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General chemistry | Part I | 1st year

2019

Practical general chemistry



Subject

Laboratory apparatus

Laboratory glassware

General instructions

Preparation of solution

Experiment (1)a- preparation of Na_2CO_3

Experiment (2)b- preparation of HCL

Experiment(3) Determination of melting point

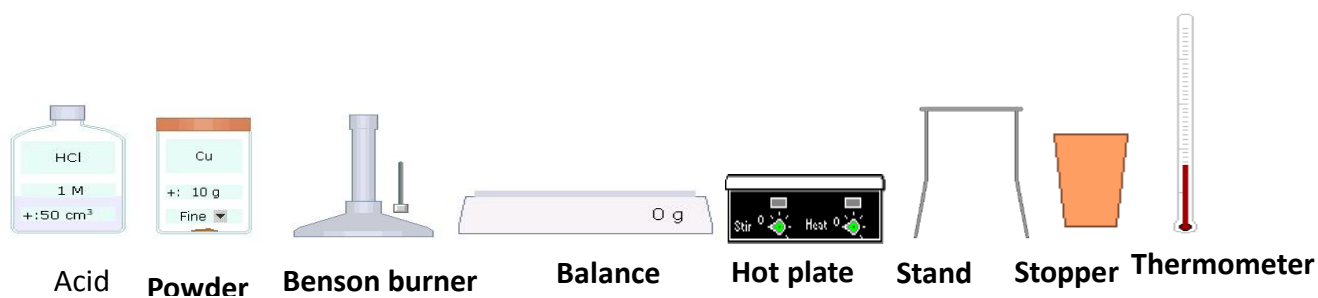
Experiment(4) Determination of boiling point

Experiment (5) Crystallization

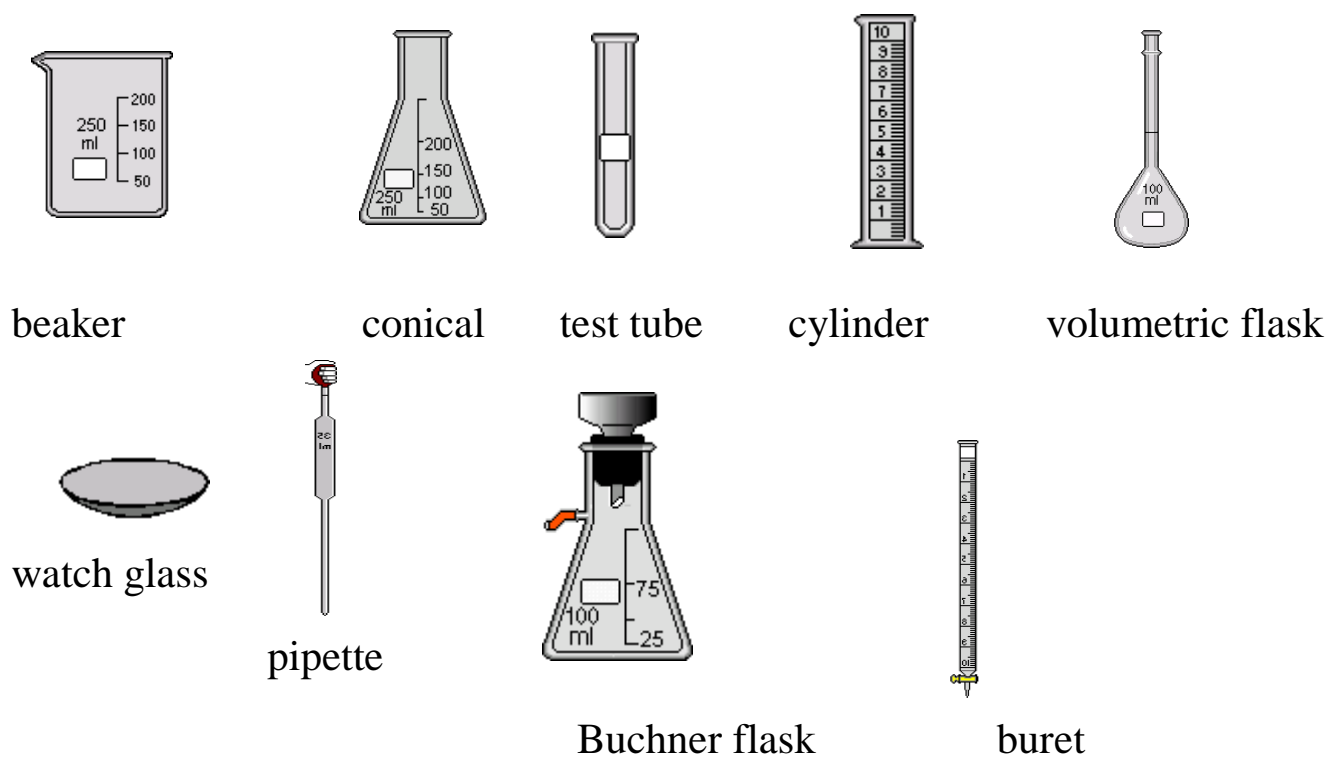
Experiment (6) Qualitative characterization of functional groups



