**KAZAN (VOLGA REGION) FEDERAL UNIVERSITY**

**ALEXANDER BUTLEROV INSTITUTE OF CHEMISTRY**

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# ORGANIC CHEMISTRY

## LABORATORY EXPERIMENTS

**Учебно-методическое пособие**

**для иностранных студентов**

**на английском языке**

**УДК 547**
**ББК 24.2**
**Б91**

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## Safety in the organic chemistry laboratory

### Safety Guidelines

The organic chemistry laboratory has the potential to be very dangerous. This is why it is extremely important that you follow all safety guidelines when you are in the laboratory. You must make sure that you understand and follow all safety precautions. Ignoring safety precautions may result in a serious injury to yourself or another student. Any student working in an unsafe manner is a danger to other students and will be asked to leave the laboratory. Always adhere to the following safety guidelines:

1. **Wear eye protection.** Chemicals can cause serious damage to your eyes, including blindness. To prevent chemicals and other hazardous substances from coming in contact with your eyes, safety glasses or goggles must be worn at all times you or someone else is working in the laboratory.

2. **Dress appropriately for lab.** The less skin that is exposed, the better! Sandals are not allowed in the laboratory because they expose your toes to chemical spills and broken glassware. You must wear a lab coat. Avoid loose-fitting clothing and accessories (e.g., scarves, ties, long necklaces, etc.) because they may cause spills or come in contact with hazardous chemicals. Hair that is longer than shoulder length must be tied back.

3. **Wear gloves.** Gloves are required at all times when you are handling chemicals and items that have been exposed to chemicals. However, once you have gloves on, this does not mean that you are free to touch everything! For example, you do not to rub your eye with a gloved hand once you have handled chemicals.

4. **Keep the lab clean and neat.** Avoid cluttering the lab bench. Clutter can lead to spills, so return items promptly to their proper locations. Do not place personal items, such as bags and backpacks, on the floor as they may cause damage other people. Promptly notify the laboratory instructor of any spills and clean them up as instructed. Spills must be cleaned up as soon as possible to prevent others from exposure to the spilled chemical. Inform the laboratory instructor of any conditions that seem unsafe.

5. **Perform only authorized experiments.** Unauthorized experimentation may expose you and your lab mates to unforeseen hazards.

6. **Do not bring food, drinks, or cosmetics into the laboratory.** Eating, drinking, or applying cosmetics in the laboratory can introduce toxic or corrosive chemicals into your system. You particularly want to avoid any contamination to your mouth or eyes.

7. **Immediately report all incidents to the laboratory instructor (teacher or lab assistant).** A incident is any injury or situation that might result in an injury. The laboratory instructor will best know how to handle the situation and may also use the information you provide to help other students avoid a similar difficulty.

8. **Be familiar with the experiment before beginning the lab.** Pay particular attention to cautions given in the procedure and by the laboratory instructor.

9. **Ask "dumb" questions.** If you are not sure about how to properly perform a procedure or handle a chemical, ask your instructor. Regardless of whatever preconceived notions you may have, asking questions does not make you look stupid, especially when safety is a concern!

**Issues Specific to Particular Experiments**

Depending on the specific experiment that you will be performing, you may need to take additional safety precautions. The following are common experiment-specific issues that you may need to be aware of:

**Responding to Incidents in the Organic Chemistry Laboratory**

In addition to following all of the above safety precautions, you must be familiar with how to handle an incident in the laboratory and be willing to provide assistance to others in emergencies. If you are the first to notice an incident or hazard in the laboratory, you should immediately alert your laboratory instructor and others that may be in harm's way.

An incident that seems minor may be much more serious than you think, and your instructor is the best person to evaluate the situation. The following are incidents that you may encounter in the organic chemistry laboratory:

1. **Broken glass and other sharp objects.** Properly dispose of broken glass. Use a hand brush and dust pan to collect the pieces, and do not attempt use your hands, even if you are wearing gloves.

2. **Cuts.** For minor cuts, wash the affected area using soap then inform your laboratory instructor. If the injury does not stop bleeding on its own, apply gentle pressure with a clean paper towel or bandage. Go to the Student Health Center if you suspect a cut may be deep, the wound continues to bleed, or it is possible that chemicals have gotten into the wound. When you return to work, be particularly diligent about wearing gloves to prevent laboratory reagents from getting into the wound.

3. **Burns.** Hold the burned area under cool running water for 10 to 15 minutes or until the pain subsides, then inform your laboratory instructor.

4. **Chemical spills.** In the event of a reagent spill, immediately notify the laboratory instructor and anybody working in the vicinity. The appropriate steps to be taken will vary, depending on the amount and identity of the reagent. If the spilled chemical is flammable, remove all ignition sources, heat sources, and equipment that could produce a spark. If a spill creates a large amount of fumes, your instructor will direct you in the proper procedure to evacuate the laboratory.

5. **Chemical spills on a person.** Deal promptly with reagent spills because many organic chemicals are fat-soluble and can be absorbed through your skin. Remove any affected clothing (if necessary, down to your underwear) and then wash the area with running water for 10 to 15 minutes. Use a sink or safety shower depending on the size of the spill.

6. **Chemicals in the eyes.** A person who has gotten reagents into their eyes will require assistance. If this happens to you, ask a lab mate to assist you operate the water flow while you hold open your eyelids. If you are wearing contact lenses, remove them under the flow of water. Ensure that the flow of water gets to the entire eye surface for no less than 15 minutes. Besure to inform your laboratory instructor, and follow up with an examination by a health care professional.

