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**ABSTRACT**

of the dissertation for the degree of Doctor of Science

**SYNTHESIS OF AMIDES AND IMIDAZOLINES,  
DEVELOPMENT OF THEIR PRODUCTION TECHNOLOGY  
AND RESEARCH AS ADDITIVES**

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## GENERAL DESCRIPTION OF WORK

**Relevance and degree of development of the topic.** The rapid development and widespread application of technology in developed countries of the world increases attention to the solution of the problem of protecting these technical equipment from atmospheric corrosion during operation and idle periods. It should be noted that since the equipment used in agriculture and military fields is stored in conservation conditions for most of the year, it must be protected from atmospheric corrosion. The main external factors that cause atmospheric corrosion are humidity, temperature and various aggressive components of the air. Since the mentioned factors differ sharply in different regions of the country, the solution of the problem of protecting metals from atmospheric corrosion is somewhat complicated.<sup>1</sup>

For this reason, the allocation of large amounts of funds for the protection of metal structures and structures from corrosion once again demonstrates the importance, significance of the problem and the importance of scientific and technical progress in this area.

Corrosion-preserving materials are used to prevent losses directly and indirectly as a result of the atmospheric corrosion process. Therefore, the creation of economically efficient and environmentally friendly atmospheric corrosion inhibitors is one of the most urgent problems.<sup>2</sup>

In this regard, in our country, attention is being paid to the development of highly effective preservative inhibitors to protect metal equipment and structures from corrosion.

It should be especially emphasized that in recent years, mainly multifunctional reagents have been used as corrosion inhibitors. Taking into account the above, the amides and imidazolines synthesized in our research work are presented in the form of

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<sup>1</sup> Abbasov, V.M. Korroziya / V.M.Abbasov. – Bakı: Savad, – 2023. – 360 s.

<sup>2</sup> Aydinsoy, E.A. A systematic review of corrosion inhibitors in marine environments: Insights from the last 5 years / E.A.Aydinsoy, Z.Z.Aghamaliyev, D.B.Aghamaliyeva, V.B.Abbasov // Processes of Petrochemistry and Oil Refining, – 2024. Vol.25, No.3, – p.793-843.

compositions. used in the creation of conservation fluids and lubricants consisting of various components. At the same time, they have also been studied as additives that improve the quality of bitumen.

It is known that bitumens are widely used in road construction in many developed countries of the world. Road bitumens are mainly obtained by oxidation of tar separated after distillation of oil. Bitumen obtained from oxidation of tar needs to be modified in order to improve its quality in order to be used mainly in the construction of transport roads. Because, if bitumen is not modified or if an additive is not added to bitumen, the dispersion process on roads built using these bitumens is faster. One of the main reasons for this is the poor adhesion of unmodified bitumen to gravel and other fillers.

For this purpose, it is especially important to add an additive that increases the adhesion of bitumens when using them in road construction.

Carrying out work in this direction is also relevant for our Republic, and the results obtained will contribute to the construction of roads in accordance with world standards.

**Object and subject of the research.** The object of the research is the synthesis of amides and imidazolines based on natural petroleum acids (NPA) and polyamines, and the development of scientific foundations of their production technology. It was considered appropriate to use NPA, which has a fairly large reserve in Azerbaijan, as a raw material for the production of amides and imidazolines.

**Aims and objectives of the research.** The aim of the dissertation work was to synthesize amides and imidazolines based on chemical raw materials that have sufficient reserves in our Republic and to study their use as a corrosion inhibitor component in the preparation of conservation fluids, as well as an additive that improves the quality of road bitumen.

