ST. PAUL'S COLLEGE, KALAMASSERY

CHEMISTRY LABORATORY MANUAL

LABORATORY MANUAL

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UG/PG CHEMISTRY LAB

The Department of Chemistry offers courses in M. Sc. and B. Sc. Chemistry along with Chemistry as a subsidiary programme for the students who have taken Physics as their main subject. The College has two labs for Chemistry practical. Each lab has sufficient number of equipment and facilities. The chemical stockroom, instrumentation room and a balance room are situated within the UG chemistry lab. The PG lab consists of physical, organic, inorganic labs and a computer lab. Both the labs are equipped with fume hoods, working benches and specialized equipment. Each student is assigned to his/her own locker which contains laboratory glassware to perform experiments.

Our laboratories with advanced equipment and facilities aid and stimulate our students for them to learn best with practical knowledge.

GUIDELINES FOR CARRYING OUT EXPERIMENTS

LAB RULES

- Bring the laboratory manual and a laboratory notebook (not loose leaf or spiral) to every scheduled laboratory session.
- Careful notes should be taken during each laboratory lecture. The instructor will generally provide information on the chemistry underlying the project, as well as advice on the techniques that you will use. Some of this information should be included in your laboratory report.
- You may work in the Chemistry PG and UG laboratories only during your regularly scheduled laboratory period and only when class is in session.
- If you miss a laboratory for a legitimate reason, you must obtain permission from your regular laboratory instructor to make-up the lab at another time. Make-up labs are only allowed when space allows and with the approval of the host professor. During the make-up laboratory, you must move your equipment to an unoccupied lab bench.
- During the first scheduled laboratory period, you will be assigned a laboratory locker to which you alone will have the combination. The locker equipment is your responsibility while you are in the course and you should be diligent to keep the equipment clean.
- Test tube brushes and soap solutions are available at each sink.
- The Chemistry Department maintains a stockroom to store the chemical and apparatus. This stockroom is open during all scheduled laboratory periods for the acquisition of equipment necessary to replace broken items.

LAB SAFETY

TRAINING AND SAFETY RULES

- Attendance is required at a presentation on laboratory safety that will be shown during your first scheduled laboratory. Each student must give the instructor a signed form indicating that the presentation was attended and that any associated training materials were examined.
- The laboratory is equipped with fire blanket, showers, eye wash, and first aid supplies.
- Learn the locations and proper use of these items.

At all times when you are working in the chemistry laboratory you should use prudent practices. Recognize that safety is, ultimately, everyone's individual responsibility. Never work alone in any laboratory.

Avoid the most common causes of accidents

- Exercise care when picking up potentially hot objects.
- Insert glass objects into rubber stoppers and corks with extreme care.

Avoid contact with laboratory chemicals

- Wear clothing that protects as much of your body as possible. Closed-toe shoes are required. All skin below the waist must be covered.
- Use department-approved eye-protection at all times.

- Keep the laboratory bench and work area orderly, clean, and free of items not related to the experiment at all times. Specifically, electronic devices are not allowed on the bench.
- Never sit on or lean against the laboratory bench.
- Use a fume hood when directed to do so.
- Food or drink should only be consumed in the lecture area of the room. Do not chew gum during laboratory sessions.
- Dispose of waste materials and excess chemicals in the appropriate containers as indicated by your instructor.

When emergencies do occur

- Always keep in mind that the first response to the exposure of the eyes or skin to a chemical is immediate, thorough irrigation with water.
- Report all accidents, however minor, to the laboratory instructor immediately.
- Know the exact location of all safety equipment and how to use it.

Preparation is important

- Perform only assigned experiments. Do not attempt to modify the written procedures unless instructed to do so.
- When conducting experiments ask yourself, "What are the worst possible things that could go wrong?" and "How will I deal with them?" Don't do the experiment until you are certain of your answers.
- Read the label on the container to be certain it contains the required chemical.

LABORATORY MANUAL

FIRST AID & EMERGENCY MEASURES

[1] Acid on clothing: Neutralize with dilute ammonium hydroxide. Then wash thoroughly with water

[2] Alkali on clothing: Neutralize with dilute acetic acid. Then wash thoroughly with water.

[3] Acid in eye: Wash thoroughly and profusely with running water. Bathe the eye with a 2 % sodium bicarbonate solution, using an eye cup. Dry with sterile gauze and put several drops of olive oil into the eye.

[4] Alkali in eye: Wash thoroughly and profusely with running water and the eyelids should be held widely open especially when caustic alkalies have entered the eye. Bathe with boric acid solution. Dry and add a drop of olive oil into the eye.

[5] Ordinary heat burns: Don't use water. Apply sodium bicarbonate, Vaseline paste or burnol and consult a doctor

[6] Acid burns: Wash first with running water and then with sodium bicarbonate solution. Cover with solid sodium bicarbonate for 10 minutes. Wash off and apply carron oil (a mixture of equal parts of lime water and linseed oil).

[7] Alkali burns: Wash first with running water and then with boric acid solution. Cover with powdered boric acid for 10 minutes. Wash off and apply sodium bicarbonate Vaseline paste.

[8] Cuts: Remove particles of glass, if any are present. Wash the wound with water. Then apply tincture of iodine and cover with a sterile bandage

[9] Poisoning by strong acid: Give plenty of water or milk. Then give two table spoons of lime water

[10] Poisoning by caustic alkalies: Give plenty of water or milk. Then give orange or lemon juice

[11] Fire: Extinguish gas burners in the vicinity. Use buckets of dry sand/fire-extinguishers to cease the fire.

LAB SAFETY RULES

- Avoid skin and eye contact with all chemicals
- Minimize all chemical exposures
- Do not engage in practical jokes or boisterous conduct in the laboratory
- Never run in the laboratory. No horseplay will be tolerated
- The use of personal audio or video equipment is prohibited in the laboratory
- The performance of unauthorized experiments is strictly forbidden
- Do not sit on laboratory benches
- Always wear a lab coat
- Laboratory safety glasses or goggles should be worn in any area where chemicals are used or stored. They should also be worn any time there is a chance of splashes or particulates to enter the eye
- Avoid skin and eye contact with all chemicals
- Do not taste or intentionally sniff chemicals
- Never consume and/or store food or beverages or apply cosmetics in areas where hazardous chemicals are used or stored
- Long hair and loose clothing must be pulled back and secured from entanglement or potential capture
- Know emergency procedures
- Never work in the laboratory without the supervision of an instructor
- Always perform the experiments or work precisely as directed by your instructor
- Never leave experiments while in progress
- Never attempt to catch a falling object
- Never point the open end of a test tube containing a substance at yourself or others
- Do not leave the Bunsen burners unattended. Make sure no flammable solvents are in the surrounding area when lighting a flame
- Turn off all heating apparatus, gas valves, and water faucets when not in use
- Keep the floor clear of all objects (eg. Ice, small objects and spilled liquids)

LABORATORY MANUAL

LABORATORY NOTEBOOKS

One of your goals in this laboratory course should be to learn to keep proper records of your work. Your laboratory reports will be based on the data in your notebook, and the more complete the data are the more likely it is that you will be able to prepare a good report. Furthermore, discrepancies and unexpected results can be accounted for only by referring to complete records of your work. In a broader sense, a notebook is essential in any research laboratory where it may be necessary to review data months or years after they were taken; hence full details are necessary.

