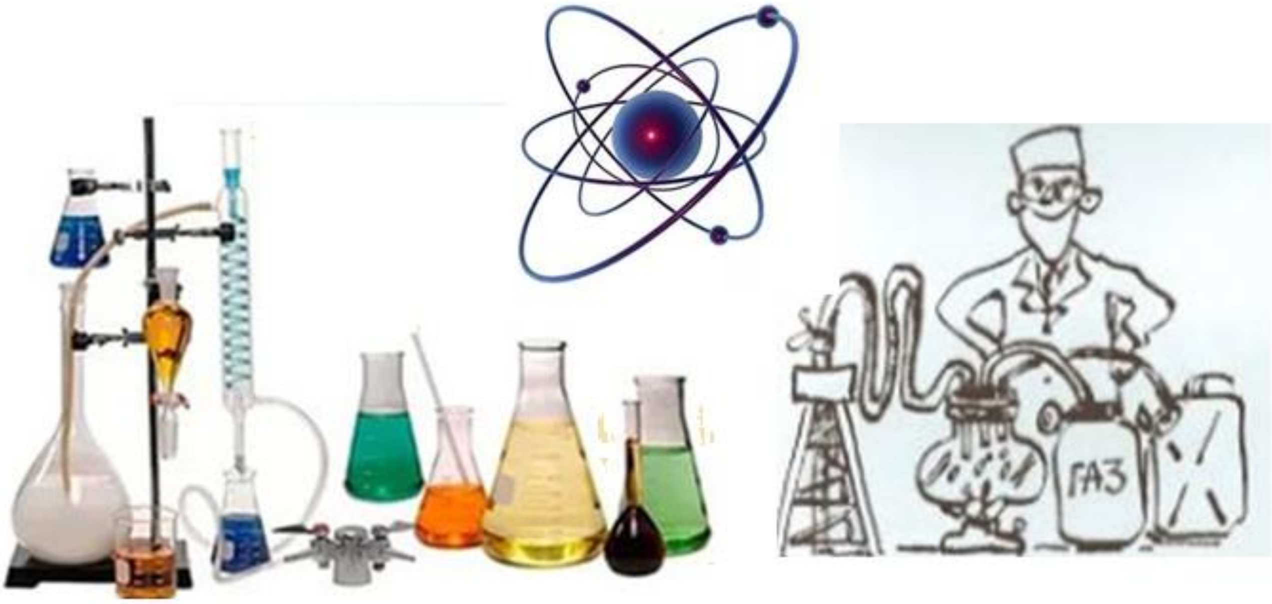
The National Technical University
"Kharkiv Polytechnic Institute"

Chemistry



Kharkiv 2017

МІНІСТЕРСТВО ОСВІТИ І НАУКИ УКРАЇНИ
НАЦІОНАЛЬНИЙ ТЕХНІЧНИЙ УНІВЕРСИТЕТ
«ХАРКІВСЬКИЙ ПОЛІТЕХНІЧНИЙ ІНСТИТУТ»

А.С. Бикова, О.В. Циганков, Є.О. Посохов

ХІМІЯ

Навчально-методичний посібник
для студентів спеціальності 185 «Добування нафти, газу та конденсату»
і спеціальності 076 «Управління та адміністрування підприємств, торгова та біржова діяльність»

Bykova A.S., Tsygankov A.V., Posokhov Y.O.

CHEMISTRY

Book for the students of specialty 185 «Oil, gas and condensate»
and specialty 076 «Management and administration of companies, trade and exchange activities»

Затверджено
редакційно-видавничою
радою НТУ «ХПІ»,
протокол № 1 від 22.06.2017 р.

Харків
НТУ «ХПІ»
2018

УДК 5.54 (076.5)

ББК 24я73

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Навчальний посібник складено для вивчення фундаментальної дисципліни «Хімія» в курсі бакалаврату. У посібнику стисло наведено теоретичний матеріал, є багато лабораторних робіт.

Розраховано на студентів спеціальності «Добування нафти, газу та конденсату» і спеціальності «Управління та адміністрування підприємств, торгова та біржова діяльність». Оскільки посібник викладено англійською мовою, він може використовуватись у ході навчання студентів-іноземців.

Бикова А.С., Циганков О.В., Посохов Є.О.

Хімія. / А.С. Бикова, О.В. Циганков, Є.О. Посохов – Харків : НТУ «ХПІ», 2018. – 89 с.

Bykova A.S., Tsygankov A.V., Posokhov Y.O.

Chemistry / Bykova A.S., Tsygankov A.V., Posokhov Y.O. – Kharkiv : NTU “KhPI”, 2018. – 89 p.

ISBN

The book presents the material for the study of fundamental discipline "Chemistry" in the undergraduate course. The manual provides concise theoretical material and includes many laboratory works.

For the students of specialty “Oil, gas and condensate” and specialty “Management and administration of companies, trade and exchange activities”. The book can be used during training of foreign students.

УДК 5.54 (076.5)
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CONTENTS

1. WORK IN A CHEMISTRY LAB

1. Only students, who have studied how to work in the chemical laboratory, have learnt section "Precautions when performing laboratory work" and have been instructed in the lab safety during the safety briefing, are allowed to perform laboratory practice. The students should confirm their knowledge of safety rules with their signatures in the safety journal. Accidents (burns, wounds, poisoning) in the laboratory usually are caused by the lack of familiarity with the relevant instructions for the protection of personnel and equipment.

2. Preparation to laboratory work is a type of independent work of students and carried out in advance. The student should:

   - preliminary to read the relevant sections of the theoretical course textbook and lecture notes in accordance with section I of laboratory work;
   - in your notebook for laboratory work write answers the questions and do the exercises that are listed in section II of this book.
   - read carefully the content of the upcoming laboratory work and, if possible, write down the experimental part (section III of the laboratory work), leaving empty space for records of observations and findings during the laboratory work.

3. All laboratory works are carried out by each student independently. Student should work at his working place, assigned to him for the duration of the laboratory workshop.

4. During the laboratory work one should follow all the lab safety rules, the sequence of actions and the quantitative ratio of substances specified in the manual.

It is forbidden to perform experiments, which are not related to the given laboratory work

5. To record the results of the experiments each student should have a separate notebook, which must indicate the name of the workshop, the student's name and initials, as well as his team (i.e. number of his academic group).

6. Entries in the notebook for laboratory work should be concise, clear and be entered immediately after the end of each experiment. Report on laboratory work should be carefully written and should contain the following information:

- ✓ the date when the laboratory work was performed
- ✓ the number and title of the laboratory work,
- ✓ paragraph numbers and the names of the experiments in the experimental section
- ✓ drawings of devices or equipment schemes
- ✓ the equations of all the reactions, which were conducted during the laboratory work
- ✓ the necessary calculations
- ✓ observations
- ✓ detailed findings
- ✓ answers to the questions and exercises in this lab

1.1 Precautions during laboratory work

1. Before the experimental work student on duty receives devices and reagents, necessary for lab work of the group, and places them in the workplaces. Only a lab notebook and necessary for work instruments and reagents can be in each workplace.
2. During the work in the chemical laboratory one should wear a robe. Long hair should be removed under a hat or collected.
3. It is **strictly** forbidden to eat food and to drink liquids in the laboratory.
4. All reagents for personal use (dilute aqueous solutions of salts, acids and bases) are on the work tables (i.e. on desktops) in special racks in flasks with pipettes. Reagents for common use during laboratory work are in a fume hood. Concentrated solutions of acids and alkalis are positioned on special trays in a fume hood. After using the reagent bottle should be immediately closed (i.e. plugged by a glass stopper) and put into place.
5. To take dry substances one should use special spoon or spatula.
6. If the reagent is accidentally taken in excess, it is not allowed to put (or pour) it back from tube to the bottle.
7. All the works related to the application or obtaining of toxic or malodorous substances, as well as works related to use of concentrated acids and alkalis, are conducted in a fume hood with activated ventilation. Also, your eyes should be protected with glasses.
8. It is forbidden to carry out from the lab: the laboratory reagents, glassware and equipment. Also, it is forbidden to conduct experiments, which are not mentioned in the methodological guidelines for the laboratory work.
9. When solutions is heated in a tube one should use a special tube holder. The tube hole must be directed away from yourself and neighbors (Fig. 1.1.1).

Figure 1.1.1

10. The smell of the substance should be determined carefully, directing air over the flask or test tube to the nose by flick of the wrist.
11. On dilution of sulfuric acid one should strictly follow to the rule - **add acid to the water!**
12. In case if acid gets onto the face or hands the acid should be immediately washed off with a strong water jet and on burned area one should put a cotton wool bandage, soaked in a dilute solution of baking soda.
13. In case if alkali gets onto the face or hands the alkali should be immediately washed off with a strong jet of water and one should put a cotton or wool bandage, soaked in a dilute solution of boric acid.
14. In case if skin is damaged with hot objects one should moisten the skin with a solution of potassium permaganate.
15. It is necessary to guard against poisoning gaseous substances such as chlorine, bromine vapors, hydrogen sulphide, carbon monoxide (II). In case of poisoning one should put the victim to the fresh air and as soon as possible the victim should see a doctor.

In all the cases (paragraphs 12-15), you must notify a teacher or an engineer on duty about the accident.

16. After finishing the work it is necessary to wash all the dishes, to give up all the dishes to the student on duty or to attendant, to clean your workspace, to turn off heaters, electric light, water and gas, to wash your hands.

1.2 First aid in the laboratory

In the laboratory, there are cases that require emergency medical assistance - cuts of the hands with glass, burns with hot objects, acids, alkalis. In particularly serious cases, you need to see a doctor.

First aid kit is available in the laboratory

1. In case of wound with glass - remove the glass fragments from the wound, remove the blood around the wound with cotton wool soaked in a weak alcohol solution or a solution of potassium permanganate. Then lubricate the edge of the wound with iodine solution and tie a bandage. In the case of severe bleeding from wound tightly tie a tourniquet above the cut. Remove the blood from the skin around the wound, apply several layers of sterile gauze and a thick layer of cotton wool, bandage the injured site.
2. When burn of the hands or face with acid or with alkali occurs, wash out the reagent with considerable amount of water for 3-5 minutes, then wash the burn either with diluted acetic acid (for alkali burn) or with soda solution (in the case of acid burns), and, then wash with water again. Do not flush acid with soap because fatty acids released during this do not allow you to remove acid well. If one get burned skin with an alkaline solution, wash the burn with water until the skin cease to be slippery to the touch. You can then apply a bandage of cotton wool soaked with alcoholic solution of tannin or 3% solution of potassium permanganate. In severe burns after first aid, contact your doctor.
3. If one get burned with hot liquid or hot object treat burned area with a freshly prepared solution of potassium permanganate, grease burnt place with ointment on a burn or with Vaseline. You can sprinkle the burn with soda and bandage it. It is helpful for small burns to moisten the cotton wool with alcohol and attach it to the place of burning for 2-3 minutes.
4. In case of chemical burns of eyes, wash the eyes with plenty of water at room temperature, then seek immediate medical attention.
5. If one is poisoned with gases (chlorine, bromine vapor, carbon monoxide (II), hydrogen sulfide), you must remove him to fresh air immediately and seek medical advice.

1.3 Rules of conduct in the event of a fire in the laboratory

1. In the event of a fire in the laboratory all electrical appliances and gas supply must be immediately turned off. Fire place must be covered with sand or covered with a fire-resistant blanket. Then, put out the fire using a fire extinguisher.
2. One should be cautious when using water to extinguish a fire, because in some cases water can increase the fire.

Graphical presentation of the rules, please, see on Fig 1.3.1:

It is forbidden to try the taste of the substance (a),
It is forbidden to take substances with hands (b)
It is forbidden to leave spilled reagents uncollected (c)
It is forbidden to leave bottles with liquids and cans with dry substances open (g);
The work should be carried out only over the table (d)

a b c d e

Figure 1.3.1

2. CHEMICAL GLASSWARE.

There are a few types of chemical glassware:
general purpose glassware, dimensional glassware and glassware of special purpose.

2.1 Glassware of general purpose

The tubes (fig. 2.1.1) have narrow cylindrical shape with a round-bottomed vessels. They are used for carrying out chemical experiments via microchemical or semi-microchemical methods based on usage of small amounts of reagents. The tubes can be simple or can be graded. The tubes are stored in wooden, plastic or metal test tube racks.

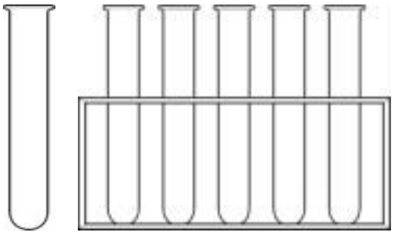


Figure 2.1.1

A droppers (fig. 2.1.2) are used to add small amounts of reagents dropwise

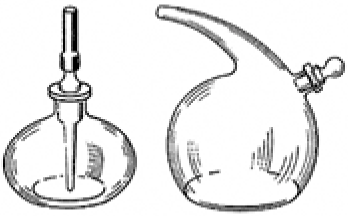


Figure 2.1.2

Glass rod with a thickness of a few millimeters and with a length of 20-30 cm is used for mixing liquids.

Watch glasses (fig. 2.1.3) are necessary in the study of crystals and, in general, when one is using small amounts of reagents, in particular, during the drip color reaction.

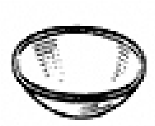


Figure 2.1.3

The funnels (fig. 2.1.4) are used for pouring of liquids, for separation of liquids, for filtering and so on.

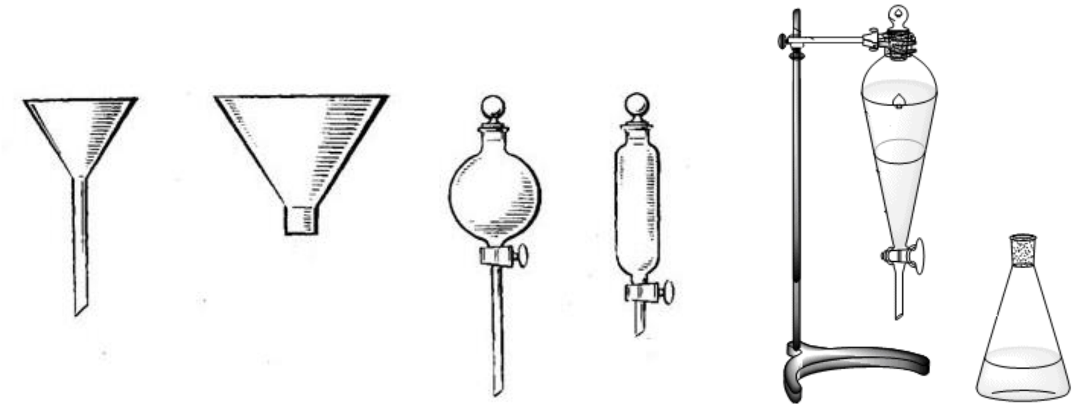


Figure 2.1.4

Chemical beakers (fig. 2.1.5) are thin-walled cylinders of different capacities, made of chemically resistant refractory glass of uniform thickness. They are used as an auxiliary glassware for stirring of solutes and for heating, mixing or draining of solutions.

Figure 2.1.5

Bulbs (fig. 2.1.6) can be in many shapes and sizes, with neck for simple rubber stopper or with neck for glass stopper.

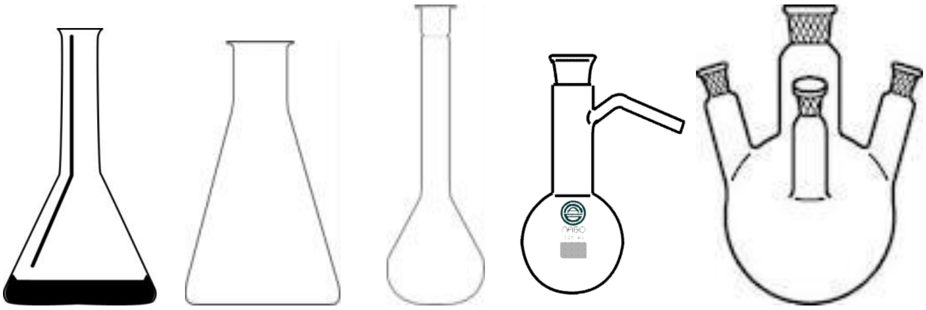


Figure 2.1.6

Wash bottles (fig. 2.1.7) are used to flush sediment from the vessel walls and to wash cuvettes.

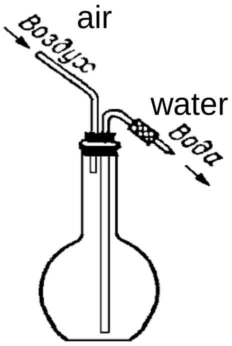
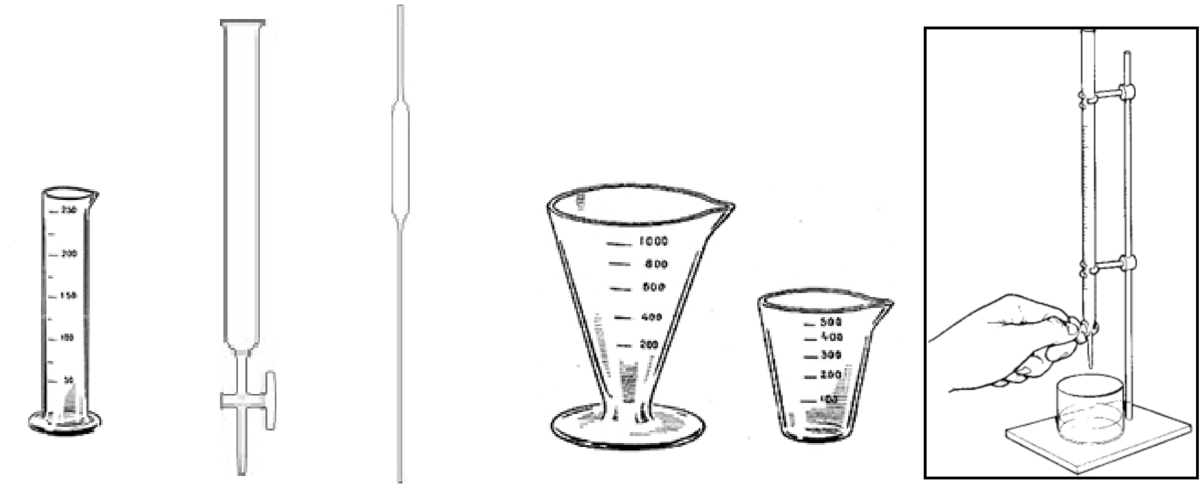


Figure 2.1.7

2.2 Measuring glassware.

Glassware used to measure the volume of liquid are called dimensional glassware.

Measuring cylinders and beakers - graduated glass vessels with the graduations indicating the volume in milliliters. They are used when measurement of the volume of fluid does not require great precision. **The pipettes, burettes** (fig. 2.2.1) **and volumetric flasks** (fig. 2.2.2) are used for precise measurements of the volume of liquid**.**



Cylinder burette pipette beakers

Figure 2.2.1

Burette - a graded glass tube with a tap (fig. 2.2.1). Burette is used to accurately measure the consumption of liquid during the titration.

Volumetric flasks (fig. 2.2.2) are intended for various analytical work: for dilution of solution, for dissolving of substances in a certain volume of a suitable solvent, for preparing of solutions with preselected concentrations, etc.

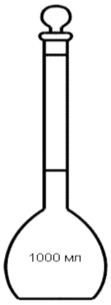


Figure 2.2.2

2.3 Chemical glassware for special purpose

Condensers (fig. 2.3.1) - devices for cooling and condensing the vapor produced by heating of various substances. They are used for distillation, extraction and other processes.

Glass condensers consist of the inner tube and the surrounding jacket (or sleeve). Inner tube of Liebig condensers is straight, and in many newer types of condensers consists of a spherical extensions or has a coil shape. Such shape allows to increase the cooling surface. The cooling water must pass through jacket (or sleeve).

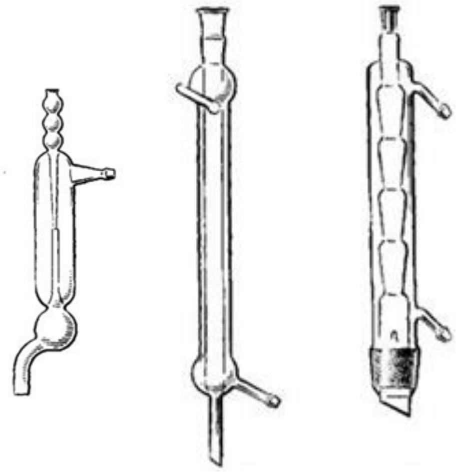


Figure 2.3.1

Waterjet pumps (fig. 2.3.2) - in such pumps a vacuum is created by the fact that air is sucked by jet of water. Vessel from which air is suctioned, is connected with a pump by thick-walled rubber hosepipe (so-called vacuum hose) because under condition of vacuum the walls of conventional rubber tubing stick together and, thus, the connection is broken. The vessels, from which air is suctioned, must be thick-walled and perfectly reliable. One should pay attention whether the vessels have even the smallest cracks.

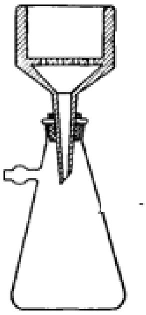


Figure 2.3.2

Bunsen flask (made of very thick glass) and Buchner funnel (porcelain) - a device (fig. 2.3.3) for filtering under vacuum

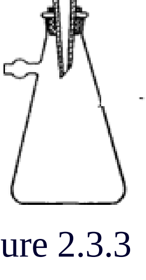
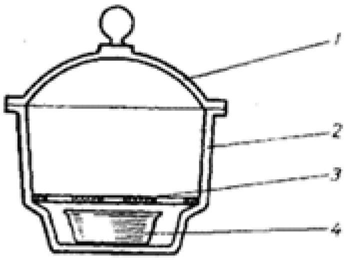


Figure 2.3.3

Exsiccators (fig. 2.3.4) - glass containers with a lid and with (or without) a tap. They are used for storing of materials, which easily absorb moisture, and for drying of substances. For this purpose, in the lower part of the desiccator the drying agents (i.e. substances which can absorb water) are placed: $CaC1_2$ (anhydrous), H_2SO_4 (concentrated), P_2O_5. A porcelain insert, on which the weighing bottles or crucibles with substance are set, is positioned over the dehydrating agent.



