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Physical Chemistry Experiments

Experimental laboratory
manual for students of higher education
in the fields of chemical technology and engineering

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The manual introduces to the basic concepts of physical chemistry (the state behavior of liquids and their mixtures, the fundamentals of chemical thermodynamics, phase equilibrium, the electrochemistry, chemical kinetics) and contains instructions on how to prepare and perform experiments on the discipline "Physical Chemistry Experiments". The experimental laboratorymanual will help the student to set up the experiment correctly and gain experience. Such experience will provide an opportunity to solve scientific and production tasks in the further independent activity of a chemical technologist.

The experimental laboratory manual is intended for undergraduate students majoring in chemical technologies & engineering

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CONTENT

INTRODUCTION

The discipline "Physical Chemistry Experiments" is outlines the basic laws and concepts of physical chemistry, i.e. the state behavior of liquids and their mixtures, the fundamentals of chemical thermodynamics, phase equilibrium, chemical equilibrium, the fundamentals of electrochemistry, chemical kinetics and the kinetics of transport processes. Conclusions from theoretical positions need to be confirmed by experience. Physical chemistry is enriched with new facts through experimental research.

The experimental laboratory main purpose of the experimental classes is to form the initial level of knowledge of students in this discipline. Experimental training will help to deepen knowledge and better mastering of the discipline, students' development of chemical thinking skills.

Task: to put into practice theoretical program material, to master the skills of work in the laboratory for further professional activity.

The student will get acquainted with the methods and techniques working in the physic-chemical laboratory. The manual will help the student to set up the experiment correctly, get information about the substance, and gain experience. Such experience will provide an opportunity to solve scientific and production tasks in the further independent activity of a chemical technologist.

Brief theoretical explanations precede the description of each experiment to clarify the meaning of the work and its significance. The student gets acquainted with the device and the principle of the devices, gets practical calculation skills when performing an experiment in the laboratory.

In this experimental laboratory manual, the student will be able to find all the necessary materials for preparing and performing experiments in the discipline "Physical Chemistry Experiments".

HOW TO PREPARE FOR CHEMISTRY LAB?

Chemistry lab is a required component of most chemistry courses. Learning about lab procedures and performing experiments helps you to learn techniques and reinforces textbook concepts. Make the most of your time in the lab by coming to lab prepared. Review these pre-lab tips before starting an experiment [1].

Complete any pre-lab assignments or homework. The information and calculations are intended to make the lab exercise quicker and easier. Know the location of the lab safety equipment and understand how to use it. In particular, know the location of the emergency exit, fire extinguisher, eye wash station and safety shower [1].

Read through the experiment before going to lab. Make sure you understand the steps of the experiment. Write down any questions you have so that you can ask them before starting lab [1].

Start filling out your lab book with information about the experiment. It's a good idea to draw out your data table in advance so all you need to do in lab is fill it in with numbers [1].

Review the Material Safety Data Sheets (MSDSs) of the chemicals you will be using during lab [1].

Make certain you have all of the glassware, materials and chemicals needed to complete the lab before starting any part of the procedure [1].

Understand disposal procedures for the chemicals and other items used in your experiment. If you are unclear about what to do with your experiment after it has been completed, ask your instructor about it. Don't throw items in the trash or dump liquids down the drain or in waste disposal containers until you are certain it is acceptable to do so [1].

Be prepared to take data in the lab. Bring your notebook, a pen and a calculator [1].

Have personal safety gear, such as a lab coat and goggles, clean and ready to use before lab [1].

HOW TO WRITE A LAB REPORT?

Lab reports are an essential part of all laboratory courses and usually a significant part of your grade. If your instructor gives you an outline for how to write a lab report, use that. Some instructors require the lab report be included in a lab notebook, while others will request a separate report. Here's a format for a lab report you can use if you aren't sure what to write or need an explanation of what to include in the different parts of the report [2].

A lab report is how you explain what you did in experiment, what you learned, and what the results meant [2]. Here is a standard format. If you prefer, you can print and fill in the science lab report template or download the pdf version of the template.

When scientists perform experiments, they make observations, collect and analyze data, and formulate generalizations about the data. When you work in the laboratory, you should record all your data in a laboratory report. An analysis of data is easier if all data are recorded in an organized, logical manner [2].

Title. All lab reports have title pages; it would be a single page that states:

- The title of the experiment.
- Your name and the group number.
- The date the lab was performed or the date the report was submitted [2].

Objectives (purpose). Usually the Introduction is one paragraph that explains the objectives or purpose of the lab. You need to state the purpose of the experiment, or why you did it [2].

Instruments and Reagents (materials). List everything needed to complete your experiment [2].

Experimental procedure. Describe the steps you completed during your investigation. This is your procedure. Be sufficiently detailed that anyone could read this section and duplicate your experiment. Write it as if you were giving direction for someone else to do the lab. It may be helpful to provide a Figure to diagram your experimental setup. The numbered steps of the procedure tell you how to carry out the activity and sometimes offer hints to help you be successful in the laboratory. Some activities have CAUTION statements in the procedure to alert you to hazardous substances or techniques [2].

Data. Always record data and observations in an organized way as you do the activity. Numerical data obtained from your procedure usually is presented as a table. Data encompasses what you recorded when you conducted the experiment. It's just the facts, not any interpretation of what they mean [2].

Figures & Graphs. Graphs and figures must both be labeled with a descriptive title. Label the axes on a graph, being sure to include units of measurement. The independent variable is on the X-axis. The dependent variable (the one you are measuring) is on the Y-axis. Be sure to refer to figures and graphs in the text of your report [2].

Results. Describe in words what the data means. Sometimes the Results section is combined with the Discussion (Results & Discussion) [2].

Discussion. The Data section contains numbers. The section contains any calculations you made based on those numbers. This is where you interpret the data and determine whether or not a hypothesis was accepted [2].

Conclusions. Most of the time the conclusion is a single paragraph that sums up what happened in the experiment, whether your hypothesis was accepted or rejected, and what this means [2].

References. If your research was based on someone else's work or if you cited facts that require documentation, then you should list these references [2].

Questions and answer. The questions in this section ask you to apply what you have learned in the activity to other real-life situations. You may be asked to make additional conclusions or research a question related to the activity"[2].

SAFETY IN THE LABORATORY

The chemistry laboratory is a place to experiment and learn. You must assume responsibility for your own personal safety and that of people working near you. Accidents are usually caused by carelessness, but you can help prevent them by closely following the instructions printed in this manual and those given to you by your teacher. The following are some safety rules to help guide you in protecting yourself and others from injury in a laboratory [3].

1. The chemistry laboratory is a place for serious work. Do not perform activities without your teacher’s permission. Never work alone in the laboratory. Work only when your teacher is present [3].

2. Study your lab activity before you come to the lab. If you are in doubt about any procedures, ask your teacher for help [3].
3. Safety goggles and a laboratory apron must be worn whenever you work in the lab. Gloves should be worn whenever you use chemicals that cause irritations or can be absorbed through the skin [3].
4. Contact lenses should not be worn in the lab, even if goggles are worn. Lenses can absorb vapors and are difficult to remove in an emergency [3].
5. Long hair should be tied back to reduce the possibility of it catching fire [3].
6. Avoid wearing dangling jewelry or loose, draping clothing. The loose clothing may catch fire and either the clothing or jewelry could catch on chemical apparatus [3].
7. Wear shoes that cover the feet at all times. Bare feet or sandals are not permitted in the lab [3].
8. Know the location of the fire extinguisher, safety shower, eyewash, fire blanket, and first-aid kit. Know how to use the safety equipment provided for you [3].
9. Report any accident, injury, incorrect procedure, or damaged equipment immediately to your teacher [3].
10. Handle chemicals carefully. Check the labels of all bottles before removing the contents. Read the labels three times: before you pick up the container, when the container is in your hand, and when you put the bottle back [3].

11. Do not return unused chemicals to reagent bottles [3].
12. Do not take reagent bottles to your work area unless specifically instructed to do so. Use test tubes, paper, or beakers to obtain your chemicals. Take only small amounts. It is easier to get more than to dispose of excess [3].

13. Do not insert droppers into reagent bottles. Pour a small amount of the chemical into a beaker [3].
14. Never taste any chemical substance. Never draw any chemicals into a pipette with your mouth. Eating, drinking, chewing gum, and smoking are prohibited in the laboratory [3].
15. If chemicals come into contact with your eyes or skin, flush the area immediately with large quantities of water. Immediately inform your teacher of the nature of the spill [3].
16. Keep combustible materials away from open flames. (Alcohol and acetone are combustible) [3].
17. Handle toxic and combustible gases only under the direction of your teacher. Use the fume hood when such materials are present [3].
18. When heating a substance in a test tube, be careful not to point the mouth of the tube at another person or yourself. Never look down the mouth of a test tube [3].
19. Use caution and the proper equipment when handling hot apparatus or glassware. Hot glass looks the same as cool glass [3].
20. Dispose of broken glass, unused chemicals, and products of reactions only as directed by your teacher [3].
21. Know the correct procedure for preparing acid solutions. Always add the acid slowly to the water [3].
22. Keep the balance area clean. Never weigh chemicals directly on the pan of the balance [3].
23. Do not heat graduated cylinders, burettes, or pipettes with a laboratory burner [3].
24. After completing an activity, clean and put away your equipment. Clean your work area. Make sure the gas and water are turned off. Wash your hands with soap and water before you leave the lab [3].
25. The below given table is helpfull in an accident situation.

Read and remember them:

Situation	Safe Response
Burns	Flush with ethanol solution or diluted solution of $KMnO_4$.
Cuts and Bruises	Treat as directed by instructions included with first aid kit
Fainting or collapse	Provide person with fresh air, have him recline in a position so that his head is lower than their body
Foreign Matter in Eyes	Flush about 15 min with plenty of water, then go to the Doctor
Severe bleeding	Apply pressure or a compress directly to the wound and get medical attention immediately
1. Spills, general 2. Acid burns 3. Base burns	1. Wash area with plenty of water, use safety shower if needed 2. Use $NaHCO_3$ 3. Use 3 % of boric acid or acetic acid

SI UNIT

The first measurements were probably based on the human body (the length of the foot, for example). In time, fixed standards developed, but these varied from place to place. Each country or government (and often each trade) adopted its own units. As science became more quantitative in the seventeenth and eighteenth centuries, scientists found that the lack of standard units was a problem. They began to seek a simple, international system of measurement. In 1791 a study committee of the French Academy of Sciences devised such a system. Called the metric system, it became the official system of measurement for France and was soon used by scientists throughout the world. Most nations have since adopted the metric system or, at least, have set a schedule for changing to it [4].

SI Base Units and SI Prefixes. In 1960 the General Conference of Weights and Measures adopted the International System of units (or SI, after the French le Systyme International d'Unites), which is a particular choice of metric units. This system has seven SI base units. In this chapter, we will discuss four base quantities: length, mass, time, and temperature [4]. One advantage of any metric system is that it is a decimal system. In SI, a larger or smaller unit for a physical quantity is indicated by an SI prefix, which is a prefix used in the International System to indicate a power of 10. For example, the base unit of length in SI is the meter (somewhat longer than a yard), and 10 -2 meter is called a centimeter. Thus 2.54 centimeters equals $2.54*10^{-2}$ meters. The SI prefixes used in our manual are presented in Table 1 [4].

Table 1. SI base units

Physical quantity	Name of unit	Abbreviation
Mass	Kilogram	kg
Length	Meter	m
Temperature	Kelvin	K
Amount of substance	Mole	mol
Time	Second	s
Electric current	Ampere	A

Some quantities are expressed as a function of more than one fundamental units known as derived units. For example, velocity, acceleration, work, energy etc. The quantities used in this manual are presented in Table 2.

Table 2. Some quantities used in this manual

Quantity with Symbol	Unit (S.I.)	Symbol
Velocity (v)	Meter per sec	ms^{-1}
Area (A)	Square meter	m^2
Volume (V)	Cubic meter	m^3
Density (r)	Kilogram m^{-3}	Kg m^{-3}
Energy (E)	Joule (J)	Kg m^2 s^{-2}
Frequency (n)	Hertz	Cycle per sec
Pressure (P)	Pascal (Pa)	Nm^{-2}
Electrical charge	Coulomb (C)	A-s (ampere – second)

Length, Mass, and Time

The meter (m) is the SI base unit of length. By combining it with one of the SI prefixes, you can get a unit of appropriate size for any length measurement. For the very small lengths used in chemistry, the nanometer (nm; 1 nanometer $=10^{-9}$ m) or the picometer (pm; 1 picometer = 10^{-12} m) is an acceptable SI unit, table 3. A non-SI unit of length traditionally used by chemists is **the angstrom (E),** which equals 10^{-10} m. (An oxygen atom, one of the minute particles of which the substance oxygen is composed, has a diameter of about 1.3 A. If you could place oxygen atoms adjacent to one another, you could line up over 75 million of them in 1cm) [4].

Table 3. Common metric length units

Unit	Symbol	Relation
meter	m	
kilometer	km	1 km = 10^3 m
decimeter	dm	1 dm = 10^{-1} m
centimeter	cm	1 cm = 10^{-2} m
millimeter	mm	1 mm = 10^{-3} m
micrometer	µm	1 µm = 10^{-6} m
nanometer	nm	1 nm = 10^{-9} m

The kilogram (kg) is the SI base unit of mass, equal to about 2.2 pounds. This is an unusual base unit in that it contains a prefix. In forming other SI mass units, prefixes are added to the word gram (g) to give units such as the milligram (mg; 1 mg = 10^{-3} g), table 4[4].

Table 4. Commonly used metric weight and mass units

Unit	Symbol	In terms of grams
gram	g	
kilogram	kg	1 kg = 10^3g
milligram	mg	1 mg = 10^{-3}g
microgram	µg	1 µg = 10^{-6}g

The second (s) is the SI base unit of time. Combining this unit with prefixes such as milli-, micro-, nano-, and pico-, you create units appropriate for measuring very rapid events. The time required for the fastest chemical processes is about a picosecond, which is on the order of how fast supercomputers can perform a single calculation [4].

When you measure times much longer than a few hundred seconds, you revert to minutes and hours, an obvious exception to the prefix–base format of the International System" [4].

Unit of temperature.

Temperature is difficult to define precisely, but we all have an intuitive idea of what we mean by it. It is a measure of “hotness.” A hot object placed next to a cold one becomes cooler, while the cold object becomes hotter. Heat energy passes from a hot object to a cold one, and the quantity of heat passed between the objects depends on the difference in temperature between the two. Therefore, temperature and heat are different, but related, concepts [5].

A thermometer is a device for measuring temperature. The common type consists of a glass capillary containing a column of liquid whose length varies with temperature. A scale alongside the capillary gives a measure of the temperature. **The Celsius scale** (formerly the centigrade scale) is the temperature scale in general scientific use. On this scale, the freezing point of water is 0^0C and the boiling point of water at normal barometric pressure is 100^0C. However, the SI base unit of temperature is **the kelvin (K),** a unit on an absolute temperature scale. (See the first margin note on the next page.) On any absolute scale, the lowest temperature that can be attained theoretically is zero. The Celsius and the Kelvin scales have equal-size units (that is, a change of 1^0C is equivalent to a change of 1 K), where 0^0C is a temperature equivalent to 273.15 K. Thus, it is easy to convert from one scale to the other, using the formula [5]:

$$T_K = (t_C \times \frac{1K}{1^\circ C}) + 273.15K$$

where T_K is the temperature in kelvin and t_C is the temperature in degrees Celsius. A temperature of 20^0C (about room temperature) equals 293 K [5].

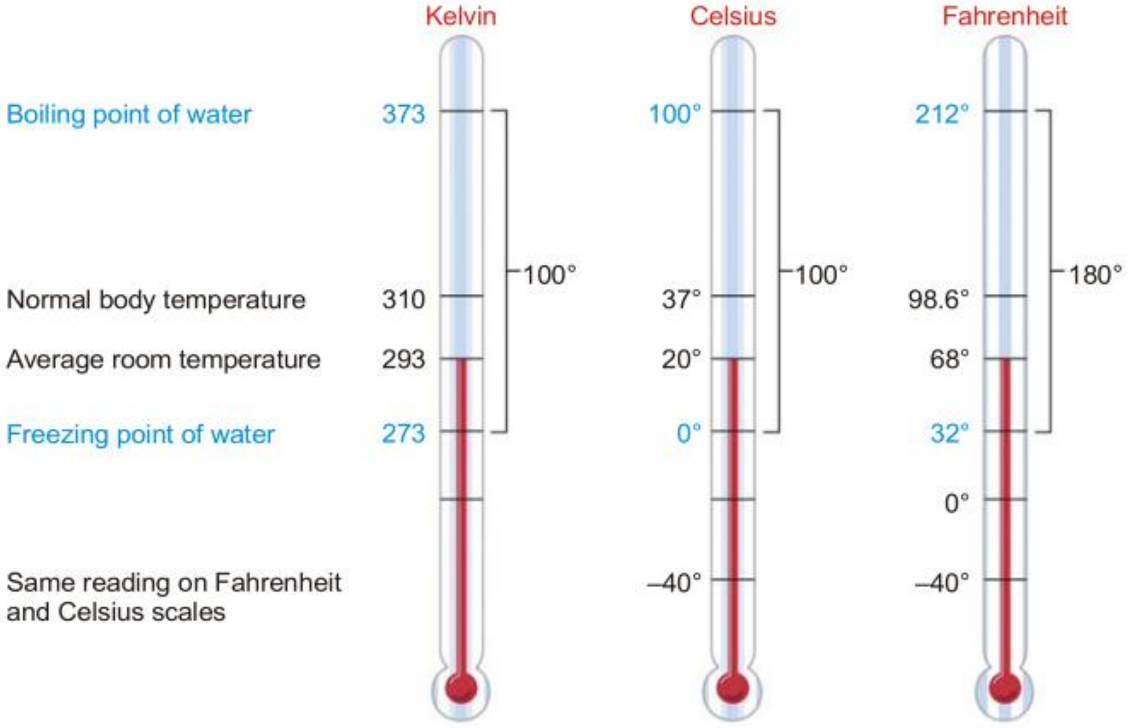


Figure 1. A comparison of Kelvin, Celsius, and Fahrenheit scales

Unit of Volume.

Volume is defined as length cubed and has the SI unit of cubic meter (m^3). This unit is too large a unit for normal laboratory work, so we use either cubic decimeters (dm^3) or cubic centimeters (cm^3, also written cc). Traditionally, chemists have used the liter (L), which is a unit of volume equal to a cubic decimeter (approximately one quart). In fact, most laboratory glassware is calibrated in liters or milliliters (1000 mL = 1 L). Because 1 dm equals 10 cm, a cubic decimeter, or one liter, equals $(10\ cm)^3 = 1000\ cm^3$. Therefore, a milliliter equals a cubic centimeter [6].

In summary,

$$1\ L = 1\ dm^3 \text{ and } 1\ mL = 1\ cm^3\ (cc)[6]$$

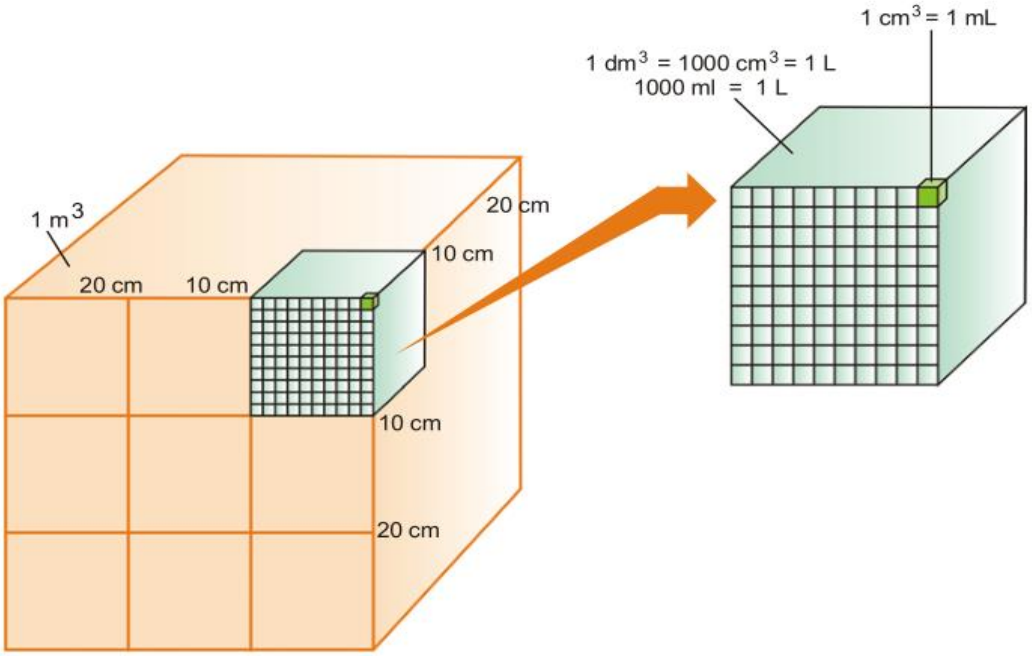


Figure 2. Relationship between length and volume

Unit of Density.

The density of an object is its mass per unit volume. You can express this as $\rho = m/V$, where ρ is the density, m is the mass, and V is the volume. Suppose an object has a mass of 15.0 g and a volume of 10.0 cm^3[6].

Substituting, you find that

$$\rho = \frac{m}{V} = \frac{15g}{10cm^3} = 1.50g/cm^3$$

The density of the object is 1.50 g/cm^3 (or 1.50 $g*cm^{-3}$) [6].

Density is an important characteristic property of a material. Water, for example, has a density of 1.000 g/cm^3 at 4^0C and a density of 0.998 g/cm^3 at 20^0C. Lead has a density of 11.3 g/cm^3 at 20^0C. Oxygen gas has a density of $1.33*10^{-3}$ g/cm^3 at normal pressure and 20^0C. (Like other gases under normal conditions, oxygen has a density that is about 1000 times smaller than those of liquids and solids.) Because the density is characteristic of a substance, it can be helpful in identifying it. Density can also be useful in determining whether a substance is pure. Consider a gold bar whose purity is questioned. The metals likely to be mixed with gold, such as silver or copper, have lower densities than gold. Therefore, an adulterated (impure) gold bar can be expected to be far less dense than pure gold [6].

Unit of Force.

Force (F) is defined as the product of mass (m) and acceleration (a).

$$F = m \times a$$

Acceleration is the change in velocity (v) per unit time (t). Velocity is the change in distance (l) per unit time. Using SI base units, we can derive the unit for acceleration.

The derived SI unit for force, then, is kg ms^{-2}. The unit is called newton and has the symbol N. Thus,

$$1\ N = 1\ kg\ ms^{-2}$$

Unit of work and heat energy.

Work has been defined as the product of the force and the distance through which it operates

$$w = f \times d$$

Because force is expressed in newtons and distance in meters, the SI units of work and energy is the newton-meter. It is also called Joule (J).

$$1J = 1\ Nm$$

Between the objects is difference of a temperature and heat is energy that flows from one object to another. The heat (quantity) transferred is best expressed in joules. But it is often given in calories (cal). One calorie is defined as exactly 4.184 joules. Thus,

$$1\ cal = 4.184\ J$$

One calorie of energy will raise the temperature of 1 g of liquid water by 1°C. The calorie is a non-SI unit, but like the joule it can be used for any form of energy. The calorie written with a capital C is equal to one kilocalorie, 1000 calories. Thus,

$$1\ C = 1000\ cal$$

Unit of pressure.

Pressure (P) is defined as the force (F) per unit area (A) exerted on a surface.
That is,

$$P = F/A$$

The SI unit Nm^{-2} is named Pascal and given the symbol Pa.

Three other units which have been traditionally used are:

atmosphere, symbol atm, is defined as the pressure exerted by a column of mercury 760 mm in height at 0ºC.

torr, symbol Torr, is defined as the pressure exerted by a 1 mm column of mercury at 0ºC.

millimeter of mercury or mm Hg, which is the height in millimeters of mercury that the pressure can support. The various units of pressure are related as

$$1\ atm = 760\ Torr = 76\ mm\ Hg = 1.013 \times 10^5\ Pa$$

Experiment 1. Determination of the heat of solution of an unknown salt

Experimental purpose

1.To determine the thermal effect of neutralization reaction of strong acid by strong base by calorimetric method;
2. To determine the total heat capacity of the system (constant of the calorimetric system);
3. To determine the integral heat of solution of an unknown salt.

Experimental principles

Chemical thermodynamics is the branch of physical chemistry which deals with transport of heat either as a result of chemical change or physical change. The thermal effect of chemical reaction is the amount of heat which absorbed or released as a result of this reaction.

The heat of neutralization of an acid is defined as the amount of heat evolved when one equivalent of an acid and one equivalent of a base undergo a neutralization reaction to form water and a salt. Similarly, the heat of neutralization of a base is the amount of heat evolved when 1 g equivalent of the base is completely neutralized by a strong acid in a dilute solution:

$$HA + BOH = AB + H_2O + 57{,}3\ kJ$$

For a constant pressure (P=const), **calorimeter** measures the heat effects of variety of reactions such as neutralization reactions, heat of solution and heat of dilutions. A coffee cup calorimeter is basically constructed from a polystyrene
cup with a lid, in which, the cup is filled with a known amount of water and a thermometer inserted measures the heat changes associated with the reaction, fig.3.

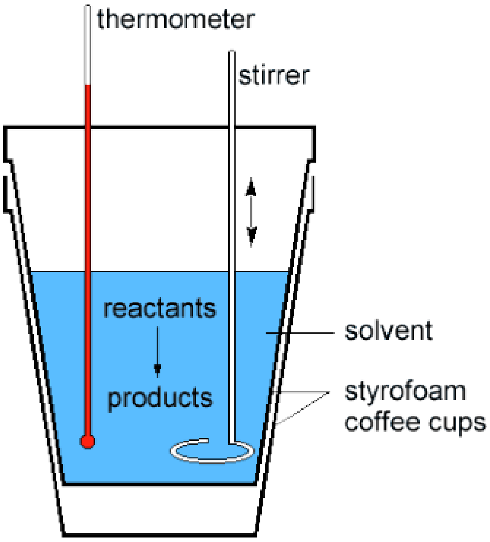


Figure 3. A coffee cup calorimeter

Heat Q is given the system and consumed to the internal energy ΔU and to perform work A:

$$Q = \Delta U + A$$

For isobar-isothermal process: p, T = const, $A_{gas} = p\,\Delta V$

Hess's law (the law of constant heat summation). This law was presented by Hess in 1840. According to this law "If a chemical reaction can be made to take place in a number of ways in one or in several steps, the total enthalpy change (total heat change) is always the same, i.e. the total enthalpy change is independent of intermediate steps involved in the change". The enthalpy change of a chemical reaction depends on the initial and final stages only.

$$Q = -\Delta H$$

The heat of reaction (or enthalpy of reaction) is actually the difference between the enthalpies of the products and the reactants when the quantities of the reactants indicated by the chemical equation have completely reacted. Mathematically, Enthalpy of reaction (heat of reaction):

$$\Delta H_{chem\ reaction} = \Sigma \Delta H^\circ_{products} - \Sigma \Delta H^\circ_{reactants}$$

$$aA + bB = cC + dD$$

$$\Delta H^\circ_A \quad \Delta H^\circ_B \quad \Delta H^\circ_C \quad \Delta H^\circ_D$$

$$\Delta H_{chem\ react} = (\Delta H^\circ_D \cdot d + \Delta H^\circ_C \cdot c) - (\Delta H^\circ_B \cdot b + \Delta H^\circ_A \cdot a)$$

Illustrations of Hess's Law.

(1) Burning of carbon to CO_2. Carbon can be burnt to carbon dioxide directly or it may first be changed to carbon monoxide which may then be oxidised to carbon dioxide.

1st way :

$$C(s) + O_2(g) \longrightarrow CO_2(g) \qquad \Delta H = -94.05\ \text{kcal}$$

2nd way :

$$C(s) + \tfrac{1}{2}O(g) \longrightarrow CO_2(g) \qquad \Delta H = -26.42\ \text{kcal}$$

$$CO(g) + \tfrac{1}{2}O_2(g) \longrightarrow CO(g) \qquad \Delta H = -67.71\ \text{kcal}$$

overall change $$C(s) + O_2(g) \longrightarrow CO_2(g) \qquad \Delta H = -94.13\ \text{kcal}$$

It is evident from above that the total heat energy evolved is the same in the two cases.

(2) Formation of Sodium hydroxide from Na. The formation of sodium hydroxide from metallic sodium presents another example of Hess's law. The process can be carried out in two ways.

1st way :

$$\begin{array}{rcll} 2\text{Na}(s)+\frac{1}{2}\text{O}_2(g) & \longrightarrow & \text{Na}_2\text{O}(s) & \Delta H = -100\ \text{kcal} \\ \text{Na}_2\text{O}(s)+\text{H}_2\text{O}(l) & \longrightarrow & 2\text{NaOH}(aq) & \Delta H = -56\ \text{kcal} \\ \hline 2\text{Na}(s)+\text{H}_2\text{O}(l)+\frac{1}{2}\text{O}_2(g) & \longrightarrow & 2\text{NaOH}(aq) & \Delta H = -156\ \text{kcal} \end{array}$$

2nd way :

$$\begin{array}{rcll} 2\text{Na}(s)+2\text{H}_2\text{O}(l) & \longrightarrow & 2\text{NaOH}(aq)+\text{H}_2(g) & \Delta H = -88\ \text{kcal} \\ \text{H}_2(g)+\frac{1}{2}\text{O}_2(g) & \longrightarrow & \text{H}_2\text{O}(g) & \Delta H = -68.5\ \text{kcal} \\ \hline 2\text{Na}(s)+\text{H}_2\text{O}(l)+\frac{1}{2}\text{O}_2(g) & \longrightarrow & 2\text{NaOH}(aq) & \Delta H = -156.5\ \text{kcal} \end{array}$$

It may be observed that the total heat evolved in carrying the reaction in two different ways is the same. The difference of 0.5 kcal is within the experimental error.

It is obvious from the above examples that by the addition of a series of chemical equations we can obtain not only the resultant products of this series of reactions but also the net heat effect. It is, therefore, clear that: thermochemical equations may be multiplied, added or subtracted like ordinary algebraic equations. For this reason, Hess's law has been of great service in the indirect determination of heats of formation.

Applications of Hess's law:

a) For the determination of enthalpies of formation of those compounds which cannot be prepared directly from the elements easily using enthalpies of combustion of compounds.

b) For the determination of heat of formation of substances which otherwise cannot be measured experimentally. The substances like methane, carbon monoxide, benzene, etc., cannot be prepared by uniting their elements. Therefore, it is not possible to measure the heats of formation of such compounds directly.

c) For the determination of enthalpies of extremely slow reactions.

d) For the determination of enthalpies of transformation of one allotropic form into another.

e) For the determination of bond energies:

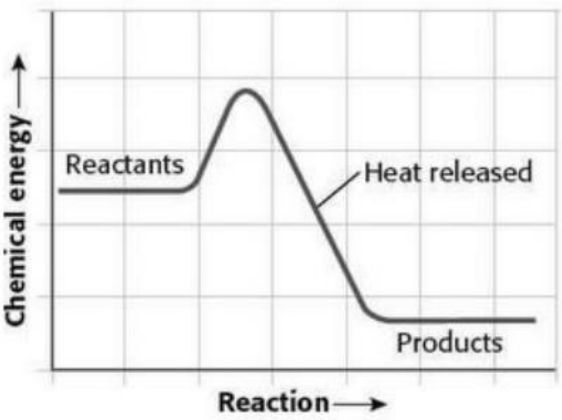
ΔH reaction = $\sum$ Bond energies of reactants – $\sum$ Bond energies of products.

f) For the determination of resonance energy.

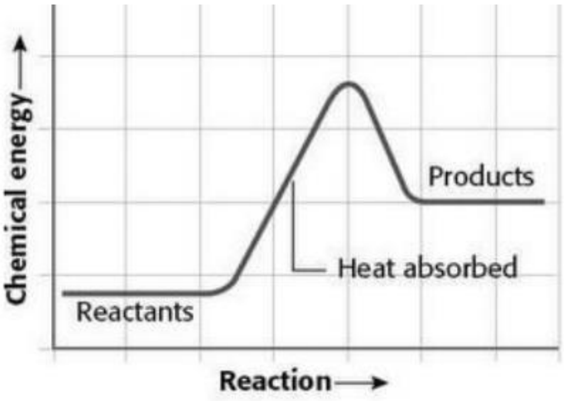
g) For the determination of lattice energy.

