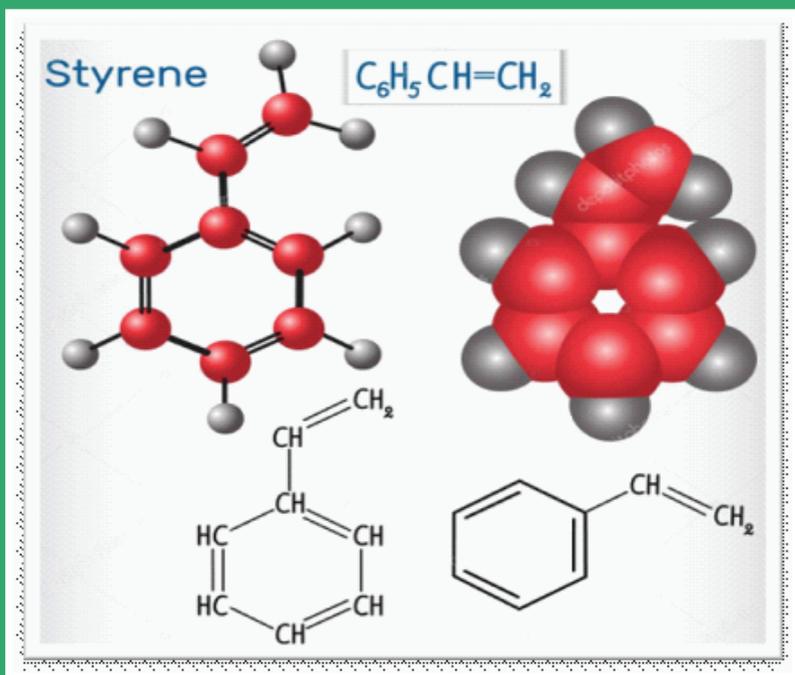


F.A. AMIROV, A.A. MAMMADOVA

CHEMICAL TECHNOLOGY AND RESEARCH METHODS OF MACROMOLECULES



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F.A. AMIROV, A.A. MAMMADOVA

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METHODS OF MACROMOLECULES**

Ankara – 2022



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METHODS OF MACROMOLECULES**

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The textbook is intended for students of the Faculty of Chemical Technology of the Azerbaijan State Oil and Industry University studying in the specialty “Chemical Engineering”, as well as engineers and technicians working in the field of chemistry.

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PREFACE

The production of high-molecular compounds, including oligomers, polymers and elastomers, is one of the areas of science that have been developing rapidly since the mid-twentieth century. This is due to the fact that the technologies for their production are relatively simple, the raw material bases are sufficient, they are cheap, light, easy to form, resistant to various temperatures and aggressive environments and have many indicators. Therefore, the global demand for macromolecules and composites obtained on their basis continues to grow by about 20-25% per year.

Currently, the demand for high-molecular compounds based adhesives, coatings, electrical insulating materials, fibers, construction materials, rubber of various purposes, inhibitors, friction materials, household and medical products, and other composites is evidence of this. For this reason, in accordance with the growing demand for high-molecular compounds, their synthesis, modification and study must meet modern requirements. According to these requirements, first of all, students should be provided with extensive information on the direction of obtaining and studying any macromolecules in laboratory conditions.

In order for students specializing in the specialty of “Chemistry” and “Chemical Engineering” in higher education institutions to be ready for production in some way, they should be able to study their physical-chemical, physical-mechanical and operational indicators along with the synthesis of high-molecular compounds obtained by polymerization, copolymerization, polycondensation, co-polycondensation and polymer analogical conversion reactions in laboratory conditions.

General theoretical information about the appropriate synthesis process is given in the textbook. This was done in

order to help students understand the purpose and essence of the practical work carried out, to choose the right direction for the analysis of the experimental results.

The tutorial consists of two sections. The first section presents laboratory work on the synthesis and modification of high-molecular compounds by various methods. This section provides examples of laboratory work related to polymerization reactions by various methods (in the solid phase, solution, suspension and emulsion), oligomerization and adhesion reactions by polycondensation, as well as polymer analogical conversion reactions.

The second section presents the main methods for studying the obtained high-molecular compounds. The textbook intended for university students studying at the faculty of "Chemical Technology" can be useful for masters, doctoral students, and for employees of research laboratories. The availability of the material base for performing laboratory work specified in the textbook was taken into account at the department. I would like to express my gratitude and appreciation in advance to the readers who expressed their critical opinion and suggestions on the textbook.

LABORATORY WORK № 1

STYRENE PURIFICATION AND BULK POLYMERIZATION

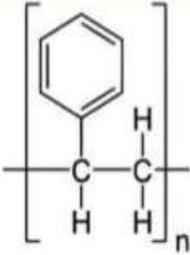
Before polymerization, styrene should be cleaned, because it may contain inhibitors (stabilizers). The inhibitor is incorporated to prevent styrene alone-induced polymerization. In addition to inhibitors, the monomer contains a small amount of ethyl-benzene, chloride, water, sulfur, aldehyde and polymer. Mixtures of styrene affect the quality of the polymer. For example, even if ethylbenzene is small, it reduces the molecular weight of polystyrene and worsens the dielectric properties.

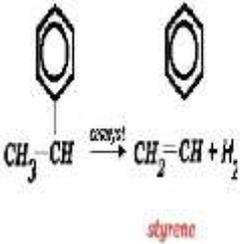
Aldehydes and peroxides are obtained as oxidation of styrene by oxygen. The bubbles in the polystyrene contained in the bulk consist of the gas contained in the monomer, for example, carbon dioxide. Carbon dioxide is obtained by the high degree of peroxide splitting. Sulfur leads to a reduction in the molecular weight of the polymer and deterioration of the light-resistance. Even in small amounts in styrene as a result of prolonged storage, the resulting polymer increases its viscosity slightly, but does not prevent the polymerization.

When used to buy copolymer from styrene, it should not contain polystyrene. When cleaning the styrene and its compounds, it is necessary to reduce the effects of air heat. Monomers (styrene) are purified by distillation in vacuum, because the boiling point of styrene is high (145 °C), and at normal pressure polymerization occurs during distillation. If there are no other impurities in styrene, except for the stabilizer - hydroquinone or tert-butyl pyrocatechol, then it can be cleaned by washing with an alkaline solution.

In this case, the separating pad is washed with 5-10% alkaline solution until the color of the alkaline solution is changed and then washed with distilled water to remove the alkaline (until neutral reaction is received). To free the water

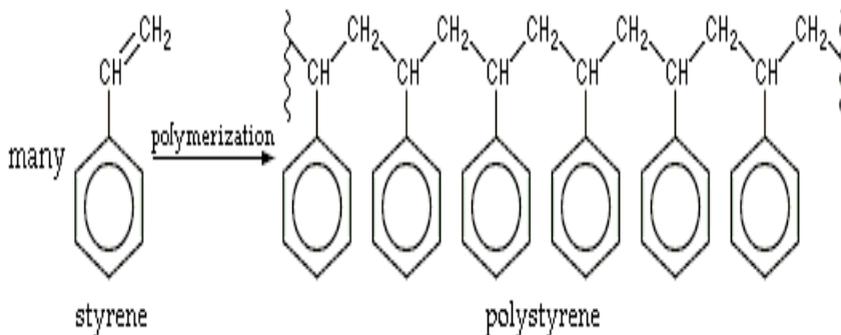
from the styrene it is necessary to freeze it at $-15\text{ }^{\circ}\text{C}$ or drain with calcium chloride.

Polystyrene	
	
Identifiers	
Abbreviations	PS
Properties	
Molecular formula	$(\text{C}_8\text{H}_8)_n$
Density	$0.96\text{--}1.04\text{ g/cm}^3$
Melting point	$-240\text{ }^{\circ}\text{C}$ (decomposes at lower T)
Thermal conductivity	$0.033\text{ W/(m}\cdot\text{K)}$ (foam, $\rho\ 0.05\text{ g/cm}^3$) ^[1]
Refractive index (n_D)	1.6; dielectric constant 2.6 (1 KHz – 1 GHz) ^[2]

Synthesis of polystyrene	
<ol style="list-style-type: none"> 1. Before we can synthesize polystyrene, we must make its monomer styrene 2. Direct catalytic dehydrogenation of ethylbenzene is the main manufacturing course of styrene 	

LABORATORY WORK № 2

POLYMERIZATION OF STYRENE IN BULK



Freshly rolled styrene is filled with thick duvet, half of a solid glass ampoule. The ampoule's mouth is soldered and heated at 170 °C for 3 - 4 hours at the thermostat. They slowly open the bulb after cooling down. Solid, transparent, colorless polystyrene is taken.

Task: 1. Calculate the output of the received polymer (in g and %).

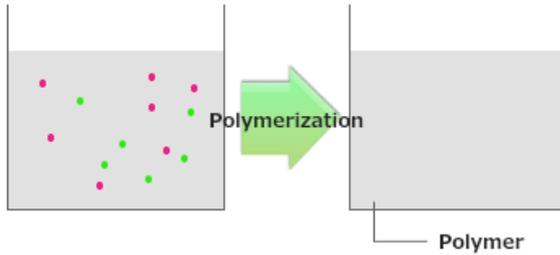
2. Examine solution of organic solvents.

3. The temperature and mol. mass.

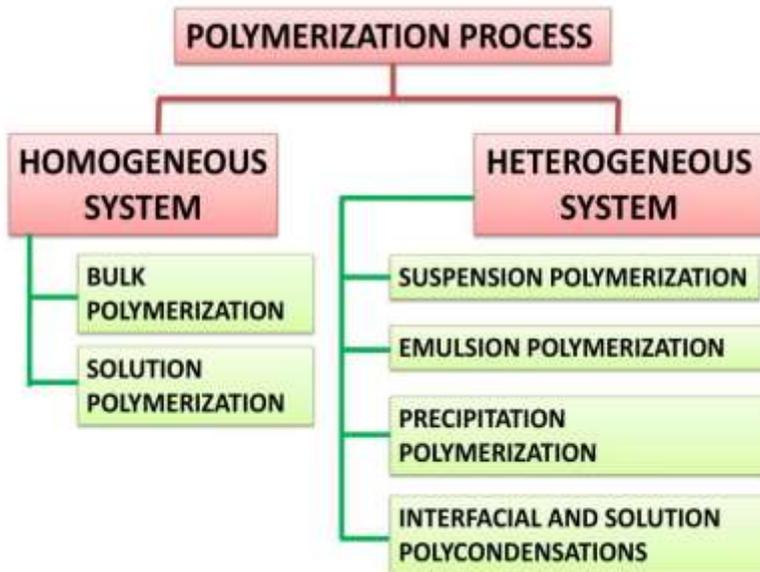
The results obtained from the experiments are transferred to the following table.

Loading, g	Output		Polystyrene characteristics		
			Solution	T _{melting} , °C	Molecular mass
Styrene	g	%			

Bulk Polymerization



- Monomer
- Polymerization Starter



LABORATORY WORK № 3

POLYMERIZATION OF STYRENE IN SOLVENT

Reagents:

Freshly cleaned styrene - 20 g

Benzoyl peroxide - 0.04 g

Benzene - 20 g

Devices: Three - throated flask, mixer, refrigerator and thermometer.

The course of action

20 g of freshly dispersed styrene mixer, refrigerant and thermometer sleeve (Figure 3.1.), added 0.04 g of inhibitor - benzoyl peroxide and the solvent in the indicated amount. Then the mixer is heated at 90-95 °C for 3-4 hours. The resulting reaction product is cooled to room temperature and precipitated by methyl alcohol.

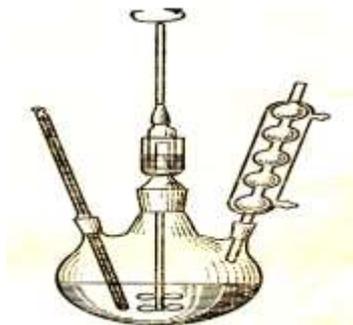


Figure 3.1. *Polymerization of styrene in solvent*

After the precipitate has settled completely, transferred to a porcelain bowl and dried in a vacuum dryer cabinet.

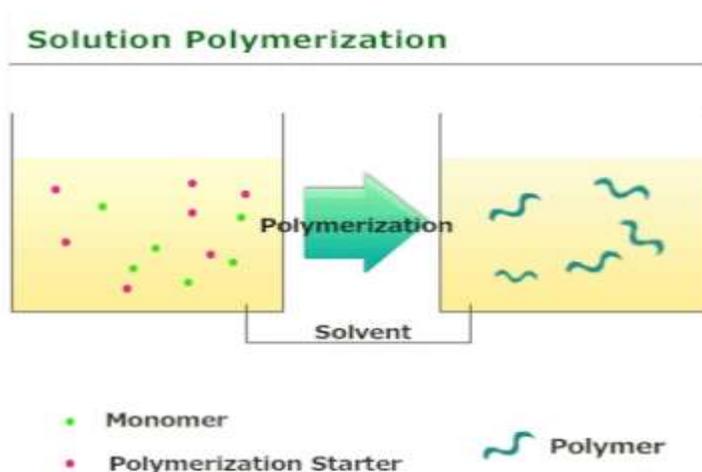
Task

1. Write the reaction of polymerization of styrene.
2. Calculate the output of the reaction product (g or %).

3. Check the reaction product solution.
4. Determine the specificity of the reaction product and calculate the molecular mass.

The results obtained from the experiments are as follows:

Loading, g			Duration of reaction, min	Polystyrene output		Polystyrene characterization	
Monomer	Initiator	Solvent		g	%	T _m , °C	Mol. mass



Pros and cons of some polymerization techniques

	Pros	Cons
Bulk	<ul style="list-style-type: none"> - easy - No contamination 	<ul style="list-style-type: none"> - Difficult to control temp. and heat transfer - High viscosity
Solution	<ul style="list-style-type: none"> -good heat transfer -easy to control reaction temp. -low viscosity -polymer produced may be used directly in the solution form 	<ul style="list-style-type: none"> - Need to use solvent –adding cost -Difficult to eliminate solvent entirely -Solvents sometimes act as chain transfer agent → leading to lower MW polymer
Suspension	<ul style="list-style-type: none"> - Good heat transfer - easy to control reaction temp. - low viscosity - polymer produced may be used directly as polymeric suspension 	<ul style="list-style-type: none"> -Need extra process in washing out suspending agent/contaminants and drying the polymer beads -Polymer beads may stick together and maybe contaminated with suspending agent -Good only for addition polymerization using <u>hydrophobic</u> free radical initiator.
Emulsion	<ul style="list-style-type: none"> - Good heat transfer - easy to control reaction temp. - low viscosity - polymer produced may be used directly as polymer latex 	<ul style="list-style-type: none"> -Need extra process in washing out emulsifier/contaminants and drying -Good only for addition polymerization using <u>hydrophilic</u> initiator.

LABORATORY WORK № 4

EMULSION POLYMERIZATION OF STYRENE

Reagents (mass fraction):

Freshly cleaned styrene - 100

Distilled water - 100

Oleic acid - 1.2

NaOH - 0.13

$(\text{NH}_4)_2\text{S}_2\text{O}_8$ - 0.3

Devices: laboratory reactor, mixer, thermometer, drip funnel, glass beaker, Buchner funnel, Bunsen 's flask and watch glass.