1- cap; 2- housing; 3- insert ;
4- cup for the absorber ;

Figure 2.3.4

2.4 Porcelain

As well as a thin-walled glassware, thin-walled porcelain is heat-resistant.

Evaporation dishes (evaporating basins, fig 2.4.1) are necessary for evaporation of solutions. Crucibles indispensable for calcining of solids, metal melting and so on. Mortar and pestle are used to grind solids.

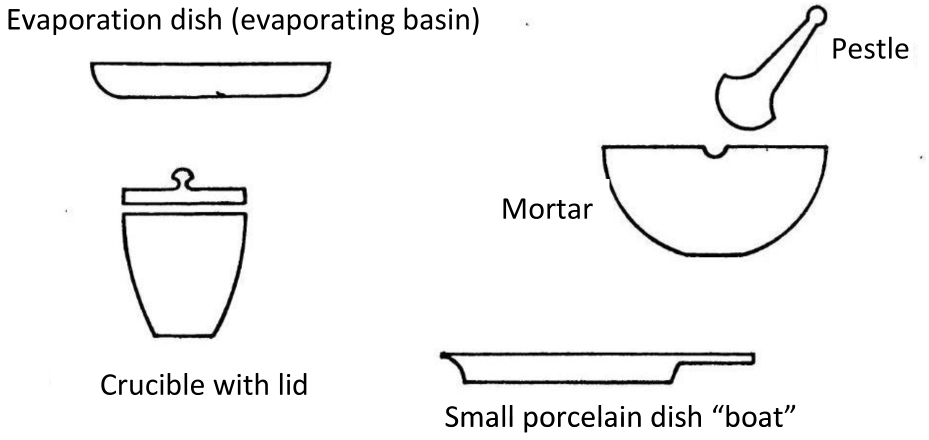


Figure 2.4.1

2.5 Preparation of glassware for work

Glassware for chemical research must be clean. Mechanical dirt should be removed with a special brush (brush). Then the glassware should be washed with plain water. If after washing with plain water one can notice indelible grease stains on the walls of the glassware, the washing process should be repeated with use of a soap solution or soda. To wash very dirty glassware one can use so called "chromium mixture" ($K_2Cr_2O_7$ solution in concentrated sulfuric acid). To prepare "chromium mixture" one should add 3% $K_2Cr_2O_7$ (as pounded powder) to concentrated sulfuric acid (1: 1). The obtained mixture in porcelain cup should be gently heated in a water bath to dissolve salt.

Treatment of glassware with chromate solution should be carried out with great care, keeping in mind that in contact with skin chrome mixture causes painful burns, and in contact with clothing destroys tissue!

A small amount of chromic mixture is used to wash inner surface of glassware. After 5-7 minutes, chromium mixture is poured into a bottle in which it is stored. Glassware is washed 3-5 times with water and then rinsed with distilled water. Do not use the brush when washing glassware with chromate.

Glassware considered clean if no water droplets are observed on the walls of glassware after water flowed down from the walls (when water is flowing down from the walls of clean glassware one can observe continuous water film on the walls).

3. Basic techniques for work with substances

3.1 Weighing

Weighing is a comparison of mass of body or substance with a mass of the weights, which is known and is expressed in specific units (mg g, kg etc.). Scales are an important instrument in the chemical laboratory, because almost none of the work can be done without determining the mass of a substance.

Depending on the accuracy with which weighing is performed, the scale is divided into the following groups:

1) for rough weighing (accurate to grams);

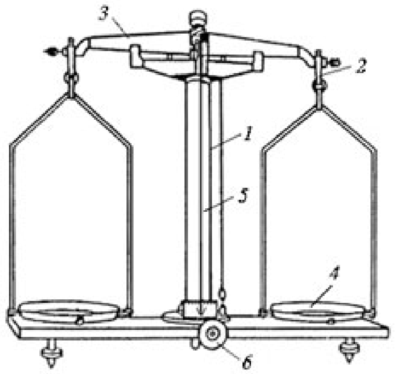
2) for the precise weighing (the accuracy from 1 to 10 mg);

3) analysis (accuracy from 0.0001 to 0.0002 g);

4) Special (assay, torsion, etc.).

Every instrument has its own set of weights (weights), located in a case in a certain sequence. Each weight is placed in a separate socket case. Weights of 1 g or more have the form of dumb-bell. Milligram weights (which, are made of aluminum sheet) have different shapes. It is easier to notice and to take the necessary weight when the weights differ in shapes from each other. In order not to contaminate the weights, they are taken only with tweezers, which are also in the case.

For accurate weighing technical-chemical scales (fig. 3.1.1) are used. Their payload can range from 200 grams to several kilograms. In contrast with the balance for coarse weighing, the balance for accurate weighing has a so-called balancing device, stop knob and nuts. With balancing device most crucial parts of the balance (balance beam and pillows) in the rest position (latched scales) are separated and do not touch rider bar. It protects the balance beam from wear and scales - from the loss of sensitivity. On weighing the scales are brought into working position by turning the stop knob. If after the turning of stop knob the balances are not in equilibrium, the balance is to be achieved by means of balancing nuts.



Technical-chemical scales :
1 - balance column; 2 - rider bar;
3 - balance beam; 4 - scale pan;
5 - pointer; 6 - stop knob

Figure 3.1.1

Analytical balance ADV-200 (fig. 3.1.2) allows one to weigh up to 0.0002 g if a load is no more than 200 g. For the scale of this type only gram weights are in a set of weights. Milligram weights have the form of rings and hung in the balance. With leverage, they are mounted on a special bracket attached to the right shoulder of the beam. The mounting is made by two rotating disks coated with these figures. Turn of the external disk can hang weights from 100 to 900 mg, while the internal rotation of the disk hung weights from 10 to 90 mg. Milligrams are determined by the arrows departing from the mark “zero” on the scale. Larger image of microscale is observed on the illuminated screen of optical device, so-called weightograph. Screen Illuminator turns on automatically when you turn the stop knob. Each mark on microscale, labeled with digit, corresponds to a load of 1 mg. The distance between two neighbouring marks is divided into 10 parts, each of which corresponds to 0.1 mg. Consequently, the reading on the scale of weightograph provides third and fourth decimal digits of the determined weight (in grams).

Figure 3.1.2

Working with weights, you must follow the following **rules**:

1) during the weighing do not slide scales from the place where they are set; do not lean on a shelf, where the scales are set;

2) Putting weighed substance and weights on the scale pans of the scales, as well as removal of the load from the scale pans are allowed only when the scales are in the rest position (i.e. the scales are latched with a stop knob).

3) before you put the item on the left pan, check for dirt on the outer surface of the item.

4) do not put the substance directly on the weighing pan, all materials are weighed in a suitable container: beakers, crucibles or watch glasses;

5) warm (or cold) objects should not be placed on the pan - weighed object must pre-cooled (or pre-warmed if the object is cold) to room temperature in the desiccator;

6) do not overload the balances (there is a limit load for each type of balances);

7) rotate stop knob slowly, slowly, cautiously;

Experiment 3.1.1. Weighing on technical-chemical balance

1. Adjust the balance. When the scales are properly installed, the tip of plummet coincides with the apex of the cone located at the steps of the

column. Some types of scales have liquid levels with an air bubble instead of plummet. If after turning on with stop knob the scales are not in equilibrium, it is achieved by means of balancing nuts. Nuts are moved when the scales are in the rest position.

2. Put weighed substance on the left pan, put weights on the right pan.
3. Take weights only with tweezers and after weighting put the weights back into their nests in the case, from which they were taken.
4. After weighing the weighing pan must stay clean.
5. When you finish weighting, check the set of weights. Set the scales in the rest position (i.e. the scales are to be latched with a stop knob).

Experiment 3.1.2 Weighing with an analytical balance

1. Connect the balance to the power supply.
2. Turn the stop knob (turn on the scales) and observe the movement of an illuminated scale. If zero on the scale does not coincide with the reference line, make them match by the slight rotation of the corrector, located a little above the wheel of stop knob. Turn off the scales with the stop knob.
3. Open the left door of the balance box, place the thing in the center of the left weighing pan. Close the door.
4. Open the right door of the balance box and begin weighing, applying weights with tweezers in the usual way. Turn on the scales (turning the stop knob) and observe the indication of the instrument (i.e. observe the movement of an illuminated scale). If the scale on the screen moves to the left of the reference line, the weighted body is heavier than the weights on the right pan. Turn of scales and put the new weights. (To ease weighting the thing should be preliminary weighted with technical-chemical scales with accuracy up to 0.01 g)
5. If the weight 1 g is in excess, remove it and proceed to the automatic laying milligram weights using disks.
6. Weights from 100 to 900 mg , i.e. hundreds milligrams, are placed on the beam with the external disc. Weights from 10 to 90 mg, i.e. weights up to hundreds of milligrams, are placed on the beam using the internal disk. When the last weight of internal drive is excessive, turn the drive one space back and let the balance settle down. On the illuminated screen of balance optical device one can see which mark on the scale coincides with a fixed line. If the value on the scale has a plus sign (+), it is added to the weight of weights if the value on

the scale has a negative sign (-), it is subtracted from the total weight of weights. Thus, we get the accuracy from 0.001 g to 0.0001 g.

3.2 Filtration

Filtration is carried out to separate the precipitate from the liquid phase during the separation of substances, purification of substances, washing of precipitate, etc.

In the simplest case, in order to separate solids from liquids the liquid is decanted from the precipitate (decantation method), in other cases - use filtration through a funnel with a filter. Various organic and inorganic substances are used as filter materials in the laboratory. Filter materials may be:

a) fiber (cotton, wool, various fabrics, synthetic fibers);
b) granular (silica sand);
c) porous (paper, ceramics, pressed glass).

The choice of filter material depends on the requirements for purity of the solution, as well as on its properties. It must be remembered that the filtering can not be used for materials in any way interacting with the filterable liquid. For example, alkalis, especially concentrated, can not be filtered through a filter made of pressed glass and other materials containing silica because SiO_2 dissolves in alkalis.

Filtration efficiency depends on the porosity of the filter, and on the difference in pressure for both sides of the filter. Filters are manufactured mostly from various kinds of filter paper, glass fiber, porous glass and teflon.
For a simple filtration a funnel with a pleated filter (fluted filter) is used.

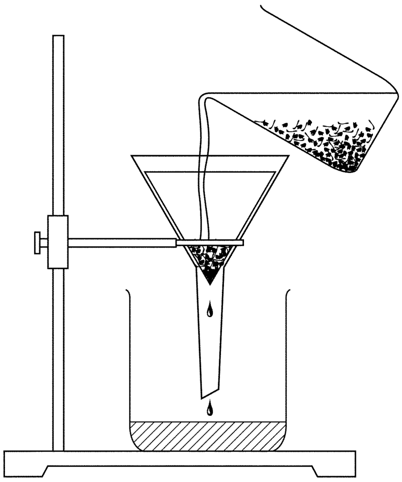


Figure 3.2.1 Filtering at atmospheric pressure

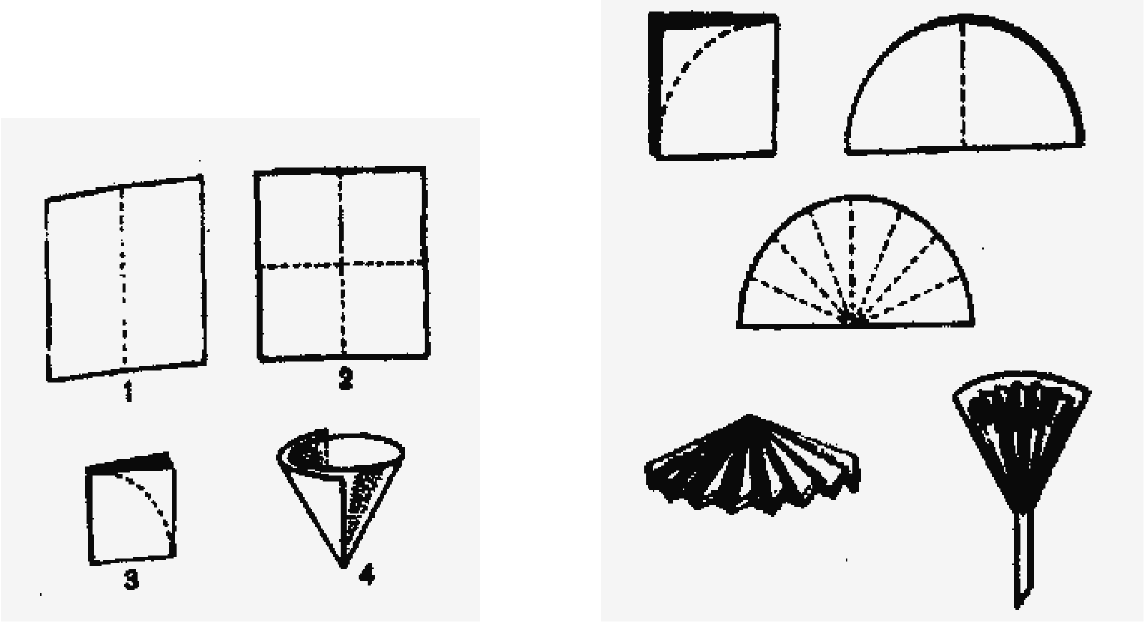


Fig. 3.2.2 Production of filters:

a) smooth b) pleated (or fluted)

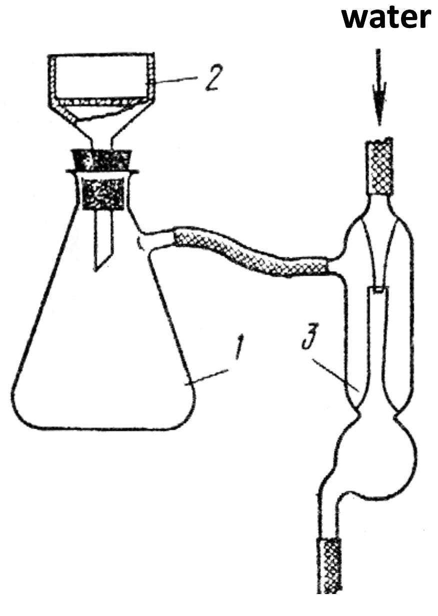


Fig. 3.2.3 Device for filtering under vacuum.
1 - Bunsen flask (filter flask);
2 - Buchner funnel (filter funnel);
3 - Water jet pump

A more efficient filtration is carried out under vacuum, which usually use two types of filter funnels: "Schott funnel" with porous glass slide and Buchner funnel with a well-fitted paper filter, connected to the Bunsen flask.

Paper filter on the funnel should be pre-moistened with a solvent, then, the solution with crystals is transferred onto paper filter.

Exhaustion of the mother liquor is performed by a water jet pump, connected to the Bunsen flask through the protective flask. The required rate of filtering is set by adjusting the jet of water in a water pump, which creates a negative pressure in the Bunsen flask.

To remove residual solution wet crystals is washed with several portions of a minimal amount of solvent with gentle stirring of crystals. Sometimes the precipitate on filter is only impregnated with the solvent, and, then vacuum suction of the precipitate is performed.

The crystals on the filter are drained from the solvent with the flat side of the glass stopper, then, the residue is sent to drying.

Experiment 3.2.1 Separation of mixture into components: the separation of insoluble solid substances from liquid substances

a) Filtration through a paper filter (smooth or *pleated).*

Funnel glass for the filtration is fixed on a stand with a ring (Fig. 3.2.1). A filter paper is placed in the funnel and wetted with water. Liquid to be filtered (part of the prepared suspension) is poured gently on the glass rod into the funnel with filter. The funnel should be installed so that its end touched the wall of the vessel for filtrate collection.

Draw a diagram and identify the equipment used for filtering through a paper filter.

b) Filtration under vacuum.

Vacuum filtration apparatus (Fig. 3.2.3), consisting of: Buchner funnel, Bunsen flask for vacuum filtration and vacuum pump (such as a water jet), is collected for the filtration under vacuum. The size of the porcelain Buchner funnel is chosen to be consistent with the expected amount of the precipitate. The more precipitate, the larger funnel must be used. In order to prevent the possibility of transferring water from the water pump to the flask, it is advisable to put a protective flask between the water pump and the Bunsen flask. Inside the funnel on its net bottom lay a circle of filter paper (cut the paper circle exactly to the inside diameter of Buchner funnel), moisten the filter with distilled water and turn on a water jet pump. Liquid to be filtered (part of the prepared suspension) is transferred to the filter (one should pour the liquid via glass rod). Filtering is finished, when dripping of the solution from the funnel stops.

Draw a diagram and identify the equipment used for filtering under vacuum.

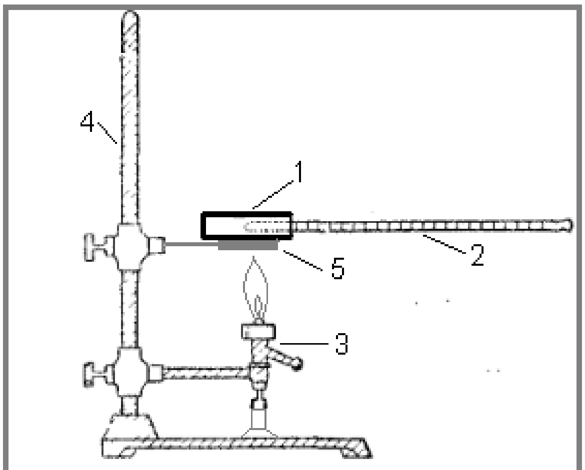
3.3 Determination of the melting point

When heated, the solid becomes liquid – i.e. the solid melts. According to the thermodynamic definition, **the melting temperature is the temperature at which the solid is in equilibrium with a liquid phase, when steam phase is saturated**. The temperature during melting, as follows from the phase rule, remains constant until all of the solid body does not turn into liquid.

Melting point (T_m) is a criterion of purity for many organic compounds. Normally, a pure substance is melted in **a very narrow temperature range** (from a few tenths of a degree to one degree), i.e. almost at the same temperature. Even slight contamination of substance substantially **lowers** the melting point, expanding the temperature range of melting up to several degrees. Consequently, the melting temperature can be used as indication of the purity of the melting material.

3.3.1 Determination of the melting point by the block method

The metal block, in which there is a hole for a thermometer, is secured to the stand by means of a ring. For the best thermal conductivity the lower end of the thermometer is enveloped with silver wire and inserted into the hole to the limit. The unit is heated by microburner **with the smallest possible flame**.



1 - metal block; 2 - thermometer;
3 - burner; 4 - stand; 5 - metal ring

Figure 3.3.0

A small amount of finely reduced (finely divided) dried substance is placed on the polished surface of the block.

Temperature rise is controlled so that it is 1 ° in 30 seconds. The temperature at which the material melts instantaneously is noted.

3.3.2 Determination of melting point by capillary method

1. The dried test substance (ground into superfine powder) is placed in a capillary tube (about 1 mm in diameter and 50-70 mm in length) sealed at one end. To place the test substance in the capillary tube, one should immerse the open end of the capillary in the test substance. In result, some amount of the test substance will be placed in the capillary tube. For correct determination of melting point the substance in the capillary should be compacted: to achieve this, the capillary with the substance should be tapped on a hard surface or repeatedly thrown into a glass tube (with a diameter of 6-8 mm and length of 50-60 mm), vertically set onto the glass surface of the table. The height of the column of substance in the capillary after compaction must be about 3 mm.

2. Then, the capillary with the substance should be attached to the thermometer with a rubber ring. The capillary is positioned so that the substance enclosed therein is at the middle of the thermometer bulb. Thermometer with capillary is placed in the instrument. Sulfuric acid or silicone oil is poured into the flask as the heat transfer medium.

3. The flask of the device is heated to the temperature, which is 10-20°C lower than the assumed melting temperature of the substance. Then, the flask is heated so that the temperature rise is 2-3°C per minute, and, at the temperature 5°C lower than the expected melting point the temperature rise is 0.5°C per minute. Carefully observe the state of the substance.

4. The appearance of the first drops of liquid substance and shrinkage of the substance are detected – this corresponds to the temperature, when melting starts. The end of melting corresponds to the temperature, when the last grains of solid substance disappear.

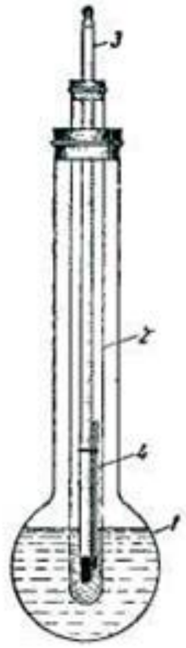


Figure 3.3.1. Device for determination of
melting point by capillary method:
1 - flask filled with heat transfer medium;
2 - tube for thermometer; 3 - thermometer;
4 - capillary with substance.

Theoretically, pure substance should have a constant melting point regardless of by whom and where the determination is carried out. However, like other methods, melting point determination is subject to the influence of a variety of factors. This may include:

- difference in the sample taken for analysis and in treatment of the sample,
- the difference in the devices,
- accuracy of the thermometer,
- a change in the rate of temperature rise,
- the difference in ambient temperature and,
- finally, the difference between the personnel performing the detection of melting point temperature: the difference in choice of detection methodology and in interpretation of the melting temperature.

Accounting and assessment of the impact of all the above factors on the experimental results are an important part of melting point determination.

3.4 Determination of density

In chemical laboratory one often has to measure the density of solutions and other liquids. In the literature of past years and in older editions of handbooks the tables of specific weights of solutions and solids are presented. This quantity was used instead of density, which is one of the most important physical quantities that characterize the properties of the substance.

The density of a substance is the ratio of mass of the substance to its volume:

$$\rho = \frac{m}{V} \qquad (3.4.1).$$

, where

ρ - density of substance;
m - mass of substance, g;
V - volume of substance, cm^{-3}.

Usually the density of the solution increases with the growth in the concentration of the solute (if it itself has a density greater than the solvent). But there are substances for which the density increase with the growth in the concentration occurs only until a certain limit, after which the density decrease occurs with the growth in the concentration.