Laboratory apparatus



Laboratory glassware



General instructions

- 1- First of all , the student should read the instructions carefully , paying attention to all details .
- 2- Attention should be paid to the relative quantities of reagent used , this depends upon the amount of test substance present .
- 3-The results and conclusion should be recorded as soon as the experiment has been done. These include all observations .

Safety laboratory rules

- 1- Drinking , eating and smoking not allowed in the laboratory . Use a laboratory coat to protect your clothes.
- 2- Use a clean apparatus to obtain good result .
- 3- Do not replace any solution in reagent bottles . Take only the minimum amount of reagent needed .
- 4- Replace the stoppers after the solution has been taken , take care not to mix the stoppers , and return the reagent bottle to its original place .
- 5- Never point a test tube of boiling liquid at your face or your neighbor , It may bump .
- 6- While using inflammable solvent like ether , benzene or alcohol , put off the flame or keep it away .
- 7- Never pipette the corrosive liquids like caustic solutions or concentrated acids or toxic and poisoning materials . Use a measuring cylinder or a buret .
- 8- If a chemical is discarded on your hand or clothes wash immediately with copious amount of water .

Abbreviations:

Precipitate	ppt.
Dilute	dil.
Concentrated	Conc.
Molar	M
Normal	N
Milliliter	ml
Equivalent weight	Eq.Wt
Molecular weight	M.Wt
Gram	g
Boiling point	b.p
Melting point	m.p



Experiment (1) :

Crystallization

Theory :

Crystallization is the most important method for the purification of solid organic compounds . The desired product is isolated from its impurities by difference in solubility .

Crystallization involves dissolving the material to be purified in the appropriate hot solvent . Insoluble impurities and colored impurities can be removed from hot solvent through the use of activated charcoal and filtration . As the solvent cools , the substance crystallizes , while the soluble impurities remain in the cold solvent .

Choosing the solvent :

The best Crystallization solvent must :

- a- Not react with the solute (inert) .
- b-Dissolve the solute when the solution is hot but not when the solution is cold .
- c-Very volatile (so it can be removed from the crystals) .
- d-Nonflammable , nontoxic and inexpensive .

materials :

Conical flask (125-250 ml), Funnel , Fluted filter paper , Filtering flask , Buchner funnel , Buchner flask Filter paper to fit Buchner funnel , Impure benzoic acid , Activated charcoal

Procedure : 1- Weigh out 2 g of impure benzoic acid , transfer it into a conical flask , then add boiling chips (to prevent bumping) .
2- Add 50 ml of solvent (water) to the sample .
3- Place on a hot plate , heat with stirring to dissolve the sample .
4- Cool the solution slightly , add 1 g of charcoal , then reheat again .
5- Filter the solution while it is hot using filtration apparatus (flask containing a few milliliters of solvent , funnel and fluted filter paper) . The solvent in this flask is brought to boil on a hot plate a long with the solution to be filtered (this is done in order to warm the filtration apparatus to prevent crystallization of the solute on the filter paper).