If the reaction is generated heat (Q>0), enthalpy of the system is lowered ($\Delta H < 0$), in this case the reaction is called exothermic.

If the reaction is absorbed heat (Q<0), the system increases the enthalpy ($\Delta H > 0$), and it called the endothermic reaction.



A In an exothermic reaction chemical energy is released, often as heat.



B In an endothermic reaction, energy from the surroundings is stored as chemical energy.

Table 5. Examples of exothermic and endothermic processes

Exothermic processes	Endothermic processes
Making ice cubes	Melting ice cubes
Formation of snow in clouds	Conversion of frost to water vapor
Condensation of rain from water vapor	Evaporation of water
A candle flame	Forming a cation from an atom in the gas phase
Mixing sodium sulfite and bleach	Baking bread
Rusting iron	Cooking an egg
Burning sugar	Producing sugar by photosynthesis
Forming ion pairs	Separating ion pairs
Mixing water and strong acids	Mixing water and ammonium nitrate
Mixing water with an anhydrous salt	Making an anhydrous salt from a hydrate
Crystallizing liquid salts (as in sodium acetate in chemical hand warmers)	Melting solid salts
Nuclear fission	Reaction of barium hydroxide octahydrate crystals with dry ammonium chloride
Mixing water with calcium chloride	Reaction of thionyl chloride ($SOCl_2$) with cobalt(II) sulfate heptahydrate

Factors which influence the heat of reaction:

There are a number of factors which affect the magnitude of heat of reaction.

1) Physical state of reactants and products: Heat energy is involved for changing the physical state of a chemical substance. For example, in the conversion of water into steam, heat is absorbed and heat is evolved when steam is condensed. Considering the following two reactions:

$$H_{2(g)} + \frac{1}{2}O_{2(g)} = H_2O_{(g)};\ \Delta H = -57.8\ kcal$$

$$H_{2(g)} + \frac{1}{2}O_{2(g)} = H_2O_{(l)};\ \Delta H = -68.32\ kcal$$

It is observed that there is difference in the value of H if water is obtained in gaseous or liquid state. ΔH value in second case is higher because heat is evolved when steam condenses. Hence, physical sate always affects the heat of reaction.

2) Allotropic forms of the element: Heat energy is also involved when one allotropic form of an element is converted into another. Thus, the value of H depends on the allotropic form used in the reaction. For example, the value of H is different when carbon in the form of diamond or in amorphous form is used.

$$C_{(diamond)} + O_{2(g)} \to CO_{2(g)},\ \Delta H = -94.3\ kcal$$

$$C_{(amorphous)} + O_{2(g)} \to CO_{2(g)},\ \Delta H = -97.6\ kcal$$

The difference between the two values is equal to the heat absorbed when 12g of diamond is converted into 12g of amorphous carbon. This is termed as heat of transition.

3) Temperature: Heat of reaction has been found to depend upon the temperature at which reaction is occurring. The variation of the heat of reaction with temperature can be ascertained by using Kirchhoff's equation:

$$\Delta HT_2 - HT_1 / T_2 - T_1 = C_P$$

Kirchhoff's equation at constant volume may be given as,

$$ET_2 - ET_1 / T_2 - T_1 = C_v$$

Orientation process, or the possibility of chemical reaction is determined by the Gibbs energy. Gibbs energy or the isobaric - isothermal potential – is a manifestation of the cumulative effect of the enthalpy H and entropy S factors:

- At any temperature: $\Delta G_T = \Delta H - T\Delta S$
- At standard condition (298K): $\Delta G = \Sigma\ \Delta\ G$ products $-$ $\Sigma\ \Delta\ G$ reactants

If $\Delta G < 0$, then the reaction is possible.

If $\Delta G > 0$, then the reaction is impossible (is the reverse reaction).

If $\Delta G = 0$, then the system is in equilibrium.

Basic laws of Thermodynamics:

- **Zeroth law**: If any two systems are in thermal equilibrium with the third system, then they are also in thermal equilibrium with each other [7].
- **First law:** First law of thermodynamic states that energy can neither be created nor be destroyed but it can only be converted from one form to another [7].
- **Second law:** This law states that “whenever a spontaneous process takes place, it is accompanied by increase in a total energy of the universe” [7].
- **Third law:** This law states that “The entropy of a perfect crystal of each element and a compound is zero at absolute zero [7].

Instruments and reagents

Instruments: calorimeter, thermometer, stirrer, beaker, measuring cylinder, Beckmann thermometer, test tube with known salt, test tube with unknown salt.

Reagents: acids – HCl, HNO_3, H_2SO_4, base – NaOH, distilled water.

Experimental content

The following section gives an example of a laboratory determination of the enthalpy change for chemical reaction in which reactants are mixed in water. After the reaction has taken place the final mixture consists of products, unused reactants and water. The principles of such measurement are as follow.

The reaction is carried out in an insulated container which ideally prevents the reaction mixture from losing heat to (or gaining heat from) the surroundings.

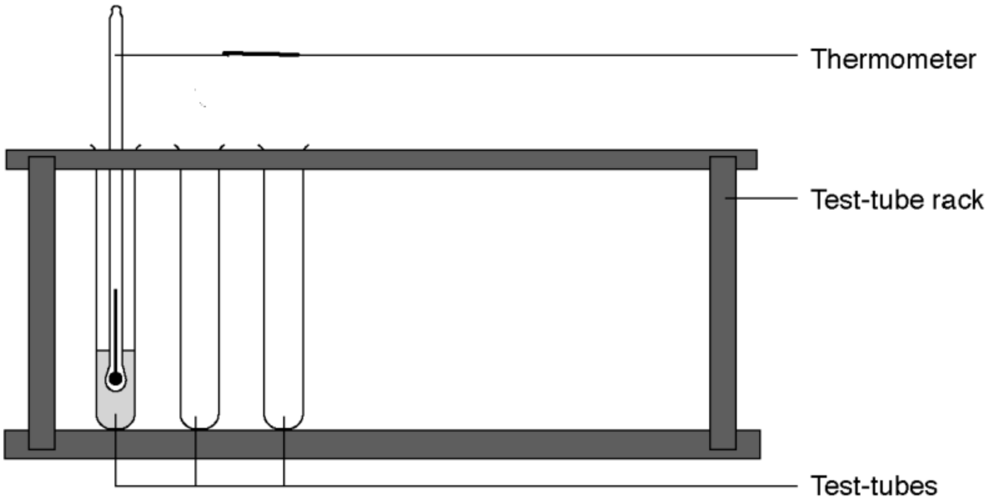
The chemical reaction should be rapid, so that the energy change complete in a short period of time. This is achieved by rapidly mixing the reactants.

An exothermic chemical reaction increases the amount of heat energy contained in the reaction mixture. This raises the temperature of the final mixture by ΔT^0C, defined as $T_{final} - T_{initial}$. An endothermic reaction decreases the amount of heat energy contained in the reaction mixture, so lowering the temperature of the final mixture.

The change in the amount of heat energy due to chemical reaction, Q joules, is calculated using the equation: $Q = m*C*\Delta T$ (note the negative sign) where m is mass and C (for pure water $4{,}184*10^3$ J/kg*^{0}C = 1 kal/g*^{0}C) the average specific heat capacity of the final mixture (mainly water) whose temperature is being measured. The value of Q is negative if the chemical reaction is exothermic (when ΔT is positive); the value of Q is positive if the chemical reaction is endothermic (when ΔT is negative).

At constant pressure, the heat change undergone produced by the chemical reaction is equal to the enthalpy change of the reaction, i.e. $Q = -\Delta H$. Note that Q and ΔH have the different sign.

1. To determine the thermal effect of the neutralization reaction, calculate the enthalpy change, please fill out the table and write thermochemical reaction equations: $aA + bB = cC + dD$ $\Delta H>0$ (endothermic reaction) or
$aA + bB = cC + dD$ $\Delta H<0$ (exothermic reaction)



1. Put 5 cm^3 of each acid in a test-tube.
2. Record the initial temperature of each acid in table.
3. Add and mix 5 cm^3 of alkali NaOH.
4. Watch what happens? How the temperature of acid is changed: increases or decreases?
5. Carefully stir, using the thermometer, and record the final temperature again.
6. Calculate the thermal effect of neutralization reaction by formula:

$Q = m*C* \Delta T$

7. All measurement results tabulated:

Volume of acid (5 ml)	Volume of base (5 ml)	Temperature		Δt, ^0C	Volume of mixture, ml	The heat effects of reaction, kJ/mol
		T_{final}	$T_{initial}$			
HCl	NaOH					
HNO_3	NaOH					
H_2SO_4	NaOH					

2. to determine the total heat capacity of the system (constant of the calorimetric system).

Weigh 5.00 grams of known salt (KCl or NH_4Cl) and pour into a clean, dry test tube. Then measure 250 ml of distilled water with a measuring cylinder and pour into a beaker. The beaker is placed in the calorimeter, fig. 4 (a, b).

Next, check the temperature. To do this, start using a stirrer to continuously and evenly mix the water in a beaker and fix the temperature every minute. After seven temperature measurements (the values should be almost the same), the substance is introduced into the calorimeter. Continue recording temperature every minute (at least 7 minutes).

After the end of the experiment, take out the Beckmann thermometer, test tube, stirrer and pour the solution out of the beaker. Wash the beaker and stirrer with distilled water.

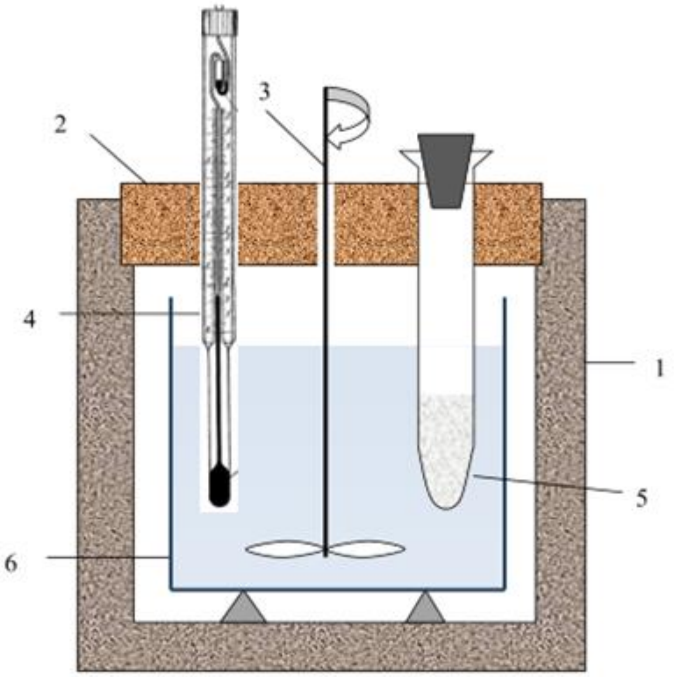


Figure 4. (a) Scheme of the calorimetric system:
1- casing; 2 - cover; 3 - stirrer; 4 - Beckmann thermometer;
5 - test-tube with salt; 6 - beaker.

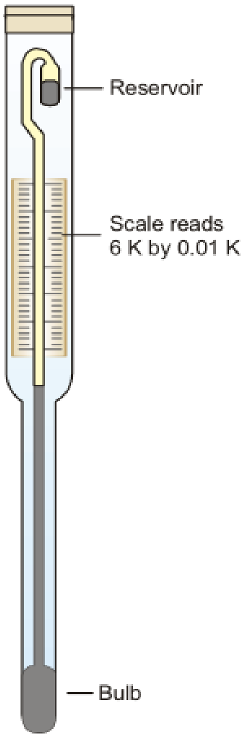


Figure 4. (b) Beckmann thermometer reading to 0.01 K

(It is differential thermometer. It is designed to measure small changes in temperature and not the temperature itself. It has a large bulb at the bottom of a finecapillary tube. The scale is calibrated from 0 to 6 K and subdivided into 0.01 K. The unique feature of this thermometer, however, is the small reservoir of mercury at the top. The amount of mercury in this reservoir can be decreased or increased by tapping the thermometer gently. In this way the thermometer is adjusted so that the level of mercury thread will rest at any desired point on the scale when the instrument is placed in the boiling (or freezing) solvent) [8].

The results of the experiment with known salt are recorded in the table 6.

Table 6. The results of the experiment with known salt

Experiment with H_2O		Experiment with known salt	
Salt____________	m (salt)________ m (H_2O)_______	Salt____________	m (salt)________ m (H_2O)_______
Time (minutes)	Thermometer readings, ^{0}C	Time (minutes)	Thermometer readings, ^{0}C
1		1	
2		2	
3		3	
4		4	
5		5	
6		6	
7		7	

Build a graph in coordinates: temperature - time and determine Δt when dissolving a known salt.

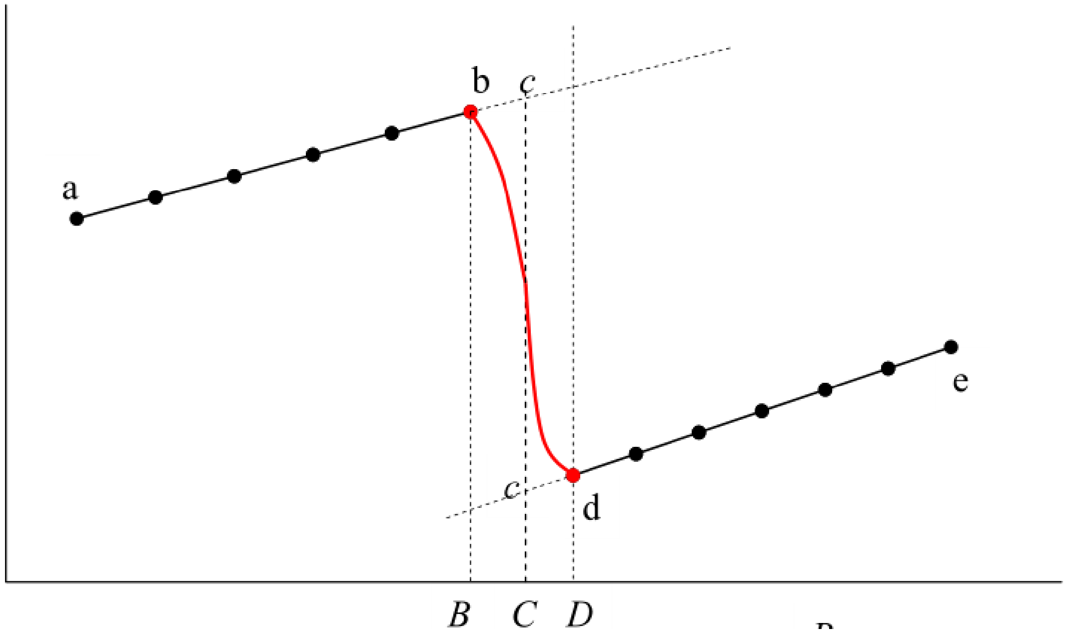


Figure 5. Graphical definition of temperature change Δt.
(Time BD divided in half, the perpendicular is drawn from point C, the perpendicular segment gives the change in temperature Δt).

The calculation of the value Q(KCl) is carried out as follows:

(1) Finding the number of moles KCl:

$$n(KCl)=m(KCl)/M(KCl),$$

where m(g) is mass KCl, M(g/mol) is molar mass KCl;

(2) Calculate the molar concentration of salt in a solution:

$$C_m(KCl)= n(KCl)/V(H_2O),$$

where $V(H_2O)$ is the volume of distilled water;

(3) By value $C_m(KCl)$ we find $\Delta H_m(KCl)$ in the table 7.

Table 7. Integral heat of dissolution of salts in water

$C_m(KCl)$, mol/kg(H_2O)	$\Delta H_m(KCl)$, kJ/mol	$\Delta H_m(NaCl)$, kJ/mol	$\Delta H_m(NH_4Cl)$, kJ/mol
0,0	17,23	3,89	14,73
0,01	17,39	4,06	14,85
0,02	17,44	4,10	14,94
0,05	17,51	4,18	15,02
0,1	17,55	4,25	15,10
0,2	17,57	4,27	15,19
0,3	17,55	4,25	15,23
0,4	17,50	4,16	15,27
0,5	17,43	4,10	15,27
1,0	17,28	3,79	15,31
2,0	16,72	3,18	15,27
3,0	16,17	2,66	15,23
4,0	15,75	2,26	15,19

(4) We calculate the value Q(KCl), J:

$$Q(KCl)= n(KCl)*\Delta H_m(KCl);$$

(5) Based on the experimental data and the definition $\Delta t(KCl)$, we calculate constant of the calorimetric system:

$$K=Q(KCl)/\Delta t(KCl)$$

3. to determine the integral heat of solution of an unknown salt.

To determine the heat of dissolution of an unknown salt, an experiment is carried out using an identical method. All readings of the Beckmann thermometer are placed in the table 8.

Table 8. The results of the experiment with unknown salt

Experiment with H_2O		Experiment with unknown salt	
Salt___________	m (salt)________ m (H_2O)_______	Salt___________	m (salt)________ m (H_2O)_______
Time (minutes)	Thermometer readings, ^{0}C	Time (minutes)	Thermometer readings, ^{0}C
1		1	
2		2	
3		3	
4		4	
5		5	
6		6	
7		7	

According to the data obtained, a graph is built and Δt_x is determined when an unknown salt is dissolved.

(1) Calculate the heat of solution of an unknown salt:

$$Q_x=K*\Delta t_x$$

(2) We calculate the specific heat of dissolution of an unknown salt:

$$q_x= Q_x/m_x,$$

where m(g) is mass unknown salt;

(3) We calculate the integral heat of dissolution of this salt:

$\Delta H_{mx}=q_x*M_x$, where M(g/mol) is molar mass salt.

Questions

1.What are the stages of the salt dissolution process? What are the thermal effects of these stages?

2. What elements does the colorimetric system consist of?
3. What is called integral and differential heat of dissolution?
4. What is the purpose of mixing during the experiment?
5. Give a name for the reaction when silver chloride (AgCl) turns grey in sunlight: $2AgCl(s) = 2Ag(s) + Cl_2$

Experiment 2. Determination of heat of combustion

Experimental purpose

1. Master the use of oxygen bomb calorimeter to measure the combustion heat of naphthalene.
2. Consolidate the relationship between constant pressure combustion heat and constant volume combustion heat.
3. Learn to use graphic methods to correct temperature changes.

Experimental principles

The heat of combustion refers to the thermal effect of the complete oxidation reaction between 1 mol of substance and oxygen. If the fuel is measured under constant volume conditions, the heat of combustion is called constant volume combustion heat, with the symbol $Q_{V,m}$ means. According to the first law of thermodynamics, the internal energy of the system changes during combustion. Transformation, internal energy change. If the system does no external work, the constant volume combustion heat is equal to the change in the internal energy of the system, that is

$$Q_{V,\mathrm{m}} = \Delta_V U_{\mathrm{m}}$$

In the same way, if the combustion heat measured under constant pressure conditions is called constant pressure combustion heat, use the symbol $Q_{p,m}$ (or $\Delta_p H_m$) table show. If the gas participating in the reaction and the gas generated by the reaction are approximated as ideal gases, then there is the following relationship:

$$Q_{p,\mathrm{m}} = Q_{V,\mathrm{m}} + \sum \nu_{\mathrm{B}}(\mathrm{g})RT$$

In the formula, $\sum \nu_B$ (g) is the algebraic sum of the stoichiometric coefficients of the gaseous substances in the products and reactants in the molar reaction formula; R for Universal gas constant; T is the thermodynamic mean temperature of the reaction.

It can be seen from the above formula that if the constant volume combustion heat of a certain substance is measured, then the heat of combustion at constant pressure can be obtained.

It is worth pointing out that, without special it is pointed out that the heat of combustion usually refers to the heat of combustion at constant pressure.

An instrument that measures the heat of chemical reaction is called a calorimeter. This experiment uses measure the constant volume combustion of naphthalene using an oxygen bomb calorimeter (shown in Figure 6) heat, and then obtain the constant pressure combustion heat of naphthalene.

The basic principle of measuring constant volume combustion heat is to combine a certain object to be measured when a mass sample is completely burned in an oxygen bomb with sufficient oxygen, the heat released the temperature of the calorimeter itself and the medium around the oxygen bomb (water for this experiment) degree increased. According to the measured temperature change value before and after combustion, it can be calculated find the constant volume heat of combustion of the sample. Its relationship is:

$$m \mid Q_{V,m} \mid / M + \mid Q_{wire} \mid m_{wire} = W_{water} \Delta T$$

In the formula, m is the mass of the substance to be measured, g; M is the substance to be measured molar mass; $Q_{V,m}$ is the constant volume combustion heat of the substance to be measured; Q_{wire} is heat of combustion per unit mass of ignition wire (-1.4 kJ $\cdot g^{-1}$); m_{wire} is a point the mass of the fire wire; W_{water} is the heat required by the combustion of substances such as the sample to increase the water and the instrument by 1°C, which is called the water equivalent.

The water equivalent of the calorimeter can be determined by a standard substance with known constant volume combustion heat (such as benzoic acid, whose Q_V = -26.0460 kJ$\cdot g^{-1}$) to calibrate. After the water equivalent of the calorimeter is known, the formula to determine the combustion heat of the substance to be tested through experiments.

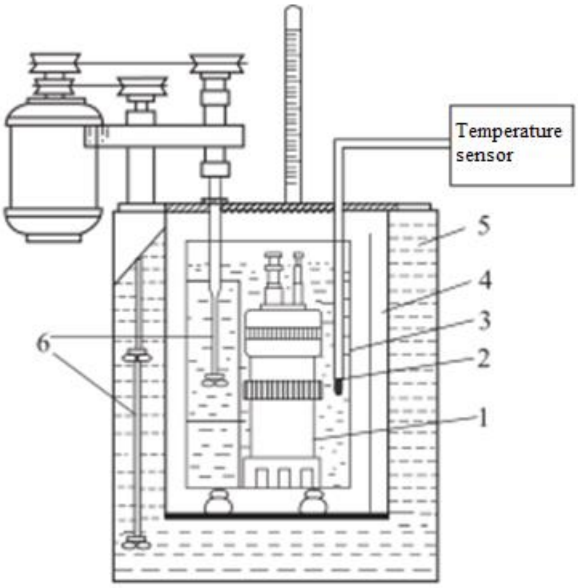


Figure 6. Schematic diagram of oxygen bomb calorimeter
1 — Oxygen bomb; 2 — Temperature sensor; 3 — Inner cylinder; 4—Air barrier; 5—Outer cylinder; 6—Agitator

The oxygen bomb is a special stainless steel container, as shown in Figure 7. The main parts include thick-walled cylinder 1, spring cover 2 and nut 3 are closely connected; the elastic cover 2 is equipped with an air inlet 4 for injecting oxygen, an exhaust hole 5 and an electrode 6, and the electrodes pass directly through the elastic body internally, it also serves as a support for the burning dish 7; in order to reflect the flame downward and make the temperature of the projectile uniform, another electrode 8 (also the air intake pipe) is also equipped with a flame shield 9.

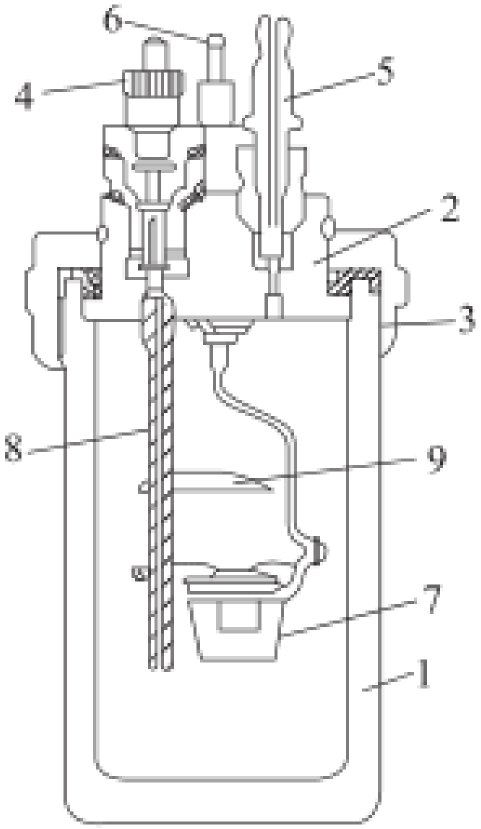


Figure 7. Structure of oxygen bomb:
1—Thick-walled cylinder; 2—Elastic cover; 3—Screw Cap; 4 — air inlet hole;
5 — exhaust hole; 6 —Electrode; 7 —Burning dish;
8 —Electrode (also the Trachea); 9 — Flame shield

In order to ensure that the sample is completely burned in it, the oxygen bomb should be filled with high-pressure oxygen. This requires oxygen bomb sealing, high pressure resistance, and corrosion resistance. When measuring powder samples, the sample must be press into flakes to prevent the sample from being blown apart when inflated or flying apart during combustion, causing solid test error.

The first key to the success of this experiment is that the sample must burn completely. Secondly, it must also be as much as possible, the heat released after combustion is transferred to the calorimeter itself and the content contained in it water and almost no heat exchange with the surrounding environment.

In order to achieve this, several measures have been taken in the design and manufacture of calorimeters, such as for example, if a shell is installed outside the calorimeter, some of the shells are constant temperature and some are insulated thermal, so calorimeters can be divided into two types: shell constant temperature type and adiabatic type.

This experiment adopts use shell thermostatic type. In addition, the calorimeter walls are highly polished to reduce thermal radiation. A barrier is installed between the calorimeter and the housing to reduce air convection. However, the heat the dissipation cannot be completely avoided, which may be due to the radiation heat from the ambient vector calorimeter.

The temperature rises due to the amount of heat, or it may be due to the heat radiated by the calorimeter to the environment. The temperature of the calorimeter decreases. Therefore, the temperature changes before and after combustion cannot be directly measured accurately is correct, it must be corrected through Reynolds diagram method (drawing method). The correction method as follows when an appropriate amount of the substance to be measured is burned, the water temperature in the calorimeter increases by 1.5~2.0℃.

The observed values before and after combustion will be record the water temperature and draw a graph to form *a b c d* line (as shown in Figure 8).

Point *b* in the figure is equivalent to the point where combustion begins, c the point is the highest temperature reading point observed. Due to the heat exchange between the calorimeter and the outside world, the curves *a b* and *c d* often tilt. incline. Suppose the temperature corresponding to point *b* is T_1 and the temperature corresponding to point *c* is T_2, take their average temperature $(T_1+T_2)/2$ as T , draw a parallel line TO with the abscissa through point T, intersect with the curve at point O, and then draw a vertical line AB through point O. This line is the extension of line *ab* and line *cd* intersects at two points E and F, then the temperature difference represented by point E and point F is the increase in the desired temperature value ΔT. As shown in Figure 8, EE ' represents the increase in the calorimeter temperature caused by the heat radiated from the environment. This part is must be deducted; and FF' represents the decrease in the temperature of the calorimeter caused by the heat radiated by the calorimeter to the environment, so this part It is a must to join. The temperature difference thus corrected represents the increase in the calorimeter temperature due to the burning of the sample.

Sometimes the thermal insulation of the calorimeter is good, the heat loss is small, and the power of the stirrer is relatively large, which often causes continuous A small amount of heat is added so that the highest temperature point after combustion is not obvious. In this case, ΔT can still be carried out according to the same method correction (as shown in Figure 9).

Figure 8 Temperature correction chart when insulation is poor

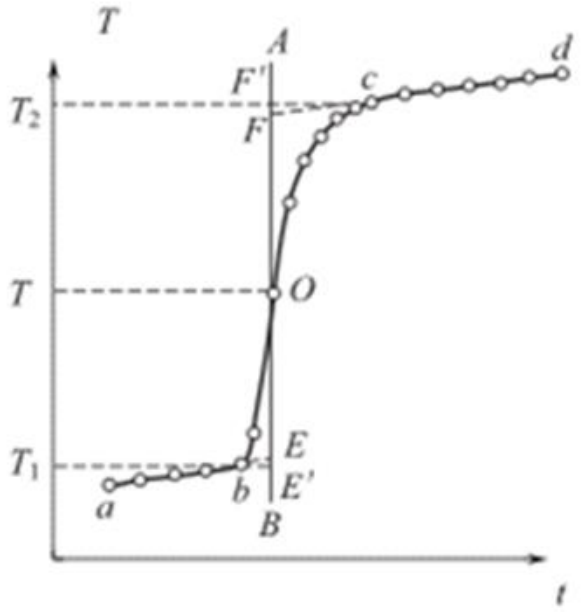


Figure 9. Temperature correction chart when insulation is good

It must be noted that when applying this drawing method for correction, the temperature of the calorimeter and the temperature of the external environment should not differ too much. (It is best not to exceed 2~3℃), otherwise errors will occur. Of course, in the process of measuring the heat of combustion, the accuracy of the calorimeter temperature measurement directly affects the results of the heat of combustion measurement.

Therefore, this experiment uses a digital temperature difference measuring instrument to measure the temperature change value of the calorimeter.

Instruments and reagents

Instruments:1 oxygen bomb calorimeter, 1 tablet press, 1 electronic balance, 1 thermometer (0~100℃), volumetric flask (1000 mL), 1 oxygen cylinder and 1 set of pressure reducing valve (for public use).

Reagents: Ignition wire, naphthalene (A.R.), benzoic acid (A.R.).

Experimental content

1. **Determination of calorimeter water equivalent (W water)**

(1) Sample tableting.

Before tableting, carefully check that the steel mold used for tableting must be clean before tableting can be carried out.

Use a bench scale to weigh about 0.6 g of benzoic acid, and use an electronic balance to accurately weigh the mass of a section of ignition wire (about 15 cm), as shown in the figure 10. Thread the iron wire into the bottom plate of the steel mold, then put the steel mold bottom plate into the mold, and pour the weighed material from above.

Take the benzoic acid sample and slowly tighten the screw of the tablet press until the sample is pressed into a tablet. Remove the supporting plate from the bottom of the mold before continuing press down to make the bottom of the mold and the sample fall off together. The shape of the pressed sample is shown in Figure 10, the debris on the surface of this sample after being accurately weighed on an analytical balance, it can be used to measure the heat of combustion.

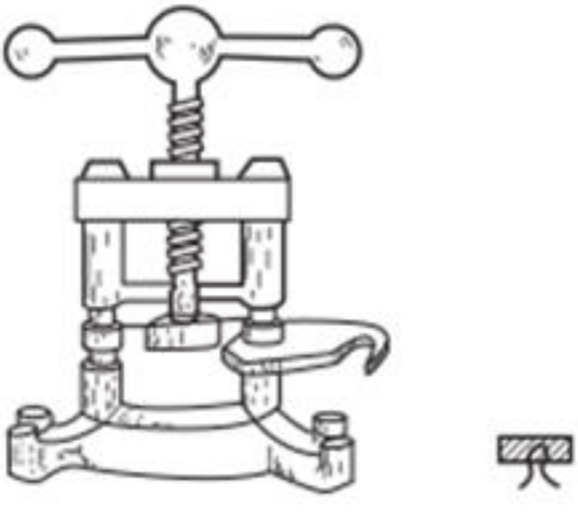


Figure 10. Schematic view of tablet press and tableting process

Figure 11. Oxygen bomb inflation diagram

(2) Install the oxygen bomb.

Unscrew the oxygen bomb cover and clean the inner wall of the oxygen bomb. Especially the stainless steel terminal at the lower end of the electrode should be cleaned wipe it clean. Hang a small quartz ceramic or metal cup, and carefully wrap both ends of the ignition wire of the pressed sheet sample tightly around the electrode. At the lower end, check that the two electrodes cannot be short-circuited. Tighten the oxygen bomb cover and tighten the oxygen pop-up port to fill with oxygen.

