2018

General Chemistry (Practical Lab)

PHAR101

Chemistry is the science that deals with the materials of the universe and the changes these materials undergo. Understanding most other fields of science requires an understanding of chemistry.

Tara Fuad Tahir
Ph.D Electroanalytical Chemistry
Faculty of Pharmacy
Tishk International University

Contents

Definition of a Laboratory

A laboratory can be a place of specialised research, clinical or diagnostic evaluation, teaching and/or learning. Laboratories are commonly used in many scientific disciplines across the University ranging from health sciences to biological and physical sciences.

Working safely in the Laboratory

Research and scientific laboratories often present a wide range of hazards to researchers and students. Having in place specific procedures for laboratories is crucial in ensuring the safety of staff and students who work in these areas.

This guideline outlines the requirements for working in a laboratory for University staff and postgraduate students.

Laboratory Safety Rules

- Food and drink (including drinking from water bottles) must not be consumed in laboratories.
- Unauthorised entry or experimentation in the laboratories is strictly forbidden.
- Staff and Postgraduates wishing to use the laboratory out of normal work hours must obtain their supervisor and the laboratory manager's permission.
- All researchers must be aware of the conditions required for the safe handling of substances and specimens being handled.
- Be aware of the safety facilities of the laboratory, i.e. location of safety showers, eyewash stations, fire extinguishers and emergency exits.
- Working spaces are to be kept clean. Broken glass sharps, and laboratory waste must be placed in the marked bins in the laboratory.
- No waste is to be left or placed in the sinks, and under no circumstance must waste be placed down the sink, unless authorised to do so.

- All spillages must be cleaned up immediately after they occur.
- Be aware of burning Bunsen burner by noting a hollow burning sound and/or the absence of a blue cone of unburnt gas.
- No samples are to be taken from, or brought into, the laboratory without the permission of your Supervisor or the Laboratory Manager.
- Pipetting by mouth is strictly prohibited.
- Defective equipment or broken glassware must be reported to the laboratory manager.
- Radioactive sources (e.g. laser, UV radioactive substance or arc lamp) must only be used following the direction and supervision of the supervisor or laboratory manager or radiation safety officer.
- Sitting on laboratory benches is prohibited. Never run in the laboratory or along corridors.
- Cover any open wounds e.g. cuts, dermatitis on hands.
- Always wash hands thoroughly before leaving the laboratory.

Personal Protective Equipment (PPE)

PPE is widely recognised as a means of protection for individuals working in an environment where all other methods of hazard control are in place and there is still a risk of injury. You must remember that PPE is the last barrier or line of defence between you and the hazardous material you are working with. The minimum PPE you are required to wear when working in University Laboratories are:

Enclosed Shoes (no bare feet, or sandals)

Lab Coat (must be individually issued, worn at all times when working in the lab, removed before leaving the lab and laundered regularly and when contamination is suspected). The

following additional PPE is provided and should be used where required / instructed / determined by a risk assessment:

- Safety Glasses / Goggles must be worn when working with hazardous chemical solutions where there is a risk of splash to your eyes or when instructed to do so by your Supervisor or the Lab Manager.
- Gloves
- Dust Mask / Respirator
- Face Shield must be worn when working with volatile hazardous chemical solutions (e.g. concentrated acids), dangerous substances that could strike/splash the face/eyes or there is a risk of solution explosion or instability causing a splash to the face/eyes and when there is a risk of dangerous objects striking the face/eyes, such as particles, glass or metal shards.

First Aid

- All accidents must be reported immediately to the supervisor/laboratory manager and recorded on the Injury, Illness and Incident Database.
- Eye injuries, whether caused by chemicals or mechanical injury or splash with a material, eye injuries are always serious. The treatment requires immediate and prolonged flushing with water (20 minutes minimum) at the eyewash station. Medical advice should be obtained for an eye injury.
- In the event of chemical or biological spills on the skin, the affected area must be washed with copious quantities of water.
- Sharps injuries – Notify supervisor/lab manager immediately. Wash the wound and encourage bleeding. Seek medical treatment.

- If you are feeling unwell or dizzy when participating in an experiment, stop immediately, sit down and notify supervisor/lab manager.

Fig.1 Typical students (female and male) at laboratory

Glasswares and Equipments Used at Lab

There are qualitative and quantitative glasswares that are used at laboratory and below are some of glasswares and equipments that are used at lab:

1. Beaker is used to transfer liquids and dissolving solid in liquid solution and their volume is not accurate

2. Watch glass is used to weigh solid chemical

3. Glass rod is used to mix solutions and dissolve solid in liquid

4. Cylinder is used to transfer liquids and their volume is not accurate

5. Funnel is used to transfer liquid and for filtration

6. Conical flask is used to transfer liquid and for titration. Their volume is not accurate

7. Washing bottle is used to clean laboratory glassware and other equipment. They are filled with appropriate cleaning liquids, and poured over the tool that needs to be cleaned

8. Reagent bottle is container made of glass or plastic or borosilicate with stopper and is intended to contain chemicals in liquid or powder form for laboratories

9. Test tube is used for qualitative analysis for small amount of solution

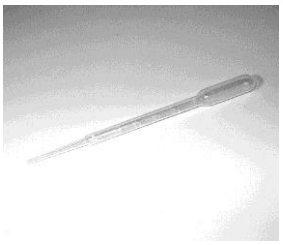
10. Centrifuge tube is used within centrifuge to separate solid from liquid