7. **Fires.** Many solvents and chemicals used in the organic chemistry laboratory are highly flammable, and a fire may occur in the laboratory. If a fire does occur, step back from the fire and then immediately notify the laboratory instructor and anybody working in the vicinity. Move flammable materials away, and turn the equipment off or remove it from the vicinity of the fire. If a fire spreads to a larger area of the bench, the laboratory instructor or other authorized persons will direct you to evacuate the laboratory and the building.

## Experiments

## Distillation

**General considerations.** Distillation is the most important and widely used method for the purification of organic liquids and the separation of liquid mixtures. The procedure involves boiling the liquid (distilland) to vaporize it, and then condensing the vapour to give the distillate. The separation of a pair of liquids whose boiling points differ by c. 50-70°C or more can be carried out by ***simple distillation***, but if the difference in boiling points is less, more complicated apparatus is required and the process is known as ***fractional distillation***. Some organic compounds that are virtually immiscible with water may be separated from non-volatile impurities including inorganic contaminants by ***steam distillation***. Some liquids have boiling points that are too high to allow distillation at atmospheric pressure without causing thermal decomposition. Reducing the pressure lowers the boiling point and thus allows very high-boiling liquids and oils to be distilled easily and safely. The technique is known as ***vacuum distillation***. Great care must always be taken when distilling organic liquids since most of them ***are flammable***. The greatest hazard is posed by those which are also very volatile (e.g. ether, b.p. 35°C). Such compounds ***must not be distilied in any way that allows their vapour to come into contact with flames*** or any other source of ignition. A further hazard is posed by ***peroxides***, which may form when certain compounds, notably ***ethers and hydrocarbons***, are exposed to air. Liquids containing peroxides ***must NOT be distilied*** until the peroxides have been removed. Materials provided for use in teaching laboratories should be peroxide-free. However, in project work it is a hazard that must always be borne in mind.

## 1. Simple distillation

A typical assembly of apparatus for simple distillation is shown in Fig. 1. A system of this type will not give a clean separation of liquids with a boiling point (b.p.) difference of less than c. *50-70°C.* If it is used for a mixture where the b.p.'s are closer, then, the more volatile component will distil over first, it will be contaminated with the higher-boiling component even in the early stages of the distillation. But this method is the best for cleaning liquid from inorganic impurities and tar. The distillation flask is usually round-bottomed for large volumes and pear-shaped for volumes of 100 ml or less. Select a flask of a size such that it is no more **than two-thirds full at the beginning of the distillation**. The flask is connected to the condenser by a distilling head (Fig 1). The distilling head is connected to the condenser, then to vacuum adapter (allonge), and finally to receiving flask.

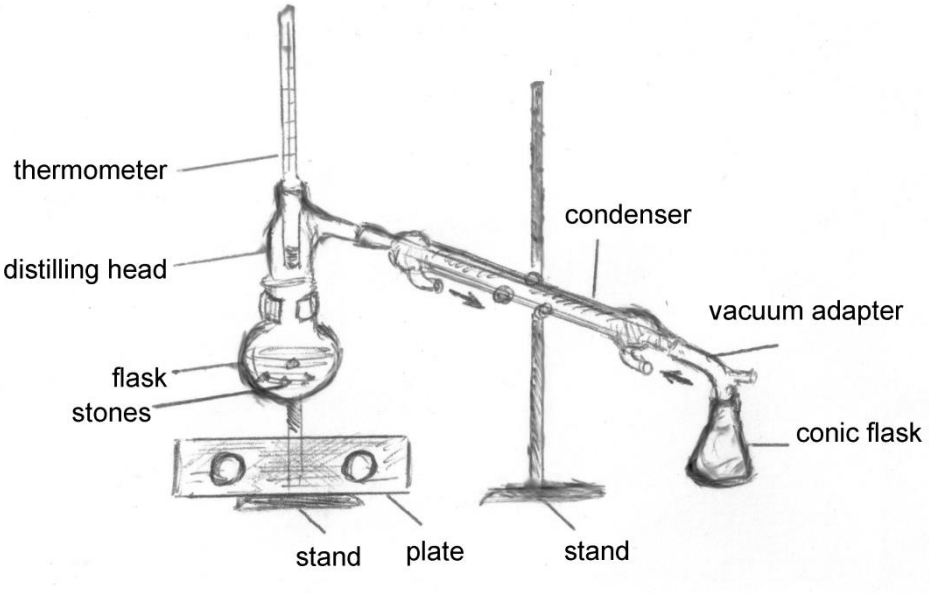



Fig. 1. Installation for simple distillation

### A. Setup:

1. Take a stand.
2. Place the heating plate on a stand.
3. Connect the holder to the stand.
4. Put the 100 ml round-bottomed flask into the holder.

5. Align the position of the flask - the distance between the flask and the heating plate should be about three centimeters.

