SOP: CLEANING OF GLASSWARE

To ensure an experiment's results are correct we need to use proper and clean glassware. But remember glassware needs only be as clean as is required for the work being done. Over cleaning, or incorrect cleaning, wastes time, equipment and money. There is no one technique that will clean all glassware – it is an art!

The best time to clean glassware – is right after use! It becomes more difficult to clean after a while. When waiting for a reaction, it is a good time to clean your glassware.

The use of a plastic container (with rubber matt at bottom with soapy washes) to clean or soak glassware in helps to prevent loss by breakage or of small glass pieces.

Cleaning glassware for experiments takes more than one step!
If glassware needs to be only cleaned with soap and water the extra 2 steps thereafter is to rinse it with water and then with distilled water. Thus it takes 3 steps!
If the glassware also contains particulate materials that first needs to be brushed or wiped off before the cleaning starts.
Remove vacuum grease with organic solvents first before removing salt deposits.

The basic consensus is like remove like! Thus for effective cleaning you MUST know what you want to clean to have an effective cleaning plan of attack in place.

You must also know what type of glassware to use for which chemicals – Borosilicate glassware (Pyrex & Kimex) is more chemically resistant than soda-lime glassware.

Glass Stripping

- Hydrofluoric acid and perchloric acid and all bases can react with Borosilicate glassware and strip the surface of glass off layer by layer.
- Some organic tars and minerals in water can also react with the glass surface.
- It can also cause and uneven glass surface causing the glassware to become soiled easier and more difficult to clean.
- It also can remove glass calibration markings making the glassware useless.

Wet glassware that is not quantitatively clean can be dried by:
1) placing it on the drying rack (or invert on a paper towel),
2) placing it in the drying oven (for items that are water-wet only, no flammable solvents) or
3) rinsing with a solvent such as acetone, methanol or ethanol and place in oven at 50ºC.

The first method (1) is preferred for drying quantitatively clean glassware (provided that the prongs of the drying rack are not inside the item, thus contaminating it). Volumetric glassware and cuvettes are never to be placed in drying ovens, even if they are not quantitatively clean. The third method is acceptable only if there is good ventilation and preferred if it is done in a fume hood.

CLEANING METHODS & STEPS

- Start off with the least and go over to the more powerful cleaning techniques and mediums until glassware is as clean as needed.
- Remember like dissolves like! Polar solvents will dissolve polar contaminants more effectively.
- Proper selection of solvent material can avoid damaging the material you want to clean and prevent a hazardous situation and forming hazardous materials.
- Typically the warmer the cleaning agent the better, faster and more effective the cleaning.

SOAPY WATER

1) Clean off grease from glassware & stopcocks
2) Rinse out glassware with a small amount of acetone
3) Prepare a warm or hot soapy water "bath" in wash basin – read instructions of the powdered or liquid detergent to be used. A non-alkaline detergent is best to be used. The concentration of detergent should be between 5 and 20% depending on the residue.
4) Place glassware in the warm/hot soapy water & submerge it totally within a washbasin under the water – may have to leave it there for several minutes; maybe even overnight.
5) Keep in mind: soaps & detergents do not dissolve oils; it can emulsify it and help to lift the dirt from the surface
6) Prevent scratching the surface of any type glass or porcelain items. **DO NOT** use scrubbing pads, metal of hard plastic brushes etc. Use laboratory bottle or test tube brushes specifically manufactured for the task. Look specifically for Teflon safe scouring pads that can be used and which will not scratch the surface.
7) After cleaning wash glassware with tap water.
8) Now wash with deionized or distilled water. The water will sheet cleanly off the glass, if it is quantitatively clean. If it does not sheet off the glass, and you desire the glassware to be quantitatively clean, first repeat the above soaking and scrubbing steps. If, after a second cleaning, bits of solid still adhere to the glass, or if there is clearly a greasy residue on the glass, more aggressive action must be taken.
9) Place glassware upside down for storage or to dry on kimwipes or any lab tissue paper.
10) If glassware needs to be used immediately and dried quickly it can be placed in a drying oven at 50ºC.
11) Alternatively you can pour about 10ml of reagent grade acetone or methanol in glassware – swirl it around & pour it into a container labelled with "Used Acetone" or "Used Methanol". This can be used again in future cleaning.
12) The soap water may be poured down the drain.

The following cleaning methods are two of the more commonly used ways to remove contaminants from glassware. They are usually used after normal cleaning has failed, and they are often used together, because each is effective at removing different types of contaminants. Care must be taken using either one because of the corrosive and flammable nature of the solutions used.