11. Tong is used for picking things up without touching them with hands or fingers

12. Test tube holder is used for holding a test tube in place

13. Centrifuge is used to separate solid from liquid or liquids that have different weights

14. Dropper is used to transfer small quantities of liquid



15. Litmus paper is used to distinguish between acids and bases

16. Filter paper is a quantitative paper used for filtering

17. Spatula is a stainless steel utensil, used for scraping, transferring, or applying powders and paste like chemicals.

18. Petri dish usually made of glass and is used for scientific experiments, especially in chemistry and biology laboratories

19. Bunsen burner is a single open gas flame equipment, which is used for heating, sterilization and combustion

20. Sensitive balance is an analytical balance that is so sensitive and can detect the mass of a single grain of a chemical substance

21. Rough balance is used for pre-weighing samples to determine the mass approximately

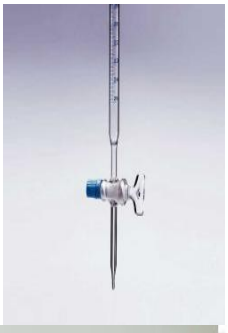
22. Water bath is made from a container filled with heated water to incubate samples in water at a constant temperature over a long period of time or to enable certain chemical reactions to occur at high temperature

23. Hot plate is a portable self-contained table top small appliance that features one, two or more electric heating elements, used to heat solution and substances

24. Thermometer is a device that measures temperature or a temperature gradient

25. Volumetric Flask is used for precise dilutions and preparation of standard solutions

26. Burette is a graduated glass tube with stopcock at one end, used in quantitative chemical analysis to measure the volume of a liquid and in titration reaction



27. Graduated pipette is used to transfer small volumes of liquid and it is not calibrated for any particular volume

28. Bulb pipette is used to transfer small and accurate volume of liquid because it is calibrated for any particular volume

29. Pipette filler is used to safely fill a pipette with solution

30. Stand is a piece of scientific equipment, to which a clamp can be attached to hold glassware

31. Clump is a device to hold an objective to stand

32. Distilled water apparatus is used to produce distilled water

Questions:

1- Classify the glasswares and equipments that are used for quantitative and qualitative analysis in the lab?
2- Write glasswares with accurate size measurement and others with inaccurate size measurement.
3- What is PPE?
4- If an acid liquid is poured on your hand during your work in the lab what are the first aid that you have to follow?

LAB -1- Measuring the density of solid compounds

Theory:
Density is a physical properties of any substance, can be expressed as mass per unit volume.

d= mass (weight) / volume
Unit of d = g/ml or g/cm^3
Kg/L or Kg/m^3

A material's density is defined as its mass per unit volume. In another way, density is the ratio between mass and volume or mass per unit volume. It is a measure of how much stuff an object has in a unit volume (cubic meter or cubic centimetre). Density is essentially a measurement of how tightly matter is crammed together. The principle of density was discovered by the Greek scientist Archimedes, but it is easy to calculate if you know the formula and understand its related units.

Finding the density of solid materials depends on their shape whether regular or irregular shapes have. For measuring density of the regular shape solid compounds, any shape has a special mathematical equation to find its volume (v) and then measuring its mass by weighing on the balance (m) and calculating the density by the above formula.

For example, take a brick of salt that weighs 433 g and have the dimensions as:

v= length x width x thickness

v= 10 cm x 10 cm x 2 cm = 200 cm^3

Thus, the density of the salt brick is d= 433 g/200 cm^3 = 2.165 g/cm^3

But about measuring the density of irregular shape solid compounds, its weight can be measured by weighing it on a balance (m), and its volume can be measured by recording the change in the volume (v) after putting the solid compound in a graduated cylinder containing a liquid (often water), then applying the above equation of density. The liquid in the cylinder must be not react and dissolve the solid compound.

One of the most common uses of density is in how different materials interact when mixed together. Wood floats in water because it has a lower density, while an anchor sinks because the metal has a higher density. Helium balloons float because the density of the helium is lower than the density of the air.

When your automotive service station tests various liquids, like transmission fluid, it will pour some of the fluid into a hydrometer. The hydrometer has several calibrated objects, some of which float in the liquid. By observing which of the objects float, the service station employees can determine the density of the liquid. In the case of the transmission fluid, this

test reveals whether service station employees need to replace it immediately, or whether the fluid still has some life in it.

The change in density can also be useful in analyzing some situations, such as whenever a chemical conversion is taking place and energy is being released. The charge in a storage battery, for example, is an acidic solution. As the battery discharges electricity, the acid combines with the lead in the battery to form a new chemical, which results in a decrease in the density of the solution. This density can be measured to determine the battery's level of remaining charge.

Density is a key concept in analyzing how materials interact in fluid mechanics, weather, geology, engineering and material sciences.

For more information watch the video below:

https://www.youtube.com/watch?v=GzdRGOuNIFg

Procedure:

1- Add approximately 40 mL of water (v_1) to a clean and dry 100-mL graduated cylinder. Record the exact volume of the water.
2- Place the cylinder and water on an analytical balance. Press the "Tare" or "Zero" button on the balance. The balance should read 0.000 g.
3- Add approximately 10 zinc pellets to the graduated cylinder. Record the new volume of the water plus zinc pellets (v_2) using the liquid level after addition of the zinc.
4- Weigh the cylinder, water, and zinc pellets on the balance. Record the mass of the zinc pellets (m).
5- Find the volume (v) of the solid 10 zinc pellets as $v = (v_2 - v_1)$.
6- For best results, repeat steps 1 - 4 twice more to obtain two additional density measurements.
7- Calculate the density of each trial by applying: $d = m / v$

Results and calculations:

Trial	Mass of Zinc (g)	Volume of Zinc (ml)	Density (g / ml)
1			
2			
3			

Name…… **Stage** First

Student Signature……..

