GLASSWARE CLEANING

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Materials Needed

Large clean sink and an undisturbed work area to allow glassware to drain and to set glassware containing acid.

Detergent (Use a non-phosphate detergent such as Sparkleen, Micro, or Octagon. Octagon can be bought from any grocery store and is fairly inexpensive.).

Brushes (thin bristle with long wire handle, thick bristle and small test tube size).

Distilled water (purchase or locate through other treatment plants in the area; if there isn't available distilled water, try a grocery store and then a pharmacy).

Sink drain (rubber or plastic dish drains are best with a rubber mat underneath to drain water into the sink; dishwasher racks also make excellent drainers).

Rubber gloves, safety clothing and safety glasses are also suggested.

STEP 1

Fill the sink with very hot soapy water, using a non-PO_4 (phosphorus) detergent. Clean glassware and sampling bottles by soaking in sink and scrubbing each item inside and outside, as well as the lips and edges of the items. Rinse each thoroughly with hot tap water to get soap film off. Drain excess water out of and off of items.

STEP 2

Fill each item to the very top with the acid designated for its use (see Section 2, Page 3) and let stand 15 minutes. Fifteen minutes is the time it takes for acid to be effective on glassware and containers. If the acid sits in the container longer than 15 minutes, it will not adversely affect its cleaning ability. If certain glassware is very dirty, let it set with acid longer or overnight. If the acid seems to be ineffective, pour it slowly down the sink drain diluting heavily with tap water. Add fresh acid and let set for the necessary time. Pour acid back into a clean glass gallon jug or other acid container to be reused. Rinse each item with tap water, followed by three (3) rinses of distilled or de-ionized water, making sure entire surface is rinsed by swirling or shaking water about before dumping down the sink.

STEP 3

Set glassware and plastic items upside down on racks or dish drains to dry. Do not let glassware set any longer than necessary to dry or it will collect dust and various organics, as well as ammonia, which contaminate the glassware and cause interference with specific test results. Some glassware can be dried effectively in ovens set at 103-105°C. Pipets and small volumetrics are easiest to dry in an oven. To use oven with metal racks, first cover the racks with aluminum foil. Replace foil as needed.

Keep glassware in dust-free, clean places, either in bench drawers or on closed-in shelves.

Specific Glassware And Cleaning Solutions Used

Chromic acid is a commonly used glassware cleaning reagent. It is prepared in a one liter container by dissolving 60 grams of potassium dichromate in approximately 150 mls of warm distilled water and then slowly adding concentrated sulfuric acid to produce a total volume of one liter Chromic Acid solution. A serious drawback to this solution is that it is quite dangerous to use. Some technicians also complain that a film is often left behind on the glassware.

Another popular solution used for glassware cleaning is Micro. A 2% solution of Micro is made by adding 200 mls of Micro to approximately 9.8 liters of distilled water. This solution is a very effective cleaner, is much safer than Chromic Acid and is not known to leave a film. It can be reused until it starts to look like dirty dish water. Micro can be purchased from:

International Products Corporation
PO Box 118
Trenton, NJ 08601-0118

Special precautions must be taken when cleaning glassware for the PHOSPHORUS and COLIFORM analyses.

Glassware to be used for the analysis of Total Phosphorus should be washed in 10% Sulfuric acid or 50% HCL. It should be allowed to soak in this acid for at least 15 minutes. Then the glassware must be THOROUGHLY rinsed with high-quality distilled water. Finally, to ensure that the glassware is ready for use it should be allowed to soak in the Combined Reagent used in the Phosphorus analysis for approximately fifteen minutes. If no blue color develops the glassware contains no phosphates and can be then rinsed once again THOROUGHLY with distilled water and set in a clean dust-free environment until used. This glassware should be dedicated for use in the analysis of Phosphorus only!

Appropriate Solutions And Specific Glassware

10% HCL	$10\%H_2SO_4$	2% Micro	$20\% HNO_3 + 10\%$ HCL
TKN Bottles & Equipment	Chloride Bottles	Test Tubes	COD Bottles & Equipment
	Volumetrics	BOD Bottles	
	Flasks	TSS Crucibles	Metals, Bottles & Glassware
	$T\text{-}PO_4$ Glassware	Pipets	
	Graduated Cylinders	Burets	
	Beakers		
		Influent Sample Bottles	
		Reagents Containers	
		ISCO Bottles & Lids	
		Very Dirty Glassware (20%)	

No Acid

Coliform Bottles

Coliform Dilution Bottles

Acid Used For Glassware Preparation

10% HCL:

Slowly add 100 mls of concentrated reagent grade HCL to 900 mls distilled water in a one (1) liter beaker. Place a beaker cover over the beaker and let cool before pouring into a storage container.

10% H_2SO_4:

Slowly add 100 mls of concentrated reagent grade H_2SO_4 to 900 mls distilled water in a one (1) liter beaker. Place a beaker cover over the beaker and let cool before pouring into a storage container.

20% HNO_3:

Slowly add 200 mls of concentrated HNO_3, nitric acid, to 800 mls distilled water in a one (1) liter beaker. Place a beaker cover over the beaker and let cool before pouring into a storage container.

Proper Care And Cleaning Of Pipets

Clean pipets should be stored in a lined drawer (with paper towel) so they do not collect dust or splashes from lab chemicals or samples. When using pipets, place them on the bench on a paper towel so the tip does not touch anything which may contaminate the pipet. If pipet is dirty or contaminated, it will contaminate the liquid (reagent, sample or acid) and its container when pipet is immersed.