6. Put into the flask 10 ml of an unknown dirty solvent using measuring cylinder.

7. Put into the flask porcelain beads or magnetic stirrer bar for uniform boiling.

8. Connect the round bottom flask to the distilling head.

9. Insert the thermometer into the distilling head.

10. Connect the condenser to water according to the Fig 1, using glycerin grease.

11. Take a second tripod / stand

12. Connect the holder to the second stand

13. Set the condenser

14. Align and connect the condenser with distilling head very carefully.

15. Connect the vacuum adapter to the condenser.

16. Connect receiving flask to the vacuum adapter.

**B. Distillation:**

1. Turn on the water valve and check the outflow of water – It should be sufficient for good cooling of condenser.

2. Turn on the heating plate.

3. The liquid will be heated and after boiling, the vapor will rise to the distilling head, and then condensed inside condenser.

4. Fix and write down the temperature of the vapor after boiling – this temperature corresponds to the boiling point of the liquid.

5. Collect about 90-95 vol % of the liquid. **Do not distill to dryness.**

6. Stop the distillation by turning off the heating plate. The water flow should be stopped only **after cooling the whole system**.

7. Measure the volume of collected liquid, the refractive index and find your liquid in handbook using two physical constants – boiling temperature and refractive index.

8. Wash all chemical glassware.

9. Make a report

**C. How to use a refractometer: (Fig. 2)**

1. Place a drop of solvent on the glass surface of refractometer.

2. Close the refractometer lid.

3. Look in the eyepiece.

4. Turn the handle until the border between the light and dark parts appears in the crosshair.

5. Look at the bottom on the green scale. Count the number of divisions on the scale.

6. The refractive index has 5 digits, for example: 1. 2345

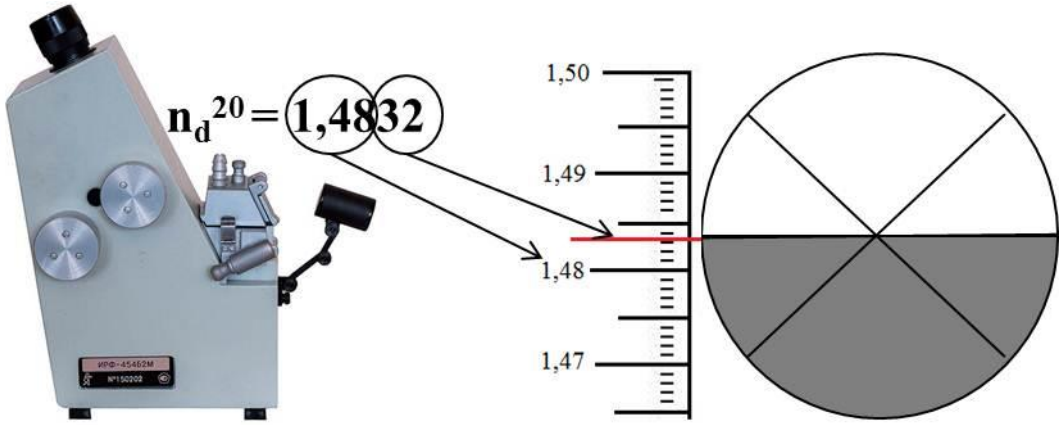



Fig. 2. Refractometer IRF-45452M

## 2. Steam distillation

Some organic compounds that are virtually immiscible with water may be separated from impurities including inorganic contaminants by steam distillation. This process is essentially a codistillation with water and is usually accomplished by passing a current of steam through a hot mixture of the material to be distilled and water. Provided that the compound possesses an appreciable vapour pressure (5 mmHg or more at 100°C) it will be carried over with the steam and, being immiscible, can be readily separated from the distillate using separating funnel. One of the advantages of steam distillation is that the temperature never exceeds the boiling point of water. This permits the purification of high-boiling substances that are too heat-sensitive. The method is also of importance in the separation of volatile products from tarry material, which is often produced during the organic reaction and cannot be removed easily by distillation or crystallization.

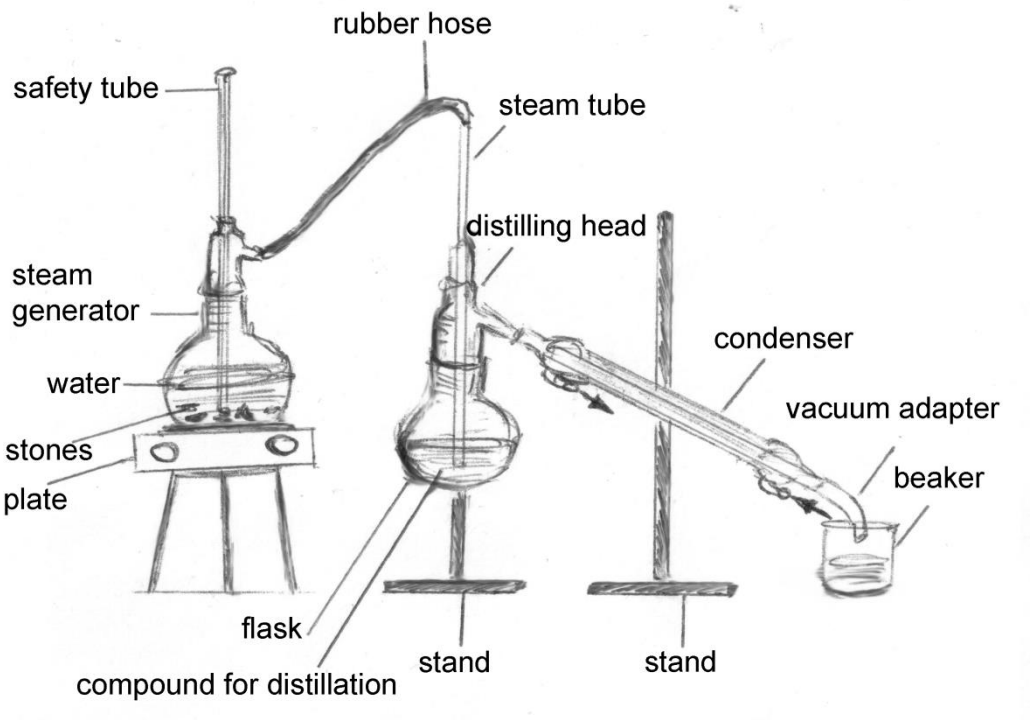



Fig. 3. Installation for steam distillation

A typical apparatus used for the steam distillation of large quantities of material is shown in Fig. 3. The material to be distilled is placed in a round-bottomed

