



## Standard Practice for Designing a Process for Cleaning Technical Glasses<sup>1</sup>

This standard is issued under the fixed designation C 912; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This practice covers information that will permit design of a rational cleaning procedure that can be used with a glass that is somewhat soluble in many aqueous chemical solutions. Typically, this type of glass is used in applications such as optical ware, glass-to-metal seals, low dielectric loss products, glass fibers, infrared transmitting products, and products resistant to metallic vapors.

1.2 In most cases, this type of glass contains high concentrations of oxides that tend to react with a number of aqueous chemicals. Such oxides include  $B_2O_3$ ,  $Al_2O_3$ ,  $R_2O$ ,  $RO$ ,  $La_2O_3$ ,  $ZnO$ ,  $PbO$ ,  $P_2O_5$ , and  $Fe_2O_3$ . The more conventional high-silica glasses are usually more chemically resistant, but the cleaning principles outlined here also apply to them.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 4 and Table 1.

### 2. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

2.1.1 *technical glass*—glasses designed with some specific property essential for a mechanical, industrial, or scientific device.

### 3. Significance and Use

3.1 Many of the low-silica technical glasses which contain soluble or reactive oxides require processing or involve applications that require cleaning. Very often these cleaning procedures have evolved over several decades and are considered an art. They usually contain numerous steps, some of questionable validity. It is the premise of this practice that cleaning glass can be more scientific. Design of a cleaning procedure should involve (1) a definition of the soil to be removed, (2) an awareness of the constraints imposed by the glass composition, and (3) a rational selection of alternative methods that will remove the soil and leave the glass in a condition suitable for

its intended application. This practice provides information to assist in step (3). General references on glass cleaning and on various methods of evaluating cleanliness and associated information has been published.<sup>2</sup>

### 4. Hazards

4.1 Many of the chemicals that can be used in cleaning glass are hazardous. This is true of most of the aqueous chemicals discussed in Section 5 and shown in Table 1 as well as the organic chemicals discussed in Section 6.<sup>3</sup>

4.2 Special care should be used with hydrofluoric acid (HF), which will react with glass generating heat. The vapors as well as the liquid destroy dermal tissue and can be fatal if inhaled.

4.3 Concentrated acids can react violently if water is added into them. When it is necessary to dilute acid, add the acid to the water slowly and with constant stirring so that heat is never allowed to concentrate locally in the solution.

4.4 Organic solvents may be flammable or toxic, or both. Threshold limit values for some common solvents are shown in Table 2. Note that the fluorocarbons are most likely to exhibit toxic effects as a result of inhalation or skin absorption. Benzene is not recommended as a solvent since it is a known carcinogen.

### 5. Aqueous Solvents

5.1 *Selection*—In using aqueous solvents for cleaning, generally two extreme choices are available. One is to select an aqueous system that dissolves the soil to be removed, but has little effect on the glass. The other is to select a system that dissolves the glass uniformly, thus undercutting the soil and leaving a chemically polished glass surface. It is best to avoid a solvent that selectively attacks the glass, dissolving only some components, or a solvent that produces a precipitate that adheres to the surface to be cleaned.

5.2 *Minimum Glass Dissolution:*

5.2.1 Water is the most frequently used aqueous solvent. Even this can attack some glasses appreciably.

5.2.2 Try to choose an aqueous system that completely removes the soil with minimal effect on the underlying glass. Obviously, to achieve this the glass composition must be

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<sup>2</sup> Campbell, D. E., and Adams, P. B., "Bibliography on Clean Glass: Supplement 1," *Journal of Testing and Evaluation*, Vol14, No. 5, September 1986 pp. 260–265.

<sup>3</sup> A useful reference is the *Handbook of Laboratory Safety*, ed., CRC Press, Inc., 2255 Palm Beach Lakes Blvd, West Palm Beach, FL 33409.