According to Figure 11connect the oxygen cylinder and oxygen meter, and connect the high-voltage copper wire tube to the oxygen bomb inlet pipe. Open the oxygen outlet at the top of the oxygen cylinder valve, at this time the pressure indicated in Table 1 is the oxygen pressure in the oxygen cylinder. Then tighten the pressure reducing valve slightly (i.e. open the pressure reducing valve to port), and at the same time open the vent pipe of the oxygen bomb to remove the air in the oxygen bomb. After the air in the oxygen bomb is removed, tighten the oxygen bomb immediately led the air pipe, and then continue to tighten the pressure reducing valve until the pressure reading on Table 2 is about 1.5 MPa. Loosen (i.e. close) the pressure reducing valve, valve, at this time the oxygen bomb is filled with approximately 1.5 MPa oxygen.

Remove the oxygen bomb, close the valve at the upper end of the oxygen cylinder, and open the pressure reducing valve bleed the remaining air from the pipes and oxygen meter.

(3) Combustion and temperature measurement:

Put the oxygen bomb filled with oxygen into the water bucket of the calorimeter (as shown in Figure 6), and then good ignition wire. Use a volumetric flask to accurately measure 2500 mL of tap water and pour it into a bucket. Close the lid and place the thermometer insert the temperature sensor probe into the water, then start the stirring motor. After the temperature rises steadily, the temperature difference meter is cleared. Every other read the digital display of the thermometer once in 1 min and continue for 10 min. Quickly press the ignition switch to ignite the power.

If the indicator light on the igniter lights up and then goes out, and the temperature rises rapidly, it means that the sample in the oxygen bomb has burned. If the indicator light does not turn on after if it goes out, it means that the ignition wire has not burned out, and the current should be increased immediately to cause combustion. If the indicator light does not light up at all or even if the current is increased, the if it does not go out and the temperature does not rise rapidly, it means that the ignition was not successful.

At this time, the oxygen bomb needs to be opened to check the cause. Self-press after the ignition switch is turned off, the reading changes to once every 30 seconds, and the temperature rises rapidly within about 1 minute. When the temperature rises to the highest point, Readings can still be taken once every 1 min, and the temperature can continue to be recorded for 10 min.

After the experiment is stopped, carefully remove the temperature sensor probe, take out the oxygen bomb, open the oxygen pop-up vent, release the remaining gas, and finally unscrew the oxygen bomb cover and check the combustion results of the sample. If there are no unburned residues in the oxygen bomb, it means the combustion is complete, otherwise, it means the combustion is incomplete and the experiment failed.

The remaining ignition wire after burning should be taken out and weighed, and the mass of the ignition wire should be calculated minus.

2. **Determination of combustion heat of naphthalene**

Weigh a portion of about 0.6 g of naphthalene, and perform experimental operations such as tableting and burning according to the above method.

After the experiment is completed, clean the oxygen bomb, pour out the tap water from the calorimeter bucket, and dry it for the next experiment.

Data recording and processing

1. Calculate the change in calorimeter temperature caused by the combustion of benzoic acid according to the graphing method. Calculate the water equivalent (W_{water}) of the calorimeter.
2. Calculate the temperature change of the calorimeter caused by the burning of naphthalene according to the graphing method, and calculate the constant volume combustion heat of naphthalene ($Q_{V,m}$).
3. Calculate the constant pressure combustion heat ($Q_{p,\ m}$) of naphthalene from the constant volume combustion heat ($Q_{V,m}$) of naphthalene.
4. Find the constant-pressure combustion heat ($Q_{p,m}$) of naphthalene from the physical and chemical data manual, and calculate the error of this experiment.

Questions

1. How to divide the system and environment in this experiment?
2. What is the relationship between the constant volume thermal effect and the constant pressure thermal effect?
3. What issues should be paid attention to when using oxygen cylinders?
4. Why do the temperature differences measured experimentally need to be corrected by the Reynolds plot method (drawing method)?

Experimental guidance

1. Be familiar with the use of the thermometer to avoid operating errors during the experiment.
2. There are two keys to the success of the experiment: first, whether the combustion is complete, for which sufficient pressure of oxygen needs to be filled; second, whether the combustion is complete the two ends of the iron wire need to be firmly connected to the two electrodes, and please be careful not to allow the iron wire and the wall of the crucible containing the medicine tablets to prevent damage short circuit.
3. The ignition current should be slightly larger to ensure a successful ignition.
4. The heat loss correction chart can also be made by another method. Select the ambient temperature T ring and make a parallel intersection parallel to the time axis. The line is at point O and the perpendicular line A B is drawn through point O. The other processing methods are the same.

Experiment 3. Determination of surface tension of solution by maximum bubble method

Experimental purpose

1. To understand the nature of surface tension, the significance of surface free energy and the relationship between surface tension and adsorption.
2. Master the principle and technology of measuring the surface tension of solution by maximum bubble method.
3. Measure the surface tension of ethanol solution with different concentrations by maximum bubble method, and understand the factors affecting the measurement of surface tension.
4. The adsorption capacity of ethanol solution at different concentrations was calculated by Gibbs formula. Calculation of molecules from experimental data of surface tension, the cross-sectional area and the thickness of adsorption layer.

Experimental principles

This property of liquids arises from the intermolecular forces of attraction. A molecule in the interior of a liquid is attracted equally in all directions by the molecules around it. A molecule in the surface of a liquid is attracted only sideways and toward the interior. The forces on the sides being counterbalanced the surface molecule is pulled only inward the liquid. Thus there is a tendency on the part of the surface molecules to go into the bulk of the liquid [9].

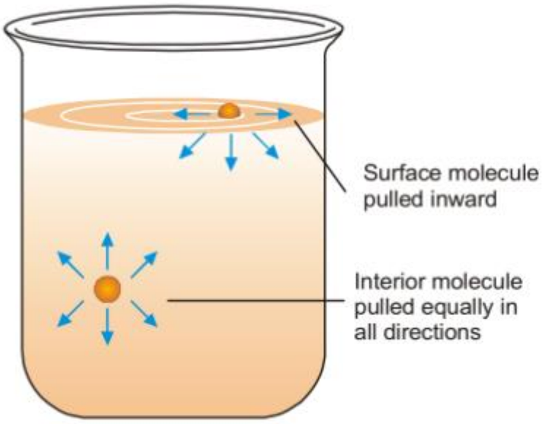


Figure 12. Surface tension is caused
by the net inward pull on the surface molecules

The inward forces on the surface molecules minimize the surface area and form a drop [9].

The liquid surface is, therefore, under tension and tends to contract to the smallest possible area in order to have the minimum number of molecules at the surface. It is for this reason that in air, drops of a liquid assume spherical shapes because for a given volume a sphere has the minimum surface area [9].

The surface tension (γ) is defined as: the force in dynes acting along the surface of a liquid at right angle to any line 1 cm in length [9].

As included in the above definition the unit of surface tension in CGS system is dynes per centimeter (dyne/cm). In SI system, the unit is Newton per meter (N/m) [9].

A change in temperature causes a change in surface tension of a liquid. When temperature increases, there is an increase in kinetic energy of liquid molecules (KE $\propto$ T), thereby decreasing intermolecular forces. It results in decrease in the inward pull functioning on the surface of the liquid. In other words, surface tension decreases with increase in temperature [9].

Table 9. Surface tension of some liquids at various temperature (dynes/cm)

Liquid	20^0C	40^0C	60^0C	80^0C
Water	72,75	69,56	66,18	62,61
Ethyl alcohol	22,27	20,60	19,01	-
Methyl alcohol	22,6	20,9	-	-
Acetone	23,7	21,2	18,6	16,2
Toluene	28,43	26,13	23,81	21,53
Benzene	28,9	26,3	23,7	21,3

The forces on the surface layer molecules and the internal (bulk) molecules of liquid are different. Internal molecules are acted on by surrounding liquid molecules. The forces cancel each other out and the resultant force is zero. The surface layer molecules are attracted by the liquid and gas phase molecules, and the resultant force points to the inside of the liquid to move the molecules inside the liquid to the surface layer (to increase the surface area of the liquid), it is necessary to overcome this attraction and do work.

When the surface area of liquid is reversibly increased at constant temperature and pressure, the free energy changes to:

$$dG_{T,p} = \delta W'_R = \gamma dA$$

$$\left(\frac{\partial G}{\partial A}\right)_{T,p} = \gamma$$

γ is the reversible work required to increase the unit surface area, that is, the number of surface layer molecules per unit area is the same.

The extra free energy of the internal molecules is called the specific surface free energy, J·m^{-2}. Because the surface layer molecules are attracted by the internal molecules, the surface layer molecules have a tendency to automatically enter the liquid macroscopically, there is a force parallel to the surface, trying to shrink the surface. When the physical meaning of γ is understood from this angle, γ is also called surface tension, and its unit is N·m^{-1}. The magnitude of surface tension γ is related to the nature of liquid, coexisting gas phase, temperature and stress related. When the temperature rises, the surface tension decreases, and when it reaches the critical state, the surface tension tends to zero.

A solution to form if a solute is added to a liquid, the surface tension of the solvent will increase or decrease. If solute is added to make the solution.

When the surface tension of the solution decreases, the concentration of solute in the surface layer is greater than that of the solution body;

On the contrary, if the surface tension of the solution is made the concentration of solute in the surface layer is less than that of the solution body. The concentration will difference between the surface layer and the solution body. When these two opposite trends reach equilibrium, the composition of the solution surface layer and the solution body is not again, this phenomenon is called surface adsorption of solution.

At a certain temperature and pressure, the surface adsorption capacity of the solution is related to the surface tension and concentration of the solution dilute to two components solution. The relationship between them can be expressed by Gibbs adsorption isotherm formula:

$$\Gamma = -\frac{c}{RT} \times \frac{\partial \gamma}{(\partial c)_T}$$

Γ is the surface adsorption amount, mol m^{-2}; γ is the surface tension, N·m^{-1}; C is the concentration of solution, mol L^{-1} ; T is thermodynamic temperature, K; R is the gas constant.

If, $\Gamma>0$, it is called positive adsorption.

$$\left(\frac{\partial \gamma}{\partial c}\right)_T < 0 \text{,}$$

If, $\Gamma<0$, it is called negative adsorption.

$$\left(\frac{\partial \gamma}{\partial c}\right)_T > 0 \text{,}$$

The isothermal curve γ = of surface tension and concentration of solution is measured F(c), the surface adsorption capacity of the solution at various concentrations can be obtained by the slope of the curve.

For single molecule adsorption, the relationship between adsorption amount Γ and concentration c can be isothermally absorbed by Langmuir

It is expressed by equation, that is,

$$\Gamma = \Gamma_\infty \frac{ac}{1+ac}$$

Γ_∞ is the saturated adsorption capacity, and a is a constant.

The reciprocal of both sides of equation can be sorted into a linear equation:

$$\frac{c}{\Gamma} = \frac{c}{\Gamma_\infty} + \frac{1}{a\Gamma_\infty}$$

When c/Γ versus c is plotted as a straight line, the slope $m = 1/\Gamma_\infty$, if N is used to represent the surface layer per unit area of saturated adsorption.

Then

$$N = \Gamma_\infty N_A,$$

where N_A is the Avogadro constant.

In saturated adsorption, each adsorbed fraction the area occupied by the molecule on the surface, that is, the cross-sectional area S of the molecule is:

$$S_0 = \frac{1}{\Gamma_\infty N_A}$$

If the density ρ and molar mass M of solute are known, the thickness of adsorption layer, that is, the length δ of molecules, can be calculated, then

$$\delta = \frac{\Gamma_\infty M}{\rho}$$

Many methods exist for determining surface tension: capillary-rise method, drop formation method, ring-detachment method, maximum bubble pressure method.

In this experiment, the maximum bubble method is used to measure the surface tension of liquid. In this method air-pressure is applied slowly through a capillary tube dipping in the experimental liquid (Fig. 13). A bubble is formed at the end of the capillary. Slowly the bubble grows and becomes hemispherical. Then it breaks away when the pressure recorded by the manometer is noted. This is the maximum pressure required to make a bubble at the end of the capillary [9].

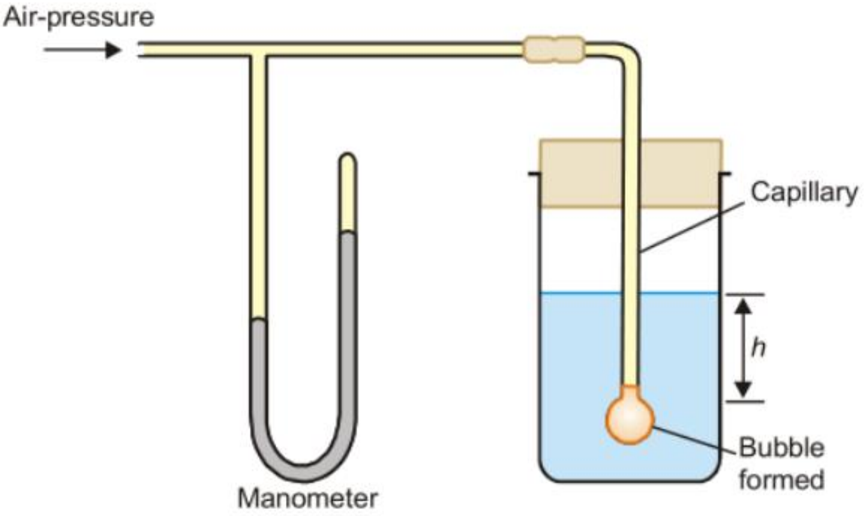


Figure 13. A simple apparatus for maximum bubble pressure method

At the moment of breaking, the forces due to maximum pressure P equals that of the opposing hydrostatic pressure P_h and the surface tension γ at the circumference of the capillary [9]. Thus,

$$P \pi r^2 = P_h \pi r^2 + 2 \pi r \gamma$$

$$P = P_h + \frac{2\gamma}{r}$$

$$P = h d g + \frac{2\gamma}{r}$$

where r = radius of capillary; d = density of the liquid; h = depth of liquid. Knowing the value of P, h, d and r, γ can be found [9].

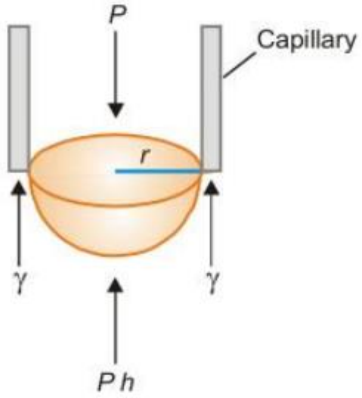


Figure 14. Applied pressure on bubble is opposed by hydrostatic pressure and surface tension

Its measuring device is as shown in the figure 15.

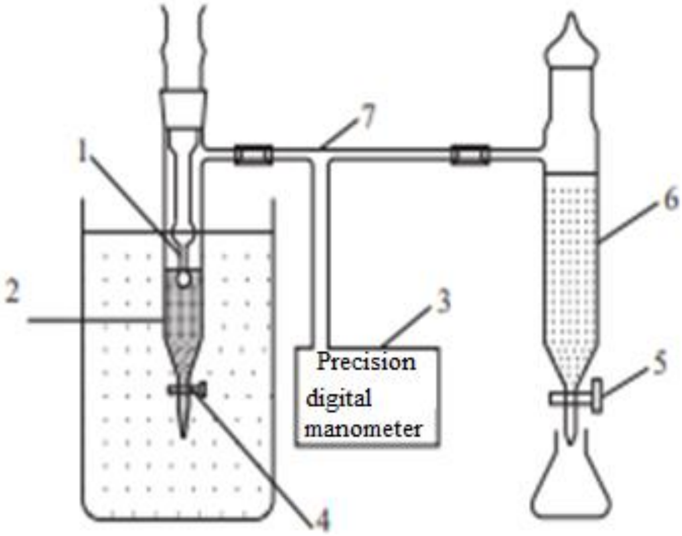


Figure 15.A apparatus for maximum bubble pressure method
1 - Glass capillary; 2 - Sample tube; 3 - Precision digital manometer;
4 - Sample tube piston; 5 - Extraction cylinder piston;
6 - Extraction cylinder; 7 - T tube

The liquid whose surface tension is to be measured is put into the sample tube 2, and the liquid level is tangent to the end face of the capillary tube 1 through piston 4, so that the liquid level rises along the capillary tube, and the piston 5 is opened to slowly decompress the system. At this time, because the pressure above the liquid level in the capillary tube (i.e., the external pressure) is greater than the pressure of the liquid level in the sample tube, the liquid level in the capillary tube gradually drops, and bubbles escape when the liquid level reaches the nozzle out. Blowing air bubbles from the capillary end under the immersed liquid surface requires additional pressure higher than the external atmospheric pressure to overcome the bubbles.

This additional pressure is proportional to the surface tension and inversely proportional to the radius of curvature of the bubble, i.e. $\Delta p = 2\gamma/R$. If the capillary radius is small, the bubbles formed are basically spherical. When bubbles begin to form, the surface is almost flat, and the radius of curvature is the largest; With the formation of bubbles, the radius of curvature gradually decreases until it forms a hemispherical shape, when the radius of curvature r = capillary radius r, the radius of curvature reaches the minimum, and according to Laplace equation, the additional pressure reaches the maximum. Bubble into one as the step grows, R increases and the additional pressure decreases until the bubble escapes.

At this time, the pressure difference delta p of the pressure gauge 3 is the additional pressure of the liquid to be measured in the capillary, that is,

$$\Delta p = \frac{2\gamma}{R'}$$

R' is the radius of curvature of the bubble.

Because the capillary radius is very small, the bubble formed is basically spherical. When the bubble begins to form, the surface is almost flat, and the radius of curvature r ' is the largest (as shown in Figure 16). When the bubble forms a hemisphere, r' is equal to the capillary radius r, and the radius of curvature reaches the minimum, and Δp is the maximum. With the further enlargement of bubbles, R' tends to increase again until it escapes from the liquid level.

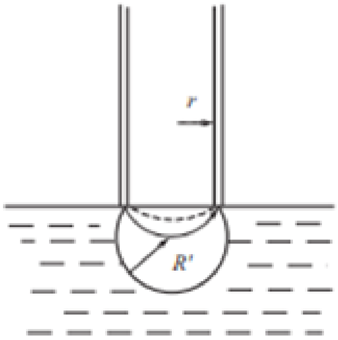


Figure 16. The bubble begins to form

During this process, the maximum additional pressure Δp_{max} is expressed as:

$$\Delta p_{max} = \frac{2\gamma}{r}$$

$$\gamma = \frac{r}{2} \Delta p_{max}$$

$$\gamma = K \Delta p_{max}$$

K can be determined by reference materials with known surface tension (for example, distilled water at 25 ^{0}C $\gamma = 71.97$ mN · m^{-1}).

In this experiment, Δp_{max} was measured with a precision digital pressure gauge, γ was calculated from equation (8), and the surface adsorption amount was calculated according to Gibbs adsorption isotherm:

$$\Gamma = -\left(\frac{c}{RT}\right)\frac{d\gamma}{dc}$$

If the curve γ = f (c) (surface tension isotherm) is drawn at constant temperature, when c increases, γ decreases significantly at first, and then gradually slows down, so that γ changes very little, and then the value of γ is constant at a certain constant. It is very convenient to use graphic method for calculation, as shown in Figure 17, passing through tangent point an as a straight line parallel to abscissa, and intersecting ordinate at point b '.

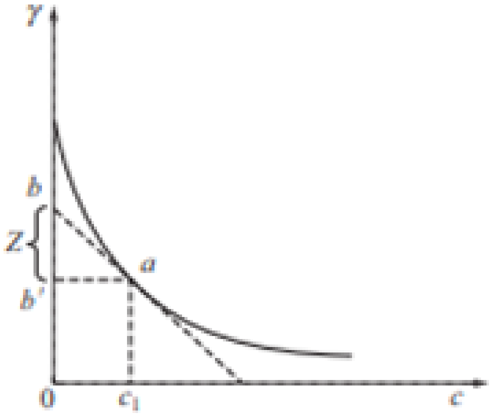


Figure 17. The graphic method for calculation

Z is the distance between the intercepts of tangents and parallel lines on the ordinate. Obviously, the length of Z is equal to $c(d\gamma/dc)_T$.

For different concentrations, a curve can be made for the corresponding Γ, and Γ = f (c) is called adsorption isotherm.

$$\left(\frac{d\gamma}{dc}\right)_T = -\frac{Z}{c}$$

$$Z = -\left(\frac{d\gamma}{dc}\right)_T c$$

$$\Gamma = -\frac{c}{RT}\left(\frac{d\gamma}{dc}\right)_T = \frac{Z}{RT}$$

Instruments and reagents

Instruments: One set of maximum bubble pressure surface tensiometer, one ear washing ball, one pipette (50mL), one pipette (5mL), one dropping funnel and one beaker (500mL).

Reagents: Ethanol, distilled water.

Experimental content

1. Instrument preparation and leak detection

Wash and dry the surface tensiometer container and capillary tube. At constant temperature, 50mL of distilled water was injected into the washed sample tube, and the open capillary tube was inserted into the sample tube so that its tip was just in contact with the liquid surface. Open the piston of the pump cylinder to reduce the pressure in the system. When the precision digital differential pressure gauge displays a certain number, close the piston of the pump cylinder. If the pressure difference of the precision digital differential pressure gauge remains unchanged within 2 ~ 3min, it means that the system does not leak air and can be tested.

Figure 18. General view of the installation

2. Measurement of instrument constants

Open the piston of the pump cylinder and make the water in the dripping funnel drip out slowly. The velocity of water flow in the dropping funnel is controlled so that bubbles escape from the tip of capillary tube into single bubbles, and the velocity of bubbles escaping from capillary tube is 8~10s for each bubble. When the bubble is just out of the tube end, when the precision digital differential pressure gauge shows the maximum pressure difference, record the maximum pressure difference, read it three times continuously, and take its average value. When the experimental temperature is found out from the manual, the surface tension γ of water, then the instrument constant K is:

$$K = \frac{\gamma_{w}}{\Delta p_{\max}}$$

3. Determination of surface tension with solution concentration

In the above system, 3.00 mL of ethanol is transferred into a pipette, and air is pumped several times with an ear washing ball (pay attention to when pumping, be sure to make the system becomes an open system), so that the concentration of the solution is uniform, and then the liquid level is tangent to the capillary end.

Methods the maximum pressure difference of precision digital pressure difference gauge was measured. Then 3.00 mL, 3.00 mL, 4.00 mL, 4.00 mL, 3.00 mL, 3.00 mL ethanol, and the pressure difference Δp_{max} was measured every time. The amount of ethanol is added until it is full and, at this time, the maximum Δ p of the pressure gauge hardly changes with the addition of ethanol. Different solutions must be determined from low concentration to high concentration were measured in turn.

4. After the experiment, clean the glass instrument and arrange the test bench.

Data Recording and Processing

Experimental temperature:

Δp_{max} of water:

the first time___; the second time___; the third time____; Average value______; K = _______________________________

C_2H_5OH		1	2	3	4	5	6	7
Δp_{max}	1							
	2							
	3							
	average							
γ, N/m								

(1) Calculate the instrument constant K, calculate the γ of ethanol aqueous solution with various concentrations, and make a table.
(2) Make Γ - c diagram, take 6 ~ 7 points on the smooth curve, use mirror image as tangent to get Z value, and then use the formula $\Gamma = -(c/RT)d\gamma/dc$ to calculate Γ value of different concentrations, and make Γ-c diagram.
(3) Calculate c/Γ. Drawing c/Γ - c isotherms, finding Γ_∞ and calculating S_0 and δ.

Questions

1. Why must the capillary tip be adjusted just tangent to the liquid surface? Otherwise, what is the effect on the experiment?
2. Why should we read the maximum pressure difference when measuring surface tension by the maximum bubble method? If bubbles escape quickly, or several bubbles come out together, will it affect the experimental results?
3. What is the influence of the radius of capillary tip selected in this experiment on the experimental determination? If the capillary is not clean, will it affect the measurement results?

Experimental Guidance

1.Ensure that the measuring system is a closed system and cannot leak air.
2. After the sample is injected into the sample tube, it should be kept at a constant temperature for about 10min.
3. The capillary must be clean, and care should be taken not to break the capillary when washing.
4. Pay attention to adjusting the piston at the lower end of the sample tube so that the capillary orifice must be tangent to the liquid surface in the sample tube. When reading the pressure difference of precision digital pressure difference gauge, the maximum pressure difference when bubbles escape individually should be taken.
5. When injecting water or solution into the sample tube, avoid liquid flowing into the hose communicated with the pressure gauge.
6. When determining ethanol solution, the order of determination of solution concentration must be from dilute to concentrated.
7. Control the bubble escape rate at the capillary orifice (for convenience of adjustment, fill the dropping funnel with water, and continue the subsequent measurement without closing the piston after adjusting the dropping speed for the first time), so as not to affect the measurement accuracy.

Experiment 4. Determination of viscosity of liquids

Experimental purpose

1. Understand the concept and significance of viscosity, and learn the measurement method of liquid viscosity.
2. The viscosity of ethanol at different temperatures was measured, and the apparent activation energy of flow was obtained.
3. Understand the structure and principle of constant temperature tank, and master the use method of constant temperature tank.

Experimental principles

Viscosity is an important property of fluid, which reflects the shear stress caused by the different velocity at each point of fluid flow. The molecular layer in contact with the stationary surface has zero velocity. The successive layers above it move with increasingly higher velocities in the direction of the flow.

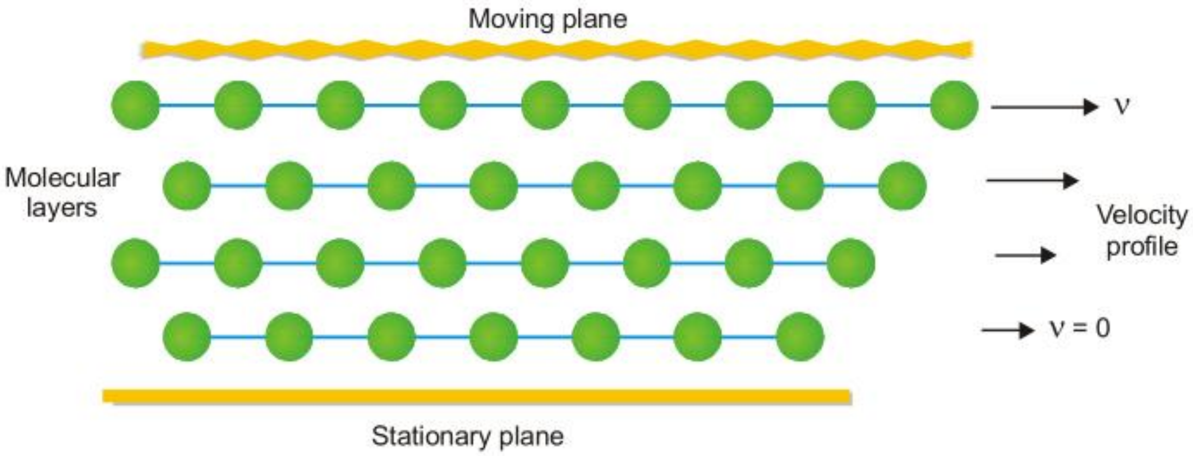


Figure 19. Flow of a liquid on a glass surface

Now consider two adjacent moving layers of a liquid (Fig. 20). Let these be separated by a distance dx and have a velocity difference dv. The force of friction (F) resisting the relative motion of the two layers is directly proportional to the area A and the velocity difference dv, while it is inversely proportional to the distance between the layers.

When many fluids are flowing, the shear stress on any differential volume element and the velocity gradient perpendicular to the flow direction in direct proportion, this fluid is called Newtonian fluid. Almost all gases and many simple liquids are Newtonian fluids, and polymer, slurry and waxy oil are common non-Newtonian fluids.

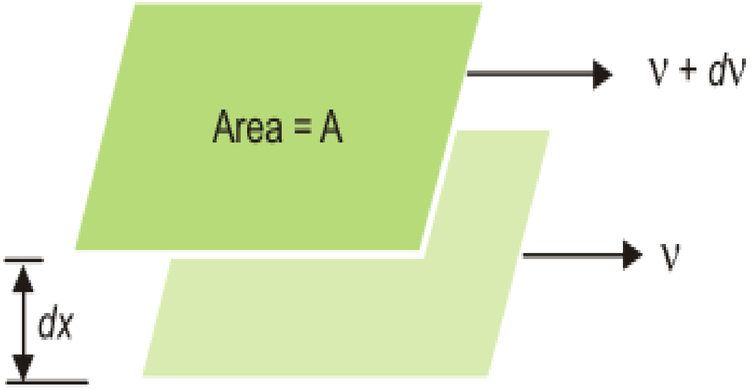


Figure 20. Two parallel layers moving in a liquid

For Newtonian fluid, the shear force F (i.e., when flowing) the relationship between internal friction) and velocity gradient dv/dx and contact area A is as follows:

That is,

$$F \propto A \frac{dv}{dx}$$

or

$$F = \eta A \frac{dv}{dx}$$

or

$$\eta = \frac{F}{A} \times \frac{dx}{dv}$$

The proportional coefficient η is called absolute viscosity degree.
The physical meaning of η is that two fluid layers with unit area separated by unit length in a fluid are separated by unit flow. The magnitude of shear force required for relative motion of velocity. When $A = 1\text{m}^2$, $dv/dx = 1\text{m}\cdot\text{s}^{-1}$, F = 1N, it is absolutely sticky. The degree η is $1\text{N} \cdot \text{s} \cdot \text{m}^{-2}$, that is, 1Pa · s.
There are three main methods to measure the viscosity of liquid:
①capillary method, which measures the time of liquid flowing in capillary;
②Falling ball method, measuring the falling time of the ball in liquid;
③ Rotating cylinder method, measuring the liquid in the same Influence of mandrel cylinders on relative rotation of cylinders.
Capillary method according to the use of the capillary viscometer is different from each other. The commonly used capillary viscometer is Ubbelli (Ubbelohde) and Ostwald. Austenitic viscometer was used in this experiment (the structure is shown in Fig. 20) measure the viscosity of Newtonian liquid.

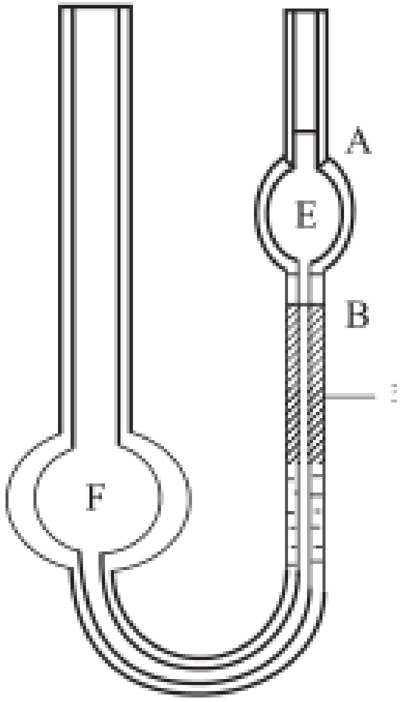


Figure 21. Austenitic Viscometer

The viscosity of the liquid can be measured by the time t of the volume V of the liquid flowing through the capillary tube quantity. When a liquid flows in a capillary viscometer due to gravity, it obeys Posser (Poiseuille):

$$\eta = \frac{\pi r^4 g h \rho t}{8Vl}$$

V is the volume of the liquid flowing through the capillary; r is the radius of capillary; ρ is liquid density; l is the length of the capillary; τ is the outflow time; h is the liquid flowing through the capillary tube average liquid column height of the body; g is the acceleration of gravity.

Liquid flows in capillaries by gravity of liquid column, which has potential energy, except the resistance consumed to overcome intermolecular friction. In addition to the force, the kinetic energy of the liquid itself is obtained at the same time, which makes the actual measured viscosity of the liquid low.

Therefore, Poselle's formula should be revised. The positive, more complete formula is:

$$\eta = \frac{\pi r^4 g h \rho t}{8Vl} - \frac{m \rho V}{8 \pi l t}$$

m is the calibration parameter of capillary end. If l > r, m = 1, viscometer determination with thin capillary tube. When the liquid flows slowly, if the outflow time is more than 100s, the second item of the above formula can be ignored. For the same viscometer, if h, r, l and V are all constants, Poselle's formula can be rewritten as follows:

$$\eta = k\rho t$$

K is called the viscometer constant (or capillary constant), and its value is affected by temperature, but it has little effect liquid viscosity.