Staff Signature……

Date

Questions and discussion:

1- How much does 1 gallon of water weigh in pounds? d H_2O = 1 g / cm^3
2- What are the factors affecting density of matter? Explain each one.
3- Are there differences in the values of densities between the three trials? If Yes explain why and if NO explain why?
4- If you have equivalent masses of aluminium (Al) and zinc (Zn) metal, which metal occupies a much smaller volume than the other? Why?

LAB -2- Measuring the density of liquid compounds

Theory:
Density is an intensive property of a substance that doesn't depend on the amount of substance present. The density of a substance is the ratio of its mass to its volume. At constant temperature and pressure, the density of a substance is constant.

d= mass (weight) / volume
Unit of d = g/ml or g/cm^3
Kg/L or Kg/m^3

To measure the density of a sample of a substance, it is necessary to measure its mass and volume. Mass is typically measured using an analytical balance, a precise instrument that relies on the force exerted by the sample due to gravity. The container to hold the sample (also used to measure volume) is weighed and tared (i.e. make it zero), so only the sample mass appears on the balance display when the sample is added to the container.

For liquids, this container is typically a volumetric flask, which has one marking that corresponds to a specific volume. The container is filled to the line with the liquid sample and weighed again after the empty flask has been tared. The measured density is the ratio of the measured mass to the volume indicated on the flask.

Gases always have much lower density than the condensed phases. Most materials have a lower density of the liquid than the solid but this isn't always true. Water has a higher density in the liquid state than the solid, so ice cubes float.

Density of a liquid depends on temperature and pressure. Temperature is related to the average kinetic energy of the atoms or molecules within the substance. The density of water decreases from 0.9970 g/mL to 0.9718 as it is heated. This makes sense because, as heat is added to the liquid water, there is greater kinetic energy of the molecules and there are also more vibrations of the water molecules. Together these mean that each H_2O unit in liquid water takes up more space as the temperature increases. Pure ethanol, CH_3CH_2OH, is another pure liquid. It is similar to water in that it is polar, with a permanent dipole moment, and forms hydrogen bonds with itself.

Changes in pressure have very little effect on the volume of a liquid. Liquids are relatively incompressible because any increase in pressure can only slightly reduce the distance between the closely packed molecules. If the pressure above a liquid is increased sufficiently, the liquid forms a solid. If the pressure above a liquid is decreased sufficiently, the liquid forms a gas.

For more information watch the video below:

https://www.youtube.com/watch?v=1dx-Tx5dPKk

Procedure:

1- Place a clean and dry 50-mL volumetric flask on an analytical balance.
2- Press the "Tare" or "Zero" button on the balance. The balance should read 0.000 g.
3- Use a burette funnel to add 45 mL of liquid ethanol to the volumetric flask.
4- Use a Pasteur pipette to add the final 5 mL of liquid, just until the bottom of the liquid's meniscus touches the marking on the flask.
5- Weigh the volumetric flask again and record the mass of the ethanol.
6- For best results, repeat steps 1 - 5 twice more to obtain two additional density measurements.

Results and calculations:

Trial	Mass of Ethanol (g)	Volume of Ethanol (ml)	Density (g/ml)
1			
2			
3			

Name…… Stage First

Student Signature……..

Staff Signature……

Date

Questions and discussion:

1- Are there differences in the values of densities between the three trials? If Yes explain why and if NO explain why?
2- Which one has more density pure water or pure ice? Why?
3- Which one has more density salt water or fresh water? Why?
4- How the temperature affects density?

LAB -3- Measuring the density to determine the concentration

Theory:

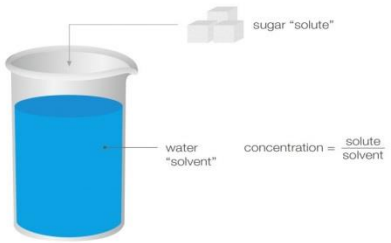
There is a relation between the concentration of a substance in a solution and the density of the solution. The concentration of the substance is referred to the amount of the substance in the solution. Concentration is expressed in different terms such as Molarity, Normality, ppm (part per million), g/L i.e. weight of substance in gram (g) that is dissolved in one liter (L) or as % w/w, % v/v and % w/v.

$$\% \text{ w/w} = \frac{\text{Weight (g) solute}}{\text{Weight (g) solution}} \times 100$$

$$\% \text{ w/v} = \frac{\text{Weight (g) solute}}{\text{Volume (ml) solution}} \times 100$$

$$\% \text{ v/v} = \frac{\text{Volume (ml) solute}}{\text{Volume (ml) solution}} \times 100$$

The density of liquids changes if ingredients change. For example: a soft drink containing sugar will have a higher density than a diet soft drink. So, sugar not only increases a liquid's density, it can also be inversely measured by knowing the liquid's density in a given liquid type. The concentration of sugar in the solution can be determined by measuring the density of the solution. Using this correlation provides useful information in the production of many beverages.