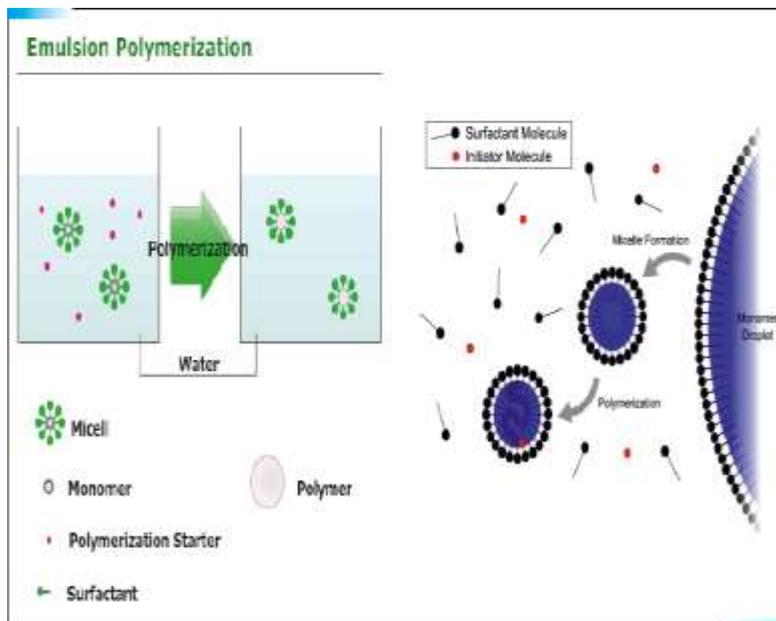
The course of action

For polymerization, 20-25 g of styrene is taken and the remaining ingredients are calculated on the above-mentioned recipe. The calculated amount of oleic acid and NaOH is dissolved in water at 25 °C in the reaction tubes (Figure 4.1.).



Figure 4.1. Emulsion polymerization of styrene

Styrene is added to the emulsion solution while constantly mixing. Monomer and emulsifier is gradually dissolved in a small amount of water after being mixed for 15-



Emulsion polymerization - the monomer is dispersed in the aqueous phase in the form of a homogeneous emulsion.

Advantages a small change in viscosity; ease to regulate heat transfer; sufficiently high reaction rates; ease of processing the finished product.

Disadvantages the need to use additional substances (surfactant, emulsifiers, etc.).

The application is carried out polymerization of vinyl chloride, butadiene, chloroprene, vinyl acetate, acrylates and methacrylates.

LABORATORY WORK № 5

SUSPENSION POLYMERIZATION OF STYRENE

Reagents:

Freshly cleaned styrene - 10 g

Benzoyl peroxide - 0.2 g

Polyvinyl alcohol - 0.34 g

Distilled water - 60 ml

Devices: Test glass, mechanical blender, refrigerator, thermometer and chemical glass.

The course of action

Polyvinyl alcohol is dissolved in distilled water at 60 °C. Then, in a separate tube, benzoyl peroxide is prepared in a styrene solution. The water-soluble polyvinyl alcohol is cooled to room temperature and adds a styrene-exciting blend. The mixer is switched on, the refrigerant is supplied with water and the test bottle is heated up to 80 °C (Figure 5.1.).

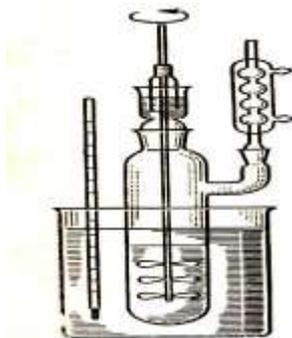


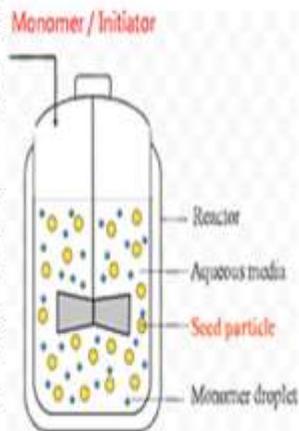
Figure 5.1. Suspension polymerization of styrene

The speed of the mixer is adjusted so that the polystyrene separates into separate grains and does not mix with the general

SUSPENSION POLYMERIZATION

In this the monomer is dispersed in a dispersing medium and polymerization occurs in the monomer droplets suspended in the dispersing medium, water soluble monomer cannot be used unless a salting out procedure is used.

This method is used with free radical polymerization where the initiator is dissolved in the monomer, which is then dispersed in water using an emulsifying agent. Polymerization is initiated in the monomer droplets dispersed in the aqueous medium.



Suspension polymerization (granular) - the monomer is dispersed in water in the form of small droplets, the monomer must be water-soluble.

Advantages control over the length of kinetic chains (narrow molecular mass distribution); efficient heat transfer; ease of separation from water; ease of processing the finished product.

Disadvantages limited use.

The application is used to produce polystyrene granules (from which polystyrene foam is obtained), polystyrene-divinyl benzene granules (for the manufacture of ion-exchange resins)

and poly-vinyl acetate granules (used for further conversion into polyvinyl alcohol).

LABORATORY WORK № 6

OBTAINING A TRANSPARENT NEOLEUCOLITE

Reagents: Freshly distilled phenol – 23.5 g
Formalin, 37% – 20.3 g
Alkali (KOH) - 3% phenol (4% aqueous solution)
Phthalic acid - 1.2% by phenol

Devices: Reverse refrigerator, thermometer, mixer, three-neck flask, water bath, porcelain bowl, test bottles, molds, thermostat and oil bath.

Progress of work

A solution of phenol, formalin and alkali is loaded into a clean, washed dry flask and the mixer is turned on. The flask is heated in a water bath (Figure 6.1.) for 2.5 hours at a temperature of 60⁰ C (an increase in temperature above 60⁰ C leads to a darkening of the color of the solution). The reaction proceeds with the release of heat. The result is a product similar to transparent yellow juice-like oligomer.

The resulting product is transferred to a porcelain bowl, heated in an oil bath and, when the temperature is 60-70⁰C, the above amount of phthalic acid is added to the juice-like to neutralize the alkali.

When the reaction product is heated in the bowl, it is necessary to stir it with a glass stick. Then the salt-containing product is poured into test tubes or special molds in hot form and heated to 80-85⁰C so that it easily separates from the juice-like oligomeric form. It turns out a juice-like oligomer with high mechanical resistance. The impact resistance of the product and its Brinell hardness are determined.

LABORATORY WORK № 7***OBTAINING NOVOLAC-TYPE
PHENOL-FORMALDEHYDE OLIGOMER***

Novolac type oligomer is used in crystalline phenol and 30 - 40% formaldehyde (formalin).

The recipe for the obtaining of novolac type oligomer is given in the following table:

Phenol, mol	Formaldehyde, mol	Catalyst, by % phenol	
		$\rho_{\text{HCl}} = 1190$ kg/m ³	$\rho_{\text{H}_2\text{SO}_4} = 1840$ kg/m ³
1.12	1	1.0	–
1.14	1	–	0.3
1.16	1	1.5	–
1.18	1	–	0.3
1.20	1	1.0	–
1.42	11	1.0	–

Reactives:

Phenol - 30-40 g

Formaldehyde - on prescription

Catalyst on prescription

Devices: Refrigerator, mixer, porcelain case, laboratory reactor and thermometer.

The course of action

In a reactor equipped with mixer and refrigerator, phenol is mixed with formaldehyde at room temperature (Figure 7.1.). Once the phenol is completely soluble, the reactor is placed in a cold water bath and the droplet catalyst is added onto the mixture. Then the water bath is heated.

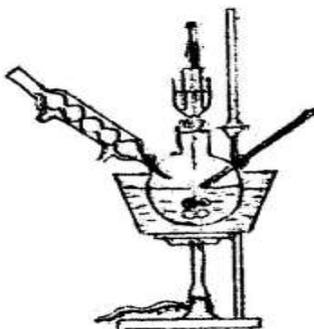


Figure 7.1. Obtaining novolac-type phenol-formaldehyde oligomer

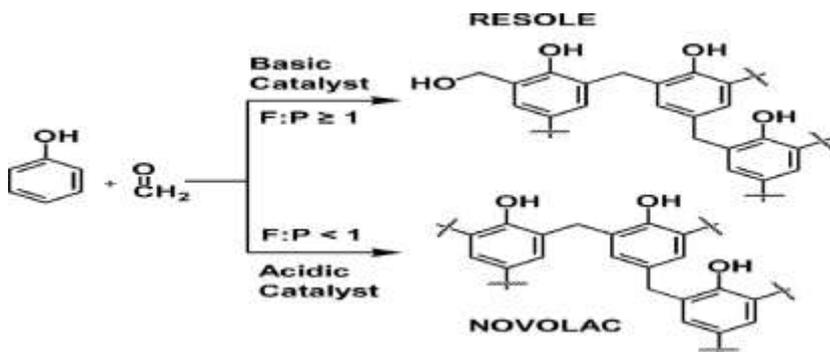
The addition of the catalyst to the heated formaldehyde mixture or rapid heating of the mixture (placed in a hot water bath) may result in the mass foaming and removal from the tube after adding the catalyst. As a catalyst, solid chloride acid or sulfuric acid is used. If the amount of catalyst is small, it is given as a liquid solution, for example, 0.0027 ml of 0.5 N H_2SO_4 catalyst. In this case it is recommended to take 10.2 ml of 0.1N H_2SO_4 (0.0049; 10.300; 0.32 g H_2SO_4) or 2 ml of 0.5N H_2SO_4 (0.0245; 2; 0.0489 g). The end of the reaction is determined by the separation of the reaction mass into two layers. The upper water is removed and the bottom layer of the oligomer poured into the chamber, washed before neutral reaction, and then dried in a vacuum drier cabinet until a constant weight.

Task

1. Write the reactions of the oligomer.
2. Calculate the amount of ingredients collected based on the given recipe by g.
3. Calculate the output of oligomer (g and %).
4. Determine the amount of free phenol in the oligomer.
5. Determine the amount of water in the oligomer.

The results obtained from the practice are summarized in the following table.

Loading			Output		Product characteristics	
Phenol, mole	37% formalin, mole	Catalyst, mole	g	Fenol accordingly, %	Water quantity, ml	Quantity of free phenol, %



#	Thermoplastic resins	Thermosetting resins
1.	They are formed by addition polymerization.	They are formed by condensation polymerization.
2.	They consist of linear long chain polymers.	They consist of three dimensional network structure.
3.	All the polymer chains are held together by weak vanderwaals forces.	All the polymer chains are linked by strong covalent bonds.
4.	They are weak, soft and less brittle.	They are strong, hard and more brittle.
5.	They soften on heating and harden on cooling.	They do not soften on heating.
6.	They can be remoulded.	They cannot be remoulded.
7.	They have low molecular weights.	They have high molecular weights.
8.	They are soluble in organic solvents.	They are insoluble in organic solvents.

LABORATORY WORK № 8

OBTAINING A RESOLE-TYPE PHENOL-FORMALDEHYDE OLIGOMER

The recipe used to obtain a resole-type oligomer is shown in the following table.

Phenol, mol	Formaldehyde (37% formalin), mol	Catalyst, according to phenol %			Boiling point of the reaction mixture
		NH ₄ OH	NaOH	Ba(OH) ₂	
1	1.10	1.5	-	-	30-45
1	1.25	1.5	-	-	30-45
1	1.20	-	1.0	-	100
1	1.25	-	-	1.5	40-60

The course of action

Phenol and formalin are added to laboratory reactor equipped with blender, refrigerator and thermometer and after mixing at room temperature for 15 min., catalyst is added by drops. Then the tube is placed in a water bath and slowly heated up to 95-98 °C. Adding the catalyst to the heated phenol-formaldehyde oligomer, or the rapid blasting of the mixture after the addition of the catalyst causes the mass to foam out of the tube. The catalyst is added as a solution. Approximately 0.5 hours later, the mixture in the flask becomes cloudy and is divided into two upper-water and sub-oligomer layers (Figure 8.1.). The upper layers are removed and the oligomer is washed with water (neutralized) till a neutral reaction and dried in a vacuum dryer at 60-65 °C. Initially, the pressure is 400 - 500 mm Hg column, then it should be 1000 - 1200 mm Hg column. The end of drying is determined by the viscosity and transparency of the oligomer.

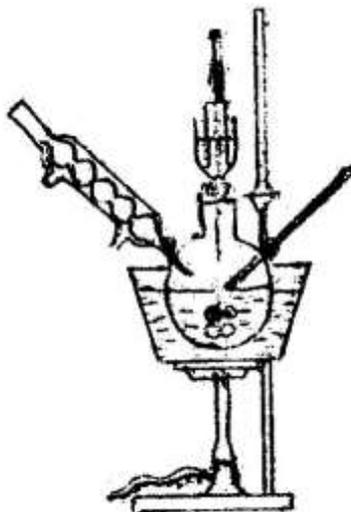


Figure 8.1. Obtaining a resole-type phenol-formaldehyde oligomer

Note: The phenol used for reaction should be clean, formaldehyde (formalin 37 - 40 % solution), ammonia and sodium calcium are taken in as a solution.

Task

1. Write the reactions of the oligomer.
2. Calculate the amount of ingredients taken by the given recipe by g.
3. Calculate the output of oligomer (according to phenol and g in %).
4. Determine the amount of free phenol in the oligomer.
5. Determine the amount of water in the oligomer.

The results obtained from the practice are summarized in the following table.

Loading			Oligo- mer output	Characteristics of the product		
Phe- nol, mol	37% forma- lin, mol	Cata- lyst, mol	g	Fenol accor- dingly ,%	Wa- ter quan- tity, %	Quantity of free phenol, %

Difference in novolac and resole	
Novolac Acid catalysed F:P < 1 Need of cross linkers Liquid in nature More amount of Phenol is present	Resole Base catalysed F:P > 1 No need of cross linkers Solid in nature Less amount of Phenol is present

Phenol-formaldehyde resin is used in the production of plywood, plywood products, chipboard and fiberboard, phenoplastics. One of the most promising areas of application of phenoplastics is shipbuilding. Already, large-sized parts are being created from them and whole hulls of small vessels, life rafts, plastic cabins and superstructures of metal vessels are being built, bulkheads, propellers and deck decking are being manufactured. Plastics are one of the main materials for electrical navigation and radio equipment of ships, ship automation equipment, communications. Phenoplastics are successfully used as decorative and finishing materials and materials for the manufacture of practical things, furniture, lighting fittings, sanitary and electrical equipment.

LABORATORY WORK № 9

PRODUCTION OF CARBAMIDE - FORMALDEHYDE OLIGOMER BY THE "BOILING" METHOD

Reactives:

Carbamide - 60 g

Formalin (40%) - 200 g

25% ammonia water - 54 g

Methyl or ethyl alcohol - 30 ml

Devices: Refrigerator, mixer, laboratory reactor, water bath, thermometer, tube, 250 ml tube, bottle or fluoroplastic beaker (any size).

The course of action

Formalin and ammonia water is added in the amount shown in the laboratory reactor (Figure 9.1.), which is equipped with a refrigerator and mechanical mixer, with a volume of 500 ml. Determine the pH of the mixture. $\text{pH} = 7.6 - 7.8$

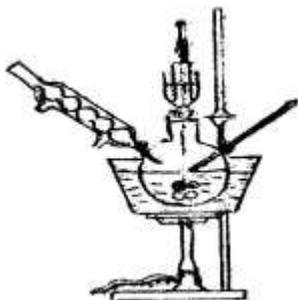


Figure 9.1. Production of carbamide-formaldehyde oligomer by the "boiling" method

If the pH is smaller than the amount shown, it is necessary to add ammonia water into the tube until desirable pH is obtained. Tube is heated with mixture for 30 minutes in a boiling water bath. If iron-hydroxide collapses at this time when

the reaction medium collapses (this occurs when the formalin is not properly clean and contains iron), the solution is filtered through a strain of paper. Then, approximately half of the carbamide is added to the filtered solution, mixed for 30 min. during heating. Then 30 ml of alcohol is added to the tube and the remainder of carbamide is heated and the reaction mixture is heated to a pH of 5.0 to 4.6. Typically, the carbamide is added and the pH value is reached approximately 3 - 4 hours after starting to warm up. The condensing product has a transparent juice appearance. Sometimes the reaction product is blurred, which is due to the presence of non-soluble methylene carbamide in the reaction medium. Methylene carbamide is formed when the reaction medium has a weak sour property. In this case, the reaction product should be filtered through a paper filter.

