature and humidity. The dust and high-velocity filters are placed ahead of the charcoal filter to protect it from being overloaded with particulate matter, thereby increasing the service life of the charcoal. This type of system permits total recirculation of air, thereby reducing the operational requirements of the air-tempering equipment and providing adequately clean air for normal operations. The service life of high-efficiency filters used for clean room and clean bench operations is also extended by precleaning the air supply for these facilities.

10.5.5 Selection of filters. The selection of filters for an air-supply purification system involves consideration of a number of factors, in order to provide adequately clean air by the most efficient and economic method. Criteria for filter selection are covered in Air Filters for Contamination Control Facilities, 10.3. The basic considerations are the identification and concentration range of the types of contaminants in the air supply and the cleanliness level required.

10.5.6 Filters for removal of gaseous contaminants. The selection of a filter media for the removal of gaseous contaminants is dependent on the types and concentrations of vapors and odors that must be removed from the air supply. Most industrial adsorbents are capable of adsorbing both organic and inorganic gases. Certain preferential characteristics and other physical properties make each one more or less specific for a particular application. Activated Alumina, silica gel, and molecular sieves are preferential to water and may best be employed in reducing the humidity when required. Activated carbon (charcoal) is preferential to nonpolar organic compounds, but its preferential adsorption properties can be partially regulated by the type of surface oxide induced on the carbon.

10.5.6.1 Selection of absorbents. The following factors are the most important in the adsorption process that must be considered in the selection of adsorbents:

(a) Particle size of adsorbent (may depend on maximum allowable pressure drop).

(b) Depth of adsorbent bed.

(c) Air velocity.

(d) Temperature of airstream and adsorbent.

(e) Concentration of contaminants to be removed.

(f) Concentration of gases not to be removed, including moisture.

(g) Pressure of the system.

(h) Removal efficiency required.

(i) Possible decomposition or polymerization of contaminants on the adsorbent bed.

(j) Regeneration capabilities.

10.5.6.1.1 Activated charcoal. Activated charcoal is the most universally used adsorbent for air purification. It has a greater surface area for a given volume than any other produce currently known. One cubic foot of activated charcoal contains approximately 200 million square feet of adsorptive surface. This tremendous surface area is but one of the factors contributing to the high adsorptive capacity for a wide range of gases normally encountered in air. Other factors include the type of raw material used and the activation process employed to control pore structure and size to some degree. The capacity of activated charcoal for any given gas is dependent on:

(a) Type of activated charcoal used.

(b) Gas being adsorbed.

(c) Other gases present.

(d) Operating conditions: temperature, pressure, humidity, etc.

10.5.7 Common gas and vapor contaminants. Some of the gases and vapors commonly encountered as contaminants in the air supply are listed below. The ones listed as high capacity are those for which activated charcoal has a high adsorptive capacity and are effectively and economically removed by standard commercially available filters. For those listed as low capacity, activated charcoal has a relatively low adsorptive capacity. The initial efficiency for effective removal of both groups of gases is high; therefore, the low capacity group is effectively removed when the concentrations are limited.

Table XVIII.

Common gas and vapor contaminants

High Capacity	Low Capacity
Acetic acid	Ammonia
Acetone	Arsine
Asphalt fumes	Carbon dioxide
Carbon disulfide	Carbon monoxide
Chlorinated hydrocarbons	Formaldehyde
Cyanide	Formic acid
Engine exhausts	Nitric oxide
Hydrochloric acid	Stibine
Hydrogen sulfide	Sulfur dioxide
Methyl ethyl ketone	
Nitric acid	
Oil vapor	
Phosgene	
Phosphine	
Smog	
Tobacco smoke vapor	

Special techniques are employed to increase the capacity and effectiveness of charcoal for removing specific gases or vapors. Such techniques include the impregnation of the activated charcoal with additives and the employment of equipment for in-place regeneration of the charcoal.

10.6 References.

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SECTION 11

TEMPERATURE AND HUMIDITY IN CLEAN ROOM FACILITIES

11.1 Factors influencing selection of temperature and humidity limits. The selection of temperature and humidity limits is both product and personnel oriented. The best conditions for the product are major considerations, but if these conditions are uncomfortable or annoying to the personnel, some compromise may be possible in some instances.

11.1.1 Location. Geographic location will obviously impart some influence on the final selection because the adding or removal of heat in areas of wide temperature extremes is an important cost item. Maintaining certain humidity ranges can be equally expensive.

11.1.2 Amount of air. The amount of makeup air is an element which also must be considered, so the decision is not only one of temperature and humidity but also of the degree of change and the gross volume.

11.1.2.1 *Air volume in non-laminar and laminar facilities.* The cubic volume of air requiring treatment for a nonlaminar airflow facility will be approximately 25 percent of that necessary for a laminar airflow installation of comparable size. However, the dwell time of the air in the nonlaminar room is at least three times greater than in a laminar-type room. The reduced dwell time will be reflected in a smaller temperature and humidity variation because the exposure to heat gain or loss is appreciably reduced. It is true that a larger volume of air will be passed through the treatment area, but it will require less power to return it to its stipulated level.

11.1.3 Heat. All sources of heat must be considered in calculating the cooling capacity needed for the facility. Two sources that are sometimes overlooked in clean room design are: (a) the heat generated by the motor/blower operation, and (b) the heat released by moving air, which was originally in the form of energy imparted to the air by the blower. Other sources such as people in the room and heat-generating operations must also be considered.

11.2 Temperature control. Temperature control in clean room areas *should be adequate to maintain a nominal* temperature of 72° F, except for those laboratory or work areas where different temperatures are necessary for product stability, in which event the required temperature should be specified. The range of temperature variation required at the control point may be 0.25° F in the most critical areas and 5.0° F in normal clean room areas.

11.2.1 Temperature monitoring. Temperature monitoring should be accomplished by conventional devices, the simplest and most accurate being a calibrated Fahrenheit thermometer. Automated devices also may be employed, but they should be checked against the thermometer at least once each 24 hours.

11.2.1.1 Nonrecording automated equipment. If nonrecording automated equipment is used, manual recording of the temperature readings should be made, just as in the case when only a thermometer is used. These readings should be taken at least once each hour, and more often if it is obvious that a radical change in the room temperature has taken place.

11.2.1.2 Automated recording devices. In the areas where a temperature-sensitive product is being processed or tested, or where exacting tolerances are required, automated temperature recording devices are recommended. When this circumstance prevails, a temperature probe should be located in the immediate proximity of the product. It is also advisable to provide an alarm or warning system that will signal temperature variations which exceed either the high or low limit.

11.3 Humidity. The requirement for relative humidity within any stipulated range should be based primarily on product needs. Relative humidities (RH) above 50 percent may introduce problems, such as rusting of parts and equipment. When abnormally high relative humidities are mandatory, the HEPA filters should have metal separators, and the filter media may deteriorate more rapidly. In addition, trouble may be encountered with the wooden supporting frame through warping and separation of the plywood.

11.3.1 Minimum RH value. A minimum value of 30% RH should be observed to preclude electrostatic charges on dielectric materials and parts. Such charges can cause serious problems in particle attraction or with explosive components.

11.3.2 Humidity monitoring. The simplest method for monitoring the relative humidity level is to use a conventional wet and dry bulb thermometer in conjunction with a psychometric chart.

Automated devices may be used to supplement the wet and dry bulb thermometer, but not to replace it. If automated equipment is employed, it should be checked against the thermometer at least once each week, or more often, if significant variations appear in the readings.

11.4 References.

1. Contamination Control Handbook, Sandia Laboratories, NASA SP-5076, 1969.

SECTION 12

CLEAN ROOM GARMENTS

12.1 Primary purpose for garmenting. The primary purpose for garmenting clean room workers is to contain as much of the particulate matter brought into the area on the person and attire of the worker as is possible. Secondarily, an adequate garment will also contain the particulate matter generated from the person and attire during the period that the garment is worn. This secondary condition is evidence that once a garment is worn, it will have particles of contamination on the inside, and unless the fabric weave is sufficiently impenetrable, it too will emit particles.

12.2 Fabrics. Selection of the fabrics used in the garments, whether purchased or rented, should be specified in detail, with the burden of proof of the contents of the fabrics resting upon the supplier of the fabrics. Not all fabricators of clean room garments maintain a laboratory for the purpose of ascertaining and certifying that they are really using the type of material specified. Cotton and linen outer garments should be excluded from a tightly controlled clean facility because of their high linting characteristics and low abrasive resistance.

12.2.1 General description of some synthetic fabrics. Following is a general description of readily available synthetic fabrics, and some specific characteristics of each:

(a) Nylon. 100-percent multiplex continuous filament, taffeta weave, having superior abrasive resistance, high absorbency, minimum linting, and outstanding wearing qualities.

(b) Nylon. 100-percent continuous filament yarn, taffeta weave, is long wearing, and has very good abrasive resistance.

(c) Nylon. 100-percent double-knitted monofilament yarn is highly pourous, very high in abrasive resistance, and has superior absorbent qualities. Used for wiping cloths.

(d) Nylon. 100-percent monofilament, lightweight tricot knit makes an excellent glove fabric. It is inexpensive, high in porosity, minimal absorbency, and is extremely resistant to linting.

(e) Nylon. 100-percent monofilament base coated with a thin film of polyvinyl chloride is completely nonporous, has no absorbancy, and was specifically developed for gloves to prevent transfer of skin contamination. Cannot be dry cleaned, and is sensitive to acetone, perchloroethylene, and similar solvents.

(f) Nylon. 100-percent continuous filament, very tightly woven taffeta, fine denier fabric, calendered, is nonporous, nonabsorbent, with high abrasive resistance. Designed to be LOX-compatible.

(g) Nylon. 100-percent stretch tricot, specifically designed for gloves. It is sensitive to abrasion, and moderately absorbent.

(h) Dacron Polyester. 100-percent polyester continuous filament yarn, plain taffeta weave, is an opaque tightly woven fabric with long wearing and acid-resistance characteristics.

(i) Dacron Polyester. 100-percent polyester continuous filament yarn in a herringbone twill to provide body and weight. It is opaque, very long wearing, very tough and sturdy, and acid resistant.

12.2.2 Combination fabrics. In addition to the above synthetic fabrics, there are a large number of combinations of dacron and viscose rayon types, which are somewhat cheaper per yard than the straight synthetic. It should be remembered that when viscose rayon is added to other yarns to produce a fabric, some of the strength is sacrificed when the fabric encounters moisture. The addition of viscose rayon will reduce the generation of static electricity, with the degree of reduction related to the percentage of rayon in the fabric.

12.3 Garment construction. All garments should be sewed with thread of 100-percent polyester continuous filament, stranded, 200 denier minimum, and for appearance, should be the same color as the garment.

All seams must be closed, double-stitched, and free of loose threads.

Garments should not have pockets. The garment should be equipped with adjustable collars and cuffs, which have stainless-steel fasteners.

12.4 Soiling and laundering. How frequently should garments be cleaned or laundered? Arriving at a good answer requires consideration of the following conditions, all of which may contribute to the contamination of the garment:

(a) Rate of personal emission varies from one individual to another.

(b) Amount of physical movement by the wearer may vary among individuals having the same work assignment.

(c) Heat and cold environments will create particulate generation rates from high to low from different persons.

(d) A high incidence of particulate generation will occur from tweeds, bulky sweaters, starched cotton, and linen fabrics.

(e) Contamination which is acquired from the operation or tools used on the job.

12.4.1 Anti static treatment. Anti static treatment applied at end of laundering cycle will reduce static electricity.

12.4.2 Fireproofing. For certain specialized activities, especially where high concentrations of oxygen gas may be present, it is recommended that garments be fireproofed.

12.5 Available garments. Garments currently available for controlled environment use are as listed:

(a) Head covers: snoods, hoods (eye and face opening).

(b) Body covers: smocks, coveralls, coveralls with hoods.

(c) Foot covers: foot socks, booties.

(d) Gloves: latex, nylon (woven), nylon (film), polyethalene.

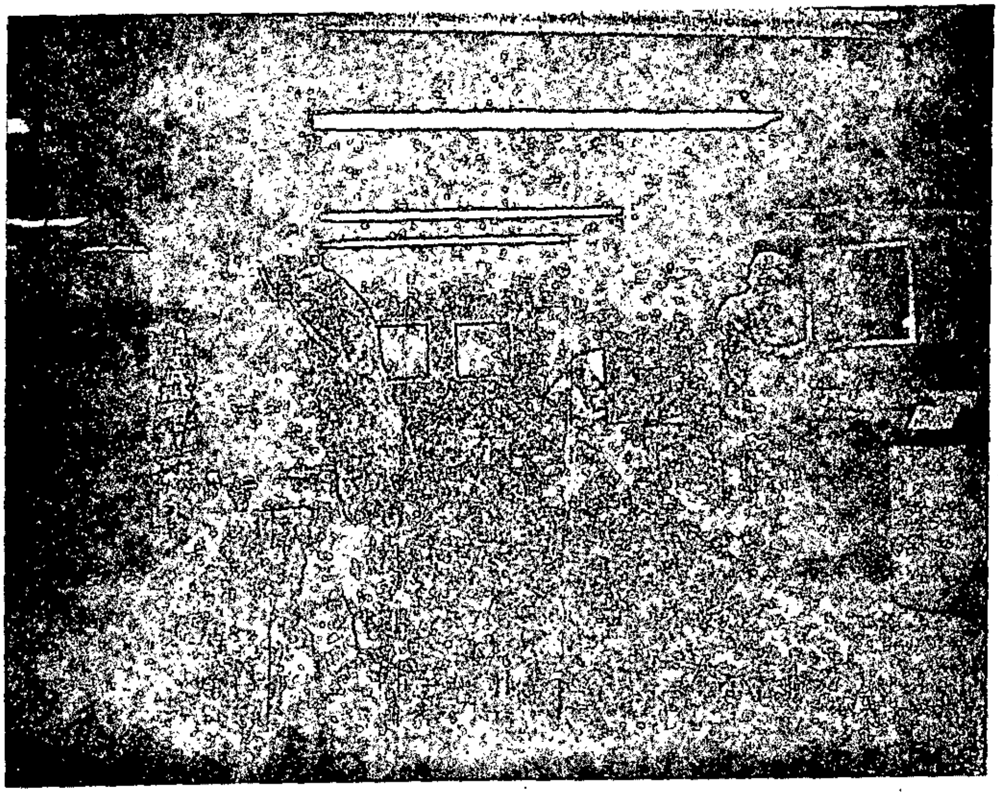

Figure 11. Garments for clean rooms
(Photograph courtesy of Technical Micronics Control, Inc., Clean Room Operations, Madison, Alabama.)

12.6 Garment specification. A garmet specification that is used in NASA programs is MSC-SPEC-C-2A, General Specification for Clean Room Garments.

12.7 References.

1. Contamination Control Handbook, Sandia Laboratories, NASA SP-5076, 1969.

2. Marquart Corporation.

3. Grumman Aerospace Corporation.

4. Technical Micronics Control, Inc., Madison, Alabama.

SECTION 13

AIRBORNE CONTAMINATION

13.1 Natural atmospheric air. Near the surface of the earth the natural atmospheric air is composed of gaseous elements, water vapor, and some particulate matter generated from natural sources. The concentration of water vapor or humidity will vary from one region to another and within any period of time. Particulate matter, both quantity and type, will vary considerably as affected by the natural conditions in a region.

Table XIX.

Composition of natural air

Constituent	Approximate Concentration	
Nitrogen (N_2)	78.03	volume per cent
Oxygen (O_2)	20.99	volume per cent
Carbon dioxide (CO_2)	0.03	volume per cent
Argon (A)	0.94	volume per cent
Neon (ne)	0.00123	volume per cent
Helium (He)	0.0004	volume per cent
Krypton (Kr)	0.0005	volume per cent
Xenon (Xe)	0.000006	volume per cent
Hydrogen (H_2)	0.01	volume per cent
Methane (CH_4)	0.0002	volume per cent
Nitrous oxide (N_2O)	0.00005	volume per cent
Water vapor (H_2O)	Variable	
Particulate matter	*Variable type and quantity	
Ozone (03)	**Variable	
Formaldehyde (HCHO)	***Uncertain	

*The types and concentrations of these substances may vary substantially from one region to another and within any period of time from natural conditions.

**From ultraviolet radiation and probably thunderstorms, concentrations will vary from 0 to 0.07 ppm.

***From biological sources or oxidation of CH_4; possible concentrations not determined.

13.1.1 Effect of human activities on natural air. The composition of natural air is substantially changed by human activities. The industrial, commercial, and domestic activities of man produce an ever increasing volume of gaseous components and particulate matter emitted into the atmosphere to pollute the air. The wide variety of contaminants produced by human activities results in changes to the concentrations of gaseous constituents of natrual air, adds a variety of new gaseous constituents, and adds a wide variety of types and quantities of particulate matter.

13.1.2 Airborne contaminant. If an airborne contaminant is defined as any airborne substance that adversely affects the contaminee, all components of the ambient air must be considered as possible contaminants. In some instances, only particulate matter of substantial size may be a contaminant; whereas, in other instances virtally all components of the air may be contaminants and must be excluded in some manner.

13.1.3 Natural and foreign airborne contaminants. Normal atmospheric air is composed of both natural air and foreign constituents. The volume of various classes of contaminants that make up the foreign constituents of atmospheric air determines the pollution level of air as it affects physiological reaction, toxicity, visibility, damage to vegetation, and soiling and damage to materials exposed to air. Pollution of air is usually the result of some activity which emits undesirable substances into air in either one or a combination of the physical states classified as "gaseous" and "nongaseous." Sources of the contaminants polluting the air are the activities taking place on the surface of the earth and are divided into two broad classes: natural and man-made.

13.1.3.1 Natural sources. Natural sources result from physical and biological processes in the earth's environment and include dusts or minerals ventilated from the soil, ash from volcanic eruptions, salt-water crystals blown from the surfaces of large bodies of water, and airborne bacteria and pollen spores from a variety of plant life. The atmosphere also absorbs quantities of solar and cosmic radiation from outer space.

13.1.3.2 Man-made sources. Man-made sources result from the varied activities of modern civilization. These involve industrial operations, vehicular, marine and aerial forms of transportation, distribution of raw materials and manufactured products, centralization of power and heat sources, together with all services necessary to the construction and maintenance of a community. The level of pollution and the classes and concentrations of various contaminants will vary widely with different geographical areas or zones. These are influenced by the air-pollution potential from the sources in the air-pollution zone, and the saturation potential of the air space over the zone.

13.1.4 Saturation potential. The saturation potential is the capacity of the air space which envelopes a pollution zone to absorb, build up, and retain the air contaminants to levels which adversely affect the areas as a whole. The saturation potential is normally realized when the rate of evacuation in the air space is less than the rate at which pollutants enter the air space. The rate of evacuation in the air space is controlled principally by the variable meterological conditions in the area; the mechanisms of evacuation are:

(a) Dilution or diffusion with uncontaminated air, facilitated by agitation or turbulence of the airflow.

(b) Dispersion of the entire polluted air vertically or horizontally, facilitated by movement of air masses.

(c) Precipitation by entrainment or nucleation of particles by water droplets in rain or fog.

(d) Normal gravity fallout.

13.1.5 Air pollution potential. The air pollution potential of a zone is the capacity for the various sources in the areas to emit contaminants in significant volume called primary contaminants, together with the capacity for these primary contaminants to interact or be modified by natural forces in the atmosphere resulting in secondary contaminants. The diversity and complexity of the sources capable of emitting such a wide variety of primary contaminants coupled with the secondary action result in virtually all chemical substances and derivatives or oxidation products known to chemical technology, and perhaps include some that are unknown. To determine what contaminants are in the air requires a sampling of the air to identify and measure the contaminants and an investigation of the sources to identify and measure the volume of the primary contaminants being emitted into the air.

The significance of the pollution of atmospheric air with its inherent variety of contaminants is principally the effect such contaminants have on a specific contaminee, including the effects on the environments in which they are found.

13.2 Classification and sources of airborne contaminants. The activities of man are the major potential source of airborne contaminants; and the greatest potential for controlling this pollution is at the source. Classification of the various types of contaminants as related to the principal sources is essential in establishing measures for controlling air pollution.

13.2.1 Contaminant classification. Classification of the various types of contaminants is essential in establishing measures for controlling air pollution. Airborne contaminants are classified by physical state as gaseous and nongaseous. Those in the gaseous state are further classified as "organic gases" and "inorganic gases." Those in the nongaseous state are known as "aerosols" and include all solid and liquid particulate matter.

13.2.1.1 Organic gases. Organic gases are compounds composed of atoms of carbon to which may be attached atoms of one or more of the elements such as hydrogen, oxygen, sulfur, nitrogen, or a halogen.

13.2.1.2 Inorganic gases. Inorganic gases result primarily from combustion reactions with oxides of sulfur and carbon monoxide formed by the oxidation of sulfur and carbon in combustible materials, and oxides of nitrogen being formed by the fixation of nitrogens at the high temperatures encountered in the combustion process.

13.2.1.3 Aerosols. Aerosols in liquid and solid form are emitted from a variety of sources both manmade and natural which include smoke, ash, soot, oil mists, and dusts and pollens in the natural state.

Table XX.

Classification of airborne contaminants

Major Classes	Subclasses	Typical Members of Subclasses
Organic gases	Hydrocarbons	Hexane, xylene, ethylene, penthane, butane, butadiene, methane
	Aldehydes and ketones	Formaldehyde, acetone
	Other organics	Chlorinated hydrocarbons, alcohols
Inorganic gases	Oxides of nitrogen	Nitrogen dioxide, nitric oxide
	Oxides of sulfur	Sulfur dioxide, sulfur trioxide
	Carbon monoxide	Carbon monoxide
	Other inorganics	Hydrogen sulfide, ammonia, chlorine
Aerosols	Solid particulate matter	Dusts, smoke, fumes, ash, soot
	Liquid particulate matter	Oil mists, entrained liquid droplets.

13.2.2 Contaminant sources. The diverse activities of man and nature which produce the contaminants discharged into the atmosphere are classified into eight general categories. The commercial or industrial activities such as production and manufacturing operations are the most significant sources of air pollution. However, the individual activities of man contribute heavily to the atmospheric pollutants, especially in densely populated areas.

13.2.2.1 Petroleum industry. This operation constitutes a significant potential for air pollution emissions, including all classes of contaminants, from the production of crude oils to the marketing of finished products.

Crude oil production encompasses those operations employed in obtaining the petroleum from the well, measuring, storing, and transferring the oil to the refinery. The contaminants consist chiefly of escaping natural gases and evaporated hydrocarbons (ethane and methane) with some carbon monoxide, oxides of sulfur and nitrogen and aerosols emitted from internal combustion engines and other combustion equipment associated with the operations.

Refinery operations and equipment include fluid catalytic cracking units, hydrocarbon storage, effluent waste disposal, flares, desulfurization and sulfur scavenging equipment. The contaminant emissions from these operations include all classes and subclasses with the major volume being hydrocarbons, oxides of sulfur and nitrogen, and carbon monoxide.

Marketing and distribution of petroleum products comprise extensive facilities such as pipelines, terminals, tank trucks, and storage and loading equipment. Contaminant emissions are chiefly hydrocarbon vaporsfrom storage tanks and transfer operations with some additional emissions from liquid spillage and from pumps and separators.

13.2.2.2 Metallurgical industry. This activity includes metal melting of both ferrous and nonferrous metals. The ferrous group includes gray iron foundries, steel foundries, and secondary steel mills. The nonferrous group includes foundries making castings of brass, bronze, aluminum, zinc, lead, and magnesium, as well as secondary smelters used in the recovery of these metals. Air contaminating emissions from these operations consist principally of aerosols including smoke, dusts and metallic fumes, with some gaseous contaminants being emitted from combustible materials burned in the processes. The volume of gaseous contaminants in some instances is quite negligible and depends on the methods used in melting the metal, the type of metals, and contaminants or impurities in the metal.

13.2.2.3 Organic solvent usage. Organic solvents are those organic liquids capable of being evaporated into the atmosphere during usage or storage. They are used to dissolve, dilute or disperse other substances without themselves chemically reacting with the other substances. The evaporation emissions of solvent vapors include all members of organic gas contaminants dependent upon the type of solvent, with the volume of vaporic emissions dependent on the volume of atmospheric exposure. Major sources may be identified by the types of equipment used which expose large volumes of solvents to vaporization. This equipment includes:

(a) Solvent storage tanks.

(b) Solvent degreasers.

(c) Spray booths.

(d) Coating, baking, and drying ovens.

(e) Dry-cleaning equipment.

(f) Solvent mixing and dissolving tanks.

(g) Solvent crystalizing equipment.

13.2.2.4 Mineral processing. The major activities in this category include asphaltic concrete batching, concrete batching, glass container manufacture, ceramic glaze and enamel frit manufacture, mineral insulation manufacture, and many other mineral processing activities. The principal contaminants released by these activities are in the aerosol class, primarily dusts and fumes from such processes as crushing, grinding, milling, conveying, bagging, mixing, and blending. Some inorganic gas contaminants will be included in some activities from combustible materials burned in furnaces, heaters, and power equipment used in conjunction with the process.

13.2.2.5 Motor Vehicles. This category involves primarily the operation of vehicles powered by gasoline or diesel engines. The exhaust from these vehicles is responsible for the largest volume of contaminants, principally carbon monoxide, hydrocarbons, and oxides of nitrogen with some aerosol emissions of smoke and carbon soot. The blowby or leakage of unburned gasoline into the crankcase contributes a substantial amount of hydrocarbon emissions with some carbon monoxide. The evaporative emissions from the fuel in the carburetor and fuel tank are principally hydrocarbon and some other organic gases. Additional particulate contaminants are emitted in the normal operation of vehicles from the cooling and braking systems to the scrubbing of tires on road surfaces.

13.2.2.6 Combustion processes. This includes those activities utilizing various fuels in a combustion reaction to produce heat and power for both domestic and commercial use. Such activity and equipment range from home space heaters and kitchen stoves to large steam electric power plants, and from the burning of combustible wastes and refuse in incinerators to open burning. The combustion of fuel in the various types of equipment to produce heat and power generates a variety of air contaminants in all classes. The composition of the fuel burned and the efficiency of the equipment used have a decisive influence on the types and quantities of air contaminants emitted. However, the principal contaminants in most operations are hydrocarbons, aerosols, and oxides of nitrogen and sulfur. The principal contaminants from incinerators and open burning are the same as for fuel burning equipment plus considerable quantities of carbon monoxide.

13.2.2.7 Other industry. This includes all other industrial and commercial activities that are potential sources of air contaminants and are capable of emitting contaminants of significant magnitude. Some of these are manufacturers of paints, varnishes, synthetic resins, and asphalt and the operation of spray booths, protective coating bake ovens, rendering plants, coffee processing, and feed and grain plants.

13.2.2.8 Natural environment. This category covers the natural sources of contaminants such as plants and animals. The principal class being aerosols such as pollens, spores, plant fibers, insect and animal parts, and bacteria and viruses. Table XX lists sources of contaminants.

Table XXI.

Sources of contaminants

	Organic Gases			Inorganic Gases				Aerosols
	Hydrocarbons	Aldehydes and Ketones	Other	Oxides of Nitrogen	Oxides of Sulfur	Carbon Monoxide	Other	Liquid and Solids
Petroleum								
Refining	#	#	N	#	#	#	#	#
Marketing	#	N	N	#	N	N	N	N
Production	#	N	N	#	N	N	N	N
Metallurgical								
Nonferrous	N	N	N	N	#	N	#	#
Ferrous								
Grey iron	N	N	N	N	N	#	N	#
Electric steel	N	N	N	N	N	N	N	#
Open hearth	N	N	N	N	N	N	N	#
Organic solvent								
Surface coating	#	#	#	N	N	N	N	#
Dry cleaning	#	#	#	N	N	N	N	N
Degreasing	#	#	#	N	N	N	N	N
Mineral								
Asphalt batching	N	N	N	N	#	#	N	#
Concrete batching	N	N	N	N	N	N	N	#
Glass and ceramic	N	N	N	N	#	#	N	#
Insulation	N	N	N	N	N	#	N	#
Motor vehicle								
Exhaust	#	N	N	#	#	#	N	#
Blowby	#	N	N	N	N	#	N	N
Evaporation	#	N	N	N	N	N	N	N
Combustion								
Heaters and boilers	#	N	N	#	#	N	N	#
Incineration	#	N	N	#	#	#	N	#
Other industry	#	N	N	#	#	#	N	#
Natural environment	N	N	N	N	N	N	N	#

- Significant Volume
N - Negligible Volume

13.3 Control techniques for atmospheric air contaminants. The capacity to prevent air pollution in a specific area is limited to reducing the potential sources through one of the following methods:

(a) Eliminating the source.

(b) Controlling source area pollution by zoning.

(c) Controlling or changing the sources.

13.3.1 Elimination. The elimination of all sources of air pollution may not be practicable or possible. Specific instances of eliminating nonessential activities or substitution of equipment or processes may be applicable.

13.3.2 Zoning. Zoning or placement of potential sources in specified areas to minimize the effects of air pollution can be quite effective. However, this method requires advance planning or movement of source activities which may not always be practicable.

13.3.3 Controlling. Controlling or changing the source may be accomplished by any one or any combination of the following actions:

(a) Institute operational and maintenance techniques to rigidly control the handling of fuels, materials, and equipment to minimize contamination emissions.

(b) Incorporate design features in equipment to efficiently utilize all materials and fuels to prevent contamination emissions.

(c) Replace or alter the composition of fuels or materials to reduce or eliminate those volatile or impure elements which cannot be utilized or collected by the equipment.

(d) Install control or collection devices which will either eliminate the contaminant through combustion or chemical reaction or collect the contaminant by entrapment.

13.3.3.1 Current air pollution control techniques. The current air pollution control technology provides a wide variety of techniques and devices to virtually the whole range of industrial activities with a considerable economic choice in the selection of suitable control methods. Most of the air pollution problems can be controlled through available techniques. Some problems still require further engineering development for an adequate solution.

13.4 Air pollution control devices. When the design and operational efficiency are inadequate to control the emissions of air contaminants, specially designed control equipment becomes necessary. Control equipment is divided into two classes, those which collect or control aerosols, those which collect or control gases.

13.4.1 Aerosols. Those which collect aerosols, solid and liquid from particulates, by one or more of the following principles:

(a) Inertial entrapment by altering the direction and velocity of the effluent.

(b) Increasing the size of the particles by agglomeration or liquid mist entrainment so as to subject the particles to inertial and gravitational forces within the operational range of the device.

(c) Impingement of particles on impact surfaces, baffles, or filters.

(d) Precipitation of contaminants in electric fields or by thermal convection.

Table XXI lists the types of devices used in controlling aerosol contaminants with some examples of the contaminants controlled and the principle of operation.

13.4.2 Gases. Those which collect or control gases by one or more of the following principles, and which are based on the physical and chemical properties of the individual gases:

(a) Combustion of volatilized gases.

(b) Absorption into reactive solutions.

(c) Absorption to an active surface area of a solid.

(d) Condensed through temperature and pressure changes.

(e) Reduced to carbon dioxide and water through high-temperature controlled combustion.

(f) Catalyzed to harmless substances.

(g) Converted by chemical reaction to by-products.

(h) Conserved and reused by means of vapor collection.

Table XXII lists the types of devices used in controlling some of the gaseous contaminants with some examples of the contaminants controlled and the principle of operation.

Table XXII.

Air pollution control devices for aerosols

Type	Principle of Operation	Examples of Contaminants Controlled	Remarks
Settling chamber	Compartment permitting gravity settling of dusts	Wood, grain, mineral dust	Simple design, low efficiency, used as precleaner.
Centrifugal collectors (dry)	Single cyclone	Wood, grain, mineral dusts	Simple construction, low efficiency.
	Multiclone	Catalyst dust, fly ash	Relatively high efficiency
	Impeller	Foundry dust	High efficiency, small space required.
Filters (fibrous, cloth or viscous)	Tubular (bag house)	Metallurgical fumes and fine dusts	High efficiency over wide range of particle size.
	Screen or frame	Ceramic dust, foundry dust	For small or intermediate size operations.
	Tubular bag with reverse jet cleaning	Carbon black, flour dust, grain dust	High filter ratios but bag wear increased.
Electrical precipitators	Single stage (high voltage)	Metallurgical fumes, catalyst	High efficiency under severe conditions, high cost.
	Two stage (low voltage)	Oil and acid mists	High efficiency, limited use.
Wet collectors (scrubbers)	Spray chamber	Rock dust, acid mist	Limited use, high nozzle presure.
	Centrifugal	Rock and sand dusts, some mists	Many variations in design.
	Inertial	Grinding dust, foundry dust	Similar to cyclone with liquid tangential sprays.
	Venturi	Chemical fumes, acid mists	High efficiency, high first cost. High velocity carrier stream through venturi.

Table XXII-A.

Air pollution control devices for gases

Type	Principle of Operation	Examples of Contaminants Controlled	Remarks
Absorbers	Packed towers	Malodors from rendering and chemical plants.	Solutions - oxidizing agents.
	Plate towers	Gases and vapors from refinery and chemical plants.	Solution - absorption oil (oil is stripped and recirculated).
	Spray towers and chambers	Hydrogen sulfide from thermal and catalytic cracking plants.	Solutions - ethanolamines, thylox, potassium phosphate, sodium phenolate, (Solutions are regenerated.)
		Sulfur dioxides from flue gases, chemical plants.	Solution - water solutions of sodium sulfite, ammonium sulfite and sulfate (replenished), dimethylamine (regenerated).
		Nitric acid from chemical plants.	Solution - alkaline solution.
Absorption	Condensation on surface of a solid	Organic solvent vapors and malodors.	Absorbents - activated charcoal, silica gel, activated alumina.
Incineration	Flares	Hydrocarbons from refineries and oil fields.	Venturi burners or steam injection for smokeless combustion.
	Fume burners	Hydrogen sulfide from refineries and chemical plants.	Sulfur dioxide is product of combustion.
		Gases and malodors from chemical plants, refineries, food processing.	High operating temperatures 900 to 1600 F.
	Catalytic combustion	Organic vapors, carbon monoxide, oil vapors, ammonia	Catalysts - platinum, nickel operating temperatures 600 to 900 F.
Vapor recovery	Vaporshpere collection	Gasoline, crude oil, other volatiles from storage tanks.	Vapor may be compressed and liquefied, or burned as fuel or flared.
Floating roofs	Reduces tank breathing losses	Gasoline, crude oil, other volatiles from storage tanks.	Closure seals required for good efficiency.

13.5 Detecting and measuring atmospheric air contaminants. Some contaminants may be visible in the atmospheric air in the form of smoke or smog. However, when the density of such contaminants is below 40 per cent capacity (No. 2 Ringlemann chart) or the contaminants are of a transparent nature, their presence is not readily apparent.

Specially designed equipment and skills for testing and measurement are required to determine what contaminants are present and what the levels of concentration are in a specific area. Within a specific geographic area or community, some information in this regard may be obtained from the federal and local agencies employed in air pollution control. Information from these agencies would include sources of air pollutants, types of contaminants and rates of emission, and types and concentrations of contaminants at air sampling stations in the area.

13.5.1 Factors to be considered in on-the-spot testing. Identifying and measuring the concentration of contaminants in a specific location or point within a plant or facility may be accomplished by on-the-spot testing should include:

(a) Variations in concentrations due to varying meteorological conditions.

(b) Hours of operation of source-emitting activities.

(c) Potential sources in the immediate vicinity.

(d) Specific types of contaminants most undesirable.

13.5.2 Air sampling and test methods. Air sampling and test methods are divided into two groups: methods applied to particulates, methods applied to gases.

13.5.2.1 Methods applied to particulates. The effectiveness of testing for particulate matter may be affected by solubility, or organic or inorganic nature of the material. However, the principal measurement parameters are size and volume or quantity. Particulate sampling devices employ the same principles used in the various types of source control equipment used by industry, but the sampling devices must be highly efficient for all particle sizes. Table XXIII lists the types of sampling devices commonly used, with their principle of operation, and some information on efficiency of collection. These devices are also used for sampling the effluent at the sources of emission.

13.5.2.2 Methods applied to gases. The testing for gaseous contaminants consists primarily in separating the contaminant gas from other gaseous media (air). Sampling techniques for gases are adapted to either the sampling of a specific gaseous compound or determining gross contaminant concentrations. Specific methods are generally used for inorganic gases, and, in most cases, involve an absorption method by bubbling through a reactive liquid agent or by exposing the contaminated air to impregnated papers and granules for colorimetric reaction. For sampling mixtures of contaminated gases, the freeze out, absorption, and grab sample techniques may be used. Table XXIV lists the types of sampling devices commonly used, with principle of operation, and some information on efficiency of collection.

13.6 Control techniques for airborne contaminants within controlled environments. The production of precise, highly reliable devices requires exacting control of the environments for manufacturing, assembly, storage, and operation of these devices. The environmental conditions relating to the ambient air that require positive control within certain predetermined limits include temperature and humidity, and airborne contamination. Facilities for the control of temperature and humidity that are currently available normally provide the controls adequate to the needs of the product. (See Section 11 for further information on temperature and humidity control.)

13.6.1 Major sources of contaminants within a controlled area. The development of facilities and equipment to control airborne contamination within a controlled environment area requires consideration of the major sources of contaminants. These include the air supply and activities within the controlled area.

13.6.1.1 Air Supply. The ambient air prior to its introduction into the controlled area must have all contaminants removed or reduced to some predetermined level of cleanness consistent with the requirements of the operation being performed. Methods and facilities that may be used to cleanse the air of gaseous contaminants and the filtration of particulate matter to desired levels are covered in Section 10.

13.6.1.2 Activities within the controlled area. A wide variety of contaminants may be generated and emitted into the otherwise clean airstream within the controlled area from numerous sources which include personnel, tools and equipment used, and the various operations performed. Some of the major sources and types of operations with types of contaminants generated are shown in Table XXV.

Table XXIII.

Sampling devices for particulates

Type	Principle of Operation	Remarks
Sedimentation and settling Chambers Jars Petri dishes Trays Gum paper	Collects particulates which settle out of the atmosphere.	For rough quantitative analysis. Generally limited to estimating dust fall trends in an area.
Inertial or centrifugal midget cyclone	Whirling, spiraling motion of the airstream causes particles to separate from the airstream by centrifugal deflection.	Simple in design, temperature resistant with low pressure drop. Close to 100-percent effective in collecting particles over 10 microns in size.
Filtration Soluble filters Insoluble filters	Suction-type blower system which draws air through specially selected filter medium. Soluble filter may be dissolved to recover particles.	Useful in collecting almost unlimited quantities of particulates for measurement of mass quantity, soiling properties, chemical analysis, counting, sizing, and measuring radioactivity.
Impingement Wet	Separates particles from the air by sudden changes in direction of the airstream impinging the particles on special surfaces.	Wet type used for collecting fine particles. Dry type limited to collecting large particles.
Cascade impactors	A series of impingement stages through which the velocities of the airstream systematically varies, sorting and collecting particles on collodion screens or microscopic slides.	Almost 100-percent efficient in sorting and collecting particles ranging in size from 0.7 to 50 microns.
Electrostatic collectors	Ionization of particles by means of a platinum electrode with particles being collected on a removable electrode of the opposite charge.	Highly efficient for particles ranging below 1 micron in size.
Thermal precipitators	Particles in a slow moving airstream between hot and cold surfaces settle on the cooler surface, collected on a coated grid for microscopic examination.	Limited to a narrow range in particle size dependent on the rate of the airstream movement.

Table XXIV.

Sampling devices for gas

Type	Principle of Operation	Remarks
Test papers	Sensitized papers that change color when exposed to certain concentrations of some gases, fumes, or dusts.	Used to test for some contaminants such as ammonia, hydrogen sulfide, phosgene, etc.
Squeeze bulb and ampules	Squeeze bulb aspirator with glass tube ampules packed with treated granules. Reagent in the granule surfaces gives colorimetric reactions for specific contaminants.	Mine safety applicance (MSA) gas testers constructed for specific contaminants. Limited to concentrations of 1 ppm or greater.
Tutweiler apparatus	Analysis by gas/liquid titrations. Consists of a burette serving as a reaction chamber with a leveling bottle and a graduate mounted on top of burette with a two-way cock.	Measures concentrations above 150 ppm of ammonia, carbon dioxide, hydrogen sulfide, and sulfur dioxide.
Reich's test	Analysis by gas/liquid titrations. Metered amount of contaminated air bubbled through a solution of water, iodine, and starch in a graduated cylinder producing color change.	Volumetric determination of sulfur dioxide only.
Impingers and gas absorption cells	Hand-operated pump aspirator impinges contaminated airstream on specially prepared surfaces or absorption cells for colorimetric reaction.	Constructed for specific contaminates such as aldehydes and chlorine.
Holide leak detector	LPG fuel tank fitted with microburner with copper ring reaction plate above the flame tip. Tubular sampling line feeds the contaminated air to burner by natural draft. Flame color identifies contaminant and estimate of concentration.	Detects and determines the concentration of halogenated hydrocarbon vapors such as carbon tetrachloride, trichloroethylene, and perchloroethylene.
Volume change measurement	Gas absorbed in a base substance. Volumetric measurement before absorption and of residual gases after absorption.	Carbon dioxide absorption in caustic-fyrite CO_2 analyzer. Oxygen absorption in pyrogallic acid-fyrite oxygen analyzer.
Explosimeters or combustion meters	Air sample aspirated through a reaction chamber with squeeze bulb and tubular sampling line. Meter and self-contained battery provides direct reading.	Used to test for combustible gases such as natural gas, carbon monoxide, and hydrocarbon vapors.

Table XXV.

Major sources and types of contaminants generated in controlled areas

Personnel	Fabrication	Assembly	Test	Handling
Bacteria Scale Hair Cosmetics Smoke Lint Fibers Body vapors Skin oils	Smoke fumes Dust Chips and burrs Chemical vapors	Wear particles Material shedding Corrosion products Sublimated materials Flux vapors Outgassing products	Test agent by products Radioactive materials Wear products	All listed in preceding columns

13.6.2 Control factors. Factors to be considered in the control of contaminants generated within a controlled area include:

(a) Adequate area to accommodate the operations and ease of access for maintenance of the facility.

(b) Architectural design for ease of cleaning and good clean down capability.

(c) Location of operations to minimize migration of contaminants from one operation to another.

(d) Selection and acquisition of tooling, equipment, and testing devices to minimize contaminant generation.

(e) Selection and training of personnel to be minimum contributors of contamination.

(f) Selection of personnel garments which are low shedding with minimum working restraint.

(g) Fabrication and assembly processes and procedures to minimize generation of contamination.

(h) Rigid enforcement of precise operating procedures and practices to minimize generation of contaminants.

(i) Employment of a monitoring system that accurately determines acceptable levels of contamination within the controlled area during all operations.

13.6.3 Characteristics that affect control. Other significant items to be considered in controlling airborne contaminants within a controlled area and maintaining acceptable levels of cleanliness are:

(a) The agglomeration characteristics of particulate matter. Particulate matter while suspended in the airstream has a significant tendency to agglomerate; wherein, small particles of matter will build larger and larger to a point where they may well exceed the allowable tolerance limits. Organic matter is more susceptible to this occurrence. The agglomeration of dissimilar materials involves the affinity of some types of matter for droplets of moisture or oils.

(b) The sizes or airborne particulates. The size of particulates as potential contaminants is significant as related to the affect on the contaminee and the methods required for filtration.

13.7 References.

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3. Contamination Control Handbook, Sandia Laboratories, NASA SP-5076, 1969.

4. Filter Application for Spacecraft Sterilization Program, Herbert M. Decker and Lee M. Buchanon, NASA SP-108, Proceedings of the National Conference on Spacecraft Sterilization Technology, 1965.

5. Method of Test for Continuous Counting and Sizing of Airborne Particles in Dust-Controlled Areas by the Light-Scattering Principle (for Electronic and Similar Applications), ASTM F 50, American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.

6. Sizing and Counting Airborne Particulate Contamination in Clean Rooms and Other Dust-Controlled Areas for Electronic and Similar Applications (5.0 Micron and Larger), ASTM F25-631, American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.

7. Standards and Guidelines for the Design and Operation of Clean Rooms and Clean Work Stations, United States Air Force, T.O. 00-25-203, August 1965.

SECTION 14

CONTROL OF SURFACE CONTAMINATION

14.1 Surface contamination. Since surface contamination is a major cause of malfunction of electrical components and tight fitting mechanical components, it is important to either remove contaminants when they exist or reduce the level to a point where they do not constitute a hazzard or degrade the function of the product.

14.1.1 Primary problem. The primary problem in the removal of surface contamination is over powering the adhesive forces that exist between a surface and a particle or droplet. The varying degrees of tenacity, strength by which particles or droplets cling to surfaces, is a major influencing factor in determining the method to be employed to remove contaminants.

14.1.2 Particle size. Another important consideration in contamination control is the size of the particle to be removed. Generally, a particular technique for cleaning a surface will probably become increasingly ineffective as a particle gets smaller.

14.1.3 Deformability. The ability of a particle to change its shape upon impact, in many cases, determines whether or not a particle will adhere to a surface. The deformation allows a larger area of the particle to come in contact with the surface enhancing adhesion. However, if large particles come in contact with a surface at high velocities, they may either be bounced away from the surface or disintegrate. Usually, if the stored elastic energy of collision is greater than adhesive forces developed upon impact, the particles will rebound. Because of this, hard particles will usually rebound more readily than deformable particles. Deformable particles use up much of their collision energy irreversibly in changing the shape of their mass.

14.1.4 How particles are deposited. There are a number of mechanisms by which contaminants are deposited on a surface. Some of the most obvious are:

(a) Deposition as a result of machining, grinding, etching.

(b) Surface to surface transfer.

(c) Gravitational settlement.

(d) Evaporation of liquid media for non-volatile contaminants.

14.1.5 General classifications of soils. The general classifications of soils most common to surface contamination are:

(a) Oils and greases.

(b) Drawing and buffing compounds.

(c) Particulate matter of all types.

(d) Metal corrosion and heat treated products.

(e) Human products (hair, skin, salts, etc.)

14.2 Purpose of surface cleaning. The purpose of surface cleaning is to rid a part of any contamination as economically as possible and without adversely affecting it. Removal can be effective only if the cleaning equipment, materials, and methods are compatible with the soils to be removed, the degree of cleanliness required, the materials of construction and configuration of the part, and the mechanism by which the soils adhere to the part surface. It should be emphasized that the removed soils must be separated and discharged in such a manner that they are not redeposited on the work surface.

There are many variables to consider in surface cleaning. For instance, the nature of the material to be cleaned, the cleaning methods and processes with varying equipment, and the consideration of further treatment to the material.

14.2.1 Design considerations. It should be noted as a design consideration that parts requiring high degrees of cleanliness, especially where components or systems require recleaning, should be capable of being recleaned. Access cleaning should be made for all critical surfaces and consideration given to possible relubrication.

14.3 Surface cleaning processes.

14.3.1 Broad classifications of processes. There are seven broad classifications of processes for removal of surface contamination any one of which may be employed in single or multistage operations. (Each classification is discussed in detail in gross or precision cleaning.)

(a) Acid cleaning.

(b) Alkali cleaning.

(c) Solvent cleaning.

(d) Emulsion cleaning.

(e) Electrolytic cleaning.

(f) Pickling and descaling.

(g) Mechanical cleaning (blast or grit, abrasive tumbling, wire brushing, and flame cleaning).

14.3.2 Liquid cleaning media. With the exception of mechanical cleaning, the classifications of processes mentioned require liquids as the cleaning media. The means by which liquids clean are:

(a) Wetting activity.

(b) Emulsification.

(c) Saponification.

(d) Deflocculation.

(e) Colloidal activity and suspension.

(f) Solvent power.

(g) pH values.

(h) Buffer activity.

(i) Alkalinity and acidity.

(j) Water conditioning.

(k) Sequestration and chelating.

(l) Rinsing.

14.3.3 Complex phenomena of liquid cleaners. A description of each of the above factors is as follows:

14.3.3.1 Wetting. In order for a surface to be cleaned it must first be wetted by a cleaning solution. Wetting action allows the cleaning solution to get in closer contact with the soil, ultimately allowing penetration of the soil and, consequently, removal.

Liquids may vary in their ability to wet a surface. The factors determining wetting capabilities of a liquid are: (1) low surface tension, and (2) low interfacial tension. Consequently, if the surface and interfacial tensions can be reduced in a particular liquid, the wetting capabilities can be increased.

Water, reputedly one of the wettest of all substances, actually has less wetting power than ammonia, acetones, benzene, and most oils. Pure water has a much higher surface tension. For example, when pure water is carefully poured on an oil surface, the surface tensions of the oil and water will resist one another resulting in the water not being able to spread over or wet the oil film. Instead, the water forms individual round droplets or pools with convex domes, a precise reflection of the forces of surface and interfacial tensions involved in the situation. But if the surface tension of the water is reduced so that it is actually less than the oil film, the water will penetrate and displace the oil. The oil then forms droplets on the surface of the water film. These droplets may not yet be completely detached from the soiled surface; however, the oily mass will have been penetrated and its adhesion weakened.

14.3.3.2 Emulsification. By definition, an emulsion is a mixture of liquids that do not dissolve in each other. In an emulsion,one of the liquids contains minute droplets of the other which are evenly distributed throughout. Cohesion forces which hold oil film to a surface are extremely strong. It is practically impossible to remove an oily film from a surface by rubbing, wiping, or scrubbing alone. Applying the water break test to a metal object that has been visibly cleaned of oil film by scrubbing will clearly demonstrate that visible cleanliness is not also chemical cleanliness. Water when applied to such a surface will break up and refuse to wet the surface. Until water refuses to break up on the surface of the object, the object will not be considered chemically clean. When water and detergent (one with an emulsifying agent) are used as a cleaning agent for oil, the process of emulsification enables cleaning. Thorough wetting results in the formation of globules of oil on the surface of the detergent film. Here the process of emulsification comes into play. The oil globules will be detached from the surface and will be dispersed throughout the cleaning solution. The adhesion of the oil to the surface will be permanently disrupted. Once so scattered, the portion of the oil removed cannot be redeposited as a film.

When part of an oily film has been removed in this manner, another layer of the film is exposed to the wetting action of the detergent. Emulsifying action then comes into play again and disperses an additional portion of the oily mass. Thus, we see that emulsification is contingent upon wetting, and further wetting is contingent upon emulsification, and the combined effect of the two actions is the loosening, breaking up, and dispersion of the oily film.

14.3.3.3 Saponification. Saponification is a process whereby fatty acids in animals and vegetable oils chemically unite with certain alkaline substances to form soap. The soap that is formed is in all appearances similar to household soap. Removal of fatty acid soil is one of the problems of detergency. Many greases are a mixture of animal fats (such as tallow) and petroleum oils. Petroleum and mineral oils cannot be saponified and, therefore, must be removed through the process of wetting and emulsification. The fatty acids do, however, react with certain alkaline detergent to form water-soluble soaps.

14.3.3.4 Deflocculation. Deflocculation is the process of changing a compound mass of particles into finely divided particles.

Many soils are solid or semi-solid in nature. Chemical materials that possess the ability to break up solids or semi-solids into smaller particles and disperse them through a liquid are termed deflocculation agents. Fuller's earth, the earliest detergent known, is a highly colloidal clay that has extreme deflocculating capabilities. Colloidal activity plays an imperative part in the removal of solids composed of insoluble solid particles. Among the soils removed by colloidal forces are: carbon, clay, road film, food deposits, and paint fumes.

The process by which deflocculation occurs is believed to involve the neutralization or breaking down of surface tension forces which allow the attraction between solid particles. Experimental data indicates that in all instances of deflocculation, surface and interfacial tensions are reduced. In order to initiate deflocculation, complete wetting of the solid is absolutely necessary.

