LABORATORY WORK №1

Introduction of the laboratory safety

Before you begin working in the chemistry laboratory, your instructor should review the safety rules and guidelines tell you what safety supplies, such as safety goggles and protective gloves you will need to use in the lab. In a first time working in a during laboratory period, the instructor will show you where safety equipment is located and tell you how to use it. As you locate each item, check it off the following list and make a note of its location and proper working, which is not expire:

1. Fire extinguishers.
2. Fire blanket.
3. Safety shower.
4. Eyewash fountain.
5. First aid supplies.
6. Spill cleanup supplies.

Every students and academician you should also learn the locations of chemicals, glassware consumable supplies (such as filter paper and boiling chips), waste containers, and various items of equipment such as balances and drying oven. If you find any glassware items with chips, cracks, or star fractures, you should have them replaced; they may cause cuts, break on heating, or shatter under stress. If necessary, clean up any dirty glassware and organize it neatly at this time.

Proper Working Efficiently

If all labs were geared to the slowest student, the objectives of the course could not be accomplished in the limited time available. Because of wide variations in individual working rates, it is usually not possible to schedule experiments so that everyone can finish in the allotted time. If you fall behind in the lab, you may need to put in extra hours outside your scheduled laboratory period in order to complete the course. The following suggestions should help you work more efficiently and finish each experiment on time:

Be prepared to start the experiment the moment you reach your work area: Don’t waste precious minutes at the start of a laboratory period doing calculations, reading the experiment, washing glassware, or carrying out other activities that should have been done at the end of the previous period or during the intervening time. The first half hour of any lab period is the most important – if you use it to collect the necessary materials, set up the apparatus, and get the initial operation (reflux, distillation, etc.) under way, you should have no trouble completing the experiment on time.

Organize your time efficiently: Schedule a time each week to read the experiment and operation descriptions and to complete the pre-lab assignment - an hour before the lab period begins is too late! Plan ahead so that you know approximately what you will be doing at each stage of the experiment. A written experimental plan is invaluable for this purpose.

Organize your work area: Before performing any operation, arrange all of the equipment and supplies you will need during the operation neatly on your benchtop, in the approximate order in which they will be used. Place small objects and any items that might be contaminated by contact with the benchtop on a paper towel, laboratory tissue, or mat. After you use each item, move it to an out-of-the way location where it can be cleaned and returned to its proper location when time permits; for example put dirty glassware in a washing trough in the sink.

Leave all chemicals where you can find them: You will understand the reason for this rule once you experience the frustration of hunting high and low for a reagent, only to find it at another's student's station in a far corner of the lab.

Take only what you need: Whenever possible, liquids and solutions should be obtained using pipets, graduated cylinders, or other measuring devices so that it will take no more than you expect to use for a given operation.

Basic Safety Rules

These basic rules provide behaviour, hygiene, and safety information to avoid accidents in the laboratory. Laboratory specific safety rules may be required for specific processes, equipment, and materials, which should be addressed by laboratory specific SOPs. Basic safety rules for laboratory conduct should be observed whenever working in a laboratory. Many of the most common safety rules are listed below:

1. Know locations of laboratory safety showers, eyewash stations, and fire extinguishers. The safety equipment may be in the hallway near the laboratory entrance.
2. Know emergency exit routes.
3. Avoid skin and eye contact with all chemicals.
4. Minimize all chemical exposures.
5. No horseplay will be tolerated.
6. Assume that all chemicals of unknown toxicity are highly toxic.
7. Post warning signs when unusual hazards, hazardous materials, hazardous equipment, or other special conditions are present.
8. Avoid distracting or startling persons working in the laboratory.
9. Use equipment only for its designated purpose.
10. Combine reagents in their appropriate order, such as adding acid to water.
11. Avoid adding solids to hot liquids.
12. All laboratory personnel should place emphasis on safety and chemical hygiene at all times.
13. Never leave containers of chemicals open.
14. All containers must have appropriate labels. Unlabeled chemicals should never be used.
15. Do not taste or intentionally sniff chemicals.
16. Never consume and/or store food or beverages or apply cosmetics in areas where hazardous chemicals are used or stored.
17. Do not use mouth suction for pipetting or starting a siphon.
18. Wash exposed areas of the skin prior to leaving the laboratory.
19. Long hair and loose clothing must be pulled back and secured from entanglement or potential capture.
20. No contact lenses should be worn around hazardous chemicals – even when wearing safety glasses.
21. Laboratory safety glasses or goggles should be worn in any area where chemicals are used or stored. They should also be worn any time there is a chance of splashes or particulates to enter the eye. Closed toe shoes will always be worn in the laboratory. Perforated shoes or sandals are not appropriate.
22. Do not utilize fume hoods for evaporations and disposal of volatile solvents.
23. Perform work with hazardous chemicals in a properly working fume hood to reduce potential exposures.

24. Avoid working alone in a building. Do not work alone in a laboratory if the procedures being conducted are hazardous.

25. The PEL and the Threshold Limit Values (TLV) will be observed in all areas. If exposure above a PEL/TLV is suspected of an ongoing process, please contact EHS immediately.

26. Laboratory employees should have access to a chemical inventory list, applicable SDSs, department laboratory safety manual, and relevant SOPs.

27. Determine the potential hazards and appropriate safety precautions before beginning any work.

28. Procedures should be developed that minimize the formation and dispersion of aerosols.

29. If an unknown chemical is produced in the laboratory, the material should be considered hazardous.

30. Do not pour chemicals down drains. Do not utilize the sewer for chemical waste disposal.

31. Keep all sink traps (including cup sink traps and floor drains) filled with water by running water down the drain at least monthly.