- 6- Cool the hot filtrate to room temperature . (do not disturb the solution).
- 7- Collect the crystals of pure product by vacuum filtration on Buchner funnel , wash it with a little of cold water , then dry .
- 8- Weight the product and calculate the % of the yield .

$$\% \text{ yield} = \frac{\text{The wight after purification}}{\text{The wight before purification}} \times 100$$

Experiment(2)

Determiration of melting point (M.P)

Theory:

Melting point is the temperature at which a substance change from a solid to liquid state. it is a physical property and can be used to help identify the substance in practice. a solid usually melt over a range of temperature rather than at one specific for this reason. it is more useful to speak of a melting point range .if the compound melts over a very narrow range .it can usually be assumed. That the compound is relatively pure besides melting over a wide range impure solid also melt at a temperature lower than that for the pure compound.

Materials:

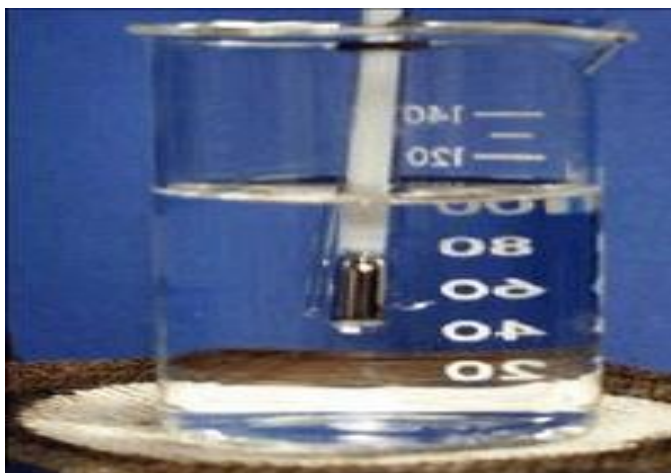
Hot plate, beaker, thermometer, capillary tube, sample (solid compound) , liquid paraffin, rubber ring, stand with clamp.

Procedure:

- 1_ Fill the capillary tube with the substance .
- 2_ Bind the capillary tube to thermometer using rubber ring.
- 3_ clamp the thermometer to a stand .immerse the thermometer in a small beaker contain liquid paraffin.
- 4_ Heat the beaker slowly on hot plate.
- 5_ recorde the temperature at which the substance starts melt(T1).



6_record the temperature at which the substance has completely melt (converted to liquid) (T₂).



DETERMINATION OF BOILING POINT

Theory: The boiling point of a compound is the temperature at which it changes from a liquid to a gas. This is a physical property often used to identify substances or to check the purity of the compound.

It is difficult, though, to find a boiling point. Usually, chemists can only obtain a boiling range of a 2 - 3°C accuracy.

Materials:

Hot plate, beaker, sodium fusion tube, thermometer, capillary tube, sample(liquid compound), liquid paraffin, rubber ring, stand with clamp.

Procedure:

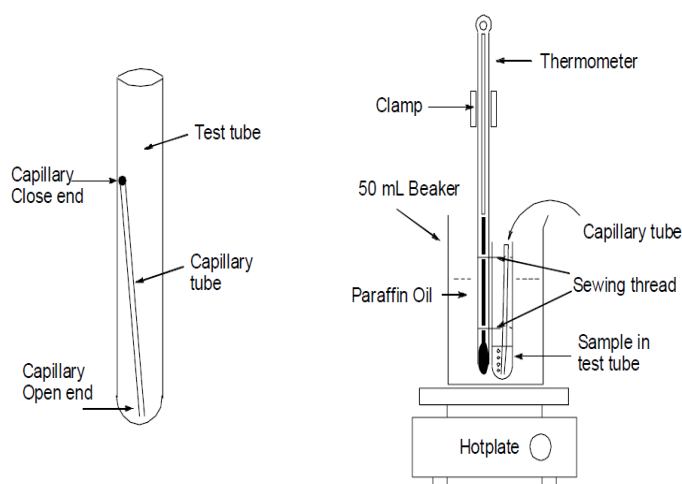
1. Place a few milliliters of a known liquid organic compound in a small sodium fusion tube.
2. Into the test tube, place the capillary tube with closed end upward.
3. Clamp the test tube to a ring stand and immerse a thermometer in the test tube. Sure to clamp the thermometer to the ring stand as well.
4. Fill a 250 mL beaker 3/4 full with liquid paraffin and place on the hot plate. The test tube and thermometer combination into the beaker of paraffin



liquid. test tube is immersed half way in the paraffin liquid .