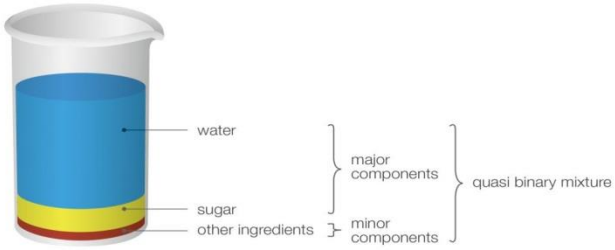


Two-component mixtures are called binary mixtures. The density of the mixture is a function of its composition. Thus, the density value of a binary mixture can be used to calculate its composition with the aid of concentration tables.

Typical two-component mixtures are e.g. alcohol-water solutions, sugar-water solutions, salt-water solutions, and acids or bases dissolved in water.

Concentration determination is also possible with so-called quasi-binary mixtures. These are mixtures containing two major components and some additional ingredients which are present in very small concentrations compared to the two main components. Due to their small impact on the bulk density these additional ingredients can be ignored.

Concentration determination is also possible if a mixture contains several components and only one of them varies while all other ingredients are constant.



Procedure:

1- Prepare different solutions of two-component binary mixtures containing (2, 4, 6, 8, 10, 12 and 14) % NaCl solutions by weighing out (2, 4, 6, 8, 10, 12 and 14) g of NaCl separately on watch glasses.
2- Transfer the weighed quantities of NaCl separately into beakers to dissolve them in 50 ml distilled water.
3- Transfer the different concentrations of solutions into 100 ml volumetric flasks and complete their volumes to 100 ml by distilled water.
4- Put stoppers on the volumetric flasks to mix them.
5- Find the density of each prepared binary mixture of NaCl solution via calculation of d= weight (g) / volume (ml) by weighing the prepared solutions of NaCl.
6- Plot a calibration curve between the density of solutions on y-axis and their concentrations % (w/v) on the x-axis on a graph paper.
7- Measure the density of unknown concentration solution as in Lab (2).
8- Find the concentration of the unknown solution from its density by plotting on the calibration curve.

Results and calculations:

Concentration of NaCl % w/v	Density NaCl solution (g/ml)
2	
4	
6	
8	
10	
12	
14	
Unknown	

Name…… Stage First

Student Signature……..

Staff Signature……

Date

Questions and discussion:

1- Indicate whether the calibration curve is linear or not? If it is linear explain why? And if not explain why?
2- If the density value of the unknown concentration solution is above the points of the calibration curve how you can find its concentration?
3- Suggest a procedure to find the concentration of two-component binary mixture ethanol-water via calibration curve method?
4- What is the condition of finding concentration of binary mixture from their density via calibration curve method?

C.O.S.H.H assessment form for use in Chemistry Laboratories

YOU MUST READ AND SIGN THIS FORM BEFORE STARTING ANY LABORATORY WORK. YOU CANNOT BEGIN THE EXPERIMENT UNTIL YOU HAVE HANDED IT TO THE LABORATORY SUPERVISOR. BY SIGNING BELOW YOU CERTIFY YOU HAVE READ THIS SHEET COMPLETELY.

Experiment name: Purification (Recrystallization) of NaCl salt sample

Subject: General Chemistry

Hazardous Chemicals

NaCl	Toxic, irritant	May cause skin irritation, causes eye irritation. Ingestion of large quantities can irritate the stomach with nausea and vomiting. Continued exposure may produce dehydration, internal organ congestion, and coma.
$BaCl_2$	Toxic, irritant	Hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation. The substance is toxic to kidneys, the nervous system, cardiovascular system and upper respiratory tract. The substance may be toxic to liver.
HCl	Corrosive, irritant	Corrosive and cause burns in contact with skin. Even contact with dilute solution can lead to serious eye damage.
Na_2CO_3	Toxic, irritant	Hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation (lung irritant). The substance may be toxic to upper respiratory tract, skin and eyes.

Risk control

When using sodium chloride, barium chloride and sodium carbonate. Wear gloves, googles and clothing to prevent skin and eye exposure.

Take care when using hydrochloric acid, causes severe burns! Wear gloves at all times. Hydrochloride acid burns, and to avoid the fume of hydrochloric acid, uses the fume hood.

Name…… Stage First

Student Signature……

Staff Signature……

Date

LAB -4- Purification (Recrystallization) of NaCl salt sample

Theory:

Recrystallization is a method used to purify solids. The crystallization method can be used to separate the solids from other solids. Recrystallization method is based on the differences of solubility between the purified substance and impurities in a particularly solvent. Purification with recrystallization method widely applied in industrial laboratories and aimed to improve the quality of a substance/material. In principle, the substance which was purified is dissolved in a solvent then it was heated and evaporated again. Impurities materials that cannot be dissolved can be separated from the solution by filtration. The impurities will be soluble in the solution.

Purification by recrystallization of solids based on the difference in solubility of the substance to be purified in certain solvents. The general procedure is often used in the recrystallization is:

1. Dissolving the impurity substances in certain solvent at near the melting point.
2. Filtering the particles of insoluble materials in hot solution condition.
3. Cooling the hot solution of the solute into the crystal.
4. Separating the crystal from the solution of supernatant.

Another thing to note on the recrystallization process is the selection of an appropriate solvent. Some of the requirements of a solvent can be used in the recrystallization process include.