It is difficult to measure the absolute value of the viscosity. Generally, the viscosity constant of the viscometer is measured by the liquid with known viscosity. The viscosity of the liquid to be measured can be measured according to the phase the outflow time measured under the same conditions is obtained:

$$\eta = \frac{\rho t}{\rho_0 t_0} \eta_0$$

η_0, ρ_0 and t_0 are the viscosity, density and outflow time of liquid with known viscosity; η, ρ and t are the viscosity of the liquid to be measured degree, density and outflow time.

Viscosity is greatly affected by temperature. For general liquids, the higher the temperature, the smaller the viscosity. The relationship between temperature and viscosity can be used the following empirical formula is expressed:

$$\ln \eta = \frac{A}{T} + B$$

A and B are empirical constants, and their values vary from liquid to liquid. Where A = ΔE/R, R is the universal constant of gas, ΔE is the apparent flow activation energy of liquid.

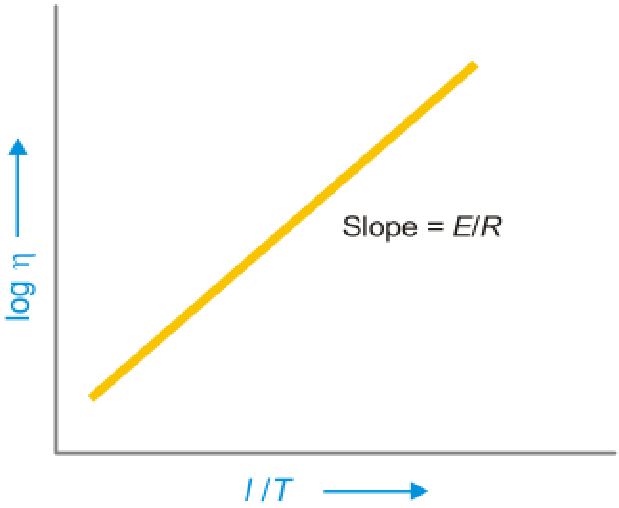


Figure 22. The plotting 1/T with ln η

A straight line can be obtained by plotting 1/T with ln η, and ΔE can be obtained from the slope of the straight line.

This experiment must be carried out at constant temperature.

Many properties of substances, such as refractive index, viscosity, vapor pressure, surface tension, electrical conductivity, adsorption quantity, electromotive force and chemical reaction rate constant are all related to temperature, so many chemical experiments must be carried out at constant temperature. This requires a thermostat with high sensitivity.

Instruments and Reagents

Instruments: One set of thermostat, two pipettes (10mL), one viscometer, one washing ball and one 1/10 stopwatch.

Reagents: Anhydrous ethanol (analytically pure).

Experimental content

1. Adjust the temperature of the thermostat to (25.0 ± 0.1) ^{0}C.

2. Take a dry and clean viscometer and place it vertically in a constant temperature tank water in the thermostat.

The surface should be immersed in the A scale of the E ball of viscometer. The viscometer should be placed in a proper position to observe the liquid flow constant temperature. The agitator speed of the tank should be appropriate, so as not to produce violent vibration and affect the measurement results. 10mL of distilled water was removed by pipette, inject the viscometer from the thick nozzle of the viscometer. After a constant temperature of 10 min, the liquid is sucked to the A-line by the upper part of the viscometer with an ear washing ball above, then let go of the ear washing ball and let the liquid flow down naturally. When the liquid level reaches the A scribe line, start the stopwatch to time. When reaching the B, stop timing and record the time when the liquid flows through the capillary. Repeat the operation three times, and the difference should not exceed each time over 1%, take the average.

3. Take out the viscometer and pour out the distilled water.

Rinse the viscometer carefully with absolute ethanol for 3 ~ 5 times. Then remove it with a pipette 10mL of anhydrous ethanol was added to a viscometer, and the flow of anhydrous ethanol at 25℃, 30℃ and 35℃ was measured according to the same method mentioned above time of capillary tube.

Data recording and processing

1.The list records the measurement data of each time.

Room temperature:__________air pressure:_____________________

Liquid name		Time, s				Pa·s
		1	2	3	average	
H_2O						
anhydrousethanol	25℃					
	30℃					
	35℃					

2.Using the outflow time of distilled water, the viscosity meter constant is calculated. The density and viscosity of distilled water can be obtained by looking up the table.

3.Calculate the viscosity of ethanol at 25℃, 30℃, and 35℃.

4.Takelnηto1/T is plotted to obtain a straight line, and the apparent flow activation energy is obtained from the slope.

Questions

1.Why does the viscometer have to be placed vertically in the thermostat?

2. How does the thickness of the capillary of the viscometer affect the experiment? 3. Why should the volume of the added standard and the measured object be the same? Why should the temperature be constant when measuring viscosity?

Experimental Guidance

1. Viscometer must be clean, sometimes trace dust, oil stain, etc. Will produce local blockage, the flow rate in the thin tube leads to a large error.

2. The diameter and length of viscometer capillary should be appropriate, so that the liquid outflow time is more than 100s.

3. The viscometer should be immersed vertically in the constant temperature tank. Do not vibrate the viscometer during the experiment, because inclination will cause the change of liquid level difference. It causes measurement errors and lengthens the flow time of liquid.

Experiment 5. Determination of the Phase Diagram of a two-component liquid-vapor equilibrium system

Experimental purpose

1. The T-x phase diagram of gas-liquid equilibrium of water-n-propanol at normal pressure was measured by boiling point meter. And find out the composition of the constant boiling point mixture and the lowest constant boiling point.
2. Master the determination method of boiling point of two components.
3. Master the measuring principle and using method of Abbe refractometer. The composition of liquid and vapor was determined by Abbe refractometer.

Experimental principles

A solution is a mixture of two or more substances dissolved in another. Solute is the substance present in the smaller amount. Solvent is the substance present in the larger amount. In aqueous solutions, the solvent is water.

A dissolved substance is a solute that is completely soluble in the solvent, resulting in the formation of a homogeneous solution. The solute can be a solid or a liquid or a gas.

Table 10. Types and examples of solution

State of solute	State of solvent	Example
Gas	Gas	Air
Gas	Liquid	Oxygen in water, CO_2 in water
Gas	Solid	Adsorption of H_2 by palladium
Liquid	Liquid	Alcohol in water
Liquid	Solid	Mercury in silver
Solid	Liquid	Sugar, Salt
Solid	Solid	Metal alloys, carbon in iron

Physical properties can be divided into two categories:

- Extensive properties (such as mass and volume) depend on the size of the sample [10].
- Intensive properties (such as density and concentration) are characteristic properties of the substance; they do not depend on the size of the sample being studied [10].

And we have discussed, solutions have different properties than either the solutes or the solvent used to make the solution. Those properties can be divided into two main groups:

1) Colligative properties depend only on the number of dissolved particles in solution and not on their identity. (Greek colligatus = Collected together) [10].

2) Non-colligative properties depend on the identity of the dissolved species and the solvent [10].

Colligative properties are depending on following factory:

1) Number of particles

- in case of non-electrolytes – number of molecules
- in case of electrolytes – number of ions
- Number of moles of solute
- Mole fraction of solute

2) For different solutes of same molar concentration, the magnitude of the colligative properties is more for that solution which gives more number of particles on ionization.

3) For different solutions of same molar concentration of different non-electrolyte solutes, the magnitude of the colligative properties will be same for all.

4) For different molar concentrations of the same solute, the magnitude of colligative properties is more for the more concentrated solution.

5) For solutions of different solutes but of same percent strength, the magnitude of colligative property is more for the solute with least molecular weight.

6) For solutions of different solutes of the same percent strength, the magnitude of colligative property is more for that solute which gives more

number of particles, which can be known by the knowledge of molecular weight and its ionization behavior.

Colligative properties include:

1. Lowering of vapour pressure of the solvent (Raoult's 1 st law).
2. Elevation in boiling point of the solvent (Raoult's 2 nd law).
3. Depression in freezing point of the solvent (Raoult's 2 nd law).
4. Osmotic pressure of the solution [11].

The essential feature of these properties is that they depend only on the number of solute particles present in solution. Being closely related to each other through a common explanation, these have been grouped together under the class name Colligative Properties [11].

A colligative property may be defined as one which depends on the number of particles in solution and not in any way on the size or chemical nature of the particles [11].

When a liquid is placed in an open vessel, it evaporates. The process by which molecules of a liquid go into the gaseous state (vapours) is called Vaporization or Evaporation. The reverse process whereby gas molecules become liquid molecules is called Condensation.

If the liquid is placed in a closed vessel a stage comes when the number of molecules escaping from the liquid is equal to the number of molecules returning to the liquid. Thus a dynamic equilibrium is established between the liquid and the vapour at the given temperature.

Now the concentration of the vapour in the space above the liquid will remain unchanged with lapse of time. Hence the vapour will exert a definite pressure at the equilibrium. The vapour pressure of a liquid is defined as: the pressure exerted by the vapour in equilibrium with the liquid at a fixed temperature, fig. 23 [12].

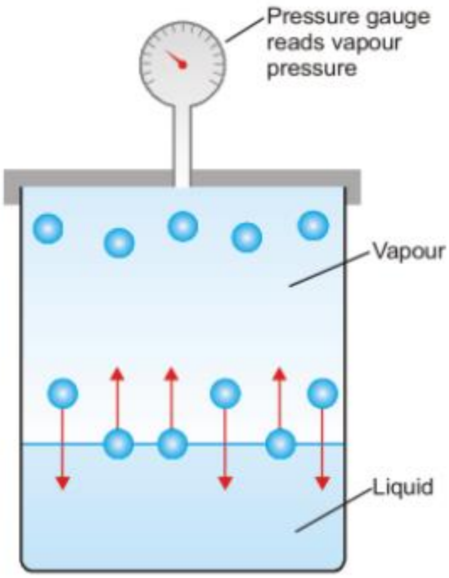


Figure 23. Illustration of vapour pressure

The vapour pressure of the pure solvent is caused by the number of molecules evaporating from its surface. When a nonvolatile solute is dissolved in solution, the presence of solute molecules in the surface blocks a fraction of the surface where no evaporation can take place [12].

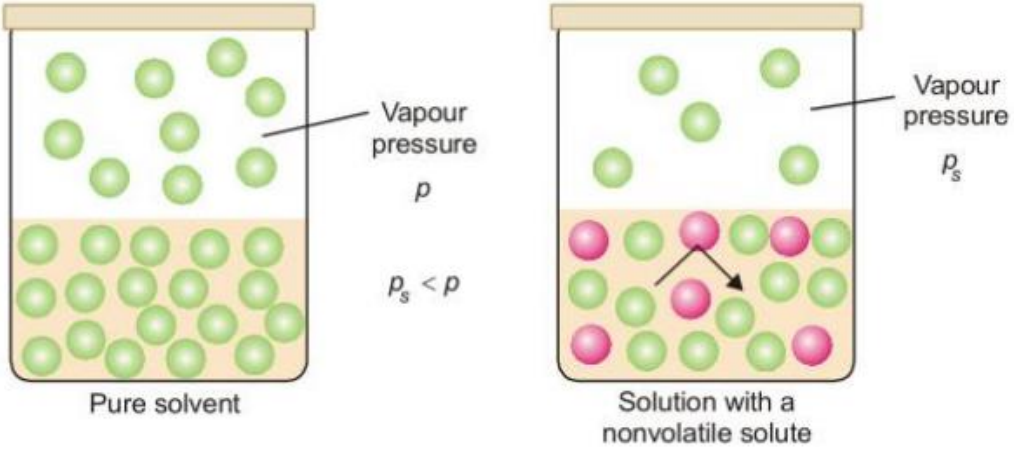


Figure 24. Lowering of vapour pressure by a nonvolatile solute

The particles of the solute block the escape of solvent molecules from the surface of the solution.

When a liquid is heated, its vapour pressure rises and when it equals the atmospheric pressure, the liquid boils. The addition of a nonvolatile solute lowers the vapour pressure and consequently elevates the boiling point as the solution has to be heated to a higher temperature to make its vapour pressure become equal to atmospheric pressure [12].

The Raoult's Law: the relative lowering of the vapour pressure of a dilute solution is equal to the mole fraction of the solute present in dilute solution [12].

Raoult's Law can be expressed mathematically in the form:

$$(p - p_s) / p = n / (n + N)$$

where n - number of moles or molecules of solute, N - number of moles or molecules of solvent [12].

When a liquid is heated, tiny bubbles are formed in it. These rise to the liquid surface and burst. The temperature at which it happens is the boiling point of the liquid [12].

The liquid vaporises into it and the vapour pressure in the bubble keeps it in form. However, the pressure of the atmosphere exerted on the liquid top tends to collapse the bubble. As the bubble goes to the surface, the vapour pressure in the bubble equals the atmospheric pressure. Thus the bubble collapses. The boiling point of the liquid may, therefore, be defined as the temperature at which the vapour pressure of the liquid is equal to the atmospheric pressure [12].

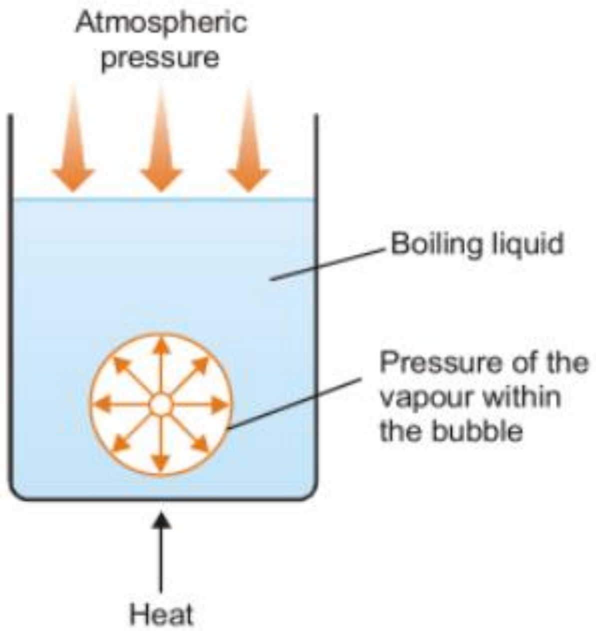


Figure 25. A liquid boils when the pressure of the vapour within the bubble equals the atmospheric pressure exerted on the bubble at the liquid surface

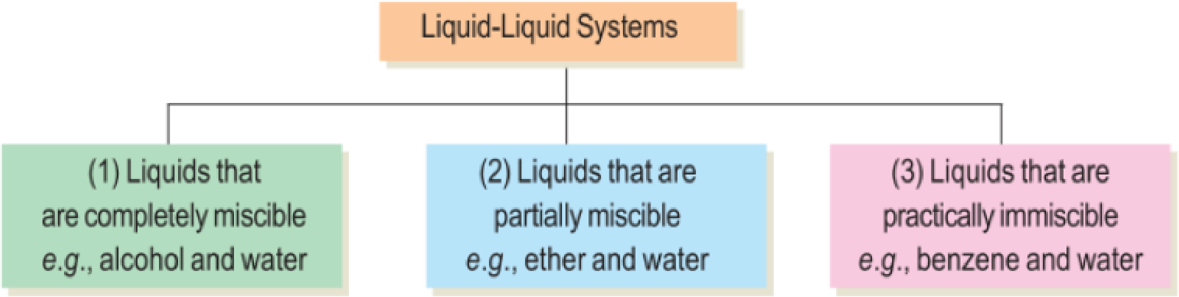
The boiling point of a liquid can be lowered by reducing the external pressure by vacuum pump. Then the vapour pressure of the liquid is equal to the external pressure at a lower temperature. The boiling point of a liquid can be increased by raising the external pressure. Thus the vapour pressure of the liquid is equal to the external pressure at a higher temperature. A domestic pressure cooker works on this principle. The pressure inside the cooker is maintained above one atmosphere and the liquid contained in it would boil at a higher temperature than 100ºC. Thus the food is cooked in a shorter time [12].

When a liquid is heated, its vapour pressure rises and when it equals the atmospheric pressure, the liquid boils. The addition of a nonvolatile solute lowers the vapour pressure and consequently elevates the boiling point as the solution has to be heated to a higher temperature to make its vapour pressure become equal to atmospheric pressure. If T_b is the boiling point of the solvent and T is the boiling point of the solution, the difference in the boiling points (ΔT) is called the elevation of boiling point [12].

$$T - T_b = \Delta T$$

The vapour pressure of a pure liquid changes with temperature. The difference of the freezing point of the pure solvent and the solution is referred to as the Depression of freezing point. Depression of freezing point is directly proportional to the lowering of vapour pressure [12].

The solutions of liquids in liquids may be divided into three classes as follows:



At room temperature, the system composed of any two liquids is called two-liquid system. If two liquids can dissolve each other in any proportion, it is called a completely miscible two-liquid system; If it can only be partially miscible, it is called partially miscible two-liquid system.

The boiling point of the two-liquid system is not only related to the external pressure, but also related to the composition of the two-liquid system. By distilling a completely miscible two-liquid system under constant pressure and measuring the composition of distillate (gas phase) and distillate (liquid phase), the composition of gas and liquid phases at equilibrium can be found out and the T-x diagram can be drawn.

If we plot the boiling point of liquid mixture against its composition and the composition of the vapour in contact with it, we get two separate curves for each type of solutions. The curves obtained for the third type are shown diagrammatically in Fig. 26.

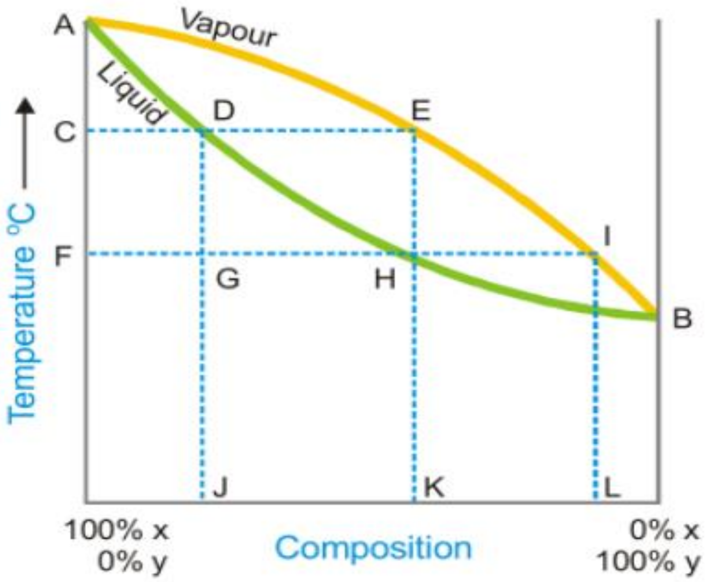


Figure 26. Curves showing the composition of vapour and liquid at various boiling temperatures

The curves AEB and ADB are the temperature composition curves for the vapour and liquid respectively. At any boiling temperature C the composition of liquid mixture is represented by J and that of the vapour in equilibrium by K.

Obviously, the more volatile component Y is present in greater proportion in the vapour than the liquid mixture. Thus the condensed vapour or the distillate will be richer in X.

If the distillate so obtained be now subjected to distillation, it will boil at F and the fresh distillate will have the composition L corresponding to I. Thus the proportion of Y in the second distillate is greater than in the first one. In this way by repeating the process of fractional distillation it is obvious that we can get almost pure Y.

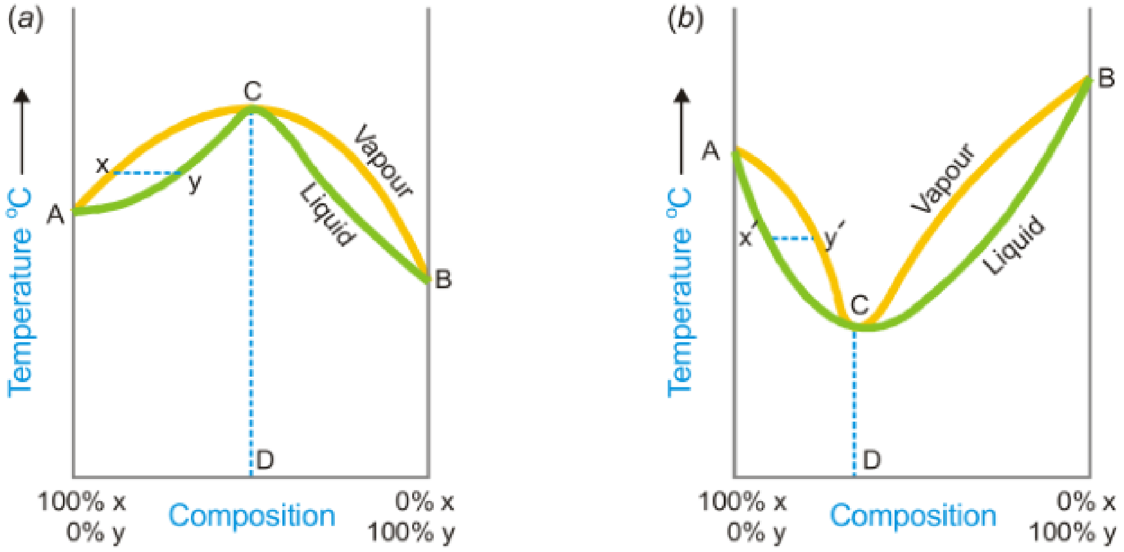


Figure 27. (a) Boiling point curves for 1st type of solutions;
(b) Boiling point curves for 2nd type of solutions.

In first type of solutions (Fig. 27, a) if we have a boiling mixture represented by Y its vapour will be poor in Y than the liquid mixture and the boiling point would gradually rise till we reach the maximum point C where the composition of liquid and vapour is the same. Here the distillation proceeds without change of composition.

Similarly, in the second type (Fig. 27, b), if we have a boiling mixture represented by the point X', the amount of Y in vapour is higher and gradually the boiling point falls to the minimum C' where the vapour and the liquid mixtures have the same composition. At this temperature the mixture boils without any change in composition. Thus it is proved that the second and first type of solutions are not capable of being separated by fractional distillation.

Usually, if the deviation between the liquid and Raoult's law is not large, the boiling point of the solution is between the boiling points of pure liquids A and B on the T-X diagram as shown in Fig. 26. However, due to the mutual influence of A and b components, the actual solution often deviates greatly from Raoult's law, and there will be the highest or lowest points on the T-x diagram. These points are called constant boiling points, and the corresponding solutions are called constant boiling point mixtures, as shown in Fig. 27 (a) and (b).

When a constant boiling point mixture is distilled, the resulting gas phase and liquid phase have the same composition, so its composition cannot be changed by distillation. In this experiment, the T-x diagram of water-n-propanol system was drawn by reflux condensation method. When the relative amount of gas and liquid is constant, the temperature of the system will remain constant, and the boiling point, that is, boiling temperature, can be read by thermometer. They are composed of equilibrium gas phases sampled and analyzed in concave grooves where vapor condenses; The composition of equilibrium liquid phase was analyzed by sampling from the liquid feeding port, and the instrument used for sample analysis was Abbe refractometer.

The refractive index of the sample is measured in the experiment, and the corresponding composition is found from the refractive index-composition working curve, and then the T-x diagram is drawn.

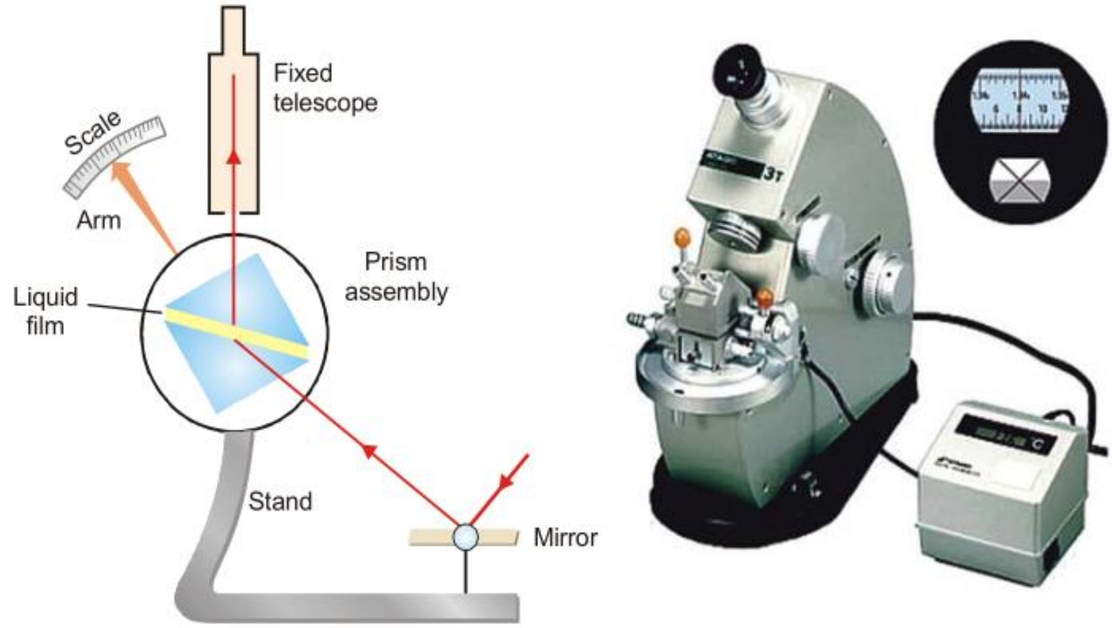


Figure 28. Abbe refractometer

Instruments and reagents

Instruments:1 set of boiling point meter; 1 thermostatic tank; 1 Abbe refractometer; 2 1mL liquid measuring tubes; Two 2 mL pipettes, two 10mL pipettes, one 20 mL pipette and one 50 ml pipette; Beaker, 4 small test tubes with plug.

Reagents: N-propanol (A.R, ≥ 99%); Water.

Experimental content

1. Adjust the temperature of the constant temperature tank to be 5 ℃ higher than the room temperature, and pass constant temperature water into Abbe refractometer.

2. Determine the relationship between refractive index and composition, and draw the working curve: 9 small test tubes were numbered, and then moved into 0.50 mL, 1.25 mL, 2.00 mL, 2.50 mL, 3.00 mL, 3.25 mL, 3.75 mL, 4.25 mL, 4.50 mL water, then 4.50 mL, 3.75 mL, 3.00 mL, 2.50 mL, 2.00 mL, 1.75 mL, 1.25 mL, 0.75 mL, 0.5 mL n-propanol, shook gently and mixed evenly to prepare 9 solutions with known concentration. The refractive index of each solution, pure propanol and pure water were measured by Abbe refractometer.

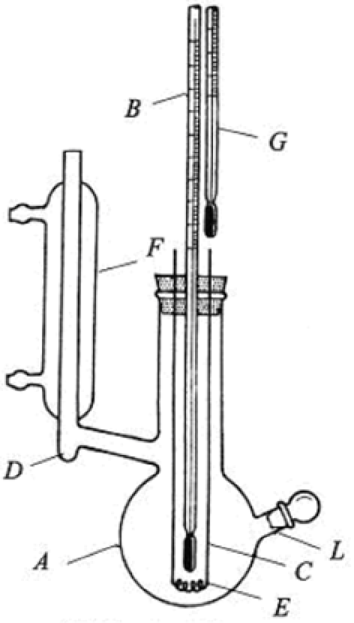
Table 11. Refractive index of water-n-propanol standard solution

X,n-propanol	0								100
refractive index									

The working curve is obtained by plotting the refractive index against the concentration (converted into mass percentage concentration according to the density of pure sample).

3. Determine the relationship between boiling point and composition of n propanol-water system.

Although the design of boiling point meter is different, its design idea focuses on how to measure boiling point and composition of gas and liquid phase correctly, and prevent overheating and fractionation. The boiling point meter we used is shown in Fig. 29.



A:A container for holding liquid
B:Measuring thermometer
C:Small glass tube
D:Small ball
E:Heating wire
F:Condensing pipe
G:Thermometer
L:Branch pipe

Figure 29. Construction of boiling point meter and determination of boiling point

This is a long-necked round-bottomed flask with a reflux condenser tube with a spherical chamber D at the bottom for collecting condensed vapor phase samples. The liquid phase sample is extracted through the branch pipe L on the flask.

In the figure E is an electric heating wire cut by 300W electric furnace wire, which is directly immersed in the solution for heating to reduce the overheating and boiling phenomenon when the solution boils. Thermometer installation must pay attention to make half of the mercury ball immersed under the liquid surface, half exposed in vapor, and a small glass tube C is sleeved around the mercury ball, so that when the solution boils, under the drive of bubbles, the gas and liquid are continuously sprayed to the mercury ball and overflowed from the upper end of the glass tube; Small glass tube C can also reduce the fluctuation of thermometer reading caused by the surrounding environment of boiling point (such as air flow or radiation from other heat sources), so the measured temperature can better represent the equilibrium temperature of gas-liquid two phases.

To analyze the composition of gas phase and liquid phase in equilibrium, it is necessary to obtain gas phase and liquid phase samples correctly. The fractionation of vapor in boiling point meter will affect the equilibrium composition of gas phase, and make the composition of obtained gas phase sample deviate from that in gas-liquid equilibrium, so it is necessary to reduce the fractionation of gas phase. The boiling point meter used in this experiment is to condense the vapor in equilibrium in the small ball D. On the premise that the solution in container A will not splash into the small ball D, the distance between the small ball D and the big ball A can be shortened as much as possible. To prevent fractionation, the volume of the small ball D can be reduced as much as possible to achieve this goal. In order to accelerate the equilibrium of the system, the liquid initially condensed in ball D can be poured back into container A.

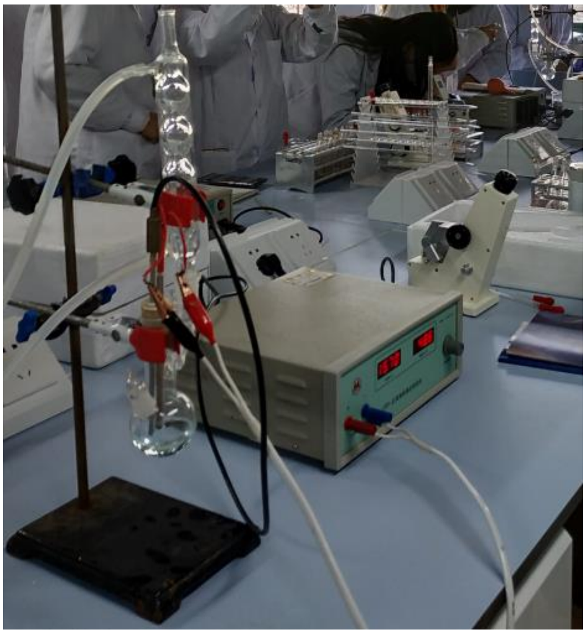


Figure 30. General view of the installation

Measurement of boiling point: Insert thermocouple into solution and connect with temperature tester for measurement. Install the boiling point meter, turn on the cooling water, and heat it to boil the solution in the boiling point meter.

At first, the condensate in the bag at the lower end of the condenser tube cannot represent the gas phase composition at equilibrium. Pour the initial condensed liquid in the bag back to the distiller and repeat it for 2 ~ 3 times.

After the solution boils and the reflux is normal and the temperature reading is constant, record the boiling point of the solution. Use capillary dropper to draw gas phase samples from gas phase condensate sampling port, put them into small test tubes with grinding port, and put the test tubes in a small flask with cold water to cool them to prevent volatilization.

The sample was dropped into Abbe refractometer and its refractive index n_g was measured. Then use another dropper to suck the solution in the boiling point meter and measure its refractive index n_L.

In this experiment, the constant boiling point is taken as the boundary, and the phase diagram is divided into left and right halves, and the phase diagram is drawn twice. The specific methods are as follows:

(1) Determination of the right half boiling point-composition relationship, tab. 12.

The boiling point of n-propanol was measured first by adding 50 mL of n-propanol into a boiling point meter, and then 0.5 mL, 1.0 mL, 1.5 mL, 2.0 mL, 2.5 mL, 4.0 mL and 6.0 mL of water were added in turn. The boiling point of solution and the refractive index ng of gas phase component and nL of liquid phase component were measured respectively by the above methods. After the experiment, pour the solution into the recycling bottle.

(2) Determination of the left half boiling point-composition relationship, tab. 13.