5. Begin to heat the hot plate slowly. As the liquid approaches its boiling point, a few bubbles will be observed flowing out of the end of the capillary tube. When steady steam of bubbles are observed, when the liquid begins to flow into the capillary tube.

6. record the temperature of the liquid as its boiling point temperature.



Apparatus set-up for boiling-point determination

Experiment (4) :

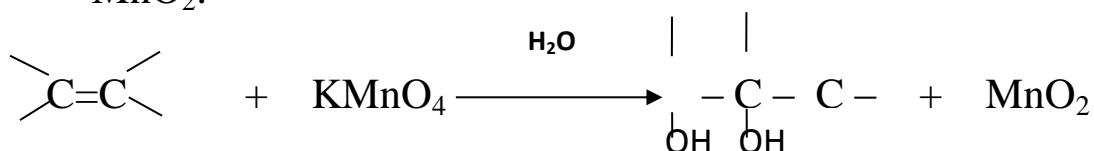
Qualitative Characterization of Functional groups

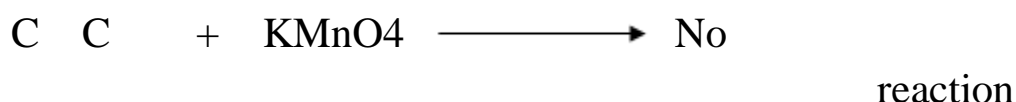
Baeyer , Lucas and Tollen's Test

A: Baeyer Test :

Baeyer test is a qualitative test for the presence of unsaturation , such as Double bonds($C=C$) .

Bayer's reagent is a cold aqueous solution of potassium permanganate , which is powerful oxidant react with the double bonds ($C=C$) or the triple bonds ($C\equiv C$) in an organic material causes the disappearance of the $KMnO_4$'s purple color and the appearance of a brown suspension of MnO_2 .





Materials :

Two test tubes , 2-Cyclohexane , 3-Cyclohexene ,Potassium permanganate solution .

Procedure :

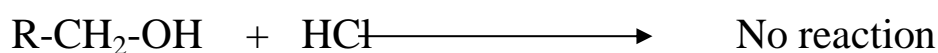
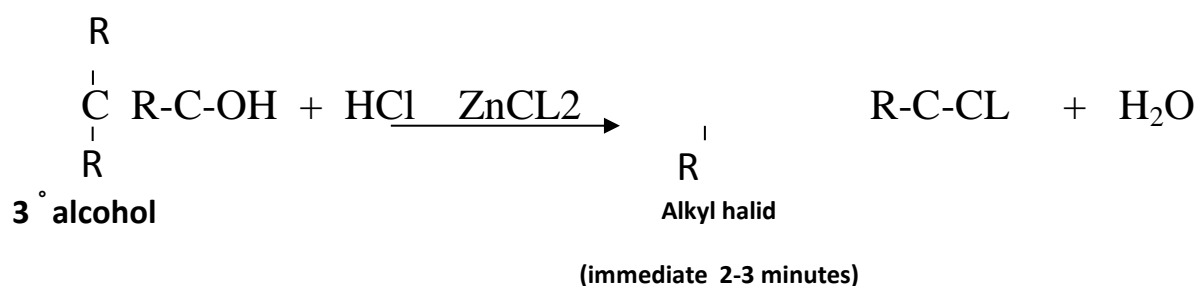
- 1- Set two clean test tubes .
- 2- Place 3 drops of cyclohexane into the first tube .
- 3- Place 3 drops of cyclohexene into the second tube .
- 4- Add 6 drops of dilute KMnO_4 solution to each tube , stir and watch .
- 5- Record your observations .

B : Lucas Test :

Lucas test is used distinguish between primary , secondary and tertiary alcohols to.