1. Providing sufficient difference in solubility between the purified substance and impurities
2. Not leaving impurities in the crystal
3. It easily to be separated from the crystals
4. It is inert (does not react easily with the crystal)

Impurities in NaCl salt are sand, soil, other ingredients like magnesium sulphate $MgSO_4$ and calcium sulphate $CaSO_4$. The purification procedure of NaCl salt includes dissolving appropriate weight of the salt in a specific volume of distilled water then filters the solution to remove sand and soil from it. Addition of a few drops of barium chloride $BaCl_2$ converts $MgSO_4$ and $CaSO_4$ to soluble salts of magnesium chloride $MgCl_2$ and calcium chloride $CaCl_2$ with remaining precipitate of barium sulphate $BaSO_4$.

$$MgSO_4 + BaCl_2 \longrightarrow MgCl_2 + BaSO_4\downarrow$$

$$CaSO_4 + BaCl_2 \longrightarrow CaCl_2 + BaSO_4\downarrow$$

The precipitate $BaSO_4$ is removed from the solution by filtration and few drops of sodium carbonate Na_2CO_3 solution is added to remove Mg^{2+} and Ca^{2+} ions as precipitates of magnesium carbonate $MgCO_3$ and calcium carbonate $CaCO_3$.

$$MgCl_2 + Na_2CO_3 \longrightarrow MgCO_3\downarrow + 2NaCl$$

$$CaCl_2 + Na_2CO_3 \longrightarrow CaCO_3\downarrow + 2NaCl$$

The excess Na_2CO_3 solution is removed by addition of few drops of dilute solution of hydrochloric acid HCl with heating the solution.

$$HCl + Na_2CO_3 \longrightarrow NaCl + H_2CO_3$$

$$H_2CO_3 \xrightarrow{\Delta} H_2O + CO_2\uparrow$$

Finding the percentage of purity and impurities by calculations:

$$\% \text{ Purity of NaCl salt} = \frac{\text{Wt. (g) pure NaCl}}{\text{Wt. (g) sample}} \times 100$$

$$\% \text{ Impurities} = \frac{\text{Wt. (g) impurities}}{\text{Wt. (g) sample}} \times 100$$

$$\% \text{ Impurities} = 100 - \% \text{ Purity of NaCl}$$

Procedure:

1- Dissolve 5g of NaCl salt sample in 20 ml of distilled water in a beaker and filtrate the solution using funnel, beaker and filter paper.
2- Add few drops of 2% $BaCl_2$ solution; note the appearance of the precipitate.
3- Filter the solution using funnel, conical flask and filter paper. Add 1-2 drops of $BaCl_2$ solution to the filtrate, if the precipitate is appeared filter again. Repeat this step until no precipitate is formed.
4- Add few drops of 5% Na_2CO_3 solution note the formation of the precipitate and filter the solution.
5- Add 1-2 drop of Na_2CO_3 solution to the filtrate of step 4, if the precipitate observed again repeat the filtration.
6- Add few drops of 0.2M HCl solution to the filtrate.
7- Evaporate the solution in a pre-weighed beaker on heater until dry crystals of NaCl salt is obtained.

8- Find the weight of pure NaCl salt by subtracting the weight of beaker and dry pure NaCl from weight of empty beaker.
9- Calculate % purity of NaCl salt.
10- Calculate % impurities in the sample by subtracting 100 from % purity of NaCl or weighing the impurities by subtracting the weight of filter paper and dry impurities from dry filter paper.

Results and calculations:

Name…… Stage First

Student Signature……..

Staff Signature……

Date

Questions and discussion:

1- Define recrystallization and conditions of the method?
2- What are examples of pure substances? Give few examples.
3- Define impurity? Give example of impure liquid sample.
4- What is the life time limit (LTL) allowed for general impurities of drug substances and products? Read the article link below:
https://juniperpublishers.com/gjpps/pdf/GJPPS.MS.ID.555570.pdf

C.O.S.H.H assessment form for use in Chemistry Laboratories

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Experiment name: Determination melting point of a solid organic compound

Subject: General Chemistry

Hazardous Chemicals

Glycerol	Irritant	Contact with molten material can result in severe burns. Direct contact of molten product to the eyes will cause thermal burns and injury. Breathing fumes in confined areas can cause respiratory discomfort and possible irritation.
Organic solid compound	Irritant	Causes severe eye irritation, redness and pain. Causes skin irritation and sensitization to skin. Harmful if swallowed. May cause irritation of the digestive tract. Causes respiratory tract irritation. May be harmful if inhaled. May cause respiratory sensitization.

Risk control

When using a chemical compound. Wear gloves, googles and clothing to prevent skin and eye exposure. Take care when using hot glycerol, causes severe burns! Wear protected gloves at all times. To avoid the fume of hot glycerol uses the fume hood.