32. Access to laboratories and support areas such as stockrooms, specialized laboratories, etc. should be limited to approved personnel only.

33. All equipment should be regularly inspected for wear or deterioration.

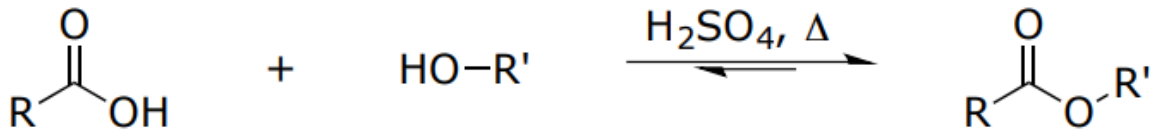
34. Equipment should be maintained according to the manufacturer's requirements and records of certification, maintenance, or repairs should be maintained for the life of the equipment.

35. Designated and well-marked waste storage locations are necessary

Lab 2-4

Esterification

Background In this experiment you will react a carboxylic acid and an alcohol under acidic conditions to form the corresponding ester. You will be assigned one of two possible esters.



carboxylic acid alcohol ester

Esters can be prepared by this method in the presence of an acid catalyst. To force the reaction equilibrium to the right (in favor of the ester), one of the starting materials must be used in excess.

As the carboxylic acid is more easily removed from the reaction mixture, it will be used as the excess reagent. Additionally, a drying tube will be used to prevent any additional water in the atmosphere from getting into the reaction.

Directions 1. Ensure that all glassware is clean and dry. (The addition of any water will adversely affect the outcome of the reaction.) If your glassware is wet, dry it in an oven before proceeding.

2. Add 45 mmol of your alcohol followed by 120 mmol of your carboxylic acid to a 25 or 50 mL round bottom flask.

3. Place a magnetic stirrer into the flask.

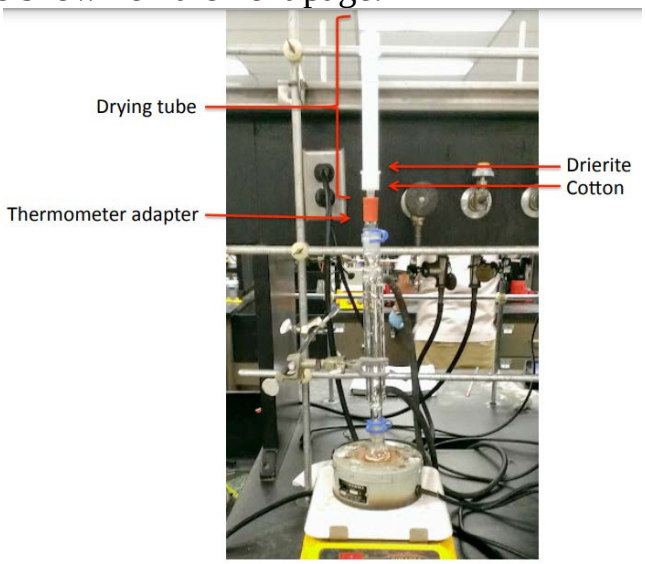
4. While stirring, dropwise add 1.0 mL of concentrated sulfuric acid under a fume hood or snorkel.

5. Prepare an apparatus for reflux using a water-cooled condenser.

6. Use a drying tube to prevent any additional water from getting into your reaction.

a. Pack the drying tube with cotton (to prevent your drying agent from falling out) and then add about 1-2 cm of drierite (calcium sulfate).

b. Attach the packed drying tube to the top of your condenser via your thermometer adapter as shown on the next page.



7. Have your instructor check your apparatus before proceeding.

8. Turn on the rheostat and adjust the heat until the reaction boils gently. (You should see the vapors condense and the liquid drip back down into the round bottom.)

9. Once you are sure your reaction is refluxing, continue heating under reflux for at least 60 minutes.

10. When the reflux period is complete, disconnect or remove the heating source and let the mixture cool.

11. Once your reaction is complete and has cooled to room temperature, disassemble the apparatus, and transfer the reaction mixture to your separatory funnel.

12. Add 15 mL of ice cold water and mix the phases by careful shaking and venting. 13. Allow the phases to separate, and then discard the aqueous layer. (Be sure you know which layer is the aqueous layer…you will need to know the densities of water and your ester.)

14. Next wash the organic layer by adding 5 mL of 5% aqueous sodium bicarbonate, then shake and vent just as you did with the water.

15. Again, discard the aqueous layer again. Wash one final time with 5 mL of saturated aqueous sodium chloride, and discard the final aqueous layer.

16. Transfer your product to a clean beaker or Erlenmeyer flask and add a scoop of anhydrous sodium sulfate to the organic layer containing the crude ester.

17. Cap the mixture and let it stand for about 10-15 minutes. If the liquid is dry, the sodium sulfate will be loose. If the liquid is not dry (as evidenced by clumping of the sodium sulfate and/or visible droplets of water), add another portion of anhydrous sodium sulfate to complete the drying.

18. Filter to remove the sodium sulfate.

19. Purify the crude ester via a microscale fractional distillation using an apparatus with a Hickman still (see figure below).

a. Obtain a microscale kit and add stainless-steel sponge to the air condenser to make a fractional distilling column.

b. Transfer your crude ester to an appropriately sized flask from the microscale kit and add a stirbar.

c. Prepare your flask for distillation using a heating mantel (or aluminum block) and stir plate.

d. Attach a fractionating column to the top of your flask.

e. Next, attach a Hickman still to the top of the fractionating column and ensure that everything is well supported with clamps.

f. Insert a thermometer into the top of your apparatus (you will need an additional clamp for the thermometer).

g. Cover your fractionating column with cotton or glass wool wrapped in aluminum foil to speed up the distillation process.

