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Cleaning Glassware
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Cleaning Glassware

Overview

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, as a synthetic chemist you need to keep your 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 doing synthetic chemistry. Chemists must be able to rely on clean glassware for their reaction. Just because something looks clean, does not mean that it 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 glassware needs to be is also an important factor. Glassware only needs to be as clean as it is required for the work being done. Over cleaning can actually waste time and money. Understanding of the chemistry can actually assist in evaluating what needs to be done. Covered here are the most basic cleaning 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 glassware in any condition. Instead, a basic understanding on the method and what it can clean is important.

One tip would be to clean the glassware just after its use. Chemists are busy and are generally balancing many tasks, but finding time in between reactions or while a reaction is running is usually a good time.

For safety reasons, each item of glassware should be inspected. Broken glassware is dangerous and should be repaired or discarded. Flasks and 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 that even when cleaning glassware with simple soap and water that eye protection is used. Dirty glassware have dangerous chemicals in them and are oddly shaped. Gloves are good protection too especially when handling harsh cleaning solutions.

Commented [ASW1]: This section is a bit terse. It should include an explanation of each of the procedures (soapy water, acid bath, base bath, etc.), what they do, why you would choose them, and when you should not use them. A lot of this is already in the manuscript, just in the procedure.

For waste disposal, all organic solutions, organic reagents, or any organic contaminated paper or solid should not be disposed through the sink or trash. All organic material should be disposed of in safety bins (to be disposed of by your on site EH&S (Environmental Health & Safety) team, and all organic liquids should be placed in an appropriately labeled liquid container (also to be picked up from EH&S). When washing with water and scrubbing with soap can go down the drain.

Removing grease in 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 and less toxic, it is the preferred choice. If the grease does not come out, then a base-bath will remove most greases used for joints or stopcocks.

Before using any water-based products, all glassware should be rinsed with organic solvent and disposed of in an organic waste container. Using a green polar solvent like acetone is a good choice. This will help to remove most organic residues from a flask before washing. When using organic solvents, remember that like dissolves like. An example of a nonpolar solvent such as hexanes is commonly used. A few commonly used polar solvents include, isopropanol (rubbing alcohol), acetone, ethyl acetate, and methylene chloride (dichloromethane).

Once removed, soapy water can be made by mixing powdered or liquid glassware cleaning reagents with tap water. Different sized brushes will help get into the curved glassware. If glassware is not going to be cleaned right away, a small tub of this soapy water could 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 then with deionized water. Then the water can be removed with acetone.

Ultrasonic cleaners are great for really 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. A base bath can be used outside of glassware includes other dirty objects like stirbars or spatulas. Since a base bath is caustic and can strip away glass, finely calibrated glassware should not be cleaned by this method. Things 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, organic reagents. All acids should be used inside of a well ventilated fumehood. Items that cannot be cleaned by a base bath like 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 inside of the reaction flask. Even if it seems dry, there can be a small thin coating from the moisture in the air. This can be removed with high heat either by placing in an oven or by heating under 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 soap and water. Then the glassware needs to be rinsed with tap water, deionized water, and then with acetone before placing on a rack to air dry. When this fails to clean the glassware, depending on the situation 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 over time developing the knowledge of what solvents or cleaning solutions to use will improve with experience.

Procedure

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

General Procedure for All Glassware (even after treatment from a method below)

Note: this method applies to stir bars, spatulas, funnels, and other reusable equipment.

1. To remove any excess organic oils, rinse glassware briefly with an organic solvent such as acetone. Acetone rinsing should go into organic waste.
2. Using 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. Finally, rinse again with acetone to remove water. This step helps to expedite the glass drying process.

General Acid Wash: 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 tossed down the drain (e.g. hydrochloric acid). Other acids like acetic acid is an organic acid and should be neutralized and combined with water-based liquid waste containers. Usually sodium bicarbonate is used to quench and neutralize acids because it comes with its own visual indicator. Since the reaction of sodium bicarbonate and acid produce CO₂ gas, when it bubbles, there is still acid in solution. When sodium bicarbonate is added and no bubbling is observed, then the solution is neutralized. This can all be placed in aqueous waste.

Commented [ASW2]: The order of cleaning is a little confusing. From what I can gather, you would do an acid bath/aqua regia, then general cleaning, then base bath (though you may not need to perform all of these). Is that correct?

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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 and swirl the solution to the dirty areas. It is sometimes useful to use a cleaning brush, pipet, spatula, or sonication.
3. The acid wash should be neutralized before adding to aqueous waste. A sodium bicarbonate solution will neutralize the acid.
4. The cleaned glassware can then be washed by the general method above.

Base Bath: 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. This base bath is strongly basic and caustic. Care should be taken. 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 disposed along with aqueous waste.

Note: glass frits should not be cleaned using a base bath.

1. In a designated cleaning bucket (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. First clean glassware according to the general method above.
3. Then carefully soak the glassware in the base bath making sure to remove as much air as possible. (Best to soak overnight).
4. When removing the glassware from the bucket, pour back the base bath solution inside of the bucket. Then take the glassware and rinse off any remaining base solution in the sink with ~~Remove as much of the solution in the bucket and rinse with~~ deionized or reverse osmosis water.
5. Dry with acetone.

Commented [ASW4]: Do you mean pour the solution back into the bucket, or something else?