Colloidal forces are important not only in the direct dispersion of solid particles but in softening and swelling resinous binders, which are a factor in the adhesion of paints, enamels, and similar coatings, and in cleaning impacted buffing compounds.

14.3.3.5 Solvent activity. Solvents may be divided into two categories: aqueous (water base), and non-aqueous (organic base). The solvent properties of water are utilized in cleaning to remove many salts, acids, sugar, and syrup deposits. In aqueous cleaning solutions, water also acts as the solvent medium for the detergent compounds, thereby liberating the energies which result in wetting, emulsification, saponification and deflocculation of soil. Water, aside from its solvent activity, also acts as a dispersal medium for oils and particles which it cannot dissolve but rather carries in suspension. Further, it serves as a means for the application of heat energy and mechanical energy of agitation, both of which are important in detergency.

The main limitation of aqueous solvents as detergent agents is that they have little or no solvent effect on oils or greases. Such ionizing solvents are described as lipophobes (fat haters). Their counterpart, the non-aqueous organic solvents, are similarly described as lipophiles (fat lovers).

Non-aqueous solvents are derived basically from petroleum, coal, and wood. These solvents are unable to dissolve many substances which are readily soluble in water; however, they possess the ability to dissolve not only oils and greases but many resins, rubber, bitumens, paraffin, waxes, and plastics. The primary advantage of organic solvents is that they are capable of formulations that are chemically neutral so that they offer a method for the safe cleaning of reactive metals, such as: aluminum, magnesium, copper, and zinc. Some of the most difficult cleaning problems such as the removal of carbon from engines and stripping of paints and organic enamels are assigned to organic solvents. Carbon deposits which form on engine surfaces are held together by resins formed during combustion of petroleum fuel. No available detergent has been found to exert a direct solvent action on carbon, but certain organic solvents are capable of softening and dissolving the resins which bind the carbon particles together into masses and hold them to the surface. A similar situation exists in the removal of paint where the pigment is insoluble but where the resinous bonding agent may be softened, swelled, or dissolved by the use of suitable solvent reagents.

Since most solvent applications have some hazards due to combustion, toxicity, or other characteristics, the use of any particular operation should be exercised with extreme caution. Guidelines for specific solvents are furnished by National Safety Council Data Sheets or Manufacturing Chemists' Association Data Sheets.

14.3.3.6 pH and its control. Assigning pH values is a method of labeling the acidity or alkalinity level of a solution. The pH value is not a measurement of the amount of acid or alkali in a solution but rather the measurement of the energy. For example, when table salt dissolves in water, a certain portion of it splits up into groups of electrically charged particles called ions which are molecules of the component sodium and chlorine. Similarly when hydrolic acid dissolves in water it splits up, or dissociates, into groups of chlorine and hydrogen ions. When the alkali caustic soda, or sodium hydroxide, dissolves in water it yields sodium and hydroxy ions. All acids share in common the ability to yield hydrogen (H+) ions in water solutions. Likewise, all alkalis yield hydroxyl (OH-) ions in water solution.

Alkalis and acids, within themselves, differ greatly in the number of hydroxyl or hydrogen ions they release per given weight in a given volume of water. A fluid ounce of 50 per cent hydrochloric acid solution would yield a far greater number of hydrogen ions than a fluid ounce of a 50 percent solution of acetic acid. An acid which yields a great number of hydrogen ions is energetic in entering chemical unions with other substances and is, therefore, described as a strong acid. In regard to alkalis, strong alkalis cause greater energetic level in entering chemical unions with other substances than do weaker alkalis. In alkalis the cause of energy is the release of hydroxyl ions and in acids the cause of energy is the release of hydrogen ions. The concentration of hydrogen and hydroxyl ions determines the relative degree of acidity or alkalinity of a water solution. In order to express the degree of acidity or alkalinity of a solution, a scale was derived based on the logarithm of the reciprocal of ion concentration, hydrogen or hydroxyl.

The values that were arrived at are known as the pH values and may be arranged on a scale from 0 to 14, with 7 as the neutral point. Values above 7 indicate degrees of acidity. Both alkalinity and acidity become more intense as values move away from the point of neutrality. Using this standard, pH 8 represents a weak alkaline solution, and a pH of 6 represents a weak acid solution. A high acid level would be represented as pH 1.5 and a high alkaline solution would be represented as pH 13.5. Each increment in pH, expressed as a whole number, since it is based on a logarithmic scale, represents a ten fold increase or decrease in acidity or alkalinity; pH 5 is 10 times stronger in acid than pH 6; pH 4 is ten times more strongly acid than pH 5 and 100 times more strongly acid than pH 6, and so forth.

Since pH is a method of stating the concentration of electrically charged groups of molecules, the most accurate method of measuring pH involves the use of a sensitive voltmeter (a pH meter) which measures the electrical potential across the charged ions and registers this in terms of pH values.

14.3.3.7 Buffer activity. The degree of acidity and alkalinity of a cleaning compound plays a part in rust and scale removal, in etching and pickling metal, in saponification, and emulsification. Buffering, the means by which a solution resists a change in the degree of acidity or alkalinity, requires very accurate pH determination. pH readings are helpful in proving comparative data on the action of a given detergent, if all other conditions have been carefully controlled.

14.3.3.8 Alkalinity and acidity. In the discussion of the pH, reference was made to the distinction between the degree and the amount of acidity or alkalinity. The active alkalinity or acidity is a factor of the extent to which the detergent dissociates completely during the course of the cleaning, the total amount of acid or alkali in the detergent is gradually made available for useful cleaning. Conversely, if a considerable amount of acid or alkali fails to dissociate or become active, it merely represents so much inert material or filler which plays no part in the cleaning and which is a source of rinsing difficulties.

In the selection of a detergent, the criterion of value is not the amount of acid or alkali by weight or volume which is offered, but the extent to which the acid or alkali can be made available for the removal of dirt or soil. A cleaning compound may consist entirely of alkaline substances, yet only 10 per cent of this amount may actually be effective in removing soil. In the formula of a scientifically balanced detergent, the choice of each component can be justified in terms of the definite function assigned to it in the cleaning operations. Moreover, the components should not merely fulfill their individual functions, but should support and reinforce each other, so that the whole compound will be more active and more stable than any of its parts.

14.3.3.9 Hard versus soft water. If distilled water could be used in the makeup of aqueous cleaning solutions, many problems could be avoided. It is impractical to use distilled water in the tremendous gallonage required for industrial cleaning operations. Local natural water sources are generally employed, and these almost invariably contain dissolved bicarbonates and sulphates of calcium and magnesium. The presence of these minerals is responsible for water hardness, and the fact that soap loses much of its efficiency in the presence of hard water has long been recognized. Soap reacts with the dissolved magnesium salts to form insoluble, adhesive precipitates. The soap which takes part in this reaction is thus unavailable for cleaning, and the gummy deposits resist rinsing and may form a more stubborn and objectionable soil than the original dirt.

14.3.3.10 Sequestration, chelating. This is the process whereby undesirable ions such as Ca++, Mg++, and heavy metals are deactivated thus preventing reactions with material that would normally form insoluble products. The most common example is the hard water scum formed when soaps or even some synthetic detergents are used. The scum formed is the reaction between the Ca++, or Mg++ ions in hard water with the soap. When the water is softened, the Ca++, and Mg++ ions become tied up or sequestered, preventing reactions.

14.3.3.11 Rinsing. Rinsing is a very basic element of cleaning procedures. There are several factors considered necessary to insure adequate rinsing.

(a) Verify contaminant free water and free flowing rinses.

(b) Audit pH of rinse water. A low pH after acid dipping and a high pH after cleaning usually indicates inadequate water flow.

(c) Low water pressure and too few rinses to provide adequate rinsing becomes more critical in winter when water is coldest. Cold rinse temperatures can vary from winter to summer. Temperatures from 35° to 50° F can set up emulsified or saponified soils on the surface of the work after cleaning with alkalines. Warm rinses following alkaline cleaning is preferred. It is recommended to be operated at a maximum temperature of 100° to 110° F to prevent dry-on of cleaner during transfer.

(d) When air agitation is used, it should be checked free of oil. It is recommended that blown air or filtered compressed air be used.

(e) Siphon breakers should be on water inlet pipes to assist air agitation.

(f) Rinse tanks equipped with dam overflows are efficient in skimming surface films. When riffle pipes are used, they should be located directly across the tank from the dam overflow.

14.3.4 Complexity of surface cleaning. Even a basic examination of the principles discussed makes it obvious that surface cleaning is and can be a very complex process involving multiple considerations, differing conditions, and almost infinite variables. All surfaces cannot be cleaned with any one given compound or material, or method of application, or one type of equipment. It could well be that one all-powerful type of solvent, when put to work in an elaborate piece of cleaning equipment, would exert sufficient detergency to perform 90 per cent of all the cleaning problems discussed, but how economic would it be? What hardships would it work on the operators of such a process?

Consequently, when the factor of efficiency is contrasted with the all important factors of safety and economy, it is increasingly evident that manufacturers of cleaning compounds must resort to a multiple line of products. Some of these may be general purpose for common metals or soils, but special purpose compounds are required for given metals and specific contaminants.

14.3.5 Cleaning of common metals. Some of the common metals and comments on cleaning them are as follows:

1. Aluminum. A light metal that possesses considerable resistance to some acids, but etches readily in uninhibited alkaline cleaners.

2. Berylium. A metal produced commercially by electrolysis of the fused chloride. Is light and silvery white and posses great strength, rigidity, and elasticity. Berylium can be safetly cleaned with heavy duty steel cleaners.

3. Brass and bronze. Copper-bearing alloys containing zinc and tin respectively. Both require uninhibited cleaners.

4. Cadmium. Cadmium is a silvery crystalline metal resembling zinc. It is usually used as a coating to protect other metals from corrosion. Cadmium is only slightly tarnished by air or water at ordinary temperatures. It may be cleaned with the same solutions used for zinc.

5. Copper. Copper may be cleaned with the same cleaners used for steel or brass.

6. Gold. This is a yellow, soft metal, and is the most malleable and ductile of all metals. Gold is a very inactive metal and does not combine directly with oxygen or corrode in the atmosphere. It is not affected by any single common acid nor by alkalies. Gold can be cleaned like steel.

7. Lead. Lead requires an inhibited cleaner. A fluoboric acid dip is best for pickling lead prior to plating.

8. Magnesium - A light metal that is safely cleaned with heavy duty steel cleaners. Magnesium will corrode in mild cleaners, but not in highly alkaline cleaners.

9. Nickel and stainless steel. Both are resistant to corrosion and can be cleaned with most products having the strength to attack the degree of soil present.

10. Rhodium. This metal is a platinum metal. It is extremely unreactive and can be cleaned like nickel.

11. Silver. Silver is a white glossy metal. It is the best conductor of heat and electricity and it is known for its ductility and malleability. Silver may be cleaned with the same cleaners used for zinc.

12. Steel and cast iron. Corrode in acids, but resist most alkalies. Steel can be cleaned with alkaline cleaners if the heavy duty variety is used.

13. Terneplate. This is lead or steel, and should be cleaned like lead.

14. Tin. Tin is a valuable plating metal because at ordinary temperatures it is permanent in air and water. Tin may be cleaned with the same cleaners used for lead.

15. Titanium. This material presents many problems and should be carefully checked out before defining a process.

16. Zinc. This nonferrous metal requires an inhibited alkaline cleaner.

17. Zirconium. Normally cleaned like steel, unless the alloy would dictate otherwise.

14.4 Determining surface contamination. Perhaps no other aspect of contamination control is as controversial or underdeveloped as verification of surface cleanliness. Many people have tried to answer the question, "How clean is clean?" The hours of discussion devoted to this question, though somewhat fruitless, are indicative of the interest and the need for additional research and development. From one of these discussions may eventually come the spark of an idea which will evolve into a relatively simple, easy to administer, reliable, and repeatable surface cleanliness test method.

A much more pertinent question is, "How clean does a part have to be to function properly and reliably?" The area of attention is thus immediately directed from generalities to specifics. In the case of microminiature electronic components, the answer to this question may not be too difficult to determine. In most other cases, however, the answer may be vague or even unknown.

It is common practice to attempt to achieve the highest practical cleanliness level. Disregarding appearance, if the part then functions properly, some reduction in cleanliness requirements may be justified. With one exception, the determining factor in establishing cleanliness requirements is function. The exception concerns microbial decontamination in the medical, pharmaceutical, and planetary quarantine fields, in which the highest attainable cleanliness levels are desired.

14.4.1 Classification of surface cleanliness tests. Apparently, the absence of any universal surface cleanliness test has lead to the development of many tests which exhibit varying degrees of effectiveness and application. There are several ways of classifying these tests; one method of classification involves direct and indirect methods for determining the degree of cleanliness. Table XXVI compares the direct and indirect surface cleanliness tests.

Table XXVI.

Comparison of direct and indirect surface cleanliness tests

Type of Test	Parameter Measured	Characteristics of Test
Direct	Presence or amount of soil remaining on surface.	The only true methods of testing for surface cleanliness; vary widely in discrimination and application; laboratory and production.
Indirect: soil removed	Amount of soil removed from a surface.	Widely used;more suitable to laboratory methods; more a measure of the effectiveness of the cleaning system than of the exact cleanliness of a surface.
Indirect: effects	Effects of unclean surfaces.	Visual means determine blemish or irregularities in subsequent operations such as plating; malfunction or failure may be the indirect result.

14.4.2 Other methods of classification. Other methods of classifying surface cleanliness tests include the following:

(a) Qualitative versus quantitative. Most tests provide qualitative results.

(b) Laboratory versus production. Generally the most accurate tests require the use of laboratory techniques and are therefore not suitable as tests which can be performed on the production line.

(c) Hydrophobic versus hydrophilic. Some tests are sensitive to only hydrophobic or hydrophilic soils, while others are sensitive to all soils.

(d) Degree of sensitivity. Varies widely among different tests.

14.4.3 Specific surface cleanliness tests. Many tests have been designed to provide a measure for surface cleanliness. A less number have been developed to a point of practical usefulness. Of those so developed, most have one or more limitations which prevent them from becoming a universally applicable and accepted surface cleanliness test.

A listing and brief description of those tests which are recognized as having application in this field are shown in Table XXVII.

Table XXVII.

Specific surface cleanliness tests

Name of Test	Test Method	Characteristics or Limitations
Visual	Examination with the unaided eye or with a microscope.	Subjective but widely used; most effective with particulate matter, least effective with invisible films; use of a highly trained microscopist increases the validity of test results.
Tissue paper or white cloth	Surface is rubbed with a piece of white tissue paper or a white cloth. Grease or soot is observable.	Limited to visible soils, insensitive qualitative test.
Water break	Normally applied after last clean water rinse. Any break in continuity or receding water film is observed as water drains off the part.	A qualitative test for hydrophobic soils; contaminates in the water lessen sensitivity; use of deionized water and a trained inspector may increase sensitivity to one-molecular thickness of contaminant.

(continued)

Name of Test	Test Method	Characteristics or Limitations
Gravimetric	A test piece is weighed before and after cleaning, or the soil remaining from the evaporated cleaning solvent is weighed.	Results show good sensitivity (5×10^{-5} gm/cm2), but are more indicative of the effectiveness of the cleaning method than surface cleanliness.
Ferrocyanide paper	Paper is immersed in a solution of NaCl, $K_3Fe(CN)_6$ and $K_4Fe(CN)_6$ in water and dried. Paper is moistened and placed on metal surface, then removed and rinsed in clear water. Clear areas on the paper are caused by soil on the metal.	Limited to ferrous metals and laboratory tests.
Copper dip	Cleaned metal panels are dipped in an acid copper surface (copper flash) solution.	Adherence, continuity, and appearance of copper flash indicate part cleanliness before the test. Limited to ferrous metals; requires an experienced operator.
Solvent ring	A drop of solvent is repeatedly deposited and picked up from the test surface. It is finally deposited on a quartz or glass slide and dried. If contamination is present, a ring will be formed on the slide.	Enables subsequent identification of soil by infrared spectrophotometry, assumes use of a high purity solvent.
Solvent	After each cleaning step, used solvent is filtered through membrane filters and examined for levels of particulate contamination. NVR is also monitored. Deionized water rinses are monitored for resistivity if ionic cleaners are used.	Commonly used indirect method which assumes part cleanliness if the solvent no longer removes contamination.
Indium adhesion	The tip of a freshly broken indium rod is forced against the test specimen surtace with a force of 10 grams. The force is maintained for periods of 1 second up to 5 seconds. The rod is then removed from the surface and force-time characteristics are recorded. The coefficient of adhesion (ratio of tensile force to break the bond to the compression force exerted to form the bond) indicates the degree of surface cleanliness.	Adaptable for production testing; a sensitive direct test, coefficient values must be established for different soils and substrates, offers rapid feedback of test data; test surface must be accessible to the indium tip.

(continued)

Name of Test	Test Method	Characteristics or Limitations
Atomizer	Surface is cleaned and dried. Water is applied as a spray with an atomizer. The droplet pattern with the advancing contact angle is observed to determine surface cleanliness.	Sensitive but only for hydrophobic soils; results affected by spray time, nozzle-to-part distance, atomizer air pressure and ambient temperature; applicable to small cross-sectional areas with stainless steel or gold. Surface must be smooth and free of wettable detergent films.
Contact angle	A light beam is directed into a water droplet on the test surface. The angle of the reflected beam indicates the contact angle or angle of incidence. Greater contact angles indicate larger amounts of contamination.	Effective only on nonwetting hydrophobic contaminants.
Ring test	A droplet of water on a surface tension ring tester is repeatedly lowered to contact the test surface. The number of contacts, or B-number, indicates surface cleanliness.	Must be performed by a trained operator to be repeatable; a measure of surface wettability.
Radioactive	A specific quantity and composition of tagged synthetic soil is applied to a test piece. After cleaning, the test piece or cleaning solution is monitored for residual tracer activity.	Primarily a test of cleaning method effectiveness; involves statistical treatment of data; highly sensitive and quantitative test.
	The formula for calculating the soil remaining is as follows: $\frac{\text{Final count}}{\text{Initial count}} \times \frac{\text{mg of initial soil}}{\text{area of test panel}} = \text{soil remaining}$	
Radiochemical	A surface contaminant is dissolved in place by a small amount of radiochemical solution. The change in rate of evaporation of the solution is indicative of the soil concentration. A stream of dry nitrogen is passed over the sample to standardize evaporation parameters.	Quantitative results are obtainable if the type of contaminant is known, and calibration curves are constructed and used as standards. Sensitivity and accuracy are sacrificed if the test surface is porous or absorbent. Variables include rate of solution, ambient temperature and pressure, rate of dry nitrogen flow, and latent heat of vaporization of the radiochemical.

14.4.4 <u>ASTM test method</u>. The American Society for Testing and Materials has developed a number of test procedures related to the field of surface cleanliness. For reference purposes, some of these tests are listed below:

Table XXVIII.

Selected ASTM test methods

ASTM Designation	Test Title
F 21-65	Test for Hydrophobic Surface Films by the Atomizer Test
F 22-65	Test for Hydrophobic Surface Films by the Water Break Test
F 24-65	Measuring and Counting Particulate Contamination of Surfaces
F 59-65T	Identification of Metal Particulate Contamination Found in Electronic and Microelectronic Components and Systems Using the Ring Oven Technique, with Spot Tests (tentative)
	Tests for Solvents
F 52-65T	Test for Silting Index of Fluids for Processing Electronic and Microelectronic Devices (tentative)
F 58-65T	Measuring Resistivity of Electronic Grade Solvents (tentative)
F 60-65T	Detection and Enumeration of Microbiological Contaminants in Water. Used for Processing Electronic and Microelectronic Devices (tentative)
D 1901-61T	Test for Relative Evaporation Time of Halogenated Hydrocarbon Solvents and their Admixtures
D 2109-64	Tests for Nonvolatile Matter in Halogenated Organic Solvents and their Admixtures

14.5 References.

1. Clean Room Technology, James W. Useller, Lewis Research Center, Cleveland, Ohio, NASA SP-5074, 1969.

2. Contamination Analysis and Control, James L. Dwyer, Reinhold Publishing Corporation, 1966.

3. Contamination Control Handbook, Sandia Laboratories, NASA SP-5076, 1969.

4. Contamination Control Principles, Sandia Corporation, NASA SP-5045, 1967.

SECTION 15

CONTAMINATION CONTROL IN GASES

15.1 Two major uses. Controlling contamination in gases and liquids requires methods applicable to the type and quantity of contaminants and the cleanliness level required for a particular application. Gases and liquids may be grouped according to their two major uses:

(a) As part of a system, such as hydraulic fluids and actuating or control gases, fuels, coolants, lubricants, and refrigerants.

(b) In processing and in support of system operations such as cleaning solvents, flush gases and liquids, cutting oils and lubricants.

15.2 Categories of gases. Gases discussed herein are those defined by the Department of Transportation as "Compressed or Liquefied" in the interest of safe handling and transportation in interstate commerce. These gases are segregated into the following major groups, as they differ in physical state when contained.

15.2.1 Nonliquefied gases. Nonliquefied gases are those which do not liquefy in containers at normal temperatures and under pressures up to 2000 to 2500 psig, normally attained in commercially used containers. However, these gases will liquefy at cryogenic temperatures (approximate range from -200 ° to -459° F).

15.2.2 Liquefied gases. Liquefied gases are those which do become liquids, to a very large extent, in containers at or near normal temperatures and under pressures ranging from 25 to 2500 psig, normally attained in commercially used containers.

15.3 Description of gases. Pertinent information on each of the compressed gases is essential to the effective application of a specific gas as part of a system or in processing and in support of system activities. Essential information is provided below:

15.3.1 Physical properties. Physical properties include boiling points, critical temperatures and pressures, specific heat, density, and other data for each gas. Table XXIX lists some of the common gases and their properties. The data given generally represent the properties of a pure gas. Therefore, the values of certain of these properties will vary somewhat with various purity levels. It should also be noted that the factors used in expressing values of properties will vary depending on the source of the data.

Table XXIX.

Low temperature physical properties of gases

Name	Formula	Molecular Weight	Normal Boiling Point			Critical Point		Triple Point or (Melting Point)		Specific Heat Cp @ 70°F, 14.7 PSIA BTU/(lb. mole) (°F)	Gas Density @ 70°F 14.7 PSIA lb./cu. ft.
			Temp. °F.	Liquid Density lb./cu. ft.	Latent Heat BTU/lb.-mole	Temp. °F.	Pressure PSIA	Temp. °F.	Pressure PSIA		
Acetylene	C_2H_2	26.04	—119.2 (3)	38.7 (3)	9180 (3)	96.0	906	—114.6	17.4	10.6	0.0680
Air (1)	(2)	28.96	—317.9	54.56	2556					7.0	0.07493
Ammonia	NH_3	17.03	— 28.1	42.6	10037	270.3	1639	—108.0	8.8×10^{-1}	8.6	0.0445
Argon (1)	A	39.95	—302.6	86.98	2804	—187.6	705	—308.9	9.99	4.98	0.1034
Carbon Dioxide	CO_2	44.01	—109.3 (3)	97.5 (3)	10854 (3)	87.9	1071	— 69.9	75.1	8.95	0.1144
Carbon Monoxide	CO	28.01	—311.9	49.3	2597	—220.4	508	—337.1	2.23	6.97	0.0725
Carbonyl Sulfide	COS	60.07	— 58.3	73.0	7961	221.0	896	(—217.8)		9.92	0.1521
Chlorine	Cl_2	70.91	— 29.3	97.4	8780	291.2	1118	—149.8	2.02×10^{-1}	8.2	0.1853
Deuterium	D_2	4.03	—417.3	10.7	540	—390.8	239	—426.0	2.48	6.97	0.0104
Ethane	C_2H_6	30.07	—127.6	33.8	6315	90.1	708	—297.9	1.70×10^{-4}	12.6	0.0783
Ethylene	C_2H_4	28.05	—154.8	35.2	5826	49.1	735	—272.5	1.70×10^{-2}	10.4	0.0729
Fluorine	F_2	38.00	—306.6	93.8	2815	—200.2	808	(—363.3)		7.49	0.0983
Freon—12	CCl_2F_2	120.92	— 21.6	92.9	8592	233.6	597	(—252.4)		17.66	0.318
Freon—13	$CClF_3$	104.46	—114.6	95.0	6670	83.9	561	(—294.0)		16.09	0.273
Freon—14	CF_4	88.01	—198.4	122.4	5160*	— 49.9	542	—299.2	1.70×10^{-2}	14.6	0.228
Freon—22	$CHClF_2$	86.47	— 41.4	88.2	8704	204.8	716	(—256.0)		13.35	0.229
Helium (1)	He	4.00	—452.13	7.798	36	—450.2	33	—455.8 (4)	7.35×10^{-1}(4)	4.98	0.01034
Hydrochloric Acid	HCl	36.46	—120.9	74.3	6948	124.5	1199	(—173.)		6.9	0.0950
Hydrogen (1)	H_2	2.02	—422.99	4.418	389	—399.8	188	—434.5	1.044	6.89	0.005209
Hydrogen Sulfide	H_2S	34.08	— 75.4	60.0	8033	212.7	1307	—122.0	3.36	8.2	0.0892
Krypton (1)	Kr	83.80	—244.0	150.6	3884	— 82.8	796	—250.9	10.62	4.98	0.2172
Methane	CH_4	16.04	—258.6	26.5	3519	—116.6	670	—296.5	1.69	8.6	0.0416
Methyl Chloride	CH_3Cl	50.49	— 11.5	62.2	9293	289.6	967	—144.0	1.27	9.97	0.133
Neon (1)	Ne	20.18	—410.7	75.35	748	—379.7	395	—415.4	6.27	4.98	0.05215
Nitric Oxide	NO	30.01	—241.0	79.3	5953	—137.2	945	—263.6	3.16	7.1	0.0777
Nitrogen (1)	N_2	28.01	—320.5	50.46	2405	—232.6	491	—345.9	1.82	6.98	0.07245
Nitrogen Trifluoride	NF_3	71.01	—199.2	96.0	4984	— 38.7	657	(—343.3)	1.77×10^{-4}		0.1864
Nitrous Oxide	N_2O	44.01	—127.2	76.8	7110	97.7	1054	—131.6	12.74	9.2	0.1146
Oxygen (1)	O_2	32.00	—297.3	71.27	2932	—181.1	737	—361.8	2.10×10^{-2}	7.02	0.08281
Ozone	O_3	48.00	—169.4	101.8	6174	10.2	793	(—314.5)			0.124
Propane	C_3H_8	44.10	— 43.7	36.2	8076	206.2	617	—305.8	8.39×10^{-11}	17.4	0.1154
Propylene	C_3H_6	42.08	— 53.8	37.5	7925	197.4	667	—301.0	2.44×10^{-8}	15.3	0.1070
Sulfur Dioxide	SO_2	64.06	13.9	89.3	10728	315.0	1142	—103.8	2.43×10^{-1}	9.6	0.1687
Xenon (1)	Xe	131.30	—163.0	190.8	5436	61.9	847	—169.2	11.82	4.98	0.3416

(1) Based on data from the Compressed Gas Association Meeting June 19, 1962

(2) Proximate composition of dry air (Mole Percent), 78.09 N_2, 20.95 O_2, 0.93 Ar, 0.03 CO_2

(3) Denotes sublimation and solid density

(4) λ (lambda) point

15.3.2 Formulation. Formulation includes elements of gas mixtures and impurities, with concentrations of each gaseous element, water, oil, and particulates in each available grade.

15.3.3 Materials of construction. Materials of construction which are suitable or not suitable for containment of the gas as related to pressure, temperature, and corrosive limitations.

15.3.4 Physiological effects. Physiological effects are odor, toxicity, threshold limit values, contact irritation to skin, eyes, etc.

15.3.5 Precautions. Precautions in handling and storage include containers, safety devices, temperatures, flammability, etc.

15.3.6 Container identification. Marking and identification of containers include methods of marking, type of data marked on containers, and color marking. These methods may vary with the different producers.

15.4 Purity levels and grades. Minimum purity levels of gases are normally defined by percentage by volume. Maximum impurities are defined by parts per million by volume. Factors for interconversion of the various concentration units are shown in Table XXX. Analysis of individual impurities may be automatically supplied with the higher purity grade or may be available on request from the producer.

15.4.1 Terms used to define grades of gases. Terms or nomenclature used by the compressed gas industry to define grades of gases relative to the purity levels vary widely among producers, and often between gases from one producer. A grade term defining a purity level for one gas may define a different purity level for another. Therefore, a single grade term defines a purity level of single gas from a single producer. The same grade term may define a different purity level when used by other producers. Some of the most commonly used terms for gas grades are listed below in alphabetic order and do not imply any relative order of purity.

Grades	
Analytical	Research
Commercial	Technical
High purity	Ultrapure
Instrument	Zero
Prepurified	

15.4.2 Impurities. Impurities found in various gases may be generally classified as gaseous and aerosol(liquid and solid particulates). Gaseous impurities are principally small concentrations of the constituents of gaseous or liquid media from which the basic gas is extracted or from other substances used in production processes.

15.4.2.1 Hydrocarbons. In some cases, forms of hydrocarbons may be an intolerable element. Sources of hydrocarbons when not a normal element are residues of cleaning solvents from containers and system equipment, and from lubricants used in processing equipment and containment systems.

15.4.2.2 Water. Water in either liquid or vapor form is usually a major significant constituent of all gases. The water content of a gas is usually reduced to an extremely low level by the normal gas production processes. However, additional amounts of water are easily introduced into the gas from the air both by inboard leakage into the system and during transfer to a container or from a container to the user's system. Another source of water is inadequately dried containers, transfer equipment, and materials and equipment used in gas systems.

15.4.2.3 Oils. Oils in liquid or vapor form may be introduced into the gas from lubricants and packing materials or sealants and inadequately cleaned equipment used in the production processes and the containment system.

15.4.2.4 Solid particulates. Solid particulates are not normally found in any significant volume in newly produced and purified gases. Principle sources of particulates are corrosion, rust, scaling, and flaking of containers used to transport the gas or to contain the gas in the user's system. For this reason, gas producers are reluctant to certify any maximum concentration of particulate matter in gases.

Table XXXI lists some commonly used gases with typical minimum purity levels that exist in the higher purity grades available from most producers. The impurities, which are those normally found, are given with the maximum values for each, and are typical of the concentrations that may be expected for the designated purity level. Actual analysis will vary with each container of a specific gas and may reveal some impurities not shown.

15.4.3 High purity levels. Producers in the compressed gas industry with highly developed, well-controlled processes for separation and purification are capable of providing gases of extremely high purity levels which meet most use requirements. Research and development in this field are constantly refining these processes to a point where some impurities can be reduced to a virtually undetectable level. The major problem, then, is maintaining the gas purity level in containers and containment systems to the ultimate use point. Initial potential sources of impurities in gases are producer storage containers, transfer equipment, shipping containers, and handling practices.

It may be reasonable to assume that a container of gas is of that purity level labeled or certified by the producer. However, when the use requirements are critical, a complete and accurate analysis by a qualified laboratory may be needed.

15.5 Gas systems. The gas user is responsible for maintaining or purifying a gas within the cleanliness level compatible with end use requirements. Gases which do not meet needed purity levels may require extensive purification facilities which are not economically feasible except for the high volume user. Hence, the most economical gas for the lower volume user is one obtained within the required purity level. Techniques for contamination control in this paragraph are limited to the user gas system.

15.5.1 Components. A gas system used for the containment, control, and distribution of a compressed gas includes the following components:

Purification equipment
Tanks
Fittings
Regulators
Valves
Gauges
Dehydration equipment
Tubing
Nozzles
O-rings and packing
Filters

15.5.1.1 Principal source of contaminants. The assemblage of the above parts and components in a system constitutes the principal source of contaminants to the gas. Types of contaminants, examples of each, and some fo the principal sources are shown in Table XXXII.

Table XXX.

Factors for interconversion of concentration units of gases and vapors

Present Units Desired Unit	Percent by Volume	Parts per Million by Volume	Moles per Liter	Milligrams per Cubic Centimeter	Milligrams per Liter	Milligrams per Cubic Meter	Milligrams per Cubic Foot
Percent by volume	-	10^{-4}	2450	24,500/M	2.45/M	2.45×10^{-3}/M	0.0863/M
Parts per million by volume	10^{4}	-	24.5×10^{-6}	24.5×10^{-6}/M	24,500/M	24.5/M	863/M
Moles per liter	4.1×10^{-4}	4.1×10^{-8}	-	1/M	10^{-3}/M	10^{-6}/M	35.3×10^{-6}/M
Milligrams per cubic centimeter	$4.1\ M \times 10^{-4}$	$4.1\ M \times 10^{-8}$	M	-	10^{-3}	10^{-6}	35.3×10^{-6}
Milligrams per liter	0.41 M	$4.1\ M \times 10^{-5}$	$M \times 10^{3}$	10^{3}	-	10^{-3}	0.0353
Milligrams per cubic meter	410 M	0.041 M	$M \times 10^{6}$	10^{6}	10^{3}	-	35.3
Milligrams per cubic foot	11.6 M	$1.16\ M \times 10^{-3}$	28,300 M	28,300	28.3	0.0283	-

How to use table:

1. Locate column along top of table which gives present unit.
2. Locate row along left of table which gives desired unit.
3. Read down and across to locate multiplying factor.
4. Multiply present quantity by factor.

Example: Given 700 ppm to convert to moles per liter:
$700 \times 4.1 \times 10^{-8} = 2.87 \times 10^{-5}$ moles/liter.

Note: In table, M is molecular weight of the gas or vapor

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Table XXXI.

Typical purity levels and impurity concentrations of gases

NAME	(a) PURITY LEVEL % VOLUME	(b) IMPURITIES IN PPM													
		Acetylene (C_2H_2)	Argon (Ar)	Carbon Dioxide (CO_2)	Carbon Monoxide (CO)	Helium (He)	Hydrogen (H_2)	Krypton (Kr)	Methane (CH_4)	Neon (Ne)	Nitrogen (N_2)	Nitrous Oxide (N_2O)	Oxygen (O_2)	Xenon (Xe)	Water (H_2O)
Argon	99.999	0.05	-	0.5	1.0	5.0	1.0	-	0.5	-	3.0	0.1	1.0	-	1.0
Helium	99.9995	0.05	-	0.5	1.0	-	1.0	-	0.5	2.0	5.0	0.1	1.0	-	1.0
Hydrogen	99.999	0.05	5.0	0.5	1.0	5.0	-	-	0.5	-	5.0	0.1	1.0	-	1.0
Krypton	99.99	0.5	-	0.5	10.0	5.0	1.0	-	5.0	50.0	25.0	0.1	2.0	25.0	1.0
Neon	99.998	0.5	-	0.5	1.0	25.0	1.0	-	0.5	-	5.0	0.1	1.0	-	1.0
Nitrogen	99.999	0.05	5.0	0.5	1.0	5.0	1.0	-	0.5	-	-	0.1	1.0	-	1.0
Oxygen	99.995	0.05	5.0	0.5	1.0	5.0	5.0	5.0	0.5	5.0	15.0	0.1	-	-	1.0
Xenon	99.99	0.5	-	5.0	10.0	5.0	1.0	25.0	5.0	5.0	5.0	0.1	5.0	-	1.0

(a) Purity levels may be equal to or greater than (≥) the values shown.

(b) Impurity concentrations may be equal to or less than (≤) the values shown.

Table XXXII.

Contaminants in gas systems

Gases and Vapors	Particulates	Water
Vapors from cleaning solvents. Vapors from assembly process materials. Atmospheric air. Products from chemical reactions of materials in the system with cleaning agents, process materials, atmospheric air, etc.	Residue from cleaning agents and processes. Residue from assembly processes and materials. Condensates from air, gases, and vapors by temperature and pressure changes. Airborne particles. Wear particles from valves, regulators, and other equipment operated in the system. Corrosion products generated in the system.	Inboard leakage of ambient atmosphere. Inadequately dried containers in systems and particulate filter equipment.

15.5.2 Design considerations. Effective contamination controls must be considered in the design stage of a gas system and implemented throughout the assembly operations. In addition to general design considerations, some characteristics unique to a gas system that should be considered are:

(a) All materials exposed to the gas should not be adversely affected by the particular gas.

(b) Parts and equipment such as regulators, valves, flow meters, safety devices, tubing, etc., should have a minimum of entrapment areas and should be suitable for the pressures involved.

(c) For oxygen systems, eliminate sharp bends in parts and system configuration to prevent erosion, hot spots, and ignition; and provide special conditioning procedures to assure removal and exclusion of all combustible substances, especially grease and oils.

(d) Seals or packing and lubricants in operating parts should be minimum contributors of contaminants to the gas system.

(e) Cleaning agents should be minimum contributors of contamination and easily removed from the system.

(f) Provide procedures and equipment to prevent contaminants from entering the system when it is opened for any reason.

(g) After system cleaning a final rinse, flush or purge should be used to assure removal of cleaning agents and residue (such as removal of hydrocarbons by steam flushing and water by heating, dry gas purging, and evacuating).

15.5.3 Requirements for gas purification equipment. The following factors should be considered in determining requirements for gas purification equipment:

(a) Specified purity level for end use.

(b) Purity levels available from the producers.

(c) Specific contaminants or impurities in gases as received that must be removed or reduced in concentration.

(d) Economics involved, such as cost of producer delivering gas of the required purity level versus cost of design, installation, and maintenance of purification equipment.

(e) Capabilities of purification equipment to remove specific impurities to the levels required.

(f) Types and concentrations of contaminants introduced by the gas system.

15.5.4 Principal contamination removal. Equipment for use in-line with a gas system to remove the principal contaminants, as well as special design equipment, is available from gas equipment companies.

15.5.4.1 Filters. Selection and design of particulate filters should consider compatibility with the gas, flow rate, pressure, and temperatures involved. Special consideration should be given to the filter media with regard to:

(a) Removal rating.

(b) Working pressure.

(c) Material.

(d) Migration or unloading.

(e) Water-vapor and oil-vapor content.

(f) Other characteristics that may contribute contaminants to the system.

15.5.4.2 Drying equipment. Some adsorptive materials used in drying equipment (for water vapor removal) may have a water content in excess of that in the gas, resulting in an increase of water content rather than the intended decrease.

Molecular sieves that have been adequately dehydrated are very effective media for drying gas to low levels of water content.

Drying equipment should be located as near as possible to the point of gas usage to preclude additional moisture being introduced between the dryer and point of use.

15.5.4.3 Other purification equipment. Other purification equipment employing various methods of absorption, adsorption, phase separation, and chemical catalysis may be used in removing or reducing gaseous impurities. Most of these processes require highly specialized techniques and complex equipment, which are not normally economically feasible for a low volume user.

15.5.5 Gas specifications. The needed cleanliness level of purity of a gas used as part of a system or process is determined by the cleanliness level requirements of the specific application. To facilitate this determination and to formulate a gas specification that will adequately describe the requirements, the following factors should be considered:

(a) Chemical and physical properties of the gas as they may affect the usage requirements.

(b) Normal impurities in the gas as they may affect usage requirements.

(c) Purity levels with concentrations of impurities of the gas normally supplied by producers.

(d) Cautions and hazards in handling, containment, and use of the gas.

(e) Facilities and methods required for monitoring or verifying purity levels or concentrations of specific contaminants.

(f) Facilities required for the removal of specific contaminants.

(g) Contamination potential of the gas containment or distribution system.

15.5.6 Procurement specification. A procurement specification for a gas should adequately describe the acceptable purity level and impurity concentrations in terms such as the following:

(a) Gas purity level - specify minimum percent by volume.

(b) Normal gas impurities - specify allowable maximum concentration by parts per million or percent by volume.

(c) Water content - specify allowable maximum concentration by dew point (°F or C) or parts per million.

(d) Other gaseous impurities that must be limited - specify allowable maximum concentration by parts per million or percent by volume.

(e) Particulate matter (fiber and nonfiber) - specify allowable maximum particle sizes with maximum allowable number per unit volume.

(f) Particulate matter (viable) - specify allowable maximum limits.

15.6 Gas analysis. Gas analysis is the identification and quantitative measurement of gas constituents. These analytical measurements are significant to the producer and user. When applied the measurements:

(a) Provide a check on production processes and controls.

(b) Determine quality of end product.

(c) Provide a comparison with specifications.

(d) Detect trace impurities and contaminants.

(e) Provide continuous analysis or monitoring for compatibility with use requirements.

15.6.1 Analytical techniques. All analytical methods and techniques are fundamentally based on the interactions between matter and energy. Energy states, characteristic of the composition of any particular substance, are readily inferred by observing the consequences of interaction between the substance and an external source of energy. External energy sources may be grouped according to their basic modes of interaction with matter. A variety of analytical techniques and instrumentation is available depending on the type of energy, the means of using the energy, and the elements to be measured. Some of the techniques for gas analysis for each mode of energy used are shown in Table XXXIII.

Table XXXIII.

Analysis techniques for gases

Group 1	
Technique	Type Interaction
Ultraviolet spectrophotometry Light scattering Infrared spectrophotometry Microwave spectroscopy	Transmission and reflection
Definition	Energy Used
Measurement of the quantity and quality of electromagnetic radiation emitted, reflected, transmitted or diffracted by the sample	Reaction with other chemicals
Group 2	
Technique	Type Interaction
Orsat analyzer Automatic titrators Impregnated tape Photometry Colorimetry Gas chromatography Combustion Other thermic reaction	Consumption of sample or reactant Measurement of reaction products Thermal energy liberation
Definition	Energy Used
Measurement of results in terms of amount of sample or reactant consumed, product formed, thermal energy liberated, or equilibrium attained	Reaction with other chemicals

(continued)

Group 3	
Technique	Type Interaction
Mass spectroscopy Nier type Omegatron Time of flight	Ion separation
Gaseous conduction	Electrical properties
Paramagnetism Nuclear magnetic resonance	Magnetic properties
Definition	Energy Used
Measurement of the current, voltage or flux changes produced in energized electrical and magnetic circuits containing the sample	Thermal or mechanical energy
Group 4	
Technique	Type Interaction
Conductivity Dew point	Thermal
Sound velocity	Mechanical
Definition	Energy Used
Measurement of results of applying thermal or mechanical energy to a system in terms of energy transmission, work done, or physical state	Thermal or mechanical energy

15.6.2 Complete analysis. Methods for completely analyzing a specific gas may employ a variety of physical and chemical techniques and instrumentation, in different combinations, to provide identification and measurement of all constituents. The basic steps of analysis for a practical application consist of:

(a) Obtaining a true representative sample of the gas stream.

(b) Separating the sample into its components or constituents or identifying those constituents of analytical interest.

(c) Determining the quantity of each constituent with relation to the whole sample or to one another.

(d) Recording the analysis results.

15.6.3 Selection of methods. The selection of methods and instrumental techniques for gas analysis involves consideration of a number of factors. The factors include:

(a) How the techniques will be employed:

  (1) Laboratory analysis.

  (2) Continuous analysis (automated).

(b) Knowledge of the probable composition of the sample:

  (1) A knowledge of the constituents of analytical interest permits selection of instrumentation for a quantitive measurement of each known element.

  (2) Identification of unknown elements requires instrumentation for both identification and quantitive measurement.

(c) The physical state of the gas, which is normally gaseous. However, some constituents or impurities may be in other states such as:

  (1) Particulates, solid.

  (2) Moisture or water, liquid.

(d) Instrument performance which includes the following:

  (1) Sensitivity. The smallest change that can be reliably detected and distinguished from background noise or drift.

(2) Range. The span of concentrations of which the instrument is capable of measuring.

(3) Accuracy. The accuracy of instrument readings with respect to the actual value being measured.

(4) Temperature effects. Ambient temperature effects on instrument and requirements to compensate for temperature of sample.

15.6.4 Instrument sensitivity. The selection of methods and instrumentation for a complete analysis instrument must be tailored to the sample composition and the information requirements. Some techniques are rather universal, permitting identification and measurement of most constituents. These are known as specific type techniques and are usually more complex and costly, and sometimes less sensitive. The less specific types are usually simpler but are less able to distinguish between related substances having similar interactions with energy. The nonspecific instruments usually have greater sensitivity to extremely small concentrations of the element being measured and are usually more adaptable to continuous analysis operations and automation. Instruments and techniques used to measure concentrations of water vapor or moisture and to size and count particulates suspended in the gas are usually limited to these applications. However, in selecting this type specifically for moisture or particulate analysis, consideration must be given to other elements in the gas that will interfere with the instrument sensitivity.

15.6.5 Moisture monitoring instruments. Moisture monitoring instruments and techniques must be capable of measurements within the range of concentrations expected and the limits required. Other elements of the gas should not interfere or affect the sensitivity of the instrument. The adaptability to continuous and automated analysis should be considered if this is a requirement. Some of the instruments or techniques for humidity or water-vapor analysis are:

(a) Dew point. Other corrosive gases and vapors having dew points relatively close will cause errors.

(b) Electrical conductivity. Sample flow must be low; may be affected by other vapors and corrosive or water soluble gases.

(c) Hygrometers. Affected by contaminants absorbed on wet-bulb wick (mechanical hygrometers are also available).

(d) Volumetric absorption. (Pneumatic bridge).

15.6.6 Instruments for measuring particulate size and quantity. Instruments and techniques for determining sizes and quantity of particulates suspended in the gas must have measurement capabilities within the ranges required. Two of the instruments and techniques are the light-scattering photometer which determines particle size or concentration level, and the microscopic measurement which consists of collecting the sample on a filter media for measurement, usually a grab sample technique.

15.7 Handling of compressed gases.

15.7.1 Regulatory standards. Compressed gas users should comply with all applicable regulatory standards in the use, handling, and storage of compressed gases. Regulatory standards include:

(a) DOT regulations on the transportation of explosives and other dangerous articles.

(b) FAA regulations on the transportation of compressed gases by air.

(c) U. S. Coast Guard regulations on the transportation of compressed gases by water.

(d) ASME code on pressure vessels.

(e) ASME code on pressure piping.

(f) Compressed Gas Association standards.

(g) Local, state, and municipal codes and standards applicable to the use and storage of compressed gases.

These standards are considered minimum requirements. Supplemental procedures for the safety of operational personnel and the protection of equipment and facilities must be compatible with these minimum requirements, and should include such other procedures that are required for a particular operation or gas usage.

Compressed gases, like high explosives, are a form of stored energy and are potentially hazardous. Strict compliance with safe-handling practices, procedures, and standards by all personnel involved will ensure control of all potential hazards and incidents.

15.7.2 Safety practices. Five basic practices interdependent in achieving a high degree of safety in a compressed gas system are:

(a) Engineering design compatible with pressure, volume, and particular gas used.

(b) Assembly practices and processes with good quality control.

(c) Proper assembly in accordance with design specifications.

(d) Proper operation in accordance with safe practices.

(e) Adequate maintenance and inspection to assure continued safety.

15.7.3 Cylinder regulations. Compressed gas containers most commonly encountered by the user are cylinders. Rules and regulations pertaining to cylinders apply equally to spheres and drums where their alternate use is authorized by DOT regulations. Cylinder safety begins with the supplier by his adherence to DOT regulations covering:

(a) Inspection and testing.

(b) Proper filling.

(c) Valves.

(d) Safety relief devices.

(e) Marking and labeling.

(f) Transportation.

15.8 References.

1. Contamination Control Handbook, Sandia Laboratories, NASA SP-5076, 1969.

2. Department of the Navy, Naval Air Development Center, Warminister, Pa. 18974.

3. Grumman Aerospace Corporation.

4. Martin Marietta - Orlando.

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SECTION 16

CONTAMINATION CONTROL IN LIQUIDS

16.1 Contaminations in liquids. In rough clean operations, contamination in the cleaning solution may be a normal condition. In precision cleaning, however, the contaminant level is of definite concern and should be monitored periodically to assure that the required purity level is maintained.

16.1.1 Primary types of contaminants. Contaminants in liquids consist of five primary types:

(a) Nonvolatile residue.

(b) Other liquids in suspension or solution.

(c) Absorbed gases.

(d) Particulate matter.

(e) Microbial matter (viable particulates).

16.1.2 Origination of contaminants. These contaminants normally originate from the following sources:

(a) Contained in the liquid as received from the producer.

(b) Generated by use in cleaning processes or system operation.

(c) Contributed by storage tanks, pipes, valves, and other system components.

(d) Contributed by exposure to ambient air (particularly high-purity water).

16.1.3 Effects of contaminants in liquids. The effects of contaminants in liquids are similar to the effects of the same contaminants in any other environment. However, the method of migration is different, and the contaminant may in some cases assume different forms. A representative list of the effects of contaminants in liquids is shown in Table XXXIV.

Table XXXIV.

Effects of contaminants in liquids

Contaminant	Effects
Nonvolatile residue	Contact resistance Increased wear Corrosion Noncompatible with LOX systems
Particulate	Increased friction, wear, and erosion Mechanical restriction Electrical malfunction Photographic process degradation Corrosion
Microbial	Deterioration of material Cross infection Creation of nonsterile conditions Water and food pollution Purity dilution of pharmaceutical products
Other liquids	Dilution; reduced effectiveness Corrosion Chemical reactions; acidity, safety hazards

16.2 Fluid systems. The performance of hydraulic systems is directly related to the level of contamination within the system. Contaminants may induce wear on critical components, affect the forces necessary to drive valves or servomechanisms, and directly obstruct hydraulic lines.

16.2.1 Types of contaminants. An effective method of contaminant removal can be designed only when the types and effects of contaminants have been identified. Some sources and effects of significant types of contaminants are shown in Table XXXV.

16.2.2 Design considerations for a hydraulic filtration system. The life of systems whose accuracy or reliability does not warrant recirculating fluid filtration may be extended by careful cleaning and periodic fluid changes. The cleaning techniques used for components in the system (ultrasonics, vapor degreasing, chemical, etc.) are discussed in precision

Table XXXV.

Sources and effects of contaminants in hydraulic systems

Types	Sources	Effects
Dirt and dust	Area of operation of the system Environment of components before assembly or during assembly During manufacture of the components, such as core sand in castings	Wear by abrasion Wear by erosion Obstructs passages Activating force of valves and servomechanisms may be increased or erratic Acts as a chemical catalyst to promote oxidation of the hydraulic fluid
Water	Condensate resulting from low operating temperatures Introduced with fluid used in the system Improper drying of system after cleaning Enters through seals immersed in water or high-humidity environments	Oxidizes some metals used in hydraulic systems Accelerates oil oxidation with a metal catalyst present
Metal particles	Generated during manufacture of components Generated during assembly of the system Generated during operation of the system	Similar to dirt and dust
Rust	Improper use of carbon steel in the system Flash rust formed during cleaning of carbon steel parts	Similar to dirt and dust
Hydrocarbons Oil oxidation	Presence of air with the hydraulic fluid Accelerated by high temperatures Accelerated by presence of fine particles Accelerated by presence of water	Similar to dirt and dust
Polymerization	Generated by high temperatures Accelerated by presence of fine particles Accelerated by presence of water	Similar to dirt and dust Increases viscosity to change forces required for the system
Sheaves	Formed in presence of Buna-N rubber, oil, and metal High temperatures accelerate the reaction Still fluid accelerates the reaction	Similar to dirt and dust Generation of sheaves may etch away metal even between close tolerance parts such as pistons and cylinder walls
Miscellaneous contaminants	Parts packing material Paint on inside of pipe Surface coatings from improper preparation and cleaning before assembly	Similar to dirt and dust

cleaning. In assembled systems, a cleaning procedure may consist of flushing the lines with a cleaning solution, rinsing with deionized water, and drying with nitrogen. This procedure, however, is only typical; various steps may need to be added or repeated.

16.2.2.1 Recirculating liquid filters. When a hydraulic system requires a recirculating liquid filter assembly, the following factors should be considered in the system design:

(a) Level of contamination permitted.

