



Designation: C912 – 93(Reapproved 2008)^{ε1}

Standard Practice for Designing a Process for Cleaning Technical Glasses¹

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^{ε1} NOTE—This standard was revised editorially in December 2012.

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.²

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.

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.

¹ This practice is under the jurisdiction of ASTM Committee C14 on Glass and Glass Products and is the direct responsibility of Subcommittee C14.02 on Chemical Properties and Analysis.

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² Campbell, D. E., and Adams, P. B., "Bibliography on Clean Glass: Supplement 1," *Journal of Testing and Evaluation*, Vol 14, No. 5, September 1986, pp. 260–265.

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, HNO₃ (Sn, Sb, Mo).

NOTE 2—W is soluble in acid but heat may precipitate it, for example, H₂WO₄.

NOTE 3—Sn⁺⁴ is soluble in hot H₂SO₄; Sn⁺² is soluble in other reagents as well.

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

NOTE 5—PbSO₄ is soluble in *hot* concentrated H₂SO₄.

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

NOTE 7—Ba is insoluble in concentrated HNO₃.

| Oxides of | HF 49 % | H ₂ SO ₄ 96 % | HNO ₃ 70 % | HCl 37 % | HBr | HI | H ₃ PO ₄ 85 % | NaOH 50 % |
|-----------|----------------|--|--------------------------|-------------|-----|----|--|--------------|
| Al | s ^A | s | s | s | i | i | i | s |
| Sb | i ^A | 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 | s ^B | 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 | s | s | s | s | s | s | 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 ^B | i | i | i | i | i | i |
| Sn | s | s | s | s | s | s | s | s |
| Ti | s | s ^B | 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 ^B | i | i | i | i | i | i |

^A s = relatively soluble, i = relatively insoluble.

^B hot

TABLE 2 Threshold Limit Values for Some Common Solvents

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

^A 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^R 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.

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 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 % SiO₂ and 5 % Na₂O could be cleaned in H₂SO₄ without appreciable glass attack even though Na₂O is very soluble in H₂SO₄; however a glass containing 50 % SiO₂ and 25 % Na₂O would probably show considerable attack by H₂SO₄. 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 % SiO₂ could probably be cleaned in this way with HNO₃, 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,