GMP 7

Good Measurement Practice for Cleaning Precision Glassware

The volume contained in or delivered from precision glassware depends on the cleanliness of the container. Glassware must be sufficiently clean to ensure uniform wetting of the entire internal surface of the standard. When clean, the walls will be uniformly wetted and the water will adhere to the surface in a continuous film. If films of dirt or grease are present, the meniscus may crinkle at the edges and liquids will not drain properly and will leave water drops on the internal walls. Lack of cleanliness can cause errors in setting a meniscus and incomplete wetting/drainage of the calibration liquid (generally water.)

Glassware that is submitted for calibration should be cleaned (and autoclaved if needed) prior to submission to the laboratory to ensure removal of all chemical, biological, radiological, or other contaminants.

If glassware is cleaned by the laboratory, appropriate inquiries should be made to determine prior and subsequent uses along with implications of certain cleaning methods. Some methods of cleaning glassware prior to calibration or use are ineffective or detrimental and may leave chemical contamination deposits that can be detected in some chemical analysis operations.

There are a number of suitable detergents (both liquid and powder), available from laboratory supply houses that do not contain phosphates. The catalog descriptions usually indicate whether or not they contain phosphates. Laboratory detergents that contain phosphates can leave a deposit on the glass that will cause water to "bead" on the surface making it appear to be dirty, and making it difficult to properly set a meniscus. Once a phosphate deposit has occurred, it is very difficult to remove. It may be removed with hot (approx. 65 °C) sodium dichromate-sulfuric acid cleaning solution. However, use of this hot solution is hazardous, is a problem for safe disposal, and is not recommended unless proper safety equipment is available (see the next section).

If acetone is used to remove oil or grease film, always follow with alcohol (ethanol) before drying or rinsing with water. Denatured methanol can leave an oily residue due to the denaturing process. Acetone, if allowed to dry, may also leave a film deposit.

The above solvents need not be used if the glass does not have oil or grease film. Mechanical shaking of water and suitable non-phosphate detergents is usually adequate for cleaning glassware.

These cleaning agents do not preclude the use of other suitable methods, of which there are several. Some have special applications that would not normally be encountered by metrologists or field inspectors.

See GLP 13, Good Laboratory Practice for Drying Containers for appropriate drying techniques. It is not necessary to dry any container marked "to deliver."

Cleaning Methods¹

Safety Note: Material safety data sheets (MSDS) should be available, studied, and carefully followed before using any of the following cleaning methods. Chromic acid solutions are not recommended for routine use because it is a hazardous waste and hazardous to health! Suitable education or training in the handling of chemicals is highly recommended.

Frequently it is desirable to give glassware a preliminary rinse or soak with an organic solvent such as xylene or acetone to remove grease, followed by a water rinse. The rinsing with water must be done thoroughly if acid will be used later to clean the glassware.

Unless autoclaving is necessary, glassware should be cleaned as soon as possible after use to avoid setting and caking of residues. Pipets, for example, may be placed in a jar containing a weak antiseptic solution immediately after use. Autoclaving is necessary to disinfect glassware that may have been used to contain potentially dangerous biological fluids.

A selection of general cleaning methods follows:

1. Fill with sulfuric acid-dichromate mixture and let stand. After removal of the mixture, rinse with distilled water at least six times. To make the cleaning mixture, dissolve (60 to 65) g of sodium- or potassium-dichromate by heating in (30 to 35) mL of water, cool and slowly add concentrated sulfuric acid to make one liter of solution. This solution is available from laboratory supply companies.

Note: Extreme care should be exercised in handling acidic solutions.

- 2. Scrub with a 1 % to 2 % hot solution of a detergent. Rinse well after brushing. A number of suitable, commercial washing compounds are available.
- 3. Fuming sulfuric acid (very hazardous material) is an excellent cleaning agent. Usually, cleaning can be accomplished by use of a comparatively small amount of acid, manipulating the vessel so that the acid comes in contact with the entire surface, and immediately emptying and rinsing.

When a piece of glassware is badly contaminated with stopcock grease (except silicone grease), it may be necessary to rinse with acetone once or twice before using one of the above methods. For silicone grease, the acetone can be omitted and the piece soaked for 30 min in fuming sulfuric acid. Warm decahydronaphthalene (decalin) also has been suggested as a solvent for silicone grease. In this case, let the piece soak for 2 h, drain, and rinse once or twice with

¹ While the metrologist is not ordinarily faced with the problems for which these procedures are necessary, they are given here in the event that they are needed.

acetone, followed by a water rinse.

Cautions

Care should be exercised when using most cleaning solutions because they can cause skin irritations or severe burns on contact. Dilute solutions become concentrated as the water evaporates; therefore, always flush the exposed area immediately with large quantities of water.

Suitable chemical-specific-resistant goggles or a face mask should be worn to protect the eyes from splashes and rubber gloves should be worn to protect the hands. It is advisable to wear an acid resistant laboratory coat or a rubber apron to protect clothing when using strong acids for cleaning. The glassware should be handled gently to avoid breakage and also to prevent spilling acids and other cleaning fluids. All cleaning should be done in a laboratory sink or on an acid-proof laboratory bench, preferably within a fume hood, to the extent possible.

Some of the cleaning materials mentioned leave minute traces or residues unless the rinsing process is carried out thoroughly. While such traces may not be harmful if the purpose of cleaning is to prepare the glassware for calibration, they can give trouble when the glassware is used in certain laboratory operations. For example, manganese and chromium compounds, even in extreme dilution, may retard or inhibit growth of micro-organisms, and traces of phosphorus may interfere with delicate tests for this element. When glassware is to be calibrated, final rinsing must be with pure distilled or deionized water.

If an article is to be dried after cleaning, as is necessary for all vessels marked "To Contain", acetone, followed by ethyl alcohol (American Chemical Society Specification) may be used. Drying may be hastened by blowing clean, dry air into the vessel (or drawing the air through the vessel). Be sure not to mix acetone with alcohol. (See GLP 13.)

Efficient air filters must be provided to remove any particles of oil or dirt from compressed air used for drying purposes.