Cleaning Glassware

Applications and Summary

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Overview

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Organic synthesis is about transforming a readily available reagent into a more valuable product. Having clean glassware is crucial for the efficiency of this process. Dirty glassware can potentially affect the reaction and make isolation of the final product more challenging. Thus, a synthetic chemist must keep the glassware spotless. The methods described here will detail different glass cleaning techniques that are regularly used to remove organics, metals, grease, and salts.

Principles

Cleaning glassware is an important part of synthetic chemistry. Chemists must be able to rely on clean glassware for their reaction. Assessing glassware by eye is not enough to confirm whether the glassware is clean. If unsure about the nature of the glassware, clean it. It is always better to take a few extra minutes to have clean glassware than to spend hours removing contaminants or have irreproducible results.

How clean the glassware needs to be is also an important factor. The level of cleanliness of the glassware depends on the type of work being done. Over cleaning can waste time and money. Understanding the chemistry can assist in evaluating how much cleaning is required for the glassware. Covered here are the most basic cleaning steps necessary for most organic chemistry reactions.

One basic principle is that "like" dissolves "like". Polar solutions dissolve polar materials and non-polar solutions dissolve non-polar grease. There is no single step that can clean any

clean is important.

One recommendation is to clean the glassware just after its use. Chemists are busy and are generally balancing many tasks, but cleaning glassware between reactions or while a reaction is running is usually a good use of time.

For safety reasons, each item of glassware should be inspected. Broken glassware is dangerous and should be repaired or discarded. Flasks and other glassware that are commonly put under vacuum or pressure should be inspected for star cracks. These are weak points on the glass that could create sudden explosions or implosions if placed under pressure. It is good practice to use eye protection even when cleaning glassware with simple soap and water. Gloves provide good protection, too, especially when handling harsh cleaning solutions.

For waste disposal, all organic solutions, organic reagents, and any organic contaminated paper or solid should not be discarded in the sink or trash. Dispose of all organic material in safety bins, which are handled by the site's Environmental Health & Safety (EH&S) team; all organic liquids should be placed in an appropriately labeled liquid container (which is also picked up by EH&S). Water used for washing the glassware and scrubbing with soap can go down the drain.

Removing grease from the joints of glassware is important. Most organic greases can be removed by non-polar solvents like hexanes or ethyl acetate. Since ethyl acetate is a much greener solvent (*i.e.*, safer, less toxic, lower environmental impact), it is the preferred choice. If the grease still remains, then a base bath will remove most greases from joints or stopcocks.

that is then disposed of in an organic waste container. Using a green polar solvent like acetone is preferred. This will help to remove most organic residues from a flask before washing. When using organic solvents, remember that like dissolves like. Hexanes are an example of a nonpolar solvent that is commonly used. Frequently used polar solvents include: isopropanol (rubbing alcohol), acetone, ethyl acetate, and methylene chloride (dichloromethane).

Once the glassware is removed, make soapy water by mixing powdered or liquid glassware cleaning reagents with tap water. Different sized brushes will help to reach and clean the inside of curved glassware. If glassware is not cleaned right away, a small tub of this soapy water can be used for long-term soaking. Using warmer water helps to dissolve and remove salts and stuck-on grease. Soapy water can be rinsed off with tap water, follow by deionized water. Then the water can be removed with acetone.

Ultrasonic cleaners are great for extremely stuck-on grease or residue. If traditional scrubbing with a brush fails, then filling the glassware with warm soapy water and placing in a sonicator will help to loosen dirt particles.

A base bath can be prepared and used to remove grease. In addition to glassware, a base bath can be used for other dirty objects like stir bars or spatulas. Since a base bath is caustic and can strip away glass, finely calibrated glassware should not be cleaned by this method. Materials like volumetric flasks, vacuum parts like vacuum stopcocks, or glass frits should not be cleaned by this method.

Acids and oxidizers can remove salts, metals, and organic reagents. All acids should be used

volumetric flasks or glass frits can be used by this method.

Finally, some reactions require water-free conditions. In these cases, it is important to remove any trace water from inside of the reaction flask. Even if it appears dry, there can be a small thin coating from air moisture. This can be removed with high heat either by placing the glassware in an oven or by heating under a vacuum.