(b) Type and amount of contaminants present.

(c) Available space.

(d) Volume and type of liquid to be filtered.

(e) Type of service and conditions of operation.

(f) Adaptability of the unit for adding auxiliary equipment.

16.2.3 Design considerations for a cleanable hydraulic system. In the design and assembly of hydraulic systems, several basic, but sometimes overlooked, factors should be considered in constructing a system that can be cleaned to the tolerances specified. These factors are as follows:

(a) The materials of construction must be such to permit cleaning to the level required with the cleaning materials and processes needed.

(b) The materials should be identified as to composition.

(c) The materials should be compatible with the liquid used in the system.

(d) Welds should be placed so as to be cleanable if at all possible.

(e) Welds should be checked out for impurities (scale, spatter).

(f) Drain and bleed-out parts should be provided for ease in cleaning.

(g) The system should not contain dead end plumbing.

(h) The components should be cleaned before assembly.

(i) The system should be assembled in a clean area, if possible.

16.2.4 Liquid filtration. Several types of particle separators are used with hydraulic systems such as gravity settling and centrifugation. However, the main method of separating particles from liquid is filtration. Two major types of filters are surface and depth, and a combination of the two may be used in recirculating hydraulic systems.

16.2.4.1 Surface filters. These filters remove solid particulate matter from liquids by a straining action which does not absolutely filter soluble contaminants and fine particles. The different types and uses of surface filters are described below:

(a) Extended area membrance-type filters. These filters are made from paper folded into an accordian pleat and enclosed within a housing. Consequently, this type of filter has a large surface area. By changing the density of the paper and the density of packing, particles of various size ranges can be removed.

(b) Bag filters. These filters are made from reinforced cloth in a configuration suitable for insertion in a housing. The filter characteristics are similar to (a).

(c) Wire screens, perforated metal, and discs of pressed sintered powdered metal. These filters are generally used to filter gross particulate contaminants and as support for other types of filter material.

(d) Magnetic fields. Magnetic fields may be used to separate magnetic materials from liquids.

16.2.4.2 Depth filters. These types of filters allow the liquid to pass through a filter medium. Absorbent and adsorbent filtering action are used in different types of depth filters.

(a) Absorbent filters. These types of filters use materials such as cotton, felt, paper, and hair. Filtration is accomplished as particles are trapped in the filter media. These filters are inactive chemically and remove only solid particles.

(b) Adsorbent filters. Adsorbent filters use materials such as charcoal, bone-black, and clay. This filter adsorbs some dissolved contaminants as well as removes particles by trapping. Adsorbent filters are, however, water-sensitive and must be used only when the system can be kept water-free.

(c) Combination filters. Combination filters can employ most of the above filter types to achieve a combination of filter effects.

16.2.5 Contaminant measurement methods. Water-vapor content of an empty system can be measured directly by using standard dew-point measurement techniques on the air in the system.

The general method for measuring particulate contamination is to collect a sample of fluid from the hydraulic system, filter it through a membrane filter, and count and size the particles. Individual components can be installed in special test systems to test for contaminants in the part or generated by the part. The components may be submersed in ultrasonic tanks to simulate the worst case of particle contamination due to vibration of the component. The Dual Volume sampling method is useful for reducing background count in these types of tests. In taking samples, many precautions must be taken to avoid particle generation and inaccurate test results.

16.3 Analysis of contaminants in liquids. Some of the contaminants found in liquids and particularly those contributed by the system were discussed in Table XXXV. Monitoring methods for viable contaminants in liquids may be accomplished in two ways.

(a) They can be passed through membrance filters and then the filters subjected to appropriate techniques to culture the organisms. Membrance filters can be obtained in pore sizes from 0.01 to 10 or more with flow rates of from 0.5 to 100 ml/min/cm^2 depending on the pore size.

(b) Liquids may be subjected to aliquot sampling and the samples tested for viable contamination. Statistical analyses can be made by the most probable number (MPN) technique to determine estimates of microbiological loading. This technique is less accurate than the use of membrane filters.

16.3.1 Procedure for the determination of particulate contamination of hydraulic fluids by the particulate count method. Particulates and other nonvolatile residue present the other greatest concern in contamination control. Gross particulate matter can be seen visually, and smaller particles can be detected by the use of membrane filters and microscopes. One method for detecting particles 5 microns and larger is described in Aerospace Recommended Practice (ARP) 598.

1. Scope. This test describes a self-checking procedure for the determination of particulate contaminant five microns or greater in size in hydraulic fluids by the particle count method. A maximum variation of two to one ($\pm$33% of the average of two runs) in results should be expected for replicate counts on the same sample, providing that the procedure is followed closely.

1.1 Outline of method. A fluid is filtered through a type HA Millipore filter disc using vacuum to impinge the contained contamination particles upon the surface of the filter. The filter disc is examined microscopically (using oblique incident lighting) to determine the amount of contaminant present in stated size ranges.

1.2 Apparatus.

(a) Pyrex filter holder, Millipore Cat. #XX 1004700, or equivalent, which includes:

  (1) Fritted glass base and rubber stopper.

  (2) A holding clamp.

  (3) A 250 ml pyrex glass funnel.

(b) A filter cover for the glass funnel to minimize contamination from the air passing through the funnel during the vacuum filtration process. The optimum cover would incorporate a Millipore filter in the cover device.

(c) Membrane filter 0.45 micron, type HA black grid 047 mm diameter Millipore, or equivalent. These to have an imprinted grid on 3.08 mm centers. Each grid square is equal to 1/100th of the total effective filtering area of the filter disc when used in the Millipore pyrex filter holder (above).

(d) Vacuum flasks.

(e) Aspirator or vacuum pump, capable of pulling a minimum 26" of mercury.

(f) Plastic petri dishes, disposable, Millipore Cat. #PD 10 047 00, or equivalent.

(g) Forceps with unserrated tips.

(h) Sample bottles, small mouth, glass, etched or otherwise permanently marked to indicate 100 ml. sample size.

(i) Microscope with mechanical stage, capable of magnification of approximately 45X and 90X. For 90X magnification, the recommended objective is 10 to 12X but at least 6X with a numerical aperture of at least 0.15. The optimum equipment is a binocular microscope with a micrometer stage. A stereo microscope should not be employed with this procedure.

(j) Measuring eyepiece - ocular micrometer - Baush & Lomb Catalog #31-16-01, or equivalent.

(k) Stage micrometer, 0.1 to 0.01 mm calibrations.

(l) Microscope lamp, high intensity, variable. This lamp is to be used as a source of oblique incident light; Leitz microscope lamp MONLA (or equivalent); 5,000-6,000 candlepower at filter surface.

(m) Wash bottles, pyrex glass.

(n) Mylar plastic films, 2" x 2" x .002".

MONLA - 6V - 5A microscope lamp with focusing illuminating lens on pillar stand with separate transformer.

1.3 Reagents.

(a) Distilled water.

(b) Acetone free, reagent grade isopropyl alcohol.

(c) Petroleum ether 30 -60° boiling range (Freon TF or equivalent may be substituted where explosive vapors are not accepted.)

1.4 Filtration of reagents.

1.4.1 Filtration process. For initial establishment of clean conditions for filtration of reagents.

(a) Clean apparatus. All apparatus should be cleaned as follows:

(1) Wash with self-rinsing type detergent and water.

(2) Thoroughly rinse with hot soft tap water.[2]

(b) Assemble pyrex filter holder with filter disc in place.

(c) Filter 100-200 ml. of isopropyl alcohol into the filter flask. Remove entire pyrex filter assembly and rinse the filter flask with the filtrate.

(d) Repeat step (c) three times.

(e) Filter a 100-200 ml. volume of petroleum ether into the filter flask. Remove the funnel assembly and rinse the filter flask with the filtrate.

[2] When filtered distilled water, filtered isopropyl alcohol, and filtered petroleum ether have been obtained, the apparatus should be cleaned as outlined in Paragraph 1.5.

(f) Repeat Step (e) three times.

(g) Filter the desired volume of the solvent.

1.4.2 Control analysis for reagent cleanliness.

(a) Clean a Pyrex filter holder and a wash bottle using the process outlined in Paragraph 1.5.

(b) Place 500 ml. of the filtered reagent in the cleaned wash bottle.

(c) Rinse the upper surface of a filter disc with the filtered reagent from the wash bottle. Place the disc on the fritted glass base and complete the assembly of the filtration apparatus.

(d) Pour a 150 ml. portion of the filtered reagent into the funnel directly from the filter flask. Filter the sample using the method for processing a blank described in Paragraph 1.7.2.1 except that the filter disc is to be rinsed only once and with a 50 ml volume of the filtered reagent from the wash bottle. If the desired cleanliness level is not obtained,[3] repeat the cleaning and filtration process using the filtered reagents on hand for the cleaning process.

1.5 Cleaning method for apparatus and sample bottles.

1.5.1 General instructions. Lint-free coats should be worn by personnel performing cleaning operations to preclude excessive fiber contamination.

1.5.2 Cleaning methods. Each item of filtration apparatus will be cleaned before each run of samples and each sample bottle and cap will be cleaned before each use by the following method.

(a) Rinse with two successive rinses of petroleum ether. (Hexane can be used as an alternate and is recommended because of its superior properties).

(b) Wash thoroughly in a solution of detergent and hot water. Rinse twice in hot tap water (soft).

(c) Rinse with filtered distilled water twice.

(d) Rinse with filtered isopropyl alcohol to remove water.

[3] Contamination content of a 150 ml. sample of filtered reagent, so processed, should be no greater than the values specified for a blank analysis. See Footnote 6. for blank analysis specifications.

(e) Rinse with filtered petroleum ether.[4]

(1) Filtration Apparatus. After rinsing with petroleum ether (hexane), hold in an inverted position for 15 seconds to allow drainage and evaporation of the petroleum ether (hexane).

(2) Sample Bottles. After rinsing with petroleum ether (hexane) allow a small quantity of petroleum ether to remain in the bottle since the vapor pressure will help preclude contamination when the bottle is opened. Rinse a previously cleaned 2" x 2" plastic film with filtered petroleum ether (hexane). Place the plastic film over the top of the bottle and install the bottle cap.

1.6 Samples. A 100 ± ml. sample is to be used for this procedure.

1.6.1 Sampling procedure. Samples for this test method should be as representative as possible of the fluid being sampled. Procedures for procuring such samples will, of necessity, have to be established by individual plants or laboratories. Extreme care should be taken to preclude the introduction of external contamination at this point. To assure reproducibility, the sampling program should be checked at the outset by the testing of replicate samples from the sampling port.

1.7 Test procedure.

1.7.1 Test information.

(a) Personnel performing contamination analyses should wear lint-free laboratory coats.

(b) Samples are to be obtained in accordance with specified sampling procedures.

(c) The filtration apparatus is to be cleaned just prior to use by the method outlined in paragraph 1.5.

(d) The microscope and its accessories should be maintained in a state of maximum cleanliness. The microscope and accessories should be protected by a dust cover when not in use.

(e) The processing and microscopic analysis of samples should be performed in as clean an area as possible within the confines of

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[4] When high humidity conditions exist, Step (e) of paragraph 1.5.2 may be followed by an additional isopropyl alcohol rinse to prevent condensation.

a modern, air-conditioned laboratory. A dust control room[5] is desirable, but not essential for validity and reproducibility. Smoking should be prohibited, both as a safety factor and to prevent the extra contamination of samples. The ingress and egress of personnel in the laboratory area should be limited.

1.7.2 Filtration procedures.

1.7.2.1 Procedure for blanks. Prior to each sample analysis, a blank analysis is to be performed on 50 ml. of filtered petroleum ether contained in a regular, clean sample bottle. The procedure used will be identical with that described below, with the exception that paragraph 1.7.2.2, step (f) will be omitted. (The total amount of petroleum ether used in the blank analysis should be 200 ml., which is approximately the volume used in the filtration of a sample.) The blank analysis is performed to determine the amount of contamination being introduced by the sample bottle, filtering process, filtering equipment, filter disc, and the filtered petroleum ether. Identify the blank as to the sample number on an identification tag and attach it to the lid of the petri dish.[6]

1.7.2.2 Procedure for samples.

(a) Using forceps, remove one filter disc from its container. Rinse the top surface of the filter disc with a stream of filtered petroleum ether from a wash bottle. Place the filter disc-printed grid side up on the fritted glass base.

(b) Immediately lower the filter funnel onto the fritted glass base, secure with the holding clamp and place cap on top of the filter funnel. (Do not slide filter funnel over the filter disc during this process.)

(c) Thoroughly agitate the sample bottle to assure that all solid particles are in suspension.

(d) Remove the sample bottle cap and plastic film. Remove the filter cap and pour sample into the filter funnel. Replace the filter cap.

[5] A hood pressure with filtered air is especially advantageous for both microscopy and filtration. At a minimum, a dust cover such as polyethylene bag, with appropriate openings, pulled over the barrel of the microscope, is required to preclude fall out of dust from the air.

[6] The maximum particle count value of a blank analysis shall be no greater than 10% of the count of an acceptable sample for a specific laboratory.

(e) Pour 100 ml. of filtered petroleum ether into the sample bottle; replace the plastic film and bottle; agitate and proceed as in (d).

(f) Rinse funnel walls with approximately 50 ml. of filtered petroleum ether (hexane) from wash bottle.

(g) Apply vacuum to the filtering apparatus. When the filtration is approximately one-half complete, release the vacuum.

(h) While some liquid still remains in the funnel, using the stream from a wash bottle, carefully wash down the sides of the funnel with filtered petroleum ether (hexane) approximately 50 ml. Replace the filter cap.

(i) Apply vacuum and allow to operate until the filter disc is completely dry. Do not rinse the funnel walls further after the filter has become dry, as this will upset the distribution of particles on the filter surface. Turn off the vacuum and simultaneously remove the holding clamp and fiter funnel so that the filter disc remains on the fritted glass base.

(j) Using forceps, carefully remove the filter disc from the top of the fritted glass base. Place the filter disc, grid side up, in a clean petri dish and replace petri dish cover. Maintain filter patch level until particles have been counted.

(k) Identify the petri dish using a sample identification tag. The test may be delayed overnight, if necessary, after completing this step.

1.7.3 Microscope analysis procedure. Particles are to be counted and tabulated in the following order: fibers, particles greater than 100 microns, 50-100 microns, 25-50 microns, and 5-15 microns. Particles smaller than 5 microns are not to be counted by this method. Fibers are defined as any particle whose length to diameter ratio exceeds 10 to 1 regardless of composition. Fibers are counted as particles and not differentiated unless their length exceeds 100 microns. The size of a particle is determined by its greatest dimension. See paragraph 1.7.4., (g).

(a) Place petri dish under the microscope dust cover and remove petri dish cover.

(b) Adjust the microscope lamp intensity to obtain maximum particle definition.

(c) A magnification of approximately 45 X shall be used for counting particles 25 microns or larger; approximately 90X for particles smaller than 25 microns. The recommended objective to obtain the 90X magnification is 10 to 12X power in conjunction with the appropriate eye-piece.

(d) Using a stage micrometer, calibrate the measuring eye-piece (ocular micrometer) for each magnification.

1.7.4 Method of counting particles. Other statistical methods may be employed provided that the method shows agreement with the values of the certified standard samples.

(a) In obtaining the number of particles of a given particle size range, the number of particles on a representative number of grid squares on the filter disc are counted. From this count, the total number of particles, which would be present statistically on the total effective filtration area of 100 imprinted grid squares, is calculated.

(b) If the total number of particles of a given particle size range is estimated to be between 1 and 50, count the number of particles over the entire effective filtering area.

(c) If the total number of particles of a given particle size range is estimated to be between 50 and 1,000, count the number of particles in 20 randomly-chosen grid squares and multiply this number by 5 to obtain the total statistical particle count.

(d) If the total number of particles of a given particle size range is estimated to be between 1,000 and 5,000, count the number of particles on 10 randomly-chosen grid squares and multiply this number by 10 to obtain the total statistical particle count.

(e) If the estimated total number of particles of a given size range exceeds 5,000, count the particles within at least ten (10) randomly-chosen unit areas.[7] To arrive at the total statistical count, the sum of the particles counted in the areas is multiplied by the calibration factor.[8]

(f) In no case shall the total number of particles in a unit area exceed 50 particles of a size range. See Figure 11 for the alternate unit areas.

[7] The basic unit area for the statistical count not based on the grid markings on the filter, when using the ocular micrometer, will be the area defined by scanning the length of an individual grid square with the length of the ocular micrometer scale or any appropriate portion of the scale.

[8] Calibration factor defined in 1.7.5.

(g) If a particle lies on the upper or left boundary line of a counting area, count this particle as if it were within the boundaries of the counting area.

(h) The largest dimension of the particle determines the size category into which the particle is placed.

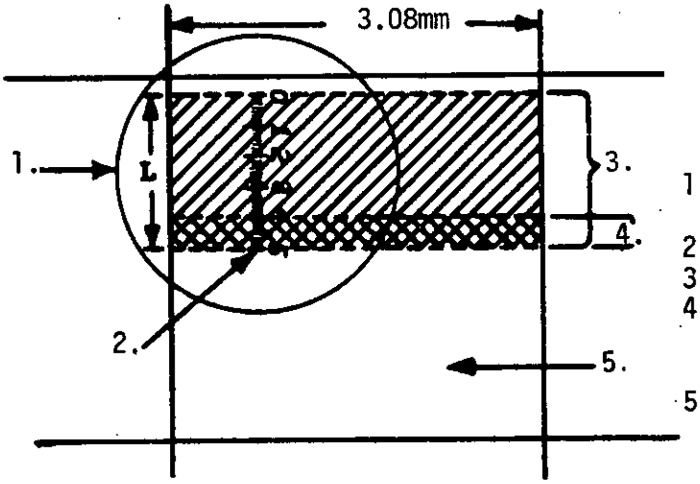
(i) In the areas of no count or no silting allowed, see silting definition in MSC-STD-C-1.

1.7.5 Calculation of calibration factor:

(a) The calibration factor is the ratio of the effective filtration area (100 grid squares or 9.6 cm^2) to the area counted.

(b) To arrive at a calibration factor, start with the microscope adjusted for the power under consideration.

(c) Using the stage micrometer, measure the length of the ocular micrometer scale which is used to define the width of the unit area. The length of the unit area is defined by the side of the grid square or 3.08 mm.



1. Field observed through microscope.
2. Ocular micrometer scale.
3. Suggested unit area.
4. Alternate unit area (for highly contaminated sample).
5. Grid square.

Figure 12. ARP 598 Possible unit areas within a grid square for statistical counting.

1.7.5.1 Formula for calibration factor. Figure 11 shows two possible unit areas within a grid square for statistical counting. The calibration factor is defined as the effective filter areas divided by the total area counted and may be calculated from the following formula:

$$F = \frac{960}{3.08 \times L \times N} \quad \text{or} \quad \frac{312}{L \times N}$$

where F = the calibration factor
N = the number of basic unit areas counted
L = the calibrated length of the ocular micrometer scale or the portion of scale used in defining unit area - in millimeters
3.08 = the side of a grid square in millimeters.

For example: Using a 10 X objective and a 5 mm ocular micrometer, counting 10 basic unit areas, the calibration factor would be

$$F = \frac{312}{0.5 \times 10} = 62.4,$$

and the total number of particles of a size range would be

$$N_T = 62.4\ N_c$$

where N_c = the number of particles actually counted in the 10 basic unit areas.

1.8 Self-checking procedure.

1.8.1 Manipulation of the microscope. Facility in the manipulation of the microscope can only be acquired by practice in perfecting focus, adjusting illumination, and sizing particles with the ocular micrometer. In order to obtain accurate results in contamination analysis, the operator must determine the capabilities and characteristics of his optical equipment and be able to use this equipment without hesitation and in such a manner as to take full advantage of these capabilities.

The widest variable in results obtained in applying the ARP is caused by the variance between companies in microscope techniques. It is therefore important, in order to establish meaningful and comparable analytical results, that the optical techniques described in the ARP be followed closely, and that these techniques be checked by means of check samples as defined in Paragraph 1.8.2. Calibration of the ocular micrometer and individual interpretation of the size range limits defined by the ocular micrometer rulings should also be checked by means of calibrated standards.

1.8.2 Check samples. Sample filter discs, permanently mounted and containing representative contaminant from hydraulic fluid systems should be used to check the optical portion of the analytical procedure. The actual particle count of these filter discs should be known within the limits of statistical accuracy and, if possible, should be verified by more than one laboratory. The procuring agency may provide these check samples or may recommend a source of suitable samples.

1.8.3 Self-checking aspect of the procedure. By employing blank analyses for checking the filtration and mounting techniques, and samples for checking the microscope counting aspect of the ARP, laboratories may provide themselves with a means for checking the repeatability and reproducibility of the procedure. By analyzing replicate samples from the same sampling part, the laboratories may determine the accuracy and reproducibility of sampling procedures.

1.8.3.1 Repeatability. (precision) The precision of results obtained with the ARP should be initially checked and periodically retested by analyzing replicate blanks, samples, and viewing fields in accordance with the ARP. The precision of results obtained in a single laboratory should also be checked by having all operators concerned occasionally analyze duplicate test samples and compare results.

1.8.3.2 Calibration. (accuracy) In order to establish meaningful inter-laboratory acceptable contamination levels and filtration efficiency data, it is important that the accuracy of the analytical procedure as employed in any individual laboratory be established by means of the calibrated industry-wide check samples. In this manner the Aircraft Recommended Procedure as described herein becomes a self-checking procedure on both an individual and inter-company scale, as well as on an industry-wide basis.

Prepared by the Contamination Control Panel
of
SAE Committee A-6, Aircraft & Missile Hydraulic
& Pneumatic Systems & Equipment

16.3.2 Nonvolatile residue (NVR) method. A more quantitative method is generally required, however, for most contamination control work. This is described in the following discussion of nonvolatile residue.

Nonvolatile residue (NVR) is defined as the soluble (or suspended material and insoluble particulate matter remaining after temperature-controlled evaporation of a filtered volatile liquid. The amount of NVR content in solvents must be controlled in cleaning processes in order to obtain desired cleanliness of the product since the NVR will remain on the product after the solvent evaporates. The final rinse stage of any precision cleaning process must utilize only solvent with an extremely low NVR content. Earlier stages in a multistage cleaning operation may be less critical in the control of NVR.

16.3.2.1 Typical contaminants in NVR control. Some typical contaminants encountered in NVR control are:

(a) Skin oils.
(b) Solder fluxes.
(c) Cutting fluids.
(d) Plasticizers.
(e) Cleaning solvents.
(f) Lubricants.

16.3.2.2 Primary NVR methods. The primary methods of NVR detection are:

(a) Gravimetric
(b) Nephelometer
(c) Solvent purity meter
(d) Chromatography
(e) Spectrophotometry.

The five primary methods for NVR detection are compared in Table XXXVI. Comparison chart NVR techniques.

16.3.2.3 Diagrams. The following diagrams depict the apparatus and steps involved in each NVR detection method. These methods are described and compared in NVR Detection Techniques.

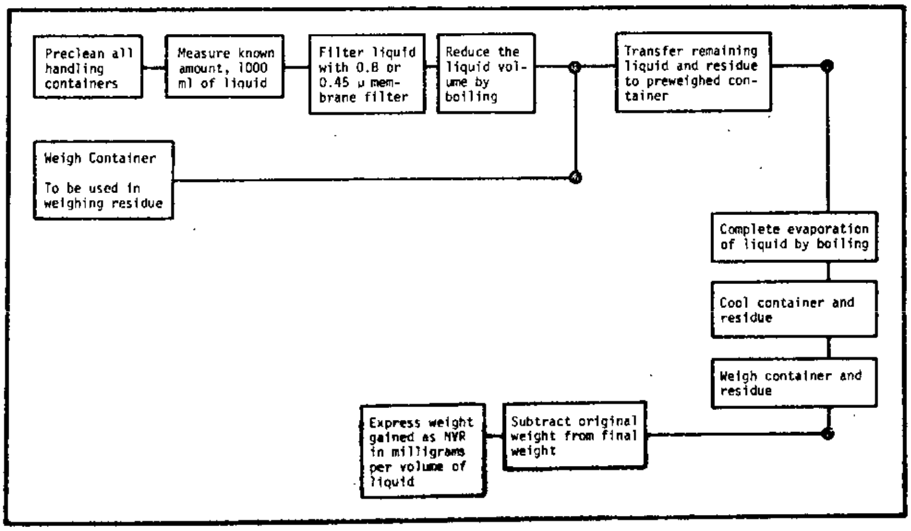


Figure 13. Typical gravimetric flow diagram

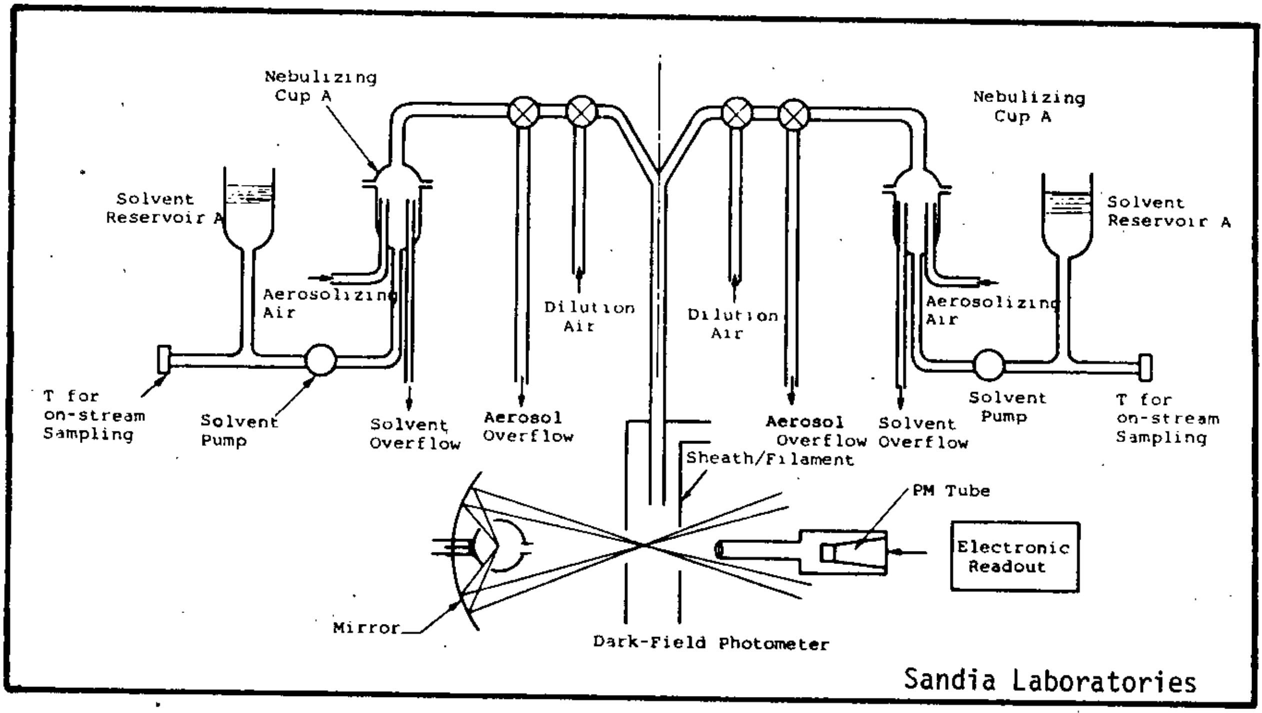


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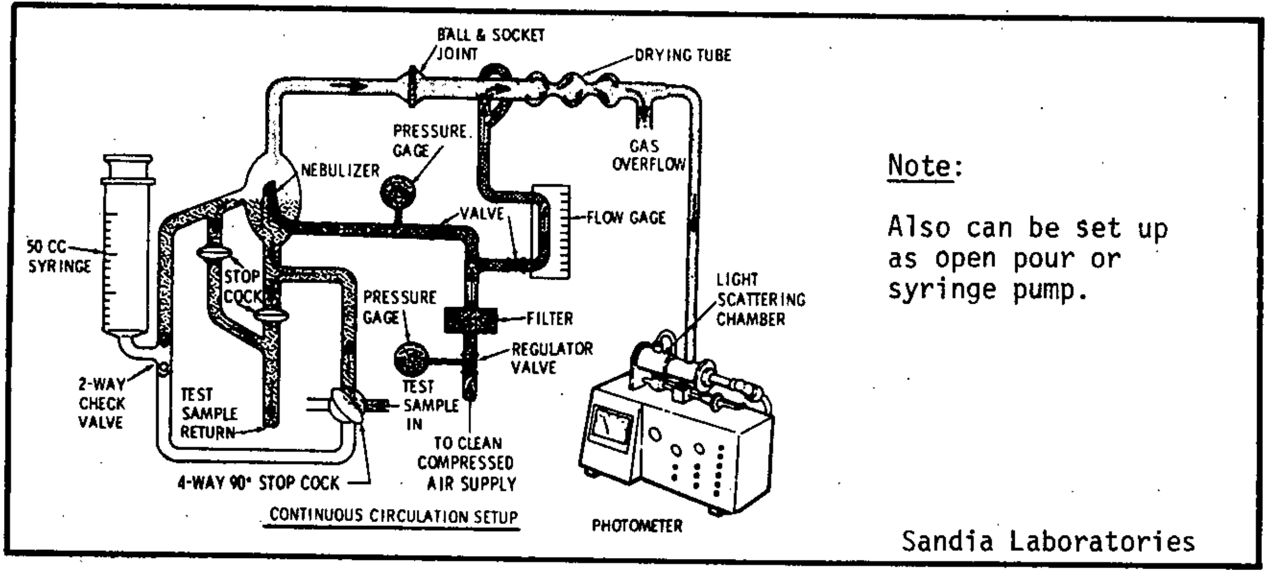
Figure 14. NVR nephelometer schematic layout



Note:

Also can be set up as open pour or syringe pump.

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Figure 15. Solvent purity meter diagram

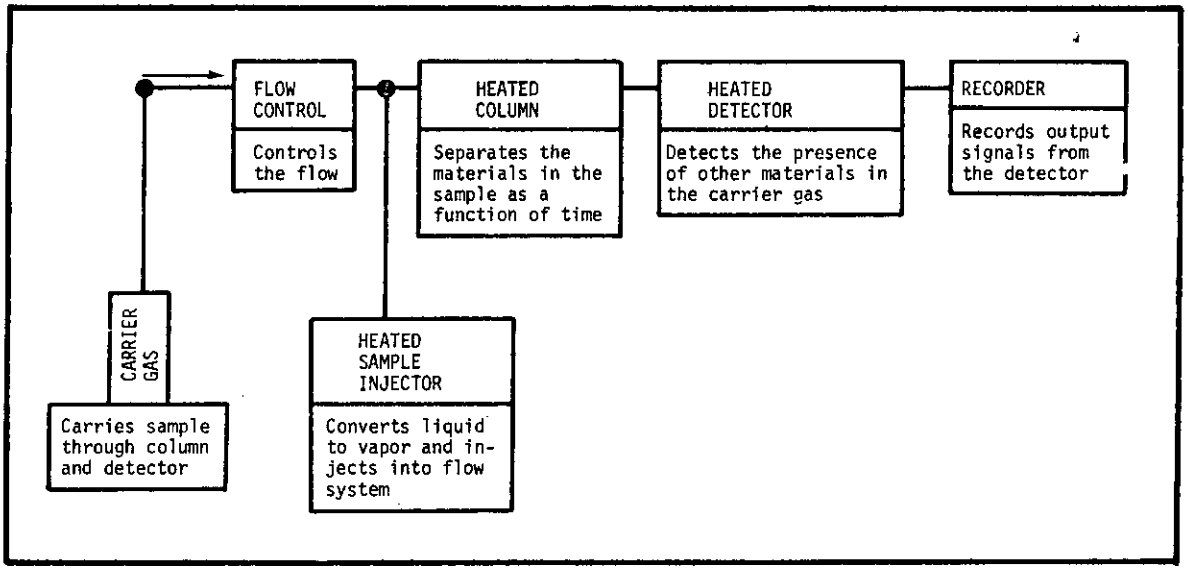

Figure 16. Block diagram of NVR chromatography

16.3.2.3.1 Chromatography. Chromatography is a method of separating the constituents of a mixture based on their differences in solubility and mobility through a packed column. The time required for each component to travel through the packed column and appear as a peak on the recording trace is its retention time. Under controlled conditions, each component will exhibit a definite retention time, and this time provides significant information to its identity.

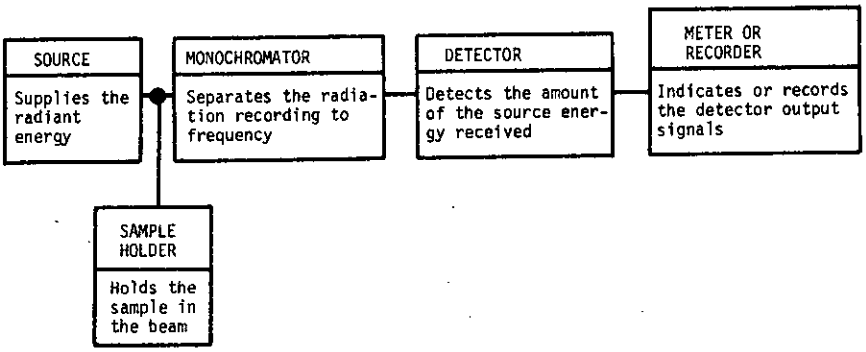

Figure 17. Block diagram of spectrophotometer

Table XXXVI.

Comparison chart for NVR detection techniques

Comparison Characteristic	Gravimetric	Nephelometer	Solvent Purity Meter	Spectrophotometry	Chromatography
Principles of operation	Performed by weighing a container and evaporation (by boiling) a known amount of solvent and reweighing the container. The weight gain is NVR.	Solvent is aerosolized by clean air and sampled by forward light-scattering photometer. As NVR content is increased, the rate of evaporation is decreased resulting in larger droplets at the point of scattering, which produces larger signal output of the photometer	Same as nephelometer	Absorption of electromagnetic radiation	Multitheoretical plate Distillation Selective adsorption
Sensitivity	0.01 ppm by weight	1 ppm	1 ppm	Varies from solvent to solvent; 1 ppm optimized	1 ppm
Time for determination	May vary from 0.75 to 8.0 hours	5 minutes	1 minute	5 minutes to 1 hour	5 minutes to 1 hour
Units of measurement	Milligrams per volume, and may be converted to ppm by weight	ppm either by weight or by volume after converting photometer reading by use of calibration curves	ppm either by weight or by volume after converting photometer reading by use of calibration curves	Percent transmittance converted to ppm or actual weight	Retention volume and direct readout are difficult
Operator aspect	Well versed in laboratory techniques, contamination control practices, and precise measurements	On development models was found simple to operate but somewhat difficult to maintain	Extremely simple to operate. Production personnel operate with no difficulty	Comprehensive knowledge of chemistry required for setup and interpretation	Comprehensive knowledge of chemistry required for setup and interpretation
General Information	Widely used, recognized by ASTM Standards, may be costly in time and labor. Batch sampling only	Developed by IITRI for NASA. Shows great promise. Batch or continuous sampling	Developed by Sandia and is being marketed by commercial firms. Batch or continuous sampling	Can be quite expensive. Quite specific in application.	May be somewhat limited in identifying the variety of contaminants in a residue

16.3.2.4 Specifications and tests for NVR.

(a) ASTM-NVR-66, Tentative Method of Test for Nonvolatile Residue of Halogenated Solvent Extract from Aerospace Components (Using Rotary Flash Evaporator).

(b) ASTM D 1025-52, Nonvolatile Residue of Polymerization Grade Butadiene.

(c) ASTM D 1353-65, Nonvolatile Matter in Lacquer Solvents and Diluents.

(d) ASTM D 2109-64, Nonvolatile Matter in Halogenated Organic Solvents.

(e) FST-2, Quality Specifications and Methods of Analysis for Freon Precision Cleaning Agent.

16.4 Removal of contaminants from liquids. Contaminants may be removed from liquids by physical, thermal, or chemical processes. The complexity of the process may be fairly simple (for some particulates) or highly involved (for liquids having similar vapor pressures).

16.4.1 Factors influencing selection of particular contaminant removal method. Contaminants may be removed from liquids in many ways, any one of which may be the most feasible for a given situation. Some of the factors which will influence the selection of a particular method are:

(a) The volume of liquid to be processed.

(b) The type of contaminants to be removed.

(c) The boiling point and vapor pressure of the liquid (and of the contaminant, also, if it is a liquid).

(d) The specific gravity of the contaminant.

(e) The degree of purity required.

(f) The time required for purification or output rate of the process.

(g) Economic considerations of space, initial purchase, operation, personnel, and maintenance.

16.4.2 Primary removal methods. The primary methods of removing contaminants from liquids are as follows:

(a) Distillation.
(b) Sedimentation and drain off.
(c) Filtrating.
(d) Centrifugation.
(e) Deionization.

A summarization of these methods are noted in Table XXXVII.

Table XXXVII.

Methods for removing contaminants from liquids

Removal Method	Principle	Type of Contaminant Removed
Distillation	Evaporation and condensation	Liquids Nonvolatile residue Particulates Microbial organisms
Sedimentation and drain off	Gravity settling	Particulates
Filtration	Straining Adsorption Absorption Magnetic attraction	Particulates Microbial organisms
Centrifugation	Gravity	Particulates Microbial organisms
Deionization	Ion exchange	Dissolved minerals in water

16.4.2.1 Characteristics of primary methods. Some of the characteristics, limitations, and applications of these methods are described as follows:

(a) Distillation. Apparatus for this method can vary from a single laboratory setup to large, sophisticated distillation systems. Along with filtration, this is probably the most universally used method for purifying liquids because it will remove most of the contaminants normally found in liquids in a relatively economical manner. Distillation plays an important role in the reclamation of manufacturing grade solvents and in the repurification of technical or precision grade solvents.

(b) Sedimentation. This is a time-consuming but effective method for removing particulate matter from liquids. It is generally applied to removing gross particulates and is not recommended when extremely high purity is required. The purity attainable is a function of:

(1) The time allowed for settling.

(2) The specific gravity of the contaminant as compared to that of the liquid.

(3) The degree to which agitation of the liquid can be controlled and minimized.

The purified liquid is usually drawn off at a point in the upper portion of the container and the sediment is discharged. An exception to this procedure is the removal of heavy sludge from industrial washers by sludge conveyors.

(c) Filtration. All filters perform the basic function of collecting and retaining contaminants. The type of filter selected for a particular application should be determined by the type and size of contaminant encountered, flow rates, and purity level desired, and the anticipated filter cleaning or replacement frequency. Some filters such as wire screens, perforated metal, and the larger pore size porous metal can be cleaned; most other types must be discarded and replaced. Filters may be used in-line to filter the entire volume of liquid in either batch or recirculating systems, or they may continuously filter only a portion of the liquid, as in the case of some ultrasonic cleaners. Membrane filters in flat or extended area configurations are used almost exclusively for filtering ultrapure liquids. Additional information on the types of filters available is discussed in Fluid Systems, Paragraph 16.2.4.1.

(d) Centrifugation. This principle is similar to that of sedimentation. The primary difference is the time required for particulate removal. High-speed centrifuges greatly accelerate removal time for even those substances with very little difference in specific gravity. Centrifugation is used widely in removing microbial organisms from a liquid.

(e) Deionization. Dissolved mineral ions can be removed from water by deionization. The Figure 18 shows a typical continuous recirculation system for deionized water. Resin bed "C" on the left removes the cationic impurities by exchanging the cationic impurities for hydrogen ions. Resin bed "A" on the right removes the anionic impurities in a similar exchange action. Mixed bed exchangers can accomplish the same result as the two-bed system and may be used downstream in addition to the two-bed system. Virtually, all dissolved contaminants can be removed from water in this manner.

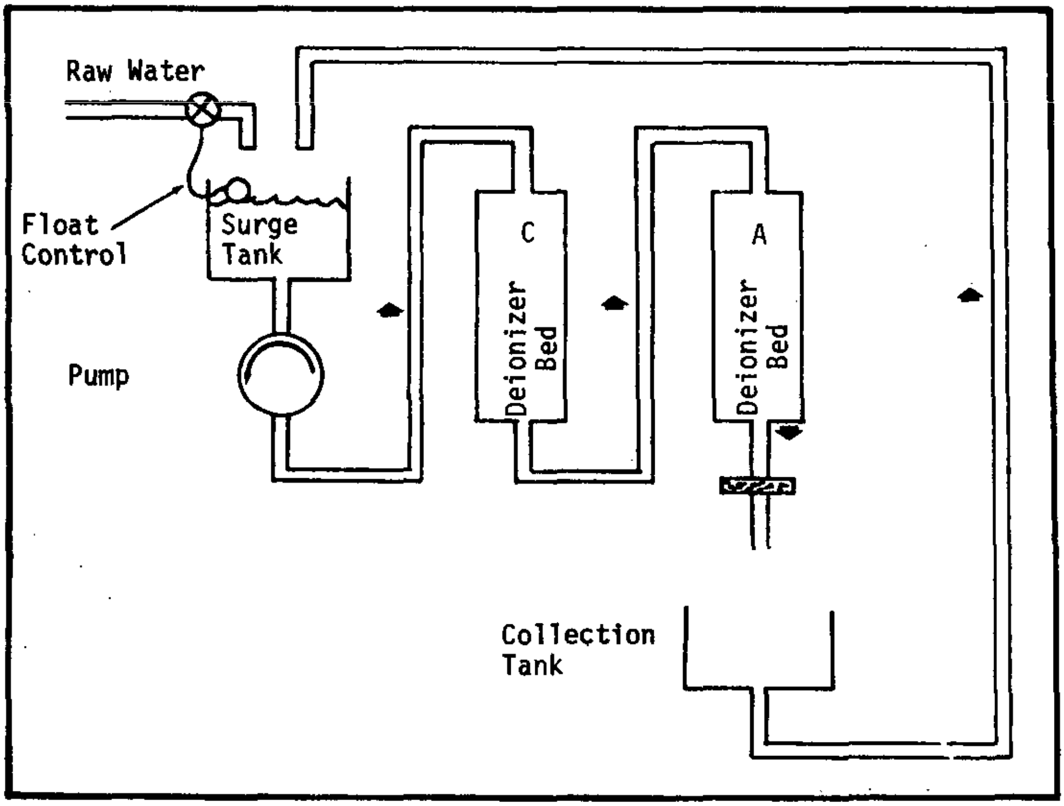

Figure 18. Typical deionized water system.

16.5 References.

1. Contamination Control Handbook, Sandia Laboratories, NASA SP-5076, 1969.

2. Department of the Navy, Naval Air Development Center, Warminster, Pa.

3. Grumman Aerospace Corporation.

4. Martin Marietta Corporation.

5. Procedure for the Determination of Particulate Contamination of Hydraulic Fluids by the Particulate Count Method, ARP 598, SAE Committee A-6, Aircraft and Missile Hydraulic and Pneumatic Systems and Equipment.

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SECTION 17

GROSS CLEANING

17.1 Gross cleaning as associated with contamination control. Gross or rough cleaning may not be normally associated with contamination control as it appears to space hardware. It is, however, often the first cleaning step in a comprehensive contamination control program. Inadequate removal of gross soils can lead to subsequent degradation of precision cleaning solutions or the failure to completely remove certain soils.

17.1.1 Mil-Std-1246A definition of gross cleaning. According to Mil-Std-1246A, gross cleaning is a cleaning method performed to achieve visible cleanliness. Gross cleaning removes contaminants such as, but not limited to, weld scale, heat treat scale, corrosion, oxide, films, oils, grease, shop soil, fuel, and carbon deposits. The cleanliness level achieved by gross cleaning does not normally require verification beyond visible appearance. (Wipe test, water break test, ultra violet inspection, special lights and mirrors, are considered aids in visual inspection.) Gross cleaning is considered a normal shop process and usually does not require special environmental controls, packaging, handling, or storage beyond accepted good practice that will not degrade the quality of the product.

17.2 Cleaning methods most frequently employed. In a general sense, the cleaning methods which are considered most frequently employed in gross cleaning are:

(a) Barrel cleaning.

(b) Abrasive blast cleaning.

(c) Mechanical cleaning.

(d) Washers.

(e) Filters.

(f) Agitation.

A description of each of the above mentioned is as follows:

17.2.1 Barrel cleaning. Barrel cleaning is accomplished by the tumbling action of parts and a medium within a rotating enclosure. The process may be used either wet or dry and with a variety of media for the removal of different soils. The amount of tumbling action varies inversely with the total mass of parts and media in the barrel. The total mass is usually set at 50 to 90 per cent of the barrel volume.

Although other cleaning methods are generally used for the removal of oil and grease, barrel cleaning can be used for this purpose. Cleaning action is provided by an alkaline solution, solvent or emulsion, or a suitable combination of the three.

17.2.1.1 Barrel descaling. Barrel descaling is more commonly used than barrel cleaning. Acid compounds are usually preferred for their faster action, and may be supplemented by wetting agents and abrasives.

17.2.1.2 Dry tumbling. Dry tumbling is a means for gross cleaning castings and forgings. Mill scale, sand, and hardened mold release materials are effectively removed by this method. After tumbling, the work must be removed from the medium. Magnetic separating devices may be used for ferrous parts and reciprocating or vibrating screens for nonferrous parts.

17.2.1.3 Factors to consider in barrel cleaning. To achieve the best results from barrel cleaning, the following factors must be considered:

(a) Barrel speed. Proper speed to regulate part movement without incurring centrifugal force sufficient to hold parts on the barrel surface.

(b) Compound. Proper for application; noncorrosive; maintained at adequate concentration.

(c) Water level. Sufficient to prevent excessive tumbling without completely filling barrels.

(d) Medium. Correct material, size, shape, and quantity to provide desired scrubbing or abrasive action.

(e) Work pieces. Proper size to be accommodated by the barrel, configuration to prevent interlocking, and composition.

(f) Flushing. Parts and barrel should be flushed after tumbling to remove objectionable residues.

17.2.2 Abrasive blast cleaning. Abrasive blast cleaning involves the forceful impingement of abrasive particles against a metal surface to remove unwanted soils. The particles are impelled toward the work surface by compressed air or by means of a high-speed bladed wheel. Both wet and dry processes are available.

17.2.2.1 Abrasive blast methods. Abrasive blast methods are commonly known by the type of abrasive they use, i.e., sand-blasting, grit blasting, and glass-bead blasting. Blast methods also employ the use of organic abrasives such as crushed corncobs, rice hulls, and walnut shells for milder abrasive action.

17.2.2.2 Protective apparel. Protective apparel and apparatus for personnel operating abrasive blasting equipment are essential. These should include a suitable combination of safety glasses, an independent air supply, protective helmet with air supply, abrasive-resistant aprons, and gauntlet gloves, as well as appropriate leg and ankle protection. In the case of dry blasting, all necessary precautions should be taken against the possibility of operators contracting silicosis. Respiratory devices should be types approved by the U. S. Bureau of Mines. Table XXXVIII compares the various wet and dry abrasive blasting methods.

Table XXXVIII.

Comparison of wet and dry abrasive blasting methods

	Wet	Dry
Type of force used	Compressed air	Compressed air High-speed mechanical blower
Abrasive media	Aluminum oxide Garnet Quartz Novaculite Silica Organic materials Glass beads	Metallic shot Metallic grit Aluminum oxide Garnet Quartz Novaculite Silica Organic materials Glass or plastic beads
Typical types of soils removed	Light rust Surface oxides Light mill scale Welding scale	Rust Mold sand Mill and heat-treat scale Welding flux Paint Carbon Weld spatter

17.2.3 Mechanical cleaning. Soil removed by mechanical means, although not usually considered to be a contamination control process, does have its place in the removal of gross soils. Abrasion or erosion is employed as the means of soil removal.

17.2.3.1 Mechanical cleaning methods. Some of the more common mechanical cleaning methods are:

(a) Grinding wheels. Used on portable or staionary grinders to remove heavy weld spatter, flash, and scale. Disc or belt sanders may be used for lighter soils.

(b) Wire brush. Either manual or power brushes are used for removal of weld, scale,and light oxides; not recommended for titanium.

(c) Rasp files. Manual operation for heavy metallic soils.

(d) Steam. Pressurized steam is effective for removal of heavy oils, grease and sludge deposits as well as most shop dirt. Ferrous metals will oxidize rapidly after steam cleaning if they are not protected.

(e) Chipping. Manual chisel and hammer operation for removing excessive weld beads, heavy weld spatter, and scale.

(f) Wiping. Usually limited to dirt, oils, and grease to prevent contamination of subsequent and more precision cleaning processes. Solvent-saturated wiping cloths are generally used.

17.2.4 Washers. Industrial or parts washers are used to remove and dispose of contamination which would be deleterious to further processing or to the end use of the product. The combination of types of washers and cleaning solutions available is nearly unlimited, but some of the standard size types will be described in this section. Because of the variety of cleaning situations encountered, many washing operations are custom-designed to fit process needs.

17.2.4.1 Characteristics of parts washers. Parts washers are characterized by various features, some of which are:

(a) Work piece size or volume capacity.

(b) Type of washing action or agitation.

(c) Number and types of stages.

(d) Degree of automation.

(e) Type of part conveyance.

(f) Type and capacity of filtration system.

17.2.4.2 Parts washers commercial nomenclature. Commercial nomenclature for parts washers includes the following types:

(a) Single Stages.

  (1) Uses immersion, spray or steam as cleaning method.

  (2) Manual or automated operation.

  (3) Usually used for batch or small quantity production.

(b) Two-Stage.

  (1) Usually spray or paddle wheel type.

  (2) One stage may be a rinse or dry operation.

(c) Multistage.

  (1) Conveyorized using rollers, endless wire-mesh belts, flight bars, monorails, or spiral rotating drums.

  (2) Normally includes several washers, plus rinsing and drying.

  (3) High quantity production.

  (4) Many combinations of washing methods and equipment available.

17.2.4.3 Cleaners for washers. Detergent, acids, alkaline, and solvent solutions are available for industrial washing applications. It is sound practice to consult materials, contamination control, and processing specialists, as well as manufacturers representatives, in planning the cleaning agent and type of operation to be employed.

17.2.5 Filtration. Since the purpose of washing is to remove unwanted soils, it is equally important that these soils be removed from the solution to prevent redeposition on the work piece. In the case of spray equipment, accumulated soil will also clog the spray nozzles.

The various filtration methods and the relative merits of each are as follows:

Table XXXIX.

Comparison of washer filtration methods

Type of Removal and Disposal	Method and Effectiveness	Relative Cost Considerations
Solution replacement	Entire solution discharged and replaced; varying degree of cleanliness from very good to poor.	Nominal for light cleaning; generally costly in solution and heat consumption, and loss of production time.
Centrifuging and filtering	Effective for many soils, but requires sizeable installation for high flow rates	High first cost and maintenance.
Edge strainers	Overflow principle; can handle large quantities of top-floating material.	Minimal solution loss; one of least costly methods.

17.2.6 Aggitation. Many factors such as solution chemistry, heat, time, and agitation are important to effective and economical parts cleaning. The subject of agitation is sometimes relegated to minor importance but is, in effect, a most important consideration. Agitation provides the functions of soil removal by physical action, carrying the soil away from the parts, and in some cases, furnishing a fresh supply of solvent to the parts.

(a) Movement of Solution.

(1) Boiling the solution sets up convection currents, but not recommended for all solutions.

(2) Compressed air provides solution movement, but may cause excessive foaming especially with detergent-type solutions.

(3) Paddles provide a continuous, large volume of cleaning solution to the parts at a low pressure; simple yet effectual, especially for fine particles and chips.

(4) Spray widely used and effective for many soils, nozzles must be arranged so that solution hits the work piece from all sides.