flask fitted with a steam glass tube and distilling head, which is connected to condenser. The condenser is connected to the receiving flack. The steam outlet from steam generator is connected to an steam glass tube using rugger tube. Since some of the steam necessarily condenses in the distillation flask, it sometimes should be heated during the distillation on a heating mantle so that it is kept about half full. **(CAUTION)** The rubber tube and steam glass tube should not contain any barrier for steam. Otherwise hot water will be uptaken from safety valve.

**A. Setup:**

1. Take a stand for steam generator.
2. Place the heating plate on a stand.
3. Connect the steam generator to the stand.
4. Take a tripod / stand for distilling flack.
5. Put the 500 ml round-bottomed flask into the holder.
6. Put into the flask a portion of orange peel from at leat 1 orange or 10 ml of turpentine (depending from your teacher) using measuring cylinder.
7. Put into the flask porcelain beads or magnetic stirrer bar for uniform boiling.
8. Connect the round bottom flask to the distilling head.
9. Insert the steam glass tube into distilling head. **Be careful - tube should not contain any barrier for steam!!!.**
10. Connect the condenser to water according to the Fig 3, using glycerin grease.
11. Take a third tripod / stand.
12. Connect the holder to the third stand.
13. Set the condenser.
14. Align and connect the condenser with distilling head **very carefully**.
15. Connect the vacuum adapter to the condenser.
16. Connect receiving flask to the vacuum adapter.

**B. Distillation**

1. Turn on the water valve and check the outflow of water – It should be sufficient for good cooling of condenser.

2. Add 3-4 fresh porcelain beads into the steam generator and turn on the heating plate of steam generator.

3. Connect the rubber tube from generator to glass steam tube.

4. After starting boiling the steam from steam generator will go into distilling flack and the process will start.

5. Collect the liquid. Finish the process when no emulsion will be detected.

6. Firstly, **very carefully** disconnect steam tube than turn off heating plate. Stop the distillation by turning off the heating plate.

7. Separate the organic phase using separating funnel (Fig.4).

8. Wash all chemical glassware.

9. Make a report.

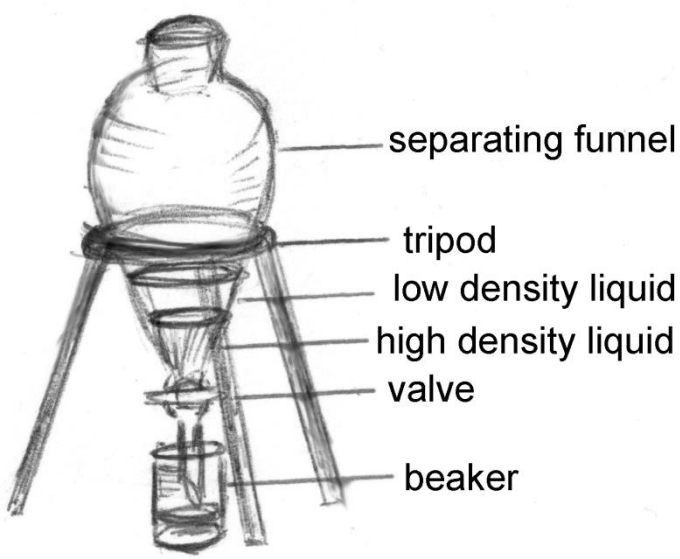



Fig. 4. Separation funnel

## 3. Crystallization

Crystallization is the most common method for the purification of organic solids that are not heavily contaminated with other substances. It is one of the most frequently performed operations in practical organic chemistry and, although it is not basically a difficult technique, **it does need much practice to do it well**.

The technique makes use of the knowledge that solid compounds are much more soluble in hot solvents than in cold ones. Thus if you prepare a saturated hot solution of compound **A** and allow it to cool, the solution will become supersaturated and the compound will separate out as crystals. If the compound is impure, for example, contains a few percent of another compound **B**, then the impurity will also dissolve in the hot solvent, but when it cools down the solution will not be supersaturated with compound **B** (because it is present in low concentration) and it will stay in solution while the major component, compound **A**, crystallizes out. Thus the pure, crystalline **compound A can be filtered off** while the impurity **B stays in solution** in the filtrate (generally known as the mother liquor).

The key to success in crystallization lies in using the best solvent, i.e. one that will dissolve the material easily when hot, but in which the major component is almost insoluble when cold, so allowing most of it to crystallize out.

So, the technique of solution recrystallization involves the following steps:

l. Selection of an appropriate solvent.