The boiling point of water was measured first by adding 40 mL of water into a boiling point meter, then 0.5 mL, 1.0 mL, 1.5 mL, 2 mL, 4 mL, 10 mL and 20 mL of n-propanol were added in turn. The boiling point of solution and the refractive index of gas phase component ng and liquid phase component nL were measured respectively by the above methods.

4. According to the refractive index of water-n-propanol standard solution, the above data are converted into the mass fraction of n-propanol, and the phase diagram is drawn.

During the experiment, It can be observed that the refractive index of vapor phase and liquid phase of n-propanol-pure water system will move in the direction of decreasing or increasing, At first, the refractive index readings of the gas and liquid phase have a small difference, and the difference gradually increases and then decreases until they are equal. It means that the lowest constant boiling point composition has been reached at this time, and this composition is a lowest constant boiling point mixture. The lowest constant boiling point of the system is about 87 ℃, and the content of n-propanol is between 69 and 71%.

Table 12. Measurement data of water-n-propanol mixed solution

Component	Boiling point/℃	Liquid phase analysis		Gas phase condensate analysis	
		refractive index	*X*, n-propanol	refractive index	*Y*,n-propanol
+0.0 mL H_2O					
+0.5 mL H_2O					
+1.0 mL H_2O					
+1.5 mL H_2O					
+2.0 mL H_2O					
+2.5 mL H_2O					
+4.0 mL H_2O					
+6.0 mL H_2O					

Table 13. Measurement data of n-propanol- water mixed solution

Component	Boiling point/℃	Liquid phase analysis		Gas phase condensate analysis	
		refractive index	*X*, n-propanol	refractive index	*Y*,n-propanol
+0.0mL n-propanol					
+0.5 mL n-propanol					
+1.0 mL n-propanol					
+1.5 mL n-propanol					
+2.0 mL n-propanol					
+4.0 mL n-propanol					
+10.0 mL n-propanol					
+20.0 mL n-propanol					

5. After the experiment, turn off the condensed water, clean the instrument, turn off the power supply and arrange the test bench.

Data Recording and Processing

1. The refractive index measured in the experiment is composed of data list and drawn into working curve. The corresponding composition is found from the working curve, and the relationship between boiling point and composition is obtained.
2. Draw T-x diagram of water-n-propanol system, and indicate the lowest constant boiling point and composition.
3. In the accurate measurement, the exposed mercury column of thermometer should be corrected.

Questions

1. In this experiment, do the constant temperature of refractometer when measuring the working curve and the constant temperature of refractometer when measuring the sample need to be consistent? Why?
2. What effect does overheating have on the experiment? How to avoid it as much as possible in the experiment?
3. Should the amount of sample added be very accurate in the continuous determination experiment? Why?
4. The D storage tank in the boiling point meter is too large or too small, what influence does it have on the measurement?
5. At equilibrium, should the temperature of gas and liquid be the same, is it actually the same, and what influence does it have on the measurement?
6. How to judge that gas-liquid has reached equilibrium? Discuss the separation of this solution during distillation.

Experimental guidance

1. When determining pure liquid samples, the boiling point meter must be dry.
2. Because the whole system is not absolutely constant temperature, the temperature of gas and liquid will be slightly different. Therefore, in the boiling point meter, the position of mercury ball of thermometer should be half immersed in solution and half exposed in vapor. And with the increase of solution amount, the position of mercury ball should be continuously adjusted.
3. In the experiment, the heating voltage can be adjusted to control the backflow speed, and the voltage should not be too large, so that the liquid to be measured can boil. The resistance wire cannot be exposed to the liquid surface, and must be immersed by the liquid to be measured.
4. In the distillation process of each sample, the equilibrium temperature will change slightly because the composition of the whole system cannot be kept constant, especially when the amount of two components in the solution is quite different. For this reason, after adding samples once, samples can be sampled and determined as long as the solution boils and refluxes normally for 1min ~ 2 min, and the waiting time should not be too long.

5. The sampling amount should not be too much each time. When sampling, the capillary dropper must be dry, and the residual liquid from the last time should not be left. The residual liquid from the gas phase sampling port should also be wiped clean.
6. During the whole experiment, the water temperature passing through the refractometer should be constant. When using the refractometer, the prism should not touch hard objects (such as droppers). Wipe the prism with mirror wiping paper.
7. For the determination of refractive index of samples, it is necessary to move quickly, spread the samples and lock the knob to ensure the accuracy of the test. When using Abbe refractometer to read data, special attention should be paid to drying the mirror surface with mirror wiping paper between the condensate sample in gas phase and the liquid sample in liquid phase.

Experiment 6. Determination of the molecular mass of a substance by cryoscopy

Experimental purpose

1. Measure the freezing point of the solution and the pure solvent experimentally [9].
2. Calculate the molecular mass of a substance by lowering the freezing point of the solution [9].

Experimental principles

This module will focus on four colligative properties of electrolyte and non-electrolyte solutions namely [9]:

- vapor pressure lowering,
- boiling point elevation,
- freezing point depression,
- osmotic pressure.

The Colligative Properties of Solution	Mathematic equation for	
	Non-electrolytes Solution	Electrolytes Solution
Vapor pressure lowering (1stRaoult's Law)	$P^0 - P / P^0 = N_x$	$P^0 - P / P^0 = iN_x$
Boiling point elevation (2nd Raoult's Law)	$\Delta t_{boil} = C_m E_{svt}$	$\Delta t_{boil} = iC_m E_{svt}$
Freezing point depression (2nd Raoult's Law)	$\Delta t_{freez} = C_m K_{svt}$	$\Delta t_{freez} = iC_m K_{svt}$
Osmotic pressure (Van't Hoff law)	$P_{osm}= C_m RT$	$P_{osm}= i C_m RT$

There are:

- E_{svt} is boiling point elevation constant, equivalent to $0.52^0C/m$ for aqueous solutions. This means that, for example, 1 mole of sugar (non-electrolyte) in 1 kilogram of water will increase the boiling point from 100^0C to $100,52^0C$. And C_m is the molal concentration of solute.
- K_{svt} is the freezing point depression constant equivalent to $-1,86^0C/m$ for aqueous solutions. Again, for example, 1 mole of sugar (non-electrolyte) in 1 kilogram of water will decrease the freezing point from 0^0C to $-1,86^0C$.
- The iVan't Hoff factor gives the number of particles per formula unit of the electrolyte solute. The degree of dissociation is associated with an isotonic factor by next ratio:

$$\alpha = (i-1)/(n-1)$$
$$i = \alpha\,(n-1) + 1$$

The vapour pressure of a pure liquid changes with temperature as shown by the curve ABC, in Fig. 31. There is a sharp break at B where, in fact, the freezing-point curve commences. Thus the point B corresponds to the freezing point of pure solvent, T_f. The vapour pressure curve of a solution (solution 1) of a nonvolatile solute in the same solvent is also shown in Fig. 31. It is similar to the vapour pressure curve of the pure solvent and meets the freezing point curve at F, indicating that $T_1$is the freezing point of the solution. The difference of the freezing point of the pure solvent and the solution is referred to as the Depression of freezing point. It is represented by the symbol ΔT or ΔT_f [9].

$$T_f - T_1 = \Delta\ T$$

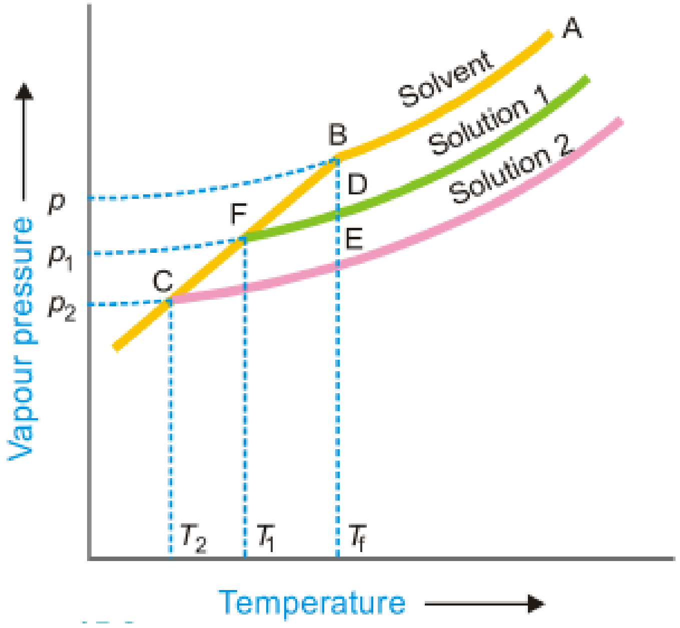


Figure 31. Relation between lowering of vapour-pressure and depression of freezing point.

When more of the solute is added to the solution 1, we get a more concentrated solution (solution 2.) The vapour pressure of solution 2 meets the freezing-point at C, indicating a further lowering of freezing point to T_2 [9].

For dilute solutions FD and CE are approximately parallel straight lines and BC is also a straight line. Since the triangles BDF and BEC are similar,

$$\frac{DF}{EC} = \frac{BD}{BE}$$

or

$$\frac{T_f - T_1}{T_f - T_2} = \frac{p - p_1}{p - p_2}$$

where p_1 and p_2 are vapour pressure of solution 1 and solution 2 respectively. Hence depression of freezing point is directly proportional to the lowering of vapour pressure [9].

$$\Delta T \propto p - p_s$$

Since p is constant for the same solvent at a fixed temperature:

$$\Delta T \propto (p - p_s)/ p$$

From Raoult's Law for dilute solutions,

$$(p - p_s)/ p = w{\cdot}M / W{\cdot}m$$

Since M of solvent is constant:

$$(p - p_s)/ p = w / W{\cdot}m$$

$$\Delta T \propto K_f \times \frac{w}{m} \times \frac{1}{W}$$

$$\Delta T = K_f \times \frac{w}{m} \times \frac{1}{W}$$

where K_f is a constant called Freezing-point constant or Cryoscopic constant or Molal depression constant. If w/m = 1 and W = 1, $K\ f = \Delta T$ [9].

Thus, Molal depression constant may be defined as the freezing-point depression produced when 1 mole of solute is dissolved in one kg (1000 g) of the solvent [9].

If the mass of solvent (W) is given in grams, it has to be converted into kilograms.

$$\Delta T = K_f \times \frac{w}{m} \times \frac{1}{W/1000}$$

$$\Delta T = K_f \times \frac{w}{m} \times \frac{1000}{W}$$

$$m = \frac{1000 \times K_f \times w}{\Delta T \times w}$$

where m = molecular mass of solute; K_f = molal depression constant; w = mass of solute; ΔT = depression of freezing point ; W = mass of solvent.

Given the value of K_f, the molecular mass of solute can be calculated [9].
The value of K_f can be determined by measurement of ΔT by taking a solute of known molecular mass (m). The constant K_f, which is characteristic of a particular solvent, can also be calculated from the relation [9].

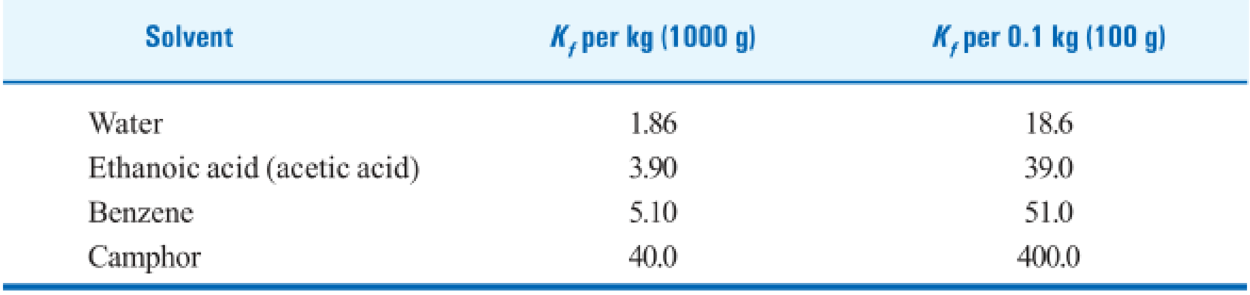
$$K_f = \frac{RT_f^2}{1000\ L_f}$$

where T_f = freezing point of solvent in K; L_f = molar latent heat of fusion; R = gas constant. Hence for water, T_f = 273 K and L_f = 336 $J{\cdot}g^{-1}$. Therefore,

$$K_f = \frac{8.32 \times 273 \times 273}{1000 \times 336} = 1.86\ \text{K kg}^{-1}$$

The molal depression constant for some common solvents are given in Table 14 [9]

Table 14. Molal freezing-point constants



Solvent	K_f per kg (1000 g)	K_f per 0.1 kg (100 g)
Water	1.86	18.6
Ethanoic acid (acetic acid)	3.90	39.0
Benzene	5.10	51.0
Camphor	40.0	400.0

Instruments and reagents

Instruments: cryoscope, Beckman thermometer, an ordinary thermometer.
Reagents: suspension of the substance, salt for preparing the cooling mixture, snow or ice, distilled water.

Experimental content

The cryoscopic method is applicable to highly dilute solutions of binary non-isomorphic systems.

Apparatus (cryoscope) consists of (1) A freezing tube with a side-arm to contain the solvent or solution, while the solute can be introduced through the side-arm; (2) An outer larger tube into which is fixed the freezing tube, the space in between providing an air jacket which ensures a slower and more uniform rate of cooling; (3) A large jar containing a freezing mixture e.g., ice and salt, and having a stirrer, fig. 32 [9].

15 to 20 g of the solvent is taken in the freezing point of the solvent by directly coding the freezing point tube and the apparatus set up as shown in Fig. 31 so that the bulb of the thermometer is completely immersed in the solvent. First determine the approximate freezing point of the solvent by directly cooling the freezing point tube in the cooling bath. When this has been done, melt the

solvent and place the freezing-point tube again in the freezing bath and allow the temperature to fall [9].

When it has come down to within about a degree of the approximate freezing point determined above, dry the tube and place it cautiously in the air jacket. Let the temperature fall slowly and when it has come down again to about 0.5° below the freezing point, stir vigorously. This will cause the solid to separate and the temperature will rise owing to the latent heat set free. Note the highest temperature reached and repeat the process to get concordant value of freezing point [9].

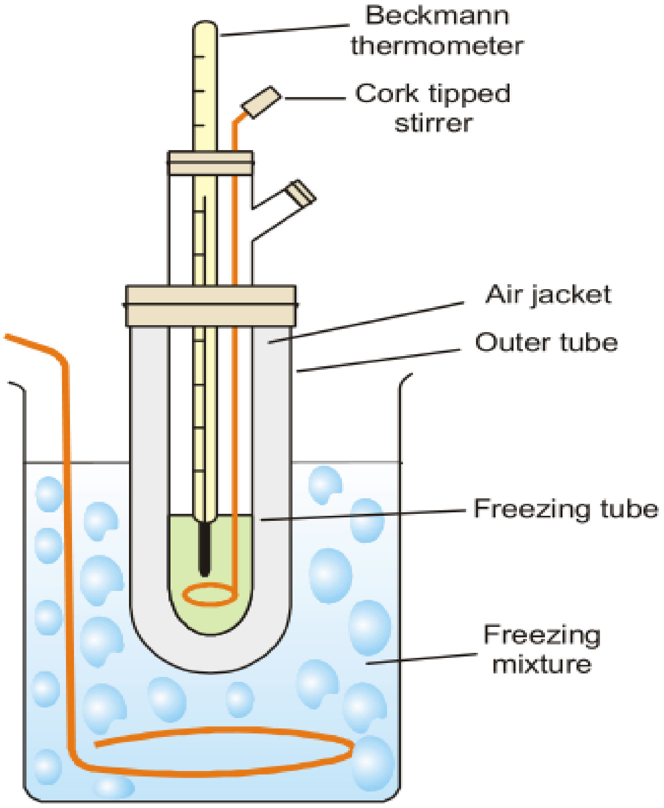


Figure 32. Diagram of the laboratory installation

The freezing point of the solvent having been accurately determined, the solvent is remolded by removing the tube from the bath, and a weighed amount (0.1–0.2 g) of the solute is introduced through the side tube. Now the freezing point of the solution is determined in the same way as that of the solvent. A further quantity of solute may then be added and another reading taken. Knowing the depression of the freezing point, the molecular mass of the solute can be determined by using the expression

$$m = \frac{1000 \times K_f \times w}{\Delta T \times W}$$

where m = molecular mass of solute; K_f = molal depression constant; w = mass of solute; ΔT = depression of freezing point; W = mass of solvent [9].

Date recording and processing

1. Determination of the freezing point of the solvent.
2. Determination of the freezing point of the solution.
3. After the experimental determination of the molecular weight of the substance, find out which substance was dispensed. The resulting molecular weight value is compared with the tabular value.

Record the measurement data and the results of their processing in the table 15.

Table 15. Experimental data and results of their processing

Solvent and its mass, g -					
The dissolved substance and its mass, g -					
The system	Crystallization temperature, ^{0}C		Lowering the crystallization temperature	The molecular mass of the solute	
	Measured	Average		The experimental determination	The tabular value
a pure solvent					
solution					

Questions

1.What is called a true solution?

2. Why does the freezing point decrease with increasing concentration of the solution?

3. What determines the isotonic coefficient?

4. What is the name of the point at which simultaneous crystallization of the solvent and solute occurs?

5. What is the name of the point where the crystallization curve and the evaporation curve intersect?

6. The i factor gives the number of particles per formula unit of the solute. What is the i factor for NaCl?

Experimental guidance

This method gives accurate results, if the following precautions are observed:

(a) The super cooling should not exceed 0.5°C.

(b) The stirring should be uniform at the rate of about one movement per second.

(c) The temperature of the cooling bath should not be 4° to 5° below the freezing point of the liquid [8].

Experiment 7. Determination of the Reaction Rate Constant of Ethyl Acetate Saponification

Experimental Purpose

1. Understand the characteristics of secondary reactions.
2. Determination of the reaction rate constant of ethyl acetate saponification by conductivity method.
3. Calculate the reaction activation energy.
4. Master the use of conductivity meter.

Experimental Principles

The saponification reaction of ethyl acetate is a secondary reaction with the following formula:

$$CH_3COOC_2H_5 + OH^- \longrightarrow CH_3COO^- + C_2H_5OH$$

If the concentration of the product is x set at time t, the kinetic equation for the reaction is:

$$\frac{dx}{dt} = k(a-x)(b-x) \qquad (1)$$

where a and b are the starting concentrations of ethyl acetate and base (NaOH), respectively; k is the reaction rate constant.

If a=b, then equation (1) becomes:

$$\frac{dx}{dt} = k(a-x)^2 \qquad (2)$$

Integral to equation (2)

$$k = \frac{1}{t} \times \frac{x}{a(a-x)} \qquad (3)$$

If the x value is measured by the experiment at different t, the k value at different t can be calculated according to equation (3). If the value of k is constant, it proves that the reaction is a secondary one.

$\frac{x}{a-x}$ is usually plotted against t, and if the result is a straight line, it is proved to be a secondary reaction, and the k value can be obtained from the slope of the line.

The concentration of the product at different times can be determined by chemical analysis (e.g. OH^- concentration in the reaction solution). It also could be determined by physicochemical analysis (such as measuring conductance), and in this experiment it is measured by conductivity.

The basis for determining the x value by the conductivity method is as follows.

(1) The conductivity of OH^- in solution is much larger than that of Ac^- (i.e., CH_3COO^-) (The conductivity of reactants and formations varies greatly). As the reaction progresses, the concentration of OH^- decreases, and so does the conductivity of the solution.

(2) In dilute solution, the conductivity κ of each strong electrolyte is proportional to its concentration, and the total conductivity of the solution is equal the sum of the conductivities of the electrolytes that make up the solution.

According to the above two points, for ethyl acetate saponification, only NaOH and NaAc are strong electrolytein reactants and products. If the reaction is under dilute solution, then

$$\kappa_0 = A_1 a$$

$$\kappa_\infty = A_2 a$$

$$\kappa_t = A_1(a-x) + A_2 x$$

where A_1 and A_2 are proportional constants related to temperature, solvent, electrolyte NaOH and NaAc properties; κ_0、κ_∞ the total conductivity of the solution at the beginning and end of the reaction, respectively (note that there is only one electrolyte at this time); $\kappa_t$isthe total conductivity of the liquid in time t. This can be obtained from these three equations

$$x = \left(\frac{\kappa_0 - \kappa_t}{\kappa_0 - \kappa_\infty}\right) a \qquad (4)$$

If the starting concentration of ethyl acetate and NaOH is equal, substitute equation (4) into equation (3)

$$k = \frac{1}{ta} \times \frac{\kappa_0 - \kappa_t}{\kappa_t - \kappa_\infty} \qquad (5)$$

Transform from Equation (5) into:

$$\kappa_t = \frac{\kappa_0 - \kappa_t}{kat} + \kappa_\infty \qquad (6)$$

Mapping κ_t to $\frac{\kappa_0 - \kappa_t}{t}$, k value can be obtained from the slope m of the line:

$$m = \frac{1}{ka} \quad \boxed{\text{or}} \quad k = \frac{1}{ma} \qquad (7)$$

The relationship between the reaction rate constant k and temperature T(K) generally conforms to Arrhenius equation, i.e.

$$\frac{d\ln k}{dT}=\frac{E_a}{RT^2}$$

If the reaction rate constants k(T_2) and k(T_1) at different temperatures are known, the activation energy E_a and the half-life of the reaction can be calculated according to Arrhenius equation.

$$\ln\frac{k(T_2)}{k(T_1)}=\frac{E_a}{R}\left(\frac{1}{T_1}-\frac{1}{T_2}\right) \tag{8}$$

The calculated E_a is the apparent activation energy of the reaction.

Instruments and Reagents

Instruments:1DDS-11A conductivity meter, 1 set of constant temperature tank, 1 set of electromagnetic stirrer and stirrer, 1 sandwich conductance bottle (as shown in Figure 5-15), 2 conical bottles with plug (250mL), 1 beaker (250mL), 1 fixed clock and 2 pipettes (20mL).

Reagents: NaOH solution ($0.02mol\cdot L^{-1}$, $0.01mol\cdot L^{-1}$) and $CH_3COOC_2H_5$ solution ($0.02mol\cdot L^{-1}$).

Experimental Content

1. **Understand and be familiar with the construction and use of DDS-11A conductivity meter.**

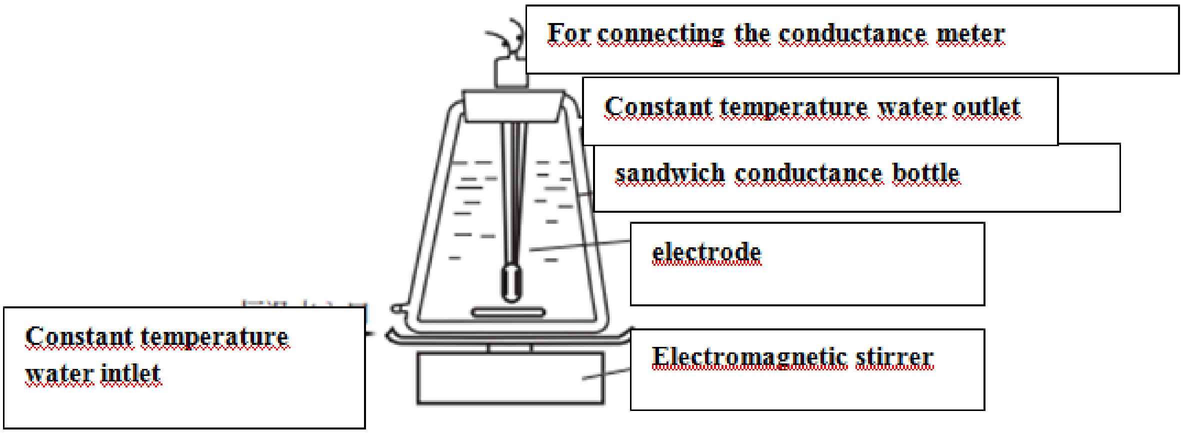


Figure 33. Experiment Device

2. Measure K_0

Adjust the bath water temperature to 25°C and connect the water inlet and outlet of the thermostat and the sandwich conductivity bottle with a hose. Take the appropriate amount the NaOH solution of $0.01mol\cdot L^{-1}$and inject it into a dry, clean sandwich conductivity flask, insert into the electrode, and the solution surface must be submerged Platinum black. Circulating constant temperature water is introduced into the sandwich layer of the conductivity bottle, and electromagnetic stirring is turned on at the same time, and its conductivity is measured after 10min, which is κ_0, write down the data.

Figure 34. General view of the installation

3.Wash and dry the conductivity bottle and set aside.

4. Calculate K_t

Take 20 mL of the NaOH solution of 0.02 mol·L$^-$1, and put it into the prepared conductivity flask and start the electromagnetic stirrer;

Take 20mL of the $CH_3COOC_2H_5$ solution of0.02 mol·L^{-1} with another pipette. and place it in another prepared conductivity flask.

After 10min of constant temperature, the NaOH solution is injected into the $CH_3COOC_2H_5$ solution conductivity flask, and the timing starts when half of the injection is injected, that is the starting time of the reaction. The conductivity value is measured starting from the timing in 2min, 5min, 10min, 15min, 20min, 25min, 30min,40min and 50min, which is the κ_t value.

5.Measure κ_0 and κ_t at 35 °C in steps 2 and 4.

Data Recording and Processing

Record the measured data in the table below.

Experiment Temperature:

k_o:

Time/min	K_1	K_o-k_1	K_o-k_1/t

(1)Process the data according to the above table and make $\kappa_t - \frac{\kappa_0 - \kappa_t}{t}$ drawing at two temperatures
(2) The activation energy is obtained from the definite integral formula (8) of the Arrhenius equation.
(3) According to the half-life formula $t_{1/2}$ = kc of the secondary reaction, calculate the half-life of this reaction at 25 °C and 35 °C.

Questions

1. Why does the concentration of ethyl acetate and NaOH solution have to be dilute enough?
2. How should k value be calculated when the starting concentration of ethyl acetate and NaOH is not equal? Try to design experiments?

Experimental Guidance

1. Ethyl acetate solution should be prepared during the experiment, and should not be left for too long. The water used in the preparation of the solution should be conductive water.
2. When the conductive bottle containing ethyl acetate solution is at constant temperature, the stopper must be plugged to prevent the ethyl acetate from volatilizing and affecting its concentration.
3. When pipetting the ethyl acetate solution, act quickly.

Experiment 8. Determination of rate constant of sucrose hydrolysis

Experimental purpose

1. Understand the relationship between the concentration of each substance and the optical rotation in the sucrose conversion reaction system.
2. Determine the rate constant and half-life of the sucrose conversion reaction.
3. Understand the basic principles of polarimeter and master its use.

Experimental principles

The sucrose hydrolysis reaction is generally carried out under the catalysis of H^+, and its reaction formula is:

$$\underset{\text{sucrose}}{C_{12}H_{22}O_{11}} + H_2O \rightarrow \underset{\text{glucose}}{C_6H_{12}O_6} + \underset{\text{fructose}}{C_6H_{12}O_6}$$

Under the condition that the catalyst H^+ concentration is fixed, this reaction should be a second-order reaction, but because the reactant water exists in large amounts, the concentration of water is basically unchanged throughout the reaction and can be regarded as a constant. Therefore, this reaction can be regarded as a quasi-first-order reaction, and the reaction rate is

$$-\frac{dc}{dt}=kc \quad (1)$$

In the formula, k is the reaction rate constant; c is the concentration of the reactant sucrose at time t.

General (1) organize and accumulate points

$$\ln c=-kt+\ln c_0 \quad (2)$$

In the formula, c_0 is the initial concentration of reactants. when c = 1/2 c_0

When c_0, t is available $t_{1/2}$ represents the half-life of the reaction. From the formula (2) available

$$t_{1/2}=\frac{\ln 2}{k}=\frac{0.693}{k} \quad (3)$$

The above formula shows that the half-life of the first-order reaction only depends on the rate constant k and has nothing to do with the starting concentration. This is the first-order reaction a feature.

How to determine the sucrose concentration c at different reaction times t? In this experiment, sucrose and hydrolysate are optically active substances. However, their optical rotation abilities are different, so the change of the optical rotation of the system during the reaction can be used to measure the progress of the reaction dissolve.

The optical rotation of the liquid depends on the type and concentration of the optically active substances contained in the solution, the nature of the solvent, the thickness of the liquid layer, the wavelength and temperature of the light source related factors.

The optical rotation of a substance refers to the property that can rotate the plane of polarization of the polarized light passing through it by a certain angle. This nature substance that are qualitative are called optically active substances. Sucrose and glucose rotate the polarization plane clockwise and are called dextral substances; fructose rotating the plane of polarization counterclockwise is called a left-handed substance.

The ability of an optically active substance to rotate the polarization plane through an angle to measure, this angle is called optical rotation, represented by α. It is generally stipulated that the optical rotation of right-handed substances is positive, and that of left-handed substances luminosity is negative. The optical rotation of a solution containing optically active substances depends on the type, concentration and solvent of the optically active substances contained in the solution.

It is related to factors such as properties, liquid layer thickness, light source wavelength and temperature. Optical rotation has great differences due to different experimental conditions, so specific optical rotation is introduced [α] concept. Specific optical rotation the degree can be expressed by the following formula:

$$[\alpha]_D^t = \frac{\alpha}{lc} \qquad (4)$$

n the formula, t is the experimental temperature, ℃; D means using sodium light D line as the light source; α is the optical rotation; l is the thickness of the liquid layer (usually (in units of 10 cm); c is the concentration [commonly the mass of the substance contained in 100 mL solution (g) to represent].

From the formula (4) It can be seen that when other conditions remain unchanged, the optical rotation α is proportional to the reactant concentration c.

Right now

$$\alpha = Kc \qquad (5)$$

In the formula, K is a factor related to the optical rotation ability of the substance, liquid layer thickness, solvent properties, light source wavelength, temperature and other factors constant.

In the hydrolysis reaction of sucrose, the reactant sucrose is a right-handed substance, and its specific optical rotation $[\alpha]^{20}_D$ = 66.6 °. In product glucose is also a right-handed substance, and its specific optical rotation $[\alpha]^{20}_D$ = 52.5°;

The fructose in the product is a levorotatory substance, and its specific rotation $[\alpha]^{20}_D$ = - 91.9 °. Therefore, at the beginning of the reaction, the system was right-handed. as the hydrolysis reaction progressed, the grape sugar and fructose gradually increase. Since the left-handed degree of fructose is greater than the right-handed degree of glucose, the right-handed angle continues to decrease, and finally after passing the zero point, it becomes left-handed. Optical rotation is proportional to concentration, and the optical rotation of a solution is the sum of the optical rotations of its components like.

The optical rotation of the solution when the reaction time is 0, t and ∞ is expressed by α respectively 0, α_t, α_∞ represent, then

α_0 = K (opposite) c_0 (means sucrose is not converted) (6)
α_∞ = K (born) c_0 (indicating that sucrose has been completely converted) (7)

Mode (6) and K in formula (7) are the proportional constants of the corresponding reactants and products respectively.

$$\alpha_t = K_{反} c + K_{生} (c_0 - c) \qquad (8)$$

From the formula (6) ~ Formula (8) can be solved together:

$$c_0=\frac{\alpha_0-\alpha_\infty}{K_{反}-K_{生}}=K'(\alpha_0-\alpha_\infty) \tag{9}$$

$$c=\frac{\alpha_t-\alpha_\infty}{K_{反}-K_{生}}=K'(\alpha_t-\alpha_\infty) \tag{10}$$

General (9) and equation (10) are substituted into equation (2) to get

$$\ln(\alpha_t-\alpha_\infty)=-kt+\ln(\alpha_0-\alpha_\infty) \tag{11}$$

From the formula (11) it can be seen that the plot of ln (α_t - α_∞) versus t is a straight line, and the reaction rate can be obtained from the slope of the straight line.

The rate constant k can then be used to obtain the half-life $t_{1/2}$. This experiment uses a polarimeter to measure α_t and α_∞value, by plotting the intercept get α_0.

Instruments and reagents

Instruments:1 polarimeter, 1 volumetric flask (50 mL), 1 polarimetric tube, 1 set of thermostatic bath, 2 Erlenmeyer flasks (100 mL) only, 1 loading scale, 1 stop watch, 1 beaker (100 mL, 500 mL) each, and 2 pipettes (25 mL).

Reagents: HC l solution (2 mol· L $^{-1}$), sucrose (A.R.).