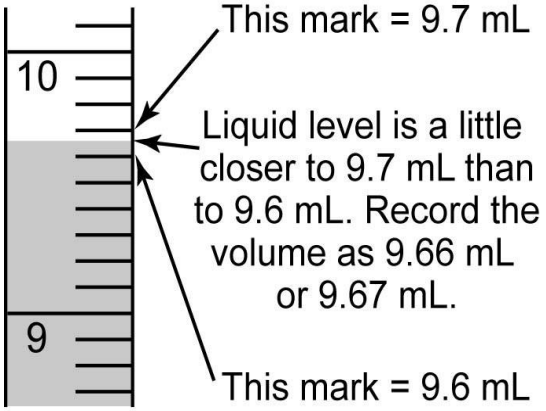
You will be required to keep your laboratory records in a hard-cover, bound notebook. The notebook should contain all experimental data and pertinent observations recorded in ink at the time they are obtained. Data may not be recorded elsewhere for later recopying in the notebook, and, in particular, loose scraps of paper are not permissible for records. The following are the requirements for your notebook:

- Each day's work should be dated. The project being performed should be indicated clearly.
- No erasures should be made, and mistakes should be crossed out with a single line but remain legible.
- Pages must not be removed from the notebook.
- The notes must be neat and orderly enough for someone else to follow them.
- Use tables to organize data whenever possible.
- The following should be included in the laboratory notebook:
- All experimental data, such as masses, buret readings, temperature, etc.
- Notable occurrences (especially phase or color changes).
- Full details of a procedure need not be recorded, but each step should be noted as it is performed.
- All mathematical computations during and after the laboratory session.

RECORDING SIGNIFICANT FIGURES IN YOUR NOTEBOOK

When recording a numeric measurement, the number of significant figures conveys how accurate the measurement is.

- When massing, record all digits on the balance. The last place has uncertainty in it (you may even see the digit change).
- When measuring volumes using graduated glassware, estimate and record one digit beyond the markings, as shown below in the figure.



EFFECTIVELY COMMUNICATING THROUGH A LABORATORY REPORT

Writing is ubiquitous in the sciences, and students primarily develop their writing skills through writing laboratory reports. As a scientist-in-training, you ultimately need to learn how to make a reasoned and articulate argument that is persuasive and based on scientific evidence; the foundation of that argument needs to be grounded in data that are presented in an organized and thorough manner. The laboratory report communicates to others the results and conclusions you obtained in performing an experiment. You should explain why the experiment was performed, how the experiment was performed, what results were obtained, how the results were analyzed, and what conclusions were reached.

EXPERIMENTAL INSTRUCTIONS

VOLUMETRIC ANALYSIS

Volumetric analysis is a widely used quantitative analytical method. As the name implies, this method involves the measurement of volume.

VOLUMETRIC PROCEDURE

[1] A solution is prepared from an accurately weighed sample of the material to be analyzed.

[2] A substance is chosen that will react rapidly and completely with the constituent that is to be determined. A **standard solution** of this substance is prepared. A standard solution is one of accurately known concentration, usually expressed as molarity with a precision of ± 0.0001 M.

[3] Some of the standard solution is poured into a **buret**. The buret is graduated (usually in tenths of a mL) so that the volume of solution that passes through the stopcock may be accurately measured.

[4] Standard solution is added slowly from a buret to the "unknown" solution, allowing the reaction to occur. This process is called **titration**, and the solution in the buret is known as the **titrant**. Ideally, the titration is continued until the reaction is complete; that is, until the amount of reactant added is exactly the amount required to react with the entire constituent that is being determined. This point is called the **equivalence** point. The equivalence point is detected by adding an **indicator** to the "unknown" solution before the titration is begun. An indicator is a substance that gives a color change at or near to the equivalence point. The point at which this change occurs is called the **endpoint**. The particular indicator that is used depends on the specific reaction involved. The titration is stopped when the endpoint is reached.

[5] The exact volume of standard solution required can be measured, from buret readings before and after the titration. Since the molarity of the standard solution is known, the number of moles of titrant can be calculated. Furthermore, from knowledge of the equation for the reaction, the number of moles of constituent present in the sample can also be calculated.

STANDARD SOLUTION

The most accurate and convenient way of preparing a standard solution is to weigh the reagent accurately, dissolve it, and dilute the solution to a definite volume in a volumetric flask. This method can be employed only if the reagent is a **primary standard**. In order to qualify as a primary standard, a substance must meet the following requirements: it must be obtainable in pure form; it must be stable both in pure form and in solution; it must be easy to dry and keep dry; and it must be soluble in a suitable solvent.

Unfortunately, many useful reagents do not meet these requirements. In such a case, the reagent is dissolved and made up approximately to the concentration desired. This solution is then standardized by titrating it against a primary standard. A solution standardized in this fashion is a **secondary standard**.

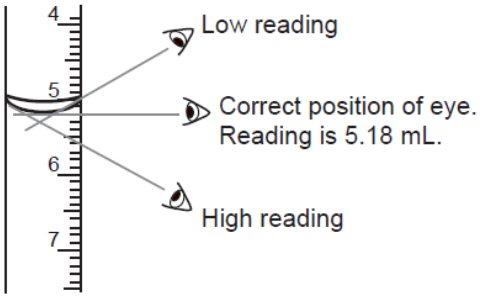
TITRATION PROCEDURE

[1] Carefully clean the buret with Alconox solution and a brush to remove all dirt and grease. Rinse the buret with several portions of tap water by partially filling it, draining a small portion through the tip, and pouring the bulk of the rinse from the top of the buret while rotating it. If any drops of water collect on the walls when the rinse is poured from the buret, the cleaning is unsatisfactory and must be repeated. Finally, rinse the buret with two or three portions of deionized water.

[2] Before filling the buret, rinse it with titrant solution 2-3 times, using about 10 mL portions.

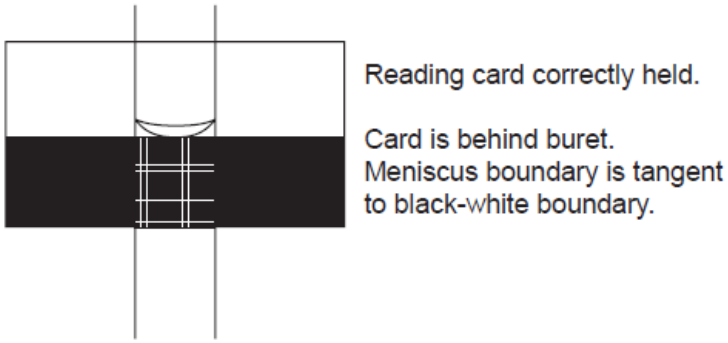
[3] Place the buret in a buret clamp attached to a large ring stand. Using a funnel, fill the buret with titrant to a level above the zero mark. Place a beaker under the buret and open the stopcock for a few seconds to remove all air from the tip. The top of the solution should now be below the zero mark.