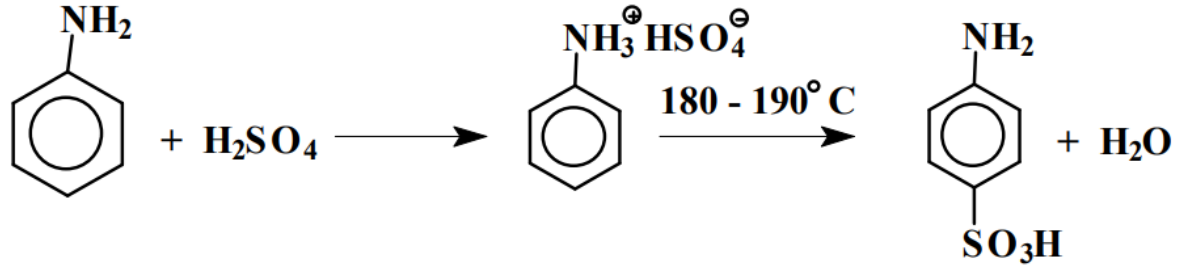
h. Have your instructor check your apparatus before proceeding.

i. Distill your ester. Be sure to remove any lower boiling contaminants from the Hickman still before you collect pure product. Don't forget to record the boiling point range of your purified ester.

20. Weigh the product, and calculate the percentage yield of the ester

Lab 5

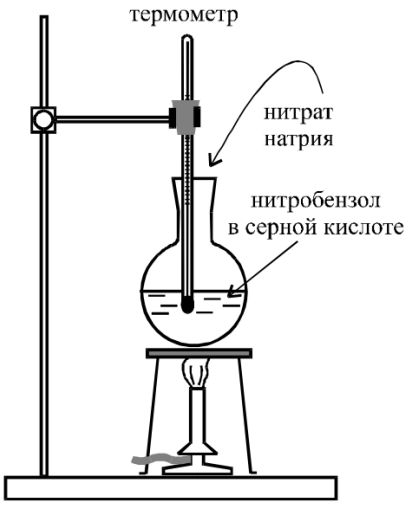
Synthesis of sulfanolic acid



анилин анилиния гидросульфат сульфаниловая кислота

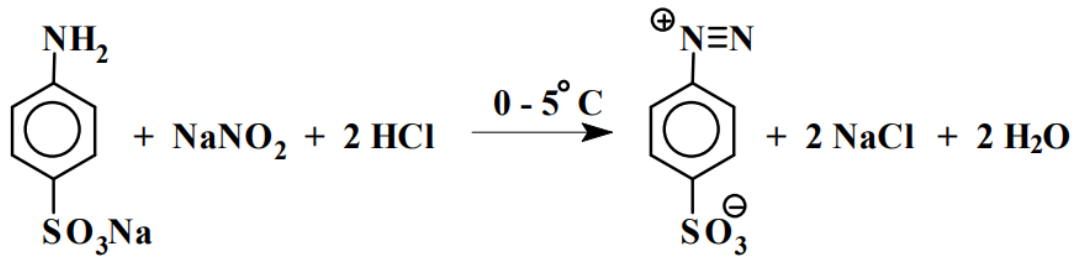
The work is done under pressure. For the synthesis, the installation used in the preparation of m-dinitrobenzene is used (Fig. 27 on p. 115). 15 ml of freshly distilled

aniline are placed in the flask and 27 ml of concentrated sulfuric acid (d = 1.84 g/cm3) are added to it in small portions so that the mixture does not overheat. The solution is heated on a grid at 180 - 190 °C until the sample, dissolved in a hot aqueous solution of alkali, ceases to release aniline (oil droplets). Sulfonation takes 1.5 - 2 hours. Overheating of the reaction mixture above 190°C leads to its charring. Upon completion of sulfonation, the reaction mass cooled to 80–90 °C is poured with stirring into 200 ml of cold water. When cooled, sulfanilic acid precipitates. The acid is filtered off on a Buchner funnel. washed twice with small portions of cold water and recrystallized from water with the addition of activated carbon. The method of recrystallization is described in the work on obtaining acetanilide (p. 88 - 89). Yield of sulfanilic acid p-H2N-C6H4-SO3H*2H2O as dihydrate 15–17 g (43–49%).



Lab 6

Synthesis of methyl orange



n-аминобензолсульфонат *n*-диазобензолсульфонат

Preparation of p-diazobenzenesulfonate*. Dissolve 2.3 g of sodium sulfanate in 5 ml of water in a beaker and add to it a solution of 0.7 g of sodium nitrite (NaNO2) in 10 ml of water (Note 1). While cooling with ice or snow and stirring, the resulting solution is poured into 10 ml of 4 N hydrochloric acid solution. Not earlier than 5 minutes after draining the solutions, a sample of the reaction mixture

is tested for the presence of some excess of nitrous acid in it according to starch iodine paper. In the absence of nitrous acid in the sample, about 0.1 g of nitrite in 1 ml of water should be added to the reaction mixture. During the diazotization, a white suspension of p-diazobenzenesulfonate is formed, which, without filtering the precipitate (Note 2), is used to obtain one of three water-soluble azo dyes: methyl orange, acid orange (β-naphtho-orange) or resorcinol yellow (tropeolin). Prior to the azo coupling, a glass with a suspension of p-diazobenzenesulfonate should be kept in a mixture of water with ice or snow.