2. Dissolution of the solid to be purified in the solvent near or at its boiling point.

3. Formation of crystalline solid from the solution as it cools.

4. Isolation of the purified solid by filtration.

5. Drying the crystals.

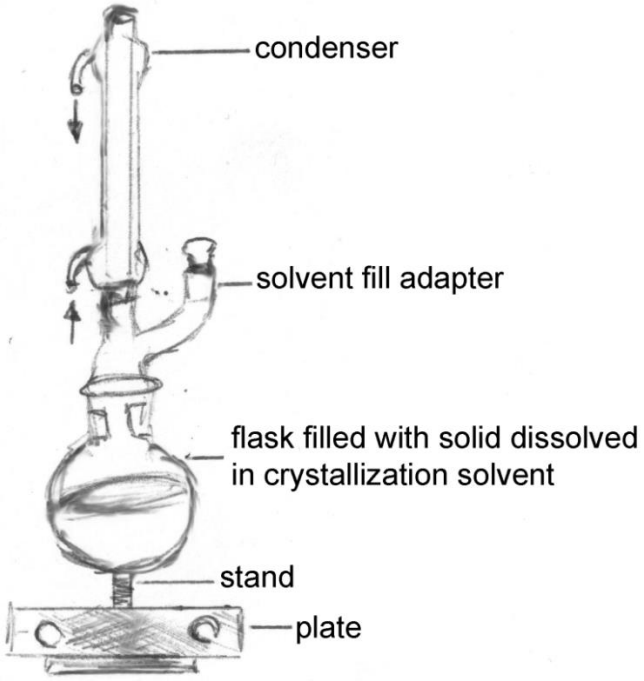



Fig. 5. Crystallization installation

A typical apparatus used for the crystallization is shown in Fig. 5. It contains conical or round bottom flask, connected to condenser, and heating plate or water bath.

**A. Setup:**

1. Take a stand.
2. Place the heating plate on a stand.
3. Put the 50-100 ml round-bottomed flask into the holder.
4. Put into the flask porcelain beads or magnetic stirrer bar for uniform boiling.
5. Connect the condenser to water according to the Fig 5, using glycerin grease.
6. Connect the round bottom flask to the condenser.

**B. Crystallization:**

1. Turn on the water valve for condenser and check the outflow of water – It should be sufficient for good cooling of condenser.
2. Put 1 gram of unknown solid into the flask

3. Add 10-15 starting amounts of solvent (ask teacher what solvent to use).

4. Heat the solution until it will start boiling.

5. Check the transmittance of liquid – it should be absolutely transmitting.

6. If it is not absolutely transmitting, put more solvent using measuring cylinder.

7. After all dissolves, turn off heating plate. Discnnec the flasc and put all liquid into beaker.

8. Put the beaker in the cold place.

9. After some time, the solid will start to form.

10. Put the solid with mother liquor into the Büchner funnel with paper filter on it (Fig 6)

11. Put chemical glasses, turn on the vacuum pump (or water pump) and connect the rubber tube from pump to the Büchner flask.

12. Wait until the end of filtering

13. Put the crystals on the filtering paper and then put them on the warm place to dry

14. After drying, measure the weight and melting point of solid.

15. Make a report.

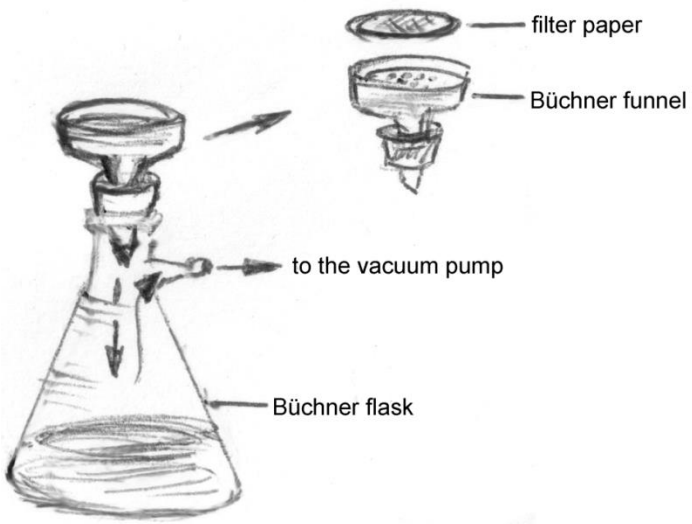



Fig. 6. Filtering system installation

## 4. Qualitative analysis of organic compounds

Organic compounds are mostly composed up of the elements like carbon, hydrogen, oxygen, nitrogen, halogens, sulphur, and rarely phosphorous. Out of these elements, C and H are generally found in organic compounds. Elements other than H and C present in the organic compound is referred as foreign elements (or hetero elements). Examples are N, S, P, and halogens.

Simultaneous detection of carbon and hydrogen:

$2CuO + C \rightarrow \Delta\ 2\ Cu + CO_2$

$CO_2 + Ba(OH)_2 \rightarrow BaCO_3 + H_2$ **Precipitation of $BaCO_3$!**

$CuO + 2H \rightarrow \Delta\ Cu + H_2O$ **Fogging the test tube by $H_2O$!**

1. A dry combustion tube of refractory glass with a volume of ~40 ml, intended for burning the sample, is closed with a rubber stopper with a gas outlet tube and fixed in a clip for test tubes.

2. The end of the outlet tube is immersed in water (1 ml), poured into another tube. Heat combustion tube by hands and check the release of air bubbles from the gas pipe (see the tube with water), which indicates the tightness of the connections of the device.