[4] **Read the buret to ± 0.01 mL**. (Because the buret is graduated to 0.1 mL, the second decimal place must be estimated.) To make this reading, it is necessary to locate the meniscus (that is, the surface of the liquid) with respect to the markings. The reading is considerably affected by the position of the eye and by the color of objects behind the buret. The variation of the apparent position of the meniscus is called **parallax**. To minimize errors due to parallax, the meniscus should be level with the eye (see the Figure given below).



Avoid parallax error by using correct eye position.

The variation of colors behind the buret can be eliminated by mounting a piece of black tape on a white card and holding it behind the buret as shown in Figure 10. If a highly colored liquid is used, it is more convenient to read the position of the **top** of the meniscus.



Reading card correctly held.

[5] Place the solution that is to be titrated in an Erlenmeyer flask and add the appropriate indicator. Position the flask under the buret. Add the titrant **slowly** from the buret while swirling the contents of the flask to assure adequate mixing. As the endpoint is approached, the titrant must be added very slowly-a drop at a time. Usually there is warning as the endpoint is approached. If the endpoint is a color change, the change is produced momentarily where the reagent drops into the solution, but fades with stirring into the bulk of solution. This fading occurs more slowly as the endpoint is approached.

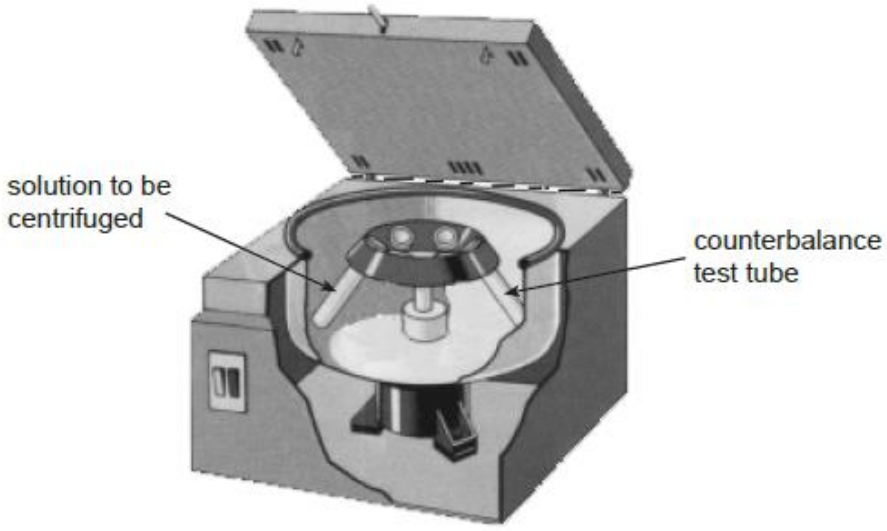
[6] If the indicator change is a very sharp one, it may be desirable to add standard solution only a half drop at a time near the endpoint. This may be done by opening the stopcock slightly until a drop begins to form on the buret tip. When the droplet has grown to a few hundredths of a mL (one drop is about 0.05 mL), it is touched to the side of the titration vessel and rinsed down with a little deionized water from the wash bottle.

[7] If an endpoint is not distinct, or if it is unfamiliar, it may be difficult to decide when the endpoint has actually been reached. In this case, record the buret reading, add another drop, and note the change produced. If the observer is still uncertain, another reading should be recorded, and another drop added. When a series of such readings have been recorded it is easier to select the endpoint in retrospect than by direct approach.

[8] When the endpoint has been reached, subtract the initial buret reading (step 4 above) from the final reading to obtain the volume of titrant used.

CENTRIFUGATION

Precipitates are separated from their mother liquors by centrifugation. Keep the centrifuge balanced by placing a counterbalancing test tube, filled with an equal volume of water, directly opposite to the test solution. The solution should be centrifuged for several minutes to pack the precipitate in the bottom of the tube. The mother liquor can be withdrawn carefully with a capillary pipet.



Counterbalance the solution to be centrifuged.

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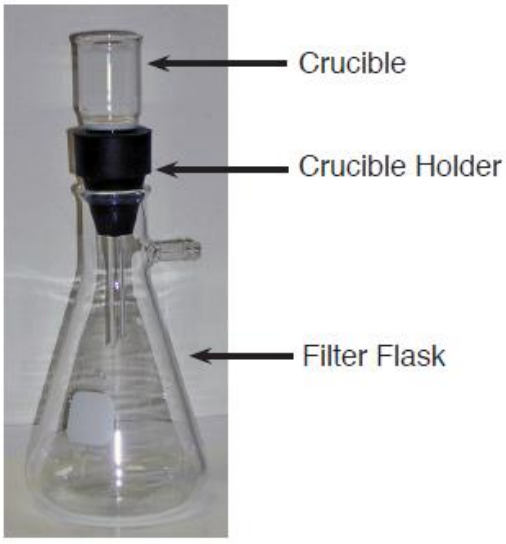
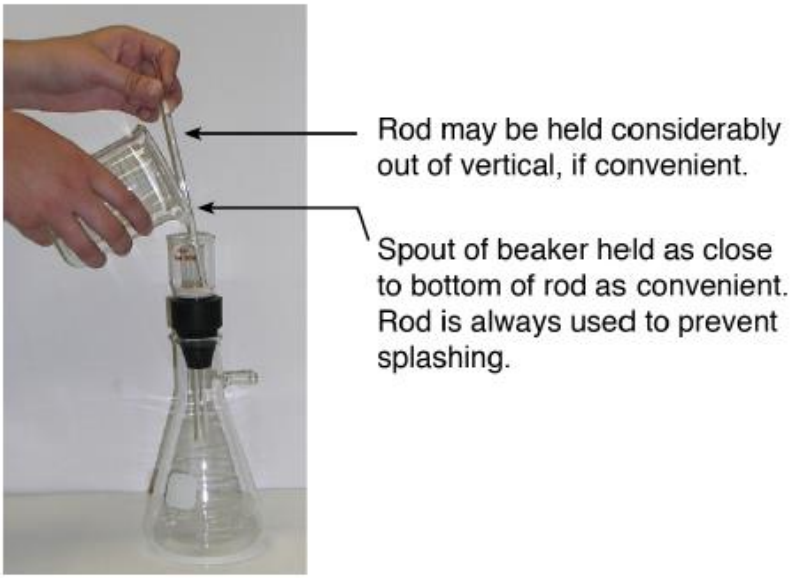
FILTERING A PRECIPITATE

The most convenient device for collection of the precipitate is a filter crucible, which is a glass crucible with a porous disc in the bottom, or a Büchner funnel with filter paper. The filter crucible, using a crucible holder or the Büchner funnel, is mounted on a filtering flask and the filtration is accomplished by suction.