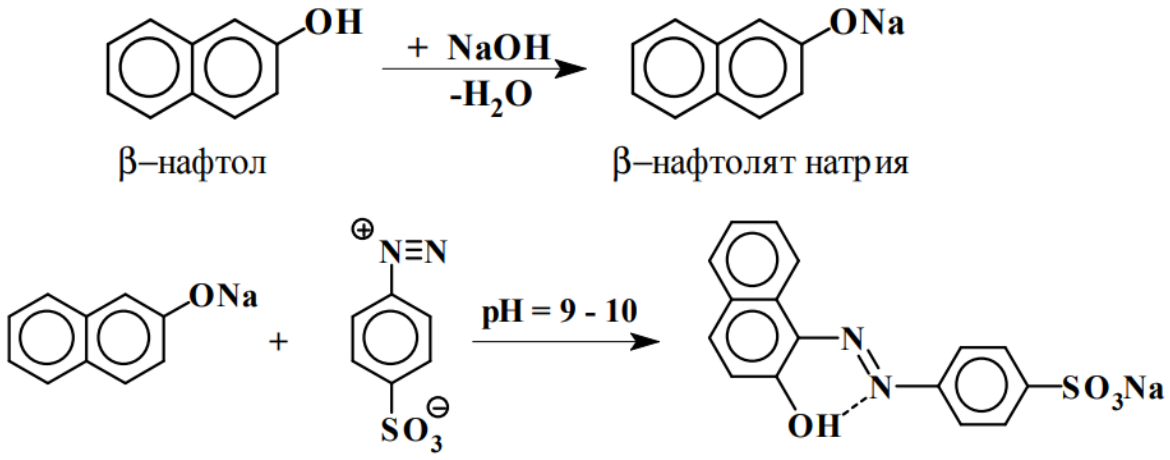
Synthesis notes

1. In the absence of a ready-made sodium sulfanilic acid reagent, its solution can be prepared from 1.7 g of anhydrous sulfanilic acid and 5 ml of 2 N sodium hydroxide solution according to the reaction:

2. In dry form, p-diazobenzenesulfonate is explosive, so do not filter out its precipitate! The resulting suspension should not be left for a long time, it should be used on the day of receipt.

Lab 7

Synthesis of acid orange (β-naphtho orange)

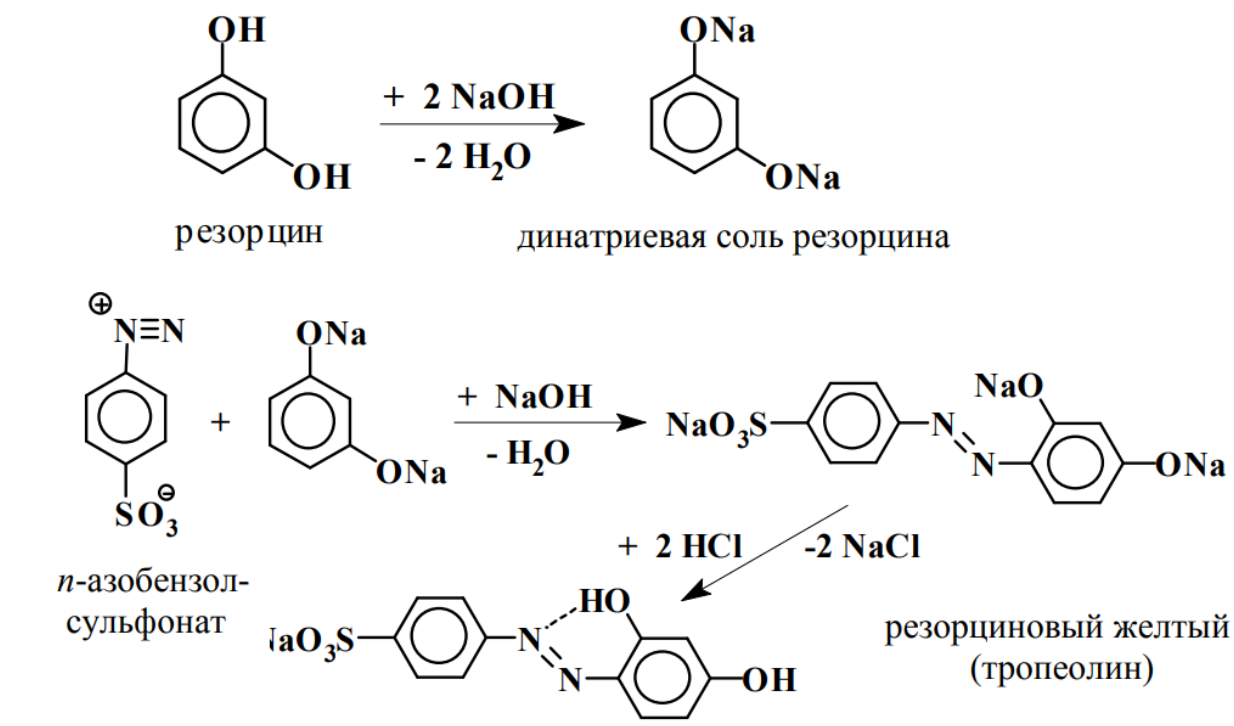


Dissolve 1.4 g of finely ground β-naphthol in 20 ml of 2 N sodium hydroxide solution with gentle heating. A solution of sodium naphtholate is cooled in a mixture of water with ice or snow, and a cooled suspension of p-diazobenzenesulfonate is added to it. After some time, the crystallization of orange-yellow leaves of the sodium salt of the dye begins. To reduce the solubility of β-naphthol-orange, 3 g of

finely ground sodium chloride is added to the mixture, heated until the salts dissolve, and the solution is allowed to cool. The precipitated dye is filtered off on a small Buchner funnel and dried to constant weight at a temperature not exceeding 60 °C. The calculation of the yield of the dye as a percentage of the theoretical is carried out taking into account the content of the active substance in the resulting preparation. Yield 80%.