50 to 70 mm Hg column at a temperature of 70 to 80 °C in the vacuum dryer of the filtered reaction product. At a pressure of 105 - 110 °C on a porcelain on the asbestos clay and evaporate until solid juice is obtained.

The obtained product is poured into glass and fluoroplastic forms, heated in a water bath or thermostat for 2-5 hours at 40 - 50 °C and then heated to 50-55 °C until hardened.

Heating is stopped when the samples are solid and separated from the sides of the forms. As a result, clear transparent oligomer is obtained.

Task

1. Write the reactions of the oligomer.
2. Calculate the output of the reaction product (in g and %).
3. Examine the reaction product soluble in organic solvents.
4. Determine the viscosity, molecular weight and melting point of the reaction product.

The results obtained from the practice are summarized in the following table.

LABORATORY WORK № 10

OBTAINING CARBAMIDE-FORMALDEHYDE OLIGOMER USING “COLD METHOD”

Reactives:

Carbamide - 60 g (1.0 mol)

Formalin (37%) - (1.75 mol)

Urotropine - 3-4 g

Oxalic acid - 0.3 g

Devices: laboratory reactor, refrigerator, mechanical mixer, thermometer, pH-meter, seated tube with volume of 100 ml.

The course of action

The formalin is poured into a laboratory reactor (Figure 10.1.), heated to 30-35 °C and mixing with 20-30 % water solution of urotropine. After 5 minutes the pH of the mixture is checked (with the universal indicator). After the mixture has a pH of 7.4 - 8.2, the carbamide is gradually added at 30 - 35 °C.

Upon completion of the carbamide solution, 20% of oxalic acid is dissolved in water. 10 minutes after delivery of oxalic acid then the pH of the mixture is re-checked.

The reaction is gradually mixed at 30 - 35 °C. When the pH is 6.8 - 7.4, polycondensation ends. If the pH exceeds this value, the polycondensation will continue to the required pH value. When the condensation temperature rises above 35 °C, a solid, non-soluble product is obtained.

Task

1. Write the reaction scheme.
2. Calculate the output of the product (q and%).
3. Check the solubility of the obtained product.

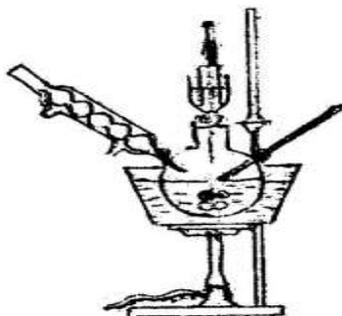
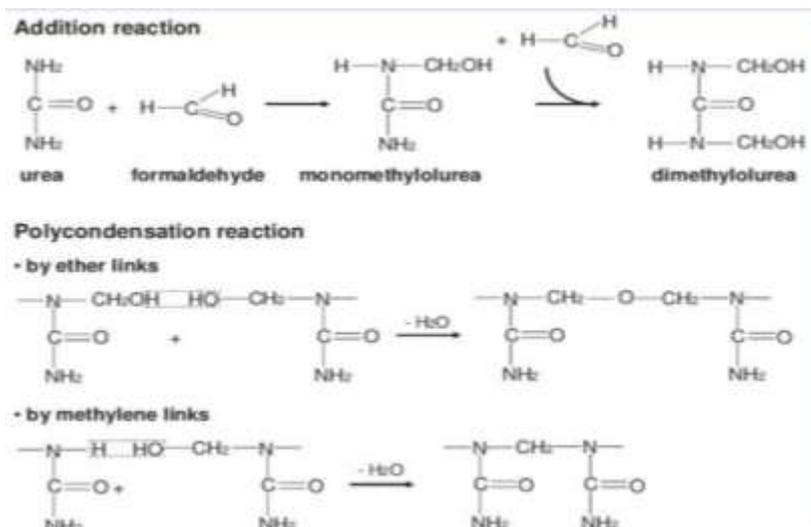


Figure 10.1. “Cold method” of carbamide-formaldehyde oligomer

The results from the experiment are as follows:

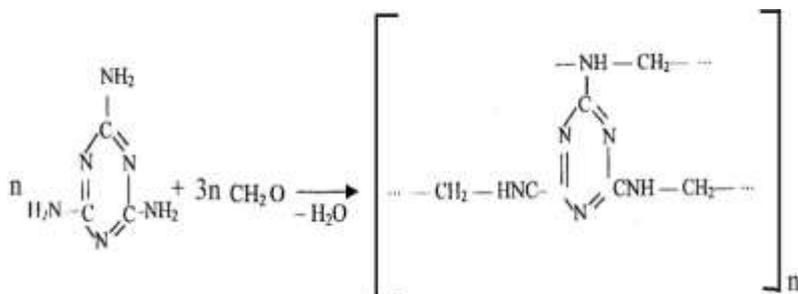
Loading, g			Oligomer output		Extraction time, hour	T _{mel} , °C	pH
Carba-mide	Forma-lin (37%)	Urotropine	g	%			



LABORATORY WORK № 11

OBTAINING MELAMINE-FORMALDEHYDE OLIGOMER

The reaction of the melamine-formaldehyde oligomer is based on the following scheme:



Reactives: Melamine

Formalin (37% water-soluble)

10% NaOH solution

Water

Devices: A four-tube cylinder with a volume of 300 ml, refrigerant, mechanical

mixer, dropping funnel, thermometer, water bath, glass flask with a

closed cap of 500 ml.

The course of action

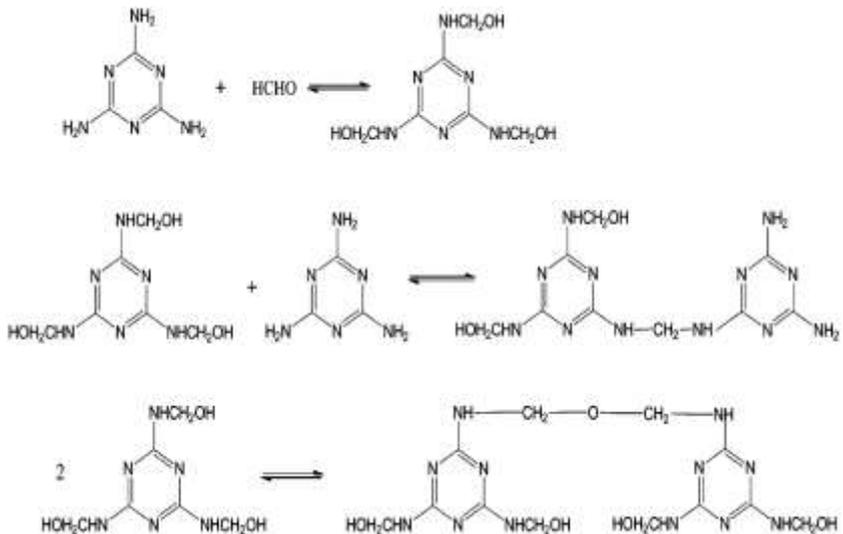
Calculated amount of melamine and formalin poured into the glass flask, the mixer is switched on, the refrigerant is supplied with water, and heated until melamine is completely melted at 60-70 °C in the tube water bath. Then, 10 % NaOH solution is added to the tube through the dropping funnel and the mixture is heated while mixing for 15-20 min at that temperature. Water is extracted, cooled and dried out from the obtained colorless, juice-looking reaction product.

Task

1. Calculate the output of the melamine-formaldehyde oligomer and the amount of separated water.
2. Check the solubility of the oligomer.
3. Determine the melting point of the oligomer.
4. Determine the molecular weight of the oligomer.
5. Make the lacquer on the basis of the obtained oligomer and determine the physical and mechanical properties of the coating.

The results obtained from the experiments are as follows:

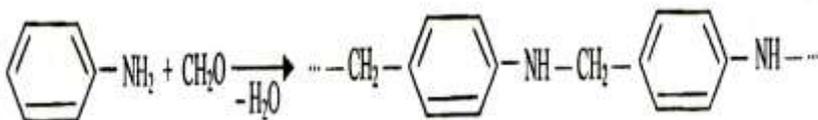
Loading, g			Output		Quantity of separated water		T _{melting} , °C	Mol. mass
Melamine	Formalin (37%)	Catalyst	g	%	ml	%		



LABORATORY WORK № 12

OBTAINING ANILINE-FORMALDEHYDE OLIGOMER

Aniline-formaldehyde oligomer reactions are carried out according to the following scheme:



Reactives:

Fresh aniline - 93 g

Formalin (37% solution of formaldehyde in water) - 91g

Acetic acid (80%) - 15 ml

Methyl alcohol - 100 ml

Devices: 500 ml volumetric tube with stopper, porcelain case, thermometer and metal plaque (9x12 cm).

The course of action

Aniline and formalin are injected into a 500 ml tube. Then the tube is closed with a stopper shaken at room temperature for 1.5 - 2 hours. The resulting white-colored amorphous substance is transferred to the porcelain case and is washed several times with water and then with methyl alcohol. The powdered substance is squeezed and the porcelain 250 ml volume is transferred to the case. 15 ml of acetic acid is added to the powder which is still moist. Then the case is heated on a low heat on asbestos wire netting, gradually increasing to 130-140 °C (the reaction can be foamed as exothermic). After 10 to 20 min. warming, the mixture begins to melt and passes into yellowish or orange color. The obtained transparent oligomeric hardness is relatively low. The oligomer is heated at the same temperature for 2 to 3 hours to increase the hardness and increase

the melting temperature. Then the mass is passed onto the iron plaque and cooled.

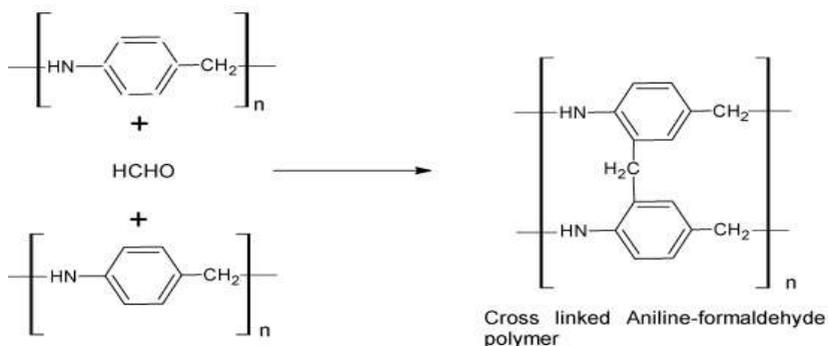
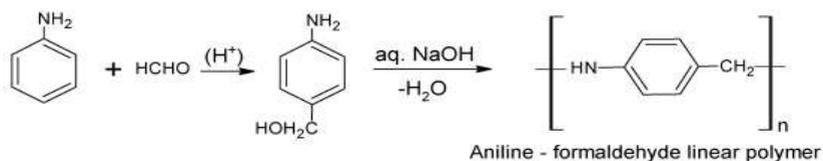
Obtained oligomer is used as a cohesive in preparation of lacquer coatings.

Task

1. Calculate the output of oligomer (in g and%).
2. Determine the solubility and melting point temperature.
3. Determine the molecular weight of the oligomer.

The results obtained from the practice are summarized in the following table.

Loading, g			Output		Quantity of water discharged		T _{mel} , °C	Mol. mass
Phenol	Formalin (37%)	Acetic acid (concentrated)	g	%	ml	%		



LABORATORY WORK № 13

OBTAINING THE RESORCINOL-FORMALDEHYDE OLIGOMER

The thermo-reactive resorcinol-formaldehyde oligomer is quickly selected by its water solubility. Its use is advantageous for phenol because it is not poisonous. This oligomer has the highest fast-hardening properties, so it can be used in medicine to cover the surface of the metal surface and cover the packaging process.

Resorcinol combined with conjugation with a single-phenolic phenol increases the hardening speed of the rubber. The reactor's high reactivity allows it to condense with its catalyzed (acid, alkaline, salt) aldehydes.

Reagents:

Resorcinol - 20 g

Formalin (37% solution of formaldehyde in water) - 20 g

Glycerin - 3 g

Paraformaldehyde - 11.5% (based on oligomer mass)

Devices: three - throated flask, refrigerator, mechanical mixer, thermometer, glass beaker, water bath, porcelain bowl, vacuum drier cabinet and viscometer

The course of action

The components needed for the reaction are placed in a triangular tube (Figure 13.1.). Then heated up to 70-75 °C in the water bath. The mixture should be kept at this temperature (pH = 7.2 ÷ 7.4) until the solid germs are mass. Then, glycerin is added to the reaction mixture, paraformaldehyde is added and poured into molds for a full hardening in a cold place.

The dried oligomer is completely soluble in acetone at ambient temperature to prepare lacquer coating on the basis of the resorcinol-formaldehyde oligomer, and then added the necessary components (filler, fixing, pigment, etc.).

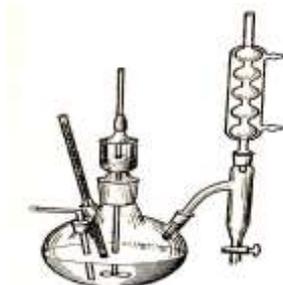


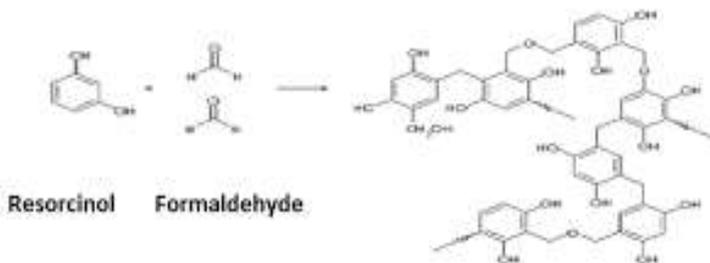
Figure 13.1. Obtaining a resorcinol-formaldehyde oligomer

Task

1. Calculate the output of the oligomer.
2. Check the solubility of the oligomer.
3. Determine the melting point of the oligomer.
4. Calculate the molecular weight of the oligomer.

The results obtained from the practice are summarized in the following table.

Loading, g				Output		T _{mel} , °C	Mol. mass
Phenol	Formalin (37%)	Glycerin	Paraformaldehyde	g	%		



Resorcinol-Formaldehyde (RF) polymer chains

LABORATORY WORK № 14

POLYCONDENSATION OF PHTHALIC ANHYDRIDE WITH ETHYLENE GLYCOL

Reactives:

Phthalic anhydride - 32.2 g

Ethylene glycol - 13.6 g

Devices: porcelain cup, glass funnel, flask, burette, porcelain case.

The course of action

32.2 g of phthalic anhydride and 13.6 g of ethylene glycol are poured into the porcelain cup. The bottle is covered with a bottle cap (Figure 14.1.) and placed in a preheated oil bath. The reaction is carried out at 180-185 °C. In the reaction process, the fluid mass is gradually transformed into a bubble mass with gradually resin and, after 4-5 hours cooling. In order to study the kinetics of the reaction in the polymer synthesis process, take samples from the reaction mixture and determine their acidity number. Examples of melting 0.5; 1; 2; 3; 4 hours later.