As much of the mother liquor as possible is decanted through the filter without disturbing the precipitate in the beaker, so that the major portion of the liquid may be filtered rapidly and before the precipitate begins to clog the filter. Figure 24 illustrates the pouring operation. It is good to keep the filter crucible always fairly full of liquid. After the filter crucible is filled, it may be necessary to interrupt the pouring and to set the beaker on the table while waiting for the crucible to empty. When pouring is interrupted, the beaker is not turned upright immediately, because there is a tendency for a drop to adhere to the outside of the spout and to run down and be lost. Instead, the beaker is tipped back only slightly and the clinging drop is transferred to the rod by touching the rod to the beaker spout. The drop adhering to the end of the rod is removed by touching it to the side of the crucible. The rod is laid across the top of the beaker with the wet end resting on the spout.



A filtering apparatus crucible



Correct method of transferring a solution to a filter

QUALITATIVE INORGANIC ANALYSIS

Qualitative chemical analysis is a branch of chemistry that deals with the identification of elements or grouping of elements present in a sample. The techniques employed in qualitative analysis vary in complexity, depending on the nature of the sample. In some cases it is necessary only to verify the presence of certain elements or groups for which specific tests applicable directly to the sample (*e.g.,* flame tests, spot tests) may be available. More often the sample is a complex mixture, and a systematic analysis must be made in order that all the constituents may be identified. It is customary to classify the methods into two classes: qualitative inorganic analysis and qualitative organic analysis.

<table>
<tr><th colspan="6">Scheme for separation of cations</th></tr>
<tr><td colspan="6">HCl or a soluble chloride, preferably NH_4Cl, added to unknown; filtered</td></tr>
<tr><td rowspan="3">precipitate: contains chlorides of lead (Pb), silver (Ag), and mercurous mercury (Hg)

$PbCl_2$ (white)
AgCl (white)
Hg_2Cl_2 (white)

Group I</td><td colspan="5">solution: H_2S passed into the acid solution; filtered</td></tr>
<tr><td colspan="2">precipitate: treated with NH_4OH; ammonium polysulfide $(NH_4)_2S_x$; and $(NH_4)_2S$; filtered</td><td colspan="3">solution: neutralized with NH_4OH and NH_4Cl; filtered</td></tr>
<tr><td>precipitate: contains cupric, lead, cadmium, bismuth, and mercuric sulfides

Group IIa</td><td>solution: contains arsenic, antimony, and tin cations

Group IIb</td><td>precipitate: contains aluminum (Al), chromium (Cr), and ferric (Fe) hydroxides

$Al(OH)_3$ (white)
$Cr(OH)_3$ (gray-green)
$Fe(OH)_3$ (brown)

Group IIIa</td><td>solution: H_2S passed into alkaline solution; filtered</td></tr>
<tr><td colspan="3"></td><td>precipitate: contains cobalt (Co), nickel (Ni), manganese (Mn), and zinc (Zn) sulfides

CoS (black)
NiS (black)
MnS (buff)
ZnS (white)

Group IIIb</td><td>solution: evaporated and NH_4OH and $(NH_4)_2CO_3$ added; filtered</td></tr>
<tr><td colspan="4"></td><td>precipitate: barium, strontium, and calcium carbonates (all white)

Group IV</td><td>solution: contains magnesium, sodium, and potassium ions

Group V</td></tr>
</table>

QUALITATIVE ORGANIC ANALYSIS

The analysis and identification of unknown organic compounds constitutes a very important aspect of experimental organic chemistry. There is no definite set procedure that can be generally applied to organic qualitative analysis. Various books have different approaches, but a systematic approach based on the scheme given below will give good results. Students should, however, consult the laboratory manual and Textbook of Practical Organic Chemistry, A.I. Vogel (4th Edition).

S.no	Experiment	Observation	Inference
	Preliminary tests		
1	**Odour:** Note the Odour of the organic compound.	(i) Fish odour (ii) Bitter almond odour (iii) Phenolic odour (iv) Pleasant fruity odour	(i) May be an amine (ii) May be benzaldehyde (iii) May be phenol (iv) May be an ester
2	**Test with litmus paper:** Touch the Moist litmus paper with an organic compound.	(i) Blue litmus turns red (ii) Red litmus turns blue (iii) No colour change is noted	(i) May be a carboxylic acid or phenol (ii) May be an amine (iii) Absence of carboxylic acid , phenol and amine
3	**Action with sodium bicarbonate:** Take 2 ml of saturated sodium bi carbonate solution in a test tube. Add 2 or 3 drops (or a pinch of solid) of an organic compound to it.	(i) Brisk effervescence (ii) No brisk effervescence	(i) Presence of a carboxylic acid. (ii) Absence of a carboxylic acid.
4	**Action with Borsche's reagent:** Take a small amount of an organic compound in a test tube. Add 3 ml of Borsche's reagent, 1 ml of Conc HCl to it, then warm the mixture gently and cool it.	yellow or orange or red precipitate	Presence of an aldehyde or ketone
5	**Charring test:** Take a small amount of an organic compound in a dry test tube. Add 2 ml of conc H_2SO_4 to it, and heat the mixture.	Charring takes place with smell of burnt sugar	Presence of carbohydrate

	Tests for Aliphatic or Aromatic nature:		
6	**Ignition test:** Take small amount of the organic compound in a Nickel spatula and burn it in Bunsen flame.	(i) Burn with sooty flame (ii) Burns with non sooty flame	(i) Presence of an aromatic compound (ii) Presence of an aliphatic compound
	Tests for an unsaturation:		
7	**Test with bromine water:** Take small amount of the organic compound in a test tube add 2 ml of distilled water to dissolve it. To this solution add few drops of bromine water and shake it well.	(i) Orange - yellow colour of bromine water is decolourised (ii) No Decolourisation takes place (iii) Decolourisation with formation of white precipitate.	(i) Substance is unsaturated. (ii) Substance is saturated. (iii) Presence of an aromatic amine or phenol.
8	**Test with $KMnO_4$ solution:** Take small amount of the organic compound in a test tube add 2 ml of distilled water to dissolve it. To this solution add few drops of very dilute alkaline $KmnO_4$ solution and shake it well.	(i) Pink colour of $KmnO_4$ solution is decolourised (ii) No Decolourisation takes place	(i) Substance is unsaturated. (ii) Substance is unsaturated.