Name…… Stage First

Student Signature……

Staff Signature……

Date

LAB -5- Determination melting point of a solid organic compound

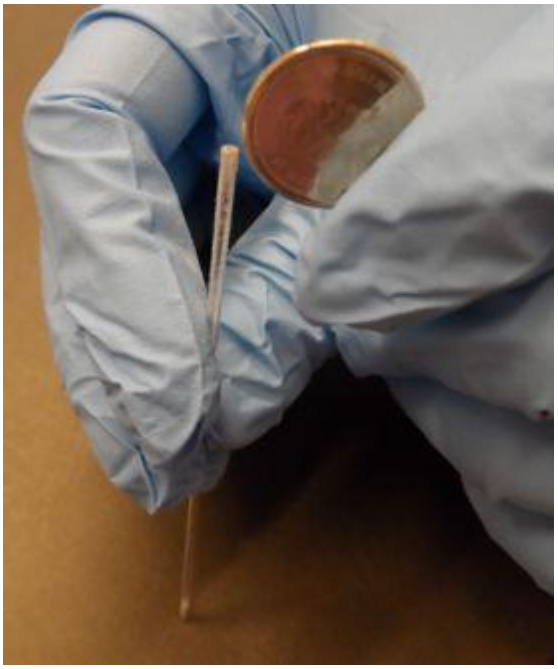
Theory:

The physical properties of a compound, such as melting point and boiling point can provide useful informations which can help in the identification of a sample or to establish its purity. The temperature at which a solid melts and becomes a liquid is the melting point. Since this requires that the intermolecular forces that hold the solid together have to be overcome, the temperature at which melting occurs will depend on the structure of the molecule involved - an example of the relationship between structure and properties. Hence, different compounds tend to have different melting points.

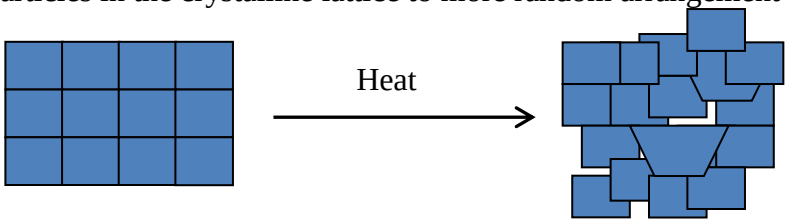
A pure, nonionic, crystalline organic compound usually has a sharp and characteristic melting point (usually 0.5-1.0°C range). A mixture of very small amounts of miscible impurities will produce a depression of the melting point and an increase in the melting point range. Consequently, the melting point of a compound is a criterion for purity as well as for identification.

The melting point of an organic solid can be determined by introducing a tiny amount into a small capillary tube, attaching this to the stem of a thermometer centred in a heating bath, heating the bath slowly, and observing the temperatures at which melting begins and is complete. Pure samples usually have sharp melting points, for example 149.5-150°C or 189-190°C; impure samples of the same compounds melt at lower temperatures and over a wider range, for example 145-148°C or 186-189°C.

The general method is to the heat the sample indirectly by placing the prepared sample (packed in a glass capillary) in or on a heated medium. Thin-walled capillary melting point tubes are used to hold melting point samples. This tube needs to be sealed at one end (pre-sealed tubes should be available in the laboratory, or, an open capillary can be sealed by inserting the tip into a Bunsen flame near the base of the flame and turning the tube in your fingers). To pack the tube, the open end is pressed gently into a small amount of the sample of the crystalline material on a watch glass or weighing paper. To transfer the crystals from the open end to the bottom of the tube, tap the bottom gently on the bench top or scratch the top edge of the tube on a small file or a coin with a milled edge. A densely packed column of crystals about 3 mm high in the tube is all that is required. A packed capillary attached to a normal mercury thermometer is shown to the left. Remember that a slow heating rate at the melting point is needed in order to get an accurate measurement. Record the temperature on the thermometer when the sample starts to melt and record the temperature again when the entire sample has melted (this gives the melting point range).



In another way melting point is the change from the highly ordered arrangement of the particles in the crystalline lattice to more random arrangement of the liquid state.



Solid organic compound

1- Highly ordered arrangement of particles
2- Low thermal energy of particles
3- Restricted motion of particles
4- Regular shape

Liquid organic compound

1- Low ordered arrangement of particles
2- High thermal energy of particles
3- Free motion of particles
4- Random shape

Procedure:

1- Enclose one end of the capillary tube by a source of heat.
2- Add a small amount of dry and finely powdered solid organic compound into a capillary tube in a packed way of about 3 mm height.
3- Place the capillary tube beside a thermometer, using a rubber.
4- Put the capillary tube and thermometer inside an oil bath gently.
5- Heat the oil bath gently by Bunsen burner.
6- Record the temperature at which the first crystal converts to liquid form, until all crystals completely converts to the liquid form.

Results and calculations:

Name…… Stage First

Student Signature……..

Staff Signature……

Date

Questions and discussion:

1- Explain why impurities lower melting point?
2- A melting point of unknown is 120-121°C, how could you tell whether it is urea or cinnamic acid as they both have same melting point?
3- Why the sample should be packed firmly in the capillary tube?
4- Can you use a water bath for a sample of m.p 150°C? Why?

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Experiment name: Determination boiling point of liquid organic compound

Subject: General Chemistry

Hazardous Chemicals

Glycerol	Irritant	Contact with molten material can result in severe burns. Direct contact of molten product to the eyes will cause thermal burns and injury. Breathing fumes in confined areas can cause respiratory discomfort and possible irritation.
Organic liquid compound	Toxic, irritant	Causes eye irritation, skin irritation and respiratory track irritation. Repeated or prolonged exposure may cause drying and cracking of the skin. May be absorbed through the skin. Ingestion may cause central nervous system depression, nausea, vomiting, diarrhea, headache, dizziness and weakness.

Risk control

When using an organic compound. Wear gloves, googles and clothing to prevent skin and eye exposure. Take care when using hot glycerol, causes severe burns! Wear protected gloves at all times.