The phthalic anhydride in the reaction mixture is fused in the process of crystallization in the cold walls of the cord. Each time you touch the walls of the lock, you need to return the crystals to the glass.

Before taking the sample, all crystals are returned to the glass. Mix the crystals well with the reaction solution and then take the sample. At the end of the synthesis, the polyurethane is determined by the acid number. The reaction is considered to be finished when the sample is easily rubbed at the end of the bottle.

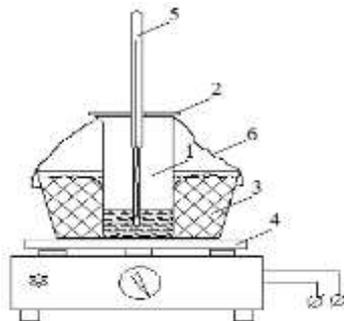


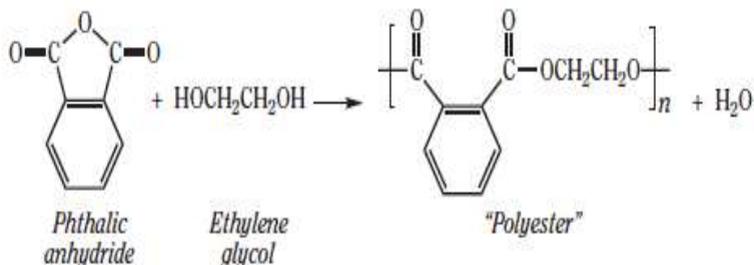
Figure 14.1. Polycondensation of phthalic anhydride with ethylene glycol

Task

1. Write polycondensation reaction of ethylene glycol with phthalic anhydride.
2. Calculate the output of the polymer obtained (in g and%).
3. Determine the molecular weight and melting temperature of the polymer obtained.

The results obtained from the practice are summarized in the following table.

Loading, g		Reaction duration, g	The output of the oligomer		Oligomer characterization	
phthalic anhydride	ethylene glycol		g	%	mol. mass	T _{melting} , °C



LABORATORY WORK № 15

OBTAINING ED-16 EPOXY OLIGOMER

Reagents:

Diphenylolpropane - 20.2 g

Alkali (NaOH) - 7,732 g

Distilled water is 43.3; 6 and 25 ml

Epichlorhydrin - 16.2 g

Devices: Refrigerator, mixer, dropping funnel, thermometer, four-necked tube, separatory funnel, Buchner funnel, test bottle, metal boards, porcelain case and vacuum dryer.

The course of action

5 g dry NaOH is dissolved in 43.3 ml of distilled water, in a tube (Figure 15.1.) with a volume of 250 ml provided with blender, refrigerant, dropping funnel and thermometer. Add 20 g of diphenylolpropane to the prepared alkaline solution. Once the diphenylolpropane has been completely soluble, the amount of epichlorhydrine is gradually added to the blend. Then in a mixed water bath at 45 °C up to 70 °C. After 45 min, the second part of the alkaline solution is added to the tube (soluble 2 g of NaOH in 6 ml of distilled water) and the mixture is heated at 82 °C for 30 minutes. Then the third part of the algae solution is added (0.73 g of NaOH and 25 ml of distilled water), the temperature is maintained at 95 °C and the fever persists for 1 h.

The obtained oligomer is transferred from the tube into chassis, washed several times with neutral reaction with water (the water should be 50 °C). Then, the reaction product taken to remove the water is dried in a drying cupboard.

To obtain solid oligomer, the reaction product should be dried at 130 °C. The dried epoxide oligomer is light-yellow. 5 % hexamethylenediamine is added and heated to 160 °C to dissolve the purchased epoxide oligomer.

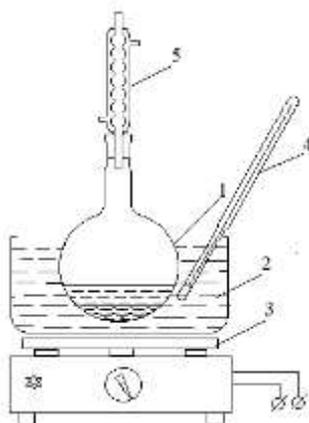


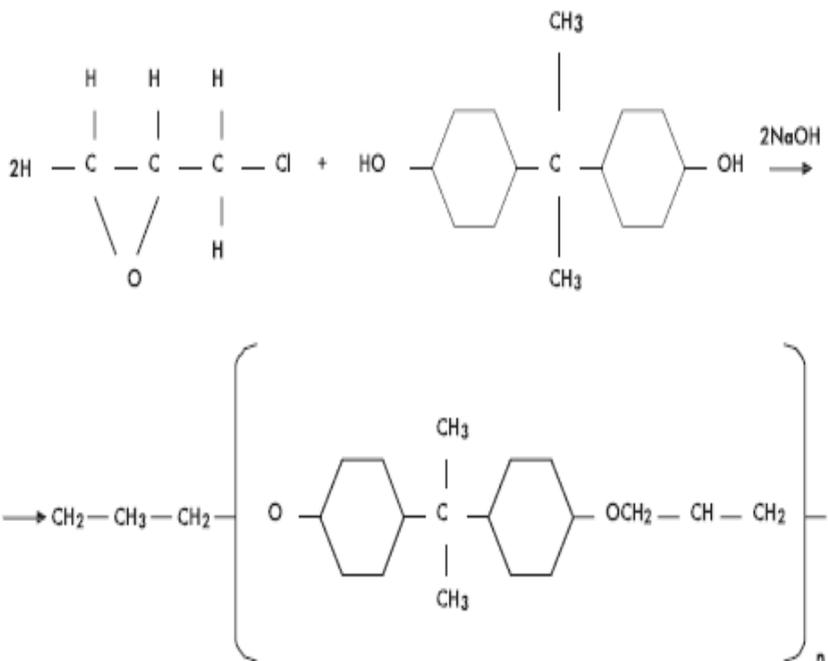
Figure 15.1. Obtaining epoxy oligomer

Task

1. Write the reactions of the oligomer.
2. Calculate the output of the product (g and %).
3. Determine the solubility of poly-epoxide in organic solvents.
4. Determine the characteristic viscosity and molecular mass of poly-epoxide.
5. Write the scheme of reaction between hexamethylenediamine and poly-epoxide.

The results obtained from the practice are summarized in the following table.

Download, g								Output		Polyepoxide Characteristics	
DFP		EXH		Alkali		H ₂ O					
mol	g	mol	g	mol	g	mol	g	g	%	solution	mol. mass



Properties of epoxy oligomers

1. Chemical stability – due to ether linkages.
2. High reactivity – epoxy/hydroxyl groups.
3. Excellent adhesion to surfaces.

4. Outstanding electrical properties – industrial applications.
5. Ability to form co-polymers with various applications.

LABORATORY WORK № 16

ACETYLATION OF CELLULOSE IN THE PRESENCE OF ACETIC ACID (HOMOGENEOUS METHOD)

Reagents:

Cellulose (3-5% moisture) - 3 g

Acetic acid - 35-40 g

Acetic anhydride - 12 g

Sulfuric acid (specific mass 1,84) - 0,8 g

Devices: thick-walled glassware, thermometer, crystallizer, Buchner flask and

watch glass

The course of action

3 g cellulose is placed in a thick-walled glassware with thermometer (Figure 16.1.). The thermometer should not touch the bottom of the bowl.

The cellulose is processed with a cooled acetylene compound of up to 3-4 °C. Composition of the mixture: 1 mass fraction (m.f.) cellulose, 4 (m.f.) acetic anhydride, 4 m.f. acetic acid, 0.1 m.f. sulfuric acid.

Acetylation mode:

3-4 °C - 30 min.

4-8 °C - 30 min.

8-12 °C - 30 min.

12-20 °C - 30 min.

20-25 °C - 30 min.

In the latter case, acetylene is continued until the fiber is completely soluble.

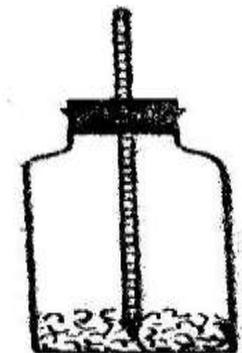


Figure 16.1. Acetylation of cellulose in the presence of acetic acid

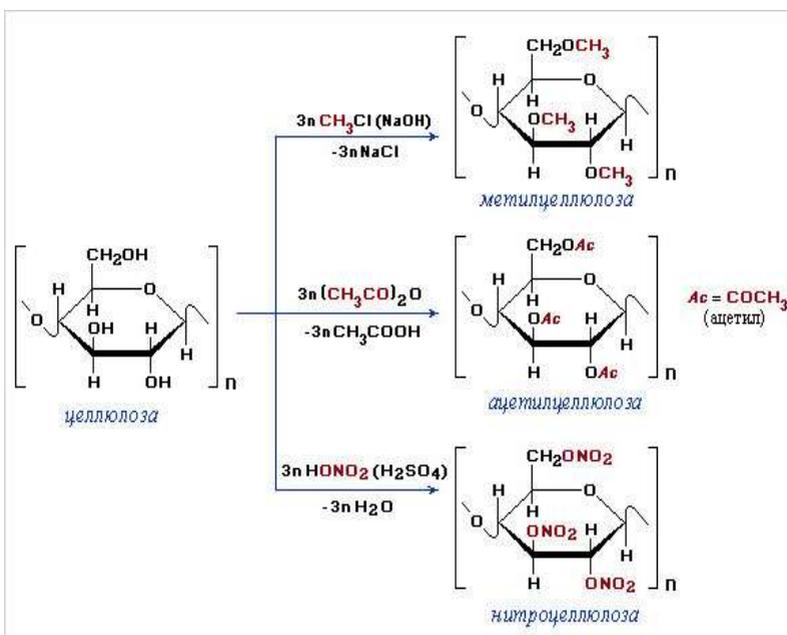
The reaction mixture is periodically mixed. After acetylation is completed, the reaction mass is rinsed with 25 - 30 ml of glacial acetic acid. In order to dissolve acetyl-cellulose, pour a solution of acetic acid (mixing) into the reaction mixture. The precipitated acetyl-cellulose is dried with warm (40-50 °C) water (check with methyl orange) and dried at a constant weight of 80-90 °C to neutral reaction.

Task

1. Write the acetylene reaction.
2. Calculate the composition of the acetylene mixture, taking into account water in cellulose and acetic acid.
3. Calculate the molar ratio of the reaction mixture $(\text{CH}_3\text{CO})_2\text{O} : \text{C}_6\text{H}_7\text{O}_2(\text{OH})_3$
4. Calculate the output of the substance (g or %).
5. Examine the substance to be soluble in organic solvents.

The results obtained from the experiments are as follows:

Check, g			Output		CH ₃ COO group % of the amount	Solution
Cellulose	Acetic acid (concentrated)	Acetic anhydride	%	g		



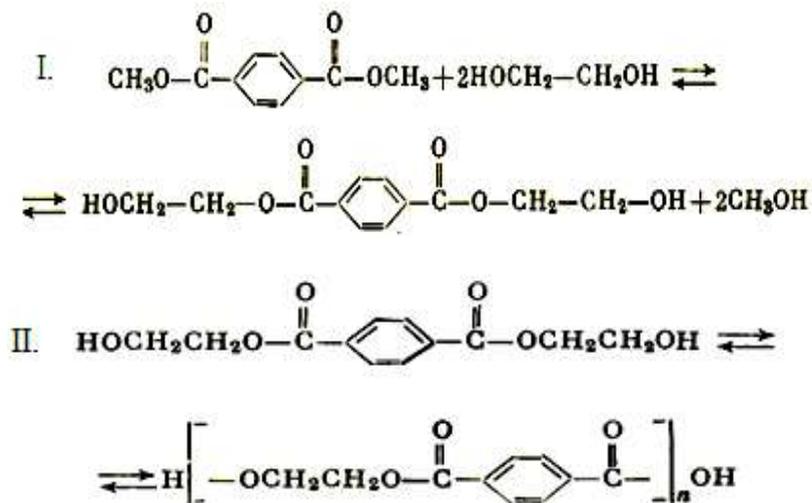
Difference between triacetate and acetate

Triacetate	Acetate or di-acetate
Contain 3 acetate groups.	Contain 2 acetate groups.
No OH groups is present.	Only one OH group is present.
More crystalline.	Less crystalline.
High melting point.	Low melting point.
It is not hydrophilic.	It is hydrophilic.
It is called primary acetate.	It is called secondary acetate.

LABORATORY WORK № 17

PREPARATION AND CURING OF UNSATURATED POLYESTERS

Polyesters are synthesized in 2 stages:



Recipe for unsaturated polyester oligomers:

Reagents	Recipes, mole		
	#1	#2	#3
Ethylene Glycol	1.1	1.1	1.1
Maleic anhydride	0.6	0.6	0.5
Phthalic anhydride	0.2	0.2	0.5
Adipic acid	0.2	-	-

The calculation is carried out for 0.4 mol of ethylene glycol, based on which the number of other components is found.

Devices: A four-neck flask, a mechanical mixer, an inert gas supply tube, sampling tubes, a burette, a pipette, a glass, a thermometer, a return refrigerator, an oil or air bath.

Progress of work

The calculated amount of reagents and hydroquinone up to 0.02% by weight of polyester is placed in a four-necked flask. The flask is heated in an oil or air bath (Figure 17.1.).

When the temperature of the bath reaches 100⁰C, an inert gas is fed into the partition. The gas supply rate is adjusted so that 5 - 6 bubbles per second pass through an intermediate container filled with glycol. Polycondensation is carried out at a temperature of 195 – 200 ⁰C. Increasing the temperature of the mixture from room temperature to 200⁰C for 40 - 60 minutes . this is achieved by heating inside. The depth of the polycondensation process is determined by the number of acidity. To determine the number of acidity after 0.5; 1; 2 and 3 hours, samples of 0.2 - 0.3 g are taken from the mixture and placed in pre-weighed test tubes.

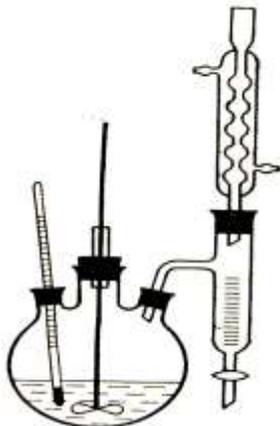
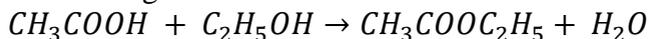


Figure 17.1. Obtaining polyesters

LABORATORY WORK № 18**ESTERIFICATION OF POLYVINYL ALCOHOL
WITH ACETIC ANHYDRIDE**

Esterification of polyvinyl alcohol with acetic anhydride follows the following scheme:



Reagents: Polyvinyl alcohol – 5 g
Acetic anhydride - 30-40 g
Sodium acetate (anhydrous) - 5 g
Inert gas (cylinder)

Devices: A round-bottomed flask with a volume of 100 ml, a reverse refrigerator, a water bath and a glass with a volume of 500 ml.

Progress of work

The above amount of polyvinyl alcohol, acetic anhydride and anhydrous sodium acetate is placed in a round-shaped flask (Figure 18.1.).

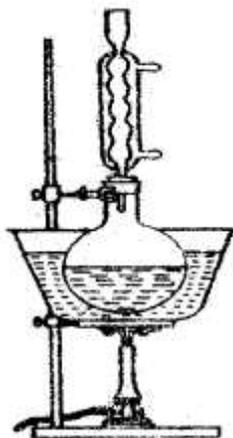


Figure 18.1. Esterification of polyvinyl alcohol with acetic anhydride

Then an inert gas is first released into the mixture in the flask, then 1.5 - 2 hours in a water bath, and then heated over low heat until the polymer is completely dissolved on an asbestos mesh (the inert gas is supplied from a tube located inside the reverse refrigerator).