	TEST FOR SELECTED ORGANIC FUNCTIONAL GROUPS		
	Test For Phenol		
9	**Neutral $FeCl_3$ test:** Take 1 ml of neutral ferric chloride solution is taken in a dry clean test tube. Add 2 or 3 drops (or a pinch of solid) oforganic compound to it. If no colouration occurs add 3 or 4 drops of alcohol.	(i) Violet colouration is seen (ii) violet – blue colouration is seen (iii) green colouration is seen	(i) Presence of phenol. (ii) Presence of α–naphthol (iii) Presence of β– naphthol
	TEST FOR CARBOXYLIC ACIDS		
10	**Esterification reaction:** Take 1 ml (or a pinch of solid) of an organic compoundin a clean test tube. Add 1 ml of ethyl alcohol and 4 to 5 drops of conc. sulphuric acid to it. Heat the reaction mixture strongly for about 5 minutes. Then pour the mixture into a beaker containing dil. Sodium carbonate solution and note the smell.	A Pleasant fruity odour is noted.	Presence of carboxylic group.
	Test for aldehydes.		
11	**Tollen's reagent test:** Take 2 ml of Tollen's reagent in a clean dry test tube. Add 3-4 drops of an organic compound (or 0.2 g of solid) to it, and warm the mixture on a water bath for about 5 minutes.	Shining silver mirror is formed.	Presence of an aldehyde
12	**Fehling's test:** Take 1 ml each of Fehling's solution A and B are taken in a test tube. Add 4-5 drops of an organic compound (or 0.2g of solid) to it, and warm the mixture on a water bath for about 5 minutes.	Red precipitate is formed.	Presence of an aldehyde
	Test for ketones		
13	**Legal's test:** A small amount of the substance is taken in a test tube. 1 ml sodium nitro prusside solution is added. Then sodium hydroxide solution is added dropwise.	Red colouration.	Presence of a ketone.
	Test for an amine.		

14	**Dye test:** Take A small amount of an organic substance in a clean test tube, add 2 ml of HCl to dissolve it. Add few crystals of $NaNO_2$, and cool the mixture in ice bath. Then add 2 ml of ice cold solution of β-naphtholin NaOH.	Scarlet red dye is obtained.	Presence of an aromatic primary amine
	Test for diamide		
15	**Biuret test:** Take A small amount of an organic compound in a test tube. Heat strongly and then allow to cool. Dissolve the residue with 2 ml of water. To this solution Add 1 ml of dilute copper sulphate solution and few drops of 10% NaOH solution drop by drop.	Violet colour is appeared.	presence of a diamide
	Test for carbohydrates		
16	**Molisch's test:** Take A small amount of an organic compound in a test tube. It is dissolved in 2 ml of water. Add 3-4 drops of alpha naphthol to it.Then add conc H_2SO_4 through the sides of test tube carefully.	Violet or purple ring is formed at the junction of the two liquids.	Presence of carbohydrate
17	**Osazone test:** Take A small amount of an organic compound in a test tube. Add 1 ml of phenyl hydrazine solution and heat the mixture for about 5 minutes on a boiling water bath.	Yellow crystals are obtained	Presence of carbohydrate

LABORATORY MANUAL

PHYSICAL ANALYSIS - CONDUCTOMETRY

Aim: To determine the strengths of strong (HCl) and weak acid (AcOH) in a given mixture conductometrically.

Apparatus: Conductivity Bridge, Conductivity cell, beaker, Pipette, Micro burette, Glass rod

Chemicals required: 0.1M HCl, 0.1M CH_3COOH, 0.5M NaOH

Principle: Conductometric titration is a type of titration in which the electrolytic/ionic conductivity of the solution continuously monitored as one reactant is added. The principle of conductometric titration is based on the fact that during the titration, one of the ions is replaced by the other and invariably these two ions differ in the ionic conductivity with the result that conductivity of the solution varies during the course of titration. The main advantages to the conductometric titration are its applicability to very dilute, homogeneous suspension, coloured solutions and to system that involve relative incomplete reactions, which cannot be used with normal indicators.

The conductivity of the solution is inversely proportional to the size of the ions .if the size of the ions is increasing then the conductivity of the solution will decrease because the mobility of the ions will decrease by increasing the size of the ions. By increasing the temperature, the mobility of the ions in the solution will increase. So temperature has a direct effect on conductance of solution.

Strong base (NaOH) neutralizes the strong acid (HCl) first rather than weak acid (AcOH) indicated by decrease in conductance. This is due to comman ion effect. Due to this, dissociation of weak electrolyte is suppressed. Hence, AcOH remains un-neutralized till the complete neutralization of strong electrolyte (HCl). Then start the neutralization of AcOH indicated by increase in conductance values. When the acid mixture is completely neutralized, further addition of base will results the increase in conductance.

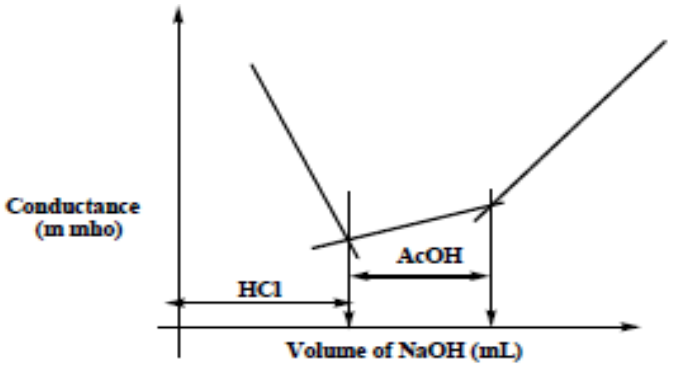
Procedure: Take 25 mL of 0.1M HCl and 25 mL 0.1M AcOH in a clean 100 mL beaker. Connect the conductivity cell to the conductivity meter. Once the conductivity meter is standardized, add 1mL of 0.5M NaOH from the burette to solution (acid mixture) containing beaker. Stir the solution carefully and note down the corresponding conductance value. Continue the addition of NaOH solution from the burette and record the conductance after every addition and tabulate the data.

Tabular form:

S. No	Volume of NaOH (mL)	Conductance (m mho)
1	0	
2	1	
3	2	
4	3	
5	4	
6	5	

Model graph:

Plot a graph between conductance values against the volume of the NaOH added. Three straight lines are obtained. The intersection of the first two lines gives the end point of strong acid and the intersection of the second and third lines gives the end point of the weak acid.



INSTRUMENTATIONS

LABORATORY MANUAL

SONICATOR

Sonication is the process of using energy to move particles around in a solution given. Typically, we do it for the purpose of cleaning or separating different substances. In the science laboratory, sonication is used to disrupt cellular membranes and as a result, release the contents of the cell. Sonication can also be used to fragment DNA, and preventing it from interfering with further sample preparation. Moreover, its other biological uses include the production of nanoparticles, liposomes, extraction of anthocyanins and antioxidants.