**TABLE 1 Relative Solubility of Various Glass Component Oxides in HF, Other Inorganic Acids, and NaOH, in Concentrated Solutions at Room Temperature**NOTE 1—Macro or minor/trace levels will determine degree of precipitation, especially in acids, for example, HNO<sub>3</sub> (Sn, Sb, Mo).NOTE 2—W is soluble in acid but heat may precipitate it, for example, H<sub>2</sub>WO<sub>4</sub>.NOTE 3—Sn<sup>+4</sup> is soluble in hot H<sub>2</sub>SO<sub>4</sub>; Sn<sup>+2</sup> is soluble in other reagents as well.

NOTE 4—Most alkali solutions must be hot to effect solution.

NOTE 5—PbSO<sub>4</sub> is soluble in *hot* concentrated H<sub>2</sub>SO<sub>4</sub>.

NOTE 6—Sb and Bi form insoluble oxychlorides in dilute HCl.

NOTE 7—Ba is insoluble in concentrated HNO<sub>3</sub>.

Oxides of	HF 49 %	H <sub>2</sub> SO <sub>4</sub> 96 %	HNO <sub>3</sub> 70 %	HCl 37 %	HBr	HI	H <sub>3</sub> PO <sub>4</sub> 85 %	NaOH 50 %
Al	s <sup>A</sup>	s	s	s	i	i	i	s
Sb	i <sup>A</sup>	i	i	s	s	s	i	s
As	s	s	s	s	s	s	s	s
Ba	i	i	s	s	s	s	s	s
Be	s	s	s	s	s	s	s	i
Bi	s	s	s	s	s	s	s	i
B	s	s	s	s	s	s	s	s
Cd	s	s	s	s	s	s	s	s
Ca	i	s	s	s	s	s	s	s
Ce	i	s	i	i	i	i	i	i
Cr	i	i	i	i	i	i	i	i
Co	s	s	s	s	s	s	s	i
Cu	s	s	s	s	s	s	s	i
Er	i	s	s	s	s	s	s	i
Eu	i	s	s	s	s	s	s	i
Gd	i	s	s	s	s	s	s	i
Ga	s	s	s	s	s	s	s	i
Ge	s	s	s	s	s	s	s	s
Au	i	i	i	i	i	i	i	i
Hf	s	i	i	i	i	i	i	i
Fe	s	s	s	s	s	s	s	i
La	i	s	s	s	s	s	s	i
Pb	i	i	s	i	i	i	s	s
Li	s	s	s	s	s	s	s	s
Mg	i	s	s	s	s	s	s	i
Mn	s	s	s	s	s	s	s	i
Mo	s	s	i <sup>B</sup>	s	s	s	s	s
Nd	i	s	s	s	s	s	s	i
Ni	s	s	s	s	s	s	s	i
Nb	s	i	i	i	i	i	i	i
Pd	s	s	i	i	i	i	i	i
P	s	s	s	s	s	s	s	s
Pt	i	i	i	i	i	i	i	i
K	s	s	s	s	s	s	s	s
Pr	i	s	s	s	s	s	s	i
Pm	i	s	s	s	s	s	s	i
Rh	i	s	s	s	s	s	s	i
Rb	i	s	s	s	s	s	s	i
Ru	i	s	s	s	s	s	s	i
Sm	i	s	s	s	s	s	s	i
Se	s	s	s	s	s	s	s	s
Si	s	i	i	i	i	i	i	s
Ag	s	s	s	i	i	i	s	i
Na	s	s	s	s	s	s	s	s
Sr	i	i	i	i	i	i	i	i
Ta	s	i	i	i	i	i	i	i
Te	s	s	s	s	s	s	s	s
Tl	s	s	s	s	i	i	s	i
Th	s	s <sup>B</sup>	i	i	i	i	i	i
Sn	s	s	s	s	s	s	s	s
Ti	s	s <sup>B</sup>	i	s	i	i	i	i
W	s	i	i	i	i	i	i	s
U	s	s	s	i	i	i	i	i
V	s	s	s	s	s	s	s	s
Yb	i	s	s	s	s	s	s	i
Y	i	s	s	s	s	s	s	i
Zn	s	s	s	s	s	s	s	s
Zr	s	s <sup>B</sup>	i	i	i	i	i	i