3. About 100 mg of the test substance, as well as 500-1000 mg of powdered copper oxide (II), are placed in a combustion tube.

4. Connect the gas outlet pipe to a combustion tube, again check the tightness of the connections of the device.

5. Put 2-3 ml of a saturated solution of $Ba(OH)_2$ into a test tube in a tripod.

6. The combustion tube is heated with a spirit lamp.

7. The formation of water is detected by the appearance of water droplets on the upper part of combustion tube, and the release of $CO_2$ (and $SO_2$) is detected by the cloudiness of the Ba (OH) solution due to the formation of a poorly soluble carbonate (and barium sulphite).

Alloying of substance with sodium. Detection of sulfur and nitrogen:

1. At the bottom of a dry 15 ml tube is placed about 100 mg of the test substance.

2. Fixe tube in a clip for test tubes.

3. Take a piece of sodium (the size of a pea) and soak the oil by dry filter paper.

4. Wipe the tip of the scalpel with a filter paper from the oil and with its help transfer sodium to a horizontally placed test tube with the substance so that the sodium must be 2-3 cm from the bottom.

5. Near the spirit lamp, place a 50 ml glass beaker, filled one third with water.

6. While holding the tube in the clamp horizontally, gently heat it with a spirit lamp so that the sodium melts.

7. **Immediately** after melting the sodium, the tube is turned vertically and shaken to make the sodium come into contact with the substance

8. The bottom of the test tube is heated at first carefully (**do not point the hole at people, gently, there is a flash!**), and in the end - to red.

9. The incandescent tube is **quickly immersed in a beaker with water** to destroy it and transfer the reaction products to the solution.

10. The resulting mixture is filtered through a funnel with a paper filter into a 3 test tubes located in a tripod. The filtrate is used for nitrogen and sulfur tests.

$$Na + C + N \rightarrow NaCN$$

$$2\ Na + S \rightarrow Na_2S$$

$$Na + C + N + S \rightarrow NaCNS$$

Nitrogen probe:

1. To 2-3 ml of the obtained filtrate placed in a separate tube, is poured 0.1 ml of a solution containing a mixture of iron (II) sulfate and iron (III).

2. The mixture is boiled for 30 seconds, than cooled under a stream of cold water, and is added concentrated hydrochloric acid dropwise under shaking after adding each drop.

3. The blue color of the mixture indicates the formation of Berlin blue $FeNa[Fe(CN)_6]$ and the presence of nitrogen in the analyte.

Sulfur probe:

1. To 1 ml of the filtrate, placed in a separate tube, several drops of a solution of lead acetate are added.

2. The appearance of a dark precipitate of lead sulfide PbS indicates the presence of sulfur in the analyte.

3. Another sample for sulfur is carried out by adding a solution of sodium nitroprusside $Na_2[Fe(CN)_5(NO)]$to the same portion of the filtrate.

4. The appearance of a bright-violet color of $Na_4[Fe(CN)_5(NOS)]$ also indicates the presence of sulfur in the starting material.

Belstein test detection of halogens:

1. The tip of the copper wire is calcined in the flame of the spirit lamp until it ceases to turn green.

2. In order to find out what the positive BELSTEIN TEST looks like, moisten the calcined end of the wire in $CCl_4$ and immediately inject wire into the flame. Pluml is colored in a bright green color in pairs of CuCl halides.

3. The tip of the copper wire is calcined in the flame of the spirit lamp until it ceases to turn green again.

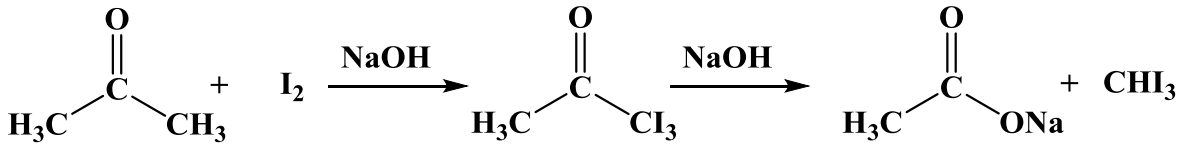
4. Then touch the analyzed solid by hot wire and inject the sample into the flame of the spirit lamp.

5. Depending on whether the flame is colored green or not, it is concluded that halogen is present or absent in the analyte.

## 5. Functional analysis

A methylketone group test:

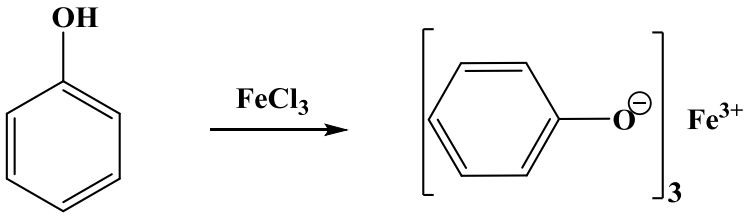
Iodoform reaction. 1 ml of solution containing iodine, dissolved in potassium iodide solution and 5 ml of sodium hydroxide solution are placed in a test tube (colour dissapears). Add 1-2 drops of 10% acetone to a discolored solution. If a methyl ketone group is present, a yellow-white solid with a characteristic odor of iodoform precipitates immediately without heating.