ORGANIC SOLVENTS

- Soap & water cannot clean everything. There are times that organic solvents are needed. There are three types: polar, non-polar & halogenated hydrocarbons (avoid as much as possible).
- Polar: alcohols & ketones – ethanol, methanol, isopropanol, acetone. Non-polar for oil & grease: various hydrocarbons such as hexane.
- Halogenated hydrocarbons are powerful degreasing materials: methylene chloride (dichloromethane).

1) Ascertain if it is a polar or non-polar contaminant
2) Remove any soaps or excess water
3) It is usually a good idea to start off with either 5ml or less acetone or dichloromethane rinse – use the lowest grade (technical). Acetone is polar and can dissolve/remove nearly all organic compounds.
4) Use a 5ml or less of the higher grade (reagent) for a final rinse.
5) If the solvent has a high boiling point – select a suitable (polar or nonpolar) solvent with a low boiling point for the final rinse to facilitate evaporation. Acetone also assists to wash away water and therefor assist in the quicker drying of equipment/glassware.

BASE BATH

This is the preferred method when silicone from stopcocks or other organic compounds needs to be removed. It is ineffective against any hydrocarbon or fluorinated grease and thus the grease will not be removed in this manner.
BUT keep in mind: 1) the alcohol is a potential fire hazard, 2) the bath's alkalinity is caustic – wear butyl gloves, 3) the bath strips glass – therefore not actually cleaning but stripping a layer of glass from the glass item, 4) **DO NOT** soak the glassware in the bath for longer than mentioned and 5) **NEVER** place volumetric items such as flasks in bath. Base baths can be used over & over until it decreases in effectivity.

1) Clean off grease as much as possible
2) Wash glassware as according to the Soap & Water Method
3) Rinse glassware with an organic solvent
4) 5L base bath: mix 4L Iso-Propanol with 200-300g KOH & add carefully 1L water.
5) The solution can also be prepared by either adding 2L litre of 95% ethanol to 120ml of water containing 120g of NaOH, or by dissolving 100g of KOH in 50 ml of water and, after cooling, make up to 1L.
6) Leave until cooled down & KOH dissolved.
7) This base bath can be stored in a plastic (OK) or stainless steel (best) container – plastic will bun & melt if alcohol catches fire. This bath will dissolve glass.
8) Clearly label as "Danger Base-Bath Solution"
9) Separate all ground joints & stopcocks before the time
10) Carefully & completely submerge items in bath and let it soak for 30min or less
11) Carefully remove items and rinse thoroughly in water – items will be slippery
12) This can be followed with a brief soak in 2-3 molar nitric acid solution to stop the hydroxyl groups of the base to attack the glass surface.

If a base bath need to be disposed of it can be neutralised and if it does not contain any metals disposed of down the drain.

NEVER soak the following items in a base bath for prolonged periods:
»Glassware contaminated with metal-containing compounds
»Glass fritted funnels
»Cuvettes
»Volumetric glassware (pipettes, volumetric flasks)
»Any glassware contaminated by an oxidizing agent
»Anything that has not been washed according to the above steps first

Glass fritted funnels and volumetric glassware can be soaked briefly with the base bath solution to remove small amounts of grease, but prolonged exposure to the caustic solution can damage these items.

ACIDS & OXIDISERS

Organic and carbonaceous materials can be removed with by acids and oxidisers. Because alkali deposits are also removed from the surface a more porous surface forms and may cause greater water absorption and cleaning problems. The problem is more likely in porcelain and soft glass containers than in borosilicate glass.

1) Clean off grease as much as possible
2) Wash glassware as according to the Soap & water Method
3) Rinse glassware with an organic solvent
4) **Aqua Regia Solution**: 3 parts concentrated HCl to 1 part concentrated HNO_3.
5) It is recommended that 1 part H_2O be added if the aqua regia will be stored to minimize the generation of Cl_2.
6) Use a small quantity of this acid mixture and swirl it in the glassware for a few minutes or place it in a warm bath until reaction ceases.
7) Dispose mixture used of as hazardous waste.
8) Wash glassware as according to the Soap & Water Method
9) Rinse with deionized water
10) Give a final rinse with alcohol & acetone to facilitate drying
11) It is the only acidic mixture that will dissolve gold and will oxidize just about everything else. Extreme caution must be used when working with aqua regia because it generates Cl_2 and NO_x gases in addition to causing severe tissue damage.
12) Rinse with deionized water
13) Give a final rinse with alcohol & acetone to facilitate drying