The resulting polyvinyl acetate solution is poured into hot water, stirred and washed until a neutral reaction (checked by methyl-orange). For complete release from acetic acid, the resulting polyvinyl acetate is dissolved in alcohol or acetone, precipitated with water. The precipitate is washed off until a neutral reaction. The resulting polymer is dried in a vacuum drying cabinet until a constant weight is reached at 70°C.

Task

1. Write a diagram of the course of the reaction.
2. It is necessary to calculate the yield (in g and%) of the resulting polymer.
3. Determine the molecular weight of the resulting polymer by osmometric method.
4. Determine the characteristic viscosity of the resulting polymer.

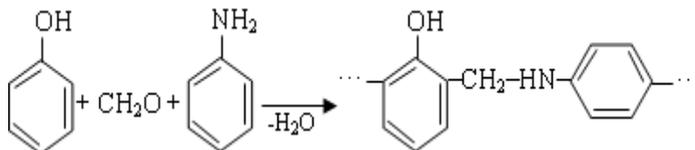
The obtained experimental results are transferred to the following table.

Loading, g			Output		Characteristics of the resulting polymer	
PVA	Acetic anhydride	Sodium acetate	g	%	$[\eta]$	mol. mass

LABORATORY WORK № 19

OBTAINING OF ANILINE-PHENOL-FORMALDEHYDE OLIGOMER

The reaction of obtaining an aniline-phenol-formaldehyde oligomer according to the following scheme:



Reagents: Aniline - 50 g
 Phenol - 20 g
 Formalin (37% formaldehyde solution in water)
 – 45 ml
 Catalyst - 0.05 g

Devices: Flask for polymer production in vacuum,
 refrigerant, vacuum pump, receiver.

Progress of work

Aniline, phenol, and formalin are poured into the polycondensation reactor (Figure 19.1.) in vacuum and mixed. The temperature is gradually increased to 150 °C and at this temperature is continuously mixed, resulting in an oligomer-like substance.

First, the water obtained as a result of the reaction is removed, then the condensation product (oligomer) is separated. The paintwork is prepared from a 50 % solution of an oligomer in a mixture of alcohol or benzene (1:1). To obtain the target compositions, various components are added to the finished paintwork.

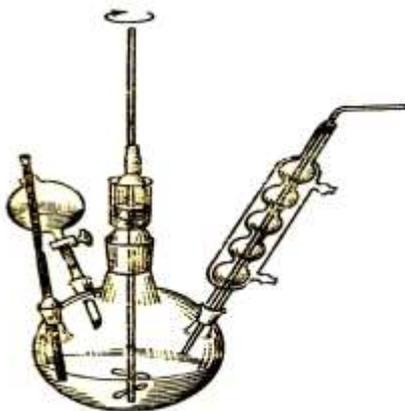


Figure 19.1. Obtaining of aniline-phenol-formaldehyde oligomer

Task

1. Calculate the output of the resulting oligomer.
2. The solubility of the oligomer should be checked.
3. It is necessary to determine the melting point of the oligomer and the molecular mass.
4. Determine the physical and mechanical properties of the finished paintwork.

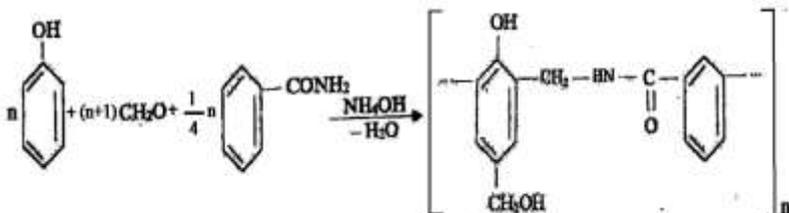
The obtained experimental results are transferred to the following table.

Loading, g			Output		Melting point, °C	Mol. mass
Phenol	Formalin	Aniline	g	%		

LABORATORY WORK № 20

OBTAINING OF A PHENOL-FORMALDEHYDE OLIGOMER MODIFIED WITH BENZAMIDE

The reaction of obtaining a phenol-formaldehyde oligomer modified with benzamide according to the following scheme:



Reagents: Phenol - 108.1 g
 Formalin (37% formaldehyde solution in water) - 43.5 g.
 25% ammonia water - 5% (depending on the amount of phenol)
 Benzamide - 44.1 g

Devices: A 300 ml four-neck flask, a reverse refrigerator, a mechanical blender, a drip funnel, a thermometer, a water bath and a porcelain bowl.

Progress of work

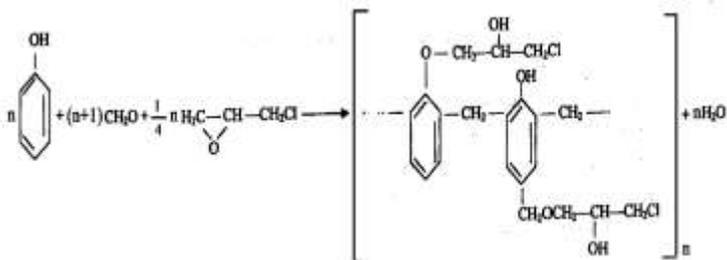
Phenol and formalin are poured into a four-necked flask (Figure 20.1.). Ammonia water is added to the reaction mixture until the pH of the medium becomes 8 - 9.

Phenol and formalin are mixed at 70 °C in the presence of a catalyst for 20 minutes. Then benzamide is added to the reaction mixture in an amount calculated in portions. By shaking the temperature of the reaction mass in the range of 90-100 °C, the polycondensation process continues until the color of the

LABORATORY WORK № 21

OBTAINING OF A PHENOL-FORMALDEHYDE OLIGOMER MODIFIED WITH EPICHLOROHYDRIN

The reaction of obtaining a phenol-formaldehyde oligomer modified with epichlorohydrin according to the following scheme:



Reagents: Phenol - 108.1 g
 Formalin (37% formaldehyde solution in water) – 43.5 g
 25% ammonia water-5% (depending on the amount of phenol)
 Epichlorohydrin - 18.4 g

Devices: A three-throated flask, a reverse refrigerator, a mechanical mixer, a thermometer, a drip funnel, a water bath, a porcelain bowl and a vacuum dryer cabinet.

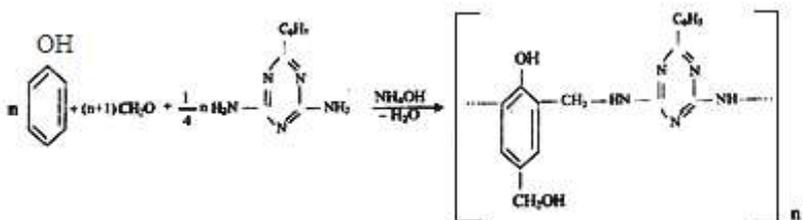
Progress of work

Phenol and formalin are poured into a three-necked flask (Figure 21.1.). 25% ammonia water is added to the reaction mixture until the pH of the medium becomes = $8 \div 9$.

LABORATORY WORK № 22

OBTAINING OF A PHENOL-FORMALDEHYDE OLIGOMER MODIFIED WITH BENZOGUANAMINE

The reaction of obtaining a phenol-formaldehyde oligomer modified with benzoguanamine according to the following scheme:



Reagents: Phenol - 108 g
 Formalin (37% formaldehyde solution in water) - 43.5 g
 Benzoguanamine - 56 g
 25% ammonia solution in water - 5%

Devices: A 300 ml four-neck flask, a reverse refrigerator, a mechanical mixer, a drip funnel, a thermometer, a water bath.

Progress of work

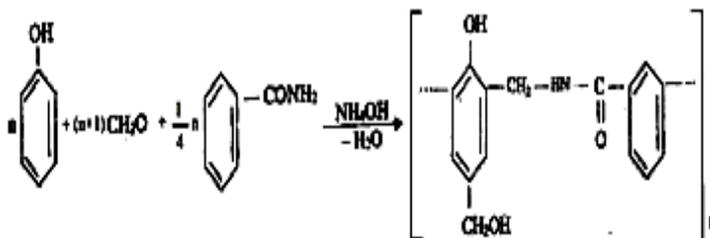
Phenol and formalin are poured into a four-necked flask (Figure 22.1.).

25% ammonia water is added to the reaction mixture until the pH of the medium becomes $= 8 \div 9$. Phenol and formalin are mixed at 70°C in the presence of a catalyst for 20 minutes, then benzoguanamine is added to the reaction mixture in an amount calculated in portions.

LABORATORY WORK № 23

OBTAINING OF A PHENOL-FORMALDEHYDE OLIGOMER MODIFIED WITH BENZYLAMINE

The reaction of obtaining a phenol-formaldehyde oligomer modified with benzylamine according to the following scheme:



Reagents: Phenol - 108 g
 Formalin (37% formaldehyde solution in water) - 43.5 g
 Benzylamine - 42 g
 25% ammonia solution in water - 5%

Devices: A three-neck flask, a reverse refrigerator, a mechanical mixer, a drip funnel, a porcelain bowl, a glass beaker, a vacuum drying cabinet.

Progress of work

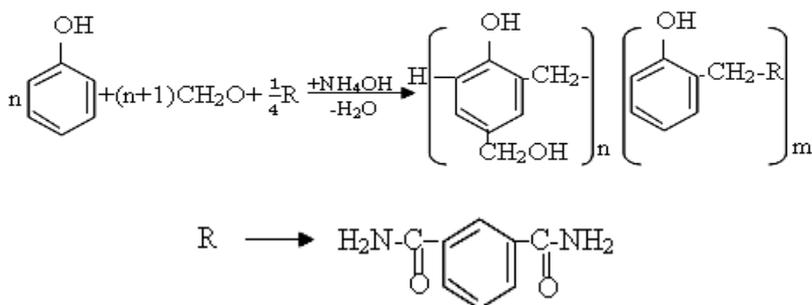
Phenol and formalin are poured into a three-necked flask (Figure 23.1). 25% of ammonia water is added until the reaction medium becomes pH = 8 ÷ 9. Phenol and formalin are mixed at 65 °C and for 15 minutes. Then the calculated amount of benzylamine is gradually added from the drip funnel.

The reaction mass is mixed at 90-95 °C for 1.0-1.5 hours. As a result, the reaction mass is divided into two parts (oligomer and water). The oligomeric layer is separated, washed and dried in a vacuum drying cabinet until a stable weight is obtained.

LABORATORY WORK № 24

OBTAINING OF A PHENOL-FORMALDEHYDE OLIGOMER MODIFIED WITH TEREPHTHALDIAMIDE

The reaction of obtaining a modified phenol - formaldehyde oligomer of terephthaldiamidlal according to the following scheme:



$$m+n=3 \div 6; \quad n=2 \div 4$$

Reagents:

Phenol - 108 g
 Formalin (37% formaldehyde solution in water) - 43.5 g
 Terephthaldiamide – 24.75 g
 25% ammonia solution in water-5%

Devices:

Three-neck flask, reverse refrigerator, mechanical mixer, drip funnel, porcelain bowl, glass beaker, vacuum drying cabinet

Progress of work

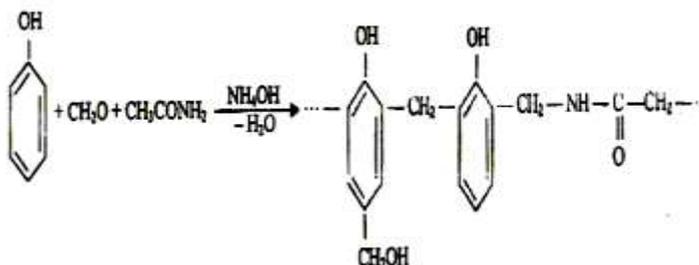
Phenol and formalin are poured into a three-necked flask (Figure 24.1.). 25% ammonia water is added to the reaction mixture until the pH of the medium becomes = 8 ÷ 9.

Phenol and formalin are mixed in 70 °C and in the presence of a catalyst for 20 minutes, then a portion-calculated

LABORATORY WORK № 25

OBTAINING OF PHENOL-FORMALDEHYDE OLIGOMER MODIFIED WITH ACETAMIDE

The reaction of obtaining a phenol-formaldehyde oligomer modified with acetamide according to the following scheme:



Reagents: Phenol - 108.1 g
 Formalin (40%) - 43.5 g
 25% ammonia water - 5 %
 Acetamide - 24.5 g

Devices: A 300 ml four-neck flask, a reverse refrigerator, a mechanical blender, a drip funnel, a porcelain bowl, a thermometer and a water bath.

Progress of work

Phenol and formalin are poured into a four-necked flask (Figure 25.1.). NH_4OH is added to the reaction mixture until the pH of the medium becomes 8-9.

Phenol and formalin are mixed at 75°C in the presence of a catalyst for 30 minutes. Then acetamide is added to the reaction mixture in an amount calculated in portions. At a reaction mass temperature of $90\text{--}95^\circ\text{C}$, the polycondensation

LABORATORY WORK № 26

DETERMINATION OF SOLUBILITY OF POLYMERS

Qualitative determination of the solution

5 ml of solvent is poured into a high-molecular compound 0.5 g of well-glued test bottle. Then, the test bottle is kept at room temperature for 2 hours, and the test bottle is repeated several times. Two hours later, the change in the test bottle is noted. The polymer may be completely resolved or inflated to a certain degree. Gradually the soluble or swelling polymer is re-tested by heating. To do this, the test bottle is combined with a refrigerator in the water bath for 0.5 hour, and then the changes are checked again.

Quantitative determination of the solution

Often, it is only necessary to clarify the solution of the polymer investigated. Solution ability can be determined by the amount of precipitator that causes the polymer solution to leak. The more sedimentary properties required for the formation of nausea, the more decisive it is, the better its solubility.

The ratio of the sediment volume to the solvent volume characterizes quantitative solubility of the polymer.

A well-scrubbed polymer and 3 ml of resin soluble are added to the convex tube at 0.05 to 0.1 g. Subsequently, the sediment is dripped down by mixing it without interruption until the nausea is removed from the burette.

Physical properties of polymers contain: density, molecular weight, degree of polymerization, molar volume, crystallinity of material, specific gravity, water absorption, water vapor permeability, viscosity, melting point, cloud point, pour point (transfer point), oil absorption of pigments.

Solvent and precipitators for different polymers and oligomers

<i>Polymer and oligomeres</i>	<i>Solvent</i>	<i>Precipitator</i>
Phenol-formaldehyde	Ethyl alcohol, acetone dioxane, tetrahydrofuran, chloroform	Water
Carbamide-formaldehyde	Water, acetic acid	Chloride acid solution
Melamine-formaldehyde	Water, acetic acid	Concentrated chloride or sulfuric acid
Aniline-formaldehyde	Acetone, cyclohexanone, quaternary carbon, chloroform	Water
Polyamide	Cresol, dimethyl-formamide	Ethyl or methyl alcohol
Epoxide	Toluene, alcohol, acetone	Water
Polyester	Acetone	Water, ethyl or methyl alcohol
Polyvinylchloride	Dichloroethane	Ethyl alcohol

When analyzing high molecular compounds, their solubility is determined first. Solution is two quantitative and qualitative:

a) Quantitative determination of the solution.

Sometimes, it is required to investigate the solubility of the polymer investigated, as well as how to solve it in several solvents. The solubility of the polymer solution can be determined by the amount of precipitator that causes the nausea to form. The greater the solubility required for the formation of nausea, the more decisive it is, the better its solubility. The ratio of the sediment to the solvent volume characterizes the quantitative resolution of the polymer. The polymer and 3 ml of the investigated solvent are added to the investigated polymer.