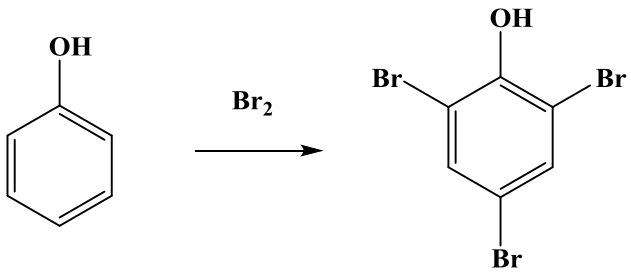

Phenolic test:

The formation of phenolic compounds is one of the features of the plant cell. Simplest diphenols are pyrocatechol, resorcinol and hydroquinone, and triphenols - pyrogallol and floroglucin. These substances are part of many natural compounds.

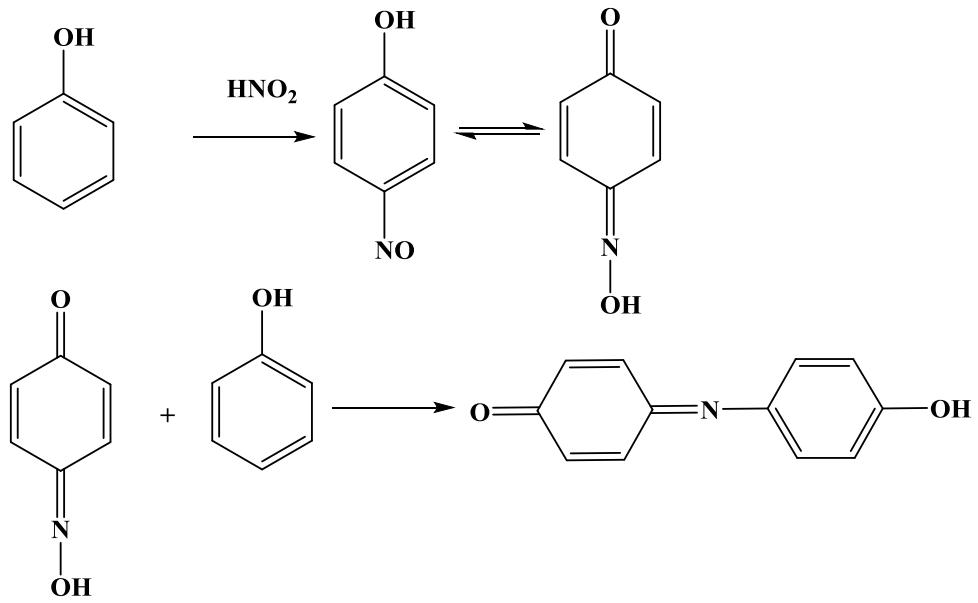
a) Reaction with iron (III) chloride. Add 2-4 drops of 2% iron (III) chloride solution to a 1 ml of 1% phenol solution placed in test tube. In test tube blue violet color appear immediately.




b) Reaction with bromine water. To 0.5-1 ml of 1% phenol solution plased on the test tube add 3-5 drops of bromine water. In the presence of phenol, a yellowish-white precipitate of tribromophenol is formed in the test solution.



c) Lieberman's reaction. This reaction is based on the formation of indophenol. In a test tube, add 1 crystal of sodium nitrite, 3 drops of concentrated sulfuric acid and 1 drop of liquid phenol. In test tube blue staining appears.




d) Nitrosoreaction of phenol. In a test tube, place 3-4 drops of concentrated sulfuric acid, 2 drops of a freshly prepared solution of phenol and cool the resulting solution. Add 1 drop of 0.1% solution of sodium nitrite. Shake. The mixture is colored green. Pour the colored liquid into a test tube with 10 drops of water - a pink-red solution is formed. Pipette 5 drops of this solution into another tube and add a few drops of 10% sodium hydroxide. The pink color changes to green or bluish. When the acid is added, the solution turns pink.

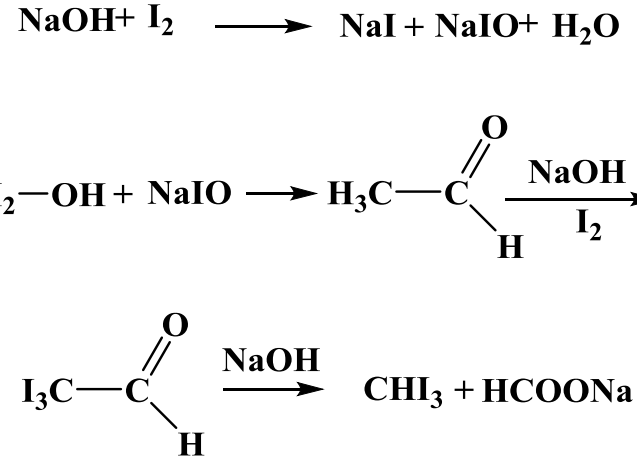
Alcohol detection reactions:

a) The Lucas test-identification of primary, secondary and tertiary alcohols. Primary, secondary and tertiary aliphatic alcohols with a carbon number of less than 6 react with concentrated hydrochloric acid in the presence of zinc chloride to produce the corresponding haloalkanes. In the Lucas probe, haloalkanes are formed at different rates, which are used to identify the initial alcohols.