EXTRA TIPS

1) Remove tungsten stains – remove with Hypoclorite solution (Chlorox bleach). The solution can be re-used many times.
2) Removing of tar from distillation flasks – best is to clean flasks immediately. Best solution is to invert flask into a beaker of acetone and let the vapour dissolve the tar. The process can be sped up by heating the acetone containing beaker in a water bath. **DO NOT** use any naked flames or hotplates. To get the solvent in the flasks neck use a flexible tube (not soluble in the acetone) to let air out as the flask is inverted into the beaker.
3) Iron stains – use equal parts of hydrochloric acid and water
4) Metal containing compound contaminant – soak in 6M HCl solution, then rinse under tap water + deionised water
5) Permanganate stains – use concentrated hydrochloric acid or a saturated solution of oxalic acid at room temperature
6) Magnesium oxide stains – 20 – 30% sodium bisulfite ($NaHSO_3$)
7) Removing Grease by placing glassware in a boiling weak solution of sodium carbonate. Acetone or another fat solvent may also be used. Strong alkalis should not be used. Silicone grease can be removed by soaking in decahydronaphthalene for 2 hours.
8) Pegboard drying is not recommended since airborne contaminants in the laboratory will be deposited on the “clean” glassware. Oven drying is suggested at 50°C to prevent burns. Open-ended glassware such as beakers should be covered with foil and stored in a dust-free cabinet.
9) It is very important that glassware is used and stored properly to prevent failure or injury. Any scratches, whether noticeable or not, greatly reduces its strength. Whenever possible, do not let glass apparatus contact metal, grit or even other glassware. Plastic stirring rods and scrapers should be used to prevent scratches and prolong glassware life. Glass should not be scribed or etched unless on a thick glass joint. This is especially important when it is to be used for pressure or vacuum work.
10) All glassware should be inspected frequently. Bring to the Glass Shop at first sign of damage for evaluation or repair.
11) Clean and dry glassware as soon as possible prior to storing. Dirty glassware gets harder to clean over time
12) Re-grease joints and stopcocks often. This will help prevent leaks, and breakage from turning poorly greased/ tight plugs
13) When not in use, remove and clean all stoppers, adapters and plugs to prevent “sticking” problems
14) Cover all metal rods with rubber tubing before storing flasks or apparatus on them
15) Have a full set of brushes at hand-brushes to fit large and small test tubes, burets, funnels, graduates and various sizes of flasks and bottles. Motor driven revolving brushes are valuable when a large number of tubes or bottles are processed. Do not use cleaning brushes that are so worn that the spine hits the glass. Serious scratches may result.
16) Scratched glass is more prone to break during experiments. Any mark in the uniform surface of glassware is a potential breaking point, especially when the piece is heated. Do not allow acid to come into contact with a piece of glassware before the detergent (or soap) is thoroughly removed. If this happens, a film of grease may be formed.
17) Cover all metal rods with rubber tubing before storing flasks or apparatus on them

HEALTH & SAFETY ISSUES

SAFETY MEASURES

1. Wear the proper PPE: labcoat for clothing & skin protection, eye protection is critical (front, top, side & bottom) against splashes, long sleeved correct gloves (to protect against any residual chemicals, cleansing agent/solvent, heat of water/solution, adsorption through skin).
2. Keep in mind that not all gloves protect against hydrocarbon solvents as it will perish the gloves.
3. Solvents can also cause: a) skin irritation, b) drying of skin, c) damage to synthetic clothing, d) cancer, e) rubber gloves to dissolve/perish
4. ALWAYS use organic solvents in a fume hood as the vapours of many solvents can have minor to major health problems & are flammable.
5. Use all acids in a fume hood.
6. Spillages: a) Use Sodium Bicarbonate to neutralise Acid spills, b) Rub Calcium Glutanate in on skin if HF spilled on it, c) Use Sodium Bisulphate to neutralise Peroxide spills
7. When using organic solvents to wash/rinse out glassware be as far as possible from any open/naked flames, hot surfaces, sparks etc.
8. Keep a moisturiser around to re-moisturize after cleaning processes.
9. Handle & wash only one item at a time – too many at once causes breakage or damage to glassware
10. Chipped and broken ends can cause cuts or more serious injury – use of fire polishing of chipped & broken ends can salvage some glassware
11. Cracked, even hair cracks can cause explosion of glassware when heated or under vacuum & pressure
12. Dispose of cracked & damage glass in the yellow bucket & any kimwipes, tissue etc. in the RUCs provided. It is a good idea to place tissues or rags in fume hood to dry before disposing of it in RUC.
13. Dispose of rinsing waste in the correct waste stream into Jerry can/s.
14. When you do glassware washing and you feel an itch immediately or burning sensation, wash/rinse skin area with large amounts of water. You also may have to seek medical help.
15. Store any surplus base or acid solutions to be used in future for washing/cleaning of glassware in closed containers which is properly labelled indicating solution & its usage.
16. NEVER heat any cleaning solution with a naked flame or hot plate, use a hot bath.