(5) Circulating pumps good for immersion cleaning if part configurations are compatible.

(b) Movement of Parts.

(1) Endless wire belts.

(2) Rotating drums or barrels; provides additional tumbling action.

(3) Oscillation or off-center rotation of suspension rods; parts must be racked or in baskets,

(4) Hydraulically activated baskets; basket is an integral part of the washer; surging action provided by lowering and raising basket into and out of solution.

17.3 References.

1. Contamination Control Handbook, Sandia Laboratories, NASA SP-5076, 1969.

2. Product Cleanliness Levels and Contamination Control Program, Mil-Std-1246A, August 18, 1967.

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SECTION 18

METAL CLEANING

18.1 Purpose of metal cleaning. The cleaning process is one of the most important aspects in preparing metals for finishing. Improper cleaning can lead to inadequately conditioned materials for further finishing processes and, consequently, may result in rejects.

18.2 Metal surface preparation. The degree of cleanliness will vary and is relative to the process being performed. In operations such as chromating, phosphating, and electropolishing, the degree of cleanliness is not as stringent as in electroplating. Setting up an equivalent cleaning cycle for phosphating and electroplating would be unreasonable and unnecessarily expensive. In the phosphating procedure, chemical action of the phosphating solution on the surface of the metal partially accomplishes some of the necessary cleaning. In other words, the determining factors for choosing a particular cleaning cycle should be two-fold, cost and function.

18.3 Considerations in determining cleaning process. There are several primary considerations in regard to cleaning procedures for particular metals. Composition, physical properties, chemistry, hardness, porosity, thermal coefficient of expansion, conductivity, melting point, specific heat,and the effect of hydrogen embrittlement are all parameters to take into account. In some instances, hardened steels and other metals like titanium which are possibly embrittled by hydrogen must be treated in a manner that will minimize or eliminate this effect.

18.3.1 Condition of basis metal. The condition of the basis metal is of great importance. It is necessary, for instance, to subject a piece of metal with heat or welding scale to much more processing than non-oxidized cold rolled steel. Also, a different cleaning process is necessary for high carbon steels as opposed to low carbon types.

18.3.2 Method/metal compatability. The method chosen as the cleaning media must be designed to be compatible with the metal. Quite frequently, specific solutions are excellent cleaners; however, unfortunately, they react adversely with some metals causing from slight etching to severe etching which is undesirable. It is, therefore, quite important to select a medium whose effects are controllable. Etching, in certain instances, is desirable if controlled. For instance, the satin or frosty finish on aluminum is an example of controlled etching. This effect is obtained by immersing the aluminum in a medium which both removes the soil and also etches the surface.

When using the process of electrocleaning, maintaining uniform current distribution is required. In electrocleaning large surface areas such as automotive bumpers and large wire goods, racking must be such that uniform current distribution is possible. The melting point of metals is a prime consideration. Metals that possess low melting points (less than 200°F) cannot be cleaned in boiling solutions. Metals with widely separated coefficients of expansion, often found in precision assemblies, can be distorted or bent in hot solution. Heavy metals are usually most efficiently cleaned if immersed for a period of time long enough to heat the metal to a temperature where cleaning is most proficient. Heat will sometimes aid in softening and melting certain soils which otherwise would not be easily removed.

18.4 Soils on metals. Generally speaking, soils can be defined as any surface substance that hinders metal finishing processes. The large spectrum of possible contaminants have been discussed in great detail in Contaminants Defined, Section 5. In a very basic sense, however, the types of soils can be broken down into two broad categories: organic and inorganic.

18.4.1 Organic.

(a) Saponifiable: vegetable and animal oils.

(b) Unsaponifiable: waxes and mineral oils.

(c) Miscellaneous: metal soaps formed in buffing operations, or inhibitors from certain acid pickles redepositing on the metal.

18.4.2 Inorganic.

(a) Polishing Compounds: abrasive, grinding, and polishing residues or grits.

(b) Smut and Scale: metallic and oxide residues.

(c) Miscellaneous: soldering flux and shop dust.

The method and procedure for contamination removal is relative to the composition, condition, physical and chemical properties of the soil as well as these same considerations applied to the metal being cleaned, as discussed previously. Frequently, a cleaning procedure can be recommended based on knowledge of the above listed properties providing there has been no chemical change after application due to aging, drying out, or heat generations.

18.5 Water as a cleaner. Whenever water is used in metal cleaning, the degree of hardness should be ascertained. Water with a hardness level exceeding 25 grains should be treated before using. Many cleaning solutions, wetting agents, or surface active agents are deactivated by the formation of insoluble calcium and magnesium products found in water. Consequently, purification treatment, for example, deionization, should be performed prior to cleaning. (See Section 19, Paragraph 19.4.1.5.)

18.6 Disposal requirements. Society has become extremely aware of pollution problems and has reached a point where industrial pollution is not going to be tolerated. Thus, it appears that pollution abatement programs will be mandatory.

Current efforts on the part of local through federal governmental agencies concerning water pollution problems have become a prime concern to metal finishing industries. Some industrial plants have already established programs to handle their wastes. In order to set up a workable disposal program the first step should be determining the various types of wastes discharged by the plant. Next, the plant should obtain a copy of the pollution regulations for the specific area from the Water Resources Commission of the State Government. Finally, a system should be engineered to segregate wastes into categories so that adequate treatment can be put into effect prior to effluent discharge from the plant.

18.7 Metal precleaning. Generally, the cleaning process is done in two phases (1) precleaning (2) final cleaning. Precleaning is necessary to assure that the final cleaning solutions are kept as clean as possible.

18.7.1 Factors to consider in choosing a cleaning medium. There are several factors to consider in choosing a cleaning medium. A few of these are:

(a) The metal to be cleaned.

(b) Level of cleanliness required.

(c) The type of soil to be removed.

Mechanical action such as scrubbing or agitation is usually desirable in cleaning methods in order to increase the efficiency of soil removal. The various mechanical cleaning methods are discussed in greater detail in Section 17, Gross Cleaning.

18.7.2 Precleaners. Precleaners may be categorized as follows:

(a) Acid.

(b) Alkaline-built detergent.

(c) Detergent.

(d) Diphase.

(e) Emulsifiable solvent.

(f) Emulsion.

(g) Solvent.

18.7.3 Final cleaners. Basically, final cleaners are electrocleaners, using either reverse, direct, or periodic reverse current.

18.8 Precleaning categories defined.

18.8.1 Acid cleaning. Acids are usually used in the removal of oil and metal oxides or to create a phosphoric coating (when using phosphoric acids) which may be used as a paint base. The composition of acid cleaners is usually either inorganic or organic acids, such as phosphoric or fluconic, along with water-miscible solvents and organic wetting and emulsifying agents. The contaminants are removed by the following means:

(a) Emulsifying.

(b) Etching.

(c) Dissolving.

(d) Wetting.

18.8.1.1 Means of acid application. The usual means of application are:

(a) Soaking

(b) Spraying

(c) Brushing

(d) Hand wiping

18.8.2 Alkaline-built detergent. The heavy duty alkaline cleaners are the most frequently used type of cleaner. The composition of alkaline cleaners consists of highly alkaline salts, such as sodium hydroxide, silicates, and carbonates, along with sequestering agents, dispersants, and various surface active agents. In cleaning with alkaline detergents, the concentration used is usually from 1/2 to 2 lb/gal. The temperature of the solution is most effective in ranges from 140° -200°F. The usual means of application are by soaking, spraying, or electrosoaking.

These cleaners are generally used in the removal of light scale, oils and smuts. They are not very effective buffing compound removers, consequently, are infrequently used for this purpose. Frequently, alkaline cleaners are used as a secondary cleaner after detergent soaks to aid in removing any residue soil or detergent film.

18.8.3 Detergents. Detergents are very frequently used in metal finishing industries, especially those industries that process buffed metals. This type of cleaner is composed of wetting agents and or soaps, sequestering agents, dispersants, buffering salts, and inhibitors. The ability of detergents to clean is a function of several factors:

(a) Wetting.

(b) Emulsifying.

(c) Dispersing.

(d) Solubilizing the soil.

Cleaning solutions containing detergents are rarely used at room temperature but most commonly at temperatures ranging from 150°F to boiling. Solution concentrations are usually maintained at from 4 to 10% by volume or 6 to 12 oz/gal.

18.8.4 Diphase cleaning. This type of cleaning incorporates two phases. One phase usually consists of water plus water soluble wetting agents and sometimes also includes inorganic salts and emulsified oils. Phase two usually consists of some suitable organic solvent.

The functioning ability of diphase cleaning is attributed to combining solvent and emulsifying properties of both phases. Two methods of application are used in diphase cleaning (1) soaking (2) spraying Of these two methods, the most efficient is spray application.

18.8.5 Emulsifiable solvents. These solvents are usually composed of petroleum derived solvents (primarily chlorinates) and surfactants (surface active agents) to render them emulsifiable. Surfactants are of major importance because they determine the type and stability of the emulsion.

Emulsifiable solvents are most often used as soak solvents followed by a spray or immersion rinse. Emulsification occurs in the rinse. The majority of the soil and solvent is removed by the water rinse which reduces the amount of soil needed to be removed in further cleaning processes.

18.8.6 Emulsion cleaning. Emulsion cleaning methods incorporate a two phase medium with one phase dispersed in the other. The two phases consist of (1) water (2) an organic solvent. Appropriate emulsifiers are used to form a water-in-oil (W/O) emulsion or an oil-in-water (O/W) emulsion. An alkaline medium is most frequently used, possessing a pH of about 7.8 to 10.0. The temperature range for this medium is 140° to 180°F. The means by which emulsion cleaners operate is by dissolving and or emulsifying the soil. The cleaning action of emulsions combines the advantages of both the water and oil phases.

This particular cleaning method is very frequently employed in precleaning buffed metals, particularly zinc-based diecastings, copper, and brass. All traces of the organic solvents and other by-products must be removed to avoid contamination of the electroplating processes; therefore, if emulsion cleaning is used in electroplating cycles it will be necessary to follow the process by alkaline spray and soak cleaning.

18.8.7 Solvent cleaning. Solvent cleaning is accomplished by spraying or soaking the part in a petroleum or chlorinated solvent. The solvent cleans by dissolving all or a part of the soil. Occasionally, the dissolution of the soluble components of a soil, such as buffing compound containing an abrasive, can leave inert residues which are very difficult to remove. Fast drying solvents tend to aggravate this problem; so for this reason, slow drying solvents are sometimes used to soften and condition the soil for removal in subsequent alkaline cleaning.

In vapor degreasing units, chlorinated solvents are usually used due to the high density of their vapors. Generally, the solvent is used hot, and cleaning is accomplished by soaking, spraying or condensation of the vapor on the part. The procedure frequently applied is, first, the equipment is soaked to wet and dissolve most of the soil, second, the part is flushed with a clean redistilled solvent.

An important consideration in solvent cleaning is the inhibition of chlorinated solvents against hydrolysis. Inhibition of hydrolysis prevents the formation of free hydrochloric acid which may occur in the presence of water and active metals such as aluminum. Any acidity in the solvent must be guarded against because it can cause etching of the part being cleaned.

18.9 Final cleaners. Although final cleaning is usually preceded by precleaning, in many instances final cleaning alone will suffice. Final cleaners are usually heavy duty alkalines (previously mentioned) which are employed with the aid of current, either direct reverse, or periodic reverse.

Final cleaning has two objectives, it enables (1) metal activation (2) complete soil removal. Metal activation is generally accomplished by using reverse current electrocleaning (the part made anodic). Soil removal is assisted by the gas scrubbing of the oxygen generated. The reverse current aids in the removal of soil and disallows the deposition of any metallic film or non-adherent metallic particles.

A fairly standard procedure after final cleaning is subjecting the part to a dilute mineral acid dip to neutralize the alkaline film on the metal surface.

Double cleaning cycles are sometimes required on parts containing heat treat, welding, or other oxides. In such circumstances, the part is usually anodically electrocleaned, pickled to remove oxide, final electrocleaned, and finally acid dipped to neutralize the alkaline. The purpose of the final electrocleaning is to remove any smut developed from the scale removal in the first pickle. The purpose of the first electrocleaning is to enhance the effectiveness of the scale removal properties of the first pickle.

18.9.1 Anodic electrocleaning. Cleaning by anodic method is a desirable means of final cleaning, primarily because of the fact that the metal being cleaned is actually dissolved as well as cleaned. The actual dissolution of the surface metal allows for the removal of metallic smut and disallows redeposition of non-adherent metallic films. Surface oxygen generation creates a scrubbing action that is helpful in soil removal. Anodic cleaning negates the possibility of hydrogen embrittlement.

18.9.1.1 The means by which anodic electrocleaning takes place. Part being cleaned is made anodic (positive) in an alkaline electrocleaner using low voltage (3 to 12 v) dc current. Current densities vary from 10 to 150 amp ft^2 (asf) depending on time. The current density depends on the type of metal and the amount of soil. Time required for cleaning is generally 1/2 to 2 minutes. There are several factors to consider in anodic cleaning. It is of utmost concern to maintain control of current density, temperature, and concentration in order to avoid etching and tarnishing. Prolonged reverse current cleaning, high current densities, high temperatures, low concentrations can cause dezincification and overetching, particularly on brass and zinc base die castings.

The reverse current alkaline cleaning method is not recommended for aluminum, chromium, tin, lead, or other metals which are soluble in alkaline electrocleaners.

18.9.2 Cathodic cleaning. Consult equipment manufacturer for ventilation requirements and control of hydrogen explosion hazard. Cleaning by the cathodic method (negative) requires the same equipment, voltage, and current densities as described in the anodic method (previously described). In using the cathodic method, hydrogen is liberated on the surface of the part (rather than oxygen as in the anodic method). The volume of hydrogen liberated at the cathode is twice that of the oxygen liberated at the anode for a given current density. Because of this the gas scrubbing cleaning effect is more efficient in cathodic cleaning; consequently, cathodic cleaning is sometimes used as a precleaner followed by anodic cleaning. (The mechanism by which soil is removed is described in previously discussed alkaline precleaning.) Soil removal is further assisted by gas scrubbing.

In cathodic cleaning the part is actually plated in a direct current cleaner. Any positively charged material is attracted to and may be reduced and deposited on the surface. Metallic film that becomes deposited is usually non-adherent but difficult to detect and remove. These films can cause poor adhesion, roughness, and staining of electroplated metals.

Parts that are susceptible to hydrogen embrittlement (such as spring steel) should not be cathodically cleaned unless the hydrogen is removed after processing. If the part is subjected to heat treatment for one hour at 400°F immediately after cathodic cleaning, the embrittling effect will be eliminated. Parts with hardness exceeding 40 Rockwell C can be embrittled and should undergo baking after plating.

Chromium contamination of cleaners is sometimes unavoidable due to the use of the same rack for chromium as well as other plating. The direct current cleaning method is more susceptible to staining from chromium-contaminated cleaners than reverse current cleaning.

The most common applications for direct current cleaning are in cleaning metals such as chromium, tin, lead, brass, magnesium, and aluminum, which are dissolved or etched by anodic cleaning.

This method is also applicable to cleaning buffed nickel prior to chromium plate. The anodic cleaning process is not advisable for nickel because it would produce a passive film, due to oxidation which would prevent the deposition of bright chromium.

18.9.3 Periodic reversal cleaning (PR). The PR electrolytic cleaning is used usually to remove smut, oxide, and scale from ferrous metals. Alkaline compounded materials possessing sequestering or chelating agents are usually used. The PR system works by making the part alternatingly cathodic and anodic using dc current at 6 to 15 v. The part may be cleaned either on a rack or in a barrel. Cleaning removes soil by combining the mechanism of alkaline cleaning, the use of reducing and oxidizing conditions, and strong metal chelation.

PR cleaning is advantageous because it eliminates the acid on certain types of parts (hinges) where entrapment of acid aggravates bleed-out after alkaline plating (brass, copper, zinc, cadmium, tin). This method also allows for the removal of oxides without the danger of etching or the development of smut usually encountered from acid pickling.

18.10 Phosphate-free cleaners. As has been previously mentioned, society has become intensely aware of pollution in the environment. Because of this concern it became necessary to establish elements of danger to the environment. It was discovered that cleaners containing phosphates were detrimental. Although they were functional as water softeners, rinsing aids, soil suspending agents, and detergency boosters, they also were instrumental in causing accelerated algae growth in waters where they were discharged. This abnormal increase in algae growth caused a biological imbalance creating water pollution problems; hence, extensive research was initiated to find a phosphate free cleaner. There are now a number of phosphate free cleaners that have proven to be very effective.

18.11 Acid dipping. Acid dipping is accomplished by immersing a metal in an acid solution. The process functions as a means of removing oxide, scale, and other acid soluble soil, and also to activate the metal surface. The acids usually used are mineral acids; however, mixtures of acid salts are used to increase both scale removal and activation.

There are two categories of acid dipping: pickling and activation. The primary purpose of pickling is to remove scale and oxide; whereas, activation acid dipping is used to neutralize the alkaline film and to dissolve any light oxide coating created by the final cleaning.

The acid that is chosen in the acid dipping method should be compatible with the metal. In other words, the results obtained should be predictable because of control. Another determining factor in acid choice is the water solubility of the metal salt formed from the reaction between the metal and the acid. As an example, sulfuric acid would not be suitable for preparing lead or leaded brass for electroplating. It is not suitable because of the formation of insoluble lead sulfate which would form a film between the basis metal and the electrodeposited metal causing poor adhesion.

DC current may be used to aid activation, increase scale removal, decrease surface roughness. The part is made cathodic for activation and scale removal purposes. The hydrogen emission at the surface of the part serves as a gas scrubber and aids in scale removal. The decreasing (leveling) of surface roughness eliminates passivity. Application of reverse current is used to remove protruding metallic slivers and smooth surfaces.

Inhibitors are not recommended in activation acid dipping because of the possibility of absorbed films which may interfere with later procedures, such as plating. Absorbed films can cause poor adhesion or cloudy plate. Inhibitors may be used in pickling. The function of the inhibitor is to reduce the severity of attack on areas that have had scale removed and to reduce hydrogen embrittlement effects.

18.12 Quality control in metal cleaning. The result of inadequate cleaning is usually part failure or rejection of part. Rejects are sometimes easily determined; however, in some instances, improper cleaning is discernible only after product failure. For instance, the film that can interfere with adhesion corrosion resistance or electrical contact is visibly imperceptible. Causes for rejects in various processes are as follows:

18.12.1 Electroplated coatings.

(a) Bare spots.

(b) Etching.

(c) Hydrogen embrittlement.

(d) Irregular plate.

(e) Pitting.

(f) Poor adhesion.

(g) Poor corrosion resistance.

(h) Roughness.

(i) Staining.

18.12.2 Electropolishing and other electrolytic procedures.

(a) Bare spots.

(b) Etching.

(c) Pitting.

18.12.3 Chemical treatments as in conversion coatings.

(a) Bare spots.

(b) Etching.

(c) Excessive graininess.

(d) Irregular color.

(e) Powdery coatings.

(f) Non adherent coatings.

18.13 Determining cause of part rejection. In the advent of a part rejection, a careful examination should be made to determine the cause of the problem. If it is determined that the problem was caused by the cleaning process, a systematic check of all control factors will usually determine the point of malfunction. When a quality control program is in practice, back tracking is expedited saving costly time.

18.13.1 Most frequent sources of error.

(a) Incorrect concentration, temperature, cleaning time.

(b) Change in the nature of the soil. Frequently, in cleaning, an unknown soil is created during a cleaning process which necessitates a change in cleaning medium and cycle.

(c) Too lengthy transfer time. Lengthy transfer times can cause dry on of cleaners. Dry on of cleaner can cause streaking and staining in final plating. Cold water rinse is not efficient in removal of dry on.

(d) Unacceptable cleaner condition. Cleaner condition can be altered by water hardness and excess soil in cleaner.

(e) Alteration in basis metal or basis metal processing.

(f) Blistering and peeling caused by mist or spray of chromic acid from chromium plating tanks settling on part, or from chromic acid seeping out of breaks in the rack coating.

18.13.2 Errors in soak cleaning.

(a) Floating oil layer in cleaning solution.

(b) Faulty agitation system.

(c) If cleaning zinc-based die castings, check if the conditions for galvanic cell exist.

18.13.3 Errors in spray cleaning.

(a) Equipment failure. Temperature gages, pump pressure, nozzles must be working properly.

(b) Excessive foaming. Excessive foaming can cause loss of solution by overflow and can also decrease the volume in the pump which could result in inadequate volume of solution.

18.13.4 Errors in emulsion cleaning.

(a) Equipment failure.

(b) Extreme emulsion soil load.

(c) Incorrect pH level.

(d) Inadequate or faulty agitation system.

18.13.5 Errors in electrocleaning.

(a) Incorrect polarity.

(b) Low concentration or temperature.

(c) Excessive voltage.

(d) Excessive current density.

(e) Improper usage of cleaning compounds or improper choice of compound.

(f) Low current density. May be caused by polarized cleaning electrodes. Electrodes should be removed and cleaned on a systematic basis.

18.13.6 Errors in rinsing.

(a) Contaminated rinses.

(b) Clogged rinse line hindering flow.

(c) pH value not within acceptable limits. High pH after cleaning and low pH after acid dipping usually indicate inadequate water flow.

(d) Inadequate number of rinses.

(e) Inadequate water pressure.

(f) Incorrect temperature to insure desired results. Water temperatures from 35°- 50°F can set up emulsified or saponified soils on the surface of a part after alkaline cleaning. Warm rinse (no more than 100°to 110°F) should be used after alkaline cleaning to prevent dry on of cleaner during transfer.

(g) Air used for agitation contaminated by oil.

18.13.7 Errors in acid dipping.

(a) Oil slicks present in acid tanks.

(b) Inadequately circulated acid solution.

(c) Acid concentration too low.

(d) Temperature too low.

(e) When using cathodic current, make certain that insoluble anodes are used. Examine the part for the presence of smut and water breaking.

(f) Care should be taken in processing two or more different alloys at the same time since in some cases potential differences can result in an alloying constituent of one plating the other. For instance, high copper aluminum may result in a copper film on low copper aluminums.

18.13.8 Insuring cleaning solution effectiveness. Obtaining maximum cleaning efficiency and economy in regards to cleaning solutions is dependent on good control. Control measures should be established and followed. Some of the major considerations are as follows:

(a) Chemical considerations.

  (1) Concentration.

  (2) Free alkalinity.

  (3) Total alkalinity.

  (4) pH.

  (5) Surface tension.

(b) Temperature.

(c) Voltage.

(d) Current density.

(e) Time.

(f) Dumping schedule.

The above factors are easily determined and maintained. Simple tests can be used to maintain chemical control. Equipment can be set up to maintain the other factors. Periodic checks of equipment will insure working order.

By determining total alkalinity, alkaline cleanness can be controlled. More sophisticated controls can be set up to include pH, surface tension and free and total alkalinity. In some cases soil load determinations may be made. In most cases, however, the determination requires lengthy analytical procedures which are difficult to justify relative to cleaning efficiency.

Emulsion cleaners (acid and alkaline) are usually controlled by pH and concentration.

Determining the proper time to dispose of a cleaner is a difficult task. The cleaner must be disposed of before it becomes contaminated enough to recontaminate parts being cleaned. The best method for this determination is practical experience because the rate at which the cleaning solution becomes contaminated is dependent on the number and severity of contamination of the parts being cleaned.

Frequently, in metal cleaning, cleaners build-up metallic impurities, such as chromium, copper, and zinc. When this build-up is determined, it is recommended that the cleaners be replaced.

Chromium is deleterious in the hexavalent state, particularly when the work is made cathodic. Reducing agents may be used to keep chromium in the reduced (trivalent) state to prolong the useful life of the cleaner.

An established quality control program is essential to maintaining production on a constant level. For a workable quality assurance program, refer to Section 21, Quality Control.

18.14 Methods and procedures for cleaning.

18.14.1 Electropolishing of metals. Basic criteria for the control of electropolish processing is as follows:

NOTE: The formula for electropolishing fluid is proprietary, there will be no specific information given. The fluid used herein is basically a phosphoric acid/cromic acid mixture and contains no other active ingredients.

18.14.1.1 Basic electropolishing finishes. The two basic types of electropolishing accomplished are as follows:

(a) Bright finish. The surface of the material processed has improved reflectivity and a decrease of RMS index.

(b) Matte finish. The surface processed has a uniform satin appearance.

18.14.1.2 Surface removal. Unless otherwise specified by the operational procedure, both finishes will effect a uniform material removal of .001 inch minimum to .0005 inch maximum.

Uniform removals of up to .010 may be effected when material removal is the primary objective for processing.

18.14.1.3 Basic controls. Although there are many variables in electropolishing that must be considered on each specific material type and finish objective, the basic controls shall be as specified in Table XXXX and Table XXXXI.

Table XXXX.

Electropolish - bright finish, material removal .0001 - .0005"

⑰

Material Type	Solution	DC Voltage	F° Temperature	Time
1. 300 series CRES	Phosphoric acid/cromic acid solution (Micron 11)* or equivalent	5-20	140/160	30/45 sec.
2. 400 series CRES		5-20	190/215	30/45 sec.
3. Inconel/Monel		5-20	150/170	60/90 sec.
4. Brass/Copper		5-20	130/150	30/40 sec.
5. Aluminum (Low Silicon)		5-20	130/150	40/60 sec.

*Technical Micronics Control, Inc., Madison, Alabama, proprietary solution.

Table XXXXI.

Electropolish - matte finish, material removal .0001 - .0005"

Material Type	Solution	DC Voltage	F° Temperature	Time
1. 300 series CRES	Phosphoric acid/cromic acid solution (Micron 11)* or equivalent	5-12	70/90	30/45 sec.
2. 400 series CRES		5-12	70/90	30/45 sec.
3. Inconel/Monel		5-12	70/90	60/90 sec.
4. Brass/Copper		5-12	70/90	30/40 sec.
5. Aluminum Alloy		5-12	70/90	40/60 sec.

*Technical Micronics Control, Inc., Madison, Alabama, proprietary solution.

18.14.1.4 Electropolishing precleaning requirements. Material to be electropolished shall be processed as follows:

(a) Vapor degrease using trichloroethylene or suitable solvent.

(b) Air Dry.

(c) If contaminants are still evident clean part in a solution of detergent and water heated to 150-170°F.

(d) Water rinse.

(e) Air Dry.

18.14.1.5 Electropolishing process.

(a) Suspend all work parts to be processed on polishing racks. Assure good conductive contact between racks and work parts by visual inspection.

(b) Completely immerse parts in the electropolishing bath preheated to the requirements of Table XXXX or Table XXXXI.

(c) Set timer for the desired time lapse and depress "start" button.

(d) When the timer reaches zero, remove parts and clean as follows:

  (1) Rinse with tap water until all visual evidence of electropolishing solution is removed.

  (2) If entrapments are evident, rinse part in a mild alkaline solution. Caution: Alkaline solution rinse can have detrimental effects on aluminum. Rinse with care.

  (3) Rinse in deionized water until pH of part is 6.0 to 8.0

  (4) Air Dry.

(e) Visually inspect all parts after electropolishing to assure uniform finish.

18.14.2 Chemical cleaning. The term chemical cleaning shall indicate pickling, passivating, descaling, deoxidizing, and other cleaning processes where surface conversion or preparation is the prime objective. These operations are usually accomplished by immerse, spray, or pump-through and the controls are as prescribed by the detailed procedure.

Parts shall be held mechanically in baskets, racks, or slings as necessary to assure full contact for the time specified by the cleaning chemical. If the configuration is such that it prevents full contact in one position, frequent movement may be necessary to assure complete cleaning.

Continue cleaning for the time and at the temperature specified by the detailed procedure. Assure, by visual examination, that cleaning was effective prior to next operation.

18.14.2.1 Cleaning procedure for stainless steel, austenitic, AISI, 300 series.

Item	Operation	Solution	Temp. °F	Time Min.	Remarks
1	Degrease	Trichloroethylene	Ambient	As Req.	Vapor or chemical degreasing may be substituted.
2	*Pickle	20±5%:1.5±.5% Nitric: Hydrofluoric	Ambient	8/15	Time may be increased as required to remove scale.
3	Rinse	Water	Ambient	As Req.	to pH of 6.0/8.0
4	Passivate	20±5%:3±1%: water Nitric: Sodium dichromate: water	Ambient	15/30	Time may be increased as required.
5	Rinse	Water	Ambient	As Req.	To pH of 6.0/8.0
6	Dry	GN_2	Ambient	As Req.	Must be visually dry

*This step shall be used only when scale or corrosion is present and provided the part has no faying surfaces or highly polished close tolerance machined surfaces. Parts that fall into this category shall be referred to the customer for disposition.

Note. This procedure is not applicable to convoluted stainless steel lined hoses. Some hoses are manufactured by helical winding and welding or brazing. Since this is not immediately apparent all convoluted hoses shall be processed as follows unless otherwise specified.

18.14.2.2 Cleaning procedure for convoluted stainless steel hoses.

Item	Operation	Solution	Temp. °F	Time Min.	Remarks
1	Degrease	Trichloroethylene or Trisodium phosphate 10±2%	Ambient	As Req.	Spray, immerse, pump through
2	Clean	25±5%: Nonionic detergent phosphoric: Nonionic detergent	150±15°F	15/20	Spray/Pump through
3	Rinse	Water	Ambient	As Req.	to pH of 6.0 to 8.0
4	Dry	GN_2	Ambient	As Req.	Visually dry

Hoses one inch or smaller in diameter may be cleaned in any position that will facilitate exercise. Manual exercise during the pump through procedure shall include all sections of the hose being flexed to its designed bend radius on all axes, working from one end of the hose to the other for three complete cycles minimum.

Hoses over one inch in diameter shall be cleaned in the vertical position. Exercise the hose.

When hoses (over 1 inch diameter) are cleaned vertically to a final clean condition in a non-controlled environment, the following handling instructions are mandatory:

(a) After hose is dry and while a light purge is continuing through the assembly, cover all openings with a polyethylene tent. The tent shall be continuously purged with GN_2.

(b) While tent purge continues disconnect GN_2 from hose and package ends. (See Section 20, Clean Packaging.)

18.14.2.3 Cleaning procedure for stainless steel, martenistic, AISI, 400 series.

Item	Operation	Solution	Temp. °F	Time Min.	Remarks
1	Degrease	Trichloroethylene or Trisodium phosphate 10±2%	Ambient	As Req.	Vapor, Spray, Immerse
2	*Pickle	20±2%: water Hydrochloric acid: water	Ambient	20 max.	
3	Rinse	Water	Ambient	As Req.	To pH of 6.0/8.0
4	Passivate	20±2%:3±1%: water Nitric: Sodium dichromate: water	Ambient	10/15	Do not allow temperature to exceed 90°
5	Rinse	Water	Ambient	As Req.	To pH of 6.0/8.0
6	Dry	GN_2	Ambient	As Req.	Visually dry

*This step shall be used only when scale or corrosion is present and provided the part has no faying surfaces or highly polished close tolerance machines surfaces. Parts in this category shall be referred to the customer for disposition.

18.14.2.4 Cleaning procedure for aluminum, bare, and all alloys.

Item	Operation	Solution	Temp.°F	Time Min.	Remarks
1	Degrease	Trichloroethylene	Ambient	As Req.	Solvent only
2	Acid Clean/ Passivate	30±5% Nitric	Ambient	8/12	This is optional and should be used for oxidation removal
3	Rinse	Water	Ambient	As Req.	Required when step 2 is used to pH of 6.0/8.0
4	Clean	Nonionic detergent	Ambient 140/160°F	As Req. 10/30	Mild brushing as required. This step not used when step 2 is applicable.
5	Rinse	Water	Ambient	As Req.	To pH of 6.0/8.0
6	Dry	GN_2	Ambient		Visually dry

18.14.2.5 Cleaning procedure for aluminum, conversion coated.

Item	Operation	Solution	Temp.°F	Time Min.	Remarks
1	Degrease	Trichloroethylene	Ambient	As Req.	Solvent only
2	Clean	Nonionic detergent: water	Ambient	As Req.	Mild brushing with soft brush as required
3	Rinse	Water	Ambient	As Req.	To pH of 6.0/8.0

Breaks in conversion coating shall be reported immediately to the customer for disposition.

18.14.2.6 Cleaning procedure for carbon and mild steel.

Item	Operation	Solution	Temp.°F	Time Min.	Remarks
1	Degrease	Trichloroethylene or Trisodium phosphate 10±2%	Ambient	As Req.	Scrub or brush as required.
2	*Pickle/	#1 20±2%: water hydrochloric: water #2 5±2%:water Citric: water #3 25±5%: phosphoric	Ambient 140 ± 10 150 ± 15	10/20 15/30 10/20	Immerse spray or pump. Immerse spray or pump. Immerse spray or pump.
3	Rinse	Water	Ambient	As Req.	To pH of 6.0/8.0
4	Dry	GN_2	Ambient	As Req.	Visually dry.

Note: Step 2 - The pickle treatment selected shall be determined as follows:

(a) #1 HCL/water - heavy scale, weld slag, and deep corrosion. When pickling with HCL, rinse and dry immediately to eliminate rust.

(b) #2 Citric/water - rust and iron ozides and light scale.

(c) #3 Phosphoric/water - light scale, rust or oxides, and as a surface treatment for parts to be painted.

18.14.2.7 Cleaning procedure for copper alloys.

Item	Operation	Solution	Temp. °F	Time Min.	Remarks
1	Degrease	Trichloroethylene	Ambient	As Req.	Solvent only.
2*	Clean	10±5%:water P-107: water	140 ± 10	15/20	Immerse spray or pump.
3	Rinse	Water	Ambient	As Req.	To pH of 6.0/8.0
4*	Clean	Nonionic detergent: water	Ambient	As Req.	Scrub or brush as required.
5	Rinse	Water	Ambient	As Req.	To pH of 6.0/8.0
6	Dry	GN_2	Ambient	As Req.	Visually dry.

Note* Steps four and five are not required if optional steps two and three are used.

18.15 References.

1. General Solution Handling, Cleaning, and Packaging Procedures, Technical Micronics Control Document B, Specification 6201, Technical Micronics Control, Madison, Alabama.

2. Metal Cleaning, John P. Homrack and William P. Innes, MacDermid, Inc., Waterbury, Conn., Metal Finishing Guidebook for 1970, Metals and Plastics Publications, Inc., Westwood, N.J.

SECTION 19

PRECISION CLEANING

19.1 Mil-Std-1246A definition. According to Mil-Std-1246A precision cleaning is a method performed to achieve a level of product cleanliness to a greater degree than is normally detected by visual means. Articles shall be visibly clean prior to precision cleaning. Precision cleaning shall be performed in a controlled environment. A controlled environment facility has the primary objective of positively controlling one or more physical, chemical, or biological variables within a specified area. The variables include but are not limited to:

(a) Personnel.

(b) Humidity.

(c) Chips, fibers, (particulate matter).

(d) Bacteria, fungi, (biological forms).

(e) Certain gases and vapors (chemical forms).

(f) Radiation.

(g) Temperature.

(h) Pressure.

(i) Air velocity.

19.1.1 Controlled environment facilities. Controlled facilities have controlled atmospheres for the protection of products, personnel, processes, experiments, equipment, or any combination thereof. Examples are:

(a) Industrial clean rooms.

(b) Special manufacturing areas.

(c) Photographic processing laboratories.

(d) Pharmaceutical laboratories.

(e) Hospital operating rooms.

(f) Germfree isolation chambers.

(g) Computer rooms.

(h) Cleanwork stations.

(i) Sterile cabinets.

(j) Metabolism chambers.

19.1.2 Classification of controlled facilities. The variables controlled and the degree of control determine the classification of the facility. The facility may be an entire building, room, or individual controlled work station. (See Section 8, Clean rooms and clean work stations.)

19.2 Precision cleaning methods. Precision cleaning methods must be capable of removing contaminants that exist but are not necessarily visible. The size of particles that are removed in precision cleaning processes are in the micron size range. The smallest particle visible by the human eye under good conditions is approximately 50 microns. (One micron is 40 millionths of an inch.)

As a need for cleanliness increases so does the need for extremely pure solvents, closely controlled techniques, clean environments, and more sensitive test methods.

There are a number of cleaning techniques used in precision cleaning. Some of the most commonly used techniques are:

(a) Immersion cleaning.

(b) Detergent cleaning.

(c) Emulsion cleaning.

(d) Etching and acid cleaning.

(e) Alkali cleaning.

(f) Ultrasonic cleaning.

(g) Electrolytic cleaning.

(h) Spray cleaning.

(i) Vapor degreasing.

(j) Flush cleaning.

Each of these cleaning processes is described in the following paragraphs.

19.2.1 Immersion cleaning. This is perhaps the simplest of all cleaning techniques. The object to be cleaned is soaked in a tank filled with a cleaning solution. The cleaning process is accomplished by the ability of the solution to weaken the adhesive bonds of contaminants.

This technique is simple and inexpensive. It is, however, not particularly efficient in removing contaminants that are strongly attached to a part. Agitation is sometimes an aid in increasing the effectiveness of immersion cleaning.

19.2.2 Detergent cleaning. Detergents are very frequently used to increase the cleaning power of water. The ability of detergents to clean is a function of several factors:

(a) Wetting. The detergent allows the water to wet (come in closer contact with the surface).

(b) Penetrating. The detergent reduces interfacial tension between surfaces and fluids and allows penetration by the cleaning solution.

(c) Breaking adhesive bonds. The adhesive bonds are broken either by displacement with detergent molecules at the interface of the surface contaminant or by the contaminant becoming dissolved in the detergent.

(d) Dispersing or emulsifying. After the contaminants have been broken loose from the surface, redeposition of the contaminant is prevented by the detergents ability to disperse particles and emulsify liquids.

(e) Rinsing. Detergents can be readily rinsed off a surface. Once particles have become dispersed or liquids emulsified they have no affinity to the surface being cleaned and consequently can be easily rinsed.

19.2.2.1 The effect of temperature on detergents. Cleaning solutions containing detergents are rarely used at room temperature but most commonly at temperatures ranging from 150°F to boiling. Solution concentrations are usually maintained at from 4 to 10% by volume or 6 to 12 oz/gal.

19.2.2.2 Types of detergents. Detergent formulations are complex. There are several detergents on the market for specific applications.

Detergents can be divided into three basic types: anionic, cationic, and nonionic. Anionic detergents form negative ions when dissolved in water; cationic detergents form positive ions when dissolved in water; nonionic detergents do not ionize in water even though they may be soluble. The charge characteristics of a given contaminant may influence the selection of the detergent.

19.2.2.3 Considerations in selecting detergents. In selecting a particular detergent there are a number of considerations. Table XXXXII lists factors that should be considered.

Table XXXXII.

Factors in selecting detergents.

Water solubility	Ability to foam
Oil solubility	Ability to be rinsed
Temperature limits	Surface tension and interfaced tension reducing power
Resistance to acids, bases, and salts	Wetting power

19.2.3 Emulsion cleaning. Emulsion cleaning is a type of immersion or dip cleaning. The cleaning solution in this case is an emulsion of water and solvent. Generally, emulsion cleaning is a precleaning process, however, it is sometimes used in precision cleaning.

19.2.3.1 Categories of emulsion cleaners. Emulsion cleaners fall into three major categories: stable emulsions, unstable emulsions, and diphase emulsions. Stable emulsions contain enough surface active agents to render the emulsion permanently stable. Unstable emulsions must be constantly agitated in order to keep the phases from separating. Diphase emulsions are really not characteristic of true emulsions. The oil phase is separated from the water phase and reamins separated floating on the water.

19.2.3.2 Efficiency of emulsion cleaners. Emulsion cleaners are not highly efficient in removing soluble particulates. The cleaning process, when using emulsion cleaners, is quick; however, there is a tendency to transfer contaminants from part to part. A unique feature of these cleaners is that they leave a residual oil film which acts as a protective coat. Emulsion cleaning will protect ferrous parts against rust for, in some cases, up to three weeks. Table XXXXIII, Comparison Chart for Emulsion Cleaners, lists the characteristics, limitations, and uses of the various emulsion cleaners.

Table XXXXIII.

Comparison chart for emulsion cleaners

Type and Uses	Characteristics	Limitations
Stable emulsion Immersion cleaning Spray cleaning	Least expensive Used at high temperatures (170 - 180°F)	Low efficiency for insoluble particulates Will form precipitators
Unstable emulsion Immersion cleaning Spray cleaning (adequate agitation necessary)	More effective than stable emulsions Better penetration of oil phase Used at low temperatures 145 - 155°F	Temperature critical – do not exceed 160°F Reacts with alkali to form soap, can cause foam (Problem when used in spray cleaning)
Diphase cleaners Immersion cleaning Spray cleaning (if emulsifiers are added)	Highest efficiency of emulsion cleaners Will tolerate temperatures up to 180°F	Requires expensive equipment

19.2.3.3 Equipment used in emulsion cleaning. Equipment necessary in emulsion cleaning is comparatively simple; heated immersion tanks or hot sprays in spray booths. Thorough ventilation is essential because of the hydrocarbon vapors. Hot and cold water rinses are necessary. The first rinse is usually cold, removing gross contaminants. The second rinse is usually hot. Parts are usually dipped by use of racks. In the case of small parts, open mesh baskets are used. Agitation is useful for effective cleaning in immersion cleaning operations.

19.2.4 Etching and acid cleaning. Quite frequently, contaminants are deeply embedded into the surface of a part. In this case, usual cleaning operations are ineffective. In order to remove the contaminants, the cleaning operation must be capable of removing a layer of the surface, thereby removing the contaminants imbedded. In metal cleaning when a thin layer of surface is removed, the acid etching is called pickling. Slags, mill scales, metal oxides are removed by deep etching.

Table XXXXIV.

Acids used in etching

Nitric acid	Oxalic acid
Hydrocholoric acid	Tartaric acid
Citric acid	Ammonium bisulfate
Acetic acid	Ammonium persulfate

19.2.4.1 Wetting agents. Wetting agents are sometimes added to the acid bath. The addition of wetting agents enables thorough contact of acid to surface. Wetting agents also reduce the possibility of bubbles adhering to surface of parts thus causing poor etching.

19.2.4.2 Temperature. Warm temperatures, 140 - 190°F, are generally used in acid baths. The usual method of application is by immersion; however, spray cleaning is sometimes used and occasionally a wipe-on and wipe-off method.

19.2.4.3 Limitations. Acid cleaning has some limitations. First, it is ineffective in removing oils or greases, in fact, if oils or greases are present acid baths will not be effective. In most cases, acid baths are inexpensive; however, acid resisting equipment is necessary and when needed for large applications can be expensive.

19.2.5 Alkali cleaning. Alkaline cleaners have the ability of removing oils and greases. They are effective on a number of contaminants.

Table XXXXV list the soils that alkaline cleaners are highly effective in removing.

Table XXXXV.

Contaminants alkaline cleaners remove

Oils	Semisolids	Particulates
Animal oils Vegetable oils Mineral oils	Greases Waxes Rust preventatives Soaps	Oxides Solder fluxes Baked oil films

19.2.5.1 Mechanisms of alkaline cleaning. There are a number of mechanisms by which alkaline cleaners remove soils. Caustic solutions saponify lipids and fatty materials converting them to soaps; they also emulsify oils and disperse particles.

19.2.5.2 Processes that utilize alkaline cleaners. There are a number of processes that use alkaline cleaners. The most common are immersion or dip cleaning. Occasionally alkaline cleaners are used for spray cleaning; however, because of the problem of foaming, spray cleaning is not as effective as immersion cleaning. Other cleaning methods that utilize alkaline compound cleaners are: barrel cleaning, steam gun cleaning,and electrolytic cleaning. Electrolytic cleaning with alkaline solutions is the most effect-ve of the alkaline cleaning methods.

19.2.5.3 Temperatures. Alkaline cleaners are generally used hot, 160 - 180°F. When using alkaline cleaners, thorough rinses are imperative because alkaline residue can cause corrosion.

19.2.5.4 Agitation. Agitation is important in immersion cleaning with alkaline compounds. Cleaning is accomplished by chemical reaction at the surface of a part; therefore, it is necessary to agitate the solution to aid in removing the soil from the surface.

19.2.6 Ultrasonic cleaning. The removal of surface soils by forces created through the implosion of vaporous bubbles (cavitation) is known as ultrasonic cleaning. This technique has been developed to the degree that it is recognized as a very effective way of removing most soils and has definite advantages over other methods. Ultrasonic cleaning is a commonly used technique in precision cleaning.

Cleaning by ultrasonic is fast and thorough; cleaning cycle times range from a few seconds to a few minutes. It is, however, more complex than ordinary cleaning methods. Its cost must be expressed in relative terms for the application. It may be the most economical means available in terms of total cost for a particular job, yet an expensive luxury and less effective than other methods when misapplied.

In addition to the characteristics mentioned above, a properly applied ultrasonic cleaning process offers the following advantages over other methods:

(a) Less heat required.

(b) Parts can be cleaned without harming surface finishes.

(c) Assemblies can be cleaned without dismantling.

(d) Soils can be removed from minute cracks, pores, and indentations.

(e) Delicate parts and assemblies can be cleaned with a vigorous, yet gentle action.

19.2.6.1 Basic equipment. An ultrasonic cleaning unit is comprised of three principal parts:

(a) A generator which produces high-frequency electrical energy.

(b) A transducer which changes the electrical impulses into high-frequency sound waves that vibrate at a frequency in the ultrasonic range.

(c) A cleaning tank to which the transducer is attached, containing the appropriate cleaning agent.

Accessories may include work-handling devices such as baskets and hooks, frequency monitoring instrumentation, heaters, dryers, and fluid filtering and recirculating equipment. Depending on the specific application, each of these accessories may increase the overall effectiveness of an ultrasonic cleaning process.

19.2.6.2 Ultrasonic cavitation. Ultrasonic cleaning utilizes the scrubbing action of imploding liquid vapor bubbles to loosen and remove soil from a part. This action is enhanced by using a cleaning solution as the conducting media.

When sound passes through an elastic medium like metal, both the sound energy and medium vibrate at the same rate. When sound passes through a liquid, the liquid ruptures or cavitates, that is, small vacuum pockets are created which almost immediately collapse. This cavitation occurs, however, only at or above the sonic intensity level of the threshold of cavitation. The rapid implosion of thousands of these cavitations provides a rapid and rigorous scrubbing action. Because cavitation can be produced essentially throughout the entire tank, it can provide a scrubbing action even in blind holes, crevices, and other normally inaccessible places.

Ultrasonic energy is transmitted into the cleaning tank by the transducer on the bottom or side of the tank. The effectiveness of the ultrasonically induced cavitation therefore largely depends on the ultrasonic power intensity in the cleaning tank.

19.2.6.3 Significant properties effecting ultrasonic cleaning. While all of the liquid physical properties will have an effect on ultrasonic cleaning, the four most significant properties are:

(a) Vapor pressure.

(b) Surface tension.

(c) Viscosity.

(d) Density.

19.2.6.4 Applications and soils removed. Allied with some of the advantages of ultrasonic cleaning mentioned previously, are the variety of applications and the effectiveness in removing a wide range of soils displayed by the ultrasonic cleaning process. The following tables present typical examples of cleaning solutions used, types of soils removed, and the types of parts that can be cleaned by ultrasonic methods.

Table XXXXVI.

Frequently used cleaning solutions

Water	Other aqueous solutions
Deionized water	Ethanol
Water + surfactants	Chlorinated hydrocarbon solvents
Water + mild acid	Fluorinated hydrocarbon solvents
Water + mild alkali	Water + solvents

Table XXXXVII.

Examples of soils removed

Small particulate matter	Graphite and drawing compounds
Light oils and grease	Microbial contaminants
Buffing and polishing compounds	Fingerprints
Resists	Heat scale
Solder flux	Abrasive dust
Coolant residue	

Table XXXXVIII.

Parts that can be ultrasonically cleaned.

Bearings	Filters
Precision gears	Semiconductors
Printed circuit boards	Microfilm
Lenses	Electrical and electronic components
Hydraulic system components	Surgical instruments

The preceding listings are not intended to be inclusive, but they indicate the variety of cleaning situations in which ultrasonic cleaning can be applied successfully.

19.2.6.5 Hazards. Contact with ultrasonically activated equipment or solutions by any part of the body could cause bone or tissue damage. Hands and other parts of the body should not be immersed in cleaning fluid when ultrasonic generator is on. Any ultrasonic cleaning system should be properly grounded to prevent electric shock. Sound levels in the audible range should be within acceptable limits.

19.2.6.6 Ultrasonically cleaning electronic components. Electronic components such as transistors, diodes , semiconductors, and assembled circuit boards have been damaged as a result of being cleaned in ultrasonic cleaners. Since it is also well established that such components can be successfully cleaned by this method, it is important to observe the rules set forth in Table XXXXIX.

Table XXXXIX.

Considerations for ultrasonically cleaning electronic components.

Parameter	Resolution
Physical structure	Components manufactured by some producers are not structurally sound enough to withstand ultrasonic cleaning. The alternatives are to clean them in another manner or use other components that can be cleaned ultrasonically.
Power intensity	Any delicate component has an upper limit for power intensity with reference to time. A maximum of 5 W/in.^{2} at a nominal 20 kHz is suggested.
Power uniformity	Cleaners with a nonuniform power intensity can expose components to higher intensities than those recommended for safe operation on electronic components.

(continued)

Parameter	Resolution
Mass of load	Electronic components, particularly circuit boards, should be ultrasonically cleaned in racked loads, not as individual parts.
Dwell time	The time the parts are exposed (immersed) to ultrasonic cavitation should be very closely controlled; 5 to 15 seconds is usually sufficient, depending on all other considerations.
Cleaning solution	The type of solution has a direct effect on the type (hard or soft) of cavitation produced. Solutions with those qualities producing soft cavitation, i.e., high-vapor pressure and low-surface tension, should be used.

Table L.

Considerations for effective ultrasonic cleaning

Factors of Operation	Remarks
Cleaning solution	Select the proper cleaning solution for the cleaning job to be done. Liquid level should be adequate to prevent damage to ultrasonic unit.
Degassing	Any solution should be degassed before use. Complete degassing time depends on the properties of the solution, especially temperature, tank depth, power intensity, and type of pulse. Shallower tanks, higher solution temperatures, and higher power intensity with a pulsed wave allows faster degassing. Soft or deionized water is more easily degassed than hard water. Partial degassing (80-85%) should take place in about 5 minutes. Complete degassing should occur after about 30 minutes, but the time may vary depending on the above factors. Heating the solvent to within 3° to 5°F of its boiling point will also accomplish degassing.
Immersing parts	Slow immersion and removal of parts from the cleaning solution will prevent the introduction of air and the bouncing back of reflective ultrasonic waves to the transducer and generator. Parts should be withdrawn from the tank while the cleaner is operating. If the ultrasonic tank emits sharp screeching sounds, the parts are being introduced too fast and the generator is put under undue stress.