For example, sulfuric acid has the highest density of 1.8415 at a concentration of 97.35%. A further increase in concentration is accompanied by a decrease in the density to 1.8315, which corresponds to 99.31%. Acetic acid has a maximum density at a concentration of 77-79% and 100% acetic acid has the same density as the 41% acetic acid.

Relative density decreases with increase in temperature, and, correspondingly, increases with decrease in temperature. On determination of the relative density it is necessary to observe the temperature at which the determination is performed, and the values obtained should be compared with reference data determined at the same temperature.

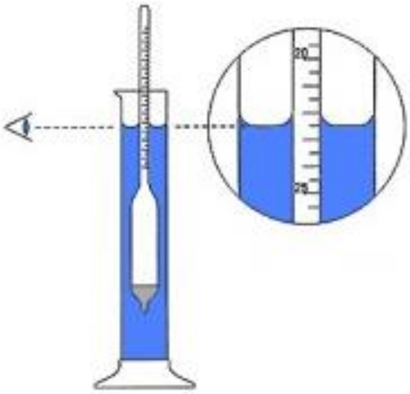


Figure 3.4.1. Determination of the density of liquid with hydrometer.

Hydrometers (fig. 3.4.1) are used for quick determination of the density of the liquid. Hydrometer is the glass float, which has a graduated scale at the top of the narrow part. The graduated scale shows the density. The lower the density of the solution, the more a hydrometer is immersed in it. In the dry narrow 100-250 ml cylinder one pours the test solution and immerses dry, clean hydrometer so that it does not touch the walls of the vessel (Figure 3.4.1). By lower meniscus one notes the scale marking, coinciding with the liquid level in the cylinder with an accuracy of ± 0,003.

Pycnometer (fig. 3.4.2) is used to determine the relative density of the liquid with a precision of four decimal places.

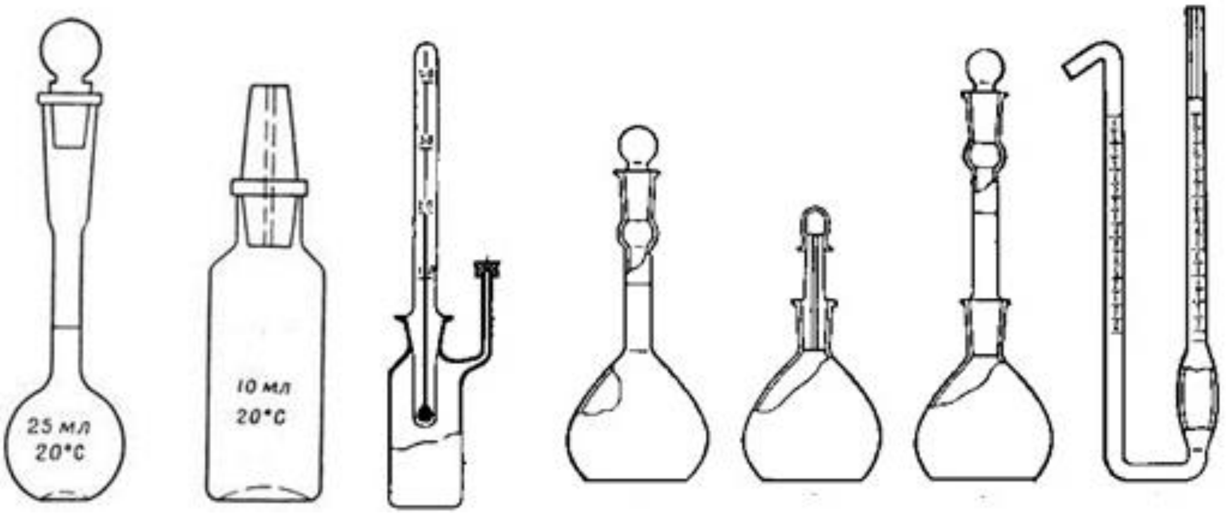


Fig. 3.4.2 Pycnometers for measurements of the density of liquid, solid and gaseous substances

Experiment 3.4.1 Density determination by pycnometric method

1. With precise scales determine the weight of the dry and clean pycnometer;
2. Fill the pycnometer with test liquid up to the mark and weigh the pycnometer;
3. Pour the liquid back into the bottle, rinse the pycnometer.
4. Fill the pycnometer with distilled water to the mark and weigh the pycnometer.
5. Calculate the density using the formula:

$$\rho_{\text{liquid}} = \frac{m_2 - m}{m_1 - m} \cdot \rho_{\text{water}} \qquad (3.4.2)$$

where: m - the mass of the pycnometer; m_1 - the mass of the pycnometer with water; m_2 - the mass of the pycnometer with the test liquid.

Pycnometers are used, for example, to determine the density of oil and oil products. Such measurements are included in measurement standards in many countries. The composition of oil and petroleum products can be roughly judged from their density. Thus, oil density can be used to judge about oil quality and cost.

Experiment 3.4.2 Determining the density of liquids using a hydrometer

Each hydrometer is intended for liquids with densities within a certain range. Since the density of each solution of the substance is a function of its concentration, then, by measuring of the density of the hydrometer, you can find the concentration of the solution from reference tables.

Procedure:

1. With the hydrometer determine the density of sulfuric acid solution (Figure 3.4.1).
2. From the table of density dependence on concentration find the concentration of the solution of sulfuric acid.
3. In the laboratory journal draw the scheme of the device for measuring of the density.

3.5 Determination of the boiling point

The temperature, at which the vapor pressure is equal to external pressure, is called **boiling point**.

The boiling point depends on the molecular weight and structure of the compound, on the nature and degree of intermolecular interaction (dispersion, ion-dipole, dipole-dipole interactions). The boiling point is highly dependent on the pressure.

To convert a boiling point at a specific pressure to boiling point at 760 mm Hg. Art., one should connect with the straight line the corresponding values on the scales A and C. The desired (i.e. required) value of the boiling point is read on the scale B. If you then connect with the line the found boiling temperature to any pressure value on the scale C, the point of intersection with the scale A gives approximate boiling point corresponding to the selected pressure.

1 mm Hg. Art. = 133.32 Pa = 1.3158·10^{-3} atm

Since the boiling temperature is strongly dependent on the pressure, the pressure and the temperature at which the boiling is observed should be specified near the figure, denoting the boiling point. If the pressure is not specified, it means that the boiling point is determined at normal atmospheric pressure of 760 mm Hg. Art.

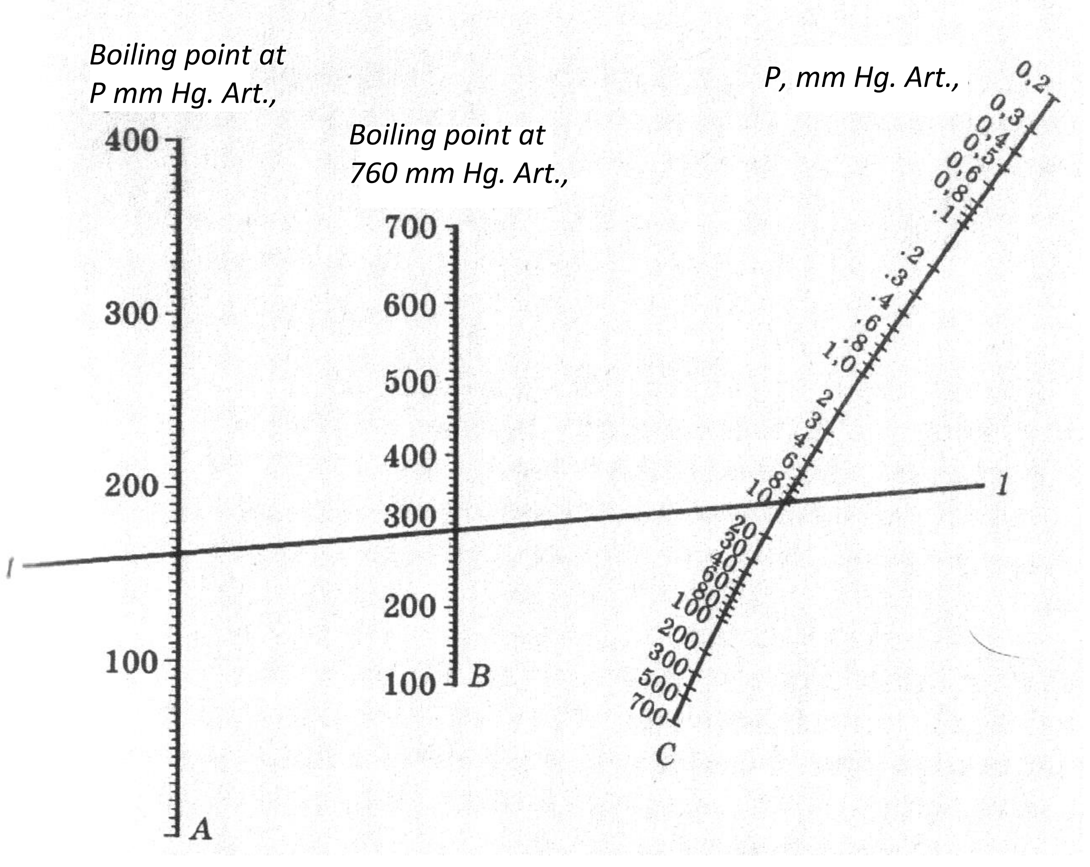


Figure. 3.5.1 The nomogram to convert the boiling temperature at different pressures

The presence of the small amount of impurities has a much smaller impact on the boiling point than on the melting temperature. Therefore, **in comparison with melting temperature (which is usually used as criterion of purity) the boiling point is not so important as a criterion of purity.**

Boiling point can be determined in vapor as well as in liquid phase in the conventional apparatus for simple distillation. For pure substances, the determination of the boiling temperature in the vapor gives more accurate results, as the vapor temperature does not depend on minor temperature fluctuations inside the liquid (such fluctuations are inevitable during direct heating of the liquid). Because of the additional hydraulic pressure, the liquid is always somewhat overheated in its lower layers. To avoid severe overheating during the determination of the boiling temperature, it is necessary to use the bath and to maintain a uniform and continuous boiling of the liquid. During the determination of the boiling temperature in vapor it takes some time until the thermal equilibrium is established.

If a sufficient amount of liquid (≥10 ml) is available, it is easier to use the distillation apparatus in order to plot a curve of boiling. During this procedure it is necessary to pay attention to the thermometer, which should be completely bathed by vapors, wetted with condensed liquid and should not be submerged too deeply into the superheated vapor.

It is convenient to determine the boiling point of a small amount of a substance (0.2-1 ml) by ***the method, suggested by Sivolobov***.

According to this method (fig. 3.5.2), a drop of the sample liquid is placed at the bottom of a wide thin-walled capillary (3-4 mm in diameter), sealed (by fusion) at the bottom end. Then, another very thin capillary, sealed (by fusion) at the top end, should be dipped in this liquid.

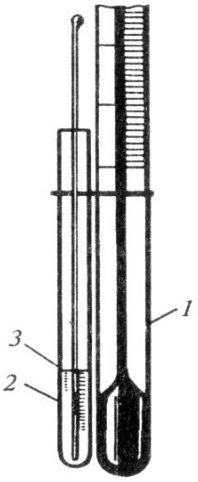


Figure 3.5.2 Device for the determination of boiling point by method of Sivolobov: 1 – thermometer; 2 - wide thin-walled capillary, sealed at the bottom end; 3 - sample liquid

Then, the capillaries should be attached to the thermometer and heated in the device for determination of the melting point. Initially, rare air bubbles are evolved from the internal capillary. When the boiling point is reached, a lot of bubbles, rising up as a steady stream, are formed. At this stage, one should stop heating. The temperature at which bubbling suddenly ceases (i.e. stops) is taken as the boiling point.

The accuracy of the boiling temperature determination by this method is about ± 1-2 ° C.

3.6 Determination of the refractive index

Along with other physical constants one can use **a refractive index *n*** for the identification of liquids and for verification of their purity.

The refractive index is determined by refractometry, which is based on the phenomenon of refraction of light at the interface. When monochromatic light beam passes from one medium to another, the speed of light is changed, and, at the interface between media the direction of the light beam changes also (Fig.3.6.1). The deviation of the beam occurs according to the law of Snell:

$$n = \frac{\sin\alpha}{\sin\beta} = \frac{c_1}{c_2}$$

Wherein: c_1, c_2 – velocity of light in the first and second media, respectively;

α, β - angle of incidence and refraction of light beams, respectively, at the transition from the first to the second medium.

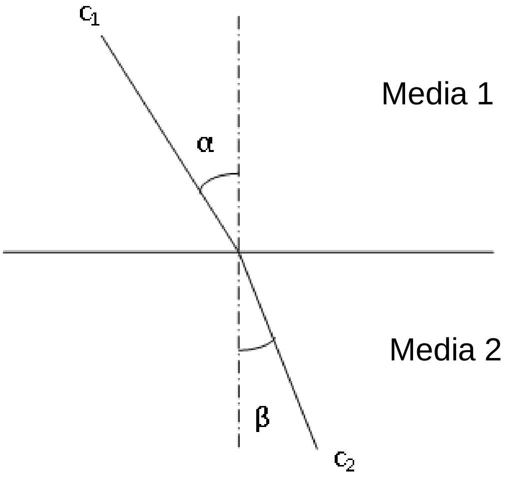
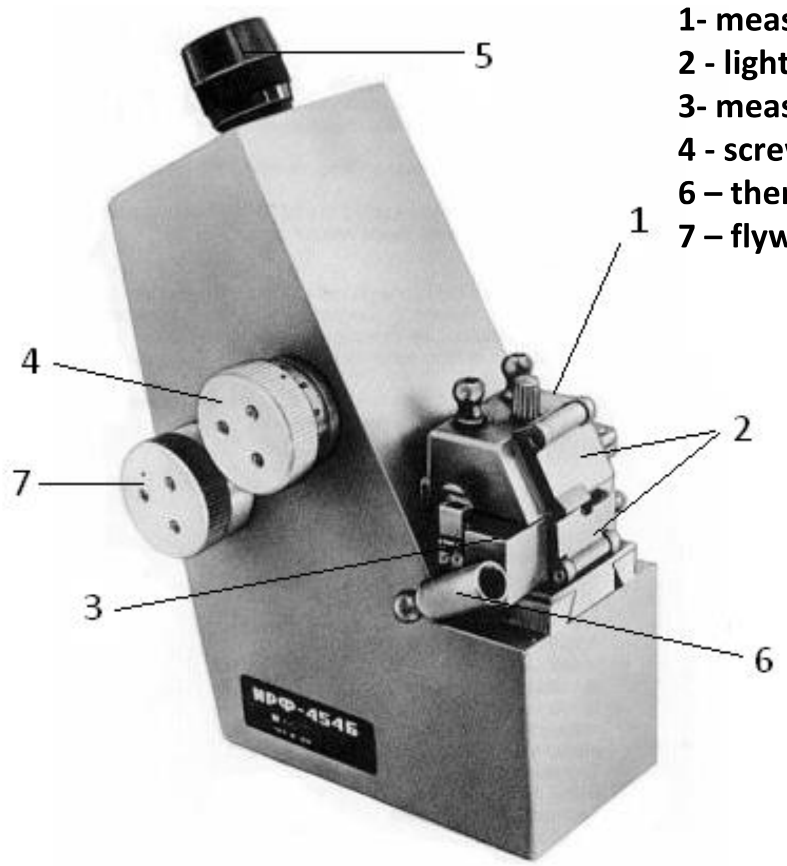


Figure. 3.6.1. Refraction of light at the interface of two media.

The value of the refractive index is dependent on temperature (temperature increase of 1°C decreases the refractive index by 0.0005) and sharply varies with wavelength and solution concentration. Therefore, the measurement is carried out at a constant temperature and monochromatic light. Impurities also affect the value of the refractive index. Typically, the value of the refractive index is measured at a wavelength corresponding to the wavelength of the sodium yellow light (D-line, $\lambda = 589$ nm) at 20°C. In this case, wavelength and temperature are necessarily specified near the refractive index (n_D^{20}).

The refractive index is determined using devices called refractometers (Fig. 3.6.2). When working on a refractometer one must open the hemisphere measuring head 1 and clean the planes of prisms with cotton wool soaked with ether. Then bring the prisms in the horizontal position.



1- measuring head;
2 - lighting mirror;
3- measuring prism;
4 - screw; 5 - eyepiece;
6 – thermometer;
7 – flywheel.

Figure 3.6.2: Refractometer IRF.

On the surface of the measuring prism 3 apply a few drops of the test liquid with pipette and close measuring head. Lighting mirror 2 is set so that the light from the source travels into light passing through the window and the lens, thus, the field of view become uniformly illuminated.

Rotating the flywheel 9, one finds in the eyepiece 5 boundary between light and shadow (fig. 3.6.3). If this boundary is blurred or colored, one achieves its clear image rotating screw 4. With the flywheel 9 one accurately combines the boundary between light and shade with a cross grid and watches the value of the refractive index on the scale (by the position of the horizontal marking of the grid). Illumination of the scale is controlled with the side mirror.

The refractive index is measured with an accuracy of four decimal places.

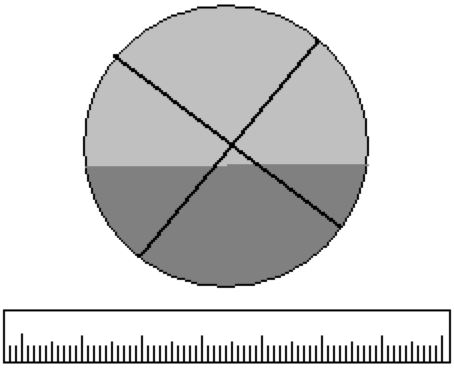


Figure 3.6.3

4. Methods for the isolation and purification of substances

Various methods are used for isolation (or separation) of the obtained compounds from the reaction mixture and for their further purification, as well as for purification of the starting reactants. The isolation of individual compounds is also mandatory when one works with natural compounds.

The most important of these techniques are based on the separation of substances by particle size or density (filtration, precipitation), or by the ability to be distributed between the two different phases (distillation, recrystallization, sublimation, extraction, chromatography).

In accordance with the standard for the grade of purity (State Standard: GOST), reagents are divided into: a) highly purified (particularly pure); b) chemically pure (reagent grade), c) pure for analysis (analytical grade), g) pure (p.); and others. The substances, suitable for laboratory work in inorganic chemistry, are labeled “reagent grade” and “analytical grade”.

4.1 Distillation

Distillation takes the most important place among the many methods of purification and separation of organic compounds. *Separation of a mixture of liquids via distillation can be achieved when vapor, generated by the distillation, has a different composition in comparison with the liquid.*

Solutions, which can not be separated by distillation, are called **azeotropic (**or **boiling without separation**), since they distilled without change in composition and boiling temperature.

***Distillation** is a process by which distilled liquid is converted into vapor via heating; the resulting vapor is condensed in the condenser, and a pure distillate enters further into the receiver.*

Due to evaporation of liquid, equilibrium between liquid and vapor, and therefore, a certain vapor pressure are established above all liquids. The magnitude of this pressure depends on the temperature and nature of the liquid. With increasing temperature, the vapor pressure above a liquid increases substantially. This can be demonstrated on the example of the water (Fig. 4.1.1).

The temperature, at which the vapor pressure is equal to external pressure, is called ***boiling point***.

The lower atmospheric pressure, the lower the boiling temperature of this liquid (see. Fig.4.1.1c). So, on top of Mount Elbrus, where the air pressure is half of the normal, ordinary water boils at 82° C (at normal pressure ordinary water boils at 100° C). Conversely, if it is necessary to raise the temperature of the boiling liquid, then it is heated at elevated pressure. On this fact, for example, the work of pressure cookers, where food containing water can be cooked at a temperature above 100° C without boiling, is based.

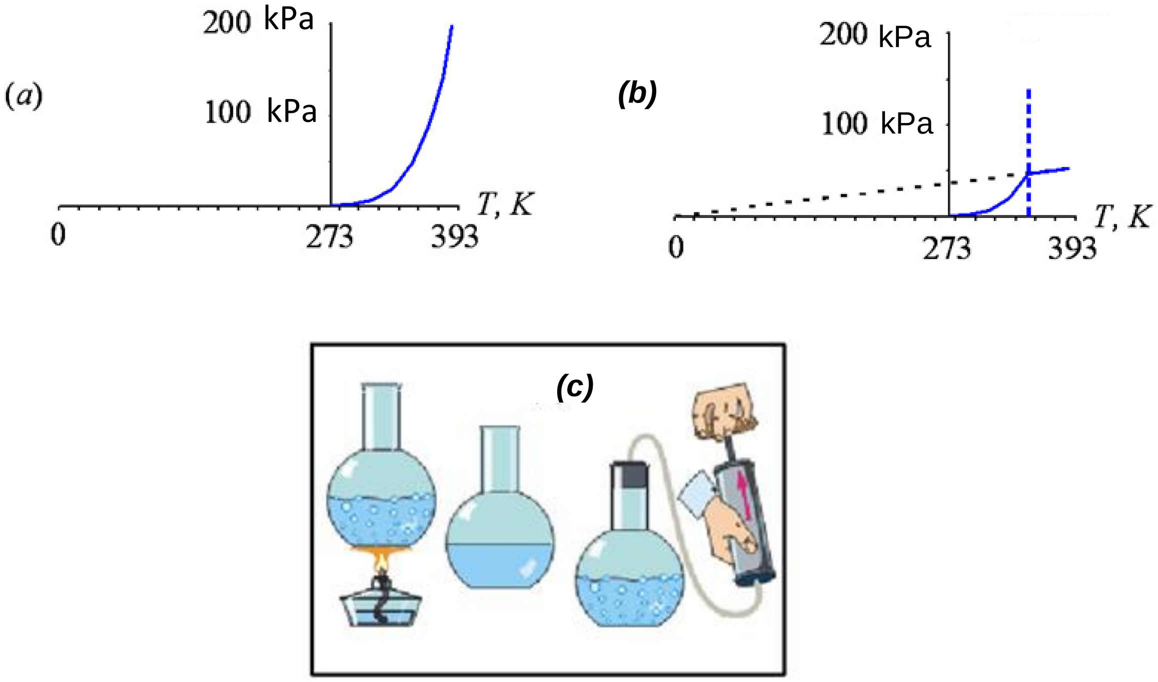


Figure 4.1.1.