Aqua Regia: Known for its properties in dissolving metals. This acid wash is strongly acidic, oxidizing, and caustic. Care should be taken. Small amounts will cause itching and must be washed off completely by rinsing area under water for 15 min. Serious spills can be neutralized with NaHCO_3 (sodium bicarbonate) and mopped up. Aqua regia also fumes and should be handled inside of a fume hood. It can also corrode stainless steel. Aqua regia should be neutralized with sodium bicarbonate before adding to aqueous waste.

1. Small batches of aqua regia can be made using a 3:1 ratio of nitric acid to hydrochloric acid ($\text{HNO}_3:\text{HCl}$). *Note:* both acids are clear but upon mixing will

fume and turn dark orange.

2. Aqua regia can be added using a pipette or carefully poured and swirled to dissolve residual metals.
3. Remove most aqua regia by pouring into a second container to be treated and disposed of in aqueous waste.
4. Clean glassware according to general method as described above.

Cleaning Glass Ground Joints: Ground joints can easily become stuck. One way to prevent this problem is to make sure all joints are clean. When clean, 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.



1. Ground glass joints should be cleaned after cleaning the glassware by one of the methods above.
2. Cleaning the inside of ground glass joints can be tricky. The best way is to use a paper towel soaked in ethyl acetate (EtOAc) and a small spatula.
3. Cleaning the outside of ground glass joints is easy using a paper towel soaked in ethyl acetate.
4. Wet the tip of a paper towel with EtOAc and use the spatula to get into the edges of the joint.
5. A gentle blow should evaporate the ethyl acetate will evaporate in seconds and reveal a frosted white ground glass joint. Any other volatile solvent will

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Commented [ASW6]: Is this paper towel also soaked in EtOAc?

Commented [ASW7]: What do you use for this? A house air or nitrogen line?

work, such as hexanes. However, ethyl acetate is much greener and safer to use than hexanes.

Removing Water: 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 the keck-clamps that secure joints between two glass fittings are usually made out of plastic and will melt if placed under heat.

1. Three methods can be used to heat glassware sufficiently enough to remove water.
 - a. Placing in an oven (used for glassware)
 - b. Using a heat gun
 - c. Over a hot flame
2. If heating using a heat gun or if using 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 and leave in the oven for roughly 30–60 min (or longer). When removing, use oven gloves and quickly cap the glassware with a septum and flow nitrogen through the glassware while hot. Using nitrogen is to help remove any outside air which contains a small amount of water vapor. Enriching in nitrogen is important to do while hot because water vapor will still be in its gas phase. If allowed to cool down, the water vapor will condense on the glass surface.
4. Once the glassware cools down to touch, ~~the inside should~~ will be enriched in an inert gas and should contain no water and will be safe to handle for reaction setup, and should be ready for the reaction.

Commented [ASW8]: Are there reasons these should or should not be used for certain types of glassware/equipment?

Commented [ASW9]: What do you do once it cools to the touch?

Representative Results

As stated before, having clean glassware is simple and fundamental in organic chemistry practice. With the knowledge of how to remove tough stains, residues, and contaminants will give the user the confidence to use glassware without worry of contamination.

Summary

Guidelines to cleaning glassware as demonstrated above leaves the chemist focused on the chemistry and less worried about contamination from glassware. The above protocol demonstrated how to clean glassware using warm, soapy water, acid wash, base bath, aqua regia, and how to dry glassware as well as tips for cleaning glass ground joints. Disposal of cleaning solutions were also touched upon. These glass cleaning procedures work for all kinds of glassware including, but not limited too: round-bottom flasks, Erlenmeyer flasks,

separatory funnels, chromatography columns, stir bars, spatulas, funnels, filters, graduated cylinders, beakers, Schlenk flasks, and test tubes.

Applications

Clean glassware goes beyond the organic chemists bench. 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 users tools for cleaning.

Chefs and cooks need clean pots and pans as well as utensils. While these pots and pans do not need acetone for finishing, usually warm soapy water is called for to remove sticky grease. Interestingly, some people suggest boiling a pot with vinegar to get it really clean and it is not too surprising that acetic acid is the major ingredient in vinegar. Baking soda is also sodium bicarbonate. The next time you clean an appliance with vinegar or baking soda, you are actually cleaning using a simple acid or base solution!

Another application where having clean equipment and glassware is needed, are those in the analytical field, namely those who quantify things need clean equipment. Cross-contamination or dirty instrumentation can give rise to unreliable data. This could be in hospitals, clinics, a crime scene, or on a manufacturing line.

There are also many products on the market sold for the purpose of cleaning glassware, as well as 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.

Materials List

Name	Company	Catalog Number	Comments
Equipment			
Fume hood			Any fume hood that complies with AIHA/ANSI Standards
Ultrasonic cleaner	Fisher Scientific	15-337-400	Or any other standard ultrasonic cleaner
Eye protection			Any goggle or glasses that complies with AIHA/ANSI standards
Lab coat			Any lab jacket that complies with AIHA/ANSI standards
Glassware or equipment			
Reagents			
Acetone	Sigma-Aldrich	179124-20L	ACS reagent
Ethyl acetate	Sigma-Aldrich	319902-20L	ACS reagent
2-Propanol	Sigma-Aldrich	278475-2L	ACS reagent
Sparkleen 1 Detergent	Fisher Scientific	S701101	
Hydrochloric acid	Fisher Scientific	A142-212	

Acetic acid	Fisher Scientific	A38-212	Glacial
Nitric acid	Sigma-Aldrich	438073-2.5L	70%
Potassium hydroxide	Sigma-Aldrich	484016-1KG	Pellets