(continued)

Factors of Operation	Remarks
Parts loading	Load parts in the ultrasonic cleaning tank such that neither the parts nor the parts basket is on the tank bottom. Heavy parts (high mass) should each be exposed to direct ultrasonic waves. If they are stacked on top of each other, the top parts may not get cleaned. The sum of the parts cross-sectional area should not exceed 75% of the tank cross-sectional area. For the most effective cleaning, total parts weight should not exceed 35 lb/kW of generator power, even though a slightly higher weight-to-kW ratio can be accommodated. Elastomers and nonrigid plastics will absorb ultrasonic energy and will produce a shadowing effect; insulated parts may have to be specifically oriented.
Container design	Incorrect basket design or a basket having too high a mass can greatly reduce the effectiveness of the best ultrasonic cleaning system. Any material more tightly woven than 50 mesh screen acts as a solid sheet, while slightly larger openings scatter the ultrasonic waves; openings larger than 1/4 in. act as open material. Hooks, racks, and beakers can also be used to support parts.
Parts positioning	Parts positioning, important in most cleaning operations, is doubly important in ultrasonic cleaning. If possible, critical areas to be cleaned should face the transducer, but racked parts should be positioned vertically rather than being stacked one on top of the other. Care must be taken so that air is not trapped in blind holes, thereby preventing liquid contact with all surfaces. Parts should be covered by at least 3/4 in. of cleaning fluid, and if possible, should be positioned at the correct depth to take advantage of the higher intensity at the antinode of the ultrasonic wave.
Noises	Audible noise of two types may be produced, a buzzing or hissing noise is caused by the shock waves produced by cavitation bubbles; screeches or squeals resulting from "beat notes" should be avoided. Beat notes can be caused by (1) moving parts in the tank too fast, (2) too violent agitation of the liquid, (3) resonant bubbles, or (4) two or more nonsynchronized generators. Introduction of a parts basket or parts into the liquid will usually eliminate beat notes from these causes. Beat notes may also be caused by cleaning chemicals which do not form clear solutions or by excessive accumulations of greasy soils which are not

(continued)

Factors of Operation	Remarks
Noises (continued)	completely soluble in the solution used; if this occurs, use a different solution or change the solution more frequently.
Filtering	In precision cleaning, particulate matter must be removed from the cleaning fluid. This may be done with a continuously recirculating filtration system simultaneously with ultrasonic cleaning. The rate of flow through a filter system should be determined through experimentation with each cleaner. A depth filter should precede the absolute filter for maximum practical effectiveness. The rate of flow is generally not over 1 to 3% of the tank volume per minute, as too high a rate will prevent the cavitation process by creating turbulence and may produce a screeching sound.

19.2.7 Electrolytic cleaning. The process of electrolytic cleaning is an effective means of metal cleaning. The parts are immersed in an electrolysis bath. A vigorous gas evolution occurs at the parts surface causing the loosening and removal of contaminants. This process is a sophistication of immersion cleaning and is much more effective than ordinary immersion cleaning.

19.2.7.1 Solutions used. In electrolytic cleaning both alkaline and acetic solutions can be used. Electrolytic pickling is the term that applies to electrolytic cleaning when an acid is the cleaning solution.

19.2.7.2 Current densities. For effective electrolytic cleaning, current densities must be at least 10 amp/ft^2 and can range to 150 amp/ft^2. However, there is a point at which an increase in current density will not increase cleaning action. In this case, the current density creates such an intense bubble formation that a semi-continuous film is formed over the surface of the part decreasing the scouring action.

Electrolytic cleaning facilities must have good ventilation. The process of electrolytic cleaning causes hydrogen and oxygen gas to be formed. These gases will combine as bubbles and miniature explosions will occur sometimes forming a mist. The mist could be harmful to workers. Accumulation of hydrogen gas is dangerous as it is flammable.

A related process is electrolytic polishing. (See Section 18, 18.14.1)

19.2.8 Spray cleaning. Generally, the solutions used in immersion cleaning can also be used in spray cleaning; however, the process of spray cleaning increases the effectiveness of the solutions. Spray cleaning is the result of three functions, first, the cleaning power of the solution, second, the force of the solution imparted on the contaminated surface, third, continuous replacement of fluid. As soils become loosened they are immediately flushed away. The spray nozzle should produce a coarse spray to lessen the influence of viscous decay thereby allowing a high kinetic energy impact to surfaces.

19.2.8.1 Equipment. The spray cleaning technique can be used in cleaning any size part. Equipment used in spray cleaning is quite varied. Some equipment has fixed spray heads and others have rotating heads. The type of equipment chosen for a particular job is relative to the parts being cleaned.

19.2.8.2 Cleaning time. The typical cleaning cycle in spray cleaning is short, 1/2 to 2 minutes.

19.2.8.3 Etching with spray cleaners. Spray cleaning techniques increase the effects of etchants and acids. After an acid or etchant comes in contact with a surface, etching ability diminishes due to chemical reaction. In spray cleaning, the expended solutions are constantly being replaced by fresh solutions thereby accelerating etching. This process also produces a uniform etching on irregular surfaces.

19.2.8.4 Versatility. Spray cleaning is a versatile process. There are no limitations to the size of parts that can be cleaned by this process. Frequently, the spray process is used on very large parts that are driven through spray tunnels. The process is also used in internal cleaning of tanks.

19.2.9 Vapor degreasing. The primary purpose of vapor degreasing is the removal of oil and grease films from surfaces. It is not an effective method of removing particulates. Frequently, vapor degreasing is used as a precleaning step for the purpose of loosening adhesive bonds of particulates that have become fixed to surfaces by oil and grease films.

19.2.9.1 Degree of cleanliness offered. Vapor degreasing produces a degree of cleanliness sufficient for many industrial operations. When used with an ultrasonic unit, it provides cleanliness levels that are adequate for precision and miniaturized parts and for subsequent electrical or electrochemical processes. A degree of cleanliness between 1.0 and 0.1 monomolecular layers of contamination has been achieved.

19.2.9.2 Degreasing equipment and process. Vapor degreasers are comprised of a tank, heating elements on the bottom of the tank, water cooled coils around top perimeter of the tank. A volatile solvent is put into the tank and heated forming a high density vapor. Vapor is not lost from the tank because of condensor tubes located at the top of the tank. When a cold part on a rock or in a mesh basket is immersed into the vapor cloud, vapor condensation is formed on the surface. The solvent vapor has a very good solvency for oil and grease films. As the vapor drips off taking with it the oils, greases, and particulates attached, more pure condensing solvent is replaced. The process continues until the part being degreased becomes heated to the point where condensation ceases.

19.2.9.3 Solvents requirements. There are a limited number of solvents that possess the properties necessary for use in degreasing units. The necessary solvent requirements are:

(a) High solvency for oils and greases.

(b) Low heat of vaporization.

(c) Low heat capacity.

(d) Boiling point high enough to produce condensation on room temperature part; low enough to produce separation from contaminants by distillation.

(e) Good chemical stability.

(f) Vapor produced should be noncorrosive and nonflammable.

19.2.9.4 Commonly used solvents. The solvents commonly used for vapor degreasing are:

(a) Trichloroethylene, most frequently used.

(b) Perchloroethylene.

(c) Trichlorotrifluoroethane.

(d) Methylene chloride.

(e) Methyl chloroform. (Requires stringent stabilizer control.)

(f) Azeotropic mixtures.

19.2.9.5 Types of vapor degreasers. There are a variety of vapor degreasers. Table LI lists several different types of vapor degreasers with their different applications.

Note: The Navy recommends the use of 1,1,1, trichloroethane (methyl chloroform), Mil-T-81533 or OT-620 in place of trichloroethylene. The Navy has procured ultrasonic filter cleaning machines which were designed and calibrated to use methyl chloroform, Mil-T-81533.

Table LI.

Types of vapor degreasers

Type of Cycle	Application
Vapor only. Dip into vapor, remove, let dry. (10 - 15 seconds)	Removal of light soils soluble in the degreasing solvent; flat surfaces or other simple shapes. Not effective for thicknesses less than 1/8".
Spray-vapor	Used primarily for compounds which would be baked on the workpiece if first subjected to vapor degreasing.
Vapor-spray-vapor. Dip into vapor, leave until condensating ceases. Spray liquid solvent on part to cool. Let condensation occur again.	More effective in removing medium light soils and insoluble contaminants, penetrating blind holes, and cleaning partially accessible complex shapes. Good for small thin parts.
Vapor-immersion-vapor	Generally used for removing medium heavy soils on parts with thin wall sections or small bulk parts in baskets or other containers.
Boiling solvent-cool solvent-vapor. Dip into boiling solvent, then warm solvent and then into vapor.	Effective for loosening and removing heavy soils and for cleaning closely basketed complex shapes. Combines gross cleaning and final cleaning.
Ultrasonic	Combined with vapor degreasing, produces a higher degree of cleanliness; applied after precleaning; followed by spray and vapor rinses; may be used for small, intricate parts, printed circuit boards, castings, miniature assemblies, and removing contamination from blind holes or remote surfaces.
Other combinations	Other cycle combinations may be devised for special cleaning needs. Experienced equipment manufacturers can furnish this assistance.

19.2.9.6 Personnel hazards.

(a) Toxic vapors are present with degreaser solvents ;therefore, adequate precautions must be taken. Excessive inhalation or adsorption may cause nausea, headaches, fatigue, and loss of sense of balance. Toxic fumes may be produced if the solvent is exposed to intense light or adjacent high-temperature operations. Adequate ventilation must be used when degreaser tanks are being cleaned. A second person should always be present during the cleaning operation.

(b) Dermatitis may result from repeated or prolonged solvent contact with the skin. The skin becomes rough, red, and dry due to removal of natural skin oils. It then cracks easily and becomes susceptible to infection.

(c) Degreaser operators may form the habit of intermittently inhaling small amounts of solvent vapor. Proper supervision must be exercised to detect and prevent such practices. Death has been attributed to the strong narcotic power of trichloroethylene vapors in high concentrations.

19.2.9.7 Vapor degreaser limitations.

19.2.9.7.1 Acidity. Solvent may decompose, become acidic, and attack copper, titanium, aluminum, and magnesium; chlorides formed by decomposed solvents and aluminum or magnesium will further contaminate electronic components. In order to lessen these effects, periodically check solvent acidity; follow prescribed degreaser cleaning schedules; use different degreasers for aluminum or magnesium and electronic components.

19.2.9.7.2 Rust. Degreased ferrous parts are highly susceptible to oxidation. Protective measures are: provide clean, dry atmospheres; apply rust-proofing solutions.

19.2.9.7.3 Heat effects on nonmetals . High solvent temperatures are deleterious to some materials such as rubber and plastic. Heat tolerances of particular materials should be determined before subjecting part.

19.2.9.7.4 Heat effects on metals. Certain polishing and buffing compounds may bake on parts at high solvent temperature. To lessen the occurrence, use spray-vapor cycle or soak or flush with other solvents and hand brush prior to degreasing.

19.2.9.7.5 Chlorides. Chlorides formed by decomposed solvents and certain metals (aluminum, magnesium, beryllium) will attack some electronic components, rubbers, and plastics, as well as the metals themselves; do not use chlorinated solvents with titanium. Check chloride effects on nonmetals; provide proper degreaser maintenance; consider separate degreasers for electronic components.

19.2.9.7.6 Thin-walled parts. Part temperature may increase to vapor temperature quickly so that desired cleanliness is not achieved. Use vapor/spray/vapor cycle or cool and repeat vapor cycle.

19.2.9.7.7 Large parts. Part may be too large for available degreaser. Degrease a portion of the part at a time; hand clean with solvents.

19.2.9.7.8 Blind holes. Depth, location or orientation may present cleaning or draining problems. Use vapor/spray-vapor cycle; ultrasonic plus vapor; rotate part to permit drainage.

19.2.9.7.9 Convoluted parts and tubing. Solvents and dissolved contamination may become trapped in convolution or tortuous passages. Parts should be fixtured to rotate during degreasing and drying; a solvent pressure flush may be required.

19.2.9.7.10 Metal castings. Due to micro- and macro- porosity, all contaminants may not be removed from the pores; prevents proper impregnation; may subsequently weep oil. If no insoluble contaminants present, recommend oven bake and combination of ultrasonic-vapor degreasing cleaning.

19.2.9.7.11 Oil impregnated powder metal parts. Bearings and bushings may be oil-impregnated for the life of the part; degreasing will remove portions of this lubricant. If cleaning is required, mild solvent surface wipe or brush; or completely degrease and reimpregnate.

19.2.10 Flush cleaning. Cleaning the internal surfaces of components is a prime concern in contamination control technology. Internal surface areas must be flush cleaned to remove contaminants.

19.2.10.1 Properties of flushing fluids. The fluid used in flushing must possess two capabilities. First, the fluid must be able to either dissolve contaminants or break adhesive bonds that bind the contaminants to surfaces; second, the fluid must then be able to suspend the contaminants and remove them.

19.2.10.2 Equipment. Flush cleaning apparatus is usually simple consisting of a reservoir of fluid, filters, and a pump. The flush cleaning apparatus should be able to operate at flow rates as high as the component being cleaned can tolerate.

19.2.10.2.2 Flush cleaning in an ultrasonic tank. Flush cleaning can be accomplished in an ultrasonic tank. The ultrasonic energy acids in breaking the particle bond and the flushing fluid removes the suspended particulates.

19.2.10.3 Cleanliness verification. Determining the cleanliness level of a part that must be flush cleaned presents a problem. There is no direct method that can be employed when observation of the surfaces is impossible. When this is the case, the technique most frequently used is monitoring the flushing fluid after it passes through the component. When the particle count in the effluent reaches acceptable limits the component is considered clean.

19.3 Choosing a cleaning technique. There is no one ideal cleaning method. Generally, in order to achieve high levels of cleanliness combinations of the various cleaning techniques previously mentioned are employed. For example, vapor degreasing is effective in removing oils and greases, however, is virtually ineffective in removing particulate material. Spray cleaning is effective in removing particulate material, however, ineffective in removing oils and greases. By combining these two techniques, one can realize the increase in cleaning effectiveness. Frequently, cleaning fluids are incompatible necessitating the complete removal of one before proceeding to another. Since this problem exists the importance of rinsing between cleaning cycles is imperative.

Determining the technique or combination of techniques used in a particular cleaning operation is an individual matter. The choice or choices depends on a number of factors, predominately, level of cleanliness necessary, design of part, material to be cleaned, and type of soil.

Table LII lists various cleaning methods and their effectiveness in soil removal.

19.4 Precision cleaning solutions. Precision cleaning solutions shall not react with, combine with, etch, or otherwise cause immediate or latent degradation of the item being cleaned. Precision cleaning fluids shall be filtered and free of residue, controlled and verified at a cleanliness level that is demonstrated to be sufficient to achieve the specific product cleanliness. Some solutions common to precision cleaning are:

(a) Halogenated solvents (nonflammable) chlorinated hydrocarbons, fluorinated hydrocarbons.

Table LII.

Comparison of cleaning method effectiveness for soil removal

Cleaner or Cleaning Method	Pigmented Drawing Compounds	Unpigmented Oil and Grease	Chips[3] and Cutting Fluids	Polishing and Buffing Compounds	Rust and Scale[4]
Emulsion	Emulsified solvent better than emulsions alone; good for this type soil.	Precleaner or preparation for other operations - not preferred as primary cleaner.	Combined immersion and spray effective; often used after alkaline; check pH.	Used on ferrous and some nonferrous materials. Effective if sprayed on most compounds; should be rinsed.	Not recommended.
Alkaline	Generally marginal; more effective with spray, agitation, or impingement.	Efficient for most soils; must have good rinse; must be inhibited for non-ferrous.	Effective and economical; must be inhibited for non-ferrous.	Widely used and least costly; agitation or spray with surfactant recommended.	Removes rust, light scale; no metal loss or hydrogen embrittlement.
Acid(1)	Effective for dried compounds, with water and spray.	Good for this soil; often used with spray; some form protective coating; etch nonferrous.	Effective when used with power spray.	Generally not recommended, except for newly applied, easily removed compounds.	Removes rust on ferrous metal parts; acid pickling removes mill scale.
Electrolytic Alkaline	Not recommended; may be used as final cleaning after other methods.	Produces very clean surfaces; usually final cleaner prior to electroplating.	Recommended for high cleanliness levels; also good final cleaning.	Used after precleaning; tendency to contaminate solution.	Faster than alkaline baths; parts require racking; can remove other soils in one operation.
Solvent(2)	Generally not suitable; sometimes used as precleaner.	Primarily precleaning; effectiveness increased by spray, agitation, and scrubbing.	Immersion, agitation, and spray effective with solvent soluble soils.	Soak, spray, or brush with solvents is effective as a precleaner.	Not recommended.
Vapor Degreasing(2)	Limited value; more effective with dip, spray, ultrasonic combinations.	Very effective; wide range of uses; repeatable; parts immerge dry.	Good only for solvent soluble soils; used with immersion, spray, and air blowoff.	Compound may bake on unless spray precedes vapor phase.	Not recommended.
Ultrasonic	Effective if properly used, but more costly.	Very effective for removing this type soil on small or precision components.	Effective for some soils; guard against chip accumulation.	Generally used as final cleaner on intricate or precision parts.	May remove light rust or scale from small or complex parts.

1. Acid cleaning is effective with many types of soils but is generally more costly than other equally effective methods.
2. Since vapor degreasing utilizes solvents, these two methods are similar. Some solvents, however, are not suitable for vapor degreasing.
3. Soils bonding chips to parts can be removed by these methods, but physical force such as gravity, spray, agitation, etc., may be needed to remove the chips from the part.
4. In removing rust and scale, other methods such as pickling, salt bath, brushing, abrasive blast, and tumbling should be considered.

(b) Ketones.

(c) Alcohols.

(d) Detergent (must be followed by deionized water rinse).

(e) Deionized water.

(f) Air or inert gas (for drying or removal of volatile fluids).

19.4.1 High purity water. Among its other vital uses, water has always been known to man as a washing agent. Today, large quantities of high purity water are needed by industry in chemical production, parts rinsing, and cleaning operations. Although water is often considered as a rinsing medium and is frequently used for this purpose, high purity water is also a highly effective solvent for some soils.

19.4.1.1 Impurities in natural water. In its natural state, water contains many impurities which inhibit or preclude its use for industrial purposes. The types and concentrations vary widely with different geographical areas but can generally be classified as:

(a) Dissolved minerals.

(b) Organic matter.

(c) Suspended particulate matter.

(d) Dissolved gases.

(e) Microorganisms.

19.4.1.2 Design of water purification systems. Any system for purifying water must be designed for its particular application. Each combination of system, use requirements, and water supply is unique in its characteristics. The use requirements will establish the maximum concentration of acceptable impurities. In order to determine the kind of system needed, however, the type and amounts of impurities in the locally available water must be carefully analyzed. In some cases where the mineral content is not excessive, it may be more economical to use private wells rather than the municipal water supply.

19.4.1.3 Mineral content. The mineral content of water is expressed in terms of "hardness", or more specifically, "grains of hardness," where one grain equals 17 ppm of calcium carbonate. Other constituents of water include silica, iron, sodium, and magnesium.

19.4.1.4 pH ranges. Natural water, as found in the United States, is relatively neutral. It ranges in pH value from 6.5 to 8.0. Generally, water found along the east coast is neutral or slightly acidic while water in midwest and western parts of the country is slightly alkaline.

The pH relative strength chart below shows the relationship of pH values to relative acidities and alkalinities. The acid region is below 7 pH, the pH values decreasing within areas of acidity. The alkaline region is above 7 pH, the pH values increasing with increasing alkalinity. Note that the relative strength changes tenfold for each unit change in pH.

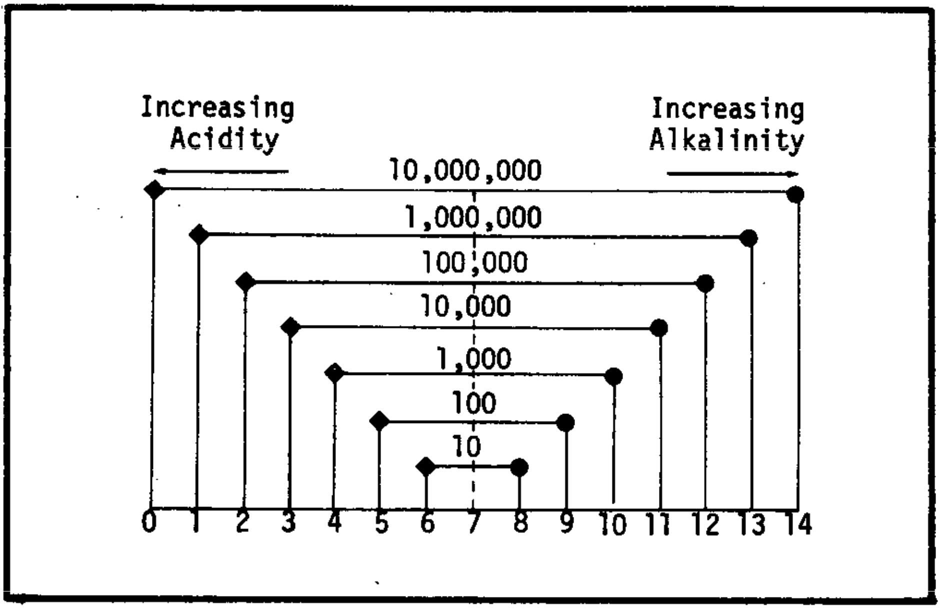

Figure 19. pH relative strength chart

19.4.1.5 Water purification methods. Many water purification methods are commercially available. Again, the correct system for any one application must be determined only after considering all parameters of that application, such as the volume of water needed, the type of use (intermittent or continuous), the degree and type of purity required, site of use, and the distribution system. Various purification techniques and their functions are listed in Table LIII.

Table LIII.

Water purification methods

Purification Method	Contaminants Removed or Function	Remarks
Distillation	Removes gross amounts of common contaminants.	Original purification method; slow; not practical for large quantities.
Activated charcoal	Removes odors, dissolved gases and residual organic material.	
Chlorination	Kills micro-organisms.	
Ultraviolet germicidal lamp	Kills exposed micro-organisms	Lamp should be checked periodically for effectiveness.
Settling	Removes coagulated materials and suspended particulate matter.	
Softening	Removes calcium, magnesium, and iron ions.	Zeolite type beds; increases sodium content and leaves neutral salts.
Demineralization	Removes cations and anions to a high-purity level.	Dual-bed for heavy contaminants; mixed-beds for lighter contaminants or combined systems.
Filtration	Removes suspended particulate and coagulated matter, and bacteria above the filter pore size.	Filters must be replaced periodically to remove contaminants and assure required volume.

Although other water purification techniques are commonly used to increase water purity, demineralization by ion exchange in resin beds or columns is a most effective method of producing high purity water. This method uses resins which have the property of exchanging the ions which make up water, H+ (hydronium) and OH- (hydroxyl), for the dissolved mineral ions in the water. Most deionization systems can be regenerated when their supply of H+ and OH- ions has been exhausted.

19.4.1.6 Electrical conductivity. The electrical conductivity (and resistance) of water varies with mineral content, thus providing a readily available means of measurement and monitoring. The conductivity of water also depends on the temperature. The dissociation of water increases as the temperature rises, so that the conductivity varies with temperature.

19.4.1.7 Recirculation of water. Many deionizing systems are designed to reuse the deionized water after it has been used for washing or rinsing purposes. The impurities picked up are generally much less and more easily removed than the impurities in raw water.

19.4.1.8 Maintaining sterility. Maintaining the sterility of high purity water is an operating consideration which requires periodic or constant surveillance and control in bacteria-sensitive systems. Flushing the system periodically with sodium hypochlorite or other liquid sterilizing agents will control bacterial growth in most instances. Bacterial growth can also be retarded if the water in the system is not exposed to air. If resins become coated with bacteria, their effectiveness may be seriously reduced or completely eliminated.

19.5 Drying. The subject of drying is closely associated with parts cleaning. Several methods are discussed in this section because each may have application depending on the cleaning situation encountered. It should be pointed out, however, that the environment during cleaning and drying, and prior to the next operation, must be sufficiently clean to maintain the level of cleanliness achieved by the cleaning operation. If superclean surfaces are required, cleaning and drying operations should be performed in a clean bench or clean room environment.

19.5.1 Three categories of drying. Drying methods are generally divided into three categories. Table LIV lists the various drying methods.

Table LIV.

Drying methods

Mechanical	Chemical	Thermal
Wiping Air blowoff Dry nitrogen blowoff Sonic	Alcohol Acetone Freon T-WD 602 Freon T-DA 35 Trisec	Ovens Infrared Light banks Vacuum ovens

Characteristics of these different methods are shown in Table LV.

Table LV.

Characteristics of drying methods

Method	Drying Action	Remarks
Wiping	Absorption by wiping material	Use lint-free cloth; usually gross or predry operation; not preferred.
Air blowoff	Physical force displaces cleaning solution; air movement aids evaporation	Air supply line must have filter, water trap and regulator; use care in not agitating other contamination in immediate environment.
Dry nitrogen blowoff	Same as above	Essentially same as above; final drying for precision work.
Sonic	Moisture concentration gradient near part surface modified by air agitation by sound waves; extraction	Not too widely used with metals; can be used with heat-sensitive parts or where high-velocity air would cause damage.
Alcohol & acetone	May combine cleaning and drying; water adsorption; rapid evaporation.	Effective in some cleaning operations; requires caution in handling; highly flammable.
Freon T-WD 602	Water emulsification Vapor evaporation	Dries traces of residual water from surfaces, small holes, and cracks; used in ultrasonic or vapor-degreasing equipment.
Freon T-DA 35	Water displacement	Removes large or small quantities of water from metals, plastics, and material combinations; used in conjunction with Freon TF in specially designed, vapor degreasers; parts must be properly oriented in the solution.
Trisec	Water displacement	Combination of chlorinated solvent and a cationic surfactant; requires modified vapor degreaser.
Ovens	Evaporation by heat	Should be recirculating, indirect gas-fired or electrically heated, with high-volume static exhaust; not applicable for heat-sensitive materials.
Infrared and heat light bank	Evaporation by heat	Not normally used if contamination control is a factor; usually a conveyorized operation.

19.5.1.1 Displacement drying. Due to the re-active newness of water displacement drying systems, some of the distinguishing characteristics of this type system are listed below:

(a) Relatively low operating temperature - 120°F.

(b) Drying cycle can be completed in 4 to 7 minutes.

(c) Nonflammable, low hazard materials.

(d) Freon T-DA 35 and water are immiscible; therefore, the water is separated and discharged without contaminating the solvent.

(e) Spot-free and contamination-free surfaces are obtainable.

19.6 Precision cleaning methods and procedures.

19.6.1 Cleaning stainless steel.

19.6.1.1 Purpose. To establish the precision cleaning methods related to small assemblies consisting of stainless steel and soft good components.

19.6.1.2 Materials.

(a) Nitrogen gas (GN_2), Mil-P-27401.

(b) Polyethylene film, Mil-P-3803.

(c) Nylon 6.

(d) Trichloroethylene solvent, Mil-T-634.

(e) Trichlorotrifluoroethane (freon).

(f) Tape, pressure sensitive PPP-T-60 or PPP-T-66.

(g) Detergent, nonionic , Mil-D-16791.

(h) Demineralized water or deionized water.

(i) Isopropyl alcohol, Fed-Spec-TT-I-735.

(j) Nitric acid.

(k) Sodium dichromate.

(l) Caustic soda.

(m) Trisodium phosphate.

19.6.1.3 Definition. Solvent shall mean trichloroethylene or freon.

19.6.1.4 Cleaning procedure. Metallic parts may be cleaned as follows:

(a) Degrease in a trichloroethylene vapor degreaser by lowering and keeping parts into solvent vapor until vapor ceases to condense on the surface of the parts. An alternative method of degreasing is thoroughly brushing the parts with a nylon brush and trichloroethylene. Other degreasing solutions such as, trisodium phosphate: water (10 ±2%: water at 140-160°F) or caustic soda: water (7 ± 2%: water at 140-160°F) may be used with caution.

(b) Immerse parts in ultrasonic cleaner which contains 2-3 oz /gal nonionic detergent and water for 10-15 minutes, or clean by brushing with a nylon-brush in detergent and water until parts are visually clean.

(c) Rinse parts with deionized water until effluent pH and influent pH are the same when checked with pH paper.

(d) Passivate. If parts require passivation the following procedure should be followed.

  (1) Immerse parts in a solution of 20 ± 5% nitric acid plus 2 ±0.5% sodium dichromate at 130-150°F for 15-30 minutes. If solution is used at ambient temperature immerse for one hour.

  (2) Rinse parts with deionized water until effluent and influent pH are the same when checked with pH paper.

(e) Dry parts with gaseous nitrogen. (Alcohol rinse before drying will lessen drying time).

(f) Forward parts to clean room for final sampling. Sample according to techniques outlined in Section 16.

(g) Package according to instructions in Section 20.

19.6.2 Cleaning carbon steel.

19.6.2.1 Purpose. To establish the precision cleaning methods related to small assemblies consisting of carbon steel and soft good components.

19.6.2.2 Materials.

(a) Nitrogen gas (GN_2), Mil-P-27401.

(b) Polyethylene film, Mil-P-3803.

(c) Nylon 6.

(d) Trichloroethylene solvent, Mil-T-634.

(e) Trichlorotrifluoroethane (freon).

(f) Tape, pressure sensitive PPP-T-60 or PPP-T-66.

(g) Detergent, nonionic, Mil-D-16791.

(h) Demineralized water or deionized water.

(i) Isopropyl alcohol, Fed-Spec-TT-I-735.

(j) Citric acid.

(k) Aqua ammonia 26° Baumé.

(l) Caustic soda.

(m) Trisodium phosphate.

(n) Sodium nitrite.

19.6.2.3 Cleaning procedure. Metallic parts may be cleaned as follows:

(a) Degrease in a trichloroethylene vapor degreaser by lowering and keeping parts into solvent vapor until vapor ceases to condense on the surface of the parts. An alternative method of degreasing is thoroughly brushing the parts with a nylon brush and trichloroethylene. Other degreasing solutions, such as, trisodium phosphate: water (10 ± 2%: water at 140-160°F) or caustic soda: water (7 ± 2%: water at 140-160°F) may be used with caution.

Warning: Many carbon steel surfaces have been painted in order to lessen corrosion problems. Degreasing will remove paint unless solution is kept from finished surfaces. If a thorough cleaning job is required, repainting may be necessary.

(b) Immerse parts in ultrasonic cleaner which contains 2-3 oz /gal nonionic detergent and water for 10-15 minutes or clean by brushing with a nylon brush in detergent and water until parts are visually clean.

(c) Rinse parts with deionized water until effluent pH and influent pH are the same when checked with pH paper.

(d) Passivate as follows:

(1) Immerse parts in a solution of 3 ± 0.5% (by weight) of citric acid in water at a temperature of 180 ± 10°F for a period of 1-3 hours, depending on amount of rust present.

(2) Allow solution to cool to 140 ± 10°F. Add sufficient aqua ammonia to raise the pH to 9 ± 1 as determined by pH paper. After pH adjustment is completed, add 0.5% (by weight) of sodium nitrite.

(3) Allow parts to remain in solution for 30 minutes.

(e) Rinse with deionized water until effluent pH approximates influent pH.

(f) Dry parts using gaseous nitrogen.

(g) Forward parts to clean room for final sampling. Sample according to techniques outlined in Section 16.

(h) Package according to instructions in Section 20.

19.6.3 Cleaning aluminum.

19.6.3.1 Purpose. To establish the precision cleaning methods related to small assemblies consisting of aluminum and soft good components.

19.6.3.2 Materials.

(a) Nitrogen gas (GN_2), Mil-P-27401.

(b) Polyethylene film, Mil-P-3803.

(c) Nylon 6.

(d) Trichloroethylene solvent, Mil-T-634.

(e) Trichlorotrifluoroethane (freon).

(f) Tape, pressure sensitive PPP-T-60 or PPP-T-66.

(g) Detergent, nonionic, Mil-D-16791.

(h) Demineralized water or deionized water.

(i) Isopropyl alcohol, Fed-Spec-TT-I-735.

(j) Nitric acid.

(k) Sodium dichromate.

(l) Caustic soda.

(m) Trisodium phosphate.

19.6.3.3 Definition. Solvent shall mean trichloroethylene or freon.

19.6.3.4 Cleaning procedure. Metallic parts may be cleaned as follows.

(a) Degrease in a trichloroethylene vapor degreaser by lowering and keeping parts into solvent vapor until vapor ceases to condense on the surface of the parts. An alternative method of degreasing is thoroughly brushing the parts with a nylon brush and trichloroethylene.

(b) Immerse parts in ultrasonic cleaner which contains 2-3 oz /gal nonionic detergent and water for 10-15 minutes, or clean by brushing with a nylon brush in detergent and water until parts are visually clean.

(c) Rinse parts with deionized water until effluent pH and influent pH are the same when checked with pH paper.

(d) Passivate. Aluminum parts which have not been conversion coated should be prossesed as follows:

  (1) Immerse parts in a solution of 30 ± 5% nitric acid at a temperature of 120 ± 5° F for 15 to 30 minutes.

  (2) Rinse parts using deionized water until pH of effluent approximates pH of influent when checked with pH paper.

(e) Dry using gaseous nitrogen.

(f) Forward parts to clean room for final sampling. Sample according to techniques outlined in Section 16.

(g) Package according to instructions in Section 20.

19.6.4 Cleaning teflon lined flexible hose.

19.6.4.1 Purpose. To establish the precision cleaning methods related to teflon lined flexible hose assemblies.

19.6.4.2 Materials.

(a) Nitrogen gas (GN_2), Mil-P-27401.

(b) Polyethylene film, Mil-P-3803.

(c) Trichloroetrifluoroethane .

(d) Trichloroethylene, technical, Fed-Spec-O-T-634.

(e) Nylon 6 .

(f) Deionized water, 50,000 OHM, min. resistance.

(g) Detergent, nonionic , Mil-D-16791.

(h) Tape , Fed-Spec-PPP-T-60.

(i) Isopropyl alcohol, Fed-Spec-TT-I-735.

(j) Nitric acid, Fed-Spec-O-N-350.

(k) Trisodium phosphate, Fed-Spec-O-S-642.

19.6.4.3 Definition. For the purpose of this procedure solvent shall mean trichloroethylene or trichlorotrifluoroethane.

19.6.4.4 Cleaning procedure.

(a) Prepare a solution containing 10 ± 2 oz trisodium phosphate and 1 ± 1/2 oz/gal nonionic detergent and add enough water to total one gallon of solution. Maintain temperature at 140 - 170°F. These same ratios will be utilized where greater quantities of solution are required.

(b) Clean by pumping prepared solution at full flow through hose assemblies for 15 minutes. While cleaning, exercise hose to its minimum bend radius on axes. Work the complete length at least 3 times.

(c) Flush with demineralized water until effluent pH is 6.0 to 8.0 .

(d) Clean and passivate end fittings only using 20 ± 5% nitric acid solution. Use brush with solution if necessary to aid in scale or corrosion removal. Immerse for 30 minutes at ambient temperature.

Caution. Do not allow nitric acid solution to come in contact with hose braid.

(e) Flush with demineralized water until effluent pH is 6.0 to 8.0 Disconnect hoses and clean each hose end by brushing with a nylon brush and flushing with deionized water. This will aid in the removal of entrapped contamination in hose interconnection fittings. After disconnecting hoses, do not let the insides dry before cleaning ends.

(f) Blow dry with gaseous nitrogen. An alcohol flush may be used to facilitate drying, or parts may be placed in a drying oven at 180 ± 10°F for 1/2 hour.

(g) Forward parts to clean room for sampling. (See Section 16.)

(h) Package according to instructions in Section 20.

19.6.5 Cleaning stainless steel convoluted hose.

19.6.5.1 Purpose. To establish the precision cleaning methods and requirements related to stainless steel convoluted hose assemblies.

19.6.5.2 Materials.

(a) Nitrogen gas (GN_2), Mil-P-27401.

(b) Polyethylene film, Mil-P-3803.

(c) Aclar film - 2 mil.

(d) Trichlorethylene, technical, Fed-Spec-O-T-634.

(e) Phosphoric acid, technical grade, Fed-Spec-O-P-313.

(f) Deionized water 50,000 OHM min. resistance.

(g) Detergent, nonionic, Mil-D-16791.

(h) Tape, Fed-Spec-PPP-T-60.

(i) Isopropyl alcohol, Fed-Spec-TT-1-735.

(j) Trisodium phosphate, Fed-Spec-O-S-642.,

19.6.5.3 Cleaning procedure for stainless steel convoluted hoses whose diameter is one inch or less. For each cleaning, rinsing, drying, and particulate sampling operation, the hose should be exercised to its minimum bend radius on all axes working the complete length at least three times. Place hose in a position that will eliminate air entrapments that might prevent total coverage of internal surface areas. Assure that a full flow is accomplished in the cleaning processes by holding hose horizontal and inspecting outlet for full diameter coverage.

(a) Degrease using trisodium phosphate 10 ± 2 oz/gal in water at 140° - 170°F Add 1 ± 0.5 oz nonionic detergent per gallon of water for wetting agent. Solution shall be pressure flushed through hoses.

(b) Rinse using filtered deionized water. Effluent water to have a pH between 6 and 8 before rinsing operation is complete.

(c) Clean by using phosphoric acid 20 ± 5% and 1/2 oz of nonionic detergent per gallon of solution. Solution shall be heated to 140° ± 10°F. Pressure flush solution through hose for 15 to 20 minutes.

(d) Rinse using filtered deionized water. Effluent water to have a pH between 6 and 8 before rinsing operation is complete.

(e) Flush with filtered isopropyl alcohol to facilitate drying operation.

(f) Dry using GN_2.

(g) Forward parts to final clean room for cleanliness test.(See Section 16.)

(h) After hose has met the cleanliness requirements it shall be dried with filtered GN_2 to a minus 40°F.

(i) Package according to instructions in Section 20.

19.6.5.4 Cleaning procedure for hoses greater than one inch diameter. Hoses in this category shall be cleaned in a position that will utilize the advantages of gravity drainage. Whenever practical, hoses shall be suspended vertically. If vertical positioning is not practical due to size or configuration, hoses shall be positioned at the most accurate angle practical for the cleaning and sampling operations. When hoses are of such a size that full flow is not attainable, spray cleaning shall be utilized. Spray cleaning shall be accomplished using a spray wand constructed so that a 360° spray pattern is produced at 60° to 90° of the longitudinal axis of the hose. The wand shall be introduced and moved from one end of the assembly to the other repeatedly as necessary to effect the cleaning operation. Chemicals and cleaning agents shall be provided to the spray head by a pumping system capable of a minimum of 40 fpm at a pressure of 480 foot head (water).

(a) Degrease using trisodium phosphate 10 ± 2 oz/gal and nonionic detergent 1 ± 5 oz/gal heated to 140° - 170°F. Spray or flow, as applicable, through the hose for 15 minutes minimum.

(b) Rinse with deionized water until effluent water has a pH of 6.0 to 8.0.

(c) Clean using phosphoric acid 20 ± 5% and nonionic detergent 2 ± 1 oz/gal heated to 130° - 150°F. Spray or flow,as applicable, through the hose for 15 minutes minimum.

(d) Rinse with deionized water until effluent water has a pH of 6.0 to 8.0.

(e) Using isopropyl alcohol, flow or spray the hose to facilitate drying.

(f) Dry by purging with GN_2.

(g) Forward hose to controlled environment for final sampling. When hose size or configuration prevents movement to the controlled environment, final cleaning and sampling may be accomplished in the pre-clean areas as follows:

 (1) Using cleaned polyethylene, form a "tent" over each opening of the hose to prevent fallout contaminants from entering the assembly.

 (2) Using trichloroethylene, spray the hose interior until all surfaces have been rinsed at least three times.

 (3) While continuing the spraying operation, take a sample of the effluent solvent for analysis.

 (4) While awaiting results of the analysis, a positive purge of GN_2 filtered to 10 microns absolute shall be maintained on the hose assembly.

 (5) After acceptance of the cleanliness sample,continue the GN_2 purge until the effluent dew point is -40°F.

 (6) During packaging, part shall have a continuous nitrogen purge. Package according to instructions in Section 20.

19.6.6 Passivating 300 series stainless steel.

19.6.6.1 Purpose. The purpose of this document is to establish a standard procedure for the passivation of 300 series stainless steel parts and assemblies.

19.6.6.2 Requirement. Stainless steel parts that have been pickled or have been wire brushed may be passivated.

19.6.6.3 Equipment.

(a) Tanks (1 or more) used should be PVC lined, glass lined, or stainless steel.

(b) Hooks, racks, etc., as required.

(c) Heaters, if used, should be quartz covered, PVC coated, or stainless steel.

19.6.6.4 Material.

(a) Prepare a solution of 30 ±5% (by weight) nitric acid (HNO_3) in water.

19.6.6.5 Pre-processing.

(a) Pickle followed by water rinse or mechanical clean followed by degreasing.

19.6.6.6 Passivation procedure.

(a) Immerse parts in a solution of 30 ±5% (by weight) nitric acid at a temperature of 150 ±10°F for a period of 30 minutes to one hour.

(b) Rinse parts with deionized water until effluent pH is the same as influent when checked with pH paper.

(c) Dry using gaseous nitrogen.

19.6.6.7 Quality assurance provisions.

(a) Titration of the solution will be accomplished weekly or every 30 hours of use, whichever is less frequent.

(b) Results should be recorded.

(c) Verify that part has been passivated according to this process standard.

19.6.6.8 <u>Solution analysis and control</u>. Nitric acid (passivating solution).

(a) Reagents

(1) 0.2N potassium hydroxide (KOH).

(2) Methyl purple indicator.

(b) Procedure

(1) Add 3.2 ml sample to a solution of 25 ml of water and 50 ml of alcohol in a 250 ml Erlenmeyer flask.

(2) Titrate resulting solution with 0.2N KOH to methyl purple end point. Record as Titration "A".

(c) Calculation of percentage of acid.

$$\% \ HNO_3 = \frac{A \times \text{Milliequivalent Wt. } HNO_3 \times \text{N of KOH} \times 100}{\text{Size of sample X specific gravity}}$$

$$= \frac{A \times .063 \times .2N \times 100}{3.2 \times 1.2}$$

19.6.6.9 <u>Verify</u>. Verify passivity as necessary by the following method.

(a) Deposit a drop of sulfuric acid and drop of 5% solution of copper sulfate on the surface to be tested. If copper deposit forms within 10 seconds, repassify.

19.6.6.10 <u>Safety precautions</u>. Because of the danger of handling HNO_3, extreme caution will be exercised at all times. Protective clothing will be worn at all times when acid is being mixed, when parts are being processed, or when parts are being inspected. Clothing will consist of the following:

(a) Plastic face shield.

(b) Rubber gloves.

(c) Rubber apron.

(d) Rubber boots.

A neutralizing bath of bicarbonate of soda and water (1 lb. per gallon) will be kept within 30 feet of the pickling operation.

19.6.7 Pickling stainless steel parts.

19.6.7.1 Purpose. The purpose of this document is to establish a standard procedure for the pickling of 300 series stainless steel parts and assemblies. 300 series stainless steel parts that are discolored, contain scale, or discoloration may be pickled by this procedure.

19.6.7.2 Equipment.

(a) Tanks (1 or more). Tanks used should be PVC lined, glass lined, or if the acid is to be discarded after a day's use, stainless steel.

(b) Hooks, racks, etc., as required.

(c) Heaters, if used, should be quartz covered, PVC coated, etc.

19.6.7.3 Materials.

(a) Prepare a solution of 20 ± 5% (by weight) nitric acid (HNO_3) in water.

(b) Prepare a solution of 2 ± 1/2% (by weight) hydrofluoric acid (HF) in water.

19.6.7.4 Pre-processing.

(a) Degrease.

(b) Mechanical clean to remove heavy scale, slag.

19.6.7.5 Procedure.

(a) Assure that the solution to be used is within the parameters of Materials, 19.6.7.3.

(b) Read safety precautions. (Paragraph 19.6.7.7)

(c) Pickle the part by one of the following methods using the guidelines of Table LVI.

  (1) Immerse. Completely cover the part by submerging in a container of pickle solution.

  (2) Drench all surfaces to be picled. All surfaces must remain wetted at all times for pickling to be uniform.

(3) Hoses or tubes may be flowed through or filled to be pickled. Flowing through a line increases the pickling process considerably. Lines should be checked every three minutes to assure that over pickling does not occur.

Table LVI

300 series stainless steel pickling process guidelines.

	Immerse	Spray	Flow
Time	30 to 45 minutes	45 to 60 minutes	15 to 30 minutes
Temperature	Ambient to 120°F	Ambient to 120°F	Ambient to 120°F
Inspection	10 to 15 minutes	10 to 15 minutes	3 minutes

(d) Flush part with water.

(e) Passivate part.

19.6.7.6 Quality assurance provisions.

(a) Titration of the solution will be accomplished weekly or every 30 hours of use, whichever is less frequent.

(b) Record results.

(c) Verify that part has been pickled according to this process standard.

19.6.7.7 Solution analysis and control.

(a) Nitric/hydrofluoric acid (pickle solution).

Reagents

(1) 1N potassium hydroxide (KOH).

(2) Silica gel ($SiO_2 \cdot xH_2O$).

(3) Methyl purple indicator.

(b) Procedure

(1) Add 5 ml sample to a solution of 25 ml of water and 50 ml of alcohol in a 250 ml Erlenmeyer flask.

(2) Titrate with a known solution of KOH (approximately 1N) to methyl purple end point. Record as Titration "A".

(3) Repeat Step 1. as listed on the previous page.

(4) Add approximately 1 gram of silica gel to solution.

(5) Titrate with a known solution of KOH (approximately 1N) to methyl purple end point. Record as Titration "B".

(c) Calculation of percentage of acid.

(1) Subtract Titration "B" from Titration "A" and record this difference as "C".

(2) $$\% \text{HNO}_3 = \frac{(\text{A-C}) \times \text{milliequivalent Wt HNO}_3 \times \text{N of KOH} \times 100}{\text{Size of sample} \times \text{specific gravity}}$$

$$= \frac{(\text{A-C}) \times .063 \times 1\text{N} \times 100}{5 \text{ ml.} \times 1.2}$$

(3) $$\%\text{HF} = \frac{\text{C} \times 1.5 \times \text{milliequivalent wt. HF} \times \text{N of KOH} \times 100}{\text{Size of sample} \times \text{specific gravity}}$$

$$= \frac{\text{C} \times 1.5 \times .020 \times 1\text{N} \times 100}{5 \text{ ml.} \times 1.2}$$

NOTE: Hydrofluoric acid should only be collected in a parafin lined or polyethylene container.

19.6.7.8 Safety precautions. Due to the danger of handling HF-HNO_3, extreme caution will be exercised at all times. Protective clothing will be worn at all times, when acid is being mixed, when parts are being processed or when parts are being inspected. Clothing will consist of the following:

(a) Plastic face shield.

(b) Rubber gloves.

(c) Rubber apron.

(d) Rubber boots.

A neutralizing bath of bicarbonate of soda and water (1 lb. per gal) will be kept within 30 feet of the pickling operation. The bath container will be painted bright green. In case of an accident, drench the affected area with this folution followed by generous amounts of water.

After handling acid or parts that have been pickled, wash all protective clothing with water.

19.6.8 Cleaning pressure sensing devices.

19.6.8.1 Purpose. To define a standard process of cleaning, sampling, and quality control procedures for pressure sensing devices. This procedure is to be used on all pressure sensing devices whether flow-through or capillary tube.

19.6.8.2 Equipment.

(a) Vacuum chamber or bell jar.

(b) Vacuum gage.

(c) Vacuum valve.

(d) Vent valve.

(e) Miscellaneous fittings.

(f) Clean sampling apparatus.

19.6.8.3 Material.

(a) Trichloroethylene, flushing grade.

(b) Trichlorotrifluoroethane, electronic grade.

19.6.8.4 Procedure.

(a) Flow-through bourdon tube.

(1) Remove the bleed valve or screw.

(2) Connect a trichloroethylene source to the part of the pressure sensing device.

(3) Flush the part at low pressure (below 20 psi) for 1 minute.

(4) Unhook the trichloroethylene flushing system and purge the part with low pressure GN_2 for 2 minutes.

(5) Connect a Trichlorotrifluoroethane source to the part that has passed a particle count of less than 10% of the specification requirements.

(6) Flow at low pressure (below 20 psi) for 30 seconds.

(7) Catch a 200 ml sample.

(8) Send sample to lab for analysis.

(9) Purge with low pressure GN_2 for 1 minute.

(b) Capillary bourdon tube.

(1) Place specimen in the cleaning apparatus as shown in Figure 19.

(2) Add enough trichloroethylene to the beaker to cover 3/4" (minimum) of the stem of the pressure sensing device.

(3) Draw a vacuum (24" Hg minimum) on the chamber for 3 - 5 minutes.

(4) Release the vacuum.

(5) Repeat the two preceding steps a minimum of three times.

(6) Discard trichloroethylene.

(7) Repeat Step 2 using trichlorotrifluoroethane filtered to 10% of specification requirements.

(8) Repeat Steps 3 and 4. This shall be the sample.

(9) Send sample to lab for analysis according to specification requirements.

19.6.8.5 <u>Quality assurance provisions</u>.

(a) Assure that the sequence of operations has been followed as specified herein.

(b) Verify that sampling media is 10% of the maximum specification requirements or less.

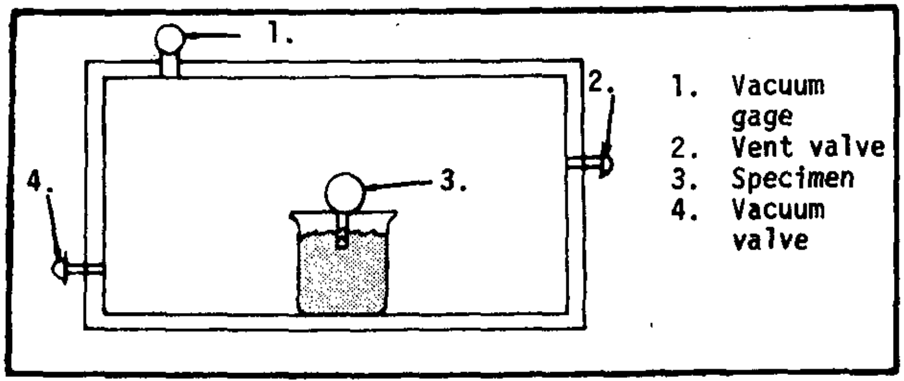

Figure 20. <u>Cleaning apparatus for pressure sensing devices</u>

19.6.9 Cleaning pneumatic systems metering valves.

19.6.9.1 Purpose. To establish the precision cleaning methods and requirements related to metering valves in pneumatic systems.

19.6.9.2 Materials.

(a) Nitrogen gas (GN_2), Mil-P-27401.

(b) Polyethylene film, Mil-P-3803.

(c) Trichlorotrifluoroethane.

(d) Trichloroethylene, technical, Fed-Spec-O-T-634.

(e) Nylon 6.

(f) Deionized water 50,000 OHM min. resistance.

(g) Detergent nonionic, Mil-D-16791-

(h) Tape, Fed-Spec-PPP-T-60.

(i) Isopropyl alcohol, Fed-Spec-TT-1-735.

(j) Nitric acid, Fed-Spec-O-N-350.

19.6.9.3 Procedure. The unit to be cleaned is a valve which is an assembly consisting of stainless steel and soft good components. Valve will be disassembled for cleaning purposes.

(a) All metallic parts will be cleaned as follows:

(1) Degrease using trichloroethylene solvent and nylon brush; scrub all accessible areas thoroughly for 2 to 3 minutes, or vapor degrease using trichloroethylene.

(2) Dry parts thoroughly by using GN_2, or air dry until all traces of trichloroethylene are removed.

(3) Clean using 1 to 2 oz/gal nonionic detergent/water and nylon brush. Scrub all accessible areas thoroughly. Parts may be ultrasonically cleaned if they are not damaged by the process.

(4) Rinse with deionized water until effluent indicates a pH between 6 to 8 when checked with pH paper.

(5) Parts may be flushed with isopropyl alcohol to facilitate drying.

(6) Blow dry with GN_2.

(7) Forward parts to final clean room.

(b) All nonmetallic parts will be cleaned in the controlled environment as follows:

(1) Clean using 1 to 2 oz/gal nonionic detergent/water and nylon brush, or ultrasonically clean.

(2) Rinse with deionized water for two to three minutes.

(3) Flush with isopropyl alcohol.