(a) Dependence of saturated water vapor pressure (i.e. steam pressure) on temperature;
(b) Dependence of water vapor pressure on temperature before (to the left from vertical dashed line) and after all the water has evaporated;
(c) Boiling liquid in flask on burner (left flask); cooled liquid in flask (middle flask); the liquid boils again on lowering of pressure in the flask (right flask).

Distillation methods are divided into two groups:

a) a simple distillation;
b) fractional distillation (rectification).

According to distillation conditions there are three types of distillation:

a) at atmospheric pressure;
b) at reduced pressure (vacuum distillation);
c) with steam.

4.1.1 Simple distillation at atmospheric pressure

Simple distillation is used for separation of liquids, which significantly differ in their volatility, for example, for distilling off the solvent from the non-volatile residue or for distillation of liquids, which greatly differ from each other in their boiling points.

Satisfactory separation is possible, provided that the difference between boiling points of distilled liquids is at least 80° C. At lower difference between boiling points the separation can be achieved using a reflux condenser (coiled condenser).

Distillation of liquids is carried out from the Wurtz flask.

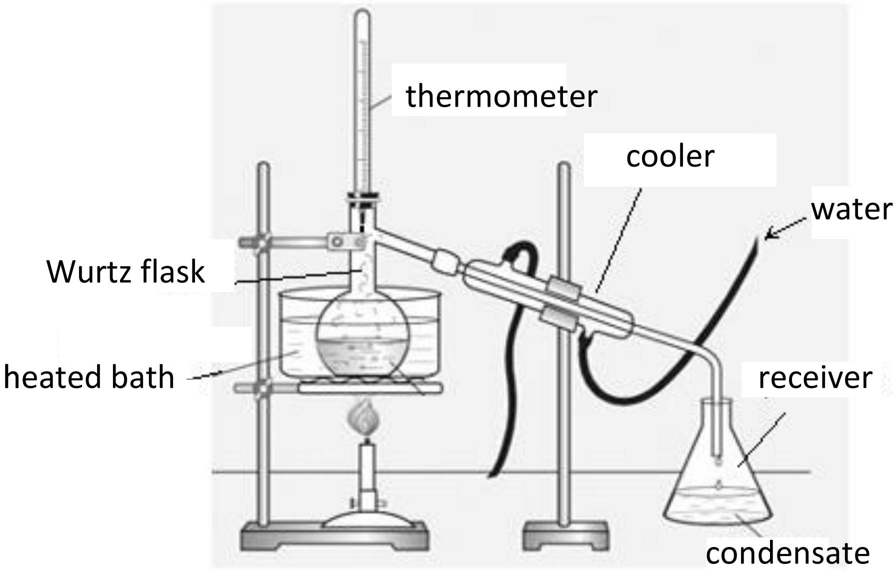


Figure 4.1.2. Scheme of device for a simple distillation;

Liquid in the flask is evaporated by using one of the types of baths (water, oil, metal-air) as a heat source for even heating and to avoid overheating. The temperature of the bath should not exceed the temperature of the distillable liquid over 25-30° C. Vapors rise in the neck of the Wurtz flask and wash the thermometer bulb with which the temperature of the vapor is watched (or observed).

Then, vapors enter into a descending (down-tending) condenser, from which the condensate flows down to the receiver.

When assembling the installation for distillation one should pay attention to the position of the mercury thermometer bulb for monitoring of the vapor temperature: the mercury thermometer bulb **should be about 0.5 cm below** the outlet tube of the Wurtz flask.

Flat-bottomed, round-bottomed and conical flasks can be used as receivers. To collect volatile liquid the receiver is usually placed in an ice bath.

It is necessary to strictly follow the rule: the interior of the equipment, not intended for work under pressure, should always be connected with the atmosphere!

Most liquids are characterized by a tendency to overheat, i.e., they can be heated above the boiling point, and boil with strong shocks. As a result of the shocks, the thermometer can be knocked out and fluid ejection (or fluid emission) from the flask can happen. These emissions can cause the loss of substance, and, also, if liquid is flammable, they can be the cause of the fire. To avoid overheating of the liquid and for uniform boiling one should put some "steam-pebbles" ("boiling chips") to the distillation flask. Small pieces of porous materials (bricks, unglazed porcelain, pumice) can be used as "steam-pebbles".

The distillation flask should not be filled more than to 2/3 of its volume. Distillation is performed with such a rate that the receiver gets not more than 1-2 drops of distillate per second. The substances, distilled in the range of 1-2° C, are considered to be almost pure. If the fluid contains a small amount of volatile impurities, they precede the main fraction (boiling in the range of 1-2° C) in the form of so-called “preliminary fraction”. The preliminary fraction should be collected in a separate receiver as long as the temperature is lower than the boiling point of the main substance. When the temperature rises to the boiling point of the main substance, one should change the receiver and to collect the main fraction. **While the main fraction is distilled, the temperature remains constant. The sharp rise in temperature indicates that the distillation of the next component (a high-boiling component) has started.**

The distillation is stopped in case of a sharp rise in temperature, or when only 1-2 ml of fluid remains in the distillation flask (i.e. when liquid just covers "steam- pebbles").

Table 4.1

Fraction	Temperature range	Volume
I (Preliminary fraction)	$t_{\text{start of distillation}} - t_{\text{boiling}}$	V_1
II (Main fraction)	$\sim t_{\text{boiling}}$	V_2
III (Remains)	$> t_{\text{boiling}}$	V_3

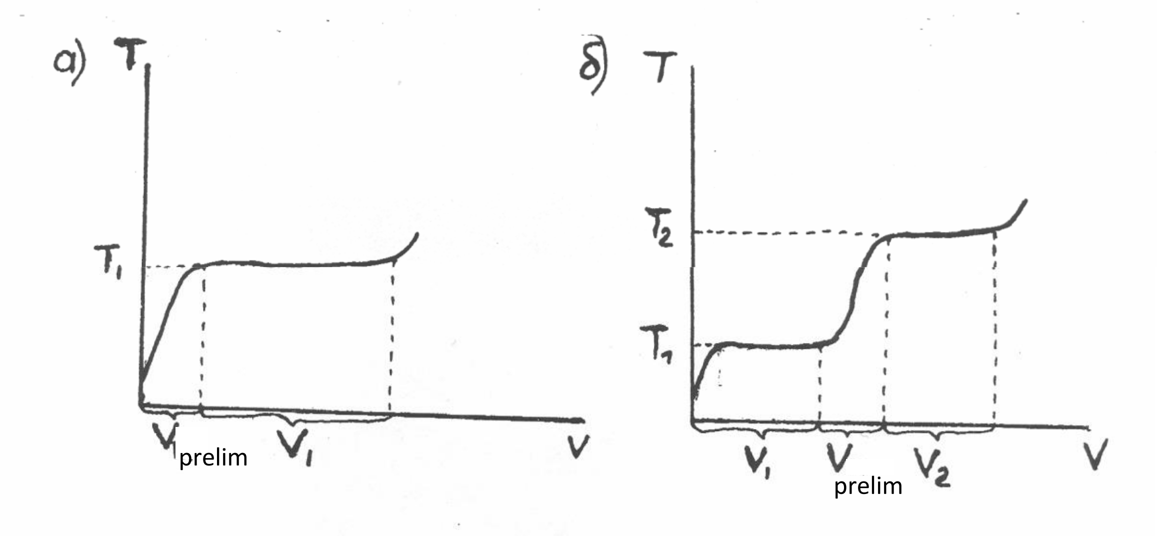


Figure 4.1.3. Plot, showing relationships between the volume of distillate and the distillation temperature:

a) purification of the individual substance;
b) separation of the mixture of the two substances.

4.1.2 Simple distillation in vacuum

Distillation of substances, partially or fully decomposing at the boiling point at atmospheric pressure, is carried out at reduced pressure (*vacuum distillation*).

A water jet and oil pumps are applied to create vacuum. For a rough estimate of the boiling point at a particular external pressure it is sufficient to know the boiling temperature at any other known pressure.

The rule of thumb for a rough estimate of the boiling temperature: ***if the external pressure decreases by one half (or by 50 percent), the boiling point is lowered by about 15° C.*** For example, substances, boiling with decomposition at 350°C / 760 mm Hg. Art., can be distilled without decomposition at about 160-210°C / 10 mm Hg. Art., at 100-130°C / 0.01 mm Hg. Art. and at 40-50°C / 0.0001 mm Hg. Art.

Using the nomogram (Fig. 3.5.1), one can check whether the observed boiling point of the substance, distilled under the low pressure, corresponds to the literature data. If we superimpose a line on nomogram, so that the line cross the right scale in the point, corresponding to the distillation pressure, and cross the scale B (scale in the middle of nomogram) in a point, corresponding to the boiling point of distilled liquid at atmospheric pressure, then, the point of intersection of this line with the left scale will show (with a certain degree of approximation) the boiling temperature of liquid in a vacuum device with achieved low pressure.

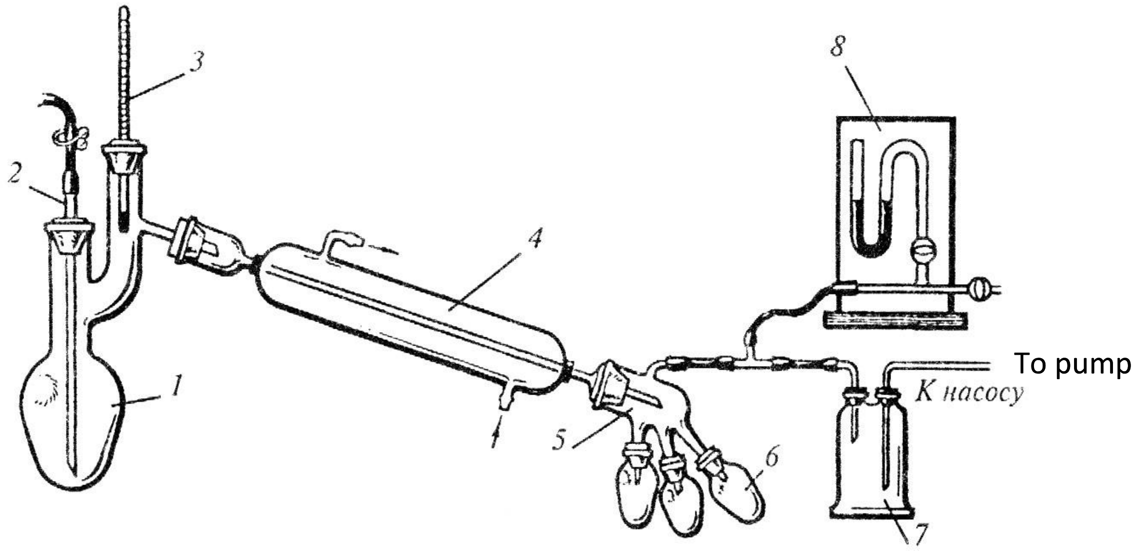


Figure 4.1.4. Installation for distillation under reduced pressure:
1 - Claisen flask or round-bottomed flask with Claisen attachment;
2 - capillary tube, connected to a rubber hose with a clip; 3 - thermometer;
4 - a cooler-condenser; 5 – allonge (rider); 6 - receiving flask;
7 - a safety bottle; 8 - the pressure gauge (or manometer).

When working with the vacuum one should follow some safety rules. Claisen flask should be filled only after the equipment has been tested for leaks. Heating should be started only after reaching the desired vacuum. The distillation in vacuum is carried out in goggles. At the end of the distillation the system must be cooled, and, then, slowly let the air in the system.

4.1.1 Experiment "Purification and distillation of liquids"

Assemble the unit according to Fig. 4.1.1. Pour solution of copper sulphate and put steam-stones in the Wurtz flask. Heat the solution to boiling point. The liquid in the flask evaporates and dissolved therein non-volatile substances remain in solution, increasing its concentration, or precipitate. Liquid vapors are condensed in the condenser and drain through allonge to the receiver. The liquid, free from dissolved solid substances, is collected in the receiver. Distill 20-30 mL of water. What is the purity of the obtained water? Does the obtained water contain copper sulfate? How to check this?

4.1.3 Fractional distillation

Fractional distillation is used to separate liquid mixtures, when the boiling temperatures of components differ from each other by less than 80° C (this means that it is not possible to separate the components of the mixture via a simple distillation).

Unlike the conventional simple distillation, during which the vapor and condensate pass through the device once in a single direction, **fractional distillation is based on interaction of the rising vapors with the flowing down condensate (reflux), produced as a result of partial condensation of vapors**. As a result of repetitive partial evaporation and condensation, the vapors are enriched with low-boiling component and a high-boiling component flows down with reflux (phlegm) in flask for distillation.

Similarly to the behavior of a pure liquid, the binary mixture of two completely-mixing with each other liquids starts to boil at a temperature at which the total vapor pressure of both components is equal to the external pressure.

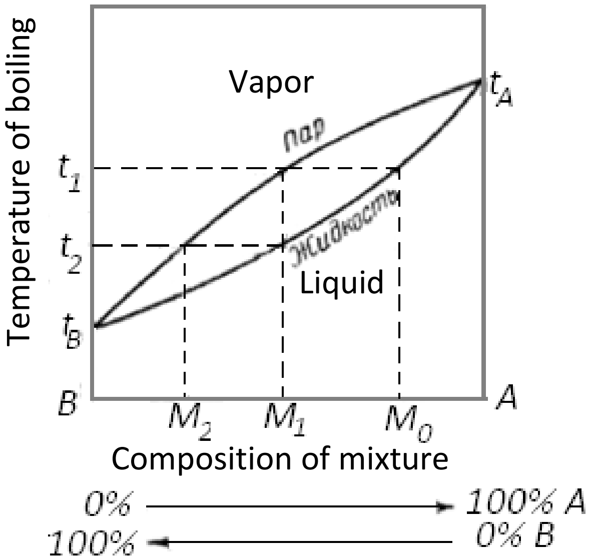


Fig. 4.1.5. Diagram of phase equilibrium

It can be seen from the diagram of phase equilibrium, that at any boiling temperature the vapor phase contains more low-boiling component, than the liquid phase; also, each boiling point strictly corresponds to certain composition of the liquid and to certain composition of the vapor. Thus, the vapor, generated from the boiling binary mixtures, always contains both components, but is enriched in more volatile one (composition M1). If to completely condense such vapor, liquid is obtained with the same composition as the vapor. The secondary distillation of this liquid gives vapor (composition M2), more enriched in low-boiling component. Consequently, as a result of frequent repetition of the phase equilibrium conditions (i.e. as a result of repetition of distillation) for each first fraction, one may ultimately receive only low-boiling component of the mixture. Accordingly, the last fraction consists of pure high-boiling component of the original mixture. This is essentially the principle of separation by fractional distillation.

The functioning of deflegmators (reflux condensers) is in partial condensation of vapor of higher boiling liquid (the partial condensation occurs in deflegmator during incomplete cooling of vapor of boiling solution). Obtained intermediate condensate is called phlegma (from the Greek “phlegma” - mucus, sputum). Phlegma flows back into the flask and the vapor become enriched in component with a lower boiling point, then, in the cooler-condenser the vapor is subjected to complete condensation. Cooling occurs due to partial condensation of the vapor and its temperature drops. As seen from the diagram of boiling, when the temperature of the vapor is lowered from t_1 to t_2, a content of low-boiling component increases from M_1 to M_2. Various types of dephlegmators are used in laboratories. Some types of dephlegmators are shown in fig.4.1.6

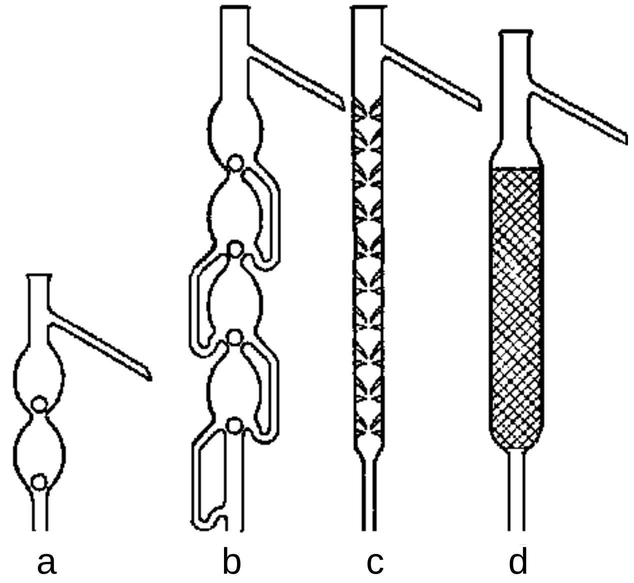


Fig. 4.1.6. Deflegmators (reflux condensers):
(a) and (b) - ball-type; (c) figured; (d) with an attachment.

Experiment 4.1.2. The separation of liquid mixtures by fractional distillation.

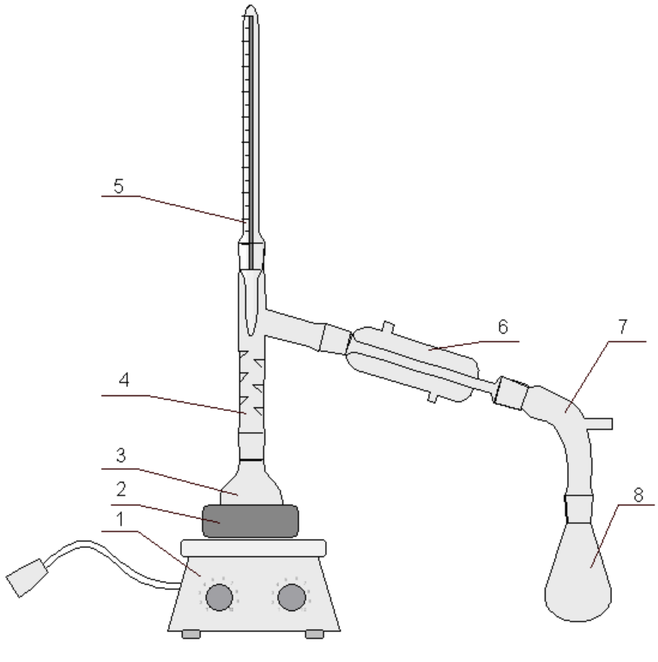


Figure 4.1.7 Apparatus for fractional distillation of liquids:

1, 2 - stove or heating mantle,
3 - flask
4 – dephlegmator (reflux condenser)
5 - Thermometer 6 – cooler-condenser,
7 – rider (allonge), 8 - receiver.

Procedure:

1. Assemble the apparatus for fractional distillation (Fig.4.1.7). Pour the liquid, received for distillation, in a round-bottomed flask. Put a few "steam-pebbles" ("boiling chips") in the round-bottomed flask. Supply water for cooling in Liebig condenser. Set thermometer just below the outlet tube. Use a water, air, or sand bath for heating of the flask with the liquid.
2. Write in the laboratory journal the volume of liquid, received for distillation.
3. Turn heating unit and adjust it to create a uniform and smooth boiling of the mixture.
4. Distillation of low-boiling liquid is performed at a constant temperature at a rate of about one drop per second. Record the temperature of the start of the distillation in the laboratory journal. Collect the distillate in the receiver until a constant temperature is achieved (V_o). At a constant boiling temperature (T_1) collect a first fraction (V_1), low boiling component of the mixture, in the first receiver. When the temperature of distillation increases collect intermediate fraction ($V_{intermidium}$) in the second receiver. When constant temperature (T_2) is established again, collect the second fraction (V_2), high-boiling substance, in the third receiver.

Mixture of substances with a difference between boiling temperatures above 100^{o}C can be distilled without intermediate fraction.

5. Record in the table the boiling temperatures and volumes of the obtained fractions.
6. The distillation is stopped when 1-1.5 ml of distilled liquid remains in the flask.

Note: It is forbidden to evaporate the liquid completely!

7. Upon completion of the distillation disable the heating device, let the device to cool, then, carefully disassemble the apparatus for fractional distillation.
8. Pass the distilled substances to lab employee, which signs the data, presented in your laboratory journal. On the base of the obtained results plot the dependence of the amount of distillate on distillation temperature (Fig. 4.1.7), calculate the percentage of the components in the initial mixture.

Table 4.2

Fraction	**Temperature range**	**Volume**	**% conent of components in mixture**
Preliminary fraction **I** **II** (intermediate fraction) **III**	**$< T_1$** **T_1** **$T_1 - T_2$** **T_2**	**V_o =** **V_1 =** **$V_{intermidium}$ =** **V_2 =**	

4.1.4 Distillation with steam

Steam distillation is used:

1) for the separation of mixtures of substances, if only one of the subsances is volatile with steam;

2) for purification of substances (in order to get rid of resinous impurities);

3) if it provides a more complete separation of volatile substances, than distillation under reduced pressure.

The principle (or essence) of steam distillation: the high-boiling, non-miscible or little miscible in water substances evaporate by passing of steam through them; then, they are condensed together with the vapor in the cooler-condenser and fall into the receiver.

In order to find out whether the substance is volatile with steam, one should heat a small amount of the substance in a tube with 2 ml of water. Above this test tube the bottom of another tube with ice is placed. If the drops, condensing on the cold bottom of the tube with ice, are turbid – the substance is volatile with steam. Steam distillation is carried out in the installation shown in Fig. 4.1.8. It consists of: a steam generator; the tube through which the steam enters into the flask; a distillation flask; a cooler-condenser and a receiver to collect distillate. Steam generator is filled with water and connected via a tee to the distillation flask, containing the substance to be distilled. The distillation flask is connected to the cooler-condenser by the glass tube. To avoid sudden transfer of liquid from the distillation flask into the receiver, the distillation flask is inclined, as shown in Fig. 4.1.8. The steam generator is heated with electrical hotplate and, simultaneously, the distillation flask is heated. As soon as the steam starts to enter in the system, fasten the clamp on the rubber tube of tee. Thus, steam enters to the distillation flask quickly via the discharge tube and, then, together with the distillable substance enters the cooler-condenser.