Lab 8

Resorcinol yellow (tropeolin)



Dissolve 1.1 g of resorcinol in 25 ml of 2 N sodium hydroxide solution. To this solution, preliminarily cooled to 5°C, a cooled suspension of p-diazobenzenesulfonate is added with stirring. The temperature during azo coupling should not be higher than 15 ° C. The resulting dye is isolated by acidifying the reaction mixture with concentrated hydrochloric acid (test on indicator paper). The dye precipitate is filtered off on a small Buchner funnel, washed on the filter with 1–2 ml of ice water and dried to constant weight at a temperature not exceeding 50 ° C. The content of the active dye in the resulting preparation is determined as described below, and the yield of pure dye is calculated as a percentage of theoretical.

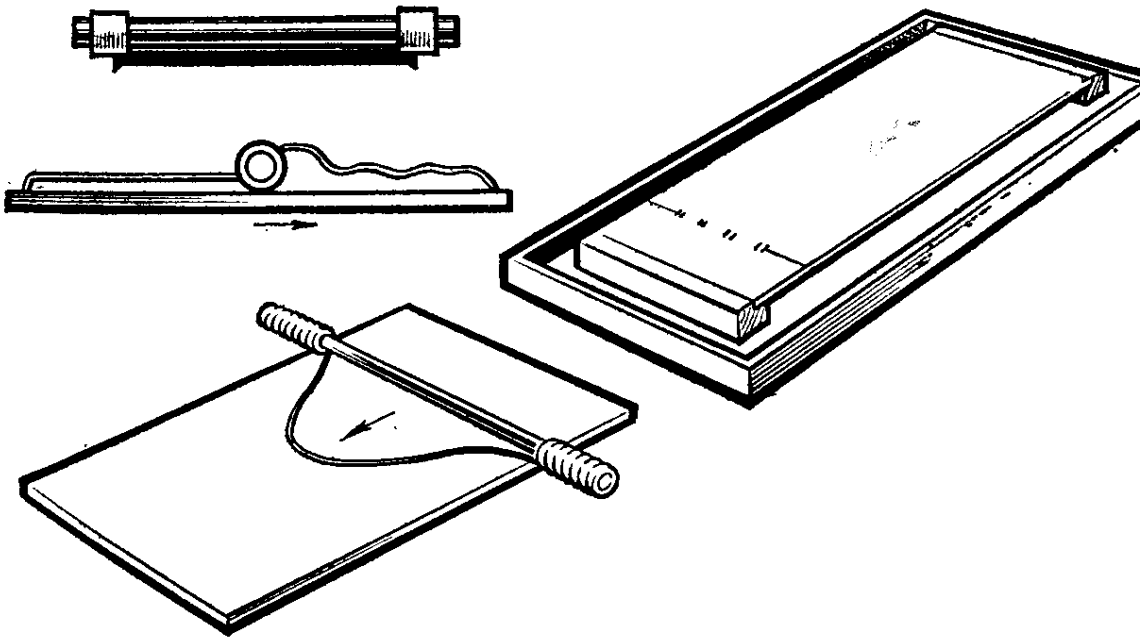
Lab 9

Thin layer chromatography

Thin layer chromatography is an effective method for separating small amounts of substances on a small adsorbent layer and in a short time. Chromatography can be carried out in fixed and non-fixed adsorbent bed. As an adsorbent for the preparation of fixed layers, oxides of magnesium, aluminum, calcium, magnesium carbonate, silica gel are used in a mixture with binders such as calcium sulfate, rice starch and water. To prepare a chromatographic plate with a fixed adsorbent layer, a mixture of adsorbent with a binder (5% by weight of the adsorbent) and water in the form of a slurry is applied to a glass plate (9X12 cm, 13X7 cm). With a special roller (see below), the mixture is evenly rolled into a layer 2 mm thick. Then the plate is dried at HO-120°C. After drying the plate, it should not have cracks.

When working in a thin loose layer, mainly aluminum oxide and silica gel are used as adsorbents. To prepare a thin, loose layer, the same glass plates are used. A layer of adsorbent is poured onto the plate and evenly leveled with a roller, slightly pressing against the glass and removing the excess adsorbent. The roller can be made from a glass rod with a diameter of 8-10 mm and a length somewhat greater than the width of the plate. Circles (up to 1 cm long) are put on the ends of the sticks from a rubber tube or polyvinyl chloride of such thickness that when the adsorbent is rolled, a layer up to 1 mm is formed. The mugs should be at such a distance from the end of the tube that after passing the roller over the plate, strips free from the adsorbent remain on both sides of it. It is possible to make a metal roller with thickened ends. And it should be superimposed on the plate. To fix the plate during the application of the adsorbent, it is convenient to have a special machine (Fig. 21).