Experimental content

1. Adjust the constant temperature bath to a constant temperature of (20.0±0.1) ℃, and then connect the constant temperature water to the constant temperature polarization tube, as shown in Fig. 35.

Figure 35. Polarization tube

2. Calibration of the zero point of the polarimeter

Figure 36. Thermostatic polarimeter

Distilled water is a non-optically active substance and can be used to check the zero value of the polarimeter point. Clean the optical polarization tube, tighten the cap at one end of the tube, and inject fill the tube with distilled water so that the distilled water forms a protruding liquid surface at the mouth of the tube.

Gently push the glass cover along the mouth of the tube, and then tighten the cover. Be careful not to cause water leakage or bubbles. Do not use excessive force when operating be careful not to crush the glass pieces. Wipe the polarization tube with absorbent paper, and then wipe the glass pieces at both ends of the tube with lens cleaning paper.

The polarimeter tube put it into the polarimeter, cover the slot cover, turn on the light source, adjust the eyepiece to make the field of view clear, and then rotate the analyzer to make it visible in the field of view.

Observe three-thirds of the visual field with equal light and dark (must be measured in a dark field), record the scale reading, repeat the operation three times, and take the average. The mean value is the zero point of the polarimeter and is used to correct the systematic error of the instrument.

3. Preparation of solution

Weigh 10 g of sucrose, dissolve it in a small amount of distilled water, and pour it into a 50 mL volumetric flask to prepare a solution. If the solution is turbid, it needs to be row filtering.

4. Determination of optical rotation α_t during sucrose hydrolysis

Use a pipette to take 25 mL of sucrose solution and place it into a 50 mL polarization tube. Pipette another 25 mL 2 mol · L $^{-1}$HCl solution quickly add the sucrose solution and start timing when half of the HCl solution is added as the starting time of the reaction. Constantly shake to fully mix the solution, quickly take a small amount of the mixture to clean the polarimeter tube twice, and then fill the polarimeter tube with this mixture (Operation use the same method as distilled water), cover tightly and tighten (check for leaks and bubbles), dry and clean the glass pieces at both ends, and put them away immediately in the polarimeter, close the tank cover. Measure the optical rotation α of the solution at different times t. Since the optical rotation α_t continuously over time changes, so measure the optical rotation α of the solution at each reaction time t

Be quick and accurate when adjusting the darkness of the three-dimensional field of view to the same after that, immediately write down the time and then read the optical rotation. At regular intervals, read the optical rotation. You can measure the first optical rotation the 5th min, 10min, 15min, 20min, 30min, 50min, and 75min after the degree value are measured once each.

5. Determination of α_∞

You can place the mixture in step 4 for 48 hours and then measure its optical rotation at the same temperature, which is the α_∞ value. for to shorten the time, you can also place the remaining mixed solution in a water bath near 60°C, keep the temperature constant for 40 minutes to accelerate the reaction, and then cool it to the experimental temperature, measure its optical rotation according to the above operation, and this value can be regarded as α_∞.

Pay attention to the water bath temperature not being too high, otherwise. Otherwise, side reactions will occur and the color of the solution will turn yellow. During the heat preservation process, volatilization of the solution should be avoided to affect the concentration.

It should be noted that after measuring 30 min, the sodium light lamp should be extinguished between each measurement to avoid damage due to overheating due to long-term use is broken, but turn on the sodium light lamp 10 minutes in advance before the next measurement to stabilize the light source. In addition, after the experiment, you should wash it immediately wipe the optical cell clean to prevent it from being corroded by acid.

Data recording and processing

1. Record the experimental data in the table below.

Experimental temperature:_____

Hydrochloric acid concentration_____

zero point:_____

α_∞______ :

Reaction time	α_t	$\alpha_t - \alpha_\infty$	$\ln(\alpha_t - \alpha_\infty)$	K

2. Plot ln $(\alpha_t - \alpha_\infty)$ against t, and find the reaction rate constant k from the slope of the obtained straight line.

3. Find α_0 from the intercept.

4. Calculate the half-life $t_{1/2}$ of the sucrose hydrolysis reaction.

Questions

1. At the beginning of the reaction, why pour hydrochloric acid into the sucrose solution instead of pouring sucrose into the hydrochloric acid?
2. Why can distilled water be used to calibrate the zero point of the polarimeter during the experiment? If no calibration is performed, will the results be affected? Ring? Why?
3. What are the characteristics of the first-order reaction? Why can a pan-type balance be used to prepare a sucrose solution?
4. How to determine whether an optically rotating substance is right-handed or left-handed?

Experimental guidance

For the construction and use of the polarimeter.

Experiment 9. Determination of the Electromotive Force of Reversible Cell

Experimental purpose

1. Determining the electromotive force of Cu-Zn galvanic cells and the electrode potential of Cu and Zn electrodes.
2. Learning the preparation methods of several electrodes and salt bridges.
3. Mastering the measurement principle of reversible cell electromotive force and the operation technology of UJ34 potentiometer.

Experimental principles

Electrochemistry is part of physical chemistry focused on the study of thermodynamic and kinetic processes in the solutions of electrolytes and melts, and in processes related to electrolysis and those occurring in galvanic cells.

When a metal is placed in a solution of its own salt one of the two processes are possible [13].

1) Metals atoms (Me) go into solution in the form of ions (Me^{n+})

$$Me \rightarrow Me^{n+} + n\bar{e} \text{ (oxidation)}$$

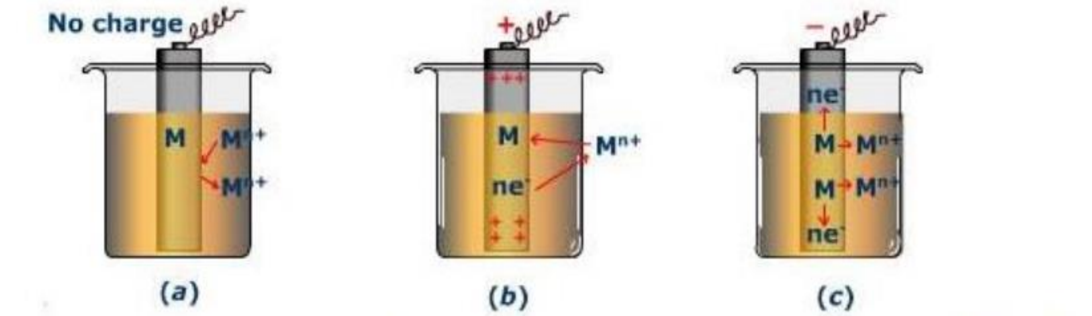
2) Metals ions from solution may deposit on the metal

$$Me^{n+} + n\bar{e} \rightarrow Me \text{ (reduction)}$$

Electrode is the material: a metallic rod / bar / strip which conducts electrons into and out of a solution. An electrode in an electrochemical cell is referred to as either an anode or a cathode:

- Anode: is the electrode where oxidation takes place, it is the negative (-) electrode (example, active metals are soluble in water).
- Cathode: is the electrode where reduction takes place, it is the positive (+) electrode (example, passive metals are insoluble in water) [13].

Three possibilities when a strip of metal M is placed in a solution containing its ions M^{n+}



If metal is oxidized in solution
$Me - ne^- \rightarrow Me^{n+}$
(dissolved in water), it is anode

If metal is reduced in solution
$Me^{n+} + ne^- \rightarrow Me$
(insoluble in water), it is cathode

A metal ion M^{n+} collides
with the electrode,
and undergoes no change

Thus a layer of positive or negative ions formed all around the metal. This layer is called Helmholtz electrical double layer, which results a potential difference between the metal ions and the solution. At equilibrium, the potential difference becomes a constant value which is known as the electrode potential of the metal. Electrode potentials are denoted by the letter E and are of two types:

1. Single electrode potential (E): it is the tendency of a metallic electrode to lose or gain electrons when it is in contact with a solution of its own salt [13].
2. Standard electrode potential (E^o): it is the tendency of a metallic electrode to lose or gain electrons when it is in contact with a solution of its salt of 1M concentration at 25°C and atmospheric pressure (1 atm = 101,325 kPa) [13].

Example (fig. 36): When a Zn rod of any length is dipped in $ZnSO_4$ solution, standard electrode is formed and the potential developed is called standard zinc electrode potential (E^0_{Zn}). The standard zinc electrode is represented as $Zn/Zn^{2+}(1M)$ [13].

The mass of solid zinc decreases during the course of the reaction. This suggests that the reaction occurring in that half-cell is $Zn(s) \rightarrow Zn^{2+}(aq) + 2\bar{e}$

The following process occurring in the cell:

$$Zn(s) \rightarrow Zn^{2+}(aq) + 2\bar{e} \text{ (anode-oxidation)}$$

$$2H^+(aq) + 2\bar{e} \rightarrow H_2\uparrow \text{ (cathode-reduction)}$$

And the measured cell voltage is 0,76 Volts [13].

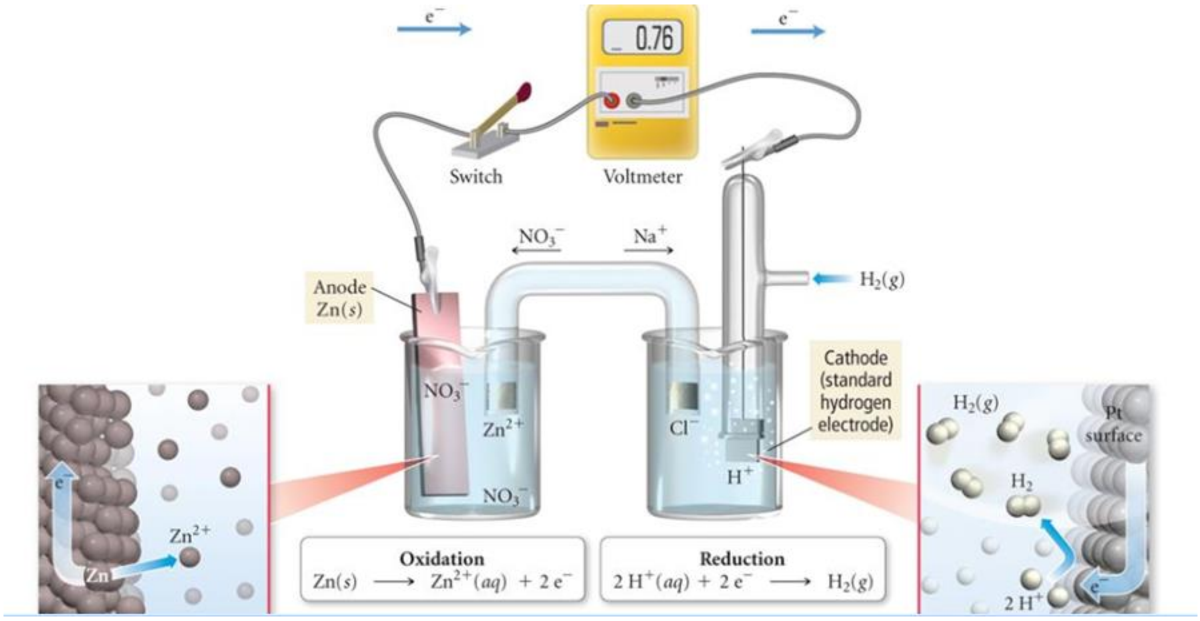


Figure 37. Measurement of electrode potential of zinc electrode

The standard electromotive force (EMF) of the cell as (for standard hydrogen electrode $E^0 = 0$ Volts):

$$E^0_{Zn\ cell} = E^0_{cathode} - E^0_{anode}$$
$$0{,}76\ V = 0 - E^0_{Zn\ cell}$$
$$E^0_{Zncell} = -0{,}76\ V$$

The functional dependence of potential of any electrode system on these factors is given by Nernst equation:

$$E_{Me} = E^{\circ} + \frac{RT}{nF}\cdot \ln Me^{+n}$$

Where E^0 - the standard electrode potential (volts), n - charge transfer number, T - absolute/Kelvin temperature, R - universal Gas Constant (8,314 kPa/mol·L), F - Faraday`s Constant (96500 Coulomb).

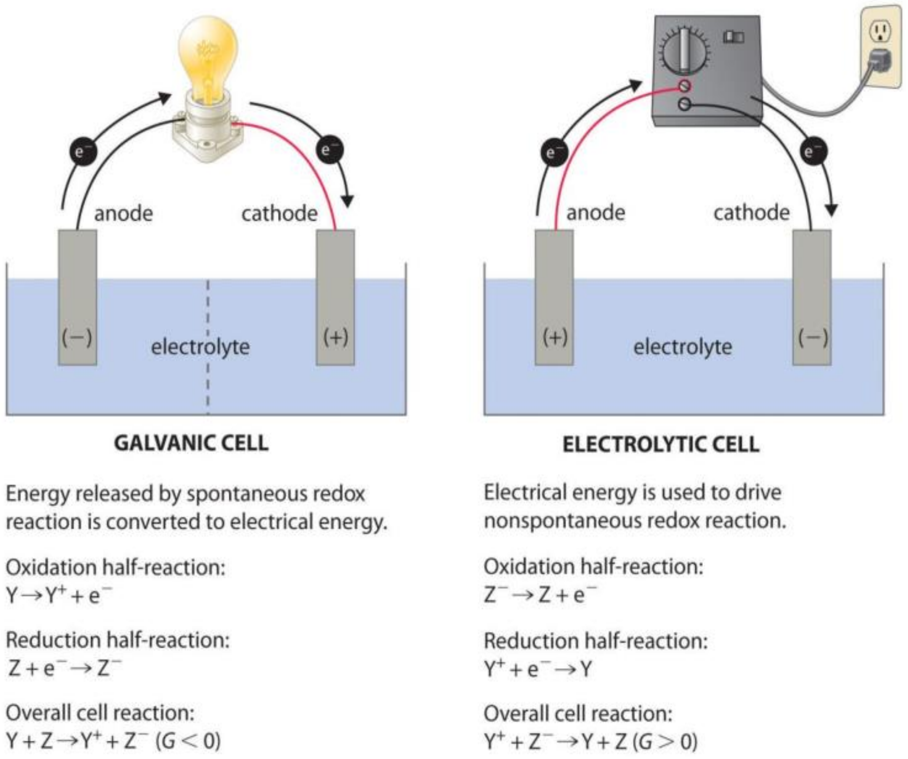
An electrochemical cell is a system consisting of electrodes that dip into an electrolyte and in which a chemical reaction either uses or generates an electric current, these reactions are called electrochemical, they happen on electrode-solution surface [13].

There are two types of electrochemical cells: galvanic cells and electrolytic cells.

Electrochemical cells that use a spontaneous oxidation-reduction reaction to generate an electric current are known as galvanic or voltaic cells.

Other type of electrochemical cell such an electrolytic cell, in it extra energy, usually in the form of voltage from a power supply, is used to drive a nonspontaneous reaction, or a reaction that would not normally occur by itself.

The two types of cells do have some things in common. Each has electrodes, which are the objects that conduct the electricity between the cell and the surroundings. Each electrode is submerged in an electrolyte– or a mixture/solution that contains ions. The electrolyte may be involved in the reaction or the ions may be used to carry the charge. An electrode is identified as being the anode or the cathode.



GALVANIC CELL

Energy released by spontaneous redox reaction is converted to electrical energy.

Oxidation half-reaction:
$Y \rightarrow Y^+ + e^-$

Reduction half-reaction:
$Z + e^- \rightarrow Z^-$

Overall cell reaction:
$Y + Z \rightarrow Y^+ + Z^-$ $(G < 0)$

ELECTROLYTIC CELL

Electrical energy is used to drive nonspontaneous redox reaction.

Oxidation half-reaction:
$Z^- \rightarrow Z + e^-$

Reduction half-reaction:
$Y^+ + e^- \rightarrow Y$

Overall cell reaction:
$Y^+ + Z^- \rightarrow Y + Z$ $(G > 0)$

In practice we distinguish reversible and irreversible electrochemical cells.

The main point of difference between an electrolytic cell and a galvanic cell (electrochemical cell) are: a galvanic cell converts chemical energy into electrical energy but an electrolytic cell converts electrical energy into chemical energy [13].

Any device that converts chemical energy into electrical energy is called a chemical power source (or cell, galvanic cell). A cell consists of two electrodes and an electrolyte solution connecting the two electrodes.

Insert the Zn sheet into the $ZnSO_4$ solution to form the Zn electrode, and insert the Cu sheet into the $CuSO_4$ solution to form the Cu electrode. Connecting the two electrodes with a salt bridge (which is filled with electrolyte) becomes a Cu-Zn cell. Reversible cells should meet the following conditions:

(1) The cell reaction is reversible, that is, the cell electrode reaction is reversible.

(2) No irreversible liquid junctions are allowed in the cell.

(3) The cell must work in a reversible condition, that is, the charging and discharging process must be carried out in an equilibrium state, that is, the current allowed through the cell is infinitesimal.

Therefore, the above conditions should be met when preparing reversible cells and measuring the electromotive force of reversible cells, and in the measurement of low accuracy, salts with close positive and negative ion migration numbers are commonly used to form “salt bridges” to eliminate the liquid junction potential. Measuring electromotive force with a potentiometer can also meet the condition that the current through the cell is infinitely small.

Instruments and reagents

Instruments: potentiometer, Cu electrode, Zn electrode, calomel electrode

Reagents:0,1 mol/L $CuSO_4$ solution, 0,1 mol/L $ZnSO_4$ solution, 1 mol/L KCl solution, agar, KCl analytical pure

Experimental content

1. Preparation of zinc electrode: the zinc electrode is polished with sandpaper to remove the oxide layer on the zinc electrode and rinsed with distilled water and taken out and rinsed with distilled water again. The processed zinc electrode is inserted directly into the 0.100 mol/L $ZnSO_4$ solution in a beaker**.**

2. Preparation of copper electrode: the copper electrode is lightened with sandpaper, then rinsed with distilled water, and inserted into the 0.100 mol/L $CuSO_4$ solution in a beaker

3. Prepare saturated KCl salt bridge: in 1 Erlenmeyer flask, add 3g agar and 100 mL of distilled water, heat on a water bath until completely dissolved, then add 30 g KCl, stir KCl thoroughly, use a dropper to load this solution into a U-shaped tube while hot, stand and wait for the agar to coagulate before use.

4. Measure the electromotive force of the Cu-Zn cell: as shown in Fig. 37, connect the Cu electrode and the Zn electrode with a salt bridge and connect the Zn electrode (negative electrode) of the cell with the negative terminal post of the potentiometer, and connect the Cu electrode (positive electrode) with the positive terminal post of the potentiometer.

Measure the electromotive force E every 3 minutes. Within 10 minutes, the change is less than 0.5 mV, and its electromotive force can be considered stable, and the average of the last several consecutive measurements is taken as the electromotive force of the cell

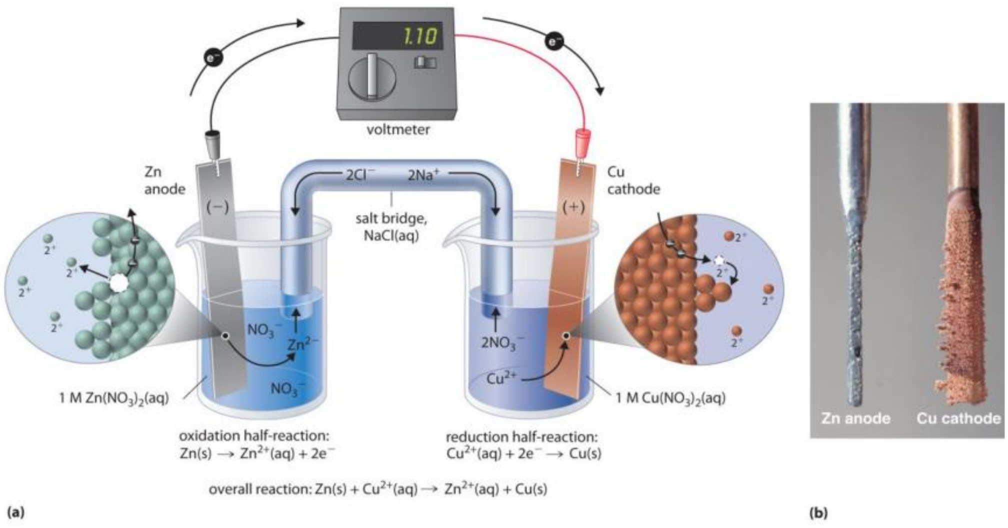


Figure 38. Schematic diagram of Zn-Cu cell

5. Measure the electromotive force of the cell composed of Zn electrode and calomel electrode: connect the two electrodes with a salt bridge, and measure its electromotive force by the same method as (4). In this cell, the calomel electrode is the positive electrode and the Zn electrode is the negative electrode.

6. Measure the electromotive force of the cell composed of Cu electrode and calomel electrode: connect the two electrodes with a salt bridge, and measure its electromotive force in the same way as (4). In this cell, the calomel electrode is the negative electrode and the Cu electrode is the positive electrode.

Data Recording and Processing

1. Data Recording:

- Cell electromotive Force:

Cell	Cell Reaction	Electromotive Force			Average
		Measured Value			
		(1)	(2)	(3)	
Cu-Zn Cell					
Cu-Calomel					
Zn-Calomel					

Electrode Potential and Standard Electrode Potential:

Electrode Name	Electrode Potential, E	
	Experimental Value	Theoretical Value
Cu		
Zn		
Calomel potential = 0,2412 V		

2. Calculate the electrode potential of the Zn electrode, $E_{Zn/Zn2+}$

3. Calculate the electrode potential of the Cu electrode, $E_{Cu2+/Cu}$

4. Calculate the electromotive force (EMF) of the Cu-Zn cell.

The EMF of the cell is:

$$EMF = E_{Cu2+/Cu} - E_{Zn/Zn2+}$$

5. Reference value: $E^0_{Cu2+/Cu} = 0{,}337$ V and $E^0_{Zn/Zn2+} = -0{,}763$ V.

Calculate the relative error.

Questions

1. What is the basic principle of electromotive force measurement by elimination method? Why can’t the electromotive force of the cell be accurately determined with a voltmeter?
2. What are the functions of potentiometer, standard cell, galvanometer and working cell?
3. How to maintain the galvanometer?
4. What are the conditions for the reference electrode? What does it do?
5. What does the salt bridge do? What electrolyte should be selected for salt bridge?

Experimental guidance

When measuring the cell electromotive force, although the elimination method is used, before the elimination point, the measurement loop will have current through, so you cannot keep pressing the electric button during the measurement process, otherwise there will always be current through the loop and the electrode will be polarized and the concentration of the solution will also change so the measured is not reversible cell electromotive force, therefore, you should press it to adjust until equilibrium.

Experiment 10. Preparation and performance testing of ion-selective electrodes

Experimental purpose

1. Understand the basic performance of ion selective electrodes and their testing methods
2. Master a preparation method of solid iodine electrode.

Experimental principles

Ion selective electrodes are developed on the basis of membrane electrodes. After the mid-1960s, with the scientific research with the development of work and production construction, new electrode membrane materials have been successfully developed, the variety of electrodes has increased sharply, and their performance has also improved a lot.

Great improvements have been made, and application methods have also been extensively developed. Ion selective electrodes have developed into important components in the field of electrochemical analysis.

Branch, in industrial field testing, environmental pollution investigation, laboratory sample analysis, medical clinical testing and solution theory research, etc. It has been widely used.

There are many varieties of ion-selective electrodes, each with its own characteristics. The iodide ion selective electrode used in this experiment is based on Ag - AgI based all-solid-state electrode.

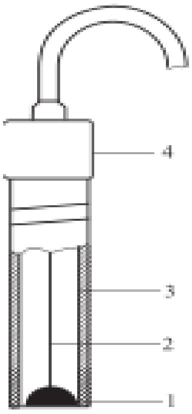


Figure 39. Schematic all solid state Ion selective electrode

1. Basic structure of iodide ion selective electrode

The structure of a commonly used all-solid-state ion-selective electrode is shown in Fig. 39. 1 is the diaphragm, which is the sensitive element of the electrode; 2 is the electrode lead; 3 is the polyvinyl chloride hard tube; 4 is the electrode cap.

The structures of ion-selective electrodes prepared by different preparation methods are not exactly the same. Preparation sub-selective electrodes often use thermal decomposition method and electroplating method. In this experiment, the electroplating method was used to coat the silver electrode. A thin film of silver iodide forms on the surface.

2. Response characteristics of ion selective electrodes

Ion selective electrode is an electrochemical sensitive element based on potential response. When the electrode is inserted into a solution containing the ions to be measured, a specific potential response value is generated at the membrane-liquid interface.

The relationship between potential response value and ion activity can be described by the Nernst equation. Like taking the iodine electrode as an example and using the calomel electrode as the reference electrode, the electromotive force of the battery can be expressed as:

$$E = E_{0} - \frac{RT}{F} \ln a_{I^-}$$

It is known that γ is the activity coefficient, so

$$a_{I^-} = \gamma c_{I^-}$$

In experiments, the testing method of fixed ionic strength is usually used. At this time, γ can be regarded as a fixed value, then the formula can be rewritten as:

$$E = E_0' - \frac{RT}{F} \ln c_{I^-}$$

It can be seen from the above formula that E and lnc there is a linear relationship between them. As long as the difference is detected the electromotive force value E when the value is E - lnc. Figure, a straight line can be obtained within a certain concentration range. From the figure, the linear range of the iodine electrode can be obtained.

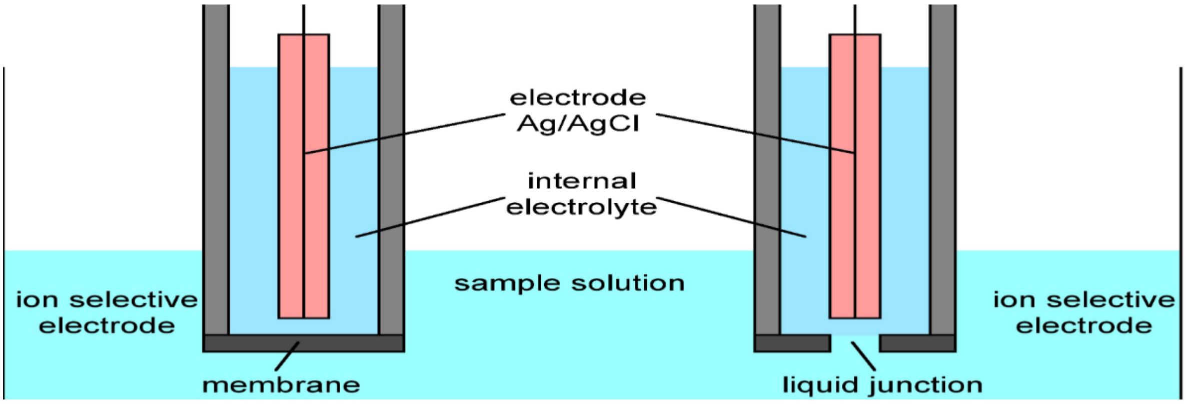


Figure 40. The principle of operation of the ion selective electrode

3. Selectivity and selection coefficient of ion selective electrode

Ion-selective electrodes have specific response characteristics for the ions to be measured, but other ions can still interfere with them to a certain extent. disturb. The selectivity of electrodes is often expressed by the selection coefficient.

If A and B represent the ions to be measured and the interfering ions respectively, then

$$E = E_0 \pm \frac{RT}{n_A F} \ln(a_A + k_{A/B} \cdot a_B^{n_A/n_B})$$

In the formula, n_A and n_B represent the charges of A and B ions respectively; $k_{A/B}$ is the selectivity coefficient of the electrode for B ions. Mode the “-” and “+” in are applicable to anion and cation selective electrodes respectively.

It can be seen that the smaller $k_{A/B}$ means, the smaller the interference of B ions on the measured ions, which also means the selection of electrodes. Usually $k_{A/B}$ value is less than 10^{-3} means there is no obvious interference.

Instruments and reagents

Instruments: pHS - 3 digital acidometer, 1 calomel electrode, 1 transistor regulated power supply, 1 platinum electrode, magnetic stirring 1 instrument, 1 metallographic sandpaper, 1 silver electrode.

Reagents: KNO_3 solution (0.2 mol· L^{-1}), potassium iodide (A.R.).

Experimental content

1. Preparation of potassium iodide standard solution

Accurately weigh 4.123 g of potassium iodide, dissolve it in twice-distilled water, transfer it to a 250 mL volumetric flask, and dilute it to the mark. KI solution is of $0.1\ mol \cdot L^{-1}$.

Figure 41. Steps of preparation of potassium iodide standard solution

2. Preparation of ion-selective electrode

The silver electrode was polished with metallographic sandpaper, and then carefully polished with lens paper. Then use the silver electrode as the anode and the platinum electrode as the cathode. Electrolysis in $0.1\ mol \cdot L^{-1}$ KI solution, voltage 1.2~1.4 V (current density is about $0.2\ mA \cdot cm^{-2}$), electrolysis for 1 h.

After the electrolysis is completed, take out the electrode, wash it, soak it in double distilled water, and use it after 1 hour.

3. Preparation of standard curve

Use KI standard solution stepwise dilution method to prepare concentrations of $2\times10^{-2}\ mol \cdot L^{-1}$, $2\times10^{-3}\ mol \cdot L^{-1}$, $2\times10^{-4}\ mol \cdot L^{-1}$, $2\times10^{-5}\ mol \cdot L^{-1}$ of KI solution.

Take 20 mL of the KI solution prepared above and 20 mL $0.2\ mol \cdot L^{-1}$ of KNO_3 solution, mix in a 50 mL beaker. Close and place the beaker on the magnetic stirrer. With the calomel electrode as the reference electrode and the ion selective electrode as the other electrode, the two electrodes. Connect it to the acidimeter, then insert it into the solution in the beaker, stir thoroughly, and read the potential value when equilibrium is reached. Measured by proceed from thin to thick.

Figure 42. General view of the installation.

Data Recording:

# solution	Concentration of KI solution, mol·L^{-1}	lg C	the potential value, V
	0.1		
	2×10^{-2}		
	2×10^{-3}		
	2×10^{-4}		
	2×10^{-5}		

4. Determination of selection coefficient

Add 10 mL 0.001 mol·L^{-1} KI solution to the beaker, measure the potential value, and then add 2 mL to the beaker 0.1 mol·L^{-1} K_2SO_4 solution, read the potential value, and then add 2 mL 0.1 mol·L^{-1} successively K_2SO_4 solution, straight until the potential value changes significantly.

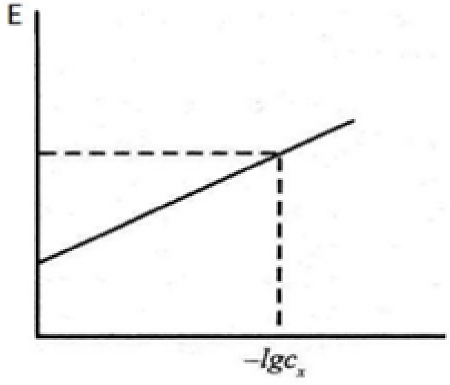


Figure 43. General view of the graph for concentration determination

Data recording and processing

1. Record each measurement data in a list.
2. Use E versus - lgc_{KI} to plot, that is, the standard curve, to determine tl concentration range.
3. Draw a graph of E versus - lgc_{K2SO4} and find out c_K at the turning point from the graph $c_{K2SO4} \cdot c_{KI} / c_{K2SO4}$

The value can be approximated by is 1×10^{-3} mol·L^{-1} I^- condition I^-/SO_4^{2-} value.

Questions

1. Why should the ionic strength of the solution be adjusted? How to adjust it?
2. Why should the test work be carried out under stirring conditions?
3. In this experiment, when the selective electrode is prepared by electrolysis, what reactions occur on the anode and cathode?

Experiment 11. The solubility of insoluble salt was determined by electrical conductivity

Experimental purpose

1. Master the principle and method of measuring solubility of insoluble salt by conductivity method.
2. Determine the solubility of $CaCO_3$ at 25 ^{o}C.
3. Consolidate the understanding of the basic concept of solution conductance and the application of conductance measurement.

Experimental principle

$BaSO_4$, AgCl, $PbSO_4$, $CaCO_3$ and other insoluble salts in water solubility is very small, with the general analysis method is difficult to directly determine its solubility, but insoluble salt in water trace dissolved part is completely ionized, so you can use the method of conductivity measurement to easily find out its solubility.