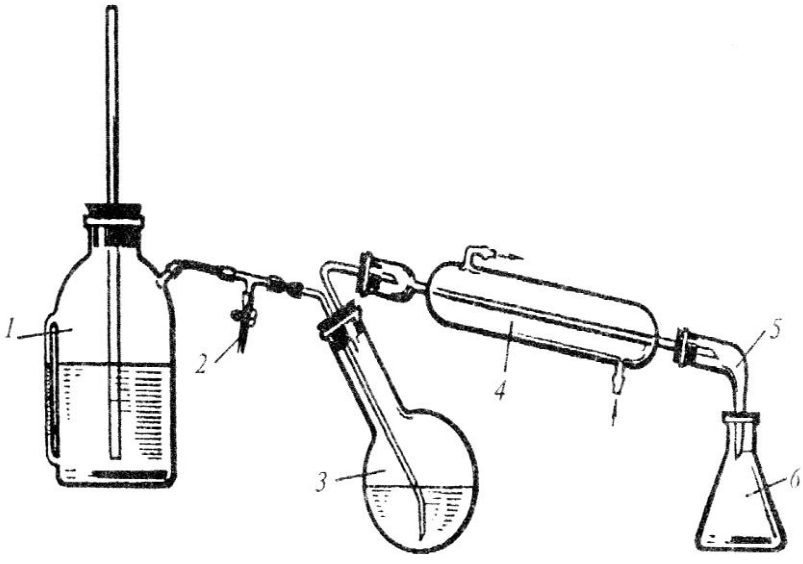


Figure 4.1.8 Apparatus for steam distillation:
1 - steam generator; 2 - tee with a clamp; 3 - distillation flask;
4 – cooler-condenser; 5 - allonge; 6 - receiving flask.

Some time later emulsion (the residue) accumulates in the receiving flask. The distillation is carried out to until the sample of the distillate does not separate into two phases upon cooling. When pure water begins to distill, one should finish the distillation, opening the clamp on the tee and stopping the heating of the steam generator. The distillate should be separated in a separating funnel and, then, distilled substance should be dried.

If the distilled sample is low-melting solid substance, one should ensure that the cooler-condenser is not clogged. Any substance that accumulates in the cooler-condenser can be quickly removed from it by removal of water from the cooling jacket for a few minutes; in result, the steam will transfer the molten solid substance into the receiver.

4.2 Recrystallization

Recrystallization is the simplest method of separation and purification of solid substances. It is based on the difference in solubility of substances in different solvents.

Crystallization method consists of the following steps:

- choice of solvent
- dissolving of solid substance in a minimum amount of boiling solvent (preparation of a saturated solution);
- filtering of the hot solution to remove insoluble impurities (if present);
- cooling the solution to form crystals;
- filtering the crystals from the mother solution and drying of the obtained crystals.

Criteria for the selection of the solvent:

- solvent must be chemically inert to the substance to be purified
- substance should not be soluble at room temperature, and has to be soluble only upon heating
- the boiling temperature of the solvent should be (if possible) below the melting point of the substance
- among the similar solvents choose non-toxic, non-flammable and non-expensive.

The general rule of solubility - "**like dissolves like**", i.e. polar compounds are more soluble in polar solvents, than in non-polar, and vice versa.

During the crystallization from combustible or volatile solvents, one commonly uses the installation for boiling (for refluxing), consisting of round-bottomed flask with reverse condenser (i.e. with Liebig condenser, see Fig. 4.2.1). If water is used as solvent, the recrystallization process is carried out in a flask or beaker.

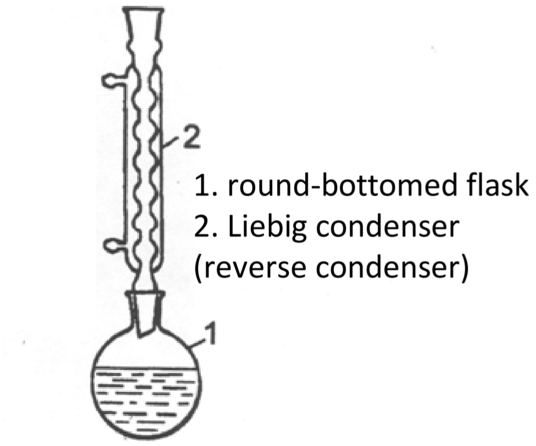


Fig. 4.2.1. Installation for boiling (for refluxing) with Liebig condenser (reverse condenser).

4.2.1 Experiment "Recrystallization"

Procedure:

1. Receive the substance to determine its melting temperature, weigh. Record the results in a laboratory register laboratory journal
2. Select the solvent.

<u>Selection of the solvent.</u>

With the end of spatula put the substance into test tube, pour 1 ml of solvent in the test tube and shake the test tube. If the substance is not dissolved or not fully dissolved, the test tube should be gently heated (*if the solvent is flammable, the tube must be stoppered with an air condenser!*). If the substance is not completely dissolved, add an additional 1 ml of the solvent and heat the test tube again (substance is considered to be insoluble in solvent if the substance is not dissolved after addition of 3 ml of the solvent). Then, cool the tube with the solute and observe the formation of crystals. This process is repeated with other solvents. It is recommended to try such solvents as water, alcohol, and any non-polar solvent (hexane, toluene).

If the substance is very well dissolved in a solvent and does not dissolve in another solvent, the crystallization is performed from a mixture of both solvents. For this, the later solvent is added dropwise to the hot solution of the substance in the former solvent by until stable dregs are formed. This solution is heated until clear and, then, allowed to crystallize.

All observations and conclusion should be written in the laboratory journal (lab notebook).

3. Place the substance in the flask, add a minimal amount of solvent (solvent should only cover the crystals) and heat up the flask. Then, during the

heating add small portions of the solvent until complete dissolution of the substance.

4. The resulting saturated solution should be cooled, precipitated crystals should be filtered and dried.

If the solution is colored or contains insoluble impurities, it should be passed through a hot funnel for the hot filtration (Fig. 4.2.2) and only then slowly cooled.

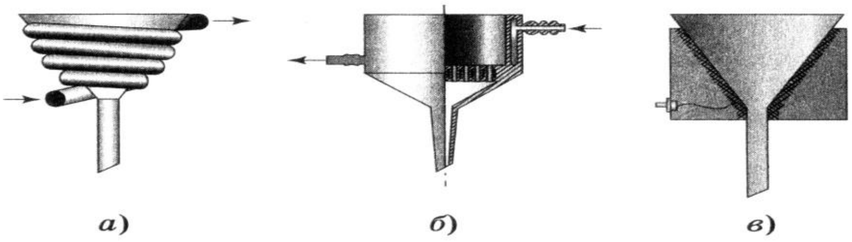


Figure 4.2.2 Funnels for hot filtration with steam (a), water (b) and an electric heating (c).

5. Weigh the obtained crystals in order to determine the loss factor.
6. Determine the melting point of the purified substance, compare it with reference data and draw a conclusion.

Experiment 4.2.2 Isolation of solute via evaporation

1. Using spatula, put a small amount of crystalline sodium chloride (salt) in 100 ml beaker. Add 50 ml of distilled water in the beaker and stir the solution until the salt is completely dissolved.

2. Put the porcelain dish on wire gauze with asbestos center, pour into the dish a small amount of salt solution and heat it to evaporate the water.

3. Put away the burner and let the dish to cool. Using spatula, collect the salt from the walls of the dish.

4.3 Sublimation

Sublimation is the evaporation of substance, when the substance is heated below its melting temperature, and, then, the condensation of vapor on the cooled surface (fig. 4.2.3). Purification of the solid substance by sublimation is possible only if the vapor pressure of the solid substance is higher than the vapor pressures of the impurities. The best results are achieved when the vapor pressure of the solid substance corresponds to the applied pressure. For example, stilbene is sublimated at a temperature of 100 ° C and a pressure of 20 mm Hg. Art.

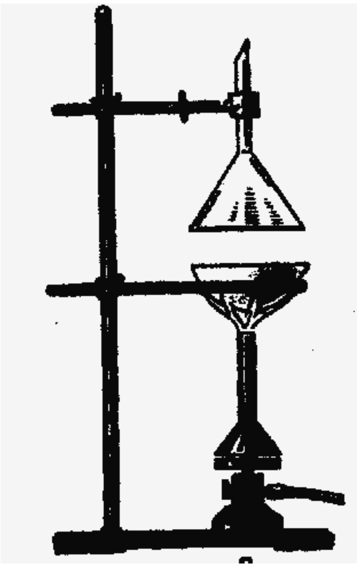


Figure 4.2.3

Sublimation is conducted under vacuum in sublimator (apparatus) or at atmospheric pressure in a porcelain dish, covered at the top by filter with numerous needle pierced holes and a glass funnel. To avoid contamination of sublimate, before sublimation the solvent and other volatile products should be removed from the substance being purified.

Experiment 4.3.1 Purification of substance by sublimation.

1. Receive contaminated substance (naphthalene, benzoic acid, iodine) from a teacher and weigh the substance. Leave 0.1 g of starting material to determine the melting temperature. In literature (in reference book) find the melting point of the pure substance.

2. A small porcelain cup cover with sheet of filter paper with fine punctures (20-30 holes) and press tightly the filter paper with inverted glass funnel (the opening of the funnel is covered with cotton).

3. Porcelain cup with sample should be carefully heated to a temperature, which is 10-20° C below the melting point of the substance. Heating should be carried out until crystals form on the surface of a glass funnel.

4. Stop the heating of installation, carefully cool it, then, collect crystals and weigh them. Determine the melting point of the samples before and after

recrystallization. Compare the obtained data with the reference. Calculate the yield of pure substance in %.

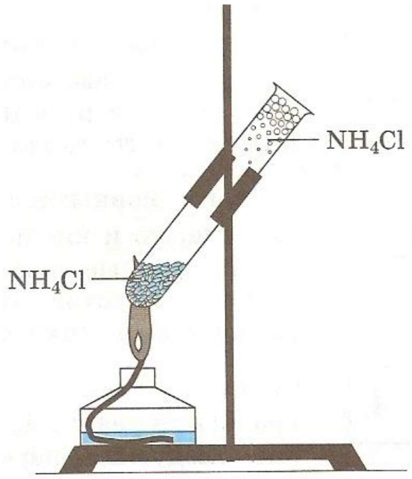


Figure 4.3.1 Sublimation of ammonium chloride

4.4 Extraction

Extraction - a method of separation of solid or liquid mixtures, based on the difference in solubility of components of the mixture in different solvents. The extraction is used for concentration, purification or separation of components of the mixture.

Depending on the state of aggregation of the extractable substance, the extraction can be divided into two types:

- extraction of solid substances (system solid - liquid);
- extraction of liquids (system liquid - liquid).

Solid phase extraction consists in extracting (i.e. taking out) of organic compounds from a solid by treatment with an organic solvent (extractant). In the course of liquid phase extraction one phase is usually an aqueous solution, the other – organic solution. The extractant should have minimum solubility in water and should be selective for the extractable substance.

Typically extraction is carried out from the water phase (neutral, acidic, basic) to solvent, immiscible with water (e.g. dichloromethane, chloroform, ethers, etc.). In the case of polar products (such as alcohols, carboxylic acids, amines) prior to extraction aqueous phase is saturated with sodium chloride (salting).

The design of the extraction apparatus and extraction techniques depend on the type of the extracted substance. So, in case of the distribution of substance between two immiscible liquids, such as aqueous and organic phase, the extraction is conveniently carried out in a separating funnel. Placed in a separating funnel, the mixture of the two liquids is shaken several times and, then, the mixture is left to exfoliate. The lower layer is drained through a tap of the separating funnel into a separate container, and, then, supernatant, remaining into the separating funnel, is drained into the other container. For complete extraction, the operation is repeated several times (small portion of fresh extractant should be added before each operation).

Application of Soxhlet apparatus is effective in the extraction of solid substances.

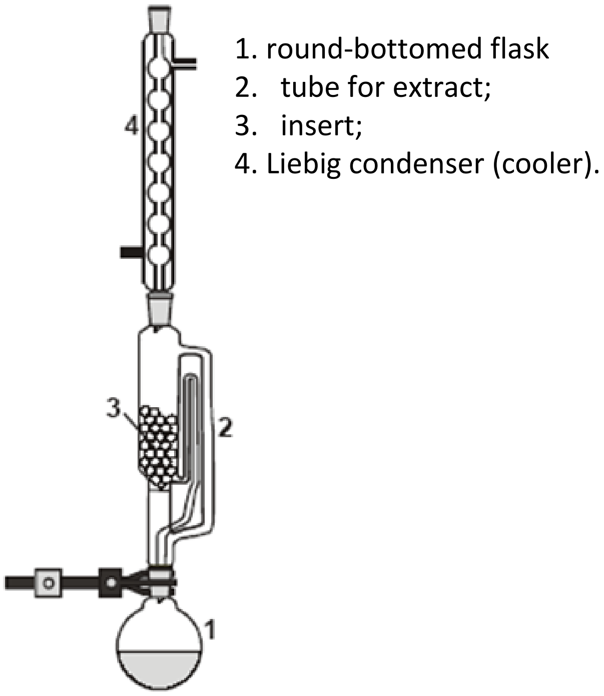


Figure 4.3.2 Soxhlet apparatus.

It consists of three parts: the flask to evaporate the solvent, a Liebig condenser and an extractor (Figure 4.3.2.). Before you start the flask is fixed on a stand, then, it is connected to the extractor. The extractor is loaded with extractable substance, wrapped in filter paper or placed in a special holder. The extractor is connected to the flask through two tubes. One of them, a wide, is intended for supplying of solvent vapor to the extractor, the second tube, curved in a knee and working as a siphon, is intended to drain the solution from the extractor into the flask. Solvent vapors, condensed in a cooler, enter the extractor, gradually filling the volume of it. When the liquid level reaches the drain pipe elbow, the extract drains back into the flask. During repetitive drain cycles the solvent becomes enriched with extractable substance.

If extractable substance is colored, then one can judge about the completion of extraction by the disappearance of the color of the solvent in any cycle of discharge. In the case where the extractable substance is colorless, a few methods are used to judge about the completion of the extraction process: in case of neutral solution, indicators are used; if the solution is not neutral, the refractive index is measured or the disappearance of the spot on a watch glass during evaporation of specially selected sample is observed. If the measured refractive index is equal to the refractive index of the pure solvent – the extraction process is completed.

5. Solutions. Ways to express composition of solution. Determination of composition of solution.

Solution is a homogeneous system consisting of two or more independent components (i.e. solution is a homogeneous mixture of two or more substances). The ratio between components of solution can vary. Components of solution are solvent and dissolved substance. Quantitatively dominant component, which do not change its state of aggregation during the preparation of the solution, is called **solvent**. The other components of the solution are called **solutes**. However, this division is arbitrary. Solutions, which contain large amounts of dissolved substance, are called **concentrated**, and, solutions with a low content of dissolved substances are called **diluted**. Concentrated solutions can be prepared only with use of readily soluble substances, while dilute solutions can be prepared with use of substances with any solubility.

The most important characteristic of solutions, affecting their properties, is a composition, i.e. the content of the solute in definite mass or in definite volume of the solution or solvent.

There are various ways of expressing of composition of solutions. Composition of the solution can be expressed in dimensionless (share, ratio) and in dimensional values (molar concentration, mass concentration, molality, titer (titre), etc.).

Concentration is the ratio of the mass or amount of a dissolved substance to the volume of the system (i.e. of the solution).

$$\omega_{solute} = \frac{m_{solute}}{m_{solution}}$$

Mass fraction - the ratio of the mass of solute to the mass of the solution. Mass fraction represents a mass of solute (in grams), which is contained in 100 grams of solution. Mass fraction is expressed as a decimal share or in percentage terms (percentagewise); for example: ω = 0.01 or ω = 1% - percentage concentration.

It is easier to measure volume of solution rather than its weight. Knowing the weight and the volume of the solution one can calculate the density of the solution:

$$\rho_{(solution)} = m_{(solution)} / V_{(solution)}.$$

$$C = \frac{\nu}{V}$$

The solution density is changed with the change in the content of dissolved substance in the solution. Using the data for a solution with a certain mass fraction of solute (usually, such data are presented in tabulated form), one can determine the density of the solution, and, vice versa, knowing the solution density one can determine the mass fraction of the substance.

The molar concentration (denoted as C) - the ratio of the amount of solute (ν) to solution volume (V). The molar concentration shows how many moles of solute are contained in one liter of solution.

Molar concentration of equivalent (so called “normal concentration” or “normality”) - the ratio of equivalents of solute (ν_{EQV}) to volume (V) of the solution. Molar concentration of equivalent represents the number of gram-equivalents of the substance in one liter of solution:

$$C_E = \frac{\nu_E}{V}$$

Experiment 5.1 Preparation of the solution with a preselected (prescribed) mass fraction of solute by use of weighted sample of solid substance and water

1. Receive an individual task from a teacher:

   Prepare an aqueous solution of ………………
   with volume ……………………………………….
   with a mass fraction of ……………………………

2. Perform the necessary calculations and determine

   - mass of solution,
   - mass of solute,
   - mass of solvent,
   - volume of solvent.

   Solute is selected from the solids in Table 5.1.1.

   If the preselected (prescribed) value of the mass fraction of the solute does not coincide with the tabulated values, then one should use *interpolation method*.

3. Using balances, weigh the calculated amount of substance to be dissolved. Measure the required volume of distilled water with a graduated cylinder. Prepare a solution in a beaker (on preparation the solution should be stirred with a glass rod). Measure the temperature of the solution with thermometer. If the temperature deviates from 20 ° C, it is necessary to heat or to cool the solution.

4. Pour the solution into a graduated cylinder of volume 50 cm^3, gently put the hydrometer into the cylinder. Observing the position of lower liquid meniscus on hydrometer scale, determine the density of the prepared solution.

Attention! *It must be remembered that during the determination of the density of the solution, the hydrometer must not touch the walls of the cylinder. After each measurement, the hydrometer should be washed, rubbed with a filter paper and carefully placed in the appropriate slot in the box.*

5. Determine the value of the mass fraction of the solute in the prepared solution (ω_{exp}) from the measured density ρ and data presented in table. 5.1.1. If necessary, use *the interpolation method*.

   (ω_{exp})………………………..

6. Compare the found value (ω_{exp}) with the value of mass fraction from the table (ω_{theor}). Determine the value of the relative error of experiment ($\eta_{relative}$):

$$\eta_{relative} = \frac{\omega_{\exp}}{\omega_{theor}} 100\%$$

7. Express quantitative composition of the solution with a preselected mass fraction in other ways. To do this, calculate the values of the mass, molar and normal concentrations and molality of this solution.

Table 5.1.1

The dependence of the density of the solution ($\rho_{20}{}^{0}$ (g/cm^3)) on mass fraction of solute

%	KOH	NaOH	NaCl	$MgSO_4$	$CaCl_2$	H_2SO_4	HCl	Na_2SO_4
2	1,017	1,021	1,013	1,018	1,015	1,012	I,008	1,019
4	1,033	1,043	1,027	1,039	1,032	1,025	1,018	1,040
6	1,049	1,065	1,041	1,060	1,049	1,038	1,028	1,061
8	1,065	1,087	1,056	1,032	1,066	1,052	1,038	1,082
10	1,083	1,109	1,071	1,104	1,084	1,066	1,047	1,103
12	1,108	1,137	1,086	1,126	1,102	1,080	1,057	1,124
14	1,126	1,153	1,101	1,148	1,120	1,095	1,068	1,146
16	1,144	1,175	1,116	1,173	1,139	1,109	1,078	-
18	1,165	1,197	1,132	1,196	1,158	1,124	1,088	-
20	1,183	1,219	1,149	1,220	1,173	1,139	1,098	-
22	1,206	1,241	1,164	1,245	1,197	1,155	1,108	-
24	1,225	1,263	1,180	1,270	1,218	1,170	1,119	-
26	1,246	1,297	1,197	-	1,288	1,186	1,129	-
28	1,267	1,306	-	-	-	1,202	1,139	-
30	1,288	1,328	-	-	-	1,219	1,149	-
32	1,308	1,349	-	-	-	1,235	1,159	-
34	1,330	1,370	-	-	-	1,252	1,169	-

36	1,355	1,390	-	-	-	1,268	1,179	-
38	1,373	1,410	-	-	-	1,286	1,189	-
40	1,395	1,430	-	-	-	1,303	1,198	-
42	1,417	1,449	-	-	-	1,321	-	-
44	1,445	1,469	-	-	-	1,338	-	-

Task 1

1. Receive the solution of salt – $NaCl$.
2. Using a hydrometer, measure the density of the solution.
3. Determine the mass fraction of the solute in the solution from the measured density and data presented in table. 5.1.1.
4. Calculate the normality (C_N), the molarity (C_M) and titer (T) of the solution.
5. Calculate the volume to which it is necessary to dilute 40 ml of this solution to obtain normality (C_N) 2.0 N.
6. Using hydrometer and calculations, check the concentration of the diluted solution.
7. Calculate the error.

Task 2

1. Receive the solution of acid – HCl.
2. Using a hydrometer, measure the density of the solution.
3. Determine the mass fraction of the solute in the solution from the measured density and data presented in table. 5.1.1.
4. Calculate the normality (C_N), the molarity (C_M) and titer (T) of the solution.
5. Calculate the volume to which it is necessary to dilute 50 ml of this solution to obtain normality (C_N) 1.2 N.
6. Using hydrometer and calculations, check the concentration of the diluted solution.
7. Calculate the error.

Task 3

1. Receive the solution of salt – Na_2SO_4.
2. Using a hydrometer, measure the density of the solution.
3. Determine the mass fraction of the solute in the solution from the measured density and data presented in table. 5.1.1.
4. Calculate the normality (C_N), the molarity (C_M) and titer (T) of the solution.

5. Calculate the volume to which it is necessary to dilute 40 ml of this solution to obtain normality (C_N) 0.5 N.

6. Using hydrometer and calculations, check the concentration of the diluted solution.

7. Calculate the error.

Task 4

1. Receive the solution of acid – HNO_3.

2. Using a hydrometer, measure the density of the solution.

3. Determine the mass fraction of the solute in the solution from the measured density and data presented in table. 5.1.1.