On a plate with an adsorbent at a distance of 1.5-2 cm from its edge, a transverse line (start line) is drawn with a stretched thread or wire parallel to the lower edge of the plate. Drops of solutions of the studied substances are applied to it with a capillary at a distance of 1.5-2 cm from each other. Up to 50 μg of the substance can be applied at one point. After applying the sample to the sorbent, the solvent is allowed to evaporate and the plate is placed in an inclined position in a cuvette, on the bottom of which the eluent is poured in a layer of 1–1.5 cm.). The eluent should touch the plate below the start line. Then the cuvette is tightly closed with a lid so that the ratio of solvents (when using a mixture) is not disturbed due to evaporation.



Рнс. 21. Нанесение сорбента на пластнику и станок для пластинок

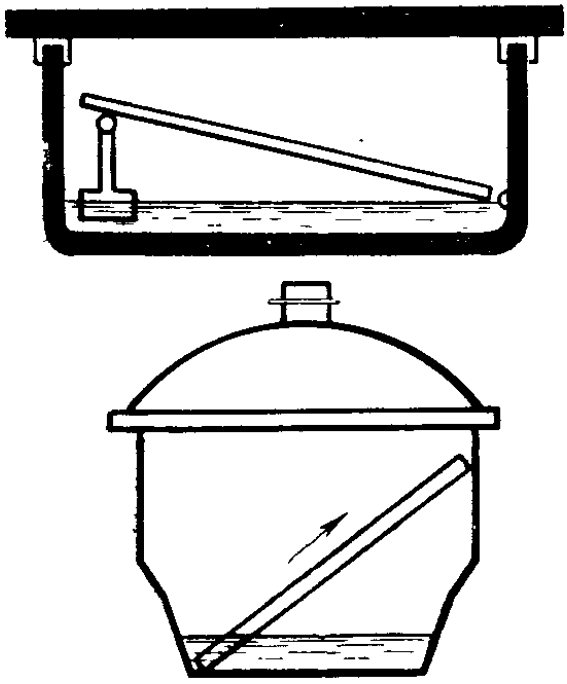


Рис. 22. Хроматографические камеры с пластинкой

When the solvent rises almost to the top of the plate, it is removed and the position of the solvent front is noted. When chromatography of colorless substances after the end of the process, the plate is dried and placed in an atmosphere of an easily adsorbed substance. In this case, the pure sorbent on the plate is colored, and the spots of the substance remain colorless. Sometimes, with such a "manifestation", the chromatograms are colored both by the sorbent and the substances, but the intensity of the color is different. Iodine vapor can serve as a "developer". Before

placing the plate in the container with the "developer", it is necessary that the solvent on the plate has evaporated. Otherwise, iodine dissolves on the plate, and the difference in adsorption is violated. The plate is placed in a vessel with crystalline iodine for 5-10 minutes. Then the plate is removed and left in the air to evaporate the excess iodine.

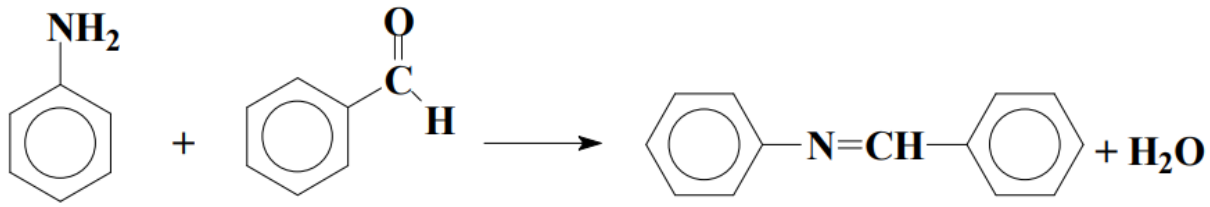
The position of substance spots after chromatography is characterized by Rf values (front ratio) and is calculated by the formula:

$$R_f = \frac{\text{Расстояние от точки старта до середины пятна хромат}}{\text{Расстояние, пройденное растворителем от линии старта}}$$

Rf is a characteristic of each substance, but depends on the quality of the sorbent and eluent. Therefore, the Rf values of the test substance are compared with the "witness" that is applied to the same plate. The "witness" is a supposed pure substance obtained by another method.

Lab 10

Synthesis of benzalaniline



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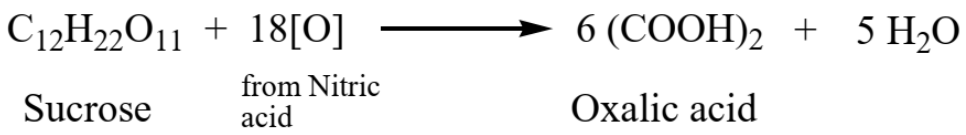
5 ml of freshly distilled benzaldehyde are poured into a 100 ml beaker and 4.5 ml of freshly distilled aniline are added to it with vigorous stirring. The reaction begins immediately, accompanied by the release of heat. After heating stops, the mixture is allowed to stand for 15 minutes, then, with stirring, it is poured into 12 ml of alcohol (ethyl or isopropyl). The solution is kept first for 10 minutes at room temperature, and then for 30 minutes at 0 ° C (in a mixture of water and snow). The crystalline mass is filtered off on a Buechner funnel and dried in air. Recrystallize benzalaniline from 85% ethanol.

Yield 7.5 g (90%), mp 52°C.