<sup>A</sup> s = relatively soluble, i = relatively insoluble.<sup>B</sup> hot

**TABLE 2 Threshold Limit Values for Some Common Solvents**

	TLV, ppm <sup>A</sup>
1,1,2-trichloro-1,2-trifluoroethane	1000
Acetone	750
Ethyl alcohol	1000
<i>n</i> -Hexane	50
Isopropyl alcohol	400
Methyl chloroform	350
Perchloroethylene	50
Trichloroethylene	50
Methylene chloride	100
Carbon tetrachloride	5

<sup>A</sup> The TLV values establish parts per million by volume of solvent vapors allowed in air for a normal work week of 8 h a day, 5 days a week. These are standards set by the American Conference of Governmental Industrial Hygienists, and the values shown in this table were effective in 1984–1985. The most recent recommended values should be consulted in “TLV’s<sup>R</sup> Threshold Limit Values for Chemical Substances and Physical Agents in the Work Environment and Biological Exposure Indices with Intended Changes for 1984–1985,” published by ACGIH, 6500 Glenway Ave., Bldg D-5, Cincinnati, OH 45211.

known. However, one cannot simply calculate glass solubility in a specific reagent. Reference to **Table 1** will then help determine if an aqueous solvent exists that will not attack the glass. The table provides guidance in selecting a solvent, but trial and error will usually be necessary also. Individual glass components do not act independently with specific solvents, in most cases, as described in **5.2.3**.

**5.2.3** It is not necessary that the glass contain absolutely none of the components that are soluble in the chosen reagent. For instance, a glass containing 80 % SiO<sub>2</sub> and 5 % Na<sub>2</sub>O could be cleaned in H<sub>2</sub>SO<sub>4</sub> without appreciable glass attack even though Na<sub>2</sub>O is very soluble in H<sub>2</sub>SO<sub>4</sub>; however a glass containing 50 % SiO<sub>2</sub> and 25 % Na<sub>2</sub>O would probably show considerable attack by H<sub>2</sub>SO<sub>4</sub>. Often this can only be determined by trial.

### 5.3 Uniform Glass Dissolution:

**5.3.1** It may be necessary to select a system that uniformly attacks the glass either because there is no other solvent for the soil or there is no solvent available that does not attack the glass. For glasses containing substantial concentrations of silica, HF or HF plus some other reagent may be a good choice. HF can often be used for cleaning provided there are no glass components that form insoluble fluorides. For non-silicate glasses, some other reagent would probably be appropriate. **Table 1** is a general guide to selection of such reagents.

**5.3.2** There are two further modifications that can allow the successful use of HF even if insoluble products form. One is to combine chemical cleaning with a mechanical cleaning process either simultaneously or sequentially. The other is to mix the HF with another acid to achieve complete solution of all products.

**5.3.3** Alkali solutions can be used as a glass solvent for cleaning, but, in most cases, it will be necessary to use them hot to achieve a sufficiently rapid reaction.

**5.3.4** Many glasses can be cleaned by the uniform dissolution process without the use of HF or alkali. Reference to **Table 1** will suggest the types of glasses to which this approach is applicable. For instance, a glass containing 60 % PbO and less than 15 % SiO<sub>2</sub> could probably be cleaned in this way with HNO<sub>3</sub>, particularly if mechanical action by polishing or rubbing is used.

### 5.4 Other Possibilities:

**5.4.1** When all else fails, organic complexing agents, either alone or in combination with other chemicals, may succeed in removing soil without damaging the glass. For instance, alkaline EDTA is a powerful complexing agent for a number of elements, such as calcium, magnesium, silicon, aluminum, lead, zinc, and barium.