4. Calculate the normality (C_N), the molarity (C_M) and titer (T) of the solution.

5. Calculate the volume to which it is necessary to dilute 40 ml of this solution to obtain normality (C_N) 2.0 N.

6. Using hydrometer and calculations, check the concentration of the diluted solution.

7. Calculate the error.

Task 5

1. Receive the solution of salt – K_2CO_3.

2. Using a hydrometer, measure the density of the solution.

3. Determine the mass fraction of the solute in the solution from the measured density and data presented in table. 5.1.1.

4. Calculate the normality (C_N), the molarity (C_M) and titer (T) of the solution.

5. Calculate the volume to which it is necessary to dilute 60 ml of this solution to obtain normality (C_N) 2.5 N.

6. Using hydrometer and calculations, check the concentration of the diluted solution.

7. Calculate the error.

Task 6

1. Receive the solution of salt – $K_2Cr_2O_7$.

2. Using a hydrometer, measure the density of the solution.

3. Determine the mass fraction of the solute in the solution from the measured density and data presented in table. 5.1.1.

4. Calculate the normality (C_N), the molarity (C_M) and titer (T) of the solution.

5. Calculate the volume to which it is necessary to dilute 40 ml of this solution to obtain normality (C_N) 0.2 N.

6. Using hydrometer and calculations, check the concentration of the diluted solution.

7. Calculate the error.

Task 7

1. Receive the solution of acid – H_2SO_4.
2. Using a hydrometer, measure the density of the solution.
3. Determine the mass fraction of the solute in the solution from the measured density and data presented in table. 5.1.1.
4. Calculate the normality (C_N), the molarity (C_M) and titer (T) of the solution.
5. Calculate the volume to which it is necessary to dilute 40 ml of this solution to obtain normality (C_N) 1.0 N.
6. Using hydrometer and calculations, check the concentration of the diluted solution.
7. Calculate the error.

Table 5.1.2

Sodium sulfate Na_2SO_4

Concentration (mass fraction, %)	Density (g/cm^3)
1	1,008
2	1,016
4	1,035
6	1,053
8	1,072
10	1,091

Table 5.1.3

Sodium chloride NaCl

Concentration (mass fraction, %)	Density (g/cm^3)
4	1,029
8	1,059
12	1,089
16	1,119
20	1,151
24	1,184
26	1,200
28	1,217

Table 5.1.4

Sulfuric acid H_2SO_4

Concentration (mass fraction, %)	Density (g/cm^3)
2	1,013
4	1,027
6	1,040
8	1,055
10	1,069
12	1,083
14	1,098
16	1,112
18	1,127
20	1,143

Table 5.1.5

Potassium dichromate $K_2Cr_2O_7$

Concentration (mass fraction, %)	Density (g/cm^3)
1	1,0052
2	1,0122
3	1,0193
4	1,0264
5	1,0336
6	1,0408
7	1,0481
8	1,0554
9	1,0628

Table 5.1.6

Potassium carbonate K_2CO_3

Concentration (mass fraction, %)	Density (g/cm^3)
1	1,007
2	1,016
4	1,034
6	1,053
8	1,071
10	1,090
12	1,110
14	1,129
16	1,149
18	1,169
20	1,190
24	1,232
28	1,276
35	1,355

Table 5.1.7

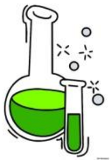
Nitric acid HNO_3

Concentration (mass fraction, %)	Density (g/cm^3)
4	1,022
8	1,044
12	1,068
16	1,093
20	1,119
20,79	1,120
22,38	1,130
23,94	1,140
25,48	1,150
27	1,160

Table 5.1.8

Hydrochloric acid HCl

Concentration (mass fraction, %)	Density (g/cm^3)
4	1,019
8	1,039
12	1,059
16	1,079
18	1,083
20	1,100
24	1,121
28	1,142
32	1,163
36	1,183



Experiment 5.2 Preparation of acid solution with a preselected (prescribed) mass fraction from acid solution with a higher concentration

1. Receive a task from a teacher. Using tables 5.1.2-5.1.8, from density of stock solution (ρ_1 – density of a more concentrated solution) determine the mass fraction of acid (ω_1) into the stock solution.
2. Using the same table for the mass fraction of the solute (ω_2) in solution, which you need to prepare, determine the density of this solution (ρ_2). Record all data in table (as shown in table 5.1.9).
3. Calculate the volume of the initial solution (V_1) that should be taken to prepare a solution with a prescribed mass fraction, which is equal to ω_2, and with the volume V_2.
4. Calculate the molar concentration, molar concentration of equivalent, molality and the titer of the solution that you have to prepare (i.e. of a preselected solution). Present the results of calculations in table (as shown in table 5.1.9). Check the calculations with the teacher.
5. Measure out the calculated volume (V_1) of a more concentrated acid solution in a small cylinder or in measuring test tube. Pour the measured amount of the acid solution into a volumetric flask of suitable volume.

6. Rinse the graduated cylinder with distilled water 1-2 times, pouring water into a volumetric flask. Then, pour distilled water into the flask to the level of 0.5 -1.0 cm below the mark, and, after that, using a pipette or a wash, add water dropwise in order to bring the solution volume up to the mark. Then, close the flask with stopper and mix the solution by turning the flask several times. Put the prepared solution in storage.

Table 5.1.9

The experimental and calculated data

Substance	Initial solution			Prtescribed (a given) solution			Composition of resulting solution			
	Density of solution	Mass fraction of solution	Volume of solution	Density of solution	Mass fraction of solution	Volume of solution	Molar concentration	Molar concentration of equivalent	Molality	Titer
	ρ_1	ω_1	V_1	ρ_2	ω_2	V_2	C_X	$C_X \times (l/z)$	B_X	T
	g/ml	%	ml	g/ml	%	ml	mol/L		mol/Kg	g/ml

Experiment 5.3 Preparation of acid solution with a prescribed molar concentration or molar concentration of equivalent

1. Receive a task from a teacher. Using tables 5.1.2-5.1.8, from density of the starting solution (ρ_1) determine the mass fraction of acid (ω_1) in the original more concentrated acid solution.

2. Calculate the volume of stock solution V_1 that you need to take in order to prepare solution with a given molar concentration C_2 (X) [or molar concentration of equivalent C_2 (l/z*X)] and with the volume V_2. Calculate the molar concentration of equivalent (or molar concentration), molality and the titer of acid solution, which you need to prepare. Present all data in table

5.1.10. Check the correctness of the calculations and show the results of the calculations to the teacher.

Table 5.1.10

The experimental and calculated data

Substance	Stock solution			Obtained solution			
	Density of solution	Mass fraction of solution	Volume of solution	Volume of solution	Molar concentration	Molar concentration of equivalent	Titer
	ρ	ω_1	V_1	V_2	C_2	$C_2(l/z)$	T
	g/ml	%	ml	L	mol/L	mol/L	g/ml

3. Measure out the calculated volume $\mathbf{V_1}$ of a more concentrated acid solution in a small cylinder or in measuring test tube. Pour the measured amount of the acid solution into a volumetric flask of 50 or 100 ml (the volume of the volumetric flask should correspond to the volume $\mathbf{V_2}$). Rinse the graduated cylinder with distilled water 1-2 times, pouring water into a volumetric flask. Then, pour distilled water into the flask up to the mark (the last 0.5 -1.0 ml below the mark add water dropwise, using a pipette or a wash). Close the flask with stopper and mix the solution by turning the flask several times.

Experiment 5.4 Determination of the concentration of solution by titrimetric method

Titration method is used to determine values of concentrations of solutions of various substances with rather good precision. In this experiment, it is necessary to determine the concentration of the acid prepared in experiments 5.1.2 or 5.1.3 or given by a laboratory assistant.

To perform the experiment, it is necessary to have: the solution of a sodium carbonate (or soda - Na_2CO_3) with a known molar concentration of equivalent; indicator - methyl orange; burette, attached to the stand; dropper; funnel; several conical flasks of volume 200 -300 ml each and a beaker to collect an excess of solution, discharging from the burette.

Burette can accurately measure any liquid volume within its capacity. Burette is graduated glass tube fitted with a tap or glass capillary, connected to the burette via rubber tube. The glass bead, stopping the drain of the liquid from the burette, is inserted inside the rubber tube. To drain liquid from the burette, rubber tube should be pulled from the bead by pressing the bead (fig.5.4.1). The burette is filled with liquid through the funnel.

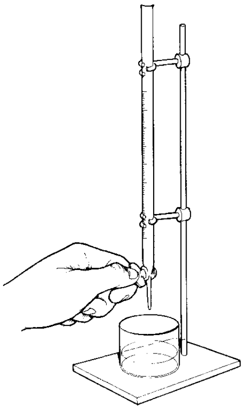


Figure 5.4.1

Pipette (fig. 5.4.2) is used to measure and to transfer a certain amount of liquid. In order to fill the pipette, the lower end of the pipette should be lowered (i.e. should be immersed) into the liquid to the bottom of the vessel, and, using a blower, fill the pipette with liquid, making sure that the tip of the pipette is always in the liquid.

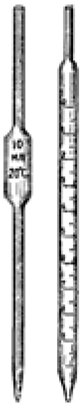


Figure 5.4.2

The pipette should be filled with liquid to the level, which is 2-3 cm above the mark, then, quickly close the upper opening with the index finger, at the same time holding the pipette with thumb and middle finger. Then, loosen the push of the index finger – the liquid begins to slowly flow out of the pipette. As soon as the lower meniscus of the liquid drops to the mark, firmly close the upper opening with the index finger again. Then, take a conical flask for titration in order to collect the liquid from the pipette. Insert the pipette into the conical flask, put the pipette on the neck of the conical flask, which is held at an angle, take up the index finger from the pipette opening, and allow the liquid to drain down on the inner wall of the flask.

Procedure:

1. Rinse clean burette with acid solution, and, then, using the funnel, fill it with the same acid to the level of 4-5 cm above zero division. Drain the excess of acid from the burette so that the lower end of the burette become free of air and the lower meniscus of the fluid become positioned at zero division of burette. After that, you can start titration.

2. Using 15 - 25 ml pipette, measure three volumes of soda solution and introduce them into three conical flasks (previously rinsed with a solution of the salt). Add 2-3 drops of Methyl Orange to each flask. Place one of the flasks with soda under the burette onto a sheet of white paper so that the tip of the burette is inside the flask. By one hand slowly pour acid from a burette into a flask and, by the other hand, constantly stir the solution via a circular motion of the flask in a horizontal plane. The place in the soda solution, where the acid enters, takes on pink color. On stirring of the solution, the pink color disappears and the yellow color of the solution is restored. When this color transition will be pretty slow, start to add acid solution dropwise, carefully mixing the solution. The titration is considered as complete, when the soda solution takes on non-vanishing pink color from a single drop of acid.

3. Record the volume of acid solution, spent for titration, with an accuracy of 0.1 ml. Count of burette marks should be performed using the bottom of the meniscus: the eye must be at the same level as the meniscus. The first titration is approximate.

4. Repeat the titration 2-3 times. Pouring acid to the burette, bring the level of acid to the level of zero mark of the burette, or, if the amount of acid, remaining in the burette, is considerable, bring the level of acid to the level of the nearest mark, which is convenient in order to determine the volume of acid, and, continue titration. Volumes of acid, consumed at titration, should not differ by more than 0.2 ml.

5. From the obtained values of the amount of acid, spent for the titration of soda, find the mean value and calculate the molar concentration of equivalent of acid and titer of acid solution:

$$C(1/z^{*}\ \text{of acid}) = \frac{V(\text{solution } Na_2CO_3) \cdot C(1/z^{*}\ Na_2CO_3)}{V(\text{of acid})}$$

$$T(\text{of acid}) = \frac{C(1/z^{*}\ \text{of acid}) \cdot M(1/z^{*}\ \text{of acid})}{1000}$$

The obtained data record in Table 5.1.11

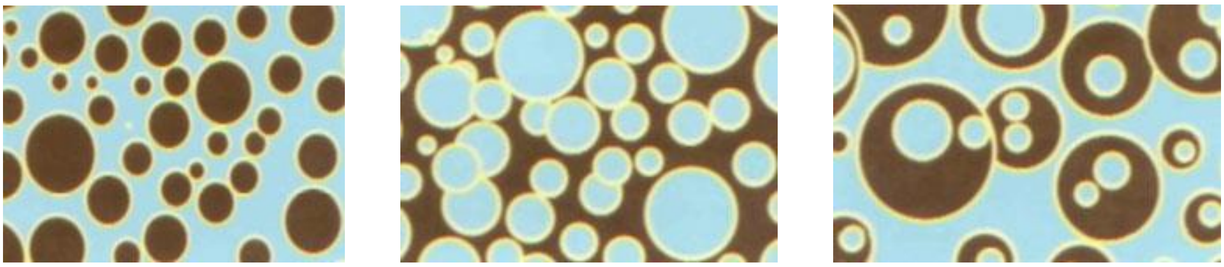
Table 5.1.11

The experimental and calculated data

Number of experiment	Volume of solution		Molar concentration of equivalent		Titre of solution of acid
	Soda	Acid	Soda	Acid	
	L	L	mol/L	mol/L	g/ml
1					
2					
3					

6. PREPARATION AND PROPERTIES OF EMULSIONS

Emulsions (fig. 6.1) - heterogeneous systems consisting of two mutually insoluble liquid phases, one of which is dispersed as droplets of size 0.1-100 microns in another continuous phase, which is a dispersion medium. Two types of emulsions are distinguished by chemists: emulsions of non-polar liquid in polar one - direct emilsions of type oil/water (O/W), and emulsions of polar liquid in non-polar one - inverse emulsions of type water/oil (W/O).



Emulsion "oil in water" ***Emulsion "water in oil"*** ***Multiphase emulsion***

Figure 6.1

The properties of the emulsions and conditions to stabilize them depend on concentration of the dispersed phase in the system. Chemists differentiate the emulsions according to the concentration of phase content: there are diluted emulsions (phase content is less than 0.1 vol. %), concentrated emulsions (phase content <74 vol. %) and highly concentrated emulsions (> 74 vol. %).

It is impossible to obtain concentrated emulsions without introduction of emulsifying additives (called **emulsifiers**) to the system. The mechanism of stabilizing action of emulsifiers depends on their chemical nature (fig. 6.2): the mentioned mechanism is different for different types of emulsions. How do emulsifiers work? Their hydrophilic "heads" are directed to the aqueous phase and the hydrophobic "tails" are directed to oil. Thus, emulsifiers are at the interface (i.e. at the boundary between phases in a heterogeneous system).

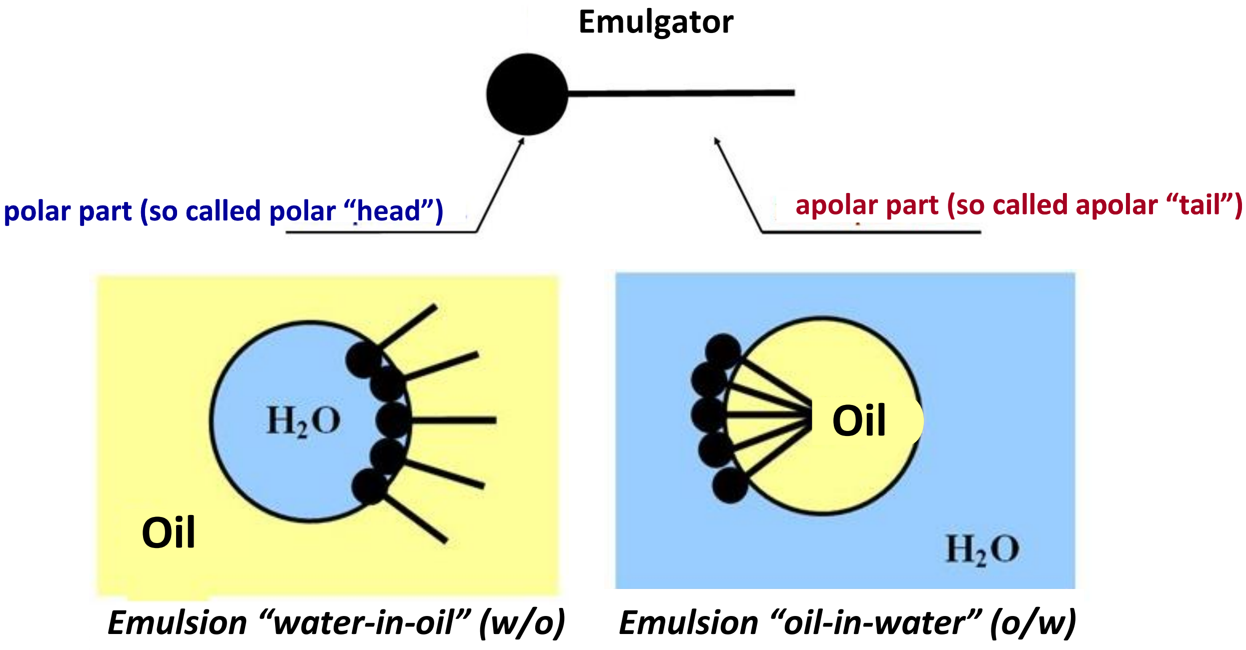


Figure 6.2

Emulsion stabilizers can be:

1. Low molecular weight surfactants. The stabilizing effect of the surfactants is due to their adsorption and to specific orientation of their amphiphilic molecules. There is rule of Bancroft: to stabilize the direct emulsions the surfactant must be readily soluble in water (soaps, containing alkali metal ions; alkylsulfates; alkylsulfonates); to stabilize the reverse emulsions the surfactant must be readily soluble in oil (soaps, containing alkaline earth metal ions). Depending on the type of surfactant (cationic, anionic, ampholytic, nonionic) emulsion particles can be uncharged or can have positive or negative charges.

2. High molecular weight surfactants. Synthetic and natural compounds with high molecular weight - polysoaps (polymers of vinyl series), proteins (gelatin, serum albumin, casein) and saponins belong to this class of stabilizers. Gel interfacial layer, having high structural viscosity, is formed as the result of adsorption of high molecular weight surfactant at the aqueous solution/oil interface.

3. Solid emulsifiers must meet the following requirements:

1) The size of the particles of powder should be very small in comparison with the drop size.

2) The particles must be able to be selectively wetted by one of the two liquids and, therefore, to concentrate at the interface. Hydrophilic particles (clay, gypsum, silica, calcites) stabilize direct emulsions, while hydrophobic particles (soot or carbon powder, plastic powders) stabilize inverse emulsions.

3) The concentration of particles at the interface must be sufficient for the formation of the phase film, which consists of densely packed particles.

The emulsion of one type can be converted into an emulsion of another type. This phenomenon is called inversion of emulsion phase. In order to cause such inversion, it is necessary to change the nature of the emulsifier. For example, benzene-in-water emulsion, stabilized with water-soluble sodium oleate, can be readily converted to water-in-benzene emulsion. To do this, add a solution of any salt of a di- or trivalent cation, such as $CaCl_2$. Calcium chloride reacts with sodium oleate and forms water-insoluble calcium oleate, which dissolves in benzene and stabilizes water-in-benzene emulsion.

In practice, the type of emulsion is determined by the following methods. In the method of dilution, a drop of the emulsion is introduced into the test tube with water. If the drop is evenly (uniformly) distributed in the water, the emulsion is of o/w type. The drop of w/o emulsion can not disperse in water. According to the method of dyeing of continuous phase, a few crystals of a water-soluble dye, such as Methyl Orange or Methylene Blue, dyed o/w emulsion uniformly over the entire volume. W/O emulsion is colored with liposoluble dye uniformly over the entire volume. Emulsion type can also be determined from the ability of emulsion to conduct electricity (conductivity method). High conductivities indicate that the dispersion medium is a polar liquid and an emulsion is of the o/w type. Low values of conductivity indicate the formation of an inverse emulsion (w/o).

OBJECTIVE: To learn how to obtain emulsions of different types and how to determine emulsion type.

Experiment 6.1 Preparation of emulsions

Pour 10 ml of hexane, colored with dye Sudan III (this dye is soluble in non-polar liquids and is insoluble in water), into two flat-bottomed flask. Add dropwise 10 ml of water into one of the flasks and, with continuous stirring, add 10 ml of 2% sodium oleate solution into the other flask. Tightly plug the flasks with stoppers and, then, vigorously shake them to obtain an emulsion (use ultrasonic dispergator). Pour the emulsions into test tubes and compare the stability of the emulsion from the 1st flask with the stability of the emulsion from the 2nd flask.

Опыт 6.2 Inversion of phases of emulsion

Transfer a half of the emulsion, stabilized with sodium oleate, to a clean flask (flask 3) and add dropwise with shaking 30 ml of 0.5% $MgSO_4$ solution to form a stable emulsion. Determine the type of emulsions, stabilized with sodium and magnesium oleate (flasks 2 and 3). Transfer 10 ml of emulsion in a test tube and note the time of its separation into two phases. The remainder of the emulsion is used to determine its type (Experiment 6.3). Record the results in the table (indicate type of emulsion before and after the addition of the electrolyte solution). Analyze the obtained data**.**

Experiment 6.3 Determination of emulsion type.

a) Place a drop of emulsion and a drop of water on a glass slide, tilt the slide glass so that the drops come into contact. If the drops merge, the dispersed phase is water, if the drops do not merge - the dispersed phase is oil.

b) Using pipette, put 1 drop of emulsion on pre-degreased surface of the slide glass and, put another drop of emulsion on the plate, covered with a smooth layer of paraffin. Drop spreads on glass in case if the emulsion is of the 1st type. Emulsion drop retains a spherical shape on the surface of the wax.

c) Apply emulsion drop on a filter paper (i.e. emulsion drop is coated on to filter paper). If the medium is water, then drop immediately soaks into paper and a greasy spot forms on the paper. A drop of the w/o emulsion is not absorbed with paper.

c) The method of dilution of phases is based on the fact that emulsion can be easily diluted with liquid, serving as dispersion medium of the emulsion. Place 2-3 ml of emulsion, stabilized with sodium oleate, into each of the two weighing bottles. Add 2-3 ml of water into one weighing bottle with the emulsion, and add 2-3 ml of hexane into the other weighing bottle with the emulsion. Observe in which weighing bottle the added liquid mixes with of the emulsion. A similar experiment is carried out with the emulsion, stabilized with magnesium oleate.
g) Pour the emulsion into two test tubes. Add a few drops of Methylene Blue (the dye that is soluble in water) into one test tube and add a few grains of Sudan I (the dye which is soluble in oil) into the other test tube. O/W emulsion is colored in blue (Sudan I is not dissolved in it). W/O emulsion is colored in red (Methylene Blue is not dissolved in it).