Name…… Stage First

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Date

LAB -6- Determination boiling point of liquid organic compound

Theory:

Boiling point**,** is the temperature at which the pressure exerted by the surroundings upon a liquid is equalled by the pressure exerted by the vapour of the liquid; under this condition, addition of heat results in the transformation of the liquid into its vapour without raising the temperature. At any temperature a liquid partly vaporizes into the space above it until the pressure exerted by the vapour reaches a characteristic value called the vapour pressure of the liquid at that temperature. As the temperature is increased, the vapour pressure increases; at the boiling point, bubbles of vapour form within the liquid and rise to the surface. The amount of energy to overcome the attraction force between the molecules of the liquid substances to convert it to vapour is called boiling point.

The boiling point of a liquid is the temperature at which the vapour pressure of the liquid equals to the applied pressure (normally 1 atm) or is a temperature at which the vapour pressure of the liquid equals to the pressure of the surrounding system. Boiling point is a characteristic physical constant of the liquid organic compounds and pure sample give us a sharp boiling point. Therefore, boiling point can be used to identify and characterize liquid organic compounds.

Generally we have two types of Boiling point apparatus; classical boiling point apparatus and Electrical (digital) boiling point apparatus. Pressure, impurities and attraction forces are factors that affecting on boiling point. The boiling point of a liquid varies according to the applied pressure; the normal boiling point is the temperature at which the vapour pressure is equal to the standard sea-level atmospheric pressure (760 mm [29.92 inches] of mercury). At sea level, water boils at 100°C (212°F). At higher altitudes the temperature of the boiling point is lower. Generally with increasing the pressure the boiling point will increase. The following table shows boiling point of water at various locations.

Location	Feet above sea level	Pressure (atm)	Boiling point °C
Top of mountain Everest, Tibet	29,028	0.32	70
Top of mountain Mckinley, Alaska	20,320	0.45	79
Top of mountain Whitney, California	14,494	0.57	85
Top of mountain Washington, New Hampshire	6,293	0.78	93
Boulder, Colorado	5430	0.80	94
Madison, Wisconsin	900	0.96	99
New York City, New York	10	1.00	100
Death Valley, California	-282	1.01	100.3

The effect of an impurity on the boiling point of a liquid varies with the characteristics of the impurities i.e. depending upon its solubility and volatility. Insoluble and non-volatile impurities don't affect the attraction forces between the molecules of the liquid. The boiling point of the mixture is the same as the boiling point of the pure liquid at the given pressure e.g. sand in water doesn't affect the boiling point of water. An impurity that is not volatile, but soluble in the liquid affects the attraction forces between the molecules and lowers the vapour pressure of the liquid above the solution, resulting in an increase in the boiling point of the impure liquid e.g. NaCl is a soluble, non-volatile impurity and the boiling point of water is elevated about 0.5128 °C.

Procedure:

1- Add about (0.5-1) ml of the liquid sample into the test tube.
2- Enclose one ends of the capillary tube; then immerse the opened side into the test tube.
3- Place the test tube beside a thermometer using a rubber.
4- Put the test tube and the thermometer into an oil bath gently.
5- Heat the oil bath slowly by Bunsen burner.
6- Record the temperature T_1 at which a rapid continuous stream of air bubble come out from the capillary tube.
7- Record the temperature T_2 at which stream of air bubble disappeared from the capillary tube.
8- Find the real boiling point of the liquid by detecting average boiling point,
   $T_{real} = (T_1 + T_2)/2$
9- Find the following link:
   https://www.youtube.com/watch?v=8b5Ha-8QGhY

Results and calculations:

Name…… Stage First

Student Signature……..

Staff Signature……

Date

Questions and discussion:

1- Explain how soluble and volatile impurity affects boiling point of a liquid organic compound?
2- What would be the boiling point of pure water on moon? Explain why?
3- Which type of boiling point apparatus is accurate? Why?
4- Is it possible to use boiling point to identify liquid organic compounds? How?

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Experiment name: Simple Distillation

Subject: General Chemistry

Hazardous Chemicals

Acetone	Toxic, irritant and flammable	Causes eye irritation, skin irritation and respiratory track irritation. Ingestion may cause nausea, stomach pain and vomiting. Prolonged or repeated contact with skin may cause redness, itching, irritation and eczema.

Risk control

When using acetone. Wear gloves, googles and clothing to prevent skin and eye exposure. Avoid keeping the acetone near the heat in an opened container.

Name…… Stage First

Student Signature……

Staff Signature……

Date

LAB -7- Simple Distillation

Theory:

Distillation is the process that includes vaporizing a liquid from a pot, condensing the vapour and collecting the condensate in a reservoir. The evaporation is an endothermic process that requires heat (external) and the condensation is an exothermic process that needs cooling (condenser to cool the vapour). This technique is very useful for separating liquids mixture when the components have different boiling points, or when one of the components will not distil. It is one of the principal methods of purifying a liquid. The method is commonly used for those liquids which are sufficiently stable at their boiling points (boil without decomposition) and contain non-volatile impurity.