Then the sediment is dropped (interrupted without interruption) until it is taken away from the burette.

b) Qualitative determination of the solution.

1 g of polymer is poured into 10 ml of solvent into the mouthpiece test bottle. Then the test bottle is kept at room temperature for 2-4 hours. Several times the test bottle stops. During this time, the change in the polymer is noted. If the degree of solubility (complete, partial, non-soluble, etc.) is determined by changing the color of the polymer or solvent, then the solubility of the polymer is re-checked by heating the mixture at a certain temperature. For this purpose, 1 g of polymer and 10 mL solvent will be placed in a tube containing 20 ml of volume equipped with a mixed refrigerator. The tube is heated in a water bath for 20 minutes, then the change is checked again. This control gives a preliminary conclusion about which class the polymer belongs.

Then the next experiment is conducted to clarify whether the polymer belongs to thermoplastic or thermosetting.

The smallest part of the polymer is combined with platinum wire and stored for 5-10 minutes in the gas flames. The color of the flame is well-marked in combustion, as well as the melting of combustion products (Figure 26.1.).



Figure 26.1. Solubility factors

LABORATORY WORK № 27

DETERMINATION OF INFLATABILITY DEGREE OF POLYMERS

Polymer solution is in two stages. The first stage of the procedure consists of macromolecule swelling.

The diffusion of the inflammatory solvent molecule into the polymer occurs as a result of the diffusion. During the inflammation, the polymer absorbs the lower-soluble soluble, resulting in the mass of the polymer and changes its mechanical properties. The volume of the polymer during the infection is increased by 1000-1500%. The interactions between the molecules are then weakened in the polymer, and macromolecules break off the basic mass of the substance and diffuse into the solvent. The result is a solution. Swelling of the polymer is measured by the inflow rate:

$$\alpha = \frac{m - m_0}{m_0}$$

Here, m_0 and m are the polymer sample, respectively, before and after the swelling.

The inflammation rate is equal to the amount of liquid (q) absorbed by the polymer of 1 gram. The given time (τ) is assigned to α , and the kinetic curve characterizing the inflating process is established (Figure 27.1.).

This dependence can be expressed in the analytic form by the following differential equation:

$$\frac{d\alpha}{d\tau} = k(\alpha_{max} - d\tau)$$

Here, α_{\max} - maximum inflatable rate; $d\tau$ - inflatable rate; k - the constant of the inflatable velocity (depends on the nature of the polymer and the solvent and the effect of the temperature).

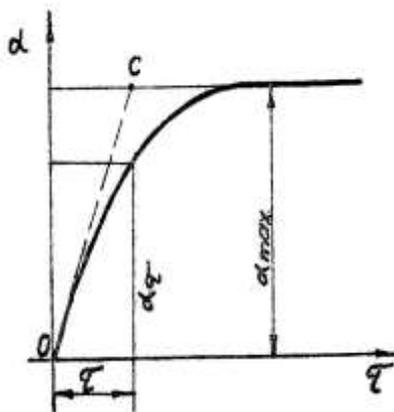


Figure 27.1. The time dependence of the inflammation rate

The course of action

Exact polymer samples are placed in a soluble container within a variety of time periods. After each time, the polymer sample is removed, the solvent is filtered and drawn. Then the inflatable rate (α) is calculated for all τ times, the kinetic curvature of the swelling is established, and the inflammation rate α_{\max} is found.

The prices received from the practice are summarized below.

The investigated polymer	Solution	m_0 , g	m , g	τ , min	α , %

LABORATORY WORK № 28

DETERMINATION OF DENSITY OF POWDERED POLYMER

The density of the polymer which is in powder, is determined by a pycnometer with a volume of 5 to 10 ml. The cleaned and dried pycnometer is drawn on an analytical scale and poured 2 to 3 ml of distilled water from the engagement line. Pycnometer thermostat (Figure 28.1.) 20 min at 20 °C stored.

Then, the residue of the water is drawn with a filter paper so that the lower meniscus touches the engagement line. The pycnometer is scrapped and filtered by filter paper.

After this operation, dried pycnometer 1-2 g the pycnometer with the substance investigated is determined by the weight of the substance. Then the pycnometer is filled with liquid that is not solved. When adding a liquid pycnometer, it is necessary to remove air bubbles from the substance and the pycnometer walls. For this purpose, the fluid should be pushed into a pink, so its level is higher than the level of the substance.

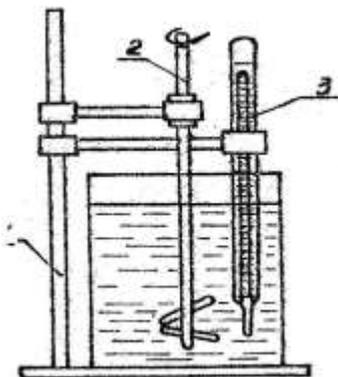


Figure 28.1. Density setting device

Such a filled pycnometer (unobstructed) is carefully heated in a water bath (30 -35 °C), released from the air bubbles by light shake. Then the pycnometer is added to the pumping line 2 to 3 mm above the liquid, 20 min. stored in the thermostat.

After that, the pycnometer is free of charge, washed and dried. Then, the pycnometer finds the weight together with the liquid.

Liquid density is determined by the following formula:

$d = (\text{the mass of pycnometer of the polymer and the pycnometer of the liquid along} - \text{empty pycnometer mass}) / (\text{mass of empty pycnometer with fluid, where the pycnometer polymer is not solved} - \text{empty pycnometer mass})$

The density of the substance is calculated as follows:

$$D = (Y \cdot d) / (Y + K-F)$$

Where Y is the mass of the substance to be investigated; d - liquid density; K - pycnometer and mass with matter of non-soluble fluid; F is the weight of pycnometer with liquid.

The results received from the practice are summarized below.

Powdered polymer	Solvent	y	d	k	F	D

LABORATORY WORK № 29

DETERMINATION OF MELTING TEMPERATURE IN THE CAPILLARY

In this way, only a small amount of the substance to investigate is needed to determine the melting point. Clean ingredients usually melt in very small temperature range. The ingredients in the mixture dissolve at low temperatures of the pure substances melting point. Because polymers are a mixture of polymer homogeneous molecules with different molecular weight, it is not possible to determine their exact melting point. Polymer has two temperatures when heated in the capillary: the dusty polymer particles begin to stick together (the temperature of the softening and the temperature of the melting point of the sample).

The course of action

The capillary 1 to 1.5 mm diameter capillary welded (1.5-2 mm high) melting temperature is included in the study material or polymer. The capillary is filled with the open end into the substance, so that the substance in question is pushed into the bottom of the capillary. For this, the capillary should be lightly touched by the finger. If the substance in question is oxidized or oxidized at high temperatures in the air, then the second end of the capillary welding is then welded.

The capillary thermometer filled with the material investigated is fixed by the rubber holder so that the substance is at the same level as the center of the thermometer. The thermometer is then inserted into the test bottle with the rubber plug, and the test bottle is positioned as a thermostat, and 2/3 part is filled with anhydrous glycerin.

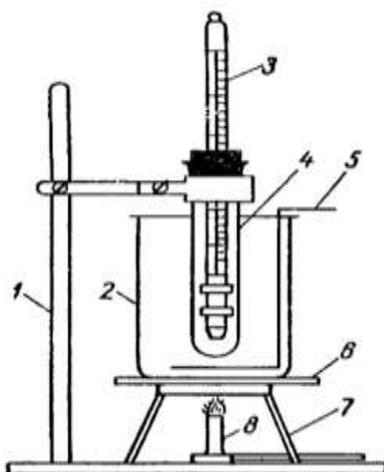


Figure 29.1. Chemical device for determining the melting point

Figure 29.1. shows the capillary melt temperature detector scheme (1 - long tube, 2 - thermometer, 3 - rubber gate, 4 - capillary, 5 - gas lamp, 6 - clamp). In this case, the thermometer should not be touched by the test bottle, and the test bottle should not touch the tube wall.

The appliance is heated over the asbestos grate so that the temperature can be increased to 1°C per minute.

Thermoplastic polymers are polymers that have the ability to soften and melt when heated and dissolve in suitable solvents due to the destruction of intermolecular bonds. Thermosetting resins are called in which, under the action of heat, reactive groups interact with each other to form strong transverse bridges (chemical bonds), while the polymer irreversibly hardens.

LABORATORY WORK № 30

DETECTING THE SOFTENING TEMPERATURE THROUGH THE UBBELODE DEVICE

Ubbelohde droplet temperature is deducted from the regular droplet temperature due to the effect of its weight on a regular heated substance. The appliance consists of a mercury cuff, a bottle-free glass thermometer at the bottom. The case is mounted on a thermometer with a metal cassette. Figure 30.1. shows the Ubbelohde thermometer, which determines the droplet.

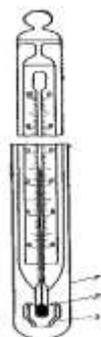


Figure 30.1. Ubbelohde thermometer that determines the degree of flushing

1 - test tube; 2 - sleeve holder; 3 - cup (for clarity, the sleeve into which the cup is inserted is removed)

The thermometer is securely fastened to the test case so that there is a gap between the bottom of the test bottle and the case. In the test box, it is placed in the tube, which is glycerol in its turn, to cover the anger thoroughly.

Prior to assigning, the bottle is removed from the crushed glaze, placed on the glass and filled with fused polymer.

The thermometer is inserted into the melted polymer and fixed into the bowl. When the polymer is completely cooled, the device is assembled and the fever starts, the temperature increase should be 1°C per minute. The softening point temperature is considered to fall when the melted polymer drops.

LABORATORY WORK № 31

DETERMINATION OF THE MOLECULAR WEIGHT OF HIGH MOLECULAR COMPOUNDS (POLYMERS)

The definition of the molecular mass of the polymer is based on the Mark Houwink equation:

$$[\eta] = KM^\alpha$$

here, $[\eta]$ - characteristic viscosity; M - molecular weight; K - fixed constant characterizing the polymer-solubility system at a certain temperature; α is a constant quantity that characterizes the mold of macromolecule and depends on the mobility of the chain.

Polymer	Solution	T, °C	k·10 ⁻⁴	α	Molecular mass interval, M·10 ⁻³
Polystyrene	Benzene	20	1.33	0.72	1.2-540
	Benzene	25	4.17	0.6	1.2-110
	Toluene	20	0.007	0.93	384-480
	Toluene	25	1.18	0.72	100-600
Polyvinyl alcohol	Water	20	3.0	0.5	8.5-1700
	Water	25	5.95	0.63	11.6-195
Polymethyl-methacrylate	Acetone	20	4.52	0.62	410-3370
	Acetone	25	0.96	0.69	410-3370
	Benzene	25	0.94	0.76	56-980
Polyvinylacetate	Acetone	20	0.75	0.75	45.0-420
	Acetone	20	0.70	0.70	68-680
	Chloroform	20	0.74	0.74	68-680

Results for k and α constants for different polymer are given in the table.

To determine characteristic viscosity, it is necessary to calculate relative and specific viscosity.

Determination of relative and special viscosity.

Oswald viscometers are used to determine the specificity. In the appropriate solvent, the polymer solution is prepared. Solution should be less volatile and good solution. The thickness of the polymer solution can range from 0.01 g /100 ml to 1 g /100 ml. A low molecular weight solution is prepared from the polymer. The polymer solution is prepared by the following procedure: a flat bottom flask with 25 or 50 ml of dried and dried volume is poured into the analytical scale of the polymer (with the preparation of 10-20 ml solution). For example, to prepare a 0.2% solution it is necessary to take 40 mg of polymer and 20 ml of solvent. The tube is clogged after the polymer and the tube is inserted. The tube stops caution every 10-15 minutes to speed up the solution and to obtain the same solid solution.

The polymer is filtered by 2 or 3 glasses after a complete solution. The filtrate is poured into 15-20 ml of pipette and washed in pre-dried viscosimeters. The diameter of the viscometer capillary should be chosen so that the duration of the cleaner solubility is less than 60 sec (as good as 100-120 sec).

Viscometer solution and solution with 20 min. The temperature should be maintained at the temperature of $20 \pm 0,10$ °C. Then, the solubility of the solvent and the solutions is determined. First, the solvent is poured into the viscosimeters and is poured into rubber viscosimeters. When the fluid begins to fall with its flow, the expiration time between the top and bottom of the pad is indicated by the stopwatch. The discharge time is determined several times for soluble solutions. Different prices, which correspond to 3-5 points for the report (prices difference should not be less than 0.2 s). After determining the run time several times, the solubility of the solvent is measured to check the capillary cleanness.

Relative viscosity is calculated by the following formula:

$$\eta_{\text{relative}} = \tau / \tau_0$$

The logarithm of the characteristic identity is equivalent to the linear function of the molecule mass.

$$\lg [\eta] = \lg k + \alpha \lg M$$

The polymer fractions (not less than 5) known to find the value of the logarithm of the k and α characteristic are shown as functions corresponding to the molecule in the graph (Figure 31.1.). The tangent angle of the straight line formed by the absciss axis of the image is equal to α . The line that cuts the straight line from the ordinate axis equals $\lg k$.

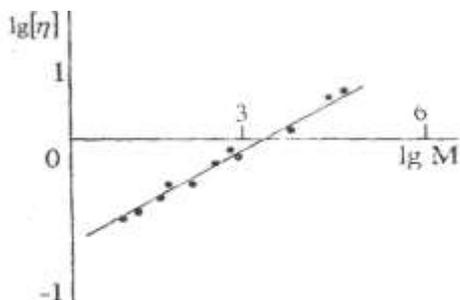


Figure 31.1. Dependence of $\lg [\eta]$ on $\lg M$

When the straight line is extended to the ordinate axis, the cut-off gives the value of the characteristic viscosity.

The value of the characteristic vomit is set in the Staudinger formula and the polymer's molecular weight is calculated.

The results obtained from the test are as follows.

Polymer	Solution	Viscosimeter №	Temperature °C	C, %	τ_0 sec	τ_0 sec	η	H_{sp}	$[\eta]_{ch}$	H_c

LABORATORY WORK № 32

DETERMINATION OF POLYDISPERSITY OF HIGH-MOLECULAR COMPOUNDS

High-molecular compounds consist of a mixture of polymer homologues with different molecular masses. To determine polydispersity, by any method, it is separated into fractions consisting of macromolecules with a relatively close molecular mass, followed by the amount of each fraction and the average molecular mass. On the basis of the information received, distribution curves are built. The separation of high-molecular compounds into fractions is based on the fact that fractions with different molecular masses of the same composition have different solubility capacity in the same solvent. The amount of solvent needed to transfer individual fractions to the solution in case of swelling is different. The lower the molecular mass of the homologues of the high-molecular compounds in the composition of the fraction, the less the amount of solvent needed to transfer that fraction to the solution. Therefore, when adding precipitators to the solution of high-molecular compounds, first of all, the fraction with the highest molecular mass is separate.

a) separation into fractions by the method of partial dissolution

In a conical flask with a volume of 250 ml, 10 g of crushed high-molecular compounds and 25 ml of solvent are placed in it and kept for 3 hours on condition of periodic mixing. The solution with a low molecular mass fraction is separated by decantation, and 25-30 ml of solvent is added on the residue of high molecular compounds. After 3-5 hours, they separate the second fraction by the same rule. Thus, high-molecular compounds are divided into several fractions: fraction solutions are collected in pre-drawn flasks. After roasting the solvent in a water bath, the remains of high-molecular compounds are dried

until a stable weight. The mass of each fraction is expressed in percent by the first mass of high-molecular compounds.

b) separation into fractions by fractional precipitation.