Glassware cleaning is a multi-step process. The most common procedure that will be effective against most dirty glassware will be to first rinse with an organic solvent, and then second, to wash and scrub with warm, soapy water. Then the glassware needs to be rinsed with tap water, deionized water, and finally with acetone before placing on a rack to air dry. When this fails to clean the glassware, depending on the experiment and the type of glassware, additional steps to clean with acid or base (or both) might need to be executed. Cleaning glassware is an art, and developing the knowledge of what solvents or cleaning solutions to use will improve with experience and time.

Procedure

General Considerations: While some of the below cleaning solutions are harmless to skin contact, it is always wise to wear chemical resistant gloves, a lab safety coat, and eye protection.

1. General Procedure for All Glassware

NOTE: This method applies to stir bars, spatulas, funnels, and other reusable equipment. It can also be used with any of the other washing methods described below.

as acetone. Acetone rinsing should go into organic waste.

2. With warm tap water, use an appropriate brush and soapy water to scrub the inside of curved glassware.
3. Remove soapsuds using warm tap water.
4. To avoid hard water stains, use deionized water or reverse osmosis water to wash away tap water.
5. Rinse again with acetone to remove water. This step helps to expedite the glass drying process.

2. General Acid Wash

NOTE: Some organic residues, metal salts, and stuck-on bases can be removed using a mild acid solution. When finished, non-oxidizing acids can be diluted and discarded down the drain (*e.g.*, hydrochloric acid). Other acids like acetic acid, which is an organic acid, should be neutralized and added to water-based liquid waste containers. Usually sodium bicarbonate is used to quench and neutralize acids because it has its own visual indicator: the reaction of sodium bicarbonate and acid produces CO_2 gas, and so bubbling indicates that there is still acid in solution. Once sodium bicarbonate is added and no bubbling is observed, then the solution is neutralized. This can all be placed in aqueous waste.

1. Make a 1 M HCl (aq.) solution or a 5% v/v acetic acid in deionized or reverse osmosis water.
2. Inside of a fume hood, add some of the acid cleaning solution to the glassware and swirl the solution to cover the dirty areas. It is sometimes useful to use a cleaning brush, pipet, spatula, or sonication.
3. Neutralize the acid wash before adding to aqueous waste (a sodium bicarbonate solution will neutralize the acid).

3. Base Bath

Note: In general, if there is a piece of glassware that cannot be cleaned by acid and soapy water, soaking the glassware in a base bath may help. The base bath is strongly basic and caustic. Care should be taken to prevent any spills. However, small amounts can be washed off completely using water. Serious spills can be neutralized with acetic acid and mopped up. The base bath should be neutralized and discarded with aqueous waste.

NOTE: Glass frits should not be cleaned using a base bath.

1. In a designated cleaning bucket (*e.g.*, 6-gallon paint bucket), add an approximately 2 M solution of KOH (potassium hydroxide) in IPA (isopropyl alcohol; 2-propanol). IPA can be substituted with methanol or ethanol. Use a cap to retard evaporation.
2. Clean the glassware according to the general method above.
3. Carefully soak the glassware in the base bath making sure to remove as much air as possible (it is best to soak overnight).
4. When removing the glassware from the bucket, pour back into the bucket any of the base bath solution inside of the glassware. Then take the glassware and rinse off any remaining base solution in the sink with deionized or reverse osmosis water.
5. Dry with acetone.

4. Aqua Regia

Note: Aqua regia is known for its properties in dissolving metals. This acid wash is strongly acidic, oxidizing, and caustic. Care should be taken to prevent any spills or skin contact. Small amounts that contact the skin will cause itching and must be washed off completely by rinsing the area under water for 15 min. Serious spills can be neutralized with $NaHCO_3$ (sodium

fume hood. It can also corrode stainless steel. Aqua regia should be neutralized with sodium bicarbonate before adding to aqueous waste.

