Designation: C912 – 93 (Reapproved 2008)

Standard Practice for Designing a Process for Cleaning Technical Glasses[1]

This standard is issued under the fixed designation C912; 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 (ε) 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.[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.

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

[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*, Vol14, No. 5, September1986 pp. 260–265.

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_2 and 5 % Na_2O could be cleaned in H_2SO_4 without appreciable glass attack even though Na_2O is very soluble in H_2SO_4; however a glass containing 50 % SiO_2 and 25 % Na_2O would probably show considerable attack by H_2SO_4. 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_2 could probably be cleaned in this way with HNO_3, 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.