10 g of crushed high-molecular compounds are added in portions to a 1000 ml flask and 200 ml of solvent is poured into it. If the dissolution goes badly, the test tube is connected to a counter-cooler and heated in a water bath. After the high-molecular compounds are completely dissolved, the drip precipitator is added to the flask at a constant temperature and mixed. The tube is held motionless for some time, when a persistent nausea is formed. The first fraction with the highest molecular weight is separated. The solution is separated from the sediment by decanting, then poured into a second test tube and the operation is repeated. The fraction isolated as a precipitate is dissolved in a small amount of solvent, transferred to a pre-weighed glass and a precipitator is added in order to completely precipitate the high-molecular compound. After that, the mixture of solvent and precipitator is removed, dried together with the precipitate in a vacuum drying cabinet at a temperature of 40-60 °C until a stable weight is obtained.

c) after the separation of a high-molecular compound into fractions, the molecular weight of each fraction is determined (you should look at the work on determining the molecular weight of polymers).

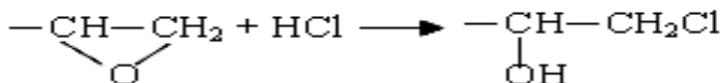
The results obtained are transferred to the following table

Fraction number	Fraction mass, g	Mass fraction of the fraction, g	Molecular weight

LABORATORY WORK № 33

DETERMINATION OF EPOXY GROUPS

Analysis of the amount of epoxy groups in a high-molecular compound is based on the reaction of epoxy groups with hydrochloric acid.



With the titration of excess acid, the amount of hydrochloric acid entering the reaction, epoxy groups corresponding to it, is calculated. It is necessary to pay special attention to the fact that the solvents used during the analysis of the amount of epoxy groups are completely dehydrated, since a very small amount of water, which can be in solvents, can affect the results of the analysis by entering the epoxy group into the reaction. One of the relatively easy-to-perform methods for analyzing epoxy groups in a high-molecular compound is as follows.

Reagents: Oligomer holding the epoxy group, 0.2 N hydrochloric acid solution in acetone, 0.1 N acetone solution, sodium hydroxide

Equipment. Erlenmeyer Flask

Progress of work

0.4-0.6 g in each of the two Erlenmeyer flasks. a sample of a high-molecular compound is poured out and a 0.2 N solution of hydrochloric acid in acetone in an amount of 30 ml is added to it (a solution of hydrochloric acid is prepared in 1600 ml of anhydrous acetone in an amount of 17 ml with a density of 1180-1190 kg/m³, dissolving solid hydrochloric acid). At the same time, an experiment is being conducted with random verification. The flasks are kept for 3 hours with the mouth closed. Then, 10 ml of anhydrous acetone and 3-4 drops of a phenolphthalein solution are added to each of the test tubes,

titrating 0.1 N of a sodium hydroxide solution remaining from an excess of hydrochloric acid.

The number of epoxy groups in a high-molecular compound as a percentage (x) is calculated using the following formula:

$$x = \frac{(V_1 - V_2) \cdot f \cdot 0,0043 \cdot 100}{g}, \%$$

here V_1 and V_2 are, respectively, the volumes of 0.1 N of NaOH solution spent on the verification experiment and titration of the sample, ml;

f - the correction factor of the NaOH solution;

0.00043 – the number of epoxy groups corresponding to 1 ml of 0.1 N NaOH solution, g;

g - the mass of the sample, g.

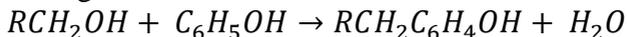
The average score is calculated based on the results of parallel experiments.

Epoxy compounds are chemicals that include epoxy rings (one or more). An epoxy ring is essentially one oxygen atom bonded to two carbon atoms. They react with amino, hydroxyl and carboxyl groups, as well as with mineral acids, forming relatively stable compounds. Epoxy compounds are widely used as chemical intermediates in the production of solvents, plasticizers, adhesives and synthetic resins. They are often used in various industries as protective coatings for metal and wood. Alpha-epoxy compounds with the epoxy group (CAC) at position 1,2 are the most reactive of all epoxy compounds and are most often used in industry. Epoxy resins after reaction with hardeners form universal thermosetting materials that have found a variety of applications, including surface coatings, electronics (sealants), lamination and bonding of various materials.

LABORATORY WORK № 34

DETERMINATION OF METHYL GROUPS

Several methods for the determination of methyl groups in the presence of phenolic hydroxides have been proposed. A. Vanscheid and B. P. Yershov. They used the Koebner method to determine and methyl groups in resole-type oligomers. In this method, phenol alcohols are condensed in the presence of HCl, resulting in diphenylmethane derivatives. Determining the amount of phenol that does not react after steam distillation allows us to calculate the number of reactive groups $-\text{CH}_2\text{OH}$. The $-\text{OH}$ groups of phenol are undergoing a change at this time. The reaction goes like this:



When determining the methyl groups with phenol, not only the methyl groups react, but also the free formaldehyde located in the primary oligomer. In addition, phenol formaldehyde reacts with the methyl groups of the oligomer in the free phenol, which is located in the oligomer. Therefore, when determining the number of methyl groups, it is also important to determine the amount of free phenol and formaldehyde.

Reagents: Phenol-80% solution (80 g in a measuring flask per 100 ml phenol is dissolved), 1 N HCl solution, 0.1 N potassium-bromide-bromate solution (2,784 g of potassium-bromate and 10 g of potassium-bromide is dissolved in 1 liter of distilled water), sodium-thiosulfate 0.1 N solution, HCl with a density of 1190 kg/m³, potassium-iodide 10% solution in water, starch 1% solution in water, bromine water (bromine is added to distilled water until a saturated solution is obtained, then 2 N HCl solution is added to the corresponding volume), ethyl alcohol-rectified.

Equipment: round-bottomed flask, reverse refrigerator and water bath

Progress of work

0.0002 g a sample of a high-molecular compound, crushed with precision, is poured into a round test tube with a volume of 150 ml and 1 ml of an 80% phenol solution is added with a pipette. The test tube is connected to a polished counter-refrigerator, placed in a boiling water bath and 1 N 10 ml of HCl solution from the refrigerator is added. It is heated for two hours, cooled if there is a refrigerator. Then this mass is transferred to another flask for running with water vapor, and the flask is washed with 20 ml of ethyl alcohol. The rest of the phenol is distilled off with water vapor, and the distillate is collected in a measuring flask with a volume of 1000 ml. Steam distillation is carried out until the distillate forms turbidity with brominated water. Then the solution is washed with distilled water to the measurement line, mixed well, 25 ml is taken from the solution and the amount of phenol is determined by the bromo-metric method.

In parallel, under the same conditions, an “emulsion-free” experiment is carried out with steam distillation of 1 ml of 80% phenol from a mixture consisting of 10 ml of 1 H HCl solution and 20 ml of ethyl alcohol.

The amount of phenol in grams (a) in the emulsion-free practice is calculated by the formula:

$$a = \frac{0,00156 \cdot V_1 - k - 100}{25} = 0,0626 \cdot V_1$$

In working practice (b) is calculated using this formula:

$$b = \frac{0,00156 \cdot V_2 - k - 100}{25} = 0,0626 \cdot V_2$$

here V_1 is the volume of 0.1 N sodium-thiosulfate solution spent on titration in experiments without a sample, ml;

V_2 - the volume of 0.1 N sodium-thiosulfate solution spent on titration in working practice, ml;

k - the correction factor so that the density of the sodium-thiosulfate solution is exactly 0.1 N;

0.0001567 - the amount of phenol corresponding to 1 ml of 0.1 N sodium-thiosulfate solution, g.

The number of methyl groups as a percentage (x) is calculated as follows:

$$x = \frac{0,33 \cdot [a - (b - e)]}{g} \cdot 100 - 1,003 \cdot c, \%$$

here a - the amount of phenol in experiments without a sample, g;

b - the amount of excess phenol detected during titration in the working experiment, g;

e - the amount of free phenol in the oligomer mass under study, g;

c - the amount of free formaldehyde in the oligomer under study, %;

0.33 - the number of methyl groups corresponding to 1 g phenol;

1.003 - the ratio of the masses of the molecules of methyl groups and formaldehyde;

g - the mass of the sample, g.

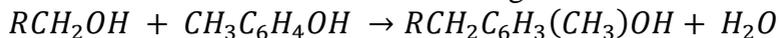
This method was modified by Martin. Thus, the amount of water formed during condensation is determined by the azeotropic displacement method, and the number of methyl groups is calculated by the amount of water formed. To get a good result, you need to select at least 10 g.

Methyl groups in phenol-formaldehyde oligomers, it can be determined by condensation with m-cresol in the presence of

p-toluol sulfonic acid or with phenol in the presence of BF_4 . The report is based on the amount of water obtained as a result of the reaction determined using the Fischer reagent.

The reaction of the interaction of methyl groups with m-cresol occurs in the presence of an acid catalyst. The resulting water is determined by the Fischer reagent.

The reaction follows the following scheme:



Reagents: Reagent solution: 500 ml of m-cresol and 15 g in 100 ml of benzene. P-toluene is dissolved in sulfonylurea monohydrate. The mixture of azeotropic distillation with benzene is dehydrated.

When the water dripping in the trap ends, the benzene is expelled by heating the solution to 150°C . The remains of a dark color from the flask are placed in a glass jar with a stopper.

Equipment: Wide-necked flask with two stoppers of 250-300 ml (one of them has 5 holes), polished micro-burette (glass) (Figure 34.1.)

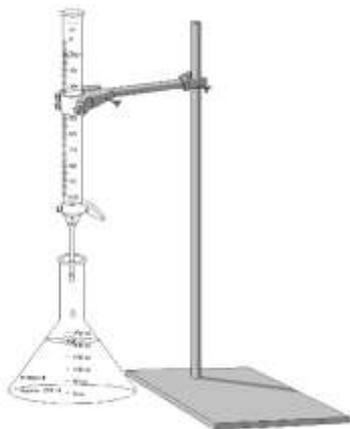


Figure 34.1. Titration device
Progress of work

0.3-0.5 g per 250-300 ml of well-dried sausage. a crushed sample of a high-molecular compound is poured, 10 ml of the reagent solution is added, the jeep is closed with a cork and put on the thermostat at 90 °C. After an hour, the test tube is cooled and 2 ml of anhydrous pyridine is added. After the second cooling, the separated water is titrated with the Fischer reagent. In parallel, an experiment is conducted under the same conditions (without a sample).

The number of methyl groups as a percentage (x) is calculated as follows

$$x = \frac{0,1723 \cdot \left(V_1 - V_2 - \frac{V_3 \cdot g}{g_1} \right)}{g} \cdot 100 - 1,003 \cdot x_1$$

here V_1 is the volume of the Fischer reagent spent on titration of a sample of a high-molecular compound, ml;

V_2 - the volume of the Fischer reagent spent on titration in experiments without a sample, ml;

V_3 – the volume of the consumed Fischer reagent when determining the humidity of a sample of a high-molecular compound, ml;

g - the mass of a sample of a high-molecular compound taken for the determination of methyl groups, g;

g_1 - the mass of a sample of a high-molecular monomolecule taken to determine its humidity, g;

x_1 - the free amount of formaldehyde, g;

0.1723 - the number of methyl groups corresponding to 1 ml of Fischer's reagent, g;

1.003 - the ratio of the masses of the molecules of methyl groups and formaldehyde.

LABORATORY WORK № 35

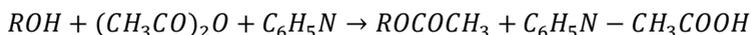
DETERMINATION OF HYDROXYL GROUPS

In high-molecular compounds, hydroxyl groups are determined by the formation of esters from the interaction of anhydride and halide-anhydrides of organic acids.

To determine the hydroxyl groups in high-molecular compounds, the method of acetylation in a pyridine solution is mainly used.

In the process of acetylation, an ester is formed from the interaction of acetic anhydride and a pyridine residue of a compound containing a hydroxyl group. In this case, the released acetic acid forms a neutral compound with pyridine, which does not allow the resulting ether to be saponified.

The reaction follows the following scheme:



When water is added, the salt is quickly hydrolyzed and free acetic acid is released. The resulting acid is titrated with 0.5 N NaOH solution together with acetic acid formed from an excess of acetic anhydride (in the presence of phenolphthalein).

The accuracy of the determination of hydroxyl groups with acetic anhydride depends on the purity of the reagents used (pyridine and acetic anhydride), as well as on the conditions of hydrolysis and the completeness of acetylation. Complete acetylation can be achieved by changing the time of the process, the density and molarity of the reagent. The nature of the sample in which the hydroxyl groups are determined also has its effect on the complete acetylation.

Reagents: A mixture of acetic anhydride and pyridine-mix 10 mass parts of acetic anhydride and 90 mass parts of pyridine containing 0.35% water, 0.5 N NaOH solution, 1% alcohol solution of phenolphthalein

Equipment: round-bottomed flask, reverse refrigerator and water bath

Progress of work

0.0002 g weighed with an accuracy of 0.1-0.3 g a sample of a high-molecular compound is placed in a round tube with a volume of 250 ml, to which 10 ml of a mixture of acetic anhydride and pyridine is added, combined with a ground anti-cooler and heated in a sand bath for two hours.

After cooling the tube, 50 ml of distilled water is added to the upper part of the refrigerator and the resulting acetic acid 0.5 N NaOH is titrated in the presence of phenolphthalein. In parallel, an experiment without samples is carried out under the same conditions.

The number of hydroxyl groups as a percentage (x) is calculated as follows:

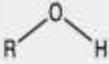
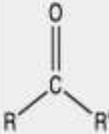
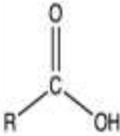
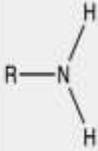
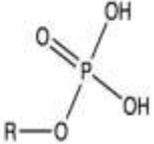
$$x = \frac{0,0085 \cdot (V_1 - V_2) \cdot k}{g} \cdot 100 = 0,85 \cdot \frac{(V_1 - V_2) \cdot k}{g}$$

here V_1 - the volume of 0.5 N NaOH solution spent on titration in an emulsion-free medium, ml;

V_2 - the volume of 0.5 N NaOH solution spent on titration in an emulsion medium, ml;

k - correction factor for the viscosity of the NaOH solution to be exactly 0.5 N;

g - the mass of the sample, g.

Functional Group	Structure	Properties
Hydroxyl		Polar
Methyl	$R-CH_3$	Nonpolar
Carbonyl		Polar
Carboxyl		Charged, ionizes to release H^+ . Since carboxyl groups can release H^+ ions into solution, they are considered acidic.
Amino		Charged, accepts H^+ to form NH_3^+ . Since amino groups can remove H^+ from solution, they are considered basic.
Phosphate		Charged, ionizes to release H^+ . Since phosphate groups can release H^+ ions into solution, they are considered acidic.

Hydroxyl group (-OH), a monovalent chemical group consisting of a hydrogen atom connected to an oxygen atom. It is an important functional group of organic compounds, such as alcohols and phenols. It should not be confused with the hydroxide ion OH^- .

LABORATORY WORK № 36**OBTAINING SULFOCATIONITE BASED ON
POLYSTYRENE**

- Reagents:** Polystyrene (granular) – 5 g.
Solvent – 65 ml
95% sulfuric acid – 40 ml
Catalyst – 0,05 g.
Sulfuric acid solutions, % (5; 25; 50; 75)
- Devices:** A flask with a 200 ml stopper, a Buchner funnel, a measuring flask, a beaker, a porcelain bowl, a water bath, a vacuum drying cabinet.