Present the results of the experiment in table.

Experiment 6.4 Influence of “dispersion medium/dispersed phase” volume ratio on emulsion type

Pour water and oil (xylene) in the volume ratios 1: 4; 2: 3; 3: 2; 4: 1; 4.5: 0.5 (the total volume of mixture is 5 ml) into the 5 test tubes, respectively. Add a few drops of the dye Sudan III and 1 ml of the emulsifier solution (2% sodium soap) to each test tube and shake it. Determine the type of emulsion.

1 : 4 = w/o; 2 : 3 = w/o; 3 : 2 = o/w; 4 : 1 = o/w; 4.5 : 0.5 = o/w.

Conclusion: if the dispersion medium is a polar liquid (in our case – water), the type of emulsion is oil-in-water (o/w). If the dispersion medium is a non-polar liquid (in our case – oil), the type of emulsion is a water in oil (w/o). In cases 1: 4; 2: 3 – the dispersion medium is oil (because the amount of oil is larger) and disperse phase – water (as the amount of water is smaller). In cases of 3: 2; 4: 1; 4.5: 0.5 – the dispersion medium is water, because it is taken in larger amount, and the dispersed phase – oil, as it is taken in smaller amount.

Experiment 6.5 Effect of viscosity of dispersion medium on the stability of the emulsion

Add 3 ml of water, 3 ml of a 10% glycerin solution and 3 ml of 20% glycerin solution to the three test tubes, respectively. To each test tube add 1 ml of xylene, 0.5 ml of sodium soap and a few drops of Sudan III.

Shake the contents of the tubes and note the time of division into layers (see Table 6.1).

Table 6.1

C of oil	Separation time, s
0.9	2.8
0.95	4.8
1.0	5.8
1.05	8.5
1.10	10.8

Conclusion: The increase in the viscosity hinders the movement of molecules, thus, the intensity of Brownian motion becomes reduced and, hence, the probability of collisions of molecules becomes smaller. Consequently, the stability of the emulsion increases.

Experiment 6.6 Preparation of concentrated emulsions of toluene in water (emulsifier - gelatin)

Prepare a 0.5% solution of gelatin in water. With stirring, add xylene (or oil) portionwise to 10 ml of this solution, heated to 40 °C. In this manner it is possible to introduce up to 10 ml of xylene. Allow the prepared emulsion to stand for about 1 hour. After such period of time it becomes jelly-like (one can not pour the obtained emulsion out from the test tube). The type of the obtained emulsion is "o / w".

Experiment 6.7 Destruction of emulsion

Separate the emulsion, remaining in the flask, into two parts. Add one part of the emulsion dropwise with agitation to a solution of 1N HCl, and the other part – to an industrial demulsifier. What is observed?

In the report it is necessary to explain the cause of the phase inversion and of the destruction of the emulsion. Also, draw the schemes of the emulsions, obtained in both cases, indicating the orientation of emulsifier molecules at the interface between the dispersed phase and the dispersion medium.

7. THE MAIN CLASSES OF INORGANIC COMPOUNDS

7.1 Chemical properties of acids.

Experiment 7.1.1 Introduction to properties of acids.

Specify the color and physical state for concentrated solutions of hydrochloric, nitric and sulfuric acids as well as for boric acid, which are in closed (plugged by stoppers) flasks, located in a fume hood.

Pour diluted solutions of these acids, available on laboratory tables, into the test tubes (pre-dissolve boric acid in distilled water) and test the effect of the solutions on methyl orange. Is the color of the indicator changed? What is the cause of the change of color?

Test the effect of boric acid on indicator solution (indicator - Methyl Red). How does the color of the indicator change? Explain the difference in the action of a solution of boric acid on the indicators with the help of the concepts of "acid strength" and "interval of change of indicator color."

7.1.2 Interactions of solutions of conventional acids with metals.

Test the effect of dilute solutions of hydrochloric and sulfuric acids on zinc and copper. After some time, note the test tubes in which the gas is released. Using the concept of "active metal", give an explanation for the observed differences in the action of zinc and copper in the dilute solutions of selected acids. Note the position of zinc and of copper relatively to the position of hydrogen in a rank of standard electrode potentials. Write the equations of the reactions. To what type can these reactions be attributed?

Experiment 7.1.3 Interaction of solutions of acids with basic oxides.

Test the action of a dilute solution of hydrochloric acid on calcium oxide powder. What is observed? Write the equation of the reaction.

Note: If calcium oxide, stored in air for a long time, is used, a side reaction, occurring with release of gas, can be observed. What is the cause of the occurrence of the side reaction?

Experiment 7.1.4 Interaction of acids with bases (reaction of neutralization).

Using tip of spatula, put a small amount of calcium hydroxide powder in test tube and add 1-2 ml of diluted solution of hydrochloric acid. What is observed? Write the equation of the reaction.

Note: If calcium hydroxide, stored in air for a long time, is used, a side reaction, occurring with gas evolution (gas release), is observed. What is the cause of the occurrence of the side reaction?

7.2 Chemical properties of bases.

Experiment 7.2.1 Introduction to properties of bases.

Specify the physical state and color for bases (hydroxides of sodium, potassium and calcium), which are in beakers in a fume hood. Using forceps, place sodium hydroxide pellet in a test tube, and, using tip of spatula, put a small amount of calcium hydroxide powder in another test tube. Add 1-2 ml of water to each test tube and stir with a glass rod. Add 1 drop of phenolphthalein to each test tube. What is the cause of the change in color of the indicator?

Experiment 7.2.2 Interaction of sodium metal with water.

Pour the distilled water into the glass vessel with thick walls and add a few drops of phenolphthalein solution. Using a scalpel, cut a piece of sodium on a sheet of filter paper, dry up it with a filter paper and, then, put the piece of sodium into the glass vessel with thick walls (Protect eyes with goggles!).

Experiment 7.2.3 Preparation and thermal decomposition of bases.

Using the solutions of copper salt (II) and sodium hydroxide, obtain copper hydroxide (II). Note the color of the precipitate. Gently heat the contents of the test tube in the flame of a gas burner. To prevent the ejection (emission) from the test tube, heat the upper part of the test tube. What is observed? Make a conclusion about the decomposition of copper hydroxide (II) when heated. Write the equations of the reactions.

Experiment 7.2.4 Preparation and properties of amphoteric hydroxides.

Pour a few drops of zinc sulfate solution in the test tube. Add a few drops of a dilute ammonium hydroxide (aqueous ammonia solution). **Caution! A deep inhalation of ammonia can lead to loss of consciousness.** What is formed in the test tube?

Divide the contents of the test tube into two parts. Using the first part, find out what happens when zinc hydroxide is in a solution of hydrochloric acid. Using the second part, find out what happens when zinc hydroxide is in sodium hydroxide solution (the solution is in excess). What is observed? Write down the equations of the corresponding reactions, taking into account that, when zinc hydroxide is dissolved in the solution of sodium hydroxide, sodium tetrahydroxy zincate $Na_2[Zn(OH)_4]$ is formed.

Experiment 7.2.5 Neutralization reaction

Pour a few milliliters of alkaline solution into a test tube, add 2-3 drops of a solution of phenolphthalein. Add dropwise a solution of acid (stirring with a glass rod) until the disappearance of color of the indicator. Write the equation of the neutralization reaction.

7.3 Preparation and properties of oxides.

Experiment 7.3.1 Preparation of basic oxide.

Put a small amount of magnesium shavings in the metal spoon and heat it in the flame of a gas burner to ignite the magnesium**. Caution! Magnesium burns very brightly.** Write the equation of the reaction. Note the color of the obtained oxide. **Let the obtained oxide to cool down** and save it for the next experiment.

Experiment 7.3.2 Interaction of basic oxide with water.

Insert the oxide, obtained in previous experiment (7.3.2), into the tube and add 1.2 ml of water and 2-3 drops of phenolphthalein. Does the color change? Write the reaction equation for interaction of magnesium oxide with water.

Experiment 7.3.3 Preparation of acidic oxide.

Put a piece of chalk in a test tube and add a few drops of solution of hydrochloric acid. What is observed? Obtain carbon dioxide in Kipp's apparatus, in which a similar reaction of hydrochloric acid with marble occurs. Write the equation of the reaction. Make a conclusion about the stability of carbonic acid.

Experiment 7.3.4. Preparation and properties of copper oxide (I)

Pour 5% copper sulfate (II) solution into 250 ml beaker to the level of about half of the beaker volume. Put the beaker on a magnetic stirrer, turn on electric heating and, with continuous stirring, add about 10 ml of a concentrated solution of hydrazinium chloride. Then, without stopping the stirring, add about 50 ml of 30% potassium hydroxide solution to the beaker. Orange-red copper oxide (I) **Cu_2O** precipitates of and gas N_2 is released:

$$\mathbf{4CuSO_4 + N_2H_5Cl + 9KOH = 2Cu_2O\downarrow + N_2\uparrow + 4K_2SO_4 + KCl + 7H_2O}$$

Then, stop the stirring, allow the suspension to settle for several minutes, then, decant the solution away from the precipitate. Several times wash the precipitate with water by decantation.

Transfer a portion of the obtained copper oxide (I) to a second beaker, and, with stirring, add dropwise 10% sulfuric acid into the beaker. Observe how the reaction mixture becomes blue (blue color is characteristic of hydrated ions of copper (II), which are the result of reaction of disproportionation) and orange-red precipitate becomes pinkish (metallic copper). Reaction occurs:

$$\mathbf{Cu_2O + H_2SO_4 = Cu\downarrow + CuSO_4 + H_2O}$$

Transfer a second portion of the copper oxide (I) to the third beaker and treat it with concentrated hydrochloric acid, adding the acid dropwise. Observe the appearance of a white precipitate of copper chloride (I) CuCl, and, then, observe the dissolving of the precipitate due to complexation:

$$\mathbf{Cu_2O + 2HCl = 2CuCl\downarrow + H_2O}$$

$$CuCl + HCl = H[CuCl_2]$$

Add concentrated (25%) ammonia solution to the copper oxide (I), remaining in the first beaker. Observe the dissolving of the precipitate as a result of formation of complex compounds:

$$Cu_2O + 4NH_3 \cdot H_2O = 2[Cu(NH_3)_2](OH) + 3H_2O$$

Experiment 7.3.5 Burning of Aluminum

Set fire to dry fuel on the asbestos sheet. Using spatula, take a small amount of aluminum powder from the weighing bottle, pour the aluminium powder in small portions on top of the burner flame. The metal bursts into flame, scattering sparks and releasing white smoke (aerosol of aluminum oxide). During the combustion of aluminum, a large amount of heat is released.

7.4 Preparation and chemical properties of salts.

Experiment 7.4.1 Interaction of salts with metals.

Pour solution of copper (II) sulfate into a test tube. Put a piece of zinc in the test tube. What is observed? Write the equation of the reaction. Note the position of copper and zinc in a rank of standard electrode potentials. Does copper interact with a solution of salt of zinc? Make a general conclusion about the interaction of solutions of salts with metals.

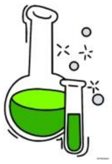
Experiment 7.4.2 Preparation of salts by exchange reaction.

Pour a few drops of solution of sodium chloride in a test tube and add 2-3 drops of solution of silver nitrate. What is the indication of this reaction? Write the equation of the reaction.

Experiment 7.4.3 Interaction of aluminum with iodine

Grind crystalline iodine in a porcelain mortar. The obtained powder of iodine mix with the dry aluminum dust in the volume ratio of 1: 1. Transfer the resulting mixture to a porcelain cup (or on iron or asbestos plate). Form the mixture into a small mound with a hole on top. Using a pipette, add 5-6 drops of distilled water in the hole. Water activates the oxidation-reduction reaction. The reaction is accompanied by considerable heat release. During the reaction, the mixture bursts into flame and purple iodine vapor is liberated.

Note. Experiment should be carried out in a hood or one should use a glass bell with big volume. If, after addition of water, the reaction does not begin within 3-5 minutes, this means that, due to the prolonged storage in air, the particles of aluminium are covered with film, which is comprised of aluminum oxide. In this case, it is possible to start the reaction of aluminum with iodine by adding of 2-3 drops of hydrochloric acid to the mixture.
Write the equation of the reaction:



Experiment 7.4.4 Interaction of iron salts (III) with thiocyanate ions

Pour 20 ml of a 5% solution of ferric chloride (III) into the beaker. Add 5% ammonium thiocyanate solution to the beaker. Observe the appearance of the blood-red color of the solution due to the formation of thiocyanate complexes of iron (III):

$$[\mathrm{Fe(H_2O)_6}]^{3+} + n\ \mathrm{NCS}^- \rightleftarrows [\mathrm{Fe(H_2O)}_{6-n}(\mathrm{NCS})_n]^{(n-3)-} + n\ \mathrm{H_2O}$$

Then, to the resulting solution add potassium fluoride solution. Observe the disappearance of the red color due to the formation of more stable colorless tetraftoroferrat (III) ions:

$$[\mathrm{Fe(H_2O)}_{6-n}(\mathrm{NCS})_n]^{(n-3)-} + 4\mathrm{F}^- \rightleftarrows [\mathrm{FeF_4}]^- + n\ \mathrm{NCS}^- + (6-n)\ \mathrm{H_2O}$$

8. ELECTROLYTIC DISSOCIATION

Electrolytes – substances conducting electric current in the molten state or in dissolved state. Electrolytes dissociate into ions in an environment of high dielectric constant (alcohols, water, etc.). The process of disintegration of the molecules into ions is called **electrolytic dissociation** (fig. 8.1).

Dissociation of electrolytes into ions is followed by solvation, i.e. by the interaction of ions with polar solvent molecules. If the solvent is water, the term solvation is replaced with the term hydration.

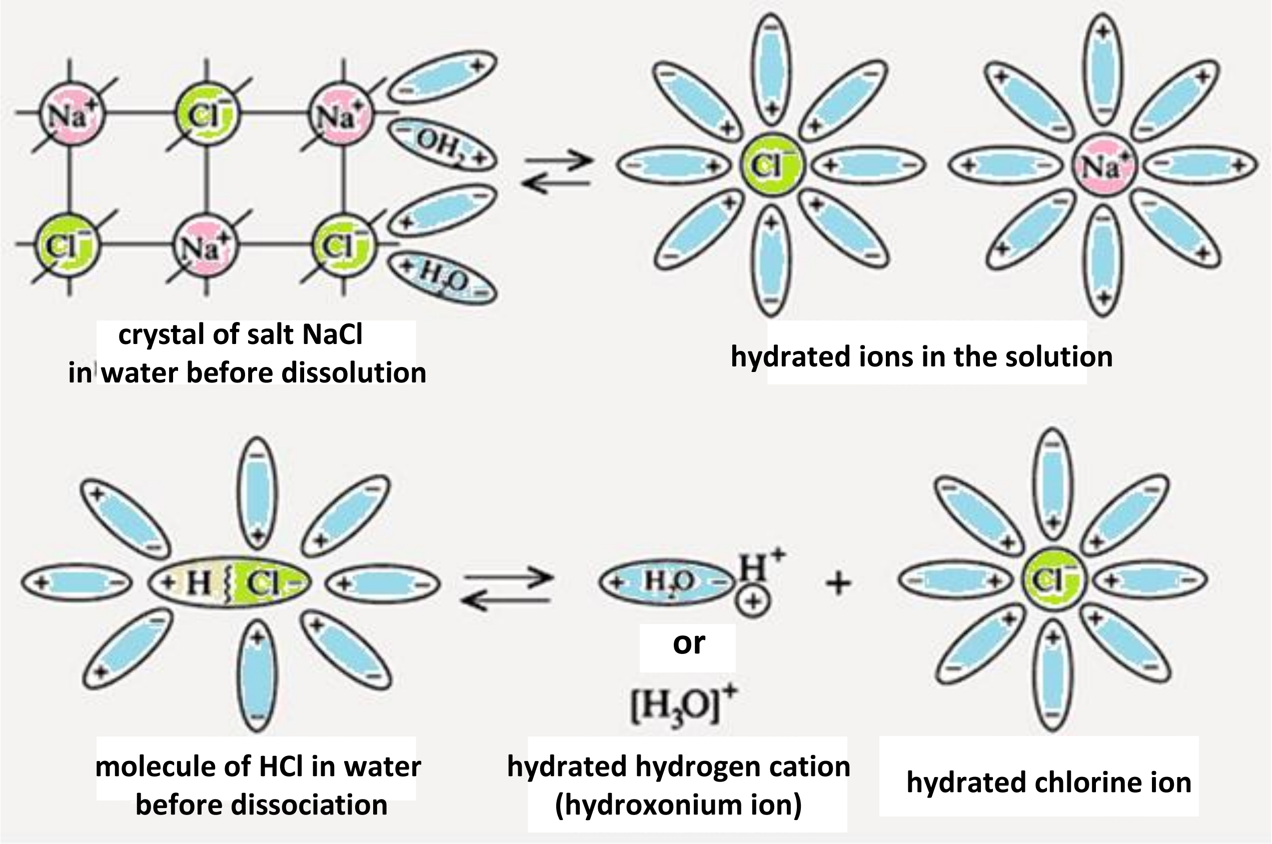


Figure 8.1. Examples of electrolytic dissociation

Electrolytic dissociation - a reversible process, and there is a balance between ions and molecules in electrolyte solutions.

$$KA \rightleftarrows K^+ + A^-$$

The degree of dissociation (α) shows the ratio of the number of molecules broken into ions (N') to the total number of dissolved molecules (N).

$$\alpha = \frac{N'}{N}$$

The degree of dissociation of the electrolyte is determined experimentally and is expressed in fractions of a unit or on a percentage basis. If $\alpha = 0$, there is no dissociation, and if $\alpha = 1$, or 100%, the electrolyte completely disintegrates into ions. If $\alpha = 20\%$, it means that out of 100 molecules of the electrolyte 20 dissolved into ions.

The degree of dissociation of electrolyte depends on the electrolyte nature, on concentration, on solvent nature, on the presence of similar ions in solution and on temperature. For the same electrolyte at a given temperature α increases with dilution of solution; at high dilutions electrolyte is completely dissociated ($\alpha \to 1$). On increase of temperature, α also increases.

According to the degree of dissociation, the electrolytes are divided into strong, medium and weak. Electrolytes, which in 0.1 M solution are characterized with $\alpha > 30\%$, are attributed to strong electrolytes. Strong electrolytes are 1) almost all the salts (except for $HgCl_2$, $SdCl_2$, $Fe(SCN)_3$, $Pb(CH3COO)_2$, and others); 2) many mineral acids such as HNO_3, HCl, H_2SO_4, HI, HBr, $HClO_4$ etc..;

3) bases of alkali and alkaline earth metals, such as KOH, NaOH, $Ba(OH)_2$ and others.

A medium electrolytes are characterized with α = 3-30% for 0.1 M solution; These include, for example, H_3PO_4, H_2SO_3, HF, $Mg(OH)_2$.

For weak electrolytes in 0.1 M solutions $\alpha < 3\%$; weak electrolytes are H_2S, H_2CO_3, HNO_2, HCN, H_2SiO_3, H_3BO_3, HClO et al., as well as the majority of bases of multivalent metals, NH_4OH and water.

The relative power of electrolyte can be measured by the electrical conductivity of its solutions.

Dissociation of bases. According to the theory of electrolytic dissociation, the bases - are electrolytes, which form only one kind of anions during the dissociation - hydroxide ions OH^-:

$$NaOH \rightarrow Na^+ + OH^- ;$$
$$Ca(OH)_2 \rightarrow CaOH^+ + OH^- ;$$
$$CaOH^+ \rightarrow Ca^{2+} + OH^- .$$

Stepwise dissociation makes possible the formation of basic and acidic salts (see. below).

Dissociation of acids. Acids – electrolytes, which form only one type of cations upon dissociation - hydrogen cations H^+.

$$HCl \rightarrow H^+ + Cl^- ;$$
$$HNO_3 \rightarrow H^+ + NO_3^- ;$$
$$H_2SO_4 \rightarrow H^+ + HSO_4^{--} ;$$
$$HSO_4^- \rightarrow H^+ + SO_4^{2-} ;$$
$$H_3PO_4 \rightarrow H^+ + H_2PO_4^- ;$$
$$H_2PO_4^- \rightarrow H^+ + HPO_4^{2-} ;$$
$$HPO_4^{2-} \rightarrow H^+ + PO_4^{3-}.$$

Dissociation of amphoteric hydroxides. In aqueous solution amphoteric hydroxides dissociate both as acids and as bases. H^+ cations and hydroxide anions OH^- are formed simultaneously during the dissociation of amphoteric hydroxides:

$$H^+ + MeO^- \;\; MeOH \rightleftarrows Me^+ + OH^-.$$

These include hydroxides of zinc $Zn(OH)_2$, aluminium $Al(OH)_3$, chromium $Cr(OH)_3$, lead $Pb(OH)_2$ and others.