A sonicator is a powerful piece of lab equipment with an ultrasonic electric generator which creates a signal to power a transducer. This transducer converts the electric signal using piezoelectric crystals i.e. crystals that respond directly to electricity by creating a mechanical vibration. The sonicator preserves and also amplifies the vibration until it passes to the probe. The sonicator operator can easily control amplitude based on properties of solution. A small probe tip produces a much intense reaction than a large probe tip. On the other hand, a large tip reaches more of the solution.

This process uses ultrasonic sound waves. During the sonication process cycles of pressure form, thousands of microscopic vacuum bubbles in the solution. These bubbles collapse into the solution in the process of cavitation. This causes powerful waves of vibration which release an enormous energy force in the cavitation field. This disrupts molecular interactions such as interactions between molecules of water. Hence it separates clumps of particles and facilitates the mixing. For example, in dissolved gas vibrations, bubbles due to the gas come together and more easily leave the solution. The energy from the sound waves creates friction in the solution, which as a result creates heat. To stop a sample from heating up and degrading, keep it on the ice before, during as well as after the sonication. If the cells and proteins are too fragile to withstand sonication, a gentler alternative is enzyme digestion or grinding with sand.

MUFFLE FURNACE

Muffle furnace refers to a type of jacketed enclosure that is used to heat a material to significantly high temperatures while keeping it contained and fully isolated from external contaminants, chemicals or substances. Muffle furnaces are usually lined with stainless steel, making them largely corrosion resistant. Muffle Furnace is box type heat treatment equipment used to change physical properties of samples at very high temperature; for example 1100 °C, 1200 °C, 1300 °C, 1600 °C and 1700 °C. These laboratory furnaces are widely used in scientific experiments in physics/chemistry laboratories, steel and paint industries, biotech companies and small industrial production etc. Their major applications include general laboratory testing, annealing, ash determination, coal analysis, leaves carbonization and lime calcination etc.

HOT AIR OVEN

Hot air ovens use extremely high temperatures over several hours to destroy microorganisms and bacterial spores. The ovens use conduction to sterilize items by heating the outside surfaces of the item, which then absorbs the heat and moves it towards the center of the item. Hot air oven is widely used in the medical industry to sterilize the equipment and other materials that are used in a laboratory. It is used for delivering the heat treatment to the product. They do not require water and there is not much pressure build up within the oven, unlike an autoclave, making them safer to work with. This also makes them more suitable to be used in a laboratory environment. They are much smaller than autoclaves but can still be as effective. Items that are sterilized in a hot air oven include: Glassware (like petri dishes, flasks, pipettes, and test tubes), powders (like starch, zinc oxide, and sulfadiazine), materials that contain oils and metal equipment (like scalpels, scissors, and blades). Place the articles at sufficient distances so as to allow free circulation of air in between them and to ensure uninterrupted airflow. Shut the door and switch on the hot air oven.

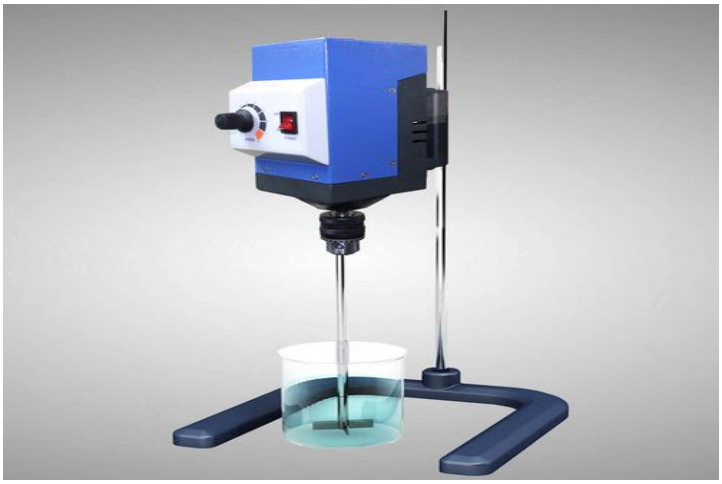
MICROWAVE OVEN

Microwave ovens can be used in laboratories for the rapid heating of material – either to dry them completely or to subject a workpiece to sudden thermal stress or electric field stress. Microwaves used for general processing may be used for heating laboratory samples, preparing solutions, drying, and heating samples or products. Determinations of the moisture content levels in soil or leaf tissue samples, for example, can be made within tens of seconds rather than hours. It is often assumed that placing a load within a microwave oven will result in it being heated evenly as well as quickly, but this is not always the case. The size and shape of a sample as well as its physical properties determine the power absorption. The range the microwave can be used for industrial, research, quality control processing is almost unlimited. The discussion on the use of microwave units specially designed for synthesis use, which are often quite expensive, becomes rather heated at times.

The water molecule is the target for microwave ovens; like any other molecule with a dipole, it absorbs microwave radiation. Microwave radiation is converted into heat with high efficiency, so that "superheating" becomes possible at ambient pressure. Enormous accelerations in reaction time can be achieved, if superheating is performed in closed vessels under high pressure; a reaction that takes several hours under conventional conditions can be completed over the course of minutes.

MECHANICAL STIRRER

Mechanical stirrer it's laboratory equipment consisting of an electric motor that drives the blades ended metal rod immersed in the mixed liquor. Laboratory mechanical stirrers are commonly used in industrial laboratories, research, cosmetic mixing and dispersing solid and liquid material, mixing at liquid-liquid system with different viscosities - from low to high. Mixers are available with different speed and advancement, equipped with a digital display or without display- depending on the requirements and needs of the client.



MAGNETIC STIRRER

A magnetic stirrer is a device widely used in laboratories and consists of a rotating magnet or a stationary electromagnet that creates a rotating magnetic field. This device is used to make a stir bar, immerse in a liquid, quickly spin, or stirring or mixing a solution. Most laboratory stirrers provide a speed range, such as 12-1800 rpm or 40-6000 rpm, but there are some single speed options. Place metal objects of any kind in a microwave oven. This includes aluminium foil and plastic coated magnetic stirrer bars. A magnetic stirrer or magnetic mixer is a laboratory device that employs a rotating magnetic field to cause a stir bar immersed in a liquid to spin very quickly and thus stirring the liquid. The rotating field created by a rotating magnet placed beneath the vessel with the liquid.

UV SPECTROPHOTOMETER

Ultraviolet-visible (UV-Vis) spectrophotometry is a technique used to measure light absorbance across the ultraviolet and visible ranges of the electromagnetic spectrum. When incident light strikes matter it can either be absorbed, reflected, or transmitted. The absorbance of radiation in the UV-Vis range causes atomic excitation, which refers to the transition of molecules from a low-energy ground state to an excited state.

Before an atom can change excitation states, it must absorb sufficient levels of radiation for electrons to move into higher molecular orbits. Shorter band gaps typically correlate to absorption of shorter wavelengths of light. The energy required for molecules to undergo these transitions, therefore, are electrochemically-specific. A UV-Vis spectrophotometer can use this principle to quantify the analytes in a sample based on their absorption characteristics.