Progress of work

The production of an ionizing oligomer based on polystyrene is carried out by a polymerological transformation reaction. 95% sulfuric acid and AlCl_3 catalyst are used for sulfonation of expanded polystyrene on carbon tetrachloride (CCl_4). For this purpose, polystyrene (5 g) is placed in a cylindrical glass container, which is crushed to a granular state, and then a solvent is added in a ratio of 1:13.

Polystyrene in granular form swells to CCl_4 within 20-30 minutes. Then the residual solvent is removed from the flask and 95% sulfuric acid (polystyrene) is added:acid=1:8) to expanded polystyrene. As a catalyst, anhydrous AlCl_3 is added to the reaction mass (by weight of 1% polystyrene), the sulfonation process is carried out at 40-50⁰C for 3-4 hours. After the end of the reaction, the sulfocationite glass is transferred into a funnel and gradually, i.e. washed with relatively weak sulfuric acid solutions of 75%, 50%, 25% and 5%. Then the sulfonated polystyrene is washed in a Buchner funnel with distilled water until a neutral reaction and kept in distilled water.

The washed grains of sulfocationite are filtered and dried first in air at 50-60⁰C, and then in a vacuum drying cabinet at 40⁰C until a stable weight is obtained.

Task

1. It is necessary to calculate the ion-exchange resins output.
2. It is necessary to determine the degree of swelling of the ion-exchange resins (in water).
3. The solubility of the ion-exchange resins should be checked.

Along with silicate or carbon-based materials, artificial exchangers have recently become increasingly important, since they have a large exchange capacity and high chemical resistance and therefore are increasingly being used.

Ion exchange resins are synthetic organic ionites, which are high-molecular polyelectrolytes insoluble in water and organic solvents, capable of exchanging mobile ions upon contact with electrolyte solutions.

Synthesis of polymer ionites is produced most often:

1) polycondensation or polymerization of monomers containing ionogenic groups to form a mesh polymer;

2) by attaching ionogenic groups to individual links of a previously synthesized mesh polymer;

3) by attaching ionogenic groups to the links of a synthetic linear polymer with its simultaneous transformation into a mesh polymer.

LABORATORY WORK № 37

OBTAINING OF SULFOCATIONITE BASED ON PHENOL-FORMALDEHYDE OLIGOMER

Reagents: Phenol-formaldehyde oligomer – 40 g.
37 % formaldehyde solution in water – 120 g.
95 % sulfuric acid – 120 g.

Devices: A flask with a 250 ml stopper, a measuring flask, a porcelain bowl, a water bath, a vacuum drying cabinet.

Progress of work

The sulfonation process is carried out in a laboratory reactor equipped with a 250 ml reflux refrigerator. First, the optimal amount of modified phenol-formaldehyde oligomer and 95% sulfuric acid are introduced into the reactor. The reactor is heated until the oligomer is completely dissolved (140°C), and then cooled to room temperature. 30 ml of a 37% formaldehyde solution in water (formalin) is immediately added to the cooled reaction mass. After that, the reaction mass is placed in an oil bath with a temperature of 110°C in a special container and the solidification process is carried out for 2 hours. The reaction mass after the solidification process is washed with distilled water to a neutral reaction.

The washed grains of sulfocationite are filtered and dried first in air at $50\text{-}60^{\circ}\text{C}$, and then in a vacuum drying cabinet at 40°C until a stable weight is obtained.

Task

1. It is necessary to calculate the yield of ion-exchange resins.
2. It is necessary to determine the degree of swelling of ion-exchange resins (in water).
3. The solubility of ion exchange resins should be checked.

LABORATORY WORK № 38

THE EFFECT OF IONITES ON SOLVENTS

It is known from the definition of ionites that they are practically insoluble in the working environment. Their solubility should be several times less than that of even slightly soluble organic monomers and inorganic substances, so these ionites should remain in solvents for a long time.

It is known that the solubility of high molecular weight compounds with a linear structure decreases with increasing molecular weight, and compounds with high molecular weight have poor solubility. The formation of bonds between macromolecules with a linear structure (intermolecular bond) leads to an increase in the mass of molecules.

The formation of a bond between two one-dimensional macromolecules increases the mass of its molecule by about two times. Thus, it is possible to obtain completely insoluble macromolecules. The solvent cannot break these bonds and separate individual macromolecules from each other by joining the lattice structure.

The addition of hydrophilic ion exchange groups leads to the hydration of macromolecules and increases their tendency to solubility. The ingress of solvent molecules into the macromolecule leads to the stretching of the mesh and the rupture of the internal bonds of the molecules.

An increase in the number of ionogenic hydrophilic groups enhances this, and an increase in the number of intermolecular bonds has the opposite effect. Thus, the main goal of synthesis is to find the optimal ratio between the active groups of ionites and groups having the opposite dispersion effect. Practically, by regulating the number of these macromolecular bonds, choosing the conditions of

polymerization and polycondensation, a compound with the maximum possible molecular weight is obtained. In all cases, a macromolecule with a geometrically irregular structure is obtained.

As a result of polycondensation and polymerization, polymer-homologous compounds with different molecular weights are always formed, even a small number of macromolecules with a linear structure are formed, including a structure with a small number of linear structural or intermolecular bonds. This type of macromolecules has solubility under certain conditions.

Ionogenic groups are introduced into the styrene and divinyl copolymer, simultaneously converting it into a mesh copolymer.

By attaching sulfhydryl groups to polymer links, ionites are obtained that are capable of electronic exchange reactions (redox reactions); by attaching acid and amino groups simultaneously, they impart amphoteric properties to ionites; by the close location of ionogenic and polar groups in polymer links, they create conditions for the formation of complexes with the absorbed ion.

The ion exchange sorption method is used to soften or desalinate water, remove salts from sugar syrups, milk, wines, fructose solutions, from solutions of tannins, hydrolysis products of agricultural raw materials waste, from solutions of various drugs (antibiotics, vitamins, alkaloids), to remove calcium ions from blood plasma before its preservation, for purification from mineral ions of solutions of organic reagents, for wastewater treatment from phenol, chromium, nickel, etc., as well as for the concentration of valuable ions in microdoses in solution, including for the extraction of ions from the discharge waters of galvanic workshops, for the capture and concentration of radioactive ions, copper ions from the wastewater of the copper-ammonia production of artificial silk.

LABORATORY WORK № 39

DETERMINATION OF IONITE CHANGE CAPACITY BY STATIC METHOD

The variable capacity of ionites is determined by the following methods.

1) *Potentiometric titration method*

The first option. Glass beakers with a volume of 150-200 ml, tightly closed with a stopper, weighing 0.25 g with a particle size of 1.0-1.0 mm added ionite. Then 100 ml of distilled water is added to one glass container, and 100 ml of an alkali solution (for H-cationite) or acid (for OH-anionite) of various concentrations (25-250 mg-eq / l) is added to the other. After the required contact time has elapsed (depending on the rate of neutralization of the ionogenic group of ionite), in the presence of ionite in each glass container, the pH of the solution with a glass electrolyte is measured. Titration curves are constructed from two or more parallel experiments when the price of the corresponding pH is taken. The pH value is indicated on the ordinate axis, and the amount of added alkali or acid (mg-eq/l) is indicated on the abscissa axis.

The second option. 1.0 g from conical flasks with a volume of 150-200 ml. ionite is included. 100 ml of 0.25 N NaCl solution is added to the first flask, 10 ml of 0.25 n alkali (or acid) solution is added to the second flask, and 20 ml of the same solution is added to the third flask, and so on. Then a NaCl solution is injected into each tube. The NaCl solution is added to each tube up to a full volume of 100 ml. Samples of strong cationite and strong anionite are kept together with the solution for a day, and samples of weak acidic and weak weak anionite-for seven days, after which the pH of these solutions is measured. According to the results of the experiment, potentiometric titration curves are constructed.

With the help of potentiometric titration curves, depending on the pH of the medium, the presence of active groups in the ionite is clarified. The degree of their dissociation, the volume of complete change of the ionite (defined as the sum of all active groups that make up the ionite and react), the volume of change of individual active groups can be determined at constant pH values of the medium. Thus, the main chemical characteristics of the ionite are refined by the method of potentiometric titration.

2) *Determination of the total replaceable capacity by a simplified method.*

1.0 g in a flask with a flat bottom volume of 300-500 ml an ionite sample is placed (cationite in H-form or anionite in OH-form). Then 200 ml of 0.1 N alkali solution (or acid solution) is added to the sample and kept for 24 hours with constant stirring. After the specified period, the ionite is separated from the solution by filtration, and part of the solution (usually 25 ml) is titrated with 0.1 N acid or alkali solution in the presence of an indicator. The price of the variation volume mg-eq/g is expressed in.

3) *Calculation of variational power by active groups.*

The above methods allow us to determine the amount of volume change for all ionogenic groups. Usually ionites consist of various active groups depending on their composition. The amount of ionite change during ionite operation depends either on one active group, or on the pH of the medium and, depending on the conditions of the process, on several active groups. For example, the cationite "KY -1" consists of the groups $-\text{SO}_3\text{H}$ and $-\text{OH}$. In most cases, the amount of change during the introduction of the "KY-1" cationite is determined based on the sulfogroups. When determining the amount of variation of natural multifunctional ionites, it is necessary to know the amount of variation for each active group. The volume of

variation for certain active groups is always a constant value, such as the total volume of ionite variation.

A relatively accurate method for determining the variational volume of ionite by active groups is potentiometric titration. According to the active groups, the volume of ionite variation is determined by another method as follows:

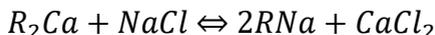
1.0 g of pre-prepared ionite (H-shaped cationite or OH-shaped anionite). it is placed in a 100 ml flask, then 100 ml of 0.1 N solution of CaCl_2 (for cationite) or Na_2SO_4 , and in some cases NaCl and KCl (for anionite) are added to the flask. With periodic stirring, the ionite is separated from the solution after contact for 24 hours. A certain part of the solution is titrated with 0.1 N alkali solution (or acid solution) in the presence of an indicator. When the pH of the medium is ≈ 7 , the color of the solution changes. Having determined the volume of variation of ionite with strong dissociating groups (mg-eq/gr) and knowing the full volume of variation of the ionite under study, based on their differences, it is possible to determine the volume of variation for other active groups. For example, in the study of sulfogroup phenolic cationites, this difference shows that the volume of variation is calculated based on the hydroxyl group of phenol. In a strongly basic polyfunctional anionite (Brand "AB-16"), the variable volume expresses all amino groups in the ionite structure, with the exception of pyridine groups. The absence of the volume of variation in weak acid cationites and solutions indicates a low degree of dissociation of ionogenic groups in ionite.

When determining various weakly dissociated groups, ionite titration in the presence of an indicator, the solution changes its color at the corresponding pH values.

4) Determination of the capacity of the equilibrium change

The equilibrium dispersive capacity of an ionite, in contrast to the total dispersive capacity, is not constant for active

groups and depends on various parameters (pH of the medium and density of the solution, the nature of the changing ions, etc.) depends. It can be seen from the above methods that the ionization equilibrium can be determined for various systems.



Determination of the volume of equilibrium change of strong acid cationites is most often carried out by the following method:

N-cationite is preliminarily converted to Na-form, and then neutralized as a complete H-cationite with a solution of 1 N NaOH. The cationite is washed with distilled water in the presence of thymolphthalein until a neutral reaction. Thus, the prepared cationite is stored dry in a tightly closed container.

1 g of the prepared cationite is sampled, placed in a tightly closed glass container with a volume of 250-300 ml, to which 100 ml of 0.1 N CaCl₂ solution is added. The sample is stored for 5-6 days. Then the calcium in the solution, which is in equilibrium, is titrated with a solution of trilon-B. To do this, 1 ml of boron buffer and 7-8 drops of indicator (chrome-dark blue) are added to 25 ml of the solution. Then the solution is gradually titrated with a 0.01 N Trilon-B solution until the color changes (from red to blue) using a micro-burette. The equilibrium volume of change is expressed in mg-eq/g.

Weak acidic cationites are pretreated with 0.5 N NaOH solution, converted to Na form, then washed with distilled water until separated from the alkali solution (tested with thymolphthalein). The equilibrium volume of change is determined by a solution of 0.1 N CaCl₂, as indicated above (as in strong acid cationite).

When determining the equilibrium shear volume for anionites with a strong base, the Cl form of anionite and a salt

solution ($\text{Na}_2\text{SO}_4\text{-a}$) are used. First, the anionite in the OH form turns into the Cl form. To do this, it is treated with 0.1 N sulfuric acid solution, after 3-4 days from the interaction, the anionite is enriched with an acid solution. The separated anionite is first washed with distilled water, and then with alcohol until the chlorine ion is completely separated. So, 1 g of the prepared anionite sample. it is placed in a 250-300 ml glass flask with a tightly closed mouth, to which 200 ml of 0.1 N Na_2SO_4 solution is added. The sample is kept for 5-6 days, then the amount of chlorine ion in the solution is determined in an equilibrium state.

Knowing the number of chlorine ions in the initial anionite and in the equilibrium solution, based on their difference, it is possible to determine the volume of the equilibrium change of the anionite based on the SO_4 ion. The equilibrium shear volume can also be determined by the method described above for weak without basic anionites (for example, when a 0.1 N solution of Cl- H_2SO_4 anionite is in contact). When comparing the potentiometric titration method with the simplified static method, the same results are observed (Table 1).

The total volume of substitution of cationites and anionites determined by different methods is shown in Tables 2 and 3.

Table 1
Determination of the volume of variation of cationites by active groups

Cationites	The total volume of variation by the static method (0.1 N NaOH), mg-eq/g	Volume of variation by sulfogroups, mg-eq/g		The calculated volume of variation based on the hydroxyl group of phenol, mg-eq/g
		0.1 N CaCl ₂ (static method)	For 0.1 N NaOH pH=6.5-7 (potentiometric method)	
“KY-1”	5.28	2.19	2.25	3.09
“MCΦ-3”	5.04	1.98	2.0	3.06
Sulfogroup	3.38	1.32	1.30	2.06

Table 2
The total volume of change of cationites determined by various methods

Cationites	With a simplified static method, mg-eq/g	According to the potentiometric titration method, mg-eq/g
“KY-1”	5.28	5.31
“MCΦ-3”	5.04	5.00
Sulfogroup	3.98	3.35

Table 3
The total volume of anionite change determined
by various methods

Anionites	With a simplified static method, mg-eq/g	According to the potentiometric titration method, mg-eq/g
“ЭДЭ-10П”	9.29	9.19
“АН-2Ф”	9.10	9.07
“АН-9”	3.57	3.60

LABORATORY WORK № 41

IONITE RESISTANCE TO ACIDS AND ALKALIS

The chemical resistance of ionite is determined as follows:

1 g of ionite is placed in a round flask equipped with a 150-200 ml reflux refrigerator. Then 100 ml of 5 N acid or alkali is added to the flask, the flask is placed on a boiling water bath, after a certain time (approximately 30 minutes) the flask is removed from the water bath, cooled to room temperature (in air) and the ionite is separated from the solution. Then the ionite is treated with an excess of acid or alkali and converted into the H-form (cationite) or OH-form (anionite), and after washing by static method, the volume of its complete change is determined. Based on the comparison of the volumes of complete conversion of ionite before and after contact with acid or alkali, the chemical resistance of ionites is determined by the formula:

$$X = \frac{SDH_K \cdot 100}{SDH_H}$$

Where SDH_K is the volume of complete change of the cationite after contact with acid or alkali;

SDH_H is the total volume of the cationite change before contact with acid or alkali.

To determine the resistance of ionites to oxidants, 1N of nitric acid and a 10% solution of hydrogen peroxide are used.

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