Lab 11

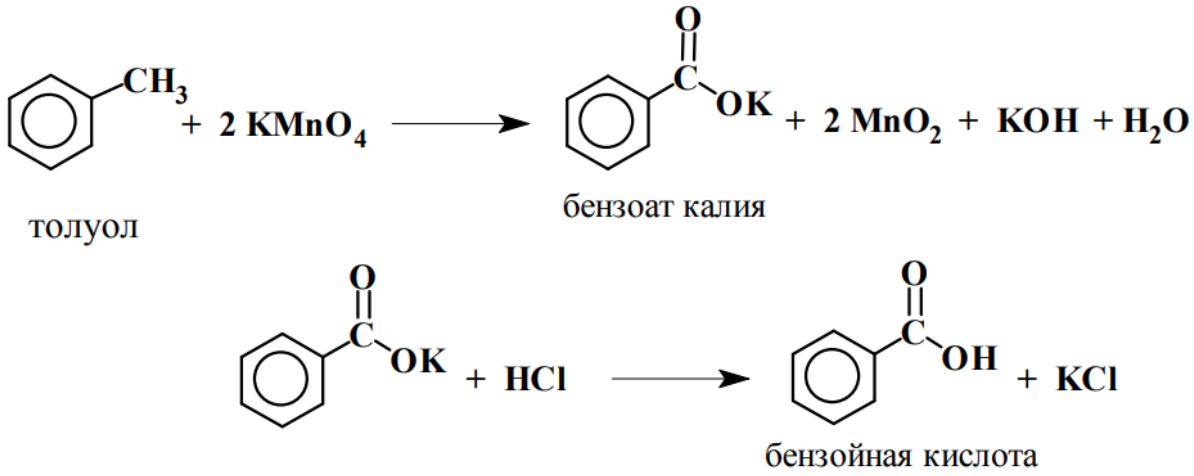
Obtaining oxalic acid



300 g of nitric acid (66%; d=1.41) are placed in a beaker with a capacity of 0.5 l, and then 30 g of sugar and 0.1 g of vanadium pentoxide are added with stirring. After some time, the release of brown oxides of nitrogen begins. Strong heating is avoided by cooling the reaction mixture with water. The solution soon turns green and after a few hours large, colorless oxalic acid crystals stand out. The reaction mixture is left under draft overnight. The next day, the crystals are sucked off through a glass filter, washed with 30 ml of cold water and recrystallized from 30-40 ml of hot water. Yield 25 g, m.p. 103°.

Lab 12

Synthesis of benzoic acid (from toluene)



In a round bottom flask with a capacity of 150 - 200 ml, equipped with a reflux condenser and a stirrer, 1 g of toluene and 3.4 g of potassium permanganate in 75 ml of water are heated on a boiling water bath. Heating is continued for 4 hours until the crimson color disappears (see note). The precipitated manganese dioxide is filtered off on a Buchner funnel and washed twice with boiling water in portions of 10–15 ml. The filtrates are combined, evaporated on a boiling water bath to a volume of 10 - 15 ml and benzoic acid is precipitated from the solution with hydrochloric acid, adding it dropwise. The precipitated benzoic acid is filtered off on a Buchner funnel, washed with a small amount of ice water, dried, and the melting point is determined. Yield: 1 g (64%), mp 121°C.

The end of the oxidation reaction is determined by the disappearance of the crimson color of potassium permanganate. To do this, a drop of the reaction mixture is applied to filter paper. If all the permanganate has reacted, a black spot of MnO2

remains at the place of application of the drop, and the spreading aqueous solution is colorless. Such a test can be carried out only if the permanganate is obviously taken in some deficiency in relation to the oxidizable substance. Otherwise, you can control the presence of the oxidizable substance in the vapors leaving the reaction flask and stop the reaction as soon as it disappears in them. This is done, for example, in the preparation of benzoic acid from benzyl alcohol. The excess of permanganate is removed by adding some reducing agent to the reaction mixture: sodium sulfite or hydrosulfite, ethyl alcohol, formaldehyde, etc. For instance,

$$2\ KMnO_4 + 3\ C_2H_5OH \longrightarrow 3\ CH_3CHO + 2\ MnO_2 + 2\ KOH + 2\ H_2O$$

этиловый спирт ацетальдегид

The acetaldehyde formed during the oxidation of ethyl alcohol does not pollute the main product, benzoic acid, since, having a low boiling point, it escapes from the reaction mixture. Using the example of the last reaction, one can show how organic oxidation-reduction reactions are usually balanced

$$2\ KMnO_4 \longrightarrow 2\ MnO_2 + 2\ KOH + 2\ O \qquad (1)$$

$$CH_3CH_2OH + O \longrightarrow CH_3CHO + H_2O \qquad (2)$$

Half-reaction (1) reflects the process of reduction of permanganate in an alkaline medium. Half-reaction (2) is the process of alcohol oxidation to aldehyde. Multiplying the stoichiometric half-reaction coefficients (2) by three and then adding equations (2) and (1), one obtains the complete equation for the oxidation of ethanol. When carrying out the synthesis of benzoic acid, vigorous stirring is required. As the reaction progresses, a heavy precipitate of manganese dioxide precipitates; therefore, even at slight overheating, shocks of an unevenly boiling mixture are observed, which can lead to cracking of the reaction vessel. To avoid this, it is expedient to put "boilers", for example, fragments of porcelain, into the flask before starting the synthesis. It should be borne in mind that the product of the oxidation of both toluene and benzyl alcohol with potassium permanganate is not free benzoic acid, but its potassium salt, dissolved in water. The free acid is isolated by acidification of an aqueous solution of potassium salt. Benzoic acid, which is slightly soluble in cold water, precipitates. It should be borne in mind that hydrochloric acid is also consumed to neutralize the alkali formed during the reaction. When calculating the amount of hydrochloric acid needed to neutralize the alkali and convert potassium benzoate to free benzoic acid, one can follow the rule: the number of acid equivalents should be equal to the number of moles of KMnO4 taken.