For example, the dissociation of $Zn(OH)_2$:

$$2\ H^+ + ZnO_2^{2-} \rightleftarrows H_2ZnO_2 \rightleftarrows Zn(OH)_2 \rightleftarrows Zn^{2+} + 2\ OH^-.$$

Dissociation of salts

1. Normal salts are electrolytes, which dissociate in aqueous solutions, with formation of metal cations and anions of acid residue. For example,

$$Na_2SO_4 \rightarrow 2\ Na^+ + SO_4^{2-}\ ;$$
$$Ca_3(PO)_4 \rightarrow 3\ Ca^{2+} + 2\ PO_4^{3-}\ .$$

1. On dissolving in water, acid salts form a metal cation and a complex anion, consisting from hydrogen atoms and acid residue:

$$KHSO_3 \rightarrow K^+ + HSO_3^- \ \ (\alpha = 1).$$

Complex anion dissociates partially:

$$HSO_3^- \rightleftarrows H^+ + SO_3^{2-} \ \ (\alpha << 1).$$

2. During dissociation basic salts form anions of acid residue and complex cations, consisting of metal atoms and hydroxy groups OH^-:

$$Al(OH)_2Cl \rightarrow Al(OH)_2^+ + Cl^- \ \ (\alpha = 1).$$

Complex cation dissociates partially:

$$Al(OH)_2^+ \rightleftarrows AlOH^{2+} + OH^- \ \ (\alpha << 1);$$
$$AlOH^{2+} \rightleftarrows Al^{3+} + OH^- \ \ (\alpha << 1).$$

3. In aqueous solution the double salt dissociate into two cations and anion of acid residue:

$$KAl(SO_4)_2 \rightarrow K^+ + Al^{3+} + 2\ SO_4^{2-}\ ;$$

$$Na_2NH_4PO_4 \rightarrow 2\ Na^+ + NH_4^+ + PO_4^{3-}\ .$$

4. Complex salts dissociate into metal cations and anions of acidic residues. For example,

$$ZnClNO_3 \rightarrow Zn^{2+} + Cl^- + NO_3^-;$$

$$AlSO_4Cl \rightarrow Al^{3+} + SO_4^{2-} + Cl^-\ .$$

The dissociation constant. To equilibrium, which is established between the molecules and ions in a weak solution of electrolyte, one can apply the laws of chemical equilibrium and write the equilibrium constant:

$$KA \leftrightarrows K^+ + A^-$$

$$K = \frac{[K^+]\,[A^-]}{[KA]} = \mathrm{const} = f\,(t)$$

The equilibrium constant, corresponding to the dissociation of a weak electrolyte, is called the dissociation constant.

The relation between dissociation constant and degree of dissociation

$$\alpha = \sqrt{\frac{K}{c}}$$

Thus, on increase of the electrolyte concentration, the degree of dissociation decreases, and, vice versa: with the decrease of the electrolyte concentration, the degree of dissociation increases.

Equilibrium in weak electrolytes obeys to the Ostwald dilution law. If the total concentration of electrolyte, for example, of a weak acid of type HA is C mol/l, whereas the concentration of H^+ and A^- ions will be determined by the expression $C\alpha$ (mol/l), $[H^+] = [A^-]$, and concentration of undissociated electrolyte is $(C\text{-}C\alpha)$, then:

$$K_D = \frac{\left[H^+\right]\left[A^-\right]}{\left[HA\right]} = \frac{Ca \cdot Ca}{C\left(1-a\right)} = \frac{Ca^2}{1-a}$$

If $\alpha \ll 1$ we obtain $K_D = C \cdot \alpha^2$. From where

$$a = \sqrt{\frac{K_D}{C}}$$

The shift of the ionic equilibrium can cause an increase or decrease in the degree of dissociation of a weak electrolyte. The shift of the ionic equilibrium can be controlled on the base of the principle of Le Chatelier. Introduction of ion of the same name to the solution, i.e., increase in the concentration of one of the reaction products, causes shift of the ionic equilibrium to the left, towards the formation of the molecules or to the reduction of the degree of dissociation of the electrolyte. In contrast, the binding of one of the ions into low-dissociating compound causes an increase in the degree of electrolytic dissociation.

Activity of ions. In solutions strong electrolytes dissociate into ions almost completely, so the concentration of ions is quite high. Forces, acting between the ions, operate even at low electrolyte concentration. The ions are not free in motion and the properties of the electrolyte, depending on the number of ions, manifest themselves less than if the ions did not interact with each other. In connection with this, the state of the ions is described with **activity** - apparent (effective) concentration of ions, whereby (i.e. by which) they act (operate) in chemical processes.

Ion activity a (mol/l) is related to molar concentration of ion in solution $C_м$ via equation:

$$a = f\, C_м,$$

where f - ion activity coefficient (dimensionless). Ion activity coefficients depend on the composition and concentration of the solution, on the nature and charge of the ion and on other conditions.

In dilute solutions at $C_м << 0.5$ mol/L ion nature has little (or marginal) effect on the values of f. There is an approximation that in dilute solutions f of the ion in the solvent depends on the charge of the ion and on the ionic strength of the solution I, which is equal to half of the sum of the products of concentration $C_м$ of each ion by the square of its charge Z:

$$I = 0{,}5\,(C_1Z_1^2 + C_2Z_2^2 + \ldots + C_nZ_n^2) = 0{,}5 \sum_{i=1,2,3,\ldots,n} C_i Z_i^{\,2}.$$

For example, for electrolyte solution of 2 M $CaCl_2 = Ca^{2+} + 2\ Cl^-$ ionic strength of the solution is

$$I = 0{,}5\,(2 \cdot 2^2 + 4 \cdot 1^2\,) = 6.$$

Values of activity coefficients of ions in dilute solutions are presented in Appendix 1. The values of activity coefficients are presented according to ion charge and ionic strength of the solution.

Laboratory work 8.1 Electrolytic dissociation

Objective

To study the electrical conductivity of electrolyte solutions and its dependence on various factors. To learn how to predict the direction of the exchange reaction in electrolyte solutions.

Equipment and Reagents

Instrument for testing of the electrical conductivity of solutions. Burette (50 ml). Wash. Conical tubes. Standard test tube rack with reagents. Sodium acetate (crystal.). Ammonium chloride. Indicators: Phenolphthalein, Methyl Orange. Solutions: acetic acid (0.1 M; 70%), ammonia (0.1 M; 25%), sodium hydroxide (0.1 M; 40%), nitric acid (0.1 M), hydrochloric acid (0.1M), potassium hydroxide (0.1 mol), sulfuric acid (0.02 mol), barium hydroxide (0.02 mol), aluminum sulphate (0.1M).

Experiment 8.1.1 The study of the electrical conductivity of solutions

After each testing of the electrical conductivity, turn off the appliance from the mains (i.e. from power) and wash electrodes with distilled water!

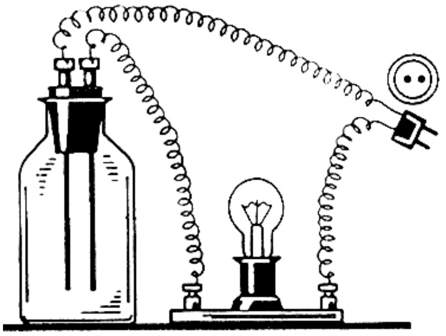


Figure 8.2. Instrument for study of the electrical conductivity of solutions

A. Pour distilled water into the device for study of the electrical conductivity of solutions (fig. 8.2). Lower carbon electrodes (pre-washed with distilled water) into the distilled water in the device. Plug the appliance into the mains (i.e. into the power network). Mark visually degree of light intensity of lamp.

B. Study the electrical conductivity (see experiment 8.1.1 A) of each of the six 0.1 M solutions: CH_3COOH, NH_4OH, HNO_3, HCl, NaOH, KOH.

Record observational data, noting the degree of the light intensity of the lamp. Taking into consideration the light intensity of the lamp, make a conclusion about each measured solution: whether the electrolyte is strong or weak.

C. Mix equal volumes of solutions: 1) a strong acid and a strong base; 2) a weak acid and a weak base. Test the electrical conductivity of the resulting solutions (see experiment 8.1.1 A). Record observational data and write the reactions in the molecular and ionic forms. Make a conclusion about the electrical conductivity of the resulting solutions.

Experiment 8.1.2. The dependence of the electrical conductivity of solution (of the degree of electrolytic dissociation) on the dilution

Pour the minimum amount of concentrated acetic acid solution, required for immersion of electrodes, into the device (see experiment 8.1.1 A). Plug the appliance into the electrical network and note the degree of the light intensity of the lamp. Then, dilute acid, slowly adding distilled water. How does the degree of the light intensity of the lamp change? How to explain the observed phenomenon?

Rinse the electrodes of the device with tap water, and, then, rinse them with distilled water. Repeat the experiment with concentrated ammonia solution. Make a general conclusion from the observational data.

Experiment 8.1.3 The change in the electrical conductivity of solution as a result of the shift of the ionic equilibrium

Study separately electrical conductivity of 0.02 M sulfuric acid solution and electrical conductivity of 0.02 M barium hydroxide solution (see experiment 8.1.1 A). Then, study the electrical conductivity of 0.02 M barium hydroxide solution in the presence of 0.02 M sulfuric acid solution. To do this, pour barium hydroxide solution into the device and add 2-3 drops of phenolphthalein solution. Using burette, add sulfuric acid solution dropwise through the funnel inserted into the instrument cover. Watch the change in color of the indicator and the degree of light intensity of lamp.

What is the electric conductivity of the solution at the time of disappearance of the color of indicator (i.e. at the equivalence point)? What happens when you add an excess of sulfuric acid to the solution? Explain the observed phenomena.

Experiment 8.1.4 The Influence of ions of the same name on the degree of dissociation of a weak electrolyte (the shift of the equilibrium of dissociation)

A. Pour dilute ammonia solution to test tube and add 2-3 drops of phenolphthalein. Divide the solution into two parts. One part of the solution should be left for comparison. To the other part of the solution add some solid ammonium chloride and stir well. Taking into consideration the principle of Le Chatelier and dissociation constants, explain the change in color of the solution.

Make a conclusion about the impact of the ion of the same name on the degree of ion dissociation of a weak electrolyte.

B. Pour 5...6 drops of 0.1 N acetic acid solution and 1...2 drops of methyl orange solution into each of the two test tubes. Then, to one of the test tubes add 2…3 crystals of sodium acetate and shake the test tube. Compare the intensity of color in the test tubes. Explain the reason for change of the color of the solution.

Experiment 8.1.5 The shift of the ionic equilibrium in saturated solution of amphoteric electrolyte

Pour ten drops of a solution of aluminum salts into test tube. Dropwise add dilute sodium hydroxide solution to the solution of aluminum salts until the appearance of the precipitate of aluminum hydroxide. Divide the precipitate into two parts (into two tubes). To one part of the precipitate add hydrochloric acid solution in excess, and, to the other part of the precipitate add concentrated alkali solution in excess. What is observed in both cases? Explain the observed phenomena in terms of the shift of the ionic equilibrium in a saturated solution of aluminum hydroxide. Write equations of ionic reactions, taking into account that the amphoteric hydroxide is poorly soluble in water.

Test questions and exercises

1. What substances are called electrolytes and non-electrolytes?

2. What are the main concepts of electrolytic dissociation?

3. Explain the process of dissociation of salts, acids and bases in terms of the structure of atoms and molecules.

4. The reactions of dissociation of electrolytes are reversible. Explain, what does this mean? How to write the equation of dissociation?

5. What electrolytes are called strong, and what electrolytes are weak? Give examples.

6. How does the ability of the electrolyte to dissociate depend on the type of chemical bond?

7. 1 liter of solution contains 0.25 g of sodium chloride. Calculate the concentration of Na^+ and Cl^- ions and the total concentration of all ions.

8. 1 liter of 0.01N formic acid HCOOH solution at room temperature contains 0.06 g of $HCOO^-$ ions. Find the dissociation constant.

9. At what concentration of acetic acid CH_3COOH in aqueous solution $\alpha = 1\%$? At what concentration of acetic acid α is 2 times more, if $K_{dis}. = 1.8.\ 10^{-5}$?

10. Taking into consideration the first stage of dissociation of hydrogen sulphide, determine α (%) for its 0.01 M solution (see. Appendix 4).

11. Write the following molecular equations of reactions in ionic form:

а) $Zn(OH)_2 + 2\ NaOH = Na_2ZnO_2 + 2\ H_2O$;
б) $Na_2ZnO_2 + 2\ HCl = 2\ NaCl + Zn(OH)_2$.

Appendix 1

Table A1 **Solubility of certain substances in water (in grams of anhydrous salt per 100 g of solution)**

Substance	**Temperature°C**								
	0	10	20	30	40	50	60	80	100
Al2(SO4)3	23,8	25,1	26,6	28,8	31,4	34,3	37,1	42,2	47,1
BaCl2	-	25,0	26,4	27,7	29,0	30,0	31,6	34,3	37,0
NaNO3	42,2	44.6	46,8	49,0	51,2	53,3	55,5	59,7	63,5
NaNO2	41,9	43,8	45,8	47,8	49,6	51,0	52,8	57,0	62,0
NaCl	26,21	26,3	26,4	26,5	26,81	27,0	27,14	27,65	28,38
Na2CO3	6,6	11,2	17,8	29,0	33,2	32,2	31,7	-	-
NaHCO3	6,45	7,58	8,76	-	-	12,67	14,09	-	-
Na2SO4	4,5	8,2	16,1	28,8	32,5	31,9			29,9
NH4Cl	23,0	25,1	27,1	29,3	31,5	33,5	39,6	39,6	43,6
NiSO4	21,4	-	-	29,8	-	33,4	35,4	38,7	43,4
KCl	22,2	23,8	25,8	27,2	28,7	30,1	31,3	33,8	36
KNO3	11,6	17,7	24,1	31,5	39,1	46,2	52,5	62,8	71,1
KMnO4	2,8	4,0	6,0	8,3	11,2	14,4	20		
K2Cr2O7	4,4	7,5	11,1	15,4	20,6	25,9	31,2	41,1	50,5
K2SO4	6,9	8,5	10,0	11,5	13,1	14,2	15,4	17,6	19,4
KAl(SO4)2	3,1	4,4	5,7	9,2	12,0	-	26,7		71,4
CuSO4	12,9	14,8	17,2	20,0	22,8	25,1	28,1	34,9	42,4
FeSO4	15,5	17,0	21,0	24,8	28,6	32,7	35,5	30,4	-
Pb(NO3)2	26,7	26,7	30,8	34,3	37,8	41,0	44,0	46,8	51,8

Dimensions of SI units and of non-system units

Meter – the length of the path traveled by light in a vacuum during 1/299792458 of second

Recommended multiples and sub-units formed from the meter: km, cm, mm, microns, nm. Along with SI units the following Common Units are approved for use:

Ångstrom Å – 10^{-10} m;
Astronomical unit (a.e.) 10,0 =$1{,}49597870 \cdot 10^{11}$ m;
inch (in); 1 inch=0,0254 m;
nautical mile (n.m.) 1 n.m. = 1852 m;
land mile (mi); 1 mi = 1609, 344 m;
parsec (pc); 1 pc = $3{,}085678 \cdot 10^{15}$ m;
light year (1 y); 1 light year = $9{,}\ 460530 \cdot 10^{15}$ m;
Fermi ; 1 Fermi = $1 \cdot 10^{-35}$;
foot (ft); 1 feet = 0,3048 m;
yard (yd); 1 yard = 0,844 m.

The second is 9192631770 periods of the radiation corresponding to the transition between the two hyperfine levels of the ground state of the cesium -133 atom (XIII General Conference of Weights and Measures, 1967).

Recommended **multiples** and **sub-units** of time: ks, ms, microsecond, nanosecond.

Common Units of time permitted for use along with the SI units: minute (min.), Hour (h), days (d).

Kelvin is equal to 1/273.16 of the thermodynamic temperature of the triple point of water (XIII General Conference of Weights and World, 1967).

The transition from the Celsius scale (° C) to Kelvin scale (T_K) is achieved by the following formula:

$$T_\kappa = t\ ^\circ C + 273{,}16.$$

Kilogram is the mass of the international prototype of kilogram of platinum iridium alloy kept in the International Bureau of Weights and Measures in Paris.

Non-system units of mass:

atomic mass unit (amu);
1 amu = $1{,}66057 \cdot 10^{-27}$ kg;

gram (g); 1 g = 1 · 10^{-3} kg;
grain (gr); 1 gr=6,479891 · 10^{-5} kg;
carat (kt); 1 carat = 2 ·10^{-4} kg;
ton (t); 1 t = 1000 kg;
apothecaries’ ounce (oz apoth.); 1 oz apoth. = 31,1035 · 10^{-3} кг;
trade ounce (oz); 1 oz = 28,3495 · 10^{-3} kg;
trade pound (lb); 1 lb = 0,45359237 kg;
pound in the Russian system of measures; 1 pound (Rus) = 0.40951241 kg;
pound (USA) [lb(US)]; 1 pound (US) = 0,4535924277 kg.

Candela – luminous intensity in a given direction from a source that emits monochromatic radiation of 540·10^{12} Hz, the energy intensity of which in that direction is 1,683 watts per steradian (XVII General Conference on Weights and Measures, 1979).

Mole – is equal to the amount of substance, containing the same number of structural elements as number of carbon atoms in the 0,012 kg of carbon-12.

When one use such unit as mole, structural elements must be specified – atoms, molecules, ions, electrons, and other particles or specified groups of particles can be considered as structural elements (the fourteenth General Conference on Weights and Measures, 1971). If the homogeneous system contains N particles, then the amount of substance:

$n(v) \frac{\mathrm{N}}{N_A}$, , where N_A – Avogadro constant, N_A = 6,0221367(36) · 10^{23} mol^{-1}.

Table A2.1 **Arbitrary SI units**

Prefix	Symbol	Factor	Prefix	Symbol	Factor
a) formed by multiplying by a factor of 10^3					
exa	E	10^{18}	milli	m	10^{-3}
peta	P	10^{15}	micro	μ	10^{-6}
tera	T	10^{12}	nano	n	10^{-9}
giga	G	10^{9}	pico	p	10^{-12}
mega	M	10^{6}	femto	f	10^{-15}
kilo	k	10^{3}	atto	a	10^{-18}
б) formed by multiplying by a factor of 10^1					
hecto	h	10^{2}	deci	d	10^{-1}
deca	da	10^{1}	centi	c	10^{-2}

1 part per million – million $^{-1}$, p pm; 1 million $^{-1}$= $1{\cdot}10^{-4}$ %.

Table A2.2 **The ratio of non-SI units of length to SI units**

Unit of length*	m	A°	ft	in
Meter*, 1 m (SI)	1	10^{10}	3,2787	3,94·10
Angstrom, 1 A°	10^{-10}	1	$3{,}2787{\cdot}10^{-10}$	$3{,}94{\cdot}10^{-9}$
Foot, 1 ft	0,305	$0{,}305{\cdot}10^{3}$	1	12
Inch, 1 in	$25{,}4{\cdot}10^{-3}$	$25{,}4{\cdot}10^{-7}$	$8{,}\ 333{\cdot}10^{-2}$	1

* derivatives: km, cm, mm, m, nm.

Table A2.3 The ratio of non-SI units of mass to SI units

Unit of weight *	kg	g	oz apoth.	lb
The kilogram, 1 kg (SI)	1	10^{3}	32,2	2,2046
gram, 1 g	10^{-3}	1	$3{,}22\cdot10^{-2}$	$2{,}2046\cdot10^{-3}$
apothecaries' ounce (1 oz apoth.), 1 ounce	$31{,}1035\cdot10^{-3}$	31,1035	1	$68{,}5715\cdot10^{-3}$
Pound (lb), pound 1	0,45359236	$4{,}5359\cdot10^{2}$	14,6	1

* derivatives: Mg, g, mg, μg; auxiliary unit - 1 ton (t) = 10^{3} kg

Table A2.4 The ratio of non-SI units of volume to SI units

Unit of volume*	m^{3}	L	bbl	pt liq	fl oz
A cubic meter,1 m^{3} (SI)	1	10^{3}	8,64842	$1{,}7597\cdot10^{3}$	$3{,}5195\cdot10^{4}$
1 Liter	10^{-3}	1	$8{,}64842\cdot10^{-3}$	1,7597	35,195
1 barrel. (bbl), dry barrel (Amer.)	0,115628	$1{,}15628\cdot10^{2}$	1	$2{,}03477\cdot10^{2}$	$4{,}0695\cdot10^{3}$
1 barrel of oil (US)	0,158988	$1{,}58988\cdot10^{2}$	1		
1 pint of liquid (pt liq; Eng.)	$5{,}68281\cdot10^{4}$	0,568281	$4{,}915\cdot10^{-3}$	1	20,00
1 fluid ounce (fl oz; Eng.)	$2{,}8488813\cdot10^{-5}$	$2{,}8488813\cdot10^{-2}$	$2{,}45727\cdot10^{-4}$	$5{,}00\cdot10^{-2}$	1

* derivatives: dm^{3}, cm^{3}, mm^{3}; auxiliary unit – liter (L) = 1 dm^{3}.

Table A2.5 **The ratio of non-SI unit of pressure**

Pressure unit*	Pa	am	mm Hg	N/mm^2
1 Pa (SI) * pascal	1	$1,02\cdot10^{-5}$	$7,5\cdot10^{-3}$	10^{-6}
1 am, physical atmosphere	$1,01\cdot10^{5}$	1	760	$9,81\cdot10^{-2}$
1 mm Hg, millimeter of Hg column	133,3	$1,36\cdot10^{-3}$	1	$1,333\cdot10^{4}$
1 N/mm^2, newton on squire millimeter	10^{6}	10,2	$7,5\cdot10^{3}$	1

* derivatives: GPa, MPa, mPa, μPa.

Table A2.6 **The ratio of non-SI units of energy, work and heat**

Unit of measurement*	J	eV	Watts	kcal
1 J (SI), joule	1	$6,242\cdot10^{18}$	1	$2,388\cdot10^{-4}$
1 eV electron volts	$1,6021\cdot10^{-19}$	1	$1,6021\cdot10^{-19}$	$3,827\cdot10^{-23}$
1 watt-second, watt-second	1	$6,242\cdot10^{18}$	1	$2,388\cdot10^{-4}$
1 kcal, kilocalorie	$4,1868\cdot10^{3}$	$2,6133\cdot10^{22}$	$4,1868\cdot10^{3}$	1

- * derivatives: kJ, MJ, GJ, TJ.

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Англійською мовою

Відповідальна за випуск Т.В. Соколова
Роботу до видання рекомендував Д.Ф. Донський

В авторській редакції

План 2018 р., поз.

Підп.до друку 2018 р. Формат 60х84 1/16. Папір офсетний.
Riso-друк. Гарнітура Таймс. Ум. друк. арк.
Наклад 100 прим. Зам. № Ціна договірна

Видавничий центр НТУ «ХПІ».
Свідоцтво про державну реєстрацію ДК № 3657 від 24.12.2009 р.
61002, Харків, вул. Кирпичова, 2

Друкарня НТУ «ХПІ». 61002, Харків, вул. Кирпичова, 2