**5.4.2** Sometimes it is necessary to use a multicomponent aqueous system to achieve the desired results. Obviously, concentrations of various reagents and temperatures at which the process can be carried out are important. It is not the intent of this practice to explore all these possibilities, but, by knowing the glass composition, the correct solvent-concentration-temperature-time conditions to effect the desired result can be devised.

### 5.5 Residues and Defects:

**5.5.1** Any reaction between a solvent and a complex mixture of oxides affects the possibility of formation of some insoluble reaction products. Agitation may help prevent their adherence to the glass. Additionally, the reagent itself is potentially a “residue.”

**5.5.2** Reaction with the glass may also leave a roughened surface (selective reaction with certain glass components), streaks (selective reaction with nonhomogeneous “cords”), or with latent grinding marks hidden by a previous polishing step.

## 6. Detergents

### 6.1 Surface Active Agents:

**6.1.1** Surface active agents accelerate the cleaning action of aqueous solutions and provide mechanisms of cleaning that water does not have by itself. Many compounds are available, usually under trade names that give no hint of their chemical nature. Selection of the best compound for a particular use is usually a matter of experimentation, since the available literature gives few clues to aid in prediction.

**6.1.2** Generally, however, such “agents” consist of long-chain organic molecules, one end of which is attracted to the soil or the substrate, or both, the other end of which is “water soluble.” They “wet” the glass surface by lowering the surface tension of water; thus decreasing the contact angle between solvent and glass and between solvent and soil. The net effect is that the particle or oily film is dislodged. They “surround” the particle or droplet to suspend or emulsify and prevent its redeposition.

**6.1.3** The activity of surface active agents is usually enhanced by the blending of two or more and by the addition of non-surface active agents (called “builders”). A compound with good emulsification will be blended with a good wetter, and built with a polyphosphate for water softening, dispersion, and micelle formation. EDTA and similar compounds are used for water softening and solubilization of inorganic compounds, soda ash, and ammonia for pH regulation and sodium silicates for achieving high alkalinity while inhibiting attack on the glass.

**6.1.4** The builders can either promote or inhibit solution of glasses, depending on whether the reaction products or the builder and the glass components are soluble or insoluble. Polyphosphates and EDTA, in particular, will chelate with and

solubilize metallic ions, promoting a preferential leaching and leaving a porous or etched surface on the glass.

6.1.5 Water-soluble surface active agents are usually long-chain organic molecules with a hydrophobic end and a hydrophilic end. The ionic nature of the hydrophilic end determines the broad basic classification of the material—if negative, it is anionic, if positive, cationic, and if the material is not ionized, it is nonionic. There are a few amphoteric materials available, and these hybrids can be either cationic or anionic, depending on the pH of the solution.

6.2 *Anionic Agents*—The oldest, and one of the most effective anionic detergents if used in “soft” water, is soap. The largest class of synthetic anionic detergents is the sulfonated hydrocarbons such as sodium dodecyl benzene sulfonate. Sulfated alcohols and polyethers, such as sodium lauryl sulfate, are also used extensively.

6.3 *Cationic Agents*—The cationic detergents are usually quaternary ammonium salts. The classic cation active surface active agent has an aryl group, a long-chain alkyl group, and two methyl groups bonded to the nitrogen atom. The cationics are not usually found in glass-cleaning detergents, probably because they might be adsorbed, causing difficulty in rinsing.

6.4 *Nonionic Agents*—The nonionics are usually produced by ethoxylating various base molecules with ethylene oxide. The ethylene oxide adduct of nonyl or isoctyl phenol is the most popular of these. Water solubility, oil solubility, detergency, surface tension reduction, and other characteristics can be adjusted by the length of the ethoxy chain, which is at the hydrophilic end of the molecule.

6.5 *Amphoteric Compounds*—The amphoteric are usually amine sulfonates, and have not had as broad use as the anionics and nonionics, probably because of the greater cost of producing them.