The instrument used in ultraviolet–visible spectroscopy is called a UV/Vis spectrophotometer. It measures the intensity of light after passing through a sample (I), and compares it to the intensity of light before it passes through the sample (I_o). The ratio I/I_o is called the transmittance, and is usually expressed as a percentage (% T). The absorbance, A, is based on the transmittance. The UV–visible spectrophotometer can also be configured to measure reflectance.

The basic parts of a spectrophotometer are a light source, a holder for the sample, a diffraction grating in a monochromator or a prism to separate the different wavelengths of light, and a detector. The radiation source is often a Tungsten filament (300–2500 nm), a deuterium arc lamp, which is continuous over the ultraviolet region (190–400 nm), Xenon arc lamp, which is continuous from 160 to 2,000 nm; or more recently, light emitting diodes (LED) for the visible wavelengths. The detector is typically a photomultiplier tube, a photodiode, a photodiode array or a charge-coupled device (CCD). Single photodiode detectors and photomultiplier tubes are used with scanning monochromators, which filter the light so that only light of a single wavelength reaches the detector at one time. The scanning monochromator moves the diffraction grating to "step-through" each wavelength so that its intensity may be measured as a function of wavelength. Fixed monochromators are used with CCDs and photodiode arrays. As both of these devices consist of many detectors grouped into one or two dimensional arrays, they are able to collect light of different wavelengths on different pixels or groups of pixels simultaneously. Samples for UV/Vis spectrophotometry are most often liquids, although the absorbance of gases and even of solids can also be measured. Samples are typically placed in a transparent cell, known as a cuvette. Cuvettes are typically rectangular in shape, commonly with an internal width of 1 cm.

COLORIMETER

A colorimeter is a device used in colorimetry that measures the absorbance of particular wavelengths of light by a specific solution. It is commonly used to determine the concentration of a known solute in a given solution by the application of the Beer–Lambert law, which states that the concentration of a solute is proportional to the absorbance.

The essential parts of a colorimeter are: a light source (often an ordinary low-voltage filament lamp); an adjustable aperture; a set of colored filters; a cuvette to hold the working solution; a detector (usually a photoresistor) to measure the transmitted light; a meter to display the output from the detector. In addition, there may be: a voltage regulator, to protect the instrument from fluctuations in mains voltage; a second light path, cuvette and detector. This enables comparison between the working solution and a "blank", consisting of pure solvent, to improve accuracy. There are many commercialized colorimeters as well as open source versions with construction documentation for education and for research. Changeable optics filters are used in the colorimeter to select the wavelength which the solute absorbs the most, in order to maximize accuracy. The usual wavelength range is from 400 to 700 nm. If it is necessary to operate in the ultraviolet range then some modifications to the colorimeter are needed. In modern colorimeters the filament lamp and filters may be replaced by several (light-emitting diode) of different colors. In a manual colorimeter the cuvettes are inserted and removed by hand. An automated colorimeter (as used in an Auto Analyzer) is fitted with a flow cell through which solution flows continuously. The output from a colorimeter may be displayed by an analogue or digital meter and may be shown as transmittance (a linear scale from 0-100 %) or as absorbance (a logarithmic scale from zero to infinity). The useful range of the absorbance scale is from 0-2 but it is desirable to keep within the range 0-1 because, above 1, the results become unreliable due to scattering of light. In addition, the output may be sent to a chart recorder, data logger, or computer.

DEEP FREEZER

Deep freezers are the testing equipment that are used to preserve and store food products, chemicals, etc. for a long period of time. Deep Freezers are used for industrial purposes as well as for household purposes. These devices are available in different sizes and shapes sometimes it is designed with compact designs and sometimes with regular designs. The specifications and functions of the instruments vary as per the requirements of the test application.

Deep freezer is highly sophisticated testing machine which designed and fitted with a cooling compressors and CFC free refrigerants. These freezers are installed to create highly effective cooling consistently inside the cabinet. The air cooling compressors of the freezer are designed with aerodynamic fans and washable condense filters which keep the internal environment free from dirt and dust. The instrument helps to look into the mechanical behavior and characteristics of the rubber and polymer and other medical or industrial products at low temperatures. A deep freezer focuses on maintaining low temperatures that help keep the products at extremely low temperatures to not only extent shelf life, but to also make sure the product does not loss quality. In contrast to a regular freezer, a deep freezer can reach temperatures of –50C to -60 Degrees Celsius within an hour to a few minutes. This helps to prepare ice to carry out various organic syntheses in lab.

FOUR DIGIT BALANCE

The 4 decimal weighing scale, normally known as Analytical Scale, or Analytical balance. The balance indicates the measured result to 4 decimal places. All our 4 decimal weighing scale has functions of units conversation, percent and counting parts. This balance used to take accurate weighing of compounds for various analyses, such as titrimetric, gravimetric analysis etc.

HOMOGENIZER

Homogenization or homogenisation is any of several processes used to make a mixture of two mutually non-soluble liquids the same throughout. This is achieved by turning one of the liquids into a state consisting of extremely small particles distributed uniformly throughout the other liquid. A homogenizer is a piece of laboratory or industrial equipment used for the homogenization of various types of material, such as tissue, plant, food, soil, and many others. Many different models have been developed using various physical technologies for disruption. The mortar and pestle, already used for thousands of years, is a standard tool even in modern laboratories.

CONDUCTIVITY METER

An electrical conductivity meter (EC meter) measures the electrical conductivity in a solution. A conductance of a solution can be measured by applying an alternating electrical current to the two electrodes present in the probe. While this electrical current is applied to the solution, the cations (+ charge) move to the negative electrode and the anions (- charge) move to the positive electrode. This movement of the ions leads to the solution to be conductive. Conductivity meter allows us to measure the level of conductivity in solutions. Conductivity is an ability of materials (solutions, metals or gases) to pass an electric current. While all materials possess the ability to pass electric currents, the degree of such ability can vary. Substances with conductive aqueous solutions are referred to as electrolytes. These electrolytes are able to break down into ions when dissolved in water, thus creating free ions in solution. Acids, bases, and salts are examples of electrolytes. Substances with non-conductive aqueous solutions are referred to as nonelectrolytes. These substances are often composed of covalent bonds, and examples include carbon-containing compounds. Solutions with high concentration of ions will exhibit high conductivity. Solutions with low concentration of ions will result in small conductivity reading. A few factors that affect conductivity measurement are temperature, concentration of ions, and the nature of ions present in solution.

PH METER

PH meter, electric device used to measure hydrogen-ion activity (acidity or alkalinity) in solution. Fundamentally, a pH meter consists of a voltmeter attached to a pH-responsive electrode and a reference (unvarying) electrode. The pH-responsive electrode is usually glass, and the reference is usually a silver–silver chloride electrode, although a mercury–mercurous chloride (calomel) electrode is sometimes used. When the two electrodes are immersed in a solution, they act as a battery. The glass electrode develops an electric potential (charge) that is directly related to the hydrogen-ion activity in the solution (59.2 millivolts per pH unit at 25 °C [77 °F]), and the voltmeter measures the potential difference between the glass and reference electrodes.

