



Cleaning Glass or Quartz Sample Containers

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Test materials that remain after an earlier thermogravimetric experiment must be removed prior to reuse of the sample container for subsequent experiments. High temperature platinum and alumina thermogravimetric analysis (TGA) sample pans are usually exposed to a high temperature propane or butane flame external to the instrument between each set of experimental runs. This approach fails with the use of glass containers that typically melt around 700 °C. Exposure to the open flame of a torch will destroy the pan. Quartz is more thermally resistant but may soften and become misshapen with exposure to the high temperatures of a flame. Glass and quartz sample containers used in thermogravimetry pose special cleaning challenges. Glass and quartz containers are cleaned in a more traditional laboratory method.

Mild Mechanical Cleaning

Glass and quartz sample containers should be cleaned mechanically as an initial cleaning step. This is best performed using a small cotton swab to remove any loose granules adhering to the surface. As with any mechanical cleaning of a delicate glass part, mechanical damage is a possibility so a delicate and light touch is a must to avoid breakage.

Ultrasonic Cleaning

Following mechanical cleaning, the next step is cleaning in an ultrasonic bath (available from most laboratory supply companies). The initial cleaning is performed for 20 minutes in a water bath to which a drop of ordinary household detergent has been added. This is followed by a 20 min rinse in a clean distilled water bath.

Examine the sample containers as they are removed from the water rinse. If all particles have been removed, then move to the drying operation below. If some particles remain, try organic solvent cleaning.

Organic Solvent Cleaning

Most organic materials are easily dissolved and removed in a bath of acetone or ethanol solvent. Soak the sample containers for 20 minutes in a screw cap bottle

containing enough solvent to cover all of the sample containers. Avoid mechanically agitation of the sample containers as this may damage the delicate glass parts.

Drying

Following the ultrasonic cleaning or the organic solvent cleaning, the sample container is removed from the bath and placed on a paper towel where it is washed with reagent-grade acetone or ethanol. (Note: Both acetone and ethanol are flammable solvent and shall be treated with care and kept away from any open flame.)

The sample containers may then be air dried until all of the organic solvent has evaporated, typically about 20 minutes.

Storage

Clean sample containers should be stored in a desiccator until ready for use.

Most commonly encountered materials will be cleaned using the mild cleaning processes described above. Occasionally, however, more aggressive procedures may be required.

Chromic Acid Cleaning

CAUTION - Chromic Acid is a toxic and corrosive solvent and is a known carcinogen (1). Do not allow the solution to come into contact with you or your clothing, for it will cause very bad burns. Protective gloves and safety glasses are required when handling chromic acid. If any is spilled on the floor or desk, neutralize it immediately with commercial-grade sodium bicarbonate (baking soda) then wash completely with water. If it is spilled on the skin, wash in running water immediately.

Chromic acid cleaning solution may be purchased from most laboratory supply houses or may be prepared by stirring about 20 g of powdered technical grade sodium dichromate with a very little water to make a thick paste. Add 300 mL of technical-grade concentrated sulfuric acid. Store in a glass-stoppered bottle. It is unnecessary to remove the residue of undissolved salt by filtration, but clear solution should be decanted from the bottle each time it is used. The solution may be repeatedly used until the reddish color of dichromate has been replaced by the green color of the chromic ion (2).

In a small beaker, just cover the glass sample containers with chromic acid cleaning solution. Allow to stand at room temperature for 20 min. Decant the cleaning solution from the mechanical parts and return the used chromic acid cleaning solution to its storage bottle. Wash the sample containers at least six times with tap water and finally with distilled water. Dry the sample containers as described above and store in a desiccator for future use.

Chromic acid cleaning solution is an Environmental Protection Agency (EPA) Toxic Substances Control Acta (TSCA) material containing “heavy metals”. It must be disposed of in an appropriate manner that complies with local, state and federal regulations.

REFERENCES

1. R. J. Lewis, Jr. (ed), *Sax's Dangerous Properties of Industrial Materials*, 10th Edition, John Wiley, New York, **2000**, 2, p. 924.
2. W. C. Pierce, D. T. Sawyer and E. L. Haenisch, *Quantitative Analysis*, John Wiley, New York, **1958**, pp. 49 - 51.

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