Lab 13

Benzyl alcohol and benzoic acid from benzaldehyde

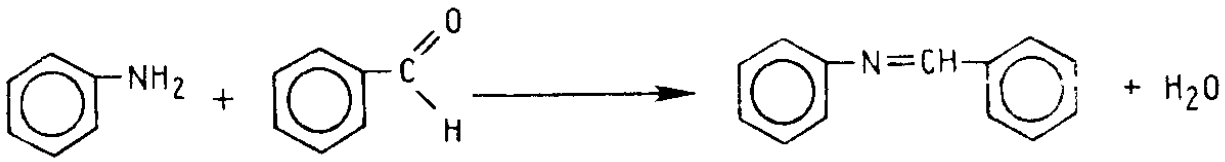
10 ml of freshly distilled benzoic aldehyde and a cooled solution of 9 g of sodium hydroxide in 6 ml of water are placed in a flat-bottomed flask with a capacity of 100 ml. The flask is cooled and shaken vigorously until a stable emulsion is formed. The flask is then stoppered with a side slit and left overnight. The resulting crystalline mass is completely dissolved with a small amount of water. (Water should be added in such an amount that extraction with ether can be carried out; benzyl alcohol partially dissolves in excess water.) The resulting solution is transferred to a separating funnel and benzyl alcohol is extracted with ether 2' times in portions of 10 ml each. The water-alkaline layer is separated and left to obtain benzoic acid.

Benzyl alcohol is extracted from ether extracts. To do this, the combined ether extracts are shaken twice in a separating funnel with 5 ml of a 40% sodium hydrosulfite solution to remove unreacted benzaldehyde. The aqueous layer is then separated and discarded. The ethereal solution in a small separating funnel is washed with an aqueous solution of sodium carbonate (to neutralize sulfurous acid, traces of which may be present in the sodium hydrosulfite solution). The resulting solution was dried with anhydrous sodium sulfate. From the dried solution, the ether is first distilled off on a water bath. Then, replacing the water cooler with an air one, benzyl alcohol is distilled, heating the flask through an asbestos mesh with a smoky burner flame. Collect the fraction at a boiling point of 206°C. Yield of benzyl alcohol 4 g.

The aqueous alkaline solution left to obtain benzoic acid is transferred to a beaker and acidified with concentrated hydrochloric acid. The precipitated benzoic acid is sucked off on a Buchner funnel and recrystallized from boiling water. Yield of benzoic acid 5 g. Melting point 122 °C.

Lab 14

Obtaining benzalaniline



In a glass with a capacity of 100 ml, equipped with a stirrer, pour 10 ml of freshly distilled benzoic aldehyde. Then, with stirring, pour 9 ml of freshly distilled aniline. The reaction begins immediately, accompanied by the release of heat. At the end of the condensation reaction (cessation of heating the mixture), the reaction mass is allowed to settle for 15 minutes. Then, with stirring, the mixture is poured into a glass with 25 ml of 95% alcohol. The solution is left at room temperature for 10 minutes. Then, cool in an ice bath for 30 min. The resulting crystalline mass is sucked off on a Buchner funnel and dried in air. Benzalanilia recrystallized from 85% alcohol. Yield of benzalannelin 15 g.

Benzalaniline is a crystalline substance, it crystallizes from carbon disulfide in the form of yellowish needles. Soluble in alcohol, ether, insoluble in water. Boiling point 300 °C; melting point 52°C.

Lab 15

Obtaining α-nitronaphthalene

Work in a fume hood!

Place 9 ml of concentrated nitric acid in a 100 ml beaker. While cooling in a cold water bath and stirring, 7.2 ml of concentrated sulfuric acid are carefully added. 8.3 g of finely ground naphthalene is added in small portions with stirring to the nitrating mixture at such a rate that the temperature of the reaction mixture does not exceed 50°C. If necessary, the mixture is cooled. If the temperature regime is not observed, by-products are formed in significant quantities: 1,5- and 1,8-dinitronaphthalenes.

After adding the entire amount of naphthalene, the reaction mass is kept for 1 h at 60°C, with constant stirring. Then the reaction mass is poured into a beaker containing 100 ml of cold water. In this case, α-nitronaphthalene solidifies in the form of a cake floating on the surface of the solution. The aqueous acid layer is drained by decantation, and α-nitronaphthalene is boiled for 15 minutes in a beaker with 50 ml of water. Washing of α-nitronaphthalene with boiling water is repeated three times. After each washing, the water is drained by decantation. After the last wash, the aqueous layer should not show an acidic reaction. Molten α-nitronaphthalene, with vigorous stirring, is poured into a glass with 100 ml of cold water, in which it solidifies in the form of reddish-yellow balls. The precipitate is sucked off on a Buchner funnel.

Recrystallization from dilute alcohol is carried out as follows. Dissolve α-nitronaphthalene in a small amount of boiling alcohol in a flask with an air reflux

condenser. Then carefully, while stirring, hot water is added dropwise until a slight turbidity appears. Again, a few drops of hot alcohol are added until the turbidity disappears and the solution is quickly cooled. The precipitated crystals of a-nitronaphthalene are sucked off on a Buchner funnel and dried in air between sheets of filter paper. Yield 10

α-Nitronaphthalene (1-nitronaphthalene) is a yellow crystalline substance, it crystallizes from ethanol in the form of needles. Easily soluble in chloroform, carbon disulfide, diethyl ether, soluble in ethyl alcohol, insoluble in water. Melting point 61.5°C; boiling point 304 °C; d - 1.331.