To conduct the experiment, in each of the three test tubes containing 3 drops of alcohol, add 6 drops of Lucas's reagent, shake and observe the changes occurring in the test tubes at room temperature for 5 minutes.

b) Oxidation of alcohols with chrome mixture. Place 2 drops of ethyl alcohol in the tube, 1 drop of a 10% solution of sulfuric acid and 2 drops of 10% potassium dichromate. The resulting solution has an orange color. Heat the solution over the flame of the spirit lamp until it begins to acquire a bluish-green color. There is a characteristic smell of acetic aldehyde.

c) Iodoform probe. To 1 ml of the test solution add 2 ml of 5% sodium hydroxide solution, and then drop by drop a 1% solution of iodine in a 2% solution of potassium iodide until a slightly yellow color is maintained. The mixture is heated for several minutes in a water bath, and a smell of iodoform is felt. When the solution is cooled, iodoform crystals are formed.




Test for the amine group (aniline):

A solution of 0.5 ml of aniline in 6 ml of hydrochloric acid (1: 3 (HCl: $H_20$)) is prepared. A drop of the resulting solution is applied to the filter paper. On the formed spot, drop a 10% solution of sodium nitrite, then a drop of N,N-dimethylaniline. After a while, there is an increasing orange staining of the spot.

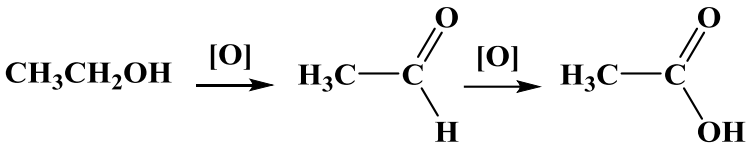
Test for aldehydes (reaction of a silver mirror):

Preparation of an ammonia solution of silver oxide: To 1ml of 1% solution of silver nitrate add a drop of water ammonia. Then a next drop of ammonia is added to dissolve the precipitate. To the resulting liquid, add 2 drops of a 2M solution of sodium hydroxide.

Add 1 ml of glucose solution to the resulting $[Ag(NH_3)_2]OH$, mix and then place for several minutes in hot water. If the test tube was clean, a mirror layer forms on the walls. Otherwise, a black precipitate of colloidal silver precipitates.

## 6. Synthesis of acetic acid

General synthesis of acetic acid goes according to equation:




**A. Setup:**

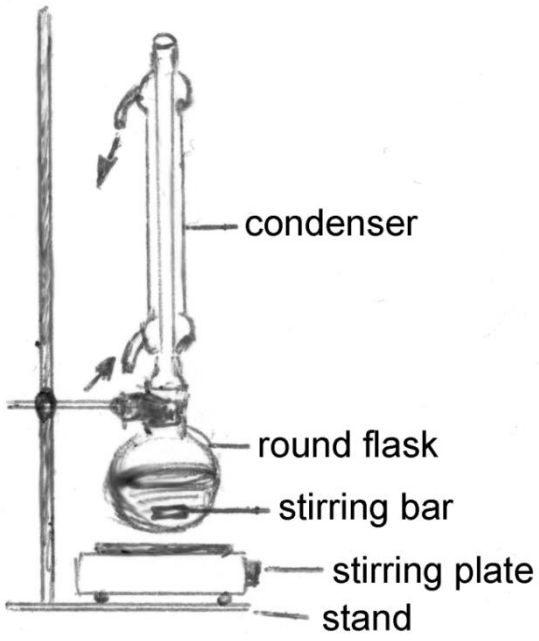



Fig. 7. Installation for synthesis of acetic acid

1. Take a stand.
2. Place the heating plate on a stand.
3. Connect the holder to the stand.
4. Take 100 ml round-bottomed flask into the holder.
5. PUT CHEMICAL GLASES ANG GLOVES
6. Pour **5 ml of water** into the 100 ml round-bottomed flask first, then slowly, with stirring, **4 ml of sulfuric acid** and **3.5 g of powdered potassium dichromate**. The resulting mixture must be gently stirred and cooled.
7. Put the 100 ml round-bottomed flask into the holder.

8. Align the position of the flask - the distance between the flask and the heating plate should be about three centimeters.

9. Put two-three boiling stones into the flask.

10. Connect the flask to a reflux condenser (connected to water, water inlet is downside, water outlet is upside of condenser).

11. The mixture consisting of **2 ml of ethanol and 6 ml of water** is slowly poured into the flask through the reflux condenser.

12. The reaction mixture is then gently heated and boiled for **15 minutes.**

13. After 15 minutes, the flask is cooled, the reflux condenser is removed

14. Connect the round bottom flask to the distilling head.

15. Insert the stopper into the distilling head.

16. Align and connect the condenser with distilling head **very carefully**.

17. Connect the vacuum adapter to the condenser

18. Connect receiving flask to the vacuum adapter.

19. Put new boiling stones into the flask.

20. Start heating

21. Collect the liquid, measure the volume

22. Calculate the yield of acetic acid according to the moles of the starting compounds.

**B. Reactions with acetic acid**

Divide the distillate into two test tubes.

1. Determine the color and smell of the distillate.
2. Determine the pH of the distillate.
3. Add magnesium tape to one tube.
4. Add a small piece of chalk to the second test tube.

**C. Reactions with ethanol**

Divide the starting ethanol into two test tubes.

1. Determine the color and smell of ethanol.
2. Determine the pH of ethanol.
3. Add a piece of magnesium tape to ethanol.

4. Add a piece of chalk to the ethanol.