To clean pipets, fill a large, approximately one liter plastic container with 2% Micro solution. Soak pipets in Micro overnight. Keep pipets upright in Micro and make sure solution has covered all of the pipet from top to bottom. Remove pipets from Micro solution, allowing pipets to drain out completely. Rinse with tap water followed by several rinses with distilled water, either by holding pipets in a stream or by letting the pipets set in another container and flush with distilled water. Be sure water rinses through the pipet.

It is best to initially put dirty pipets in a container filled with tap water rather than let the sample or reagent solution dry on the pipet. This procedure allows easier cleaning later and prolongs the shelf life of the 2% Micro cleaning solution.

Coliform Sample Bottle Preparation

Wash sample bottles in the same manner as other glassware, using hot soapy water and a brush that fits inside the coliform bottle. Rinse out with tap water followed by at least two rinses of distilled water. Let dry upside down on drying rack. Wash caps the same way. Make a 10% solution of sodium thiosulfate by adding 10 grams $Na_2S_2O_3$ to 100 mls distilled water. Pipet four (4) drops of this 10% mixture into each 250 ml sample bottle and put cap on loosely. Do not tighten down bottle cap until bottles have been autoclaved or heated (glass bottles only) for the necessary time and then cooled completely. Place bottles upright in autoclave, pressure cooker or oven, keeping sodium thiosulfate on bottom of bottle. Autoclave or pressure cook for 15 minutes at 250°F, approximately 15 psi.

Pipetting

Begin by selecting a clean and dry pipet, making sure the tip is not broken. When pipet tip is broken, it will be difficult to use and may hold an inaccurate amount of liquid, so dispose of it. If pipet is clean but still has distilled water inside, draw up reagent to be used and discharge it, repeating this procedure again to clear any traces of water which would dilute the reagent or sample.

When using any **volumetric pipets**, use the following procedure:

> Submerse pipet just below liquid surface and, using pipet bulb, draw liquid above the line on the pipet. Quickly place one finger on the top of the pipet and remove pipet from liquid. Wipe off excess moisture on the outside of the pipet with a Kimwipe. Holding pipet vertically at eye level, slowly allow the liquid, drop by drop, to run out down to the top of line, not below it. With a Kimwipe or paper towel, touch the tip of the pipet with a corner of the towel if a drop of liquid adheres to the tip at this point. Allow exact amount of liquid to run out by placing the tip against the inside of the vessel and hold for 15 seconds (or specified time) after liquid is out to completely drain pipet. Most volumetric pipets will include specific instructions near the top of the pipet. Example: T.D. at 20°C 15 seconds. This means the pipet is designed to deliver an exact amount of solution if at 20°C and allowed to drain for 15 seconds. Never blow or shake the last drop from a volumetric pipet!

When using **graduated pipets**, the procedure is somewhat different:

> Immerse the pipet below the liquid surface and draw liquid up above the zero line. Holding pipet vertically at eye level, allow liquid to run down to the level of the zero line. Discharge the desired amount of liquid into the vessel and dispose of remaining liquid; do not put it back in the reagent bottle. Graduated pipets with measured lines to the tip of pipet require that liquid be blown out completely.
>
> Generally, serological pipets are marked with either a single white ring or double white rings near the top of the pipet. The single white ring signifies that the pipet is calibrated "To Deliver" and should not be blown out. Pipets with two white rings must be blown out in order to deliver the specified volume.

Burets

To **prepare** buret for use:

> Rinse buret three times with reagent, using 5 to 10 milliliters (mls) each time for a 50 milliliter (ml) buret. To do this, pour reagent in from the top with the stopcock open, shut off the stopcock and rotate the buret to rinse all sides, tip the buret and allow to drain with stopcock open.

To **fill** buret for use:

> Pour reagent in with stopcock open until liquid (reagent) level is above the lowest line level on the buret and then close off. This maneuver keeps air bubbles from entering the buret. Fill the buret to above the zero line. Place in a buret stand and allow excess to run out slowly, by opening up the stopcock, a small amount, until the level reaches the zero line.

When reagent has been left in buret overnight, allow all liquid in the buret to run freely out and refill with fresh reagent before titrating.

The titrating method may vary between persons but generally one hand swirls the Erlenmeyer while the other hand controls the amount and speed in which the reagent is discharged.

Titrate to the end point described in the test procedures, until sample changes color, loses color, etc. Use back titrant, if available, to verify the endpoint. If titration is correct, read the level of the reagent to 0.05 mls.

Measuring Liquids

Glassware such as beakers or flasks should NEVER be used to measure samples, chemicals, reagents, or distilled water where exact measures are necessary. Marks on these types of glassware should only be used as rough measures and estimates. When making up acids for cleaning or when needing an approximate amount and where relatively large inaccurate amounts are needed, it is easiest to use such glassware.

Volumetric flasks are used to measure out exact volumes when preparing reagents and standards, or diluting samples. Fill to level of the line inscribed on neck.

Volumetric pipets can be used when the exact amount is required in direction such as "add 10.0 mls or 10.00 mls" in any method. Graduated pipets can be used when the exact amount is not as critical to the test involved.

Burets are the most accurate devices for measuring in-between volumes that cannot be measured by the standard sizes of volumetric pipets (e.g., 18.75 mls).

The following pages show examples of common laboratory glassware. Taken from "Operation of Wastewater Treatment Plant Volume II".