The electrolyte solution is the second type of conductor, conducting electricity through the migration of positive and negative ions. The magnitude of its ability to conduct electricity is often expressed in terms of conductance (reciprocal of resistance):

$$G = \frac{1}{R} \quad (1)$$

Where: *G* is the conductance, the unit is Siemens (S); *R* is resistance, in ohms (Ω).

According to the relation of conductance to resistance, there are:

$$\boldsymbol{G} = \boldsymbol{\kappa}\left(\frac{\boldsymbol{A}}{\boldsymbol{L}}\right) \quad (2)$$

$$\boldsymbol{\kappa} = \boldsymbol{G}\left(\frac{\boldsymbol{L}}{\boldsymbol{A}}\right) \quad (3)$$

Where is the conductivity or specific conductivity (reciprocal of resistivity), which is equivalent to the conductivity, when the cross-sectional area of the conductor A=l m^{2} and the length L = 1 m, κin S$\cdot$m^{-1}.

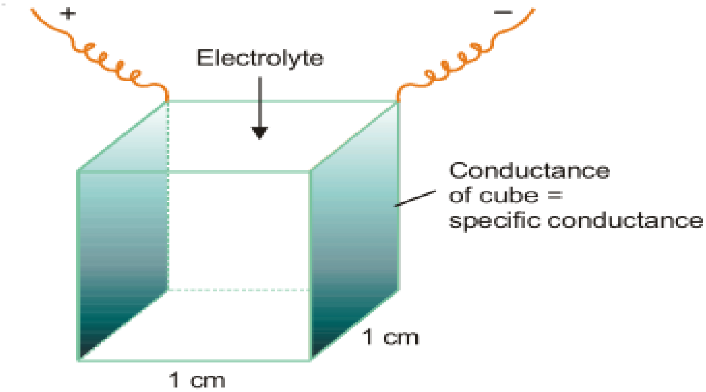


Figure 44. Diagrammatic illustration of definition of specific conductance

In order to compare the conductivity of electrolyte solutions, molar conductivityΛ_m is often used. A solution containing l mol of electrolyte is placed between two parallel electrodes that are lm apart, and the conductivity of this solution is called molar conductivityΛ_m and is expressed in $S \cdot m^2 \cdot mol^{-l}$.

At a certain temperature, the concentration of the electrolyte solution $c(mol \cdot m^{-3})$, the relationship between Λ_m and the conductivity is:

$$\Lambda_m = \kappa / c \quad (4)$$

The solubility of insoluble salt can be calculated by equation (4).

Since the solubility of insoluble salt is very small, salt is a strong electrolyte, the saturated solution can be regarded as infinite dilution, and the molar conductivityΛ_m of the saturated solution can be approximately considered to be equal to the molar conductivity of the infinite dilution solution Λ_m^∞ of the insoluble salt.

$$\Lambda_m \approx \Lambda_m^\infty$$

According to Kohlrausch's law of independent motion of ions, the infinite diluted molar conductivity of $CaCO_3$ can be obtained by adding and:

$$\Lambda_m^\infty = \Lambda_m^\infty(\frac{1}{2}Ca^{2+}) + \Lambda_m^\infty(\frac{1}{2}CO_3{}^{2-})$$

The conductivity is obtained by determining the solution conductance *G* and substituting it into equation (3). For the determined conductance electrode, it is a constant, called the conductance cell constant.

A simple type of conductance cell used in the laboratory is shown in Fig. 45. The electrodes fitted in the cell are made of platinum plates coated with platinum black. These are welded to platinum wires fused in two thin glass tubes. The contact with copper wires of the circuit is made by dipping them in mercury contained in the tubes.

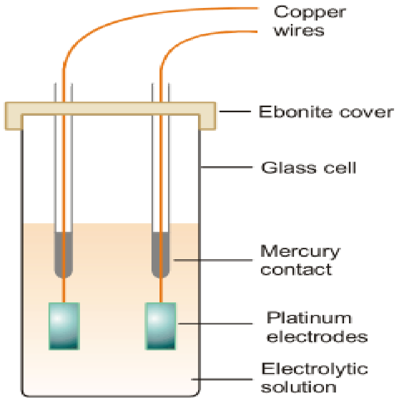


Figure 45. A Conductance cell

The conductance cell constant can be determined by measuring the conductance of an electrolyte solution with known conductivity.

The known conductivity of the standard KCl solution into the conductivity cell, measured its conductivity *G*, from the known conductivity can be calculated the conductivity cell constant.

It must be noted that since the solubility of insoluble salt in water is very small, the conductivity of its saturated solution is actually the sum of the conductivity of salt and water:

$$\kappa_{solution} = \kappa_{salt} + \kappa_{water} \quad (5)$$

Therefore, it is necessary to measure the conductivity of the water κ_{water} used to prepare the solution κ_{salt} before it can be obtained.

After the measurement, the saturation concentration κ_{salt} c (mo1·m^{-3}) of insoluble salt in water at the temperature can be obtained by substituting formula (4), and the solubility of the insoluble salt can be obtained by conversion.

The temperature has an effect on the conductance, and the experiment should be carried out at constant temperature. The conductivity meter is used to measure the conductivity directly in this experiment.

Instruments and reagents

Instruments:1 super constant temperature tank, 1 conductance electrode, a DDS-IIA type conductivity meter, 1 conductance meter, 1 conical bottle (200 mL), 2 thermostatic bottles.

Reagents: standard potassium chloride solution (0.02mo1 ·L^{-1}), $CaCO_3$ (A.R.).

Experimental content

1. **Adjust the thermostat temperature to 25^{0}C±0.14^{0}C**.

2. **Determine the conductivity of redistilled water**

Soak the conductance electrode and thermostatic bottle with distilled water and redistilled water 2 to 3 times each in turn, and add about 30 mL of redistilled water to the thermostatic bottle. After constant temperature, the conductivity of redistilled water at 25^{0}C was measured three times, and the average value was taken.

3. **Determine the conductivity of $CaCO_3$ solution**

Take about 1 g of solid $CaCO_3$ into 400 mL conical bottle, add about l00 mL of heavy distilled water, shake and heat to boil, pour away the clear liquid to remove soluble impurities. Repeat in the same way twice. Add about 100 mL more heavily distilled water and heat it to a boil to dissolve it thoroughly. Reduce to room temperature naturally, then place in a thermostatic tank for 20 min to allow the solids to settle. Soak the conductive electrode and the conical bottle with the upper clarified saturated $CaCO_3$ solution for 2 to 3 times, and then fill the conical bottle with an appropriate amount of clarified saturated $CaCO_3$ solution. After constant temperature, measure its conductivity, change the solution and measure twice, and take the average value.

Figure 46. A DDS-IIA type conductivity meter

Data recording and processing

1. List records the measurement data.
2. From the measured conductance of 0.02 mol·L^{-1} standard KCl solution and the conductivity of the standard solution at the experimental temperature, the conductivity cell constant is calculated with formula (3).
3. The conductivity of $CaCO_3$ calculated by formula (5) according to the conductivity of redistilled water and saturated $CaCO_3$ solution.
4. Refer to the table to calculate the infinite dilution molar conductivity of $CaCO_3$ at 25^{0}C.
5. Calculate the concentration (mol·m^{-3}), solubility (mol· L^{-1}) and solubility product of saturated $CaCO_3$ solution by equation (4).

Questions

1. How to determine the conductivity cell constant?
2. Why choose 100 Hz alternating current instead of direct current when using Wheatstone bridge method to determine the conductance of solution?
3. Why should the saturated $CaCO_3$ solution be boiled several times?
4. Can I use ordinary distilled water when preparing saturated $CaCO_3$ solution?

Experimental guidance

Distilled water is a poor conductor of electricity, but because of dissolved impurities, such as carbon dioxide and soluble solid impurities, its conductance is very large, affecting the results of conductance measurement, so it is necessary to treat distilled water and use heavy distilled water. This experiment requires that the conductivity of water should be less than 1×10^{-4} S·m^{-1}.

Experiment 12. Determination of specific surface (solution adsorption method)

Experimental purpose

1. Understand the principles and methods of determining specific surface area by solution adsorption method.
2. Use methylene blue aqueous solution to determine the specific surface of activated carbon.

3. Master the method of measuring the concentration of solutions using spectrophotometry.

Experimental principles

When a solid surface is exposed to a gas or a liquid, molecules from the gas or the solution phase accumulate or concentrate at the surface.

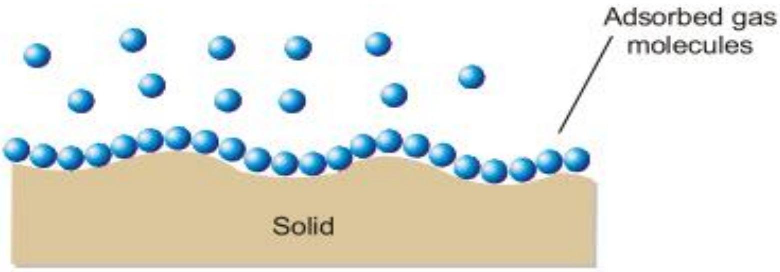


Figure 47. Adsorption of a gas at a solid surface.

The phenomenon of concentration of molecules of a gas or liquid at a solid surface is called adsorption. The substance that deposits at the surface is called Adsorbate and the solid on whose surface the deposition occurs is called the Adsorbent.

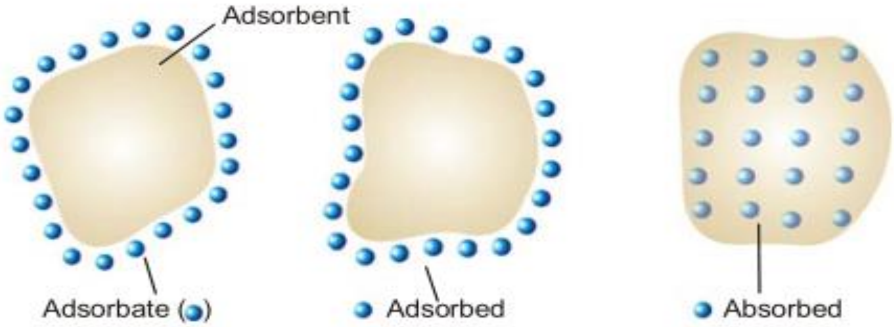


Figure 48. Adsorption absorption (illustration).

The adsorption of a gas on a solid adsorbent in a closed vessel is a reversible process. The amount of the gas adsorbed depends on equilibrium pressure (P) and temperature.

The relationship between the equilibrium pressure of a gas and its amount adsorbed on the solid adsorbent at any constant temperature is called an Adsorption isotherm. It may be given in the form of an equation or graphical curve.

Porous or finely divided solid substances can also adsorb dissolved substances from solution. Thus activated charcoal is used to remove colored impurities from solutions. Charcoal will also adsorb many dyestuffs. When a solution of acetic acid is shaken with activated charcoal, part of the acid is removed by adsorption and concentration of solution is decreased. Again, precipitates obtained in qualitative analysis often act as absorbents. For example, magnesium hydroxide when precipitated in the presence of the dye-stuff magneson forms a blue ‘lake’.

Adsorption from solution generally follows the same principles as laid down for adsorption of gases by solids and is subject to the same factors. Thus,

(1) Some adsorbents specifically adsorb certain solutes more effectively than others.

(2) An increase of temperature decreases the extent of adsorption.

(3) An increase in surface area increases the extent of adsorption.

(4) Adsorption of solutes also involves the establishment of an equilibrium between the amount adsorbed and the concentration of the solute in solution.

The precise mechanism of adsorption from solution is not clear. However, there is a limit to the adsorption by a given mass of adsorbent and hence possibly adsorption takes place unless a unimolecular layer is formed.

There are many methods for measuring the specific surface of solids. Currently, BET method, chromatography and electron microscopy are commonly used. These methods generally require complex equipment or long experimental times. The solution adsorption method determines the specific surface of solid substances, although it does not.

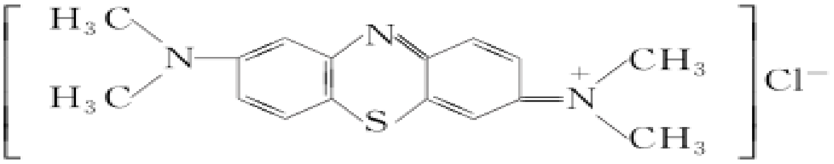
If the above method is accurate, the instrument is simple, the operation and calculation are convenient, and multiple samples can be measured at the same time, it is the best way to understand solids a simple approach to adsorbent performance.

For the basic principles of the solution adsorption method, please refer to the chapter on surface chemistry in the physical chemistry textbook.

The adsorption of water-soluble dyes has been used to determine the solid specific surface. Among all dyes, methylene blue has the largest adsorption tendency towards. Studies have shown that within a certain concentration range, the adsorption of methylene blue by most solids is monolayer adsorption, which is consistent with Langmuir type. But when the concentration of the original solution is too high, multi-molecular layer adsorption will occur; when the concentration of the solution is too small, adsorption cannot reach saturation. Therefore, the original solution concentration and the equilibrium concentration after adsorption equilibrium should be selected in an appropriate range.

The degree is 2g · about L^{-1}, the concentration of the equilibrium solution is not less than 1g· L^{-1}.

The molecular structure of methylene blue is



There are three orientations for the adsorption of methylene blue:

- plane adsorption, with a projected area of 1.35 nm^2;
- side adsorption, with a projected area of 1.35 $nm^2$is 0.75 nm^2;
- terminal group adsorption, projected area is 0.395 nm^2.

For non-graphite activated carbons, methylene blue may not it is plane adsorption but end group adsorption. According to the experimental results, in the case of monolayer adsorption, 1 mg methylene blue the area covered can be calculated as 2.45 m^2.

Methylene blue aqueous solution is blue, and its concentration before and after adsorption can be measured by spectrophotometry.

Spectrophotometry is a powerful tool for analyzing substances that have color. According to the Beer's law, the absorbance (*A*) value is directly proportional to the molar concentration of the absorbing substance if the same cuvet and a set wavelength are used for all measurements. A plot of absorbance versus concentration (*A*~*c*) data is linear; a calculated slope and absorbance data can be used to determine the molar concentration of the same absorbing species in a solution of unknown concentration from the linear relationship.

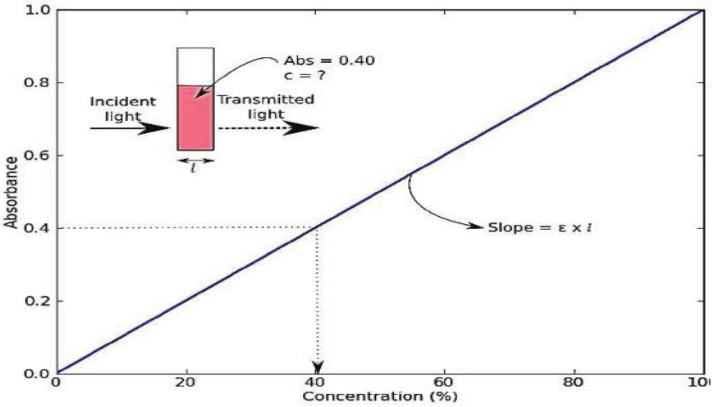


Figure 49. Find out a concentration of unknown solution

The solid adsorbent in solution the adsorption amount Γ when the liquid reaches adsorption equilibrium can be calculated based on the change in solution concentration before and after adsorption:

$$\Gamma = \frac{(c_0 - c)V}{m} \qquad (1)$$

In the formula, Γ is the adsorption capacity, usually referring to the amount of solute adsorbed per gram of adsorbent; c_0 is the concentration of the solution before adsorption (original concentration); c is the concentration of the solution when reaching adsorption equilibrium (equilibrium concentration); V is the volume of the solution; m is the mass of the adsorbent.

If the area covered by 1 mg methylene blue is calculated as 2.45 m^2, then the specific surface of the adsorbent a_s (m^2 ·g^{-1}) is:

$$a_s = \frac{(c_0 - c)V}{m} \times 2.45 \qquad (2)$$

In the formula, c_0 and c are the original solution concentration and equilibrium solution concentration respectively, g· L^{-1}; V is the volume of the solution, mL; m is the mass of adsorbent, g.

The results measured by the solution adsorption method have certain errors, mainly because the non-spherical adsorbates appear on the surface of various adsorbents during adsorption.

The orientation of the surfaces is not the same, so the projected area of each adsorbate molecule is very different. Therefore, the value measured by the solution adsorption method should be calibrated by other methods. The measurement error of the solution adsorption method is generally about 10%.

Instruments and reagents

Instruments:722 type spectrophotometer 1 set, volumetric flask (1000 mL 2 pieces, 100 mL 3 pieces), 1 Conisoscillator, colorimetry tube (25 mL) 2 pieces. Reagents: methylene blue solution (2g·L $^{-1}$) (original solution), standard methylene blue solution (0.1g· L^{-1}).

Experimental content

1. Activation sample

Place the granular activated carbon in a porcelain crucible, activate it in a muffle furnace at 500°C for 1 hour, and then cool it in a desiccator spare.

2. Solution adsorption

Accurately weigh about 0.1 g of activated carbon, add it to a 25 mL colorimetric tube, and then add 2g· L $^{-1}$ of base blue solution (original solution) 25 mL. Place the colorimetric tube on a conisoscillator and oscillate for 4 to 6 hours (or shake it thoroughly and then place it for a while) week) to allow adsorption to reach equilibrium.

3. Preparation of methylene blue standard solution

Use a pipette to transfer 0.5 mL, 1 mL, and 2 mL of the standard methylene blue solution into a 100 mL volumetric flask, and dilute to scale.

4. Treatment of original solution and equilibrium solution

In order to accurately measure the concentration of the original solution, take 2g · L $^{-1}$ original solution 1 mL put into 1000 mL volumetric flask and diluted to scale. Take 1 mL of the equilibrium solution that reaches adsorption equilibrium, add it to a 1000 mL volumetric flask, and dilute to the mark.

Figure 50. Treatment of original solution and equilibrium solution

5. Measure optical density

Select 665 nm as the working wavelength, use distilled water as the blank solution, and measure the three prepared methylene blue standards respectively.

Figure 51. Measure optical density

The optical density value of the solution, as well as the optical density value of the diluted original solution and the equilibrium solution. Measurement of optical density of each sample the measurement should be repeated three times and the average value should be taken.

Data recording and processing

1. Record each measurement data in a list.
2. Plot the concentration of three methylene blue standard solutions against the optical density, and the straight line obtained is the working curve.
3. Use the optical density of the diluted original solution and the optical density of the diluted equilibrium solution to find the corresponding values on the working curve. The concentration value is then multiplied by the dilution factor 1000 to obtain the original solution concentration c_0 and equilibrium solution concentration.
4. Calculate the specific surface area of activated carbon according to formula (2).

Questions

1. Why is the original solution concentration of methylene blue chosen at 2g·around L^{-1}, after adsorption equilibrium, the concentration of methylene blue solution should be 1g· around L^{-1}?
2. If the concentration of methylene blue solution is too low after adsorption equilibrium, how should the experimental operation be changed?

GLOSSARY

A

absorption

The disappearance of a wave into a medium. When a wave is absorbed, the energy transferred by the wave is converted into another form of energy, usually thermal energy.

acceleration

The rate at which velocity changes over time.

acid

A substance that can donate a proton to another substance and has a pH below 7.

acoustics

The scientific study of sound; the behavior of sound waves inside a space.

air resistance

The fluid friction due to air.

alloy

A solid mixture composed of a metal and one or more other substances.

alternating current AC

Electric current that reverses direction at regular intervals.

ampere amp

The unit of measurement of electric current, which is equal to one coulomb per second. The number of amps flowing through a circuit equals the circuit's amperage.

amplification

The strengthening of an electrical signal, often used to increase the intensity of a sound wave.

amplitude

The maximum distance that a disturbance causes a medium to move from its rest position; the distance between a crest or trough of a wave and line through the center of a wave.

analog

Represented by a continuous but varying quantity, such as a wave. In electronics, analog information is represented by a continuous but varying electrical signal.

atom

The smallest particle of an element that has the chemical properties of that element.

atomic mass

The average mass of the atoms of an element.

atomic mass number

The total number of protons and neutrons in an atom's nucleus.

atomic number

The number of protons in the nucleus of an atom.

B

base

A substance that can accept a proton from another substance and has a pH above 7.

Bernoulli’s principle

A statement that describes the effects of movement on fluid pressure. According to this principle, an increase in the speed of the motion of a fluid decreases the pressure within the fluid.

binary code

A coding system in which information is represented by two figures, such as 1 and 0.

bioluminescence

The production of light by living organisms.

boiling

A process by which a substance changes from its liquid state to its gas state. The liquid is heated to a specific temperature at which bubbles of vapor form within the liquid.

boiling point

The temperature at which a substance changes from its liquid state to its gas state through boiling.

bond energy

The amount of energy in a chemical bond between atoms.

buoyant force

The upward force on objects in a fluid; often called buoyancy.

C

calorie

The amount of energy needed to increase the temperature of one gram of water by one degree Celsius.

carbohydrate

A type of carbon-based molecule in living things. Carbohydrates include sugars and starches used for energy or as structural materials, Carbohydrate molecules contain carbon, hydrogen, and oxygen atoms.

catalyst

A substance that increases the rate of a chemical reaction but is not consumed in the reaction.

centripetal force

Any force that keeps an object moving in a circle.

chemical change

A change of one substance into another substance.

chemical formula

An expression that shows the number and types of atoms joined in a compound.

chemical property

A characteristic of a substance that describes how it can form a new substance.

chemical reaction
The process by which chemical changes occur. In a chemical reaction, atoms are rearranged, and chemical bonds are broken and formed.
circuit
A closed path through which charge can flow.
coefficient
The number before a chemical formula that indicates how many molecules are involved in a chemical reaction.
collision
A situation in which two objects in close contact exchange energy and momentum.
compound
A substance made up of two or more different types of atoms bonded together.
compound machine
A machine that is made up of two or more simple machines.
computer
An electronic device that processes digital information.
concave
Curved inward toward the center, like the inside of a spoon.
concentration
The amount of solute dissolved in a solvent at a given temperature.
condensation
The process by which a gas becomes a liquid.
conduction
The process by which energy is transferred from a warmer object to a cooler object by means of physical contact.
conductor
1. A material that transfers energy easily.
2. A material that transfers electric charge easily.
convection
A process by which energy is transferred in gases and liquids, occurring when a warmer, less dense area of gas or liquid is pushed up by a cooler, denser area of the gas or liquid.
convex
Curved outward, like the underside of a spoon.
cornea
A transparent membrane that covers the eye.
covalent bond
A pair of electrons shared by two atoms.
crest
The highest point, or peak, of a wave.
cycle
n. A series of events or actions that repeat themselves regularly; a physical and/or chemical process in which one material continually changes locations and/or forms. Examples include the water cycle, the carbon cycle, and the rock cycle.
v. To move through a repeating series of events or actions.

D

data

Information gathered by observation or experimentation that can be used in calculating or reasoning. Data is a plural word; the singular is datum.

decibel dB

The unit used to measure the intensity of a sound wave.

degree

Evenly divided units of a temperature scale.

density

A property of matter representing the mass per unit volume.

diffraction

The spreading out of waves as they pass through an opening or around the edges of an obstacle.

diffuse reflection

The reflection of parallel light rays in many different directions.

digital

Represented by numbers. In electronics, digital information is represented by the numbers 1 and 0, signaled by a circuit that is either on or off.

dilute

adj. Having a low concentration of solute.

v. To add solvent in order to decrease the concentration of a solution.

direct current DC

Electric current that flows in one direction only.

Doppler effect

The change in perceived pitch that occurs when the source or the one who hears the sound is moving.

E

echolocation

The sending out of high-pitched sound waves and the interpretation of the returning echoes.

efficiency

The percentage of the input work done on a machine that the machine can return in output work. A machine’s output work divided by its input work and multiplied by 100.

electric cell

A device that produces electric current using the chemical or physical properties of different materials. A battery consists of two or more cells linked together.

electric charge

A property that allows one object to exert an electric force on another object without touching it. Electric charge can be positive or negative: positive charge is a property of the proton, while negative charge is a property of the electron.

electric current

A continuous flow of electric charge, which is measured in amperes.

electric field

An area surrounding a charged object, within which the object can exert an electric force on another object without touching it.

electric potential

The amount of potential energy per unit charge that a static charge or electric current has. Electric potential is measured in volts and is often called voltage.

electric power

The rate at which electrical energy is generated from, or converted into, another source of energy, such as kinetic energy.

electromagnet

A magnet that consists of a piece of iron or steel inside a coil of current-carrying wire.

electromagnetic spectrum EM spectrum

The range of all electromagnetic frequencies, including the following types (from lowest to highest frequency): radio waves, microwaves, infrared light, visible light, ultraviolet light, x-rays, and gamma rays.

electromagnetic wave EM wave

A type of wave, such as a light wave or radio wave, that does not require a medium to travel; a disturbance that transfers energy through a field.

electromagnetism

Magnetism that results from the flow of electric charge.

electron

A negatively charged particle located outside an atom’s nucleus. An electron is about 2000 times smaller than either a proton or neutron.

electronic

adj. Operating by means of an electrical signal. An electronic device is a device that uses electric current to represent coded information.

n. An electronic device or system, such as a computer, calculator, CD player, or game system.

element

A substance that cannot be broken down into a simpler substance by ordinary chemical changes. An element consists of atoms of only one type.

endothermic reaction

A chemical reaction that absorbs energy.

energy

The ability to do work or to cause a change. For example, the energy of a moving bowling ball knocks over pins; energy from food allows animals to move and to grow; and energy from the Sun heats Earth’s surface and atmosphere, which causes air to move.

energy efficiency

A measurement of usable energy after an energy conversion; the ratio of usable energy to the total energy after an energy conversion.

enzyme

A type of protein that is a catalyst for chemical reactions in living things.

evaporation

A process by which a substance changes from its liquid state to its gas state by random particle movement. Evaporation usually occurs at the surface of a liquid over a wide range of temperatures.

exothermic reaction

A chemical reaction that releases energy.

experiment

An organized procedure to study something under controlled conditions.

F

fiber optics

Technology based on the use of laser light to send signals through transparent wires called optical fibers. This technology is often used in communications.

field

An area around an object where the object can apply a force-such as gravitational force, magnetic force, or electrical force-on another object without touching it.

fluid

A substance that can flow easily, such as a gas or a liquid.

fluorescence

A phenomenon in which a material absorbs electromagnetic radiation of one wavelength and gives off electromagnetic radiation of a different wavelength.

focal length

The distance from the center of a convex lens to its focal point.

focal point

The point at which parallel light rays reflected from a concave mirror come together; the point at which parallel light rays refracted by a convex lens come together.

force

A push or a pull; something that changes the motion of an object.

freezing

The process by which a substance changes from its liquid state into its solid state.

freezing point

The temperature at which a substance changes from its liquid state to its solid state through freezing.

frequency

The number of waves that pass a fixed point in a given amount of time, usually one second; the number of cycles per unit time.

friction

A force that resists the motion between two surfaces in contact.

fulcrum

A fixed point around which a lever rotates.

G

gamma rays

Part of the electromagnetic spectrum that consists of waves with the highest frequencies; electromagnetic waves with frequencies ranging from more than 1019 hertz to more than 1024 hertz.

gas

Matter with no definite volume and no definite shape. The molecules in a gas are very far apart, and the amount of space between them can change easily.

generator

A device that converts kinetic energy, or the energy of motion, into electrical energy. Generators produce electric current by rotating a magnet within a coil of wire or rotating a coil of wire within a magnetic field.

gravity

The force that objects exert on each other because of their mass.

grounding

The creation of a harmless, low-resistance path-a ground-for electricity to follow. Grounding is an important electrical safety procedure.

group

A vertical column in the periodic table of the elements. Elements in a group have similar properties.

H

half-life

The amount of time it takes for half of the nuclei of a radioactive isotope to decay into atoms of another element.

heat

1. The flow of energy from an object at a higher temperature to an object at a lower temperature.
2. Energy that is transferred from a warmer object to a cooler object.

hertz Hz

The unit used to measure frequency. One hertz is equal to one complete cycle per second.

horizontal

Parallel to the horizon; level.

horsepower hp

The unit of measurement of power for engines and motors. One horsepower equals 745 watts.

hydrocarbon

A compound that contains only carbon and hydrogen.

hypothesis

A tentative explanation for an observation or phenomenon. A hypothesis is used to make testable predictions.

I

image

A picture of an object formed by rays of light.

incandescence

1. The production of light by materials having high temperatures.
2. Light produced by an incandescent object.

inclined plane

A simple machine that is a sloping surface, such as a ramp.

induction

The build-up of a static charge in an object when the object is close to, but not touching, a charged object.

inertia

The resistance of an object to a change in the speed or the direction of its motion.

infrared light

Part of the electromagnetic spectrum that consists of waves with frequencies between those of microwaves and visible light.

inorganic compound

A compound that is not considered organic. All compounds that do not contain carbon are inorganic, as are some types of carbon-containing compounds.

insulator

1. A material that does not transfer energy easily.
2. A material that does not transfer electric charge easily.

intensity

The amount of energy of a wave, per wavelength. Intensity is associated with the amplitude of a sound wave and with the quality of loudness produced by the sound wave.

interference

The meeting and combining of waves; the adding or subtracting of wave amplitudes that occurs as waves overlap.

ion

An atom or group of atoms that has a positive or negative electric charge.

ionic bond

The electrical attraction between a negative ion and a positive ion.

isomer

Any of two or more compounds that contain the same atoms but that have different structures.

isotope

An atom of one element that has a different number of neutrons than another atom of the same element.

J

joule (jool)

A unit used to measure energy and work. One calorie is equal to 4. 18 joules of energy; one joule of work is done when a force of one newton moves an object one meter.

K

kilowatt kW

A unit of measurement for power equal to 1000 watts.

kilowatt-hour kWh

The unit of measurement for electrical energy equal to one kilowatt of power over a one-hour period.

kinetic energy

The energy of motion. A moving object has the most kinetic energy at the point where it moves the fastest.

kinetic theory of matter

A theory stating that all matter is made of particles in motion.

L

laser

A device that produces an intense, concentrated beam of light that can be brighter than sunlight. Lasers are often used in medicine and communications.

law

In science, a rule or principle describing a physical relationship that always works in the same way under the same conditions. The law of conservation of energy is an example.

law of conservation of energy

A law stating that no matter how energy is transferred or transformed, it continues to exist in one form or another.

law of conservation of mass

A law stating that atoms are not created or destroyed in a chemical reaction.

law of conservation of momentum

A law stating that the amount of momentum a system of objects has does not change as long as there are no outside forces acting on that system.

law of reflection

A law of physics stating that the angle at which light strikes a surface (the angle of incidence) equals the angle at which it reflects off the surface (the angle of reflection).

lens

A transparent optical tool that refracts light.

lever

A solid bar that rotates, or turns, around a fixed point (fulcrum); one of the six simple machines.

lipid
A type of carbon-based molecule in living things. Lipids include fats and oils used for energy or as structural materials.

liquid
Matter that has a definite volume but does not have a demit shape. The molecules in a liquid are close together but not bound to one another.

longitudinal wave
A type of wave in which the disturbance moves in the same direction that the wave travels.

luminescence
The production of light without the high temperatures needed for incandescence.