(4) Holding parts with clean forceps, pressure flush with 500 ml of pre-filtered isopropyl alcohol.

(5) Forward sample to analytical laboratory for particulate analysis.

19.6.9.4 Cleanliness tests.

(a) Test according to nonvolatile residue (NVR) tests. (See Section 16 for NVR test methods.)

(b) Reassemble valve in accordance with manufacturer's instructions.

(c) Package according to instructions outlined in Section 20.

19.6.10 Cleaning pneumatic systems pressure transducers.

19.6.10.1 Purpose. To establish the precision cleaning methods and requirements related to pressure transducers in pneumatic systems.

19.6.10.2 Materials.

(a) Nitrogen gas (GN_2), Mil-P-27401.

(b) Polyethylene film, Mil-P-3803.

(c) Tape pressure sensitive, Fed-Spec-PPP-T-60.

(d) Trichlorotrifluoroethane.

(e) Aluminum foil, heavy duty.

(f) Deionized water 50,000 OHM min. resistance.

(g) Detergent, nonionic, Mil-D-16791.

(h) Nylon 6.

19.6.10.3 Precleaning. The unit to be cleaned is a pressure transducer which is an assembly consisting of stainless steel and soft goods.

(a) Degrease using trichlorotrifluoroethane solvent by rinsing internal areas thoroughly for 2 to 3 minutes.

(b) Clean using 1 to 2 ounces/gallon nonionic detergent/water and nylon brush, where possible. If electrical connections allow, part may be ultrasonically cleaned.

(c) Rinse using deionized water until all traces of detergent are removed. The effluent water shall have a pH between 6 to 8.

(d) Dry using dry GN_2 or placing in a drying oven at 180 ± 10°F for 30 minutes or longer if required (determine visually).

19.6.10.4 Final cleaning.

(a) Forward parts to final clean room.

(b) Rinse all areas several times with trichlorotrifluoroethane filtered to 10 microns absolute.

(c) Proceed to cleanliness test for sampling.

(1) Test for particle count.

(2) Test according to nonvolatile residue (NVR) tests as defined in Section 16.

(d) Drying of the component shall be accomplished in a drying oven set at 299°F for 30 minutes or longer if required (determine visually.

(e) Package according to instructions outlined in Section 20.

19.6.11 Cleaning stainless steel fittings in pneumatic systems.

19.6.11.1 Purpose. To establish the precision cleaning methods and requirements related to stainless steel fittings in pneumatic systems.

19.6.11.2 Materials.

(a) Nitrogen gas (GN_2), Mil-P-27401.

(b) Polyethylene film, Mil-P-3803.

(c) Saran wrap, dow chemical corporation.

(d) Trichloroethylene solvent, 0-T-634.

(e) Nitric acid 42° Baume 0-A-88.

(f) Aluminum foil, heavy duty.

(g) Deionized water 50,000 OHM min. resistance.

(h) Detergent, nonionic , Mil-D-16791.

(i) Trichlorotrifluoroethane.

19.6.11.3 Precleaning. The units to be cleaned are fittings which are one piece assemblies fabricated of stainless steel.

(a) Degrease using trichloroethylene and nylon brush all external and internal areas thoroughly for 2 to 3 minutes or by trichloroethylene vapor degreasing.

(b) Blow dry with GN_2.

(c) Passivate using 30 ± 5% nitric acid (HNO_3) plus 2.0 ± 0.5% sodium dichromate and water. Immerse the part for 1 hour at room temperature (65° - 90°F).

  (1) Component may be descaled if needed to remove corrosion prior to passivation. Extreme care should be exercised in pickling fittings in order not to damage sealing surfaces or threaded areas.

(d) Rinse using tap water until outlet pH is between 6 and 8 when checked with pH paper.

(e) Clean using 1 to 2 oz/gal nonionic detergent/water and nylon brush; scrub all accessible areas thoroughly. Ultrasonic cleaning can also be employed.

(f) Flush with deionized water until effluent pH is between 6.0 to 8.0 when checked with pH paper.

(g) Blow dry with GN_2.

(h) Visually inspect and assure that parts are free of grease, scale, corrosion, and other contaminants prior to entering the clean room.

19.6.11.4 Final cleaning.

(a) Forward part to final clean room.

(b) Holding the part with clean forceps, rinse all areas several times with trichloroethylene or other suitable solvents filtered to 10 microns absolute.

(c) Proceed to cleanliness test for sampling.

  (1) Establish particulate count.

  (2) Perform nonvolatile residue (NVR) test as defined in Section 16.

(d) Drying of the component shall be accomplished in a drying oven set at 200°F for 30 minutes or longer if required (determine visually), or by purging with GN_2.

(e) Package according to instructions as outlined in Section 20.

19.6.12 Cleaning pneumatic pressure regulators.

19.6.12.1 Purpose. To establish the precision cleaning methods and requirements related to pneumatic pressure regulators.

19.6.12.2 Materials.

(a) Nitrogen gas (GN_2), Mil-P-27401.

(b) Polyethylene film, Mil-P-3803.

(c) Trichlorotrifluoroethane.

(d) Trichloroethylene, technical, Fed-Spec-O-T-634.

(e) Nylon 6.

(f) Deionized water 50,000 OHM, min. resistance.

(g) Detergent, nonionic , Mil-D-16791.

(h) Tape, Fed-Spec-PPP-T-60.

(i) Isopropyl alcohol, Fed-Spec-TT-I-735. I-735.

19.6.12.3 Cleaning. The unit to be cleaned is a pressure regulator which is an assembly consisting of gauges, soft goods and brass components. Regulator will be disassembled for cleaning purposes.

(a) All metallic parts will be cleaned as follows:

(1) Degrease using trichloroethylene solvents and nylon brush; scrub all accessible areas thoroughly for 2 to 3 minutes, or vapor degrease using trichloroethylene.

(2) Blow dry with GN_2.

(3) Clean using 1 to 2 ounces/gallon nonionic detergent/water and nylon brush; scrub all accessible areas thoroughly. Ultrasonic cleaning can also be employed.

(4) Flush with deionized water until effluent indicates a pH between 6 and 8 when checked with pH paper.

(5) Blow dry with GN_2.

(6) Visually inspect and assure that parts are free of grease, scale, corrosion, and other contaminants prior to entering the clean room.

(7) Forward parts to final clean room.

(8) Holding the parts with clean forceps, rinse all areas several times with trichlorotrifluoroethane filtered to 10 microns absolute.

(9) Proceed to cleanliness test for sampling.

(b) All nonmetallic parts will be cleaned as follows:

(1) Clean using 1 to 2 oz /gal nonionic detergent/water and nylon brush ; scrub all accessible areas thoroughly. Rinse with deionized water. Ultrasonic cleaning process may be used.

(2) Flush with isopropyl alcohol.

(3) Holding parts with clean forceps, pressure flush with 500 ml. of pre-filtered isopropyl alcohol.

(4) Forward sample to analytical laboratory for particulate analysis.

(5) Dry parts by purging with GN_2.

19.6.12.3.1 Method "A", cleaning of gauges.

(a) Insert polyethylene tubing into extreme end of bourdon tube with other end of tubing connecting to trichlorotrifuoroethane pressure source.

(b) Flush with trichlorotrifluoroethane (freon).

(c) Sample.

(d) Dry by connecting GN_2 to polyethylene tubing, purge for 2-3 minutes.

19.6.12.3.2 Method "B", cleaning of gauges. This process applies to gauges which cannot be cleaned per Method "A".

(a) Refer to Figure 20 for test set-up.

(b) Connect gauge to test set-up.

(c) Apply vacuum (20 inches of mercury, minimum) to vacuum flask thus evacuating air from bourdon tube on gauge.

(d) Return flask to ambient pressure thus pulling solvent (trichlorotrifluoroethane) up into bourdon tube on gauge.

(e) Repeat Step (c) to remove solvent from bourdon tube.

(f) Cycle vacuum flask five times by repeating Steps (c) through (e).

(g) For cleanliness test, replace trichlorotrifluoroethane in vacuum flask with trichloroethylene which has been filtered through a .45 membrane filter.

(h) Cycle vacuum flask two times by repeating Steps (c) through (e).

(i) Remove trichloethylene from flask and check.

(j) Dry by removing trichlorotrifluoroethane and cycling vacuum flask by repeating Steps (c) through (e).

19.6.12.4 Cleanliness tests. Test according to nonvolatile residue (NVR) tests as defined in Section 16.

19.6.12.5 Drying and reassembly. Drying of the components shall be accomplished in a drying oven set at 180° ± 10°F for 30 minutes or longer if required (determine visually).

Regulator shall be reassembled and functional tested to assure that no damage has occurred during cleaning.

19.6.12.6 Packaging. Package according to instructions outlined in Section 20.

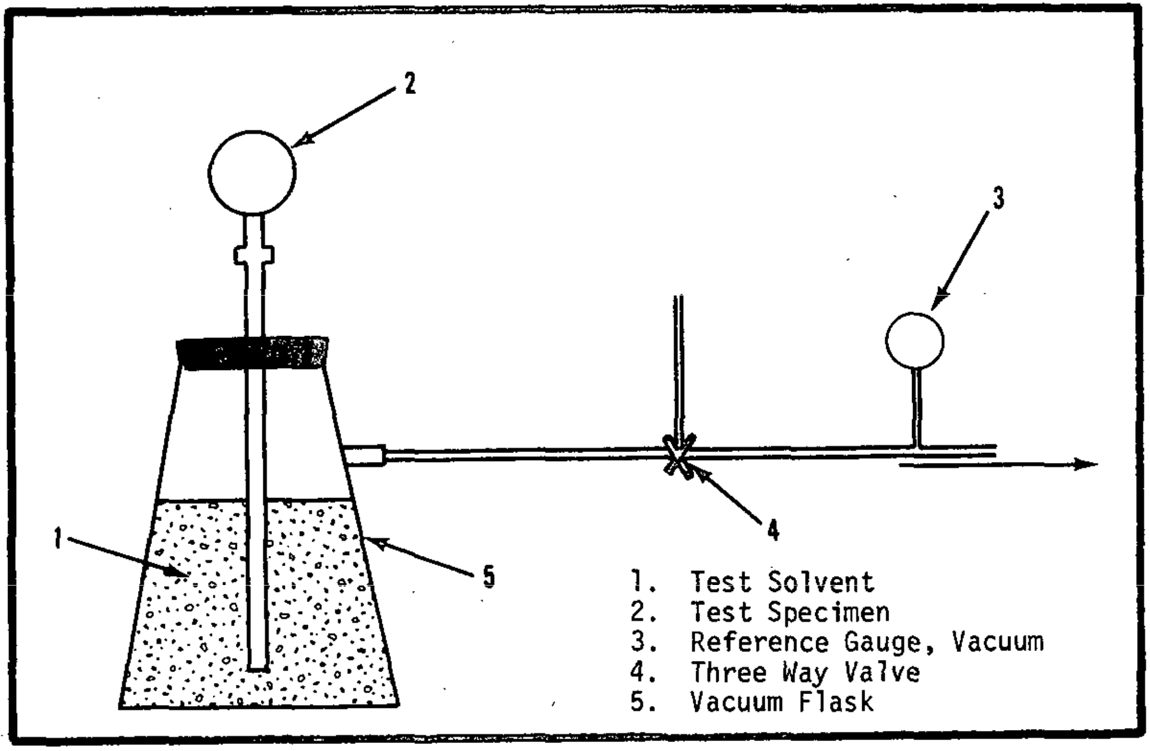

Figure 21. Test set-up for cleaning pneumatic pressure gauges

19.6.13 Cleaning cap, tube fitting, and chain used in pneumatic systems.

19.6.13.1 Purpose. To establish the precision cleaning methods and requirements related to cleaning of cap, tube fitting, and chain as used in pneumatic systems.

19.6.13.2 Materials.

(a) Nitrogen gas (GN_2), Mil-P-27401.

(b) Polyethylene film, Mil-P-3803.

(c) Trichlorotrifluoroethane.

(d) Trichlorethylene, technical, Fed-Spec-O-T-634.

(e) Nylon 6.

(f) Deionized water 50,000 OHM min. resistance.

(g) Detergent, Mil-D-16791.

(h) Tape, Fed-Spec-PPP-T-60.

19.6.13.3 Precleaning. The unit to be cleaned is a fitting which is an assembly consisting of stainless steel.

(a) Degrease using trichlorethylene solvents and nylon brush; scrub all accessible areas thouroughly for 2 to 3 minutes, or vapor degrease.

(b) Clean using 1 to 2 oz/gal nonionic detergent/water and nylon brush; scrub all accessible areas thoroughly or ultrasonically clean.

(c) Flush with deionized water until effluent indicates a pH between 6 and 8 when checked with pH paper.

(d) Blow dry with GN_2.

(e) Visually inspect and assure that parts are free of grease, scale, corrosion, and other contaminants prior to entering the clean room.

19.6.13.4 Final cleaning.

(a) Forward parts to final clean room.

(b) Holding the parts with clean forceps, rinse all areas several times with solvent filtered to 10 microns absolute.

(c) Proceed to cleanliness test for sampling.

19.6.13.5 Cleanliness tests. Perform nonvolatile residue (NVR) test as defined in Section 16.

19.6.13.6 Drying. Drying of the components shall be accomplished in a drying oven set at 180° ± 10°F for 30 minutes or longer if required (determine visually), or by purging with GN_2.

19.6.13.7 Packaging. Package according to instructions outlined in Section 20.

19.6.14 Cleaning nonmetallic materials.

19.6.14.1 Purpose. To establish the precision cleaning methods and requirements related to nonmetallic materials including natural rubber, teflon, nylon, Kel-F, polyethylene, and any other plastic or synthetic rubber materials.

Caution. Determine if the material to be cleaned is adversely affected by the cleaning solutions.

19.6.14.2 Preprocessing procedure.

(a) Decontaminate using cold tap water; flush until pH of effluent is within one-half pH unit of influent.

(b) Detergent clean with a suitable general purpose detergent cleaner using nylon brushes as necessary.

(c) Spray rinse with hot (150° - 180°F) tap water followed by rinse with high purity water.

(d) Dry with hot (120°F max.) nitrogen gas.

19.6.14.3 Final cleaning procedure. Spray rinse with compatible cleaning agent for one-half minute maximum.

19.7 References.

19.7.1 Government.

19.7.1.1 Federal.

1. Acetone, Reagent Grade, O-A-51.

2. Acid, Hydrochloric (Muriatic), Technical Grade, O-A-86.

3. Acid, Nitric, Technical Grade, O-A-88.

4. Cleaning Compound Alkali Type, P-C-436.
5. Ethyl Alcohol (Ethanol); Denatured Alcohol; and Proprietary Solvent, O-E-760.
6. Hydrofluoric Acid, Technical, O-H-795.
7. Nitrogen, Technical, BB-N-411.
8. Orthophosphoric (Phosphoric) Acid, Technical, O-O-670.
9. Petroleum Ether, O-E-751.
10. Phosphoric Acid, Technical Grade, O-P-313.
11. Sodium Dichromate, Technical Grade, O-S-595.
12. Sodium Phosphate, Tribasic, Technical Anhydrous, Dodecahydrate, and Monohydrate, O-S-642.
13. Tape; Pressure Sensitive Adhesive, Waterproof for Packaging and Sealing, PPP-T-60.
14. Trichlorethane, O-T-620.
15. Trichloroethylene, Technical, O-T-634.

19.7.1.2 John F. Kennedy.

1. Cleanliness Levels, Cleaning Protection, and Inspection Procedures for Parts, Field Parts, Assemblies, Subsystems, and Systems for Fluid use in Support Equipment, KSC-SPEC-KSC-C-123 (D).

19.7.1.3 George C. Marshall Space Flight Center.

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2. Cleaning and Passivation of Bellows and Ducts, Drawing #10419901.
3. Cleanliness Levels, Cleaning and Inspection Procedures for Components Parts of Gas Bearing and Slosh Measuring Systems, Drawing #10419906.
4. Cleanliness of Components for use in Oxygen, Fuel, and Pneumatic Systems, MSFC-SPEC-164.

5. Cleanliness Levels, Cleaning, Protection, and Inspection Procedures for Parts, Field Parts, Assemblies, Subsystems, and Systems for Pneumatic Use in Support Equipment, Drawing #10M01671.

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7. Hydraulic System Components and Hydraulic Fluids for Space Vehicles, Cleaning, Testing and Handling, MSFC-PROC-166B.

8. Nitrogen, Space Vehicle Grade, MSFC-SPEC-234.

9. Polytetrafluoroethylene (Teflon) Molded and Extruded Parts, Sheets, Rods, and Tubing, MSFC-SPEC-236.

10. Procedure for Cleaning, Testing, and Handling of Onboard Hydraulic System Components and MIL-H-5606 Hydraulic Fluid, Drawing #20M85015.

11. Solvent, "Freon", Precision Cleaning Agent, MSFC-SPEC-237A.

12. Trichloroethylene, Technical Specification For, MSFC-SPEC-217.

19.7.1.4 Military.

1. Aluminum Foil, Mil-A-148.

2. Compound, Cleaning, Washing Machine (For Aircraft Metal Parts), Mil-C-5543.

3. Detergent, Nonionic, Mil-D-16791.

4. Isopropyl Alcohol, Grade A, Mil-A-10428.

5. Nitric Acid, Mil-C-110298.

6. Propellant Pressurizing Agent, Nitrogen, Mil-P-27401.

7. Sodium Nitrite, Mil-S-322.

8. Trichloroethylene, Oxygen Propellant Compatible (by flushing methods), Mil-T-27602.

19.7.1.5 Ordnance Corps. U.S. Army Missile Command.

1. Cleanliness of Components for Use in Liquid Oxygen, Fuel, and Pneumatic Systems, Drawing #10509305.

19.7.2 Nongovernmental.

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SECTION 20

CLEAN PACKAGING

20.1 Introduction. The advent of precision, microminiature, and high reliability components provided the impetus for developing clean packaging materials and techniques. Concurrently, the other essential ingredient for clean packaging, clean working environments, was developed.

20.2 Clean packaging defined. Packaging in general constitutes enclosing an item in some material to protect it from some environmental condition. Clean packaging is more definitive in that it protects critical areas from specific contaminants or contaminating environments and requires individual analysis of each packaging situation. It may involve sealing ports or openings into a specific area or enclosing an entire assembly. It may include items ranging from microminiature parts and components to complete assemblies and systems.

20.3 Theory of clean packaging. The theory of clean packaging is simple: the package must be capable of maintaining the cleanliness requirements of the packaged item, and this condition must be maintained throughout handling, storage, and transportation until further assembly or use. In order to achieve this end, the following conditions must exist:

(a) The packaging material must be cleanable and precision-cleaned prior to use.

(b) The cleanliness level of the cleaned packaging material must be maintained from the point of cleaning until a closure is effected.

(c) The packaging operation must be accomplished in a clean environment.

(d) Stringent clean packaging techniques must be observed.

(e) The package must be constructed and sealed so as to prevent the entrance of contaminants from outside the package.

(f) The packaging material itself must not contribute contamination, except within controlled and acceptable limits.

In many fields of endeavor, a great emphasis is placed on achieving a specified cleanliness level. Unless this cleanliness level is maintained, usually through clean packaging, the clean effort is nullified.

20.4 Preparation for clean packaging. Many factors in addition to packaging materials and methods must be considered if the desired result is to be achieved. If clean packaging requirements are critical, it is preferable that a specification be prepared for each item or entity to be so packaged. However, due to the large number of parts or assemblies involved in some programs, it may be more practical to establish some system of classes or methods into which most of the items can be conveniently categorized. The determination of the type of grouping adopted will depend on conditions such as the following:

(a) The degree of cleanliness required.

(b) The material composition of the item being packaged.

(c) The configuration of the item being packaged.

(d) The intended use of the item, including other materials with which it will be in contact, criticality of the operation.

(e) The extent and type of handling, storage, or transport anticipated prior to use.

These and other points are discussed in the following paragraphs.

20.4.1 Determination of cleanliness levels. In determining the cleanliness level to be maintained by clean packaging, primary consideration should be given to the item or class into which the item belongs and its functional cleanliness requirements. Other considerations are the availability of environmentally controlled areas and the degree to which the packaging materials are cleanable, resistant to sloughing of particles, and capable of excluding adverse environmental conditions.

The cleanliness level requirement, along with the degree of handling and transport involved, may also have a distinct bearing on the type and amount of cushioning and packing materials used to protect the contents of the clean package.

It is common practice to establish cleanliness levels for an entire functional system when extensive and complex projects are involved. Each contractor contributing to such a project often establishes his own standards of cleanliness to meet the system requirements. This method of operation, though usually satisfactory, is less preferable than a full coordinated establishment of cleanliness levels among the various contractors. The latter method would probably reduce conflicts, recleaning and repackaging, and additional costs in subsequent assembly, inspection, and testing operations.

20.4.2 Preparing the item to be packaged. It should be sufficient to point out here that any item to be clean-packaged must be properly cleaned and dried prior to packaging, and that its clean condition must be maintained until a clean packaging closure or enclosure is effected.

Particular attention should be given to the removal of moisture and cleaning agents from the components prior to clean packaging. This can usually be accomplished by the application of heat, vacuum, a dry purge gas, or some suitable combination of these methods.

20.4.3 Selecting or preparing the packaging material. The availability of packaging materials which can meet the requirements for clean packaging is limited by three conditions: (1) the material must be cleanable to the degree needed, (2) the material must exhibit a low particle sloughing tendency, and (3) it must possess the capability for effecting a hermetic seal in a manner that will not contribute to contamination of the package.

Packaging materials which have found wide application are plastic films such as polyethylene, polyamide (nylon), and fluorohalocarbon (aclar). These films may not possess all of the required characteristics to the degree desired; however, they are adequate for many applications. The attributes of these films for clean packaging are generally considered to be:

(a) Polethylene. Moisture vapor barrier; medium sloughing.

(b) Nylon. Minimal sloughing; flex-resistant.

(c) Aclar. Moisture vapor barrier; particles are LOX-compatible.

Packaging films are not manufactured in a clean condition, but clean films are available from several convertors and clean produce suppliers throughout the country. Clean film can be procured in various forms such as roll stock, bags, shrouds, and tubing.

Small sections of film, including bags, may be effectively cleaned by the user who has adequate cleaning and drying equipment. Precision grade trichlorotrifluoroethane is the cleaning agent normally used for film cleaning. Cleaning film on a large scale, however, requires rather elabrate, mechanized cleaning equipment and a clean room in which further packaging of the clean film is accomplished. In no case can the clean film be exposed to ambient factory environmental conditions after it leaves the cleaning equipment. Therefore, the cleaning equipment must be located in the clean room, which in most cases is not advisable, or the clean film must issue directly from the cleaning equipment into the clean room through an appropriately protected pass-through. The type of installation described above would be economically feasible only for the very large user or commercial convertor of clean film.

20.4.4 Packaging facilities, equipment, and personnel. In order to accomplish its objectives, all clean packaging operations must be carried on in an environmentally controlled area which provides an adequate degree of protection for both the item and the clean packaging material. The degree of protection needed should be consistent with the product cleanliness requirements, and may range from a Class 100 (Fed. Std. 209a) clean room to a nonlaminar airflow clean room with some lesser degree of particulate and environmental control. Any preparation of clean packaging material, such as, unpacking from original container, cutting, trimming, initial heat sealing, should be done in the exhaust or downstream portion of the clean room. Items connected with these operations should be brought into the immediate vicinity of the clean parts only during actual use.

All necessary equipment and supplies for clean packaging must be as clean as possible, in good operating condition, and readily accessible for use. All cutting devices must be well maintained in a sharp condition to minimize particle generation and ragged material edges.

As in any contamination control activity, people are a most important ingredient to successful clean packaging. Anyone being introduced to a clean packaging operation should receive, as a minimum, some degree of formal and on-the-job training in the following essential aspects of his job:

(a) The philosophy of contamination control.

(b) The specific need for clean packaging.

(c) The proper operation of equipment and use of materials.

(d) The specification requirements of the product.

(e) The regulations governing attire and conduct in an environmentally controlled area.

Personnel should be selected for this function on the basis of both their mechanical aptitude and their understanding and willingness to conform to established practices and regulations.

The proper utilization of personnel in clean packaging, however, is not solely dependent on training. Competent supervision is equally important.

20.5 Packaging materials. The adequacy of any material to protect an item from being contaminated is dependent on the cleanliness level that must be maintained. This can cover an extremely wide range of levels. To better relate material performance criteria to required cleanliness levels, the principal qualities that a material should possess are categorized as follows:

(a) Barrier to external environments. A good barrier should be capable of excluding those contaminants in the external environments which would cause contamination of the packaged item.

(b) Minimum contributor of contamination. An ideal material would not in itself contaminate the packaged item or adjacent environment by release of particles from sloughing or contact-abrasion or from emission of vapors.

(c) Compatible for use in hazardous environments. A material which would not contribute to an explosive, flammable, corrosive condition when exposed to environments containing concentrations of oxygen and explosive mixtures, volatile vapors, hypergolic propellants, and other flammable substances.

An evaluation of any group of materials to meet the requirements for a specific application of clean packaging may reveal that no one material possesses all of the required qualities and characteristics. This is especially true as the required cleanliness level becomes more stringent.

The preservation of a precision-cleaned item by a packaging technique may require materials and methods which are currently not available, unknown or untried. Situations of this nature call on the ingenuity of the personnel involved to develop the techniques that will enable them to meet the requirements.

The following paragraphs discuss the types and characteristics of the packaging materials currently available and commonly used, with current information on significant limitations and qualities for clean packaging.

20.5.1 Barrier materials. Materials used in the clean package as a barrier to contaminating environments may be categorized in several ways. For simplicity and practicality, these materials are grouped according to structural type and package application.

20.5.1.1 Structural type. These groups consist of the following:

(a) Rigid. This group includes metals, glass, ceramic, and rigid plastics which are cleanable to the degree required, provide adequate environmental protection, and are minimal contributors of contaminants. They may be used as intimate and environmental packages.

(b) Nonrigid or flexible. This group consists principally of metal foil and plastic films which are cleanable to some degree. The barrier qualities will vary with different materials. The sloughing of particles due to abrasion and flexing is prominant in all materials of this type, especially in those of lower density. These may be used as intimate and environmental packages and as intimate cushioning material.

20.5.1.2 Package application. The three basic elements or parts which make up a complete clean package are as follows:

(a) Intimate package. The material used for this part of the clean package requires the same degree of cleanliness as the clean item. The material may contact the clean surfaces or is environmentally adjacent to those surfaces. It should be capable of providing a hermetic seal to external contaminants and environments.

The rigid materials of high density and hard finish can provide the highest degree of cleanliness and effective barrier qualities. Some impediments to the use of rigid materials may be in forming to a particular configuration, sealing, and securing the item within the enclosure to prevent damage to the item and container and still maintain the required cleanliness.

Nonrigid or flexible materials are limited in the degree of cleanliness they can maintain due to their sloughing characteristics. Metal foils are cleanable to a good degree, but flexing and abrasion will create considerable particle generation. Plastic films of high density and smooth surface finish are readily cleanable, and they generate fewer particles than the low-density films. Many of the plastic films are heat-sealable and are effective barriers to certain environments.

(b) Environmental package. This part of the clean package is used to provide an additional barrier to specific environments which may not be provided by the intimate package material, and to provide the intimate package with protection from the various environments encountered during handling operations and storage.

The sloughing characteristics of flexible film material used for this package may be of less significance, and emphasis can be placed on those characteristics that provide the best environmental barrier. Many of the plastic films are effective barriers. The rigid materials possess all the barrier qualities of the films and, in addition, provide greater physical protection from forces both within and outside the container.

(c) Intimate cushioning. Clean items which have sharp edges, protrusions, or external threaded portions may require some form of cushioning to protect the intimate and environmental package from puncture or damage. The material used for this part of the package requires the same degree of cleanliness as the item because of its intimate contact with the item. Where a plastic film is used as the intimate package, the same material is commonly used for cushioning by placing a number of layers over the areas required.

20.5.2 Ancillary materials and accessories for clean packaging. In addition to the basic materials for closures, enclosures, and cushioning of a clean package, certain other materials are necessary to form a complete clean package. Most of these items and material will be used in the clean packaging operation either internal to the package or adjacent to it. Therefore, the cleanliness of the items and the handling procedures must be compatible with the cleanliness level of the clean packaging operation. The following items are commonly used in clean packaging operations; however, special situations may require additional items which should be given the same considerations for cleanliness.

20.5.2.1 Tape, adhesive-backed. May be used in some instances for securing cushioning material to the item, sealing closures or wraps, and securing other items to the package. The tape material and the adhesive should not generate contaminants or degrade the package material.

20.5.2.2 Ribbons and ties (nonadhesive). May be used for the same purposes as the adhesive-backed tape and may be preferred to eliminate the possibility of contamination from the adhesive. Material of this type should be cleanable and nonshedding to the degree required.

20.5.2.3 Identification labels and tags. Normally required for proper and complete identification of the clean package and item. They should be preprinted to preclude the need for marking materials in the clean packaging area. Labels, tags, and markings should be of materials which are nonfading and minimal in generation of contaminants.

20.5.2.4 Desiccants and humidity indicators. When static dehumidification is required in the clean package, desiccants may be used with provisions to ensure that the clean item is not contaminated by desiccant dust or the desiccant bag material. Desiccants contained in bags, which are impermeable to desiccant dust and noncorrosive are available. Similar measures should be taken to protect the clean item from contamination by humidity indicators, when used.

20.5.2.5 Purging gases. Inert gases, such as dry nitrogen used for purging the clean package prior to sealing the closure, shall be of a purity equal to or better than the cleanliness level of the item. The impurities to be considered include water vapor, hydrocarbons, and particles that would contaminate the clean item. Filtration of the gas shall be adequate to ensure removal of all contaminating particulates.

20.5.2.6 Integrity seals. Tamperproof-type seals or decals may be used to detect violation of the integrity of a sealed clean package or closure. These items shall be of such material and configuration that their application does not result in deterioration or violation of the closure.

20.5.2.7 Intermediate package materials. Items of a fragile nature should be provided physical protection after being clean-packaged and prior to extensive handling. Materials used for this purpose include cushioning, compartmentalized containers, environmental barrier covers or wraps. Materials of this type should be applied outside the clean packaging area.

20.6 Characteristics and properties. Packaging materials have certain characteristics and properties which are significant to clean packaging. The characteristics as related to the requirements of clean packaging are described in the following paragraphs.

20.6.1 Strength. Should be adequately resistant to penetration by tearing, parting or piercing from forces either external or internal during normal handling operations. Rigid materials provide the greatest strength. The flexible foils and films are more susceptible to damage.

20.6.2 Permeability. Should have a low degree of permeability to the gases, vapors, and liquids which may be encountered. Some rigid materials and high density plastic films are essentially impermeable and have a low water-vapor-transmission-rate (W-V-T-R).

20.6.3 Formability. The capability of forming to a desired shape or configuration. The flexible foils and films are the most easily formed. The rigid materials require more extensive performing or molding operations.

20.6.4 Resistance to oil and grease. The resistivity to reaction when in contact with oils, grease, and other petroleum products which result in deterioration and penetration of the material. Metals and metal foils may be the most resistive with some of the plastic films having a high degree of resistance.

20.6.5 Temperature range. The range of temperatures within which the material does not deteriorate or degrade as a barrier. Includes the tendency of some films to block or stick together or stick to the packaged item. Metals and metal foil provide the greatest usable temperature range. The low density plastic films block at relatively low temperatures, which may be a limiting factor for some applications.

20.6.6. Sealability. The capability of sealing the material to itself or other material to form a hermetic closure. The method of sealing should not contribute contamination to the interior of the closure. Rigid materials and metal foils may require compressive or adhesive materials, which may not be applicable in some instances. Heat sealing is the simplest and most common method of joining plastic films. The major techniques commonly used are thermal, dielectric, and ultrasonic.

20.6.7 Transparency. A transparent material may be desirable in some instances to facilitate identification and inspection of the packaged item. Other situations may require the exclusion of light necessitating the use of opaque materials. Many of the plastic films are naturally highly transparent although others are pigmented to aid in their identification.

The metals and metal foils are the most opaque.

20.6.8 Cleanability. The material should be capable of being cleaned to the same level of cleanliness specified for the item being packaged. Rigid materials with smooth surface finishes can provide the highest degree of cleanliness. Metal foils and high-density plastic films with hard and smooth surface finishes may achieve a good degree of cleanliness when properly cleaned.

20.6.9 Sloughing or shedding. All materials have a tendency to slough off particles to some degree when subjected to certain conditions. Rigid materials with a hard, smooth surface finish are less susceptible to sloughing. Flexible materials will slough particles to variable degrees when subjected to motion, vibration, flexing, or abrasion. The size and quantity of particles generated depend on the type of material, surface finish, and the amount of activity seen by the material. Flexible materials are therefore limited in their use to applications where the particles generated do not exceed the limits specified.

20.6.10 Static charge. The electrostatic charge formed and retained on the surface of a material whenever two objects contact one another and then are separated results in two undesirable conditions: (a) the charged surface attracts contaminating particles from the adjacent environment, and (b) the high-energy discharge is capable of igniting electroexplosive devices and flammable materials and causes interference in the operation of sensitive electronic components.

The amount of charge that a material may acquire is dependent on the surface areas involved and in the types of materials as related to their position in the triboelectric series. A triboelectric series chart of some common materials is shown in Table LVII. The material uppermost in the series becomes positively charged when contacted or rubbed by a material lower in the series, which becomes negatively charged. Clean packaging materials should possess adequate anti-static properties particularly when they are used with explosive devices.

Table LVII.

Triboelectric series of common materials

Positive (+) Charge

Asbestos
Glass
Mica
Wool
Cat fur
Nylon
Viscose
Rayon
Lead
Silk
Fiberglass
Aluminum
Paper
Cotton
Wood, iron
Chrome
Acetate
Lucite
Dacron
Vinyl (PVC)
Polyethylene
Teflon
Nickel, copper, silver
Brass, stainless steel
Sulfur
Platinum, mercury
India rubber

Negative (-) Charge

Effective antistatic properties can be incorporated in fabrics and plastic films by adding antistatic agents. Two techniques commonly used for adding the antistatic agents are: (a) coating the material or (b) incorporating an internal additive during the manufacturing process.

Films and fabrics coated with an antistatic agent are not considered acceptable for clean packaging due to a tendency of the antistatic particles to slough. In some instances, the antistatic properties of the coating agent are degraded considerably when exposed to certain environments.

Plastic films with an antistatic agent as an internal additive are considered satisfactory for clean packaging materials. Care must be exercised in the film formulation with an antistatic agent to prevent sacrificing other characteristics or properties of the film. The retention of antistatic properties for an extended period of time will vary with the different plastics, the agent used, and the environments to which it is exposed. Antistatic polyethylene and nylon films show some loss of antistatic properties when exposed to strong fluorescent lighting and exposure to ultraviolet light. Direct sunlight accelerates this loss considerably.

20.6.11 Flammability. The degrees of flammability may be expressed as flammable or burning, self-extinguishing or slow burning, and nonflammable or nonburning. Clean packaging materials should be either nonflammable or self-extinguishing to assure integrity of the package and reduce hazardous conditions.

Flame retardancy of plastic films, which do not normally possess the desired degree of this characteristic, may be achieved by adding flame-retardant chemicals to the formulation of the plastic. These additives are generally classified into two types: (a) additives which are blended physically with the material, and (b) modifiers which are reactive and unite chemically with the plastic. The additive types may be used in many different plastics, while the reactives are usually tailored for a specific plastic. Combining flame-retardant additives with the various plastics may affect other characteristics such as color, flexibility, tensile strength, and softening point. Therefore, careful formulation is essential to prevent degradation of other essential characteristics.

20.6.12 Interaction with hazardous materials. The effect that the packaging material may have when in contact with certain hazardous materials is a required consideration. The resulting interaction may be evident by temperature increases, ignition, combustion, dissolution.

The materials which present probable hazardous interactions include: hypergolic propellants (such as unsymmetrical dimethylhydrazine, UDMH; monomethylhydrazine, MMH; nitrogen tetroxide, N_2O_4; liquid oxygen, LOX; gaseous oxygen, GOX; chemicals; volatile liquids; and other explosive and highly flammable materials.

20.7 Evaluation of plastic films. Evaluating materials for the qualities essential to clean packaging reveals that such materials readily available and economically practicable are somewhat limited.

The plastic films being more economical are widely used; however, those which possess the qualities essential to intimate closures for precision-cleaned items are quite limited. The plastic films which are cleanable to a reasonable degree and limited in sloughing are currently the most commonly used.

Films used for clean packaging purposes are available from the producers normally as only commercial clean which is inadequate for this application. Precision-cleaned films are available which can meet the cleanliness requirements for many clean packaging applications. The levels of cleanliness as specified by the supplier of such films should be checked to assure conformance with the requirements of the specific application. Some of the films which possess the necessary qualities for clean packaging are described in the following paragraphs:

20.7.1 Polyethylene. This film is widely used because it is available economically in many types, sizes, and thicknesses. Its disadvantages are a lower strength than most films and a higher degree of sloughing. It distorts readily when in contact with oils.

(a) Polyethylene, ordinary, clean. Low to medium density polyethylene film, natural color, 6-mil thickness, without talc or starch slip agents. (Antistatic and fluorescent types are available, and colored for identification.)

(b) Polyethylene, antistatic, clean. Pink nonfluorescent polyethylene film, 6 mils thick, containing an internal organic antistatic additive throughout the film which renders all surfaces sufficiently conductive to bleed off static charges and eliminates particle attraction or spark-discharge hazard due to static electricity. Films surface treated or sprayed with antistatic compounds are not acceptable, since such coatings rub off or interfere with heat sealing.

(c) Polyethylene, daylight fluorescent, clean. Bright fluorescent pink polyethylene film, 6 mils thick, containing a pigment which fluoresces orange under black light, for ease in identifying those particles sloughed from the film surface of a bag or wrap as opposed to those from other sources.

20.7.2 Polyamide (nylon 6). This film is high in abrasion and flex resistance, and thus creates the least self-contamination problems among films in common use. A limitation of Nylon 6 is that it does not provide a good moisture barrier. This characteristic can be used to advantage when a desiccant is used between the intimate and environmental packages. Because it is a poor barrier to moisture vapor, however, a nylon intimate package normally requires an environmental package such as polyethylene.

(a) Nylon, ordinary, clean. Nylon 6 (or blends of nylon 6 and nylon 6, 6) polyamide films, natural color, 2-mil thickness, without talc or starch slip agents. Nylon film is most resistant to sloughing of particles due to flex and abrasion of all films in clean packaging use, and is therefore preferred as the intimate package in contact with all clean surfaces or items except those parts destined for LOX or GOX service.

(b) Nylon, antistatic, clean, heat stabilized. Orange fluorescent transparent nylon film, 2 mils thick, containing an organic antistatic agent throughout the film to eliminate static charging (see Polyethylene, antistatic, clean), plus a heat stabilizer enabling the film to withstand dry heat sterilization cycles of 300°F for 12-hour periods and up without marked degradation. Ordinary nylon browns and embrittles in short periods under this temperature.

(c) Nylon, daylight fluorescent. clean. Yellow-green fluorescent nylon film, 2 mils thick, containing a pigment which fluoresces blue-white under black light, to aid in identifying those particles sloughed from the intimate package surface as opposed to those from other sources.

20.7.3 Fluorohalocarbon (aclar). Aclar and teflon are compatible with LOX. Aclar 33C, 22A, and 22C meet the requirements of MSFC-Spec-456 for packaging all pneumatic components requiring LOX-compatibility, but

Aclar 33C is generally used because of superior cleanability. Aclar has the disadvantage that it abrades more readily than nylon and, therefore, has more tendency toward self-contamination. However, sloughed particles of Aclar are LOX-compatible while particles from other films and foils can constitute a potential explosion hazard under impact in LOX service.

(a) Aclar, clean. Fluorohalocarbon copolymer, largely chlorotrifluoroethylene copolymerized with small quantities of other fluorocarbons, unplasticized and without additives. It is clear and colorless, 2 mils thick (although other gages such as 5 mils are available and very occasionally called out), and is available in three grades: Aclar 22A, 22C, and 33C. All grades are certified LOX-compatible under impact test (MSFC-Spec-106A), but Aclar 33 is preferred for packaging parts designed for LOX service due to its superior resistance to cleaning solvents.

20.7.4 Polyester (mylar). This film has excellent resistance to abrasion and to grease and oils, and relatively low permeability of gases and water vapor. Particle sloughing resulting from flexing is somewhat higher than nylon, and sealing by normal heat-sealing methods is difficult to achieve. This film may be preferred in some applications provided the required cleanliness level is attainable and an effective seal of the package can be achieved.

20.7.5 Aluminum foil. Although this is not a plastic film it is sometimes considered for use with the plastic films for clean packaging applications. Household grade aluminum foil has an oil film on the surface that is not acceptable for clean packaging or for LOX or GOX use. Clean oil-free foils are available which can pass the LOX impact test as a sheet sample, but the high rate of particle sloughing from abrasion and flexing produces aluminum particles which are not compatible with LOX under impact. It is not heat-sealable, it is opaque, the surface oxides may be abrasive to precision finishes, and contact with dissimilar metals can contribute to corrosive action. Aluminum foil as an intimate packaging material may find very limited application. However, as an environmental package, or intermediate package, its high resistance to grease, oils, acid, heat, nonflammability, and limited permeability of gases and vapor may make it desirable in some applications. Where an opaque material is required, aluminum foil or an aluminized surface on a plastic film may fulfill this requirement.

20.7.6 Composite films. The structuring of two or more different types of films to form a composite film has the advantage of combining in one film the characteristics of two or more individual films to provide the needs for a particular application. Two methods of structuring are utilized for composite films: the mechanical lamination and the extruded or formed composites.

(a) Mechanical lamination consists of sandwiching together the different types of films with special adhesives, by thermal lamination, or extrusion coating one film on another.

(b) The extruded composite films are two or more films produced simultaneously, and formed into a multilayer structure while still in amorphous and semimolten states during the manufacturing process.

20.8 Closure and packaging methods. The preceding paragraphs have discussed the general preparations and materials for clean packaging. The next step in clean packaging is the application of these materials in such a manner as to provide a sealed, identified, and contamination-protected item. Depending on the item and its cleanliness requirements, packaging can range from a fairly simple operation to a complex process.

Packaging techniques have been developed which, when coupled with a knowledge of the elements of clean packaging, can produce a package as clean as the material technology presently permits. Unfortunately, so far as particulate contamination is concerned, material development has barely kept pace with cleanliness requirements and lags behind surface cleaning capabilities.

A complete clean package may consist of one or more elements dependent on the particular requirements. These elements are: (a) intimate cushioning, used as required; (b) intimate package, used to exclude exterior contaminants and maintain cleanliness of item; (c) environmental package, used over the intimate package when that package does not provide adequate environmental protection. The following paragraphs describe the closure and packaging methods for constructing these elements of a clean package.

20.8.1 Intimate cushioning. The purpose of cushioning in clean packaging is to protect the intimate package material from excessive abrasion or rupture due to contact with the item. It differs from the usual concept of cushioning in that it does not necessarily serve as a shock mitigating medium. Any protrusion, sharp corner, or externally threaded section should be adequately cushioned.

Because of the quantity of sharp edges involved, male threaded sections present a unique challenge to clean packaging. Normally they are covered with one or more layers of the same clean material used for the intimate package. Surface area should be kept to a minimum consistent with the need for completely covering the threads and securing the material to the item. There appears to be no specific agreement on how tightly the material should be drawn over the item. It is believed, however, that best results are attained when the material is neither too tight nor too loose but is gently handformed over the section. This technique reduces both the material surface area and the amount of movement between the material and the item, but it does not draw the material tight over the sharp edges.

Cushioning material may be secured to the item in several ways as described below. The exact method will depend on several factors such as item configuration, cleanliness requirements of the item external surfaces, and the amount of handling and transportation anticipated.

20.8.1.1 Taping the cushioning to the item. This method involves securing the cushioning materials to the item at all common points. It is most often used on external protrusions which lead to internal surfaces of a component or assembly, and when the cleanliness requirements are primarily intended for the internal surfaces.

That portion of the tape in direct contact with the part will probably leave an undesirable residue which may have to be removed by further cleaning when the cushioning is removed. The length of the tape used should be sufficient to make an effective bond. If the tape is applied around a protrusion, it should overlap itself by at least 1/4 of the circumference of the protrusion.

20.8.1.2 Taping only over the cushioning material. This is similar to Method 20.8.1.1 above, except that the tape does not contact the surface of the item. The cushioning is applied in the same manner, hand-formed over the protrusion, and held in position while the tape is secured. This method eliminates the problem of adhesive residue on the item.

The cushioning must be of sufficient length to allow space for taping. The tape must be stretched and kept under tension as it is applied in order to provide adequate holding power when the tension is released. In this case, the tape should overlap itself by at least 1/2 the circumference of the protrusion.

20.8.1.3 Tying the cushioning to the item. Strips, ribbons, or monofilament cord of clean packaging material may be used to secure the cushioning if tape is not acceptable. This is applied in much the same manner as described in 20.8.1.2 above. While this method offers the advantage of eliminating the tape, it also has two distinct disadvantages. It presents more opportunity for particle sloughing both from the increased amount of material and from the tying operation. Further, unless the tie is strongly knotted, it may come loose during subsequent handling.

20.8.2 Intimate package. Significant factors in determining the methods and procedures for preparing an effective package are cleanliness requirements, shedding of the barrier material, and size and configuration of the item. Some typical methods of making an intimate package are described in the following paragraphs. Variations and combinations of these simple package forms should be considered to provide the most effective package for any given application. In all packaging operations, the item and the package material must be handled in a manner that will essentially preclude contamination.

20.8.2.1 Film wrap. The entire item is enclosed in a plastic envelope, bag, or tube. For this type package, all surfaces of the item must be cleaned to the same level.

The film should be gently hand- or vacuum-formed around the item to achieve the minimum of package volume. Seal the film as close as possible to the item without causing undue stress which might damage either the item or the film.

Prior to final sealing of the package, the interior of the package and the item may be purged by directing a stream of filtered dry nitrogen or other inert gas into the package at a pressure, volume, and period of time which are adequate to completely replace the original atmosphere of the package with the gas. The final seal of the package should be accomplished immediately after purging to assure a maximum entrapment of the purging medium.

Some typical applications of this type of film package are shown in the preceding three figures.

20.8.2.2 Film closure. By this method, the open connections and ports leading to cleaned interior surfaces of parts, ducts, fittings are closed with plastic film.

One or more layers of clean film are placed over the opening and formed around the outside of the part and secured by tape or tie placed over the film and drawn tight. A film tube of adequate size to fit over the opening and extend down the side of the part may be used in place of film sheet.

Adhesive-backed tape may be used to secure the film snugly around the part to effect the seal. The tape should be placed entirely on the film and not extend onto the part unless its contact is specifically acceptable for subsequent operations and assembly. However, it should be noted that a good hermetic seal may not be effected. Therefore, additional environmental wraps to provide the necessary protection may be required.

Prior to final sealing of the opening(s), the interior of the part may be purged with filtered dry nitrogen or other inert gas. Purging should be performed in a manner to ensure that the inside atmosphere of the part and the closure(s) is completely replaced with the gas. Final sealing of the closure(s) should be performed immediately following the purging.

20.8.2.3 Film closure with protective disc or cover. The size of openings in parts may require some form of protection for the plastic film closure. Discs or slip covers made of rigid plastic or similar, smooth, clean material may be used. If the disc or cover will be in contact with the part, metals should be carefully selected to preclude the possibility of galvanic corrosion.

The disc should have a slightly larger outside diameter than the opening on which it is to be used. With the film closure drawn smooth over the opening, the disc is centered over the opening on top of the film. Tape is placed around the perimeter of the disc, partially on the disc and partially on the film covering the outside of the part, with the tape drawn tight to hold the disc securely in place. In some situations, the disc may be placed directly over the opening, the film drawn smooth over the disc, and the tape placed over the film to hold the disc and film in place. A second wrap of tape may be required near the end of the film to provide a better seal and hold the film in place.

Covers used should be of adequate size to slip over the outside diameter of the part at the opening. With the film closure drawn smooth over the opening of the part, the cover is slipped over the film and part with the head of the cover seated firmly against the end of the opening. The covered flange is sealed to the film with tape placed partially on the flange and film and around the circumference of the closure. In some instances, the cover may be placed directly over the opening with the film closure over the cover in a manner similar to the disc and film closure.

Purging when required should be performed as described for the film closure.

20.8.2.4 Gasket and place closure. Bolted flange-type connections and openings leading to precision-cleaned interior surfaces may be sealed using a gasket and closure plate.

The gasket should be made of clean film or other material compatible with the end use of the item and of adequate thickness to provide a compression seal to the flange. One or more sheets of gasket material may be used to obtain the desired thickness.

The diameter of the gasket and closure plate should be equal to the outside diameter of the flange. Holes should be of equal size and have the same hole pattern as the corresponding holes in the flange.

The closure plate should be a rigid material of adequate thickness and flatness to assure uniform compression of the gasket on the flange.

If attachment hardware such as bolts and nuts is of a metal dissimilar to that of the flange, bushings and washers of nylon or similar material should be used to prevent galvanic corrosion. The bolts and bushings should fit loosely in the holes to preclude galling or shredding.

Purging the interior of the item with filtered dry nitrogen or other inert gas may be accomplished prior to sealing by directing the gas into another opening in the part and allowing the gas to escape around the gasket, or allow space between plate/gasket and flange to direct the gas into the interior at this point. Purging should be performed in a manner to completely replace the inside atmosphere with the gas.

Immediately following the purging operation, the closure should be made with the film gasket over the flange face and the closure plate over the gasket. With holes aligned, attachment hardware is installed and torqued to recommended values for the size and type used. This is followed by placing tape over the circumference of the plate, gasket, and flange.

20.8.2.5 Threaded cap or plug closure. Threaded connections with openings leading to clean interior surfaces may be sealed with a threaded plug for female thread connections or a threaded cap for male thread connections. O-rings or gaskets may be used in conjunction with the caps and plugs for effective sealing. This type of closure has limited application due to the inherent characteristic of particle generation by the connecting and disconnecting of the threaded closure. Careful selection of the closure material and thread profile will aid in minimizing particle generation, and preventing galvanic corrosion.

A clean cap or plug should be carefully mated to the connection by applying a minimum of torque to seat and seal the closure. Excessive torque increases the probability of particle generation in the threads due to galling and shredding of the thread surfaces.

Prior to sealing the closure, the interior of the item may be purged.

20.8.2.6 Containers of rigid material. Enclosure of a part or device in a container of this type is frequently employed as a specially designed encapsulated package in which the device is mated to the package in a manner to enhance its operation in the completed system. However, the utilization of rigid materials for the preservation of cleanliness of parts on a more temporary basis offers some advantages and disadvantages described earlier.

20.8.3 Environmental package. The selection of the type of material for the environmental package depends on the type and degree of protection needed for its contents. Polyethylene film is frequently used for this purpose. Containers formed of rigid materials may be applicable for this type package and will provide additional physical protection when required.

The operations involved in applying and sealing the environmental package are essentially the same as described for the intimate package. Film wraps and closures should be formed over the intimate package and sealed in a similar manner but not to interfere with or damage the intimate package. Rigid containers may require additional intimate cushioning material, such as dimple wrap, around the intimate package in the container. Sealing may be accomplished using adhesive-backed tape over the juncture of the container parts.

20.8.3.1 Variations of environmental packages. Some variations of the application of the environmental package are described below. The requirements of the individual packaging situation will dictate the particular methods.

(a) Desiccant and humidity indicators may be enclosed with the environmental package. The desiccant should be of the nondusting type in cleanable bags, and should conform to Mil-D-3464, Type II. Desiccant bags should be wiped clean with a solvent-dampened cloth before use.