6.6 *Other Additives*—Additives that enhance the cleaning action of organic solvents have not received as much attention as water-soluble additives. The dry cleaning industry uses coupling agents and water-in-oil emulsifiers to incorporate water in solvents for the purpose of removing water-soluble solids from fabrics; detergents have been developed for lubricating oils; and amines are used to accelerate the action of paint strippers; but otherwise there are few such materials commercially available.

6.7 *Residues*—All detergent compounds could potentially leave residues. Cationic detergents are the most likely to be a problem since they can readily bond to the surface. An acid rinse will usually remove such a residue.

## 7. Nonaqueous Solvents

### 7.1 Types of Solvents:

**TABLE 3 Relative Solvent Power of Some Organics (Removal of Stearic Acid from Glass by a 30-s Soak at the Boiling Point)**

Solvent	Stearic Acid Remaining, %
Combination No. 1 <sup>A</sup>	35.0
Combination No. 2 <sup>A</sup>	2.4
Combination No. 3 <sup>A</sup>	1.4
Trichlorotrifluoroethane	74.0
Acetone	1.3
Methanol	0.30
Hexane	44.0
Methyl chloroform	1.6
Benzene	6.7
Isopropanol	0.60
Trichloroethylene	0.80
Perchloroethylene	1.0

<sup>A</sup> See Table 4 for description.

7.1.1 *Hydrocarbons* such as hexane can be used to remove oils and other nonpolar contaminants from glass. However, for the removal of adsorbed polar compounds or intermediate size particulate matter (0.1 to 1000 μm), more active solvent systems such as those shown in Table 3 are required to overcome the intermolecular forces bonding the contaminants to the glass surface.

7.1.2 *Polar Compounds* such as low molecular weight alcohols (ethanol, isopropanol, and so forth) and ketones (acetone, methyl ethyl ketone, and so forth.) can be used to remove adsorbed polar material (Table 3) and intermediate-size particulate matter (0.1 to 1000 μm) from glass surfaces. The solvent/surface interactions may be such that the solvent can successfully compete for adsorption sites with the contaminants. These polar compounds are generally poor solvents for oils, greases, and so forth, and require cosolvents for optimum cleaning performance (see 7.1.4). Flammability limitations may also restrict the use of these polar compounds as cleaning agents, unless used in conjunction with nonflammable cosolvents.

7.1.3 *Halocarbons* are generally divided into chlorocarbons and fluorocarbons (including chlorofluorocarbons). Chlorocarbons such as trichloroethylene, perchloroethylene, and methylene chloride are powerful degreasing agents. Several of these chlorocarbons are highly effective in removing adsorbed polar organic matter from glass (Table 4). Fluorocarbons and particularly chlorofluorocarbons may possess much of the cleaning power of chlorocarbons and can be handled with considerably greater safety. The cleaning efficiency of chlorofluorocarbons can be markedly improved by the incorporation of relatively small quantities of polar cosolvents (see 7.1.4).

7.1.4 *Mixtures of Azeotropes of Polar Compounds and Fluorocarbons*—As described in 7.1.2, polar compounds are

**TABLE 4 Useful Solvent Combinations**

Type Combination	A	B	% B
Azeotrope	trichlorotrifluoroethane	methylene chloride	50
Azeotrope	trichlorotrifluoroethane	acetone	11
Azeotrope	trichlorotrifluoroethane	denatured ethyl alcohol	4
		{water	6
Emulsion	trichlorotrifluoroethane	{surfactant	2.5
Blend	trichlorotrifluoroethane	denatured ethyl alcohol	35
Blend	trichlorotrifluoroethane	isopropanol	35

quite effective in removing adsorbed polar contaminants and particulates (0.1 to 1000 μm) from glass. When small quantities of these polar compounds are combined with a fluorocarbon such as trichlorotrifluoroethane, the resultant combination possesses the added cleaning power of the polar compound with the lack of flammability of the trichlorotrifluoroethane. These combinations of solvents are also less aggressive on other materials such as plastics and elastomers. Some useful combinations are listed in Table 4.