M

machine
Any device that makes doing work easier.

magnet
An object that attracts certain other materials, particularly iron and steel.

magnetic domain
A group of atoms whose magnetic fields align, or point in the same direction. Magnetic materials have magnetic domains, whereas nonmagnetic materials do not.

magnetic field
An area surrounding a magnet within which the magnet can exert a force. Magnetic fields are concentrated into a pattern of lines that extend from the magnet’s north pole to its south pole.

magnetic pole
One of two ends of a magnet where the magnetic force is the strongest. Every magnet has two poles.

magnetism
The force exerted by a magnet. Opposite poles of two magnets attract, or pull together, whereas like poles of two magnets repel, or push apart.

mass
A measure of how much matter an object is made of.

matter
Anything that has mass and volume. Matter exists ordinarily as a solid, a liquid, or a gas.

mechanical advantage
The number of times a machine multiplies the input force; output force divided by input force

mechanical energy
A combination of the kinetic energy and potential energy an object has.

mechanical wave
A wave, such as a sound wave or a seismic wave, that transfers kinetic energy through matter.

medium
A substance through which a wave moves.
melting
The process by which a substance changes from its solid state to its liquid state.
melting point
The temperature at which a substance changes from its solid state to its liquid state through melting.
metal
An element that tends to be shiny, easily shaped, and a good conductor of electricity and heat.
metallic bond
A certain type of bond in which nuclei float in a sea of electrons.
metalloid
An element that has properties of both metals and nonmetals.
meter
The international standard unit of length, about 39.37 inches.
microwaves
Part of the electromagnetic spectrum that consists of waves with higher frequencies than radio waves, but lower frequencies than infrared waves.
mixture
A combination of two or more substances that do not combine chemically but remain the same individual substances. Mixtures can be separated by physical means.
molecule
A group of atoms that are held together by covalent bonds so that they move as a single unit.
momentum
A measure of mass in motion. The momentum of an object is the product of its mass and velocity.
monomer
One of many small, repeating units linked together to form a polymer.
motion
A change of position over time.

N

nanotechnology
The science and technology of building electronic circuits and devices from single atoms and molecules.
net force
The overall force acting on an object when all of the forces acting on it are combined.
neutral
Describing a solution that is neither an acid nor a base. A neutral solution has a pH of 7.

neutron
A particle that has no electric charge and is located in an atom’s nucleus.
Newton’s first law
A scientific law stating that objects at rest remain at rest, and objects in motion remain in motion with the same velocity, unless acted on by an unbalanced force.
Newton’s second law
A scientific law stating that the acceleration of an object increases with increased force and decreases with increased mass.
Newton’s third law
A scientific law stating that every time one object exerts a force on another object, the second object exerts a force that is equal in size and opposite in direction back on the first object.
nonmetal
An element that is not a metal and has properties generally opposite to those of a metal.
nucleic acid
One of several carbon-based molecules that carry an organism’s genetic code. One of the nucleic acids DNA- contains the information needed to construct proteins.
nucleus
The central region of an atom where most of the atom’s mass is found in protons and neutrons.
0
ohm Q
The unit of measurement for electrical resistance.
Ohm’s law
The mathematical relationship among current, voltage, and resistance, expressed in the formula I = V/R (current = voltage/resistance).
optics
The study of light, vision, and related technology.
orbit
The elliptical path one celestial body follows around another celestial body. An object in orbit has a centripetal force acting on it that keeps the object moving in a circle or other ellipse.
organic compound
A compound that is based on carbon.

P
parallel circuit
A circuit in which current follows more than one path. Each device that is wired in a parallel circuit has its own path to and from the voltage source.
particle
A very small piece of matter, such as an atom, molecule, or ion.

pascal Pa
The unit used to measure pressure. One Pascal is the pressure exerted by one newton of force on an area of one square meter, or one N/m^2.

Pascal's principle
A statement that says when an outside pressure is applied at any point to a fluid in a container, that pressure is transmitted throughout the fluid with equal strength.

period
A horizontal row in the periodic table of the elements. Elements in a period have varying properties.

periodic table
A table of the elements, arranged by atomic number, that shows the patterns in their properties.

pH
The concentration of hydrogen ions in a solution; a measurement of acidity.

photosynthesis
In green plants, the endothermic process in which light is absorbed and used to change carbon dioxide and water into glucose and oxygen.

physical change
A change in a substance that does not change the substance into a different one.

physical property
A characteristic of a substance that can be observed without changing the identity of the substance.

pitch
The quality of highness or lowness of a sound. Pitch is associated with the frequency of a sound wave- the higher the frequency, the higher the pitch.

plastic
A polymer that can be molded or shaped.

polar covalent bond
The unequal sharing of electrons between two atoms that gives rise to negative and positive regions of electric charge.

polarization
A way of filtering light so that all of the waves vibrate in the same direction.

polymer
A very large carbon-based molecule made of smaller, repeating units.

position
An object's location.

potential energy
Stored energy; the energy an object has due to its position, molecular arrangement, or chemical composition.

power
The rate at which work is done.

precipitate

n. A solid substance that forms as a result of a reaction between chemicals in two liquids.

v. To come out of solution.

pressure

A measure of how much force is acting on a certain area; how concentrated a force is. Pressure is equal to the force divided by area.

primary colors

Three colors of light-red, green, and blue-that can be mixed to produce all possible colors.

primary pigments

Three colors of substances--cyan, yellow, and magenta that can be mixed to produce all possible colors.

prism

An optical tool that uses refraction to separate the different wavelengths that make up white light.

product

A substance formed by a chemical reaction. A product is made by the rearrangement of atoms and bonds in reactants.

protein

A macromolecule in living things that is made of smaller molecules called amino acids.

proton

A positively charged particle located in an atom’s nucleus.

pulley

A wheel with a grooved rim that turns on an axle; one of the six simple machines.

pupil

The circular opening in the iris of the eye that controls how much light enters the eye.

R

radiation

Energy that travels across distances in the form of electromagnetic waves.

radioactivity

The process by which the nucleus of an atom of an element releases energy and particles.

radio waves

The part of the electromagnetic spectrum that consists of waves with the lowest frequencies.

reactant

A substance that is present at the beginning of a chemical reaction and is changed into a new substance.

reactive

Likely to undergo a chemical change.

reference point
A location to which another location is compared.

reflection
The bouncing back of a wave after it strikes a barrier.

refraction
The bending of a wave as it crosses the boundary between two mediums at an angle other than 90 degrees.

regular reflection
The reflection of parallel light rays in the same direction.

resistance
The property of a material that determines how easily a charge can move through it. Resistance is measured in ohms.

resistor
An electrical device that slows the flow of charge in a circuit.

resonance
The strengthening of a sound waves when it combines with an object's natural vibration.

respiration
The exothermic process by which living things release energy from glucose and oxygen and produce carbon dioxide and water.

retina
A light-sensitive membrane at the back of the inside of the eye.

robot
A machine that works automatically or by remote control.

S

saturated
Containing the maximum amount of a solute that can be dissolved in a particular solvent at a given temperature and pressure.

scattering
The spreading out of light rays in all directions as particles reflect and absorb the light.

screw
A simple machine that is an inclined plane wrapped around a cylinder. A screw can be used to raise and lower weights as well as to fasten objects.

seconds
A unit of time equal to one-sixtieth of a minute.

series circuit
A circuit in which current follows a single path. Each device that is wired in a series circuit shares a path to and from the voltage source.

short circuit
An unintended and undesired path connecting one part of a circuit with another.

simple machine
One of the basic machines on which all other mechanical machines are based. The six simple machines are the lever, inclined plane, wheel and axle, pulley, wedge, and screw.

solar cell
A type of technology in which light-sensitive materials convert sunlight into electrical energy.

solid
Matter that has a definite shape and a definite volume. The molecules in a solid are in fixed positions and are close together.

solubility
The amount of solute that dissolves in a certain amount of a solvent at a given temperature and pressure to produce a saturated solution.

solute
In a solution, a substance that is dissolved in a solvent.

solution
A mixture of two or more substances that is identical throughout; a homogeneous mixture.

solvent
In a solution, the substance that dissolves a solute and makes up the largest percentage of a solution.

sonar
Instruments that use echolocation to locate objects underwater; acronym for “sound navigation and ranging.”

sound
A type of wave that is produced by a vibrating object and that travels through matter.

specific heat
The amount of energy required to raise the temperature of one gram of a substance by one degree Celsius.

speed
A measure of how fast something moves through a particular distance over a definite time period. Speed is distance divided by time.

states of matter
The different forms in which matter can exist. Three familiar states are solid, liquid, and gas.

static charge
The buildup of electric charge in an object caused by the uneven distribution of charged particles.

sublimation
The process by which a substance changes directly from its solid state to its gas state without becoming a liquid first.

subscript
A number written slightly below and to the right of a chemical symbol that shows how many atoms of an element are in a compound.

substance
Matter of a particular type. Elements, compounds, and mixtures are all substances.

suspension
A mixture in which the different parts are identifiable as separate substances; a heterogeneous mixture.
system
A group of objects or phenomena that interact. A system can be as simple as a rope, a pulley, and a mass. It also can be as complex as the interaction of energy and matter in the four parts of the Earth system.

T
technology
The use of scientific knowledge to solve problems or engineer new products, tools, or processes.
temperature
A measure of the average amount of kinetic energy of the particles in an object.
terminal velocity
The final, maximum velocity of a falling object.
theory
In science, a set of widely accepted explanations of observations and phenomena. A theory is a well-tested explanation that is consistent with all available evidence.
thermal energy
The energy an object has due to the motion of its particles; the total amount of kinetic energy of particles in an object.
thermometer
A device for measuring temperature.
transformer
A device that uses electromagnetism to increase or decrease voltage. A transformer is often used in the distribution of current from power plants.
transmission
The passage of a wave through a medium.
transverse wave
A type of wave in which the disturbance moves at right angles, or perpendicular, to the direction in which the wave travels.
trough
The lowest point, or valley, of a wave valle El puntomasbajo de unaonda.

U
ultrasound
Sound waves with frequencies above 20,000 hertz, the upper limit of typical hearing levels in humans, used for medical purposes, among other things.
ultraviolet light
The part of the electromagnetic spectrum that consists of waves with frequencies higher than those of visible light and lower than those of x-rays.

V

vacuum

A space containing few or no particles of matter.

variable

Any factor that can change in a controlled experiment, observation, or model.

velocity

A speed in a specific direction.

vertical

Going straight up or down from a level surface.

vibration

A rapid, back-and-forth motion.

visible light

The part of the electromagnetic spectrum that consists of waves detectable by the human eye.

volt V

The unit of measurement for electric potential, which is equal to one joule per coulomb.

The number of volts of an electric charge equals the charge's voltage.

volume

An amount of three-dimensional space, often used to describe the space that an object takes up.

W

watt W

The unit of measurement for power, which is equal to one joule of work done or energy transferred in one second. For example, a 75 W light bulb converts electrical energy into heat and light at a rate of 75 joules per second.

wave

A disturbance that transfers energy from one place to another without requiring matter to move the entire instance.

wavelength

The distance from one wave crest to the next crest; the distance from any part of one wave to the identical part of the next wave.

wedge

A simple machine that has a thick end and a thin end. A wedge is used to cut, split, or pierce objects, or to hold objects together.

weight

The force of gravity on an object.

wheel and axle

A simple machine that is a wheel attached to a shaft, or axle.

work

The use of force to move an object over a distance.

X, Y, Z

x-rays

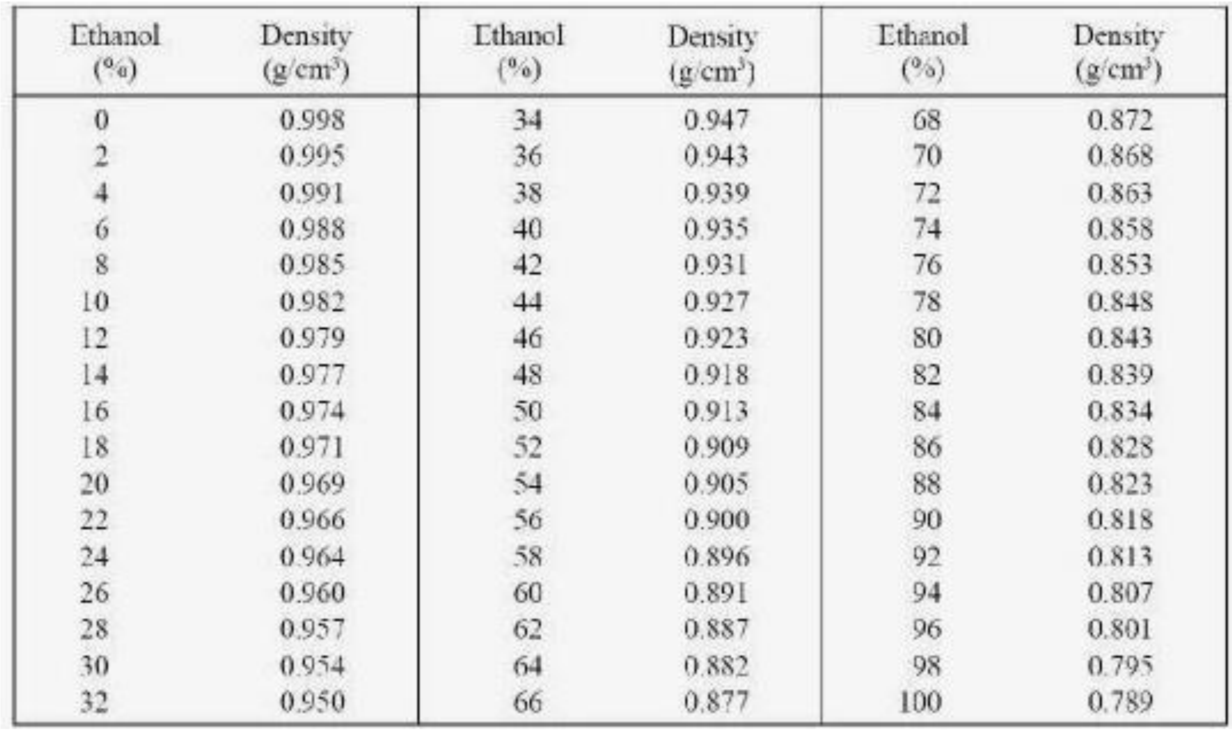
The part of the electromagnetic spectrum that consists of waves with high frequencies and high energies; electromagnetic waves with frequencies ranging from more than 1016 hertz to more than 1021 hertz.

APPENDICES

Properties of water

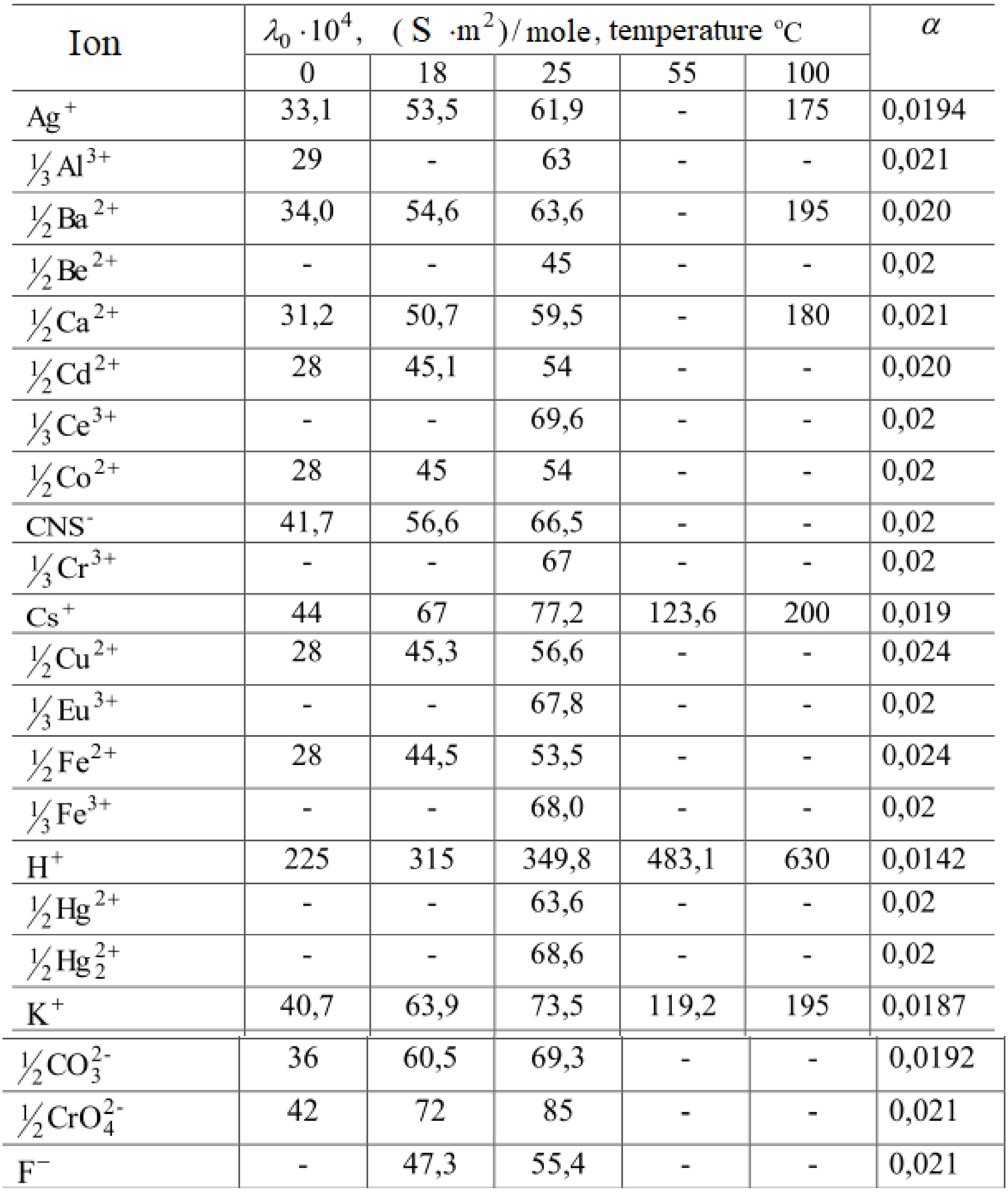
Density: 0.99984 g/cm^3 at 0°C
0.99970 g/cm^3 at 10°C
0.99821 g/cm^3 at 20°C
0.98803 g/cm^3 at 50°C
0.95840 g/cm^3 at 100°C
Enthalpy (heat) of vaporisation: 45.054 kJ/mol at 0°C
43.990 kJ/mol at 25°C
42.482 kJ/mol at 60°C
40.657 kJ/mol at 100°C
Surface tension: 74.23 J/m^2 at 10°C
71.99 J/m^2 at 25°C
67.94 J/m^2 at 50°C
58.91 J/m^2 at 100°C
Viscosity: 1.793 mPa·s at 0°C
0.890 mPa·s at 25°C
0.547 mPa·s at 50°C
0.282 mPa·s at 100°C
Ion-product constant, K_w(or K_{H2O}): 1.15×10^{-15} at 0°C
1.01×10^{-14} at 25°C
5.31×10^{-14} at 50°C
5.43×10^{-13} at 100°C
Specific heat (C_s): 4.2176 J/(g·°C) at 0°C
4.1818 J/(g·°C) at 20°C
4.1806 J/(g·°C) at 50°C
4.2159 J/(g·°C) at 100°C

Density of ethanol

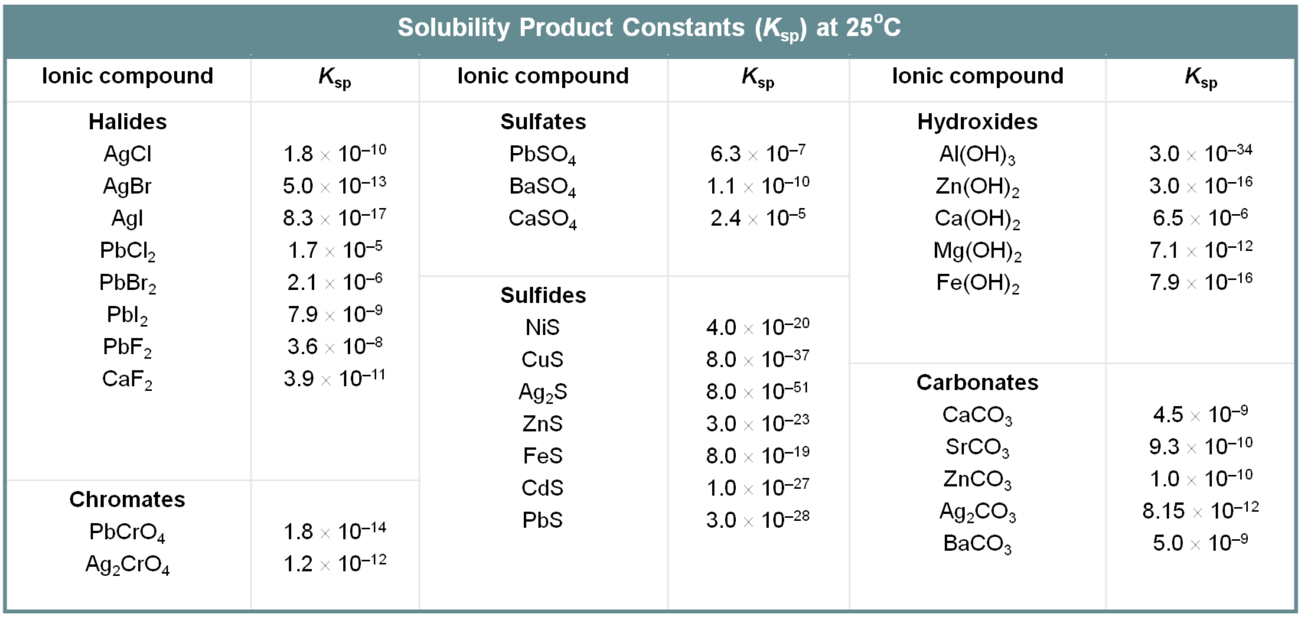


Ethanol (%)	Density (g/cm^3)	Ethanol (%)	Density (g/cm^3)	Ethanol (%)	Density (g/cm^3)
0	0.998	34	0.947	68	0.872
2	0.995	36	0.943	70	0.868
4	0.991	38	0.939	72	0.863
6	0.988	40	0.935	74	0.858
8	0.985	42	0.931	76	0.853
10	0.982	44	0.927	78	0.848
12	0.979	46	0.923	80	0.843
14	0.977	48	0.918	82	0.839
16	0.974	50	0.913	84	0.834
18	0.971	52	0.909	86	0.828
20	0.969	54	0.905	88	0.823
22	0.966	56	0.900	90	0.818
24	0.964	58	0.896	92	0.813
26	0.960	60	0.891	94	0.807
28	0.957	62	0.887	96	0.801
30	0.954	64	0.882	98	0.795
32	0.950	66	0.877	100	0.789

Specific electrical conductivity for 0.01M, 0.02M, 0.1M aqueous solution of potassium chloride, Ohm^{-1}·cm^{-}



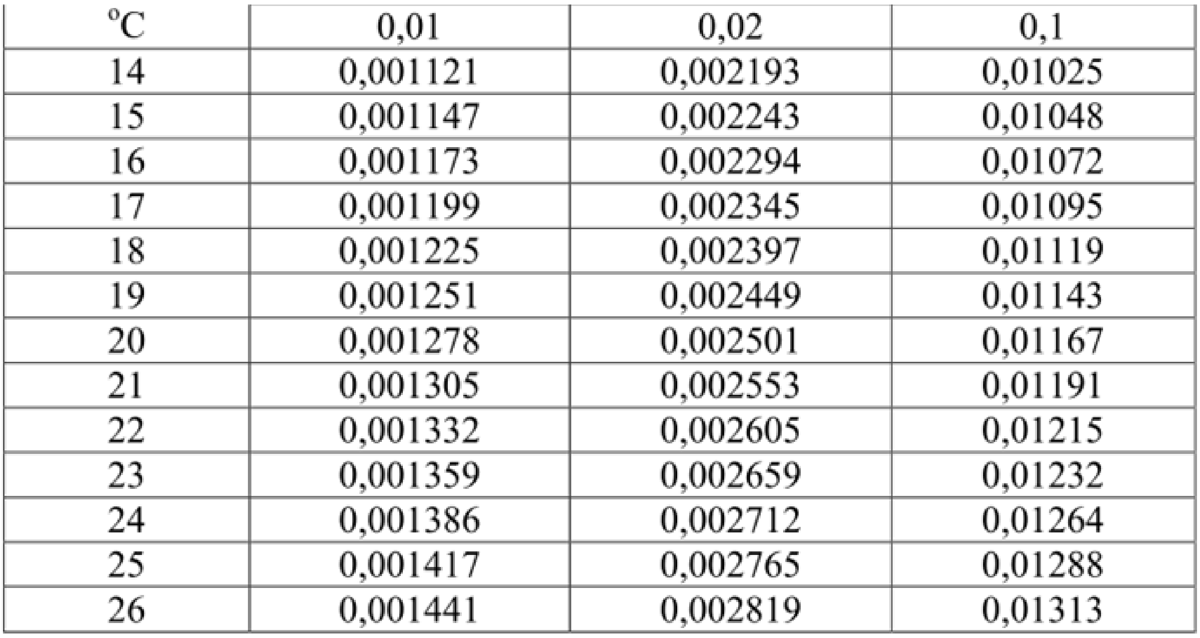
Ion	$\lambda_0 \cdot 10^4$, $(S \cdot m^2)$/ mole, temperature °C					α
	0	18	25	55	100	
Ag^+	33,1	53,5	61,9	-	175	0,0194
$\frac{1}{3}Al^{3+}$	29	-	63	-	-	0,021
$\frac{1}{2}Ba^{2+}$	34,0	54,6	63,6	-	195	0,020
$\frac{1}{2}Be^{2+}$	-	-	45	-	-	0,02
$\frac{1}{2}Ca^{2+}$	31,2	50,7	59,5	-	180	0,021
$\frac{1}{2}Cd^{2+}$	28	45,1	54	-	-	0,020
$\frac{1}{3}Ce^{3+}$	-	-	69,6	-	-	0,02
$\frac{1}{2}Co^{2+}$	28	45	54	-	-	0,02
CNS^-	41,7	56,6	66,5	-	-	0,02
$\frac{1}{3}Cr^{3+}$	-	-	67	-	-	0,02
Cs^+	44	67	77,2	123,6	200	0,019
$\frac{1}{2}Cu^{2+}$	28	45,3	56,6	-	-	0,024
$\frac{1}{3}Eu^{3+}$	-	-	67,8	-	-	0,02
$\frac{1}{2}Fe^{2+}$	28	44,5	53,5	-	-	0,024
$\frac{1}{3}Fe^{3+}$	-	-	68,0	-	-	0,02
H^+	225	315	349,8	483,1	630	0,0142
$\frac{1}{2}Hg^{2+}$	-	-	63,6	-	-	0,02
$\frac{1}{2}Hg_2^{2+}$	-	-	68,6	-	-	0,02
K^+	40,7	63,9	73,5	119,2	195	0,0187
$\frac{1}{2}CO_3^{2-}$	36	60,5	69,3	-	-	0,0192
$\frac{1}{2}CrO_4^{2-}$	42	72	85	-	-	0,021
F^-	-	47,3	55,4	-	-	0,021



Solubility Product Constants (K_{sp}) at 25°C

Ionic compound	K_{sp}	Ionic compound	K_{sp}	Ionic compound	K_{sp}
Halides		**Sulfates**		**Hydroxides**	
AgCl	1.8×10^{-10}	$PbSO_4$	6.3×10^{-7}	$Al(OH)_3$	3.0×10^{-34}
AgBr	5.0×10^{-13}	$BaSO_4$	1.1×10^{-10}	$Zn(OH)_2$	3.0×10^{-16}
AgI	8.3×10^{-17}	$CaSO_4$	2.4×10^{-5}	$Ca(OH)_2$	6.5×10^{-6}
$PbCl_2$	1.7×10^{-5}			$Mg(OH)_2$	7.1×10^{-12}
$PbBr_2$	2.1×10^{-6}	**Sulfides**		$Fe(OH)_2$	7.9×10^{-16}
PbI_2	7.9×10^{-9}	NiS	4.0×10^{-20}		
PbF_2	3.6×10^{-8}	CuS	8.0×10^{-37}		
CaF_2	3.9×10^{-11}	Ag_2S	8.0×10^{-51}	**Carbonates**	
		ZnS	3.0×10^{-23}	$CaCO_3$	4.5×10^{-9}
		FeS	8.0×10^{-19}	$SrCO_3$	9.3×10^{-10}
Chromates		CdS	1.0×10^{-27}	$ZnCO_3$	1.0×10^{-10}
$PbCrO_4$	1.8×10^{-14}	PbS	3.0×10^{-28}	Ag_2CO_3	8.15×10^{-12}
Ag_2CrO_4	1.2×10^{-12}			$BaCO_3$	5.0×10^{-9}

Specific electrical conductivity for 0.01M, 0.02M, 0.1M aqueous solution of potassium chloride, Ohm^{-1}·cm^{-1}



°C	0,01	0,02	0,1
14	0,001121	0,002193	0,01025
15	0,001147	0,002243	0,01048
16	0,001173	0,002294	0,01072
17	0,001199	0,002345	0,01095
18	0,001225	0,002397	0,01119
19	0,001251	0,002449	0,01143
20	0,001278	0,002501	0,01167
21	0,001305	0,002553	0,01191
22	0,001332	0,002605	0,01215
23	0,001359	0,002659	0,01232
24	0,001386	0,002712	0,01264
25	0,001417	0,002765	0,01288
26	0,001441	0,002819	0,01313

Standard electrode potentials of metals

Li^+/Li	$Li^+ + \bar{e} = Li$	– 3,02	Al^{3+}/Al	$Al^{3+} + 3\bar{e} = Al$	– 2,34
Rb^+/Rb	$Rb^+ + \bar{e} = Rb$	– 2,99	Mn^{2+}/Mn	$Mn^{2+} + 2\bar{e} = Mn$	– 1,05
K^+/K	$K^+ + \bar{e} = K$	– 2,92	Zn^{2+}/Zn	$Zn^{2+} + 2\bar{e} = Zn$	– 0,76
Ba^{2+}/Ba	$Ba^{2+} + 2\bar{e} = Ba$	– 2,90	Cr^{3+}/Cr	$Cr^{3+} + 3\bar{e} = Cr$	– 0,71
Sr^{2+}/Sr	$Sr^{2+} + 2\bar{e} = Sr$	– 2,89	Fe^{2+}/Fe	$Fe^{2+} + 2\bar{e} = Fe$	– 0,44
Ca^{2+}/Ca	$Ca^{2+} + 2\bar{e} = Ca$	– 2,87	Cd^{2+}/Cd	$Cd^{2+} + 2\bar{e} = Cd$	– 0,40
Mg^{2+}/Mg	$Mg^{2+} + 2\bar{e} = Mg$	– 2,71	Co^{2+}/Co	$Co^{2+} + 2\bar{e} = Co$	– 0,29
Ni^{2+}/Ni	$Ni^{2+} + 2\bar{e} = Ni$	– 0,25	Cu^+/Cu	$Cu^+ + \bar{e} = Cu$	+ 0,52
Sn^{2+}/Sn	$Sn^{2+} + 2\bar{e} = Sn$	– 0,14	$Hg_2^{2+}/2Hg$	$Hg_2^{2+} + 2\bar{e} = 2Hg$	+ 0,79
Pb^{2+}/Pb	$Pb^{2+} + 2\bar{e} = Pb$	– 0,13	Ag^+/Ag	$Ag^+ + \bar{e} = Ag$	+ 0,80
$H^+/\ {}^1/_2H_2$	$H^+ + \bar{e} = {}^1/_2H_2$	± 0,00	Pd^{2+}/Pd	$Pd^{2+} + 2\bar{e} = Pd$	+ 0,83
Sb^{3+}/Sb	$Sb^{3+} + 3\bar{e} = Sb$	+ 0,20	Hg^{2+}/Hg	$Hg^{2+} + 2\bar{e} = Hg$	+ 0,86
Bi^{3+}/Bi	$Bi^{3+} + 3\bar{e} = Bi$	+ 0,23	Pt^{2+}/Pt	$Pt^{2+} + 2\bar{e} = Pt$	+ 1,20
Cu^{2+}/Cu	$Cu^{2+} + 2\bar{e} = Cu$	+ 0,34	Au^{3+}/Au	$Au^{3+} + 3\bar{e} = Au$	+ 1,42

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Навчальний посібник знайомить з основними поняттями фізичної хімії (поведінка станів рідин та їх сумішей, основи хімічної термодинаміки, фазова рівновага, електрохімія, хімічна кінетика) та містить інструкції щодо підготовки та проведення експериментів з дисципліни «Фізико-хімічні експерименти». Посібник допоможе студенту коректно поставити експеримент та отримати досвід. Такий досвід надає можливість вирішувати наукові та виробничі завдання в подальшій самостійній діяльності хіміка-технолога.

Посібник призначено для студентів бакалаврату з хімічних технологій та інженерії.

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