(b) Identification labels may be attached in the external portion of the package when a plastic film wrap or bag is used. After sealing the package, the label is placed within the excess portion of the film and sealed to retain the label.

(c) Purging the environmental package may be accomplished prior to sealing, by directing a stream of filtered dry nitrogen or other inert gas into the package in a manner to assure complete replacement of the inside atmosphere with the gas. In some cases, the environmental package is slightly pressurized with the purge gas to provide a cushioning effect. In others, it is hand- or vacuum-formed around the intimate wrap.

20.9 Sealing. Methods for effectively sealing packaging materials to assure the integrity of the enclosed clean item depend on the type of material and the closure or wrap. Sealing methods described in the following paragraphs are some which have general application for the common types or closures. Each particular packaging situation requires individual consideration to select or develop the most effective method of sealing.

20.9.1 Heat sealing. Heat sealing is the simplest and most commonly used method for joining plastic films of the same type or different types and for joining plastic films with some other materials. Heat sealing eliminates the need for adhesives and other forms of mechanical fasteners.

The major techniques of heat sealing which are currently in use are thermal, dielectric, and ultrasonic. There is a preferred method for each type of film; however, many of the films can be sealed by more than one method. An effective hermetic seal can be achieved on most of the plastic films by at least one of these methods. The following table lists some of the plastic films with the methods that may be used for sealing.

Table LVIII.
Plastic film heat-sealing methods

Film Type	Dielectric	Thermal	Ultrasonic
Polyethylene	0	X	X
Polyamide (nylon)	X	X	X
Polycarbonate	0	X	X
Polyester	X	0	X
Polypropylene	0	X	X
Polyurethane	X	X	0
Polyvinyl chloride	X	X	0
Fluorohalocarbon	X	X	X
X - sealable 0 - not sealable			

20.9.1.1 Thermal sealing. Thermal sealing is the direct application of heat to the films for a sufficient period of time for the films to melt and fuse together. The preferred sealing method for clean packages is thermal impulse sealing wherein a die is quickly heated after coming in contact with the material and the proper pressure is applied. This heating cycle is then followed by a cooling period with the material held in place by pressure on the die, which assures an effective seal without distortion or separation.

In the hot-bar method the film to be sealed is placed between two plates or dies, one or both constructed with cartridge or strip heaters. Temperature-sensing devices are mounted close to the sealing surface of the dies to control the temperature, as effective control of the temperatures determines the strength and quality of the seal. A press is employed to apply pressure to the dies for the time required for the films to melt and fuse, and then the dies are separated and the sealed film allowed to cool. Continuous seams can be made by passing the films between hot rollers under pressure or over a hot platen, and then between cold rollers under pressure. Films which can be effectively sealed by this method are generally those which can be removed hot from the die without distortion or separation of the seal. Nylon and Aclar are difficult to seal by the constant heat, hot-bar method.

In both methods the flatness of the die faces, which contact the material, is important to ensure uniformity of pressure applied in all areas for a homogeneous seal. Materials of construction for dies should be similar throughout to avoid bimetallic distortion due to differences in the thermal coefficient of expansion of dissimilar materials. Effective use of insulating materials and barriers is required for good temperature control.

20.9.1.2 Dielectric sealing. This method utilizes radio frequency (RF) to produce the heat and fusion process. The films being joined act as the dielectric between the plates of a capacitor which are the die face and the press platen. The plates are connected to an RF generator which induces molecular vibrations within the plastic and the resulting friction produces heat uniformly throughout the film seal area. Since the plates or dies are cool, heat loss to the plate cools the film surfaces so that only the interfaces melt and fuse together forming a bond.

The frequency allocated by the Federal Communications Commission (FCC) for unlimited operation of this equipment is 27.12 MHz. This frequency provides effective sealing of most materials sealed by this method. Operation at other frequencies required that the radiation be maintained below the level of interference specified by the FCC.

The major advantages of the dielectric method of sealing are the speed, ease, and simplicity of producing effective seals with good reproducibility and reliability. After sealing, the material can be cooled under pressure to achieve maximum seal strength and quality.

Dielectric sealing is normally used only in the manufacturing process for high volume plastic products, due to the high investment required for the device and the shielded facilities. It is not used generally for clean packaging applications.

20.9.1.3 Ultrasonic sealing. Joining plastics by ultrasonics employs high frequency mechanical vibration to produce molecular motion or jostling at the interface. The localized heating results in a natural molecular bond without deforming, distorting, or changing the properties of the material. The material to be joined is placed between a solid anvil and an ultrasonic tool, which provides the mechanical vibrations. The quality of the bond formed depends on the pressure of the tool on the work surfaces, the length of time the vibrations are impressed, and the amplitude of the tool vibration. The thickness of the material and the type of plastic must be considered in designing the sealing procedure.

Ultrasonic sealing can be used for plastic films, but it is generally considered too expensive and too slow a process for this purpose. It is used extensively for bonding rigid plastics.

20.10 Tape, ribbons, and ties. Some types of closures may be sealed adequately by placing adhesive-backed tape, ribbon, or other ties over a flexible film and drawing it tight around an outside portion of the item and securing in place. In some instances, adhesive-backed tape may be used, partially on the package material and partially on the item, to secure a seal. In other instances, the tape may be used to seal the opening in the package material.

The use of adhesive tapes should be in a manner that will not contribute to the contamination of the clean item or surface.

20.11 Compression seals. Gaskets, O-rings, and similar type materials may be used to form a seal over or around a closure on an item or between two parts of a rigid container. Material used for this type seal should be clean and of a type that will not contribute contamination.

A means for compressing the seal is required, and the mating surfaces must be relatively uniform to assure uniform pressure over the seal area.

20.12 Integrity seals. Seals or decals of a tamperproof type may be applied over the closing seal area of a package. These seals are used as a means of detecting the violation or tampering with the clean package seal. Some form of marking is usually included on this seal such as date and place used or other identification.

The requirements for using integrity seals and the method of application should be specified for each particular type of package seal.

20.13 Verification of clean packaging. Since the objective of clean packaging is to maintain a specified cleanliness level of the enclosed item, some inspection or testing techniques must be applied to verify that this cleanliness level is, in fact, maintained. Assuming that the item is clean prior to packaging, there are two general types of verification procedures that may be employed. One is monitoring the cleanliness of the intimate packaging materials prior to use and observing clean packaging practices during subsequent packaging operations. The other is monitoring the cleanliness of functional operation of the item upon removal from the clean package. Each of these procedures is described in the following paragraphs.

20.13.1 Monitoring packaging materials and operations. Plastic films are the most commonly used intimate packaging materials capable of being cleaned to the level required for clean packaging. Surface cleanliness of the other materials, when used, may be monitored as described in Section 14, Control of Surface Contamination. The most generally accepted method for monitoring the cleanliness of clean packaging films is set forth in Specification MSFC-Spec-C-25. Information from this specification is extracted as follows:

20.13.1.1 Visual inspection. No evidence of oil, grease, water, solvents, paints, ink, dirt, metal chips, decals, preservatives, or other foreign matter shall be permitted on either the external surfaces or the internal surfaces of packaging materials when inspection is made with the unaided eye. Surfaces shall contain no particulate matter greater than approximately 50 microns in size and should be free from all films other than known innocuous films.

20.13.1.2 Preparation for test. The bag shall be heat-sealed across the open end. Using surgical scissors or other extremely sharp blade (to minimize particle generation when cutting), one corner of the bag shall be cut off so that an opening not over 3/4 inch in length is created.

Plastic tubing for precision packaging applications shall be sealed at both ends of a length to give an inside test area of approximately one square foot and sampled by rinse test as for bags.

Plastic film (flat roll stock) shall be cut carefully with surgical scissors or other sharp blade to a length and width of 12 inches. The sections shall be folded in half, sealed into a bag form in such a manner as to minimize exposure of the interior to airborne particles, and sampled by rinse test as for bags.

20.13.1.3 Rinsing. Through the opening, 100 ml of solvent (MSFC-Spec-237A) per square foot of interior surface shall be introduced from a wash bottle or similar apparatus. A bag having less than one square foot of interior surface shall be considered as one square foot. The opening shall then be held shut by a practical means. The exterior of the bag shall then be rinsed down with the same agent to prevent exterior particles from being picked up when the bag is decanted. The cleaning agent within the bag shall be agitated by a gentle but rapid sloshing.

20.13.1.4 Collection of sample. The cleaning agent within the bag shall be poured out through the same opening (held shut during rinsing) onto a microporous 0.45- or 0.8-micron membrane filter.

20.13.1.5 Testing. The effluent of the rinse test shall be examined for particulate matter by the particle count method in accordance with ARP 598, or equivalent. The nonvolatile residue of the solvent rinse shall be determined in accordance with ASTM D2109-64 or an equivalent procedure.

20.14 Clean packaging quality control. Monitoring the clean packaging operation itself is a function of the packaging supervisor. Although this is not a recognized quantitative means of verifying cleanliness, it is nonetheless a practical means of verifying that all reasonable measures have been taken to assure a clean package.

20.14.1 Control factors. Some of the conditions that can be monitored by the supervisor are suggested below:

(a) Nothing should be permitted to enter the clean room except essential materials, equipment, and authorized personnel.

(b) Personnel should be properly attired.

(c) Clean packaging materials (film, bags, etc.) should be covered when not in use.

(d) Heat-sealed closures should be made as soon as possible after an item is inserted into a bag.

(e) Tools and equipment (knives, cutters, heat-sealer bars, and rollers) should be cleaned frequently or as dictated by use.

(f) Protective covers on clean materials should be resealed if contents will not be withdrawn for a significant period or if they are transported from one clean area to another.

(g) Ancillary materials such as desiccant and labels should be brought into the immediate packaging area only when and in the quantities needed for use.

20.14.2 Affectiveness of clean packaging. In addition to monitoring the cleanliness of packaging materials and operations, another method is widely used to verify clean packaging, or what may more accurately be called the effectiveness of clean packaging. This is done by monitoring the cleanliness of the part immediately after its removal from the clean package. Several means of monitoring in this fashion are described below:

(a) External surfaces and accessible internal surfaces may be visually inspected for contaminants such as particles, moisture, corrosion, scale, and oil. The presence of these contaminants usually indicates improper or inadequate cleaning prior to packaging or rupture of the package during handling or transport. A special light source or borescope is usually needed to inspect internal surfaces.

(b) External surfaces may also be inspected with a microscope or by any suitable means of verifying surface cleanliness as described in Section 14, Control of Surface Contamination. The selection of a particular test method will usually depend on:

1. (1) The degree of cleanliness required.
2. (2) The size and configuration of the item.
3. (3) The type(s) of contaminants of interest.
4. (4) The materials of construction.

(c) In some systems or assemblies, internal and inaccessible surfaces and confined areas may be monitored by administering a purge gas and testing the gas as it is expelled from the system. A dry, filtered, inert gas should be used for this purpose. The three contaminants most commonly monitored by this method are:

(1) Particulates entrapped on a membrane filter, and sized and counted by use of a microscope and ocular micrometer.

(2) Moisture measured by a moisture meter in parts per million

by volume, by the dewpoint method in degrees centigrade (°C) or other acceptable methods.

(3) Condensable hydrocarbons measured by the scrubber method or other acceptable method.

Precision clean solvents may also be used in some systems in this same manner to test for particulates and nonvolatile residue.

While this method may be acceptable for some systems, it is not suitable for others because of two serious limitations. Contaminants may be trapped in pockets or dead-end plumbing and therefore are not evident, and test pressures applied to the system may be only a fraction of the actual working pressure.

(d) Electrical components and assemblies may be given a functional or continuity check. Any malfunction discovered by this method, however, might be attributable to handling damage.

Another type of monitoring sometimes used involves removing clean packaged components from their package, assembling them into a system, and conducting a functional test of the complete system. This may be sufficient for some applications, but it is not a true test of clean packaging effectiveness because it does not consider handling damage or contamination introduced during the assembly process.

20.15 Prodedures.

20.15.1 Packaging procedure for service media. The following materials for packaging shall apply with regards to the service media.

(a) All component parts, sub-assemblies, assemblies, etc., utilized in oxygen systems, will require Aclar film 33C, Allied Chemical Corp., as environmental wrap and/or outlet port covering.

(b) All other system parts will require Nylon 6, E. I. DuPont deNemours & Co., or equal, as environmental wrap or outlet port covering.

20.15.1.1 Method I (small components):

(a) For use on, but not limited to: Fittings, (unions, tees, elbows, etc.) seals (O-rings, torus rings, gaskets, etc.), springs, nuts, bolts, washers, pins, etc.

(b) Environmental bagging will be accomplished as follows:

(1) Purge bag with GN_2.

(2) Place part into bag.

(3) Gently but effectively compress bag with hands to force out excess GN_2.

(4) Heat seal bag.

<u>Note</u>. Due to applicable vendor requirements it may be necessary to pull vacuum on environmental bag. Process as follows.

(5) Insert vacuum pump tube needle, at a corner location of environmental bag, pull vacuum until bag collapses firmly about enclosed part, remove needle and immediately heat seal corner of bag.

(6) Apply tamperproof seal, at heat sealed end of bag, if required.

(c) Overwrap bagging will be accomplished as follows (one or two overwrap bags as required):

(1) Select, or fabricate, proper size bag.

(2) Place environmental bagged part into overwrap bag.

(3) Gently but effectively compress bag with hands to force out excess air.

(4) Place certification card into bag.

(5) Heat seal bag.

<u>Note</u>. Due to applicable vendor requirements, it may be necessary to pull vacuum on overwrap bag. Process as follows.

(6) Insert vacuum pump tube needle, at a corner location of overwrap bag, pull vacuum until bag collapses firmly about enclosed bagged part, remove needle and immediately heat seal corner of bag.

<u>Note</u>. In the event a second overwrap bag is required, repeat steps (1), (2), (3), and (5). Do not pull vacuum.

(7) Place bagged part(s), protected by dimple wrap, or equal, in shipping carton and prepare for shipment.

20.15.1.2 Method II:

(a) For use on, but not limited to: Valves, gages, regulators, transducers, instruments, etc.

(b) Application of environmental wrap will be accomplished as follows:

(1) Apply a double film of environmental wrap over inlet and outlet ports, fitting ends (attached to valves, gages, etc.), apertures, etc.

(2) Secure environmental wrap with tape.

(c) Application of overwrap will be accomplished as follows:

(1) Apply a film of overwrap, over environmental wrap (tape) and secure with tape.

Note. The size of the part, being packaged by Method II, will be the deciding factor with regards to overwrap bagging. When feasible, parts that measure 4 inches or less in any direction, shall be placed in an overwrap bag. Process as follows.

(2) Select or fabricate proper size bag.

(3) Place environmental taped part into overwrap bag.

(4) Gently but effectively compress bag with hands to force out excess air.

(5) Place certification card into bag.

(6) Heat seal bag.

(7) Do not pull vacuum.

(8) Place parts (bagged) (not bagged) properly protected, in shipping carton and prepare for shipment.

20.15.1.3 Method III:

(a) For use on, but not limited to: tubing, flex hose, pipe, duct, expansion joint, etc.

(b) Application of environmental wrap will be accomplished as follows.

Note. Prior to application of environmental wrap, on tube assemblies, back to backs, etc., that contain movable "B" nuts and sleeves, move "B" nuts and sleeves approximately 2 inches and secure with tape.

(1) Apply a double film of environmental wrap over ends of hose, tube assemblies, back to backs, etc.

(2) Secure environmental wrap with tape.

(c) Application of overwrap will be accomplished as follows:

(1) Apply a film of overwrap, over environmental wrap (tape) and secure with tape.

Note. The size of the part, being packaged by Method III, will be the deciding factor with regards to overwrap bagging. When feasible, parts (tubes, back to backs, etc.) that measure 6 inches or less in length shall be placed in an overwrap bag. Process as follows.

(2) Select, or fabricate, proper size bag.

(3) Place environmental taped part into overwrap bag.

(4) Gently but effectively compress bag with hands to force out excess air.

(5) Place certification card into bag.

(6) Heat seal bag.

(7) Do not pull vacuum.

(8) Place parts (bagged) (not bagged), when feasible, properly protected, in shipping carton and prepare for shipment.

Note. Parts with large flange areas (4 inches and greater) may require special handling and processing. When this is the case a special packaging and handling process will be issued with regards to part involved.

20.16 References.

1. Clean Packaging, Technical Micronics Control, Document B.

2. Contamination Control Handbook, Sandia Laboratories, NASA-SP 5076, 1969.

SECTION 21

QUALITY ASSURANCE

21.1 Introduction. The general consideration of quality assurance refers to a system or methods of measuring and comparing the quality of a processing plant's production against established standards. Control of processes and maintenance of the ultimate in finished product quality are among the more important phases of plant management. Quality assurance supplies the production phase with detailed analytical information to aid in proper operation and control of equipment and processes to assure optimum quality, maximum output, and safe operating conditions.

The following quality assurance outline is a workable program that has proven to be extremely efficient in a particular case. It is offered merely as an example, however, may be used as a guide to establish a quality assurance program to satisfy a particular need.

21.2 Applicability. Compliance with the quality requirements of customers specific needs is of great consequence. In order to insure conformance to these requirements, it is mandatory that a quality assurance program is established and maintained. The criteria established within the program serve two purposes: (1) enables the customer to be aware of company intentions and program implementation (2) offers a mandatory directive to employees to be utilized daily in the performance of services.

The requirements for an established quality assurance system is a part of virtually every contract and sub-contract. It is the intent of this procedure to authorize the initiation and use of the quality assurance procedures.

It shall be the responsibility of the quality assurance department to assure that each obligation of contractual documents related to services be fulfilled through implementation of the written instruction within the procedures of the program.

Quality control management shall assume the responsibility of review of new contractual documents and shall initiate necessary action to assure that satisfactory instructions are written to comply with new requirements.

Preparation of procedures of the quality assurance program may be made by any individual or department within the company; however, final release responsibility shall be assumed by the quality assurance manager or his designated representative.

21.3 Requirements. The general control of facility cleanliness and systems shall be in accordance with the requirements established in this quality assurance program and according to specifications.

Recording of all data related to the controls described shall be accomplished by the entry of objective evidence by the cognizant inspector on a controlled environment data log posted in or near the quality managers office for daily use. This log shall contain all the control point requirements of this document and shall be supported by such back-up data as required to depict complete evidence of these quality assurance program requirements.

21.4 Controlled environment area.

21.4.1 Pressurization. The CE shall be pressurized to a minimum of 0.05 inches H_2O above the ambient atmosphere. Each successively cleaner CE area shall be pressurized above the less clean CE area adjacent to it, at least .01 inches water.

Once the cleaning facility has been placed in operation, pressurization shall be maintained continuously.

21.4.1.1 Pressurization failure. In the event of pressurization shutdown or failure the following precautions shall be taken:

(a) All cleaning, assembly, and testing operations shall be suspended.

(b) Parts, components, and assemblies in process shall be covered to reduce chance of contamination from fallout and particles which may be breathed into the facility due to pressure loss.

(c) Every possible precaution shall be taken to prevent excessive contamination of the area.

(d) When pressurization is again achieved, aerosol tests of the area shall be made before resumption of processing, assembly, and testing. Vacuum clean and mop down area prior to resumption of operations when indicated by aerosol checks. Record results on applicable form and validate with initials and quality assurance acceptance stamp.

21.4.2 Vacuum cleaner system. The vacuum cleaner system shall be maintained and cleaned bi-monthly or as required to enable optimum efficiency.

21.5 Demineralized water system. The specific resistance of the demineralized water shall not be less than 50,000 ohms. The demineralized water shall be tested on a regular scheduled basis to verify specific resistance of 50,000 ohms.

21.6 pH value. The pH value shall be maintained between 6.0 and 8.0 and shall be checked and recorded as required by operational needs.

21.7 Filters. Systems filters shall be changed subsequent to repairs or cleaning; operations shall be made upstream from the filter.

21.8 Drying purging and testing gases used on clean components. Drying, purging, and testing gas shall conform to Mil-P-27401. Conformance to Mil-P-27401 is assured by certification and affidavits on file at the supplier's plant and in quality assurance office.

21.8.1 Gas storage system. Gas storage system shall be checked as follows:

(a) Dew point and particulate analysis shall be checked in accordance with approved quality requirements and:

  (1) At any time that system integrity is in question.

  (2) After change of the last supply filter.

21.9 Cleaning solution. The compound chemicals used to mix a solution shall be carefully measured.

(a) Solution concentration shall be checked as determined by use, and results recorded.

(b) Conditions that do not comply with specifications shall be corrected prior to further processing.

21.10 Drying oven. Temperature of the drying oven is controlled by thermostat. The accuracy of the thermostat shall be checked once each month of operation or portion thereof and recorded. Variation in excess of 10 degrees F between the thermostat and the standard shall be cause for replacement or recalibration of the thermostat. Thermostat shall be checked at 150 degrees F.

21.11 Testing airborne contaminants. Tests shall be made once each day of operation and at closer intervals at the inspector's option. Results of these tests shall be recorded and action taken to rectify unsatisfactory conditions will also be recorded. Particulate contamination shall not exceed requirements.

(a) Evaluation of airborne contaminants shall be made as specified.

(b) Ultraviolet check shall be made as specified.

21.12 Visual inspection. Visually inspect to determine the acceptability of cleaned parts and controlled environment areas, and to determine whether or not contamination control procedures are effective and adequate. Visual examination shall not be the only basis for acceptance of parts, components, or assemblies.

(a) Damage. Examine the parts for nicks, dents, abrasions, scratches, scarring, burrs, etc., which may occur during fabrication, assembly, cleaning, shipping, packing. Determine if damage will be detrimental to the function and life of the end product.

(b) Contamination. Visually examine for particulate matter. Check for the presence of oils, grease, preservatives, corrosion products, weld slag, shop and other dirt, and other material foreign to the item.

(c) Deterioration. Examine parts for deterioration, aging, softening, erosion. Determine if damage is due to excessive or improper cleaning, processing, storage, handling, or application of incompatible cleaning solution.

Fuel system equipment, pipes, and components may have a light film resulting from rust-proofing treatments.

21.12.1 Ultraviolet examination. By use of an ultraviolet light source of 2500 to 3700 angstrom units, examine all accessible surfaces of the specimen to determine the presence of petroleum-type hydrocarbons. Ultraviolet examination will be used to detect contamination, generally hydrocarbon in nature. Excessive contamination, as determined at this point, shall be cause for rejection regardless of fluid or airborne particle analysis results. Aided visual inspection will be utilized whenever necessary or required.

The area where this inspection is performed shall be capable of a complete blackout.

All petroleum-type hydrocarbons will not fluoresce under the specified wave length (2500-3700) angstrom units) of this test. Examples of such materials which do not fluoresce are RP-1 fuel, RP-4 fuel, and most other kerosene-type fuels.

21.12.1.1 Procedure when fluorescent surface areas detected. Component parts which are found to have fluorescent surface areas after cleaning shall be subjected to the following procedure. (Note: This test shall not be used when fibers, lint, or any free fluorescent matter as evidenced by a wipe test are found present on part surfaces.)

(a) Collect samples from fluorescent surfaces and perform laboratory analysis for positive identification.

(b) When positive identification has been established and the materials identified are known to be compatible with liquid oxygen, the rejection may be waived.

(c) If positive identification cannot be established, the part shall remain rejected, until the material has been removed.

21.12.1.2 Alternate method.

(a) Wet filter papers (Whatman No. 42, S and S No. 6202, or equal) and swab fluorescent areas successively with:

  (1) Chlorinated aliphatic solvent (e.g., trichloroethylene, carbon tetrachloride).

  (2) Water miscible solvent (e.g., isopropyl alcohol, acetone, diethyl ether).

  (3) Aromatic solvent (e.g., toluene, xylene).

  (4) A paraffinic solvent (e.g., hexane, petroleum ether).

(b) When no evidence of fluorescence is transferred to the filter paper, the fluorescent area so tested shall be acceptable.

(c) When fluorescence is transferred, the part shall be rejected, the fluorescence removed and recleaned through the complete cleaning cycle.

These requirements are not applicable to hydraulic oil areas.

21.13 Personnel indoctrination. The door to the CE areas shall be kept closed at all times. There shall be one man for each shift in charge of each area. This man shall be given full responsibility and authority for seeing that the requirements of this specification are fulfilled.

It is imperative that all personnel associated with CE operations, including supervisors, technicians, and plant maintenance personnel receive a thorough indoctrination in the purpose and practices of CE operations. Each individual subsequently assigned to CE work will receive thorough indoctrination. The CE indoctrination will cover the following subjects:

(a) Personnel restrictions due to excessive skin conditions or temporary respiratory ailments.

(b) Visitor authorization and control. Anyone entering CE area without proper authorization will be ushered out of those areas and shall remain out until authorization is granted.

(c) Personnel cleanup practice before entering the CE work area.

(d) Contamination preventive clothing: specification for material and styling, recommended laundry procedure, storage, handling, care, and use.

(e) Restrictions for bringing into the CE area (personal or work associated) contamination generating materials or articles.

(f) Cleaning procedures for parts, materials, tools, equipment for use in CE area.

(g) Protection procedures for storage of work in progress.

(h) Housekeeping standards and procedures, schedules, materials, and equipment for use in CE area.

(i) Planned and emergency maintenance and service procedures, schedules, tools, and equipment for CE areas.

21.14 CE area control procedures. The following restrictions and controls are applicable to all classes.

21.14.1 Control of personnel entering CE area.

(a) Permission for visitors to enter CE will be strongly discouraged and held to an absolute minimum. No one will be permitted to enter the CE without first receiving an indoctrination on CE conduct, going through the entire cleanup process, and donning the full set of dust preventive clothing as required by specifications.

(b) Continuous sneezing and coughing will not be permitted in the CE. Persons with colds or allergy conditions shall be assigned temporary jobs outside the CE until they are sufficiently recovered.

21.14.2 Control of personnel leaving CE area.

(a) Personnel leaving the CE for any reason will remove smocks and all other dust preventive clothing and leave these garments in the personnel change room. Upon re-entry into the CE change room, the entire cleanup procedure will be carefully and completely repeated.

21.14.3 Lunch and breaks. Snacking and eating is absolutely forbidden in all classes of CE and in areas provided for cleanup of personnel or material preparatory to entering the CE. No edibles, including candy, chewing gum, or soft drinks shall be taken into the CE.

21.14.4 Restrictions of articles permitted in CE area.

(a) Only those tools, fixtures, test equipment required for current workloads will be kept in the CE area.

(b) Abrasives such as steel wool, emery cloth and sand paper, files will not be permitted in CE. Corrosive materials and cleaning solvents are prohibited except those specifically authorized in work specifications for use in the CE area.

(c) Ordinary paper pads, notebooks, manuals, or writing paper of any description will not be permitted. Ordinary writing pencils, pens, erasers, crayons, or chalk will not be permitted. Notes and records will be kept on lint-free watchmaker paper, plastic sheet, or equal, using ball point pens or other suitable non-dust generating materials. Blueprints, work specifications and other instructions will be printed on or covered with plastic film or other lint-free, non-dust generating materials.

(d) Paper towels (except Kimwipes), and cloth towels, are prohibited in CE, personnel and material cleanup rooms, and locker rooms. Bar and powdered soaps or detergents are prohibited. Only liquid detergents shall be used.

(e) Personal articles, such as, keys, coins, knives, nail files, cigarettes (tobacco), lighters, matches, pencils, watches, handkerchiefs, kleenex, combs, cosmetics, candy, and chewing gun will not be permitted in CE areas.

(f) If considered necessary, personnel may be permitted to keep billfolds and similar valuables on their person, but they will be kept in pockets under dust preventive clothing and will not be removed in the CE.

(g) Jewelry, such as necklaces, lockets, earrings, wrist watches, bracelets, large rings, shall not be worn in the CE. Small rings, such as wedding bands, may be permitted if they present no work hazard. Pins and broaches may be permitted provided they are covered and remain covered by dust preventive clothing.

(h) Personal clothing which tends to produce a great deal of lint, such as angora sweaters or linty stockings, shall not be worn in CE.

21.15 Personal cleanup requirements before entering any CE area. The following procedures will apply at all times to all personnel including supervisors and visitors whenever they enter a CE.

(a) Topcoats, raincoats, overshoes, umbrellas, lunches, street shoes, and other such personal articles will be removed and placed in an outer cloak room.

(b) Personnel working in Class 100,000, 10,000, and 100 CE areas will be provided with an extra pair of personal shoes (sneakers) of ordinary commercial type, simple design and comfortable for standing to be used exclusively for wear in the CE. The extra pair of personal shoes will be kept in the CE locker room when not in use.

(c) Personnel working in Class 100,000, 10,000, and 100 CE areas will use castile liquid soap and lukewarm water, wash hands thoroughly to remove dust, cigarette ashes, loose skin flakes, loose hair, skin oils and cosmetics. Dry washed areas with a hot air blower or lint free cloth.

(d) Cosmetics will not be worn or carried into a CE at any time.

(e) Glasses, if worn, will be washed and dried with a lint free towel.

(f) Enter change room and vacuum shirt collars, waist area, pant cuffs and feet.

(g) Don special contamination preventative clothing as listed below:

  (1) Booties.

  (2) Cap; being sure to cover as much hair as possible.

  (3) Coveralls; secure leg opening snuggly and tuck under bootie tops. Do not allow upper section of coverall to touch floor.

21.16 Dust preventive clothing. In general, the clothing will incorporate the following features:

(a) The garments must be available in enough sizes and be sufficiently adjustable to fit all employees as required.

(b) French and fell seams will be used throughout the garments leaving no open ends of fabric which could become frayed and give off lint and loose strands.

(c) All seams will be double stitched using thread of the same material as the garment cloth.

(d) Adjustable exits (sleeves, collars, etc.) will be incorporated to give a snug fit when worn.

(e) Pockets will not be put on any dust preventive clothing.

(f) The cap must fit snugly around the head, covering all of the hair.

(g) The boots must be high enough to cover and hold the coverall pant legs and should have reinforced soles.

(h) Hand covering, when required, must be lint-free, comfortable during use, and enable the user to maintain a delicate finger touch.

(i) The garments will be made from a synthetic fabric material, of a long fiber monafilament type, nonflammable, nonshedding, dust free, preferably dacron polyester.

(j) Garments worn in CE areas will be changed once every 16 hours of use, more often if they become soiled. All CE clothing will be kept in the locker room and stored in polyethlene bags separate from the employees street clothing.

21.17 Cleaning of parts, material, tools, and equipment for CE room use. No tools, parts, or other material will be carried into the CE area, except those items required for the workload or maintenance which have been properly processed. All parts shall be delivered in suitable containers unless restricted by size.

(a) Small hand tools, parts, material, and sub-assemblies will be cleaned and carefully packaged in lint-free, non-shedding plastic containers prior to entry into the CE. Extreme care will be taken to insure that all solvents and all loose particles are removed from all parts and inacessible crannies of the item before they are packaged for CE use.

(b) Portable test equipment, jobs, fixtures, parts, and sub-assemblies which cannot be normally cleaned due to size, material composition, or critical finishes will be thoroughly vacuumed. Entry of these materials into a CE area will be accomplished through an air lock.

(c) All assembly tools used in the CE will be cleaned daily. This operation should be accomplished either at the beginning or end of the work shift. Each CE worker will clean his tools and bench area to insure cleanliness. He will continually inspect and clean his work in progress in accordance with the technical instructions for that item.

21.18 Protecting partly completed work from dust. All tools, parts, and assemblies on workbenches will be kept covered with suitable covers of metal or plastic except when they are actually being worked on. Dust free plastic bags or molded chip proof plastic trays and covers are particularly well suited for protection purposes.

21.19 Maintenance of CE area. The air conditioning and air filtration systems will be kept in continuous operation 24 hours per day every day including weekends and holidays except for brief period of time necessary to accomplish maintenance and repair of the systems.

Following are the minimum requirements for cleaning Class 10,000 and 100 and will be applied to a reasonable degree for Class 100,000.

21.19.1 Daily routine.

(a) Using sponge and pail, wash workbenches, counters, and table tops with detergent (liquid Joy) and water.

(b) Clean floors using central vacuum cleaning system and mop with detergent and water as required.

(c) Clean windows using detergent and water and wipe dry with lint-free wiping material, if necessary.

(d) Doors and walls shall be spot-cleaned with sponge and solvent.

(e) All waste containers shall be emptied daily outside the CE area.

21.19.2 Monthly routine.

(a) Thoroughly wash walls, floors, and ceilings with liquid detergent and water. This thorough cleaning shall be accomplished at a time when CE area is not in cleaning, assembly, or testing operations.

21.20 Work restrictions.

(a) Polishing, grinding, drilling, etc., which produce contamination particles through abrasion, shall not be performed in Class 100,000, 10,000, and 100 CE areas.

(b) Failure of the air conditioning system shall be cause for immediate work stoppage and evacuation of the area in which the failure occurs.

(c) Repairs and maintenance of CE area air conditioning systems, gas systems shall be scheduled for periods when cleaning, testing, and assembling is not in process. When repairs are completed a, thorough cleaning-up shall be performed before CE operations are resumed.

21.21 Equipment requirements for controlled environment areas.

21.21.1 Operation and maintenance of controlled environment area.

1. Vacuum cleaning equipment, commercial industrial type.
2. Pail, plastic.

3. Detergent, liquid Joy, or equal.
4. Sponge, polyeurathane.
5. Mop, cellulose.

21.21.2 <u>Microscopic examination of filter disc for particulate contamination</u>.

1. Microscope, Unitron Binocular, mechanical stage, wide field 10X eyepieces, 4.6X and 10X objective lens, capable of 46X and 100X magnification. This microscope features all substage adjustments for both stage and focus which is better suited for CE work because the microscopists hands do not endanger samples.
2. Ocular micrometer, measuring eyepiece scale with 50 or 100 equal divisions.
3. Stage micrometer, 0.01 to 0.10 mm graduations.
4. Light source, variable intensity.
5. Microscope filter disc holder.
6. Forceps, unserrated tip.
7. Filter disc, Millipore; type HABG and AABG on 3.08 mm centers and equal to 1/100th of the total effective filter area.

21.21.3 <u>Determination of airborne particles in CE areas</u>.

1. Aerosol filter, open type, Millipore No. XX50 047 10.
2. Vacuum source, capable of pulling 25" of mercury constantly.
3. Flowmeter, 40 LPM minimum capacity.
4. Forceps, unserrated tips.
5. Filter disc, Millipore, type AABG, 0.8 mm micron rating 47 mm diameter.
6. Petri dish, plastic, disposal, Millipore Cat. #PD-1004700, or equal.
7. Detergent, Liquid Joy or equivalent.
8. Solvent, trichloroethylene or equal, filtered to requirements.
9. Wash bottle; Millipore solvent filtering dispenser catalogue No.

XX66-025-00.

21.21.4 Analysis of liquid samples for particulate contamination.

1. Pyrex filter holder; Millipore Cat. #XX10 047 00 includes: fritted glass base, rubber stopper, holding clamp, and 300 ml. pyrex graduated glass funnel.
2. Vacuum flask, 1 liter capacity.
3. Vacuum pump, capable of pulling a minimum of 26" mercury.
4. Forceps, unserrated tip.
5. Filter disc, Millipore, 047 mm diameter, type HA.
6. Filter disc, Whatman No. 42.
7. Petri dish, disposal, Millipore.
8. Wash bottle, Millipore solvent filtering dispenser, Catalogue No. XX66-025-00.
9. Detergent, Liquid Joy, self-rinsing, or equal.
10. Plastic film.
11. Alcohol, isopropyl, conforming to Mil-A-10428.
12. Solvent, Trichloroethylene.

21.21.5 Analysis of liquid samples for nonvolatile residue.

1. Oven, stainless steel interior, laboratory, convection type.
2. Flash evaporator, or equal.
3. Analytical balance chain, Ainsworth type LC., or equal.
4. Aluminum moisture dish, E. H. Sargent and Co. #S-25725.
5. Beaker, pyrex glass, 800 ml.
6. Wash bottle, Millipore solvent filtering dispenser Catalogue No. XX66-025-00.
7. Graduated cylinder, 500 ml.

8. Tweezers, laboratory.
9. Desiccator, Cenco, #12691, or equal.
10. Vacuum source, capable of pulling 25" mercury.
11. Acetone, reagent grade, nonvolatile residue content shall not exceed 02.00 mgs per 500 mls.
12. Desiccant, calcium chloride or equal.
13. Wiping material, Kimwipes or equal.
14. Aluminum foil, Mil-A-148.

21.21.6 <u>Analysis of part cleaning solutions</u>.

1. Drying oven, range 50° - 250° C.
2. Analytical balance, read to 0.1 milligram.
3. Flash evaporator.
4. Desiccator.
5. Barnsted purity meter.
6. Buret, capacity 50 ml.
7. Buret, support (single).
8. Buret, support (double).
9. Hydrogen ion (pH) meter, accuracy ±0.01 pH unit.
10. Hot plate.
11. Magnetic stirrer.
12. Filter pump.
13. Beaker, capacity 800 ml.
14. Bottle with screw cap, polyethylene, capacity 8 oz.
15. Bottle with stopper, capacity 1000 ml.
16. Bottle with stopper, capacity 150 ml.

17. Bottle, dropping, capacity 60 ml.
18. Bottle with stopper, weighing, capacity 60 ml.
19. Volumetric flask, capacity 2000 ml.
20. Erlenmeyer flask, capacity 250 ml.
21. Boiling flask, 1000 ml.
22. Filtering flask (no inside tube), capacity 1000 ml.
23. Filtering flask with side tube, capacity 1000 ml.
24. Filtering flask with side tube, capacity 500 ml.
25. Pipet, capacity 100 ml.
26. Pipet, capacity 20 ml.
27. Pipet, capacity 1.6 ml.
28. Pipet, capacity 5 ml.
29. Pipet, capacity 1.2 ml.
30. Pipet, capacity 1.3 ml.
31. Graduated cylinder, capacity 100 ml.
32. Graduated cylinder, capacity 500 ml.
33. Gas washing bottle, with fritted discs.
34. Filter holder (millipore) part #2.
35. Funnel, long stem, 125 mm diameter.
36. Funnel, long stem, 50 mm diameter.
37. Thermometer (30° - 124° F), total immersion.
38. Hypodermic syringe, 30 cc.
39. Ring clamp.
40. Watch glass, diameter 150 mm.
41. Micro slides.

42. Wash bottle, millipore solvent filtering dispenser, Catalogue No. XX66-025-00.

43. Petri dishes.

44. Polyethylene hose, vacuum.

45. Rubber stopper, two hole, size #7.

46. Rubber stopper, two hole, size #8.

47. Acetic acid, glacial, reagent, A.C.S.

48. Barium chloride, A.C.S.

49. Brom cresol green, sodium salt (.04% in distilled H_2O).

50. Carbon tetrachloride.

51. Chromic acid.

52. Gramercy universal-indicator.

53. Hydrochloric acid, reagent, A.C.S.

54. Iron sulfate.

55. Isopropyl alcohol.

56. Lead acetate solution (5%)

57. Leak detector, sherlock 5 second - type 1.

58. Methyl-purple (pH 4.8 - 5.4).

59. Methyl red (pH 4.4 - 6.0) liquid.

60. Methyl red (pH 4.2 - 6.2) powdered.

61. Nitric acid.

62. O-phenanthrolene ferrous sulfate complex ("Ferroin").

63. Oxalic acid.

64. Phenolphthalein (1%in methanol).

65. Phosphoric acid, 85% reagent grade, A.C.S.

66. Potassium acid phthalate, primary standard, A.C.S.

67. Potassium chloride, A.S.T.M. saturated solution.

68. Potassium ferricyanide, crystal, A.C.S.

69. Sodium carbonate,,crystal, A.C.S.

70. Sodium chloride, crystal, A.C.S.

71. Sodium dichromate, crystal, A.C.S.

72. Sodium hydroxide, pellets, A.C.S.

73. Sodium hydroxide solution (.5N).

74. Sulfuric acid, reagent, A.C.S.

75. Sulfuric acid solution, (.5N).

76. Thymolphthalein,(pH 9.4 - 10.6).

21.21.7 Particulate tests for analysis of compressed gases for use in testing, drying and purging.

1. Millipore filter holder, Cat. #XX4504700 or an approved equal.

2. Millipore filter, type HA47 mm diameter.

3. Flowmeter, capable of 10 SCFM low.

4. Binocular microscope with a mechanical stage.

5. Measuring eyepiece, ocular micrometer.

6. Stage micrometer, with 0.1 mm to 0.01 mm calibrations.

7. High intensity microscope lamp.

8. Solvent, trichlorethylene, or approved equal.

21.22 Laboratory sampling techniques. Unless adequate attention is given to proper sampling techniques no amount of refinement of measurement procedures will provide valid, reproducible results. Sampling errors fall in several major categories:

21.22.1 Sampling representation. To be meaningful and reproducible, samples should be representative of the entire fluid system under test when the system is operating normally. Samples from the bottom (or top) of a hydraulic pump will not fully represent contamination in fluid power lines. Samples of more viscous fluids should be taken from areas of high turbulence where particles are mixed throughout the fluid cross section. When it is necessary to sample a static system where the contents of the system cannot be thoroughly mixed, a multi-level sample should be taken.

21.22.2 Sampling valves. Some types of sampling valves such as gasketed globe valves are natural particle generators and must be avoided. Valves with small orifices (needle valves) which tend to act as filters are also to be avoided. Ball valves, plug valves, and quick release valves are among the acceptable types for sampling purposes.

21.22.3 Fluid sampling. Whenever possible, all fluid sampling should be taken on a full flow basis with all of the fluid stream passing through the millipore filter in a closed filter holder. The type and size of holder should be selected for flow required and allowable pressure drop. A stainless steel high pressure filter holder is satisfactory for flow of gas up to about 500 fpm at pressure differentials as high as 15,000 psi. Sampling procedure is obvious although at flows above 10 lpm (depending on apparatures used) there will be some unevenness of distribution of collected particulates which will require modification of technique for optical particle counting.

21.22.4 Sampling contamination. Sampling equipment, containers, analytical apparatus, and filters must be clean if subsequent measurements are to be a valid index of the fluid system being tested rather than extraneous or background contamination.

21.22.5 Sampling adequacy. Adequacy depends both upon the level of contamination of the system being measured and type of measurement being employed. In general, sufficient material must be sampled so that the collected contaminant will be clearly measurable at dangerous levels of contamination. Thus, 100 ml would represent an adequate sample from a high performance hydraulic system. The contamination should be measured by microscope (particle counting). One to five gallons is a proper sample for turbine fuel and is measured gravimetrically (weight of contaminant). A minimum of ten cubic feet shall be the measurement of the air taken as a sample in a clean room.

21.23 Sampling requirements.

21.23.1 Microscope selection. Selection of a microscope is dependent upon the size and number of particles to be counted. These factors in turn, require the proper lens combination, magnification, and type of microscope.

The two types of microscopes normally used in counting particles are binocular and stereo; each is specially suited for certain applications.

Stereo microscopes, as a rule, have larger numerical apparatures, a wider field range, and lower magnifications and resolving power factors than binocular microscopes. Because of these characteristics, the stereo microscope is more suitable for sizing and counting particles 100 microns and greater in size. In these ranges, resolving power is less important and the wide field range of the stereo microscope enables rapid scanning.

21.23.2 Guidelines in selecting proper magnification. The necessary magnifications for viewing particles are determined by the size of the particles to be counted. The following are general guidelines used to select proper magnifications.

Particle Size	Approximate Magnification
Less than 25 microns	100X
25-50 microns	100X
Greater than 50 microns	45X

21.23.3 Resolving power. When scanning particle size ranges less than 50 microns, resolving power becomes an important factor. Resolving power is defined as the ability to distinguish two adjacent points. Resolving power factor is determined by dividing the total magnification by the numerical apperture of the objective.

21.23.4 Calibrating ocular micrometer.

(a) Insert stage micrometer on microscope stage and bring into sharp focus using focus adjusting knobs.

(b) Adjust light source intensity and position for maximum definition of the stage micrometer as viewed through the microscope.

(c) Adjust ocular micrometer until it appears parallel to the stage micrometer and superimpose the ocular micrometer over the stage micrometer and align both scales at the zero marks.

(d) Read the total number of divisions on the stage micrometer subtended by by a specific number of divisions on the ocular micrometer scale.

(e) Using the following formula, calculate the number of microns represented by each division of the ocular micrometer:

$$N = \frac{Nm}{Ns}$$

Where N = Number of microns represented by each division of the ocular micrometer.

Nm= Number of microns subtended on the stage micrometer by a number of divisions of the ocular micrometer.

Ns= Number of divisions on the stage micrometer.

(f) Calibrate the ocular micrometer for each magnification of the microscope and post the following information on or near the microscope:

S/N______________power___________=______________microns

Tech_____________power___________=______________microns

Date_____________power___________=______________microns

21.24 Microscope scanning methods.

21.24.1 Absolute scanning method.

(a) Using the microscope's horizontal and vertical traversing stage adjustments, systematically scan and count all particles on the entire filtering area of the sample filter disc scanning each specified size range separately. The absolute scanning method is required for examination of certain sample filter discs because:

(1) It has been determined that samples taken with high pressure filter holders tend to collect particulate contaminants in an uneven distribution mostly near the O-ring sealing surface.

(2) Sample filter discs with low particulate population levels preclude any statistical count because of the probability of gross inaccuracies.

21.24.2 Statistical scanning method (for use on hydraulic samples). To obtain the number of particles of a given size range, the number of particles on a representative number of grid squares on the sample filter disc is counted. From this count, the total number of particles which would be present statistically on the total effective filtration area of 100 imprinted grid squares is calculated.

(a) Using the microscope's horizontal and vertical traversing stage adjustments, rapidly scan the sample filter disc to assure that particulate contaminant is evenly distributed. Estimate the particulate population before proceeding with a count.

(b) If the total number of particles of a given size range is estimated to be between 1 and 50, use the absolute scanning method.

(c) If the total number of particles of a given size range is estimated to be between 50 and 1000, count the number of particles within 20 randomly chosen grid squares. Multiply this number by 5 to obtain the total statistical particle count.

(d) If the total number of particles of a given size range is estimated to be between 1000 and 5000, count the number of particles within 10 randomly chosen grid squares. Multiply this number by 10 to obtain the total statistical particle count.

(e) If the estimated total number of particles of a given size range exceeds 5000, count the particles within at least 5 randomly chosen grid squares. To arrive at the total statistical count, the sum of the particles counted in the grids is multiplied by 20.

(f) Record results on appropriate data sheet.

21.25 Particle sizing and counting procedure.

(a) Select a microscope in accordance with specified requirements.

(b) Select eyepiece and lens objective combination in accordance with specified requirements.

(c) Using forceps, position sample filter disc on filter disc holder and place on microscope stage.

(d) Select correct magnification(s) to be used for sample filter disc examination according to methods previously stated.

(e) Determine if microscope has been calibrated within the previous six months for each magnification to be used. Calibrate according to methods previously described if any of the following conditions exist:

  (1) Microscope is new and has never been calibrated.

  (2) Microscope has not been calibrated within the preceding six months.

  (3) Damage has occurred, repairs or replacements made that might

have effected previous calibrations.

(f) Adjust microscope focus and oblique light source to obtain maximum clear particle definition.

(g) Size and count the particulate contamination according to one of the scanning methods previously specified. The correct method for scanning a specific sample filter disc shall be selected according to requirements.

(h) Cleanliness of the microscope and the area where it is used affect the results of filter disc examinations. The microscope shall be protected by a suitable covering when not in use. The CE examination area or laboratory shall conform to Mil-STD-1246A.

(e) Record results of particle count on appropriate data sheet and validate by initials or quality control stamp.

21.26 Determination of airborne particles in CE areas.

21.26.1 Equipment preparation.

(a) Petri dishes. Clean petri dishes by washing with wash bottle containing solvent.

(b) Forceps. Rinse forceps with solvent prior to each use.

(c) Wash bottle and Millipore solvent filtering dispenser (Catalogue No. XX66-025-00). Aerosol filter holder assembly shall be prepared by the following procedure.

(d) Thoroughly wash and rinse disassembled filter holder with prefiltered solvent from wash bottle.

(e) Using plastic or stainless steel tubing, connect outlet port of filter holder to inlet port of flowmeter and outlet port of flowmeter to vacuum source.

(f) Mount filter holder outlet plate on support stand in upright, vertical position approximately 36 inches from floor.

(g) Using cleaned forceps place prepared blank sample filter disc grid side up on screen support of filter holder outlet plate.

(h) Carefully assemble filter holder by placing inlet plate on outlet plate and twist to lock in place securing filter disc.

21.26.2 Ambient air analysis.

21.26.2.1 Blank sample.

(a) Using forceps, carefully remove one filter disc from its container and thoroughly rinse the grided surface of the filter disc with a forceful stream of solvent.

(b) Microscopically count and size all particulates on the filter disc according to procedure outlined in earlier discussion.

(c) Acceptable blank sample filter disc count shall not exceed limits specified in table below.

Size Range in Microns	Acceptable levels
5 - 25	10
25 -100	3
+100	0
Fibers	0

(d) Place acceptable filter disc in a clean petri dish, cover, and label as "blank sample" along with the particle count results.

21.26.2.2 Sampling. The ambient air is collected by impinging airborne particulate matter on a filter disc at bench level in close proximity to activity areas.

(a) Apply vacuum to filter holder assembly and adjust flow to 28 liters per minute.

(b) Allow test to continue for 10 minutes (total sample shall equal 10 cubic feet).

(c) Release vacuum.

(d) Twist to release locking ring of filter holder and remove inlet plate from outlet plate of the filter holder and vacuum train.

(e) Using forceps, carefully remove filter disc and place in a clean petri dish.

21.26.2.3 Filter disc analysis.

(a) Microscopically count and size all particulate on the filter disc.

(b) Subtract blank sample count from ambient air sample count and compare with allowable limits as specified.

(c) Record results in daily log.

21.27 Analysis of solvent samples for particulate contamination. Liquid analysis shall be performed as specified in this document to give an accurate picture of the contaminants present. Using a vacuum source, the sample is filtered through a filter disc impinging particulate contaminant upon the filter disc surface. The filter disc is then examined microscopically to count the particulate content. Record results on applicable form and validate with initials or quality control acceptance stamp.

21.27.1 Material requirements.

(a) CE laboratory meeting the requirements of Mil-Std-1246A.

(b) Halocarbon solvents and alcohol filtered to a 20 micron level (absolute).

(c) Demineralized water filtered to a 40 micron level (absolute) with a minimum specific resistance of 50,000 ohms and neutrality in pH range of 6.0 to 8.0.

(d) Filter disc shall be selected as applicable to the fluid being analyzed as follows:

  (1) Fuel and hydraulic fluids: Millipore filter disc, type HABG or type HAGG 047 mm.

  (2) Solvent and demineralized water: Whatman No. 42 filter disc or Millipore disc in fuel and hydraulic fluids.

21.27.2 Preparation of equipment.

21.27.2.1 Cleaning. Clean sample bottle and filter funnel as specified. Sample bottles shall be cleaned per step (a) below daily. Filter funnel shall be initially cleaned as specified in steps (a) through (f) below daily.

(a) Wash with solution of detergent and hot tap water.

(b) Rinse with hot tap water.

(c) Rinse twice with demineralized water.

(d) Rinse with prefiltered alcohol to remove moisture.

(e) Invert and allow to dry.

(f) Store inverted in designated area.

21.27.2.2 Analysis setup.

(a) Connect filtration flask to vacuum source.

(b) Insert rubber stopper and the fritted glass base of the filter holder into the opening at top of filtrator.

(c) Thoroughly rinse the inner wall of the filter funnel with wash bottle solvent.