7.2 Methods of Application:

7.2.1 Vapor Rinse (Ultrasonics)—Vapor rinsing is one of the most important techniques because when properly utilized the solvent is always clean, thus, the only residue possible is the solvent itself. The precise technique used for solvent cleaning of glass by vapor rinsing depends on the type of glass article and the nature of contamination encountered. A general procedure, however, can be outlined to illustrate the cleaning process utilizing the equipment shown in Fig. 1 as follows:

- (1) Vapor rinse,
- (2) Wash (hot solvent),
- (3) Vapor rinse and spray, and
- (4) Air dry.

Azeotropic solvent blends such as those described in Table 5 can be used effectively in this application since the liquid and vapor compositions do not change with evaporation.

7.2.2 Cold Cleaning (Ultrasonics)—In some situations where production volumes of glass articles are large or parts are too small to vapor rinse well, cold cleaning techniques are useful. Apparatus for cold cleaning is shown in Fig. 2. The procedure for cleaning with this equipment is as follows:

- (1) Vapor rinse in area (5),
- (2) Ultrasonically clean in (1),
- (3) Rinse in sumps (2) and (3),
- (4) Spray rinse in (4),
- (5) Vapor rinse in (5).

Cold cleaning is an effective way to utilize nonazeotropic solvent blends and solvent/surfactant systems (Table 4). The cleaning solution is placed in area (1) and pure solvent is put in the boiling sump of the rinse section.

7.3 Residues—Organic solvents may leave some residue on glass. Evaporation cannot be relied on to effect complete removal. Heating above the combustion point of the compound will almost totally remove it if it contains no inorganic components. However, such treatment can alter the glass surface to condense hydroxyl radicals to silanol bonds making the glass more hydrophobic.

8. Physical Methods

8.1 Mechanical Movement of Soil—A variety of methods, ranging from scrubbing with a cloth to ultrasonic energy, can be used to mechanically remove soil from the surface of glass.

8.1.1 Scrubbing, wiping, and brushing are probably the crudest methods. However, they may be quite effective if the soil is readily dislodged. A judicious combination of one of these techniques with an aqueous or organic solvent will often add to the effectiveness of the mechanical methods. Obviously, if misused, damage to the glass surface can occur.

8.1.2 Polishing and buffing can be varied over a wide range. They may include a very soft abrasive, such as calcium carbonate, or a solvent which simply interacts with the soil, or both. Or they may actually involve removal of a very thin layer of surface glass in order to ensure that all soil has been dislodged. This process may alter the glass surface chemically and physically in such a way that it is unacceptable.

8.1.3 Mechanical methods that cause virtually no physical damage to the glass surface include ultrasonics, high-pressure spray, spin wash, and air jets. The first three must obviously be used in conjunction with a liquid, which can either be aqueous or organic. Ultrasonics is most effective when all conditions are optimized, for example, the solvent is degassed, the temperature is the best for solvent and ultrasonic activity, the part holder is properly designed, and the frequency is optimum. Jets should be used only with filtered gas whether this is air or some other gas such as nitrogen. The use of ionized gas or spin drying will prevent the redeposition of small particles by electrostatic attraction.