The lucas reagent is a solution of zinc chloride (ZnCl_2) in concentrated HCL .

The test depends on a difference in the rate of reaction of these alcohols with Lucas reagent (tertiary>secondary> primary).



1° alcohol



Positive test :

Appearance of a cloudy second layer or emulsion denotes formation of an insoluble alkyl halide .

Materials :

Three test tubes , Lucas reagent , 1- butanol , 2- butanol , 3- t – Butyl alcohol

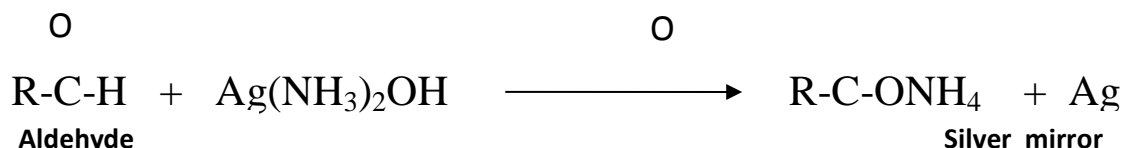
Procedure :

- 1- Place 1ml of Lucas reagent in each of three test tubes .
- 2- Add 4 drops 1- butanol to one test tube , shake and note whether the mixture gets cloudy and how long it takes .
- 3- To the second test tube add 4 drops of 2- butanol , shake and note how long it takes the tube to get cloudy (if cloudiness does not appear place the tube in a hot water bath and observe) .
- 4- To the third test tube add 4 drops of t- butyl alcohol , shake and note how long it takes the tube to get cloudy .
- 5- Record your observations.

C: Tollens' Test :

This test identifies aldehydes , and is mainly used to distinguish between aldehydes and ketons .

Tollen's reagent (Ag^+ in aqueous NH_3) oxidize aldehydes to carboxylic acids . Metallic silver is produced as a shiny mirror on the walls of the reaction flask or test tube ,where as ketons are generally inert toward the oxidation .



Materials :

Two test tubes , 5% Silver nitrate solution , 10% Sodium hydroxide solution , 1 M Ammonia solution , Formaldehyde , Acetone .

Procedure :

- 1-Place 2 ml of 5% Silver nitrate solution (AgNO_3) in each of two test tubes.
- 2-Add 1 drop of 10% Sodium hydroxide solution (NaOH) to each tube . A brown precipitate of Ag_2O will appear .
- 3- To each test tube add 1 M Ammonia solution (NH_4OH) , drop by drop , with shaking until the precipitate just dissolves .
- 4-To the first tube , add 3 drops of formaldehyde solution , stir gently .
- 5-To the second tube , add 3 drops of acetone , stir gently .
- 6- Record your observations .

Preparation of solutions

Standard solution : is a reagent of known concentration that is used to carry out the titration . Therefor , we need to consider the desirable properties for such solutions , how they are prepared , and how their concentration are expressed .

Desirable properties of standard solution

The ideal standard solution for titrimetric method will :

- 1- Be sufficiently stable so that it is only necessary to determine its concentration .
- 2- React rapidly with the analyte .
- 3- React completely with the analyte.
- 4- Undergo a selective reaction with the analyte that can be described by a simple balanced equation .

Methods of expressing concentrations

Concentration : refers to exact amount of solute presence in a given volume of solvent .



1- Solid compounds

a- Molarity (M) :- The number of moles of solute per liter of solution .
A capital letter (M) is used to abbreviate units of mol/L.

$$M = \frac{\text{moles of solute}}{\text{liters of solution}}$$

$$M = \frac{\text{weight of solute (g)} \times 1000 \text{ (ml/L)}}{\text{molecular weight (g/mol)} \times \text{volume (ml)}}$$

a- Normality (N) : The number of equivalents of solute per liter of solution . A capital letter (N) is used to abbreviate units of equivalent / L .

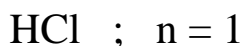
$$N = \frac{\text{weight of solute (g)} \times 1000 \text{ (ml/L)}}{\text{equivalent weight (g/eq)} \times \text{volume (ml)}}$$

$$\text{Equivalent weight} = \frac{\text{molecular weight}}{n}$$

n= represent in

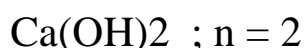
1- acids : number of (H⁺) ions.