(d) Using forceps remove one filter disc from its container.

(e) Prepare filter disc for media to be analyzed.

  (1) Millipore filter disc. Rinse grided surface of the filter disc with a forceful stream of solvent from wash bottle.

  (2) Whatman filter disc. While securely holding filter disc in a vertical position with forceps, snap or strike one side of the disc sharply to dislodge any particles that might be present.

(f) Place the filter disc on the fritted glass base of filter holder.

  (1) Milipore filter disc shall be positioned on the fritted glass base so that the grided surface is up and visible.

  (2) Whatman filter disc shall be positioned to make sure that the surface of the disc that was snapped or struck is positioned down on the fritted glass base.

(g) Place the filter funnel on the fritted glass base and secure with holding clamp.

21.27.3 Analysis procedure.

21.27.3.1 Blank sample analysis. When specified in applicable operating or acceptance procedures, perform a blank sample analysis. The blank sample analysis is performed prior to actual sample analysis to determine the accumulated amount of contamination being introduced by the sample bottle, filtering equipment, filter disc, solvent, and the filtration process. The method and amount of solvent used in the blank sample analysis is the same as that used in actual sample analysis.

(a) Perform blank analysis.

(b) Cover the petri dish and identify as blank sample including other pertinent information related to the specific liquid test sample to be analyzed such as date and technician.

21.27.3.2 Test sample analysis.

(a) Thoroughly agitate the test sample bottle to assure that all solid particles are in suspension.

(b) Remove cap from sample bottle.

(c) Apply vacuum to filtrator.

(d) Pour test sample into the filter funnel.

(e) When filtration of liquid test sample is complete, release vacuum.

(f) Using wash bottle, lightly rinse inner wall of filter funnel with solvent or alcohol.

Caution. Do not direct stream from wash bottle directly into filter disc.

(g) Apply vacuum to filter funnel until filtration is complete.

Note. The Millipore filter disc will change to a lighter shade of color when dry.

(h) Simultaneously remove holding clamp and filter funnel from the filter holder.

(i) Release vacuum.

(j) Using clean forceps, carefully remove the filter disc from fritted glass base and place in a clean filter holding device.

(k) Microscopically analyze the filter disc. Count particulate in various size ranges.

(l) Record results on appropriate form and validate with initials or quality assurance stamp.

21.27.4 Quality assurance sample requirement. Any combination of parts may be combined to provide one to five square feet of area. The analysis will be read to the most stringent requirement.

21.27.4.1 For components over 1.0 but less than 5.0 square feet in surface area. A test solution of 500 ml (minimum) shall be used to determine particle population and size and the nonvolatile residue on the critical surfaces of the quality control samples. The samples shall be selected from components having critical surface areas over 1.0 through 5.0 square feet.

21.27.4.2 For components with a surface area greater than 5.0 square feet. A maximum of 100 ml of test solution for each square foot of surface area shall be used to determine conformance to particle population and size and nonvolatile residue content. If more than 500 ml of test solution is used, the total used shall be thoroughly agitated and a 500 ml sample shall be taken for the analysis.

21.27.4.2.1 Alternate sampling procedure for components with surface areas greater than 5.0 square feet. Some customers may require an alternate sampling procedure for components having an internal surface area greater than five square feet.

(a) Take a sample equivalent to the compound volume.

(b) Agitate the sample and take a 500 ml representative sample.

(c) Process the sample per paragraph 20.27.3.2 and using the following formula calculate the number of particles in each category:

Actual number of particles per square foot =

$$\frac{\text{Total particles counted X Total Volume of Solution taken}}{\text{Number of square feet sampled X Total Volume of Solution analyzed.}}$$

21.27.5 Acceptance inspection.

21.27.5.1 Examinations (to be performed after parts have passed particulate and NVR requirements). Surfaces of all cleaned components that will contact the service medium shall be visually inspected for the presence of moisture and foreign matter such as corrosion, scale, dirt, oil, grease, and similar foreign matter. The presence of visible contamination shall be cause for rejection and shall necessitate recleaning of the component. Scale-free discoloration due to welding and passivation is permitted.

21.27.5.2 Acidity and alkalinity test (to be performed on final water rinse of precleaned parts). All surfaces that have been cleaned and flushed shall be tested for acidity and alkalinity with pH indicator paper while surfaces are wet from the final demineralized water rinse. When surfaces are dry, a few drops of demineralized water will satisfactorily wet the surfaces to permit testing. When tested, the pH value shall be within a range from 6.0 to 8.0 pH. Record results on applicable form and validate with initials or quality control acceptance stamp.

21.27.6 Procedure for determination of contamination level of hydraulic fluids. Contamination testing of hydraulic fluids shall be performed during cleaning of the fluid and prior to connection of test supplies to assemblies (pumps, actuators, servo valves, power packages, vehicle systems). Since the check on particle contamination in the fluid is the same for assemblies and systems the following procedure will apply:

21.27.6.1 Fluid sample. Obtain fluid sample as follows:

(a) Thoroughly clean sample point with filtered solvent from a wash bottle.

(b) Open sampling valve and allow a minimum of 100 ml of hydraulic fluid (300 ml when possible) to drain from the sample point into a waste container; then, without moving bleed valve catch 100 ml fluid in a pre-cleaned bottle.

21.27.6.2 Testing contamination level. To test the contamination level of hydraulic fluid and control sample, proceed as follows:

(a) Using unserrated forceps, remove one filter disc from its container. Rinse the grid-printed side of the filter disc with a stream of filtered solvent. Place the filter disc, grid-printed side up, on the fritted glass base.

(b) Rinse pyrex glass funnel with a stream of filtered solvent and immediately lower the filter funnel onto the fritter glass base and secure with holding clamp. Do not slide the filter funnel over the filter disc during assembly and disassembly of funnel.

(c) Apply a vacuum and draw the sample fluid through the filter disc. Dilution of the sample with reagent trichloroethylene is recommended.

(1) To determine environmental and laboratory analytical equipment contamination not attributable to the assembly or system that is being tested, periodically take a control sample to reflect cleanliness level of sample bottles, filtering equipment, and effectiveness of membrane rinsing.

(2) After 100 ml sample has passed through filter disc assembly, rinse the inner walls of the sample bottle with 10 to 15 ml of solvent; pass this solvent rinse through the filter disc.

Caution. Never strike the filter disc with the stream of the solvent.

(d) When the filter disc is dry, release vacuum and remove filter disc from filter holder and insert in a clean petri dish, cover, and label.

(e) Perform microscopic examination.

(f) Record number, size, and color of particles, and whenever possible, indicate whether the particles are metallic, nonmetallic, wax-like for each filter disc.

(g) If particle size limitations as specified are not met, cleaning procedures shall be repeated until requirements are met. Failure to meet specified requirements shall be cause for rejection.

21.28 Analysis of compressed gases for use in testing, drying, and purging.

21.28.1 Cleanliness level and inspection methods for drying and preservation gases. This document specifies the cleanliness requirements at gas utilization points for gases used in cleaning, testing, drying, and preservation operations on space vehicle systems, subsystems, assemblies, components, and support equipment. Gases having super-critical cleanliness requirements (such as for gas bearing) are specifically exempt. Included are the procedures, materials, and equipment required to verify that gas distribution system cleanliness is adequate to assure the specified cleanliness level of gases at the gas utilizing points.

21.28.1.1 General requirements.

(a) Prior to use for testing, drying, purging of space vehicle parts or components, gases shall be evaluated to assure that the required contamination levels are maintained. Gas distribution system components shall have been cleaned in accordance with pneumatic system requirements prior to installation. Lubricants used in valves or on fittings shall be approved LOX compatible materials and applied with discretion.

(b) Gas utilization points may be evaluated for contamination levels each time prior to use or qualified over a period of time and checked at regular intervals for reliability.

(c) The gas contamination level includes requirements for moisture and particulate content. All requirements must be satisfied for the gas to be acceptable for use.

(d) When any gas contaminant exceeds the specified limits, corrective action shall be initiated. Often the contamination level can be corrected by purging the system for a period of time at a high flowrate. If such purging is not effective, an effort shall be made to determine the contamination source. If the contaminant is not readily eliminated, the system shall be rejected and withdrawn from service until disposition is made.

21.28.2 System qualification requirements.

(a) Gas utilization points subject to periodic use only, as in pressure checkout complexes for stages or instrument units, shall be checked each time they are activated to assure contaminant levels are acceptable. Record results on applicable form and validate with initials or quality control acceptance stamp.

(b) Gas utilization points which are daily or continually used shall be tested to establish particulate, moisture, and hydrocarbon qualification. Periodically thereafter, they shall be tested to assure reliability. Record results on applicable form and validate with initials or quality control acceptance stamp.

(c) When, for any reason, a system is broken into or a reliability sample exceeds the specified limits, the affected system shall be requalified.

(d) Gas systems in the process of being qualified may be used provided all contaminant levels are acceptable prior to use.

(e) All utilization points in "bottle" system shall be checked each time bottles are replaced, in addition to regular checks. Record results on applicable form and validate with initials or quality control acceptance stamp.

21.28.3 Particulate and moisture analysis.

(a) Analyze the gas for particulate and moisture content daily for five consecutive working days. Record results on applicable form and validate with initials or quality control acceptance stamp.

(b) During the five day period if particulate and moisture content in any sample exceeds the specified limits start the period over.

(c) Once the gas moisture and particulate content has qualified for five consecutive days, reliability tests shall be made once weekly. Record results on applicable form and validate with initials or quality control acceptance stamp.

(d) Moisture content shall be analyzed by use of Alnor Dew Pointer, Model 7300, or equal. Moisture content shall be a minimum of minus 65 degrees Fahrenheit.

Note. Dew point will be performed on any component in which there is a possibility of water vapor being trapped.

21.28.4 Particulate content determination.

(a) Gas sample sizes shall be minimum of 30 standard cubic feet (scf), unless otherwise specified, and shall be filtered through a 10 micron absolute rated filter.

(b) Particulate content of the gas sample shall not contain particles greater than 10 microns.

(c) For systems with special particulate requirements, the system requirements shall dictate the limits.

(d) Solvents and alcohol used in this procedure shall be prefiltered to remove all particles greater than 50 microns. Use a wash bottle to dispense the solvent or alcohol.

21.28.5 Preparation of equipment. Equipment to be used in making particulate tests shall be made ready for use in a Class 100 CE area. When not in use, the equipment shall be stored in a CE area.

(a) Disassemble the high pressure filter holder by removing the four capscrews using the assembly tool. Separate the inlet plate from the outlet plate.

(b) Thoroughly wash and rinse the parts of the filter holder with abundant amount of alcohol or trichlorotriflouroethane. Be especially careful to rinse the inlet plate and port.

Note. If the filter holder is visibly contaminated, wash first using detergent and demineralized water.

(c) Cover the inlet port of the filter holder with several thicknesses of solvent-rinsed plastic film; secure with a rubber band.

(d) Using forceps, remove a filter disc from the container. Carefully rinse the grid marked surfaces of the filter disc with solvent.

(e) Perform blank analysis.

(f) With the back-up screen in place, position the blanked filter disc on the outlet plate of the filter holder, grid side up.

(g) Carefully position the inlet plate to the outlet plate to avoid moving the filter disc and install the four capscrews securely.

21.28.6 Sampling.

(a) Purge the sampling port at the rate of approximately 10 standard cubic feet per minute (scfm) for a minimum of two minutes prior to sampling. Systems not having 10 scfm flow capability shall be purged at the maximum flowrate.

(b) Reduce the flowrate to approximately 0.5 scfm.

(c) Remove the plastic film covering from the filter holder inlet port. Fold the film to protect the inside surfaces from contamination; save for reuse after sampling.

(d) Using the necessary fittings, carefully attach the inlet port to the sample port. Attach the flowmeter downstream of the filter holder.

(e) Increase the flowrate to between 7 and 8 scfm and allow to flow for 4 minutes or until a minimum of 30 scf has passed through the filter disc.

(f) Reduce the flowrate to approximately 0.5 scfm and disconnect the test apparatus.

(g) Replace the plastic film cover on the filter holder inlet port.

Note. Although particles should be impinged on the filter disc surface, do not jar filter holder. Hold the filter holder with the inlet port up.

(h) Reduce gas system flow to zero.

21.28.7 Analysis.

(a) Transport the filter holder to a Class 100 CE laboratory area.

(b) Holding the filter holder in an upright position, inlet port up, carefully remove the four capscrews.

(c) Carefully separate the inlet plate from the outlet plate.

(d) Using forceps, carefully remove the filter disc from the outlet plate and position it on the microscope filter disc holder.

(e) Examine filter disc under a microscope for particulate contamination.

(f) Count particles. Determine if the contamination level meets the particulate limit specified for the item being dried or tested.

21.29 Analysis of liquid samples for nonvolatile residue. The filtered sample, a 500 ml nominal volume shall be analyzed for nonvolatile residue content as follows:

(a) Transfer 500 ml of the solvent into a clean, degreased 800 ml beaker.

(b) Evaporate the solvent to 10 ml volume in a steam bath or by using a flash evaporator.

(c) Transfer the solvent to a constant weight (within 0.3 mg) aluminum moisture dish and weigh to the nearest 0.1 mg.

(d) Continue evaporation by placing aluminum moisture dish inside a constant temperature oven which has normalized at 225° + 5°F.

(e) Allow moisture dish to remain inside the oven until the solvent is evaporated to dryness; not to exceed a maximum of 1.5 hours.

(f) Remove the aluminum moisture dish from the oven, and place in a desiccator for cooling, a minimum of 5 minutes.

(g) After cooling, remove the aluminum moisture dish from the desiccator and weigh to the nearest 0.1 mg.

(h) Return the aluminum moisture dish to the oven, normalize at 225 225° + 5° F for 0.5 hours.

(i) Repeat paragraphs (e) and (f). If the difference in weights taken in paragraph (f) and (h) is greater than 0.3 mg, repeat paragraph (g), (e), and (f) until weighing difference of 0.3 mg or less is obtained.

(j) Compare results obtained from the used sample solvent with the results obtained from the unused solvent. The difference between the weights shall not be greater than the increase specified for the affected component (based on significant surface area.)

Note. In some instances, total drying cannot be accomplished. When this occurs, the sample contains excessive contaminants and shall be recorded as rejected along with other pertinent information as to color, amount of residue, overtime involved, etc.

21.30 Cleaning solvents, final sampling. All solvents used for final cleaning and sampling shall be tested to the requirements of the controlling specification prior to use.

21.31 Packaging, quality control. Packaging inspection shall be performed to assure correct packaging materials and methods.

21.32 Preparation and analysis of cleaning solution.

21.32.1 Preparation of standard solutions.

21.32.1.1 <u>0.3N - ferrous sulfate (Fe SO_4)</u>.

(a) Dissolve 83.5 g of $FeSO_4 \cdot 7H_2O$ in 700 ml of distilled H_2O. Add 60 ml of concentrated H_2SO_4 and cool the solution.

(b) Transfer the material to a liter volumetric flask and dilute to the mark with distilled water.

(c) Transfer to a dark colored stock bottle.

(d) To standardize, dry about 1.5g of potassium dichromate ($K_2Cr_2O_7$) at 110° C for 2 hours on a watch glass.

(e) After drying, transfer material to a stoppered weighing bottle and cool in a desiccator.

(f) Weigh 3 samples of 0.35 g each to four significant figures.

(g) Dissolve each sample in 25 ml distilled water in a 250 ml flask.

(h) Add 10 ml concentrated H_2SO_4 and cool the sample.

(i) Add 4-10 drops of o-phenathroline ferrous sulfate indicator.

(j) Titrate with the $FeSO_4$ to a reddish endpoint. Record the volume used for each sample.

(k) Calculate the normality of the $FeSO_4$ by the following equation:

$$N\text{-}FeSO_4 = \frac{\text{grams } K_2\,Cr_2\,O_7}{\text{ml } FeSO_4 \times 0.04904}$$

(l) Average the three volumes and express the normality to three significant figures. If the resultant normality is approximately 0.300, the $FeSO_4$ may be used without adjusting the normality.

(m) Mark the stock bottle as follows:

Material and normality
Date of Standardization

(n) Record results on applicable form and validate with initials or quality control acceptance stamp.

21.32.1.2 <u>IN - sodium hydroxide (NaOH)</u>.

(a) Dissolve 42 g of sodium hydroxide pellets, reagent grade, in 500 ml of recently boiled and cooled H_2O.

(b) Add enough $BaCl_2$ to precipitate any carbonate that may be present. Allow the $BaCl_2$ to settle.

(c) Filter through a #42 Whatman filter into a liter volumetric flask and dilute to the mark with CO_2 free distilled H_2O. Transfer to a stock bottle.

(d) To standardize, dry about 30 mg, of potassium acid phthalate ($KHC_8H_4O_4$) at 105°C for two hours on a watch glass.

(e) Weigh 3 samples of 8.6 g each to four significant figures.

(f) Dissolve each sample in 100 ml of recently boiled and cooled distilled H_2O in a 250 ml flask.

(g) Add 4-6 drops of phenolphthalein indicator to each and titrate with the NaOH with constant stirring, to a pink endpoint. Record the volume of NaOH used for each sample.

(h) Calculate the normality of the NaOH by the following:

$$\text{N-NaOH} = \frac{\text{grams } KHC_8H_4O_4}{\text{ml NaOH} \times 0.20423}$$

(i) Average the three volumes and express the normality to three significant figures. If the resultant normality is approximately 1.000, the NaOH may be used without adjusting.

(j) Mark stock bottle:

Material and normality
Date of standardization

(k) Record results on applicable form and validate with initials or quality control acceptance stamp.

21.32.1.3 <u>1N - hydrochloric acid</u>.

(a) Measure 84 ml of hydrochloric acid, reagent grade, approximately 12 N, into a liter volumetric flask. Dilute to mark with distilled water.

(b) Transfer to stock bottle.

(c) To standardize, pipet 3 samples of 10 ml each of standard 1N - NaOH into 250 ml erlenmeyer flask.

(d) Add 100 ml of distilled water and 4-6 drops of methyl orange indicator solution to each.

(e) Titrate with the HCl to an orange endpoint; shake constantly. Record the volume of HCI used for each sample.

(f) Calculate the normality of the HCl by the following equation:

$$N - HCl = \frac{10 \times N\text{-}NaOH}{ml\ HCl}$$

(g) Average the three volumes and express the normality to three significant figures. If the resultant normality is approximately 1.00 the HCl may be used without adjusting the normality.

(h) Mark the stock bottle as follows:

Material and normality
Date of standardization

(i) Record results on applicable form and validate with initials and quality control acceptance stamp.

21.32.2 Standardization of prepared solutions.

21.32.2.1 Make standard $FeSO_4$ (used to analyze HNO_3-HF mixture).

(a) Dissolve 176.5 g $FeSO_4 \cdot 7H_2O$ in 300 ml distilled water.

(b) Add 500 ml of 50% volume of H_2SO_4 (50% H_2SO_4 - mix 1 volume of concentrated H_2SO_4 with 1 volume of distilled water) stir constantly (cool if necessary).

(c) Transfer the material to a 1 liter volumetric flask and dilute to the mark with distilled water.

21.32.2.2 Make standard HNO_3.

(a) Dilute 41 ml concentrated HNO_3 to 1000 ml in 1 liter.

21.32.2.3 Standardize HNO_3.

(a) Pipet 20 ml of standard HNO_3 into a 500 ml flask. Add 50 ml of distilled water and 4-6 drops of methyl red indicator solution.

(b) Titrate with standard 1 N-NaOH to a yellow end point (indicator change from red to yellow). Record volume NaOH used.

(c) Calculation:

ml NaOH x 3.1508 = grams HNO_3/liter.

21.32.2.4 Standardize $FeSO_4$ to final factor for HNO_3.

(a) Measure 100 ml concentrated H_2HO_4 into a 500 ml flask. Place flask in a cooling bath and cool to ambient temperature.

(b) Pipet 10 ml of HNO_3 (standard HNO_3) slowly under the surface of the H_2SO_4 into the pipet and allowing to drain.

(c) Titrate with standard $FeSO_4$ to the brown or red ferroin end point. Record volume of $FeSO_4$ used.

(d) Calculation:

$$\text{Each ml } FeSO_4 = \frac{0.01 \text{ x g } HNO_3\text{/liter}}{\text{ml } FeSO_4 \text{ used}}$$

$$= \text{Factor for } HNO_3$$

21.32.3 Analysis of cleaning solutions.

Note. Titration of cleaning solution will be performed within the time interval specified.

21.32.3.1 Analysis of mixed nitric acid - hydrofluoric acid solution nitric acid (HNO_3).

(a) Determine specific gravity of sample at 60°F, record.

(b) Pipet 50 ml sample into a liter volumetric flask and dilute to the mark with distilled water.

(c) Measure 100 ml concentrated H_2SO_4 into a 500 ml flask.

(d) Pipet 20 ml of diluted sample (paragraph above) slowly under the surface of the H_2SO_4. Rinse the pipet by drawing some of the H_2SO_4 into the pipet and allowing to drain.

(e) Titrate with standard $FeSO_4$ to the brown or red ferroin end point. Record volume of $FeSO_4$ used.

(f) Calculate HNO_3

$$\frac{\text{ml } FeSO_4 \text{ x factor for } HNO_3 \text{ x 100 by wt.}}{\text{Sp. Gr.}} = \%HNO_3$$

(g) Record results on applicable form and validate with initials or quality control acceptance stamp.

21.32.3.2 Hydrofluoric acid (HF).

(a) Pipet 5 ml sample into a 500 ml flask and add 50 ml of distilled water. Add 4-6 drops of methyl red indicator solution.

(b) Titrate with standard 1 N-NaOH to a yellow endpoint (indicator change from red to yellow). Record volume of NaOH used.

(c) Calculation:

$$\frac{\dfrac{\text{ml NaOH x N-NaOH ml FeSO}_4\text{ x factor for HNO}_3\text{ x 2.001}}{\text{5 ml sample} \qquad \qquad 0.063}}{\text{Specific Gravity}} = \%\text{HF by wt.}$$

(d) Record results on applicable form and validate with initials and quality control acceptance stamp.

21.32.3.3 Analysis of nitric acid (HNO_3) (passivating bath) 5% by weight.

(a) Determine specific gravity of the solution at 60°F and record.

(b) Pipet 25 ml sample into flask and add 100 ml demineralized water.

(c) Add 4-6 drops methyl red indicator and titrate with standard 1 N-NaOH to yellow endpoint. Record volume of NaOH used.

(d) Calculation:

$$\frac{\text{ml NaOH x N-NaOH}}{\text{Sp. Gr. of Sample}} \text{ x } 0.252 = \%\ \text{HNO}_3 \text{ (pure) by wt.}$$

(e) Record results on applicable form and validate with initials or quality control acceptance stamp.

21.32.3.4 Analysis of nitric acid (HNO_3) 30% by weight).

(a) Determine specific gravity of the solution at 60°F and record.

(b) Pipet 5 ml sample into flask and add 100 ml demineralized water.

(c) Add 4-6 drops of methyl red indicator and titrate with standard 1 N-NaOH to yellow endpoint. Record volume of NaOH used.

(d) Calculations:

$$\frac{\text{ml NaOH x N-NaOH}}{\text{Sp. Gr. of Sample}} \times 1.260 = \%HNO_3 \text{ (pure) by wt.}$$

(e) Record results on applicable form and validate with initials or quality control acceptance stamp.

21.32.3.5 Analysis of trisodium phosphate ($Na_3\ PO_4\ 12H_2O$) solution.

(a) Determine specific gravity of the solution at 60°F and record.

(b) Pipet a 50 ml sample into a flask and add 100 ml demineralized water.

(c) Add 4-6 drops of phenolphthalein indicator and titrate with standard 1 N-HCl to a clear endpoint. Record volume of HCl used.

(d) Calculations:

$$\frac{\text{ml HCl x N-HCl}}{\text{Sp. Gr. of Sample}} \times 0.760 - \%\ Na_3\ PO_4\ 12H_2O \text{ by wt.}$$

(e) Record results on applicable form and validate with initials or quality control acceptance stamp.

21.32.3.6 Analysis of mixed nitric acid - sodium dichromate solution (passivating bath).

(a) Determine the specific gravity of the solution at 60°F and record.

(b) Record results on applicable form and validate with initials or quality control acceptance stamp.

21.32.3.7 Determine $Na_2\ Cr_2\ O_7$.

(a) Pipet a 10 ml sample into a 250 ml erlenmeyer flask and add 50 ml demineralized water.

(b) Add 10 ml of concentrated H_2SO_4 and cool the sample to ambient temperature.

(c) Add 4-6 drops of O-phenanthroline ferrous sulfate indicator and titrate with the standard 0.3 N-$FeSO_4$ used.

(d) Calculation:

$$\frac{\text{ml } FeSO_4 \text{ x N - } FeSO_4}{\begin{matrix}\text{Sp. Gr.}\\ \text{(Anhydrous)}\end{matrix}} = 0.437 = \%\ Na_2Cr_2\ O\ _7 \text{ (by wt.)}$$

(e) Record results on applicable form and validate with initials or quality control acceptance stamp.

21.32.3.8 Analysis of hydrochloric acid (HCl) solution.

(a) Determine specific gravity at 60°F.

(b) Pipet 10 ml sample into flask.

(c) Add 50 ml distilled water and 4-6 drops methyl red indicator.

(d) Titrate with standard NaOH (1.030 N) to a yellow endpoint. (red to yellow)

(e) Calculation:

$$\frac{\text{ml NaOH} \times 1.030\ \text{N} \times 0.036465}{\text{Sp. Gr.} \times 10\ \text{ml sample}} \times 100 - \%\ \text{HCl (by wt.)}$$

$$\frac{\text{ml NaOH}}{\text{Sp. Gr.}} \times \%\ \text{HCl (by wt.)}$$

21.33 Scrubber test (condensable hydrocarbon contamination).

(a) Clean the three gas washing bottles with liquid detergent and rinse thoroughly with demineralized or distilled water. Rinse again with acetone, dry, and rinse at least three times with previously blanked carbon tetrachloride. Do not dry with compressed air.

(b) Fill the gas scrubbing bottles with 100 ml of blanked distilled carbon tetrachloride and seal the tops with rubber bands. Connect the bottles in series with new rubber tubing. The tubing shall be approximately 1-1/2" long to prevent having excess tubing exposed to carbon tetrachloride vapor.

(c) The gas supply that is to be sampled shall have a flow rate of approximately four liters per minute when the test is first started. After a few minutes, increase the gas flow rate to five or six liters per minute or as prescribed. Use care to prevent the carbon tetrachloride from blowing from one scrubber to the other. If this occurs, begin test again using new rubber tubing for the connections.

(d) Connect the wet test meter to the exit side of the scrubbers before the test is started. The exhaust from the wet meter shall be vented to out-of-doors because of the toxicity of carbon tetrachloride.

(e) Do not permit the carbon tetrachloride to evaporate to less than 50 ml. When adding carbon tetrachloride to the scrubbers, release the rubber band on scrubber number 1, detach the inlet line, and immediately separate the top and bottom sections of the scrubber. This prevents the carbon tetrachloride in the scrubbers from blowing over due to internal pressure. Carbon tetrachloride can be added without completely removing the fritted tube from the scrubber. Reseal scrubber number 1 and proceed to scrubbers numbered 2 and 3 in the same manner.

Caution. Carbon tetrachloride is highly toxic. Avoid inhalation and prolonged or repeated contact with skin. Use with adequate ventilation. If carbon tetrachloride is heated to decomposition, phosgene and other dangerous gases are produced. These decomposition products must be avoided.

(f) After approximately 600 liters of gas have been scrubbed, release the pressure and remove all rubber connections from the bottles. Cover the bottles with aluminum foil until the solution is transferred to the beaker.

(h) Clean an 880 ml beaker in the same manner used to clean the gas scrubbing bottles. Transfer the sample quantitatively to the beaker by washing each section of the bottles at least three times with the blanked carbon tetrachloride.

(i) Evaporate the sample to approximately 2 ml using a low heat near the end of the evaporation cycle and transfer the sample to a 5 ml volumetric flask.

(j) Dilute the sample to a volume of 5 ml with distilled carbon tetrachloride, and determine the net absorption on an infrared spectrophotometer using the 3.42u band. Determine the concentration of oil in the sample by comparing the absorption value of the sample to the calibrated curve.

(k) Correct the volume of gas to standard conditions and calculate the weight from its density. Calculate the quantity of oil in the gas in ppm by weight. Use the following calculations to find the volume of gas at standard temperature and pressure, weight of gas, and ppm of oil in the gas:

$$\text{Volume of gas in liters} \times \frac{\text{barometric pressure in mm}}{760\ \text{mm}} \times \frac{273.1}{273.1 + \text{average temperature (degrees centigrade}} =$$

Volume of gas at standard temperature and pressure.

Volume of gas at standard temperature and pressure times density of gas = weight of gas.

$$\frac{\text{g of oil times } 1{,}000{,}000}{\text{weight of gas (g)}} = \text{ppm of oil in gas}$$

21.33.1 Rejection and resubmittal.

21.33.1.1 Individual rejection. Parts, field parts, assemblies, subsystems, or systems that fail the visual inspection or the test shall be rejected and reprocessed.

21.33.1.2 Lot rejection. If any acceptance sample fails to meet the requirements specified, a second sample shall be selected and tested. If any item from the second sample fails to meet the requirements, the entire lot represented by the sample shall be rejected.

21.33.1.3 Resubmittal. Before resubmittal of rejected lots, full particulars concerning previous rejection and corrective measures taken to prevent recurrence of the failure shall be made available.

21.34 Pre-clean inspection. Precision operation will be accomplished in accordance with the procedure in the instructions of the purchase order, specification or customer's request. All cleaning agents used will be in accordance with the specifications.

21.34.1 Pickling and passivation.

(a) Verification of solutions and their mixture.

(b) Verification of the time limitations for each solution.

21.34.2 Part disassembly. No part shall be disassembled without at least one of the following:

(a) Manufacturer's drawing or procedure.

(b) Customer's furnished procedure.

(c) Written customer approval to disassemble without either of the above.

21.34.3 After disassembly. When components are disassembled:

(a) Verify their condition and note it on contamination control process sheet.

(b) Insure that all parts for a specific component are kept together.

21.34.4 Degreasing.

(a) Insure teflon and metal parts are degreased in trichloroethylene.

(b) Insure rubber goods are degreased in isopropyl alcohol only.

21.34.5 Precleaning inspection includes:

(a) Visual inspection and aided visual inspection.

(b) pH factor of cleaned parts will be between 6.0 - 8.0.

21.35 Inspection of final cleaning and analytical sampling.

(a) Assure cleaning to correct specification per customer's purchase order.

(b) Assure drying to correct dewpoint (-65°F minimum) if required.

(c) Assure sampling for:

  (1) Particle analysis.

  (2) Non-volatile residue content if required.

(d) Record results above on the contamination control process sheet.

(e) The analytical laboratory will be operated by a quality control technician.

21.35.1 Component functional test. Persons performing functional test on valves, hoses, filters, etc., shall exercise caution at all times; maintain to the best of their knowledge and ability, utilizing equipment available to the fullest extent, a safe test condition. During a functional test, only the personnel involved in the test will be in the functional test room.

21.35.1.1 Test cell and final assembly area.

(a) Reassembly: Component shall not be reassembled without at least one of the following:

  (1) Manufacturer's drawing or procedure.

  (2) Customer's furnished procedure.

  (3) Written customer approval to reassemble without either of the above.

(b) Test: Components which require functional tests will be tested in accordance with applicable drawings, specification, or customer's request.

21.36 References.

21.36.1 Government.

21.36.1.1 Federal.

1. Ethyl Alcohol (Ethanol); Denatured Alcohol and Proprietary Solvent, O-E-760.
2. Nitrogen, BB-N-411.
3. Plastic Films, Polyethylene Type II, L-P-378.
4. Trichloroethylene, Technical Grade, O-T-634.

21.36.1.2 Military.

1. Aluminum Foil, Mil-A-148.
2. Nitrogen, Propellant Pressurizing, Mil-P-27401.
3. Nonionic Detergent, Mil-D-16791.

21.36.1.3 George C. Marshall Space Flight Center.

1. Carbon Tetrachloride Scrubber Method for Analysis of Condensable Hydrocarbon Contamination of Compressed Gases, MSFC-Proc-245.
2. Cleanliness Level Requirements and Inspection Methods for Determining Cleanliness Level of the Bearing Gas Supply and Flash Measuring Systems, MSFC-Proc-195.
3. Cleanliness Levels, Cleaning and Inspection Procedures for Component Parts of Gas Bearing and Flash Measuring Systems, Specification For, Drawing #10419906.
4. Cleanliness Levels, Cleaning Protection and Inspection Procedure for Parts, Field Parts, Assemblies, Subsystems, and Systems for Pneumatic Use in Support Equipment, Specification for, Drawing #10M01671.
5. Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems, MSFC-Spec-164.
6. Contamination Control and Environmental Protection of Space Launch Vehicles and Associated Equipment, Procedure For, MSFC-Proc-151.

7. Design and Operational Criteria of Controlled Environment Areas, Standard For, MSFC-Std-246.

8. Gases, Drying and Preservation, Cleanliness Level, and Inspection Methods, MSFC-Proc-404.

9. Hydraulic System Components and Hydraulic Fluids for Space Vehicles, Cleaning, Testing, and Handling, MSFC-Proc-166B.

10. Nitrogen, Space Vehicle Grade, MSFC-Spec-234.

11. Procedure for Cleaning, Testing, and Handling of Onboard Hydraulic System Components and Mil-H-5606 Hydraulic Fluid, Drawing #20M85015.

12. Solvent, Precision Cleaning Agent, MSFC-Spec-237A.

21.36.1.4 National Aeronautics and Space Administration.

1. Inspection System Provisions for Suppliers of Space Materials, Parts, Components and Services, NCP-200-3.

2. Quality Program Provisions for Space System Contractors, NPC-200-2.

21.36.1.5 U. S. Army Missile Command.

1. Cleanliness of Components for Use in Liquid Oxygen, Fuel, and Pneumatic Systems, Specification, Drawing #10509305.

21.36.2 Nongovernmental.

1. Cleaning Facility (Document "A"), TMC-Spec-6200.

2. Detail Procedures (Document "D") for Contractors Individual Requirements, TMC-Spec-1000X.

3. General Solution Handing, Cleaning and Sealing Procedures (Document "B"), TMC-Spec-6201.

4. Procedure for the Determination of Particulate Contamination of Air in Dust Controlled Spaces by the Particle Count Method, ARP 743, Society of Automotive Engineers.

5. Procedure for the Determination of Particulate Contamination of Hydraulic Fluids by the Particle Count Method, ARP 598, Society of Automotive Engineers.

6. Test Method for Determining the Degree of Cleanliness of the Downstream Side of Filter Elements, Procedure For, ARP 599. Society of Automotive Engineers.

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APPENDIX I
RECENT DEVELOPMENTS

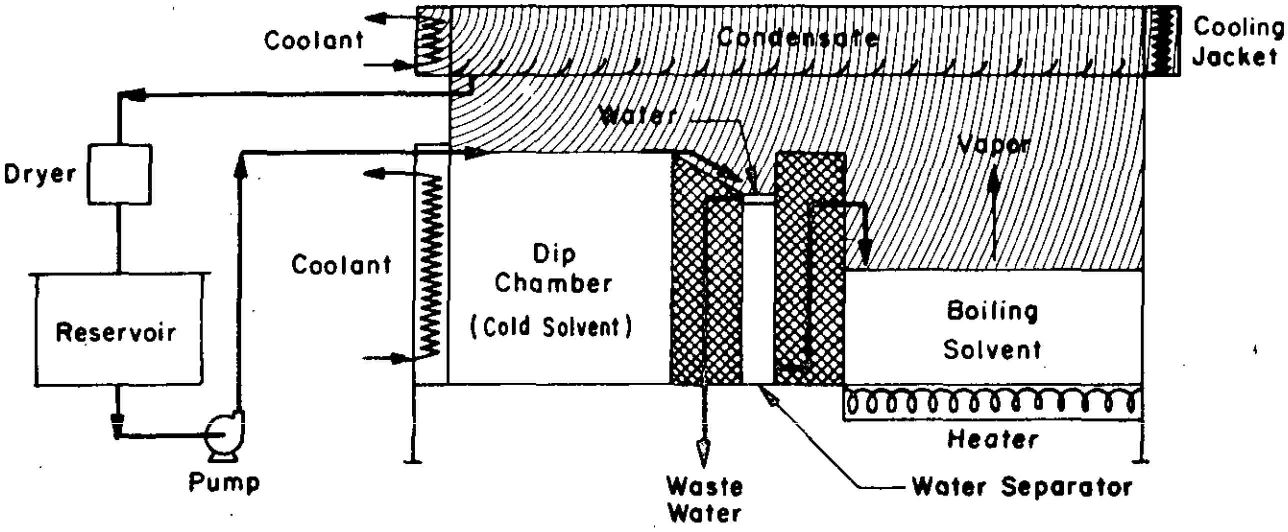
A dewatering process recently developed by Allied Chemical Corporation, Genesolv Solvent Drying System, can be used for removal of water from electronic devices. This system is based on water displacement principle.

The system is unique in that it uses only one solvent D-1,1,2 trichloro-1,2,2 triflouroethane (Genesolve) and simple equipment. The solvent does not dissolve the water, but replaces it with a solvent film which in turn evaporates at a temperature below 120°F. The system is designed in such a way that replaced water is continuously removed. As the solvent level drops, more can be added to restore the level.

This drying system is excellent for dewatering:

a) *Integrated circuits*
b) Printed circuit boards
c) Optical lenses
d) Silicon wafers
e) Glass plates
f) Metal parts

A schematic of the system is shown below.



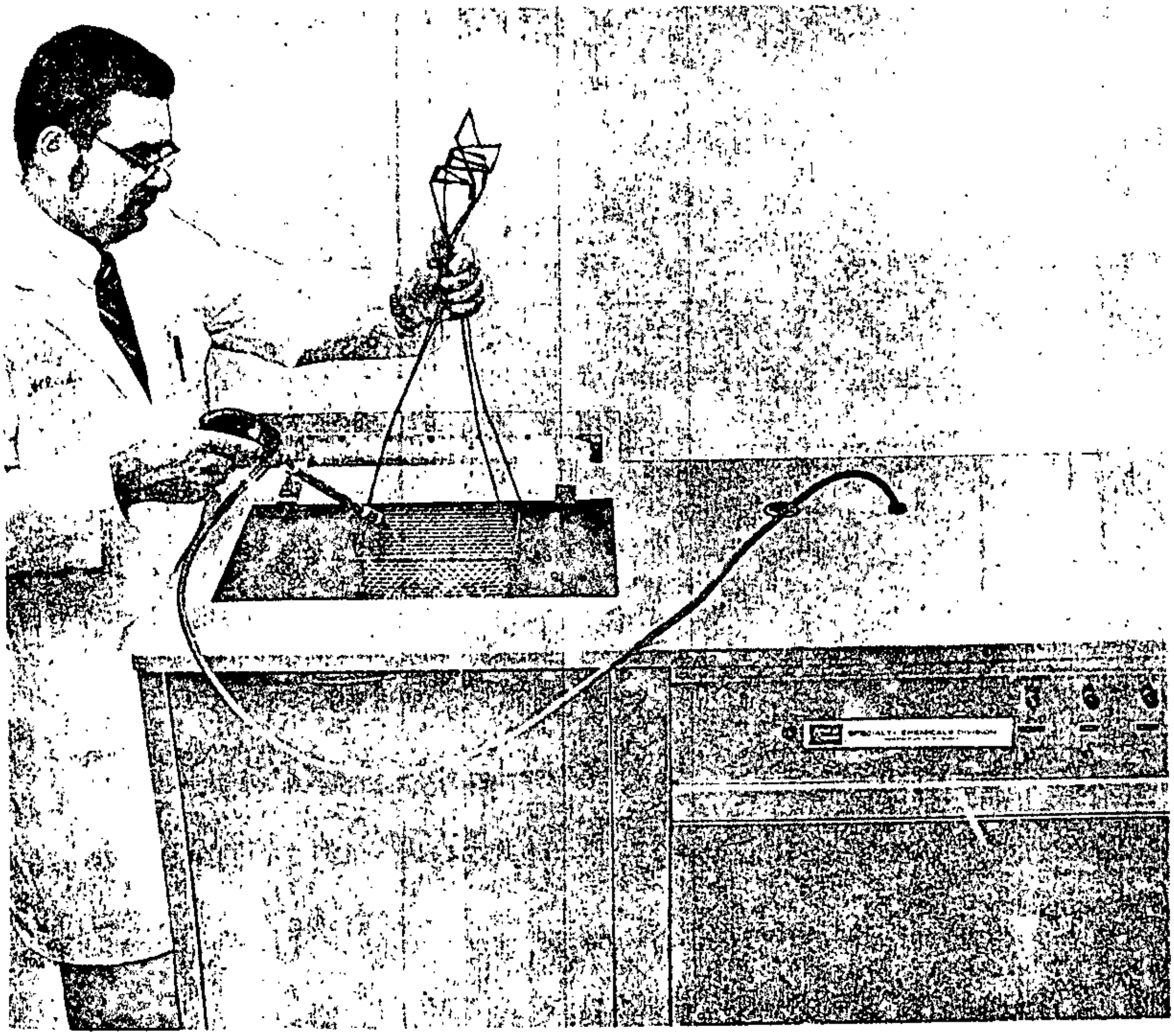
Suggested drying procedure. Different substrates require different drying procedures. Metal parts have high heat capabities and thus dry most easily. Silicon and glass have lower heat capacities and hydrophilic surfaces and require longer exposures to the drying solvent. The following procedures have produced satisfactory dewatering of metals, silicon and glass:

Metal parts.

a) Place parts in an appropriate carrier.
b) Dip in cold solvent sump for 30 seconds.
c) Hold in vapor phase for 1-2 minutes.
d) Remove from drying system.

Silicon and glass parts.

a) Place parts in an appropriate carrier.
b) Hold in vapor phase for 15 seconds.
c) Hold in solvent spray for 15 seconds.
d) Dip in solvent for 15 seconds.
e) Hold in solvent spray for 15 seconds.
f) Hold in vapor phase for 30 seconds.
g) Remove from drying system.



APPENDIX II

USEFUL CHARTS

Square footage chart

Fittings, number of fittings/sq. ft. of surface area

SIZE	CAPS	TEES	ELLS	UNIONS
1/8	120	56	65	75
1/4	77	33	46	66
3/8	52	28	42	56
1/2	30	19	30	36
5/8	26	10	15	20
3/4	17	7	10	14
1	14	4	7	9

Flex hose, smooth feet of hose/sq. ft. of surface area

SIZE	1/8"	1/4"	3/8"	1/2"	5/8"	3/4"	1"
FEET	30.6	15.3	10.2	7.6	6.2	5.1	3.8

Flex hose, convoluted

SIZE	1/8"	1/4"	3/8"	1/2"	5/8"	3/4"	1"
FEET	15.3	7.7	5.1	3.6	3.1	2.6	1.7

Tubing, feet of tubing/sq. ft. of surface area

SIZE		1/8"	1/4"	3/8"	1/2"	5/8"	3/4"	1"
GAUGE	.035	69	21	12	8.9	6.8	5.6	4.1
	.049	139	25	14	9.5	7.3	5.9	4.3
	.065		32	15.5	10.3	7.5	6.2	4.4

Square footage chart (cont'd)

Valves, hand, solenoid, check

SIZE	1/4"	1/2"	3/4"	1"
FEET	1/4	1/3	2/3	1

Regulators, 1/3 sq. ft. (most methods)

O-Rings, 5 or less = 1/10 sq. ft. (small)

"O" Rings approximate square footage chart for use on "O" rings approx. 1/8" thick

SIZE	NO. equal to 1 Sq. Ft.
1/2"	160
1	90
1 1/2"	63
2"	50
2 1/2"	41
3"	35
3 1/2"	30
4"	26
4 1/2"	23
5"	22
5 1/2"	19
6"	18

Note: For purpose of particulate and NVR results, 5 or less small "O" Rings = 1/10 sq. ft.

Informational tubing data

Tube size (inches)	Tube I.D. (inches)	Volume/100 ft.			Wt. of trichloroethylene in 100 ft. column (lbs)	Static pressure 168 ft. column (psi)
		(gal.)	(in.^{3})	(ft.^{3})		
1/4	.180	.180	30.4	.0176	1.62	107.5
5/16	.2425	.24	55.2	.0319	2.93	107.5
3/8	.305	.38	87.0	.0505	4.65	107.5
1/2	.430	.76	174.0	.101	9.3	107.5
5/8	.555	1.27	292.0	.169	15.5	107.5
3/4	.652	1.75	402.0	.233	21.4	107.5
7/8	.777	2.47	568.0	.329	30.2	107.5
1	.902	3.35	768.0	.445	40.8	107.5
1 1/4	1.152	5.45	1250.0	.725	66.5	107.5
1 1/2	1.370	7.8	1775.0	1.03	95.0	107.5
1 3/4	1.620	10.8	2470.0	1.43	131.0	107.5
2	1.870	14.4	3300.0	1.91	176.0	107.5
3	2.870	33.9	7770.0	4.5	414.0	107.5
4	3.810	60.0	13700.0	7.95	730.0	107.5
5	4.813	95.0	21800.0	12.6	1160.0	107.5
6	5.761	136.0	31500.0	18.1	1665.0	107.5

Rigid tubing inside surface area (sq. ft.)

O.D.	Wall thk.	Surface area for lengths specified					
		1 ft.	2 ft.	3 ft.	4 ft.	5 ft.	6 ft.
.125	.020	.0222	.0445	.0667	.0890	.1112	.1335
.250	.020	.0549	.1099	.1649	.2199	.2748	.3298
.250	.035	.0471	.0942	.1413	.1884	.2355	.2826
.250	.049	.0398	.0796	.1194	.1592	.1989	.2388
.375	.028	.0835	.1670	.2505	.3341	.4176	.5011
.375	.035	.0798	.1597	.2395	.3194	.3992	.4791
.375	.049	.0725	.1450	.2175	.2900	.3626	.4351
.500	.035	.1126	.2251	.3377	.4503	.5629	.6754
.500	.065	.0968	.1937	.2906	.3874	.4843	.5812
.750	.049	.1707	.3414	.5121	.6828	.8535	1.0241
.750	.095	.1466	.2932	.4398	.5864	.7330	.8796
1.000	.035	.2435	.4870	.7304	.9739	1.2174	1.4608
1.000	.049	.2361	.4723	.7084	.9446	1.1807	1.4168
1.000	.065	.2778	.4555	.6833	.9111	1.1388	1.3666
1.250	.035	.3089	.6178	.9268	1.2357	1.5446	1.8535
1.250	.049	.3016	.6032	.9048	1.2064	1.5080	1.8096
1.250	.065	.2932	.5864	.8796	1.1728	1.4660	1.7562

Rigid tubing inside surface area (sq. ft.) (continued)

O.D.	Wall thk.	Surface area for lengths specified						
		7 ft.	8 ft.	9 ft.	10 ft.	20 ft.	30 ft.	100 ft.
.125	.020	.1557	.1780	.2002	.2225	.4450	.6675	2.225
.250	.020	.3848	.4398	.4947	.5497	1.0995	1.6492	5.497
.250	.035	.3298	.3769	.4240	.4711	.9422	1.4135	4.711
.250	.049	.2786	.3183	.3581	.3979	.7959	1.1938	3.979
.375	.028	.5646	.6681	.7516	.8351	1.6703	2.5054	8.351
.375	.035	.5589	.6388	.7186	.7985	1.597	2.3955	7.985
.375	.049	.5076	.5801	.6526	.7251	1.4503	2.1755	7.251
.500	.035	.7880	.9006	1.0132	1.1257	2.2515	3.3772	11.257
.500	.065	.6780	.7749	.8717	.9685	1.9371	2.9057	9.685
.750	.049	1.1948	1.3655	1.5362	1.7069	3.4138	5.1207	17.069
.750	.095	1.0267	1.1728	1.3194	1.4660	2.9321	4.3982	14.660
1.000	.035	1.7043	1.9478	2.1913	2.4347	4.8695	7.2992	24.347
1.000	.049	1.6529	1.8891	2.1252	2.3614	4.7228	7.0842	23.614
1.000	.064	1.5944	1.8221	2.0499	2.2777	4.5553	6.8330	22.777
1.250	.035	2.1625	2.4714	2.7803	3.0892	6.1785	9.2677	30.892
1.250	.049	2.1112	2.4127	2.7143	3.0159	6.0319	9.0478	30.159
1.250	.065	2.0525	2.3457	2.6389	2.9321	5.8643	8.7964	20.321

Flow rates for flushing and circulation operations

Tube size (inches)	Tube I. D. (inches)	Velocity (ft./sec)	Flow (gpm)	Pressure drop/100 ft (psi)
1/4	.180	4	.317	27.3
5 5/16	.2425	4	.58	18.1
3/8	.305	4	1.0	14.0
1/2	.430	4	2.0	8.8
5/8	.555	4	3.4	6.4
3/4	.652	4	4.0	5.4
7/8	.777	4	6.4	4.3
1	.902	4	8.8	3.6
1 1/4	1.152	4	14.0	2.57
1 1/2	1.370	4	20.0	2.07
1 3/4	1.620	4	30.0	1.715
2	1.870	4	35.0	1.385
3	2.870	4	84.0	.86
4	3.810	4	156.0	.61
5	4.813	4	200.0	.46
6	5.761	4	300.0	.36

Note: It is recommended that tubing and pipe larger than 2 inches be cleaned prior to assembly and installed as a clean part.

Conversion chart

Water Vapor By Volume PPM	Dewpoint Temp. °F	Water Vapor By Volume PPM	Dewpoint Temp. °F	Water Vapor By Volume PPM	Dewpoint Temp. °F	Water Vapor By Volume PPM	Dewpoint Temp. °F
6	-83.5	20	-67.5	60	-51.8	180	-34.5
7	-82.0	24	-65.0	70	-49.5	200	-33.0
8	-80.0	28	-63.0	80	-47.0	250	-29.0
10	-78.0	30	-62.0	90	-45.5	300	-26.0
12	-74.5	35	-59.5	100	-44.0	400	-21.0
14	-72.5	40	-57.7	120	-41.0	500	-17.0
16	-70.5	45	-56.0	140	-38.5	600	-14.0
18	-69.0	50	-54.0	160	-36.1	700	-11.0

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IDENTIFICATION OF INTEREST IN DOCUMENT BY ACTIVITY

Custodians:

Army - MI
Navy - AS
Air Force - 84

Review Activities:

Army - EL, MR, MU, WC, AT, AV, ME
Navy - AS, SA, SH
Air Force - 11, 12, 13, 15, 16, 17, 19, 43, 79, 84

User Activity:

Army
Navy
Air Force

Preparing Activity:

Army - MI

Project No. - FSC 3694-0025

Defense Supply Agency:

GS, PS, ES

Civilian Agencies:

NASA - MSFC, KSC, MSC, GSFC
DOT - FAA
GSA

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STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

(See Instructions – Reverse Side)

1. DOCUMENT NUMBER

2. DOCUMENT TITLE

3a. NAME OF SUBMITTING ORGANIZATION

b. ADDRESS *(Street, City, State, ZIP Code)*

4. TYPE OF ORGANIZATION *(Mark one)*

☐ VENDOR

☐ USER

☐ MANUFACTURER

☐ OTHER *(Specify)*: ____________

5. PROBLEM AREAS

a. Paragraph Number and Wording:

b. Recommended Wording:

c. Reason/Rationale for Recommendation:

6. REMARKS

7a. NAME OF SUBMITTER *(Last, First, MI)* – Optional

b. WORK TELEPHONE NUMBER *(Include Area Code)* – Optional

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