8.2 Other Physical Methods:

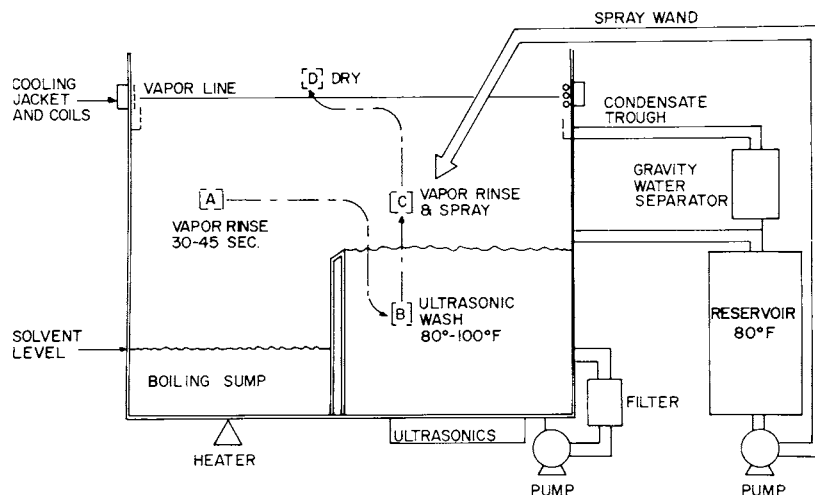


FIG. 1 Schematic of Vapor Cleaning System

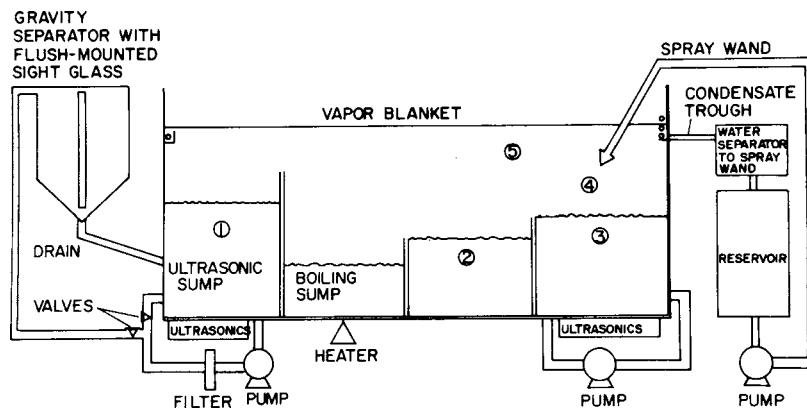


FIG. 2 Schematic of Cold Cleaning System

8.2.1 Combustion can be used for removal of organic materials except those which have inorganic components. Compounds, organic and inorganic, that burn or vaporize at temperatures below the softening point of the glass can be removed in this manner. Localized heating may, however, cause the glass to break. In some cases, it may even be desirable to heat the glass to soften the surface, so long as it is recognized that the physical and chemical properties of the surface may change in the process.

8.2.2 Finally, contaminants may be removed by physically entrapping them in a material that does not stick too tenaciously to the surface. Collodion or simply a sticky tape has been used for this purpose.

## 9. Designing a Cleaning Process

9.1 Each cleaning process must be tailored to the particular needs. The following factors should be considered: (1) glass composition, (2) contaminants present, (3) contaminants that are undesirable, and (4) requirements of the surface of the finished part.

9.2 Given this information, the designer of the process has a wide variety of options. That is, he may design a step that involves two or more techniques, or he may use two or more techniques sequentially. For instance, he could combine ultrasonic agitation with a solvent to reduce surface tension. Or he could use an organic solvent to remove a greasy component of the soil followed by water to remove a salty deposit.

9.3 Combining several techniques into one step or sequentially is done for only two reasons. The first is to remove some component of the soil that was not removed in prior steps. The second is to remove prior cleaning agent or some contaminant left by it. Ideally, the first step should be so designed as to contribute no further problems.

9.4 However, it is often impossible to avoid a residue from a cleaning process. When using an aqueous solvent, water may not be sufficient for removing the residue. An acidic or basic rinse may be required. Bear in mind that this also can leave a residue. Water itself can leave a residue if it is not initially pure or it has picked up contaminants from cleaning. This can be minimized or eliminated if the water is blown off with filtered ionized gas rather than allowing droplets to dry in place. Detergents, especially cationics, leave traces on the glass surface after cleaning. These may be best removed by rinsing in a basic or acidic solution depending on whether the detergent is anionic or cationic.

9.5 It is important if not essential to confer with the glass producer before implementing a particular process to ensure that no unforeseen undesirable effects will occur.

## 10. Keywords

10.1 cleaning; glass; solubility; solvents; technical

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