According to the differences in boiling points between the liquids in a mixture, distillation process is classified into four types:

1- Simple distillation
2- Fractional distillation
3- Vacuum distillation (distillation at reduced pressure)
4- Steam distillation

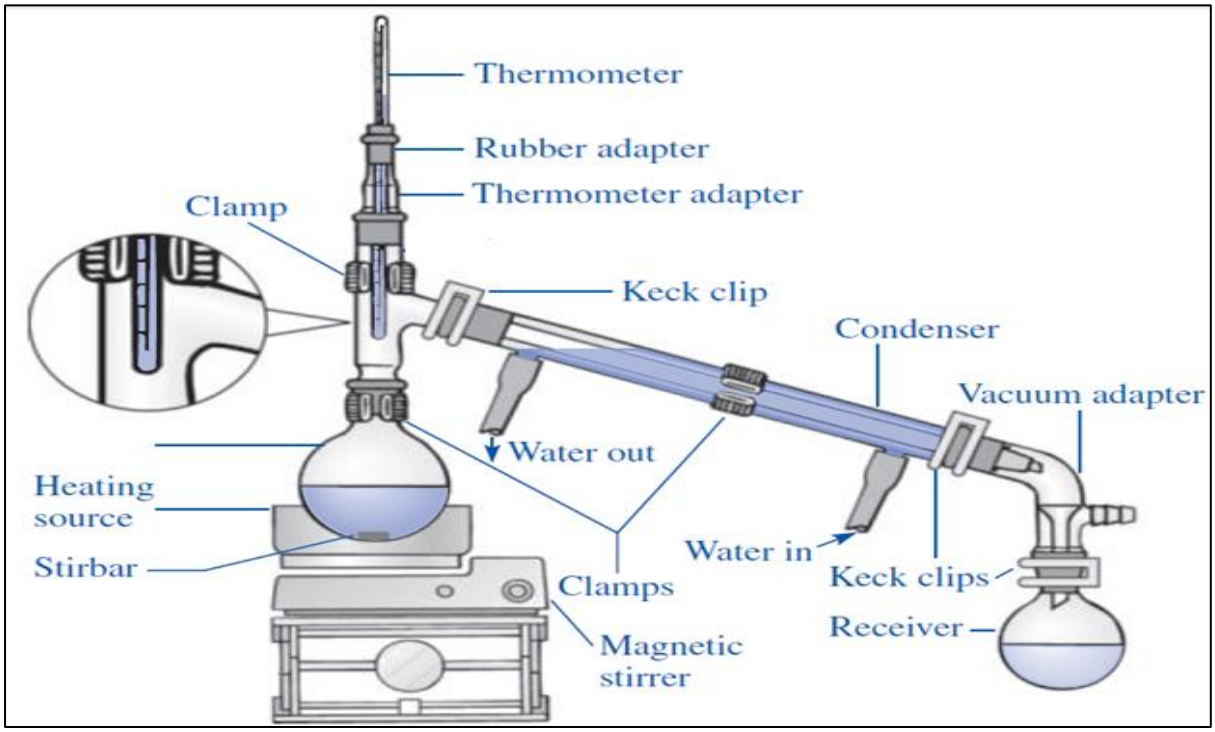
Simple distillation is useful for separating liquids boiling below 150 ˚C at 1 atm. The liquids should dissolve in each other and the difference in boiling point between various liquid components should be at least 25˚C (i.e., water from salt water solution).

Fractional distillation is used for separating liquids mixture in which boiling points of the volatile components differ by less than 25˚C from each other.

In the separation of a mixture of liquids with boiling points above 150˚C at 1 atm the technique of vacuum distillation is used. Steam distillation is mainly used to isolate oils from natural components.

Simple distillation is frequently employed for determining the boiling point of a liquid or for purifying a liquid which is contaminated by another liquid whose boiling point is either lower or higher by 25˚C or more. The apparatus used in distillation process are a round bottom flask (distilling flask) in which the impure liquid is heated (pumic stones are placed to avoid superheating and bumping), a water condenser (condenser) through which the heated vapour is cooled to the liquid state, and a receiver (receiving flask) in which the purified liquid, called the distillate, is collected (Fig. 2). A thermometer of suitable temperature range is fitted into the neck of the distillation flask by a rubber adapter. The bulb of the thermometer should be in the centre of the neck of the flask and slightly below the level of condenser joint. When the flask is heated, the temperature rises gradually and the liquid starts boiling when its vapour pressure becomes equal to atmospheric pressure. The vapours get condensed as the

pass through the condenser. Only the liquid which distils at a constant temperature is collected in a receiver.



Procedure:

1- A mixture composed from A (acetone) and B (water) liquids with boiling point (56 and 100) °C respectively is heated using simple distillation apparatus.
2- The lowest boiling point liquid is vaporized and elevated from the solution until it reaches the top of the system, with recording its real boiling point by the thermometer.
3- The elevated vapour is converted to the liquid form by condensing the vapour in the condenser through cooling it and then collecting the distillate at the receiver.
4- Measure the volume of distillate liquid of lowest boiling point using cylinder.
5- The liquid with highest boiling point is remaining at the distillation flask. Measure its volume.

Results and calculations:

	Boiling point °C	Collected volume ml
Liquid (A) Acetone		
Liquid (B) Water		

Name…… **Stage** First

Student Signature……..

Staff Signature……

Date

Questions and discussion:

1- If the thermometer is set too high in the apparatus, the temperature reading will not be accurate. Too high or too low, and why?
2- What is the purpose of a boiling stone? When should they be used?
3- How much liquid can one put in a distilling flask? Why?
4- When should a simple distillation be used and when must a fractional distillation is used?