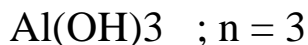
ex:



2- Bases : number of (OH⁻) ions .

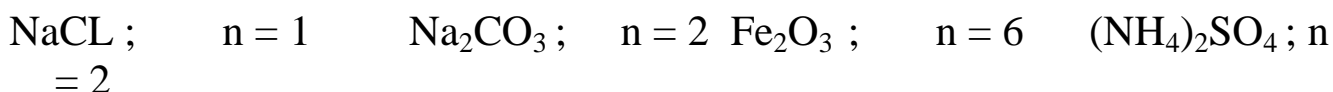
ex:





3- salts : number of atoms or groups which can be ionized oxidation state of ions

ex:



2- liquid compounds

Since acids are liquids so it will be prepared in a way different than that for solids . First check the stock solution and see the density (specific gravity) , percentage (%) and molecular weight then calculate the molarity (M) or normality (N) .

$$M = \frac{\text{Density} \times \%(\text{in decimal}) \times 1000}{M . wt}$$

$$N = \frac{\text{Density} \times \% (\text{in decimal}) \times 1000}{eq. wt}$$

Dilution law :

dilution To prepare dilute solution from concentrated one we use the law which defined as :**The number of moles (equivalents) of diluted both and concentrated solution must be equal .**

$$(M_1 V_1)_{\text{conc.}} = (M_2 V_2)_{\text{dil.}}$$

$$(N_1 V_1)_{\text{conc.}} = (N_2 V_2)_{\text{dil.}}$$

Experiment (5) :

a) Preparation of 0.1 N sodium carbonate (Na_2CO_3) .

To prepare 0.1 N of primary standard solution of Na_2CO_3 we should know the volume of solution , after that we calculate the weight required to prepare this solution .



$$\text{Wt (g)} = \text{Normality} \times \text{Eq.wt} \times \text{Volume} / 1000$$

In this lab we need 100 ml of (0.1 N) Na_2CO_3 , M.wt = 106 g/mol

$$\text{wt (g)} = 0.1 \times 106 \times 100 / 1000 = 0.53 \text{ g}$$

Materials and Equipments :

Balance , 100 ml volumetric flask , funnel , beaker , anhydrous Na_2CO_3 and distilled water .

Procedure :

- 1-Weight out accurately 0.53 g of anhydrous Na_2CO_3 .
- 2-Dissolve in small quantity of D.W and transfer quantitatively to 100 ml volumetric flask .
- 3- Complete to the mark and shake well .

Experiment(6);

Preparation Of 0.1 N hydrochloric acid (HCL) .

Since HCL liquid is it so will be prepared in this way :

$$\text{N} = \text{Density} \times \% \text{ (in decimal)} \times 1000 / \text{eq.wt}$$

Density 1.2 g / ml

% 36 %

M. wt . 36.5 g/mol

Eq.wt. 36.5 g/eq

$$\text{N} = 1.2 \times 0.36 \times 1000 / 36.5 = \dots\dots\dots \text{Eq / L conc . of HCL}$$

Then we calculate the volume of the conc. HCL required to prepare the dilute one .

$$(\text{N}_1 \text{ V}_1) \text{conc.HCL} = (\text{N}_2 \text{ V}_2) \text{dil.HCL}$$

Materials and Equipments :

100 ml volumetric flask , pipette , conc. HCL , beaker , D.W.

Procedure :

- 1-Calculate the concentration of the conc. HCL
- 2-Take accurately the volume (.....ml) of conc. HCL



3-Put in small quantity of D.W. and transfer quantitatively to 100 ml volumetric flask .

4-Complete to the mark with D.W. and shake well .

References:-

- Selected experiments in organic chemistry second edition . Leila Itanaineh Abdelnoun , Ahmed Qasem Hussein.

- Vogel"s

Textbook of practical organic chemistry Fifth edition . B-S-Furniss A-J Hannafordr.

- Fundamentals of analytical chemistry .F.James Holler Stanleyr. Crouch.