1. Make small batches of aqua regia using a 3:1 ratio of nitric acid to hydrochloric acid (HNO_3:HCl).

   NOTE: Both acids are clear but upon mixing will fume and turn dark orange.
2. Add aqua regia by pipette or by careful pouring, and swirl to dissolve residual metals.
3. Remove most aqua regia by pouring it into a second container that will be treated and disposed of in aqueous waste.
4. Clean glassware according to the general method as described above.

5. Cleaning Glass Ground Joints

Note: Ground joints can easily become stuck. One way to prevent this problem is to make sure all joints are clean. When clean, the ground glass joints are frosted white in appearance. Clear joints usually indicate small amounts of oils and grease that can contaminate spectroscopic data and the desired compound.

2. Clean the inside of ground glass joints (this can be tricky). The best way is to use a paper towel soaked in ethyl acetate (EtOAc) and a small spatula.
3. Easily clean the outside of ground glass joints by using a paper towel soaked in ethyl acetate.
4. Wet the tip of a paper towel with EtOAc and use the spatula to reach the edges of the joint.
5. Observe that the ethyl acetate evaporates in seconds and reveals a frosted white ground glass joint.

   NOTE: Any other volatile solvent will work, such as hexanes. However, ethyl acetate is much greener and safer to use than hexanes.

6. Removing Water

Note: Some reactions are very water sensitive. Even the moisture from the air can lead to a failed reaction. Therefore, it is important to drive off water and to keep outside air away from the inside of the glassware. Some glassware like reflux condensers are fragile and cannot be placed under vacuum; for this reason, an oven is preferred. Be aware that the keck-clamps that secure joints between two glass fittings are usually made of plastic and will melt if placed under heat.

1. Use one of the three methods to heat glassware sufficiently enough to remove water:
   a. An oven (this is used for glassware)
   b. A heat gun
   c. Over a hot flame
2. If heating using a heat gun or a flame, place the glassware under vacuum (with the stir bar). This pulls the water vapor out of the flask while drying.
3. If using an oven, place the glassware with the stir bar in the oven and leave for roughly

with a septum and flow nitrogen through the glassware while it is still hot. The nitrogen helps to remove any outside air, which contains a small amount of water vapor; it is important to perform this step while the glassware is hot because water vapor will still be in the gas phase. If allowed to cool down, the water vapor will condense on the glass surface.

4. Safely handle the glassware for reaction setup once the glassware cools down to touch (at this point, the inside of the glassware will be enriched in an inert gas and should contain no water).

Results

Clean glassware is simple and fundamental in organic chemistry practice. The knowledge of how to remove tough stains, residues, and contaminates provides the confidence to use glassware without worry of contamination.

Applications and Summary

By following the guidelines to cleaning glassware, the chemist can focus on the chemistry and worry less about contamination from glassware. The above protocol demonstrated how to clean glassware using warm, soapy water, acid wash, base bath, and aqua regia, and how to dry glassware, as well as advice for cleaning glass ground joints. Disposal of cleaning solutions were also discussed briefly. These glass cleaning procedures work for all kinds of glassware including, but not limited to: round-bottom flasks, Erlenmeyer flasks, separatory funnels, chromatography columns, stir bars, spatulas, funnels, filters, graduated cylinders, beakers, Schlenk flasks, and test tubes.

Having clean equipment in general is important for all types of applications within and outside of chemistry and science. In each case, it is important to consider the material of the item being cleaned and the tools required for cleaning.

We use simple acid and base solutions to clean materials in everyday life. For example, chefs need clean pots and pans, and while these pots and pans do not need acetone for finishing, usually warm, soapy water can remove sticky grease. Interestingly, some people suggest boiling a dirty pot with vinegar to achieve a strong clean, and it is not too surprising that acetic acid is the major ingredient in vinegar. Additionally, baking soda is sodium carbonate and a common house-hold cleaning tool.

Another example of an application where having clean equipment and glassware is essential is quantification analysis for those in an analytical field. Cross-contamination or dirty instrumentation can give rise to unreliable data, affecting many different areas of work; this could be in hospitals, clinics, a crime scene, or on a manufacturing line.

Finally, cleaning products are sold on the market for everyday use for glassware, with the added purpose of cleaning bathroom tiles, windows, and mirrors. These are sometimes filled with organics and may be mildly acidic or basic. This combination can remove grease and water stains.

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