LABORATORY GLASSWARES

BOTTLE, WEIGHING

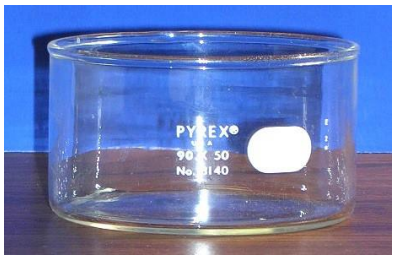
BURRET

CONDENSER

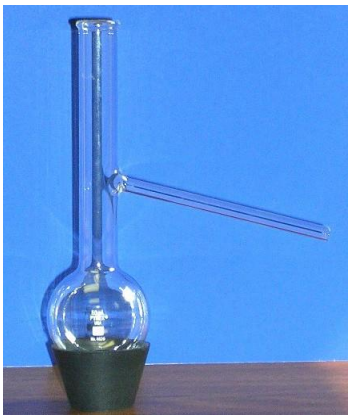
CRUCIBLE HOLDER, WALTER

CRUCIBLE, PORCELAIN, WITH LID

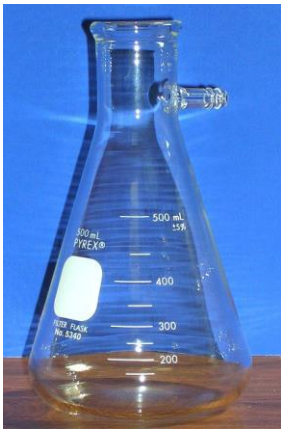
DISH, EVAPORATING



DISH, RECRYSTALLIZING

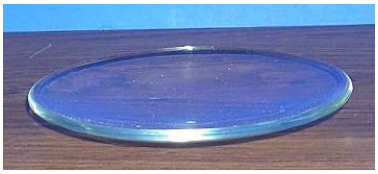


FLASK, DISTILLING

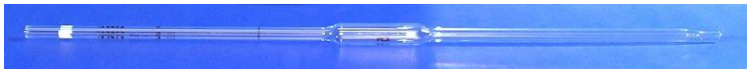


FLASK, FILTER

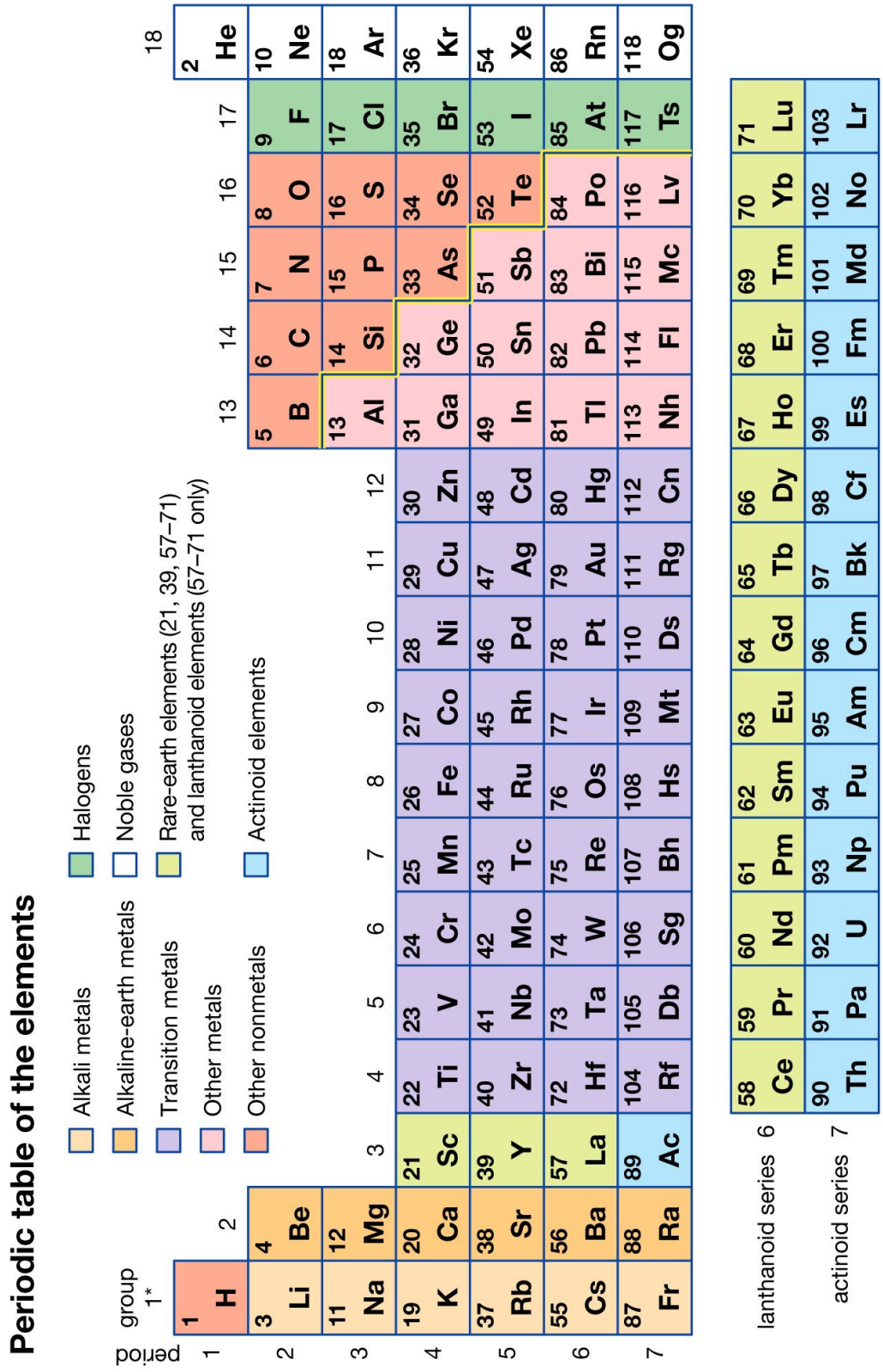
FLASK, VOLUMETRIC

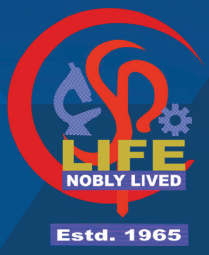


GLASS, WATCH



PIPET


Periodic table of the elements
Alkali metals
Alkaline-earth metals
Transition metals
Other metals
Other nonmetals
Halogens
Noble gases
Rare-earth elements (21, 39, 57–71) and lanthanoid elements (57–71 only)
Actinoid elements
group
period
lanthanoid series
actinoid series



ST. PAUL'S COLLEGE, KALAMASSERY

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