To achieve the set objectives, the following scientific studies were conducted:

- preparation of preservation fluids and lubricants using amides,

imidazolines, metal salts of NPA (Co, Ni, Zn, Mn and Ba) obtained from the interaction of natural petroleum acids with Berolamine-20, nitrocompound of tetradecene-1 and various compositions based on paraffin as components;

- study of the physicochemical properties of preservation fluids, determination of their corrosion protection ability by various methods;

- testing of the prepared preservation fluids and lubricants in “Q-4” thermohumidity and new modern “CORROSIONBOX-1000E” experimental chambers;

- checking the quality indicators of bitumen by adding amides and imidazolines synthesized in different molar ratios based on natural petroleum acids, polyethylenepolyamine (PEPA) and Berolamine-20 (BA-20) to road bitumen;

- testing the quality indicators of bitumen by adding distilled natural petroleum acids, cubic residue of distillates of natural petroleum acids and amides synthesized on the basis of PEPA to road bitumen;

- testing the quality indicators of bitumen by adding compositions of distilled natural petroleum acids, cubic residue of distillates of natural petroleum acids and amides synthesized on the basis of Berolamine-20 with various oil distillates to road bitumen.

**Research methods.** The testing process of preservation compositions was carried out in the CORROSIONBOX-1000E experimental chamber, in the "Q-4" thermo-humidity chamber, in seawater and 0.001% H<sub>2</sub>SO<sub>4</sub> environment on steel plates within the framework of existing standards. Analysis of the structural composition of the components used in the preparation of preservation liquids was carried out using <sup>1</sup>H NMR, IR-Fourier spectroscopy methods and other modern physicochemical methods.

In order to study the effect of adding an additive to road bitumen on the quality indicators of road bitumen, the brittleness temperature was determined in the BPA 5 device of the Anton Paar company, the tensile strength in the DDA3 device, and the depth of needle penetration in the PNR 12 device.

### **The main provisions put forward for defense:**

- Amide and imidazoline derivatives based on NPA and polyamines, metal salts of NPA and nitro compounds were synthesized based on appropriate reagents.

- Compositions of amide and imidazoline derivatives with metal salts of NPA were added to various brands of oil distillates (T-30, T-22, T-46 and T-1500) and studied as preservation fluids.

- Compositions prepared in optimal proportions based on amide and imidazoline derivatives, metal salts of NPA and nitro compounds were added to mineral oil distillates (T-30, T-22, T-46 and T-1500) in various concentrations and studied as preservation fluids.

- Lubricants with different compositions were prepared by adding liquid paraffin to preservation fluids that showed high results and the corrosion protection ability of metal plates was tested.

- The physical and chemical properties of the prepared new composition of conservation fluids and lubricants, the material balance for the organization of their production, the principle technological scheme of the process and recommendations for their application in practice were given.

- The effect of amide and imidazoline derivatives prepared with metal salts of NPA as additives on road bitumen was tested.

- The effect of amide and imidazoline derivatives in various mineral oils on road bitumen was tested.

**Scientific novelty of the research.** For the first time, amide and imidazoline derivatives were synthesized based on NPA and Berolamine-20 and tested in a modern "CORROSIONBOX-1000E" experimental chamber as preservation fluids, and high results were achieved. At the same time, the synthesized amide and imidazoline derivatives were added to road bitumen as additives and their effect on the quality indicators of bitumen was studied, in particular, it was determined that they further improved its adhesion.

Thus, 10% solutions of compositions prepared in a ratio of (1:1:1) based on amides obtained from the interaction of natural petroleum acid with Berolamine-20, Co salt of NPA and nitro compound in mineral oils were studied as a preservation fluid and several times higher indicators than required (90 days, GOST 9054-

75) were obtained. Thus, the preservation fluids prepared on the basis of these compositions (experiments were carried out in the "Q-4" thermo-humidity chamber) had a corrosion protection effect on "polad-3" brand metal plates for 235, 350 and 467 days.

The sulfoderivative of fatty acid (sunflower fatty acid) imported from Azersun was synthesized, its Ca-salt was obtained and applied as a hydrophilizer to mineral powders.

**Theoretical and practical significance of the research.** High-quality conservation fluids, conservation lubricants, and additives to road bitumen were prepared based on natural petroleum acids obtained as a by-product and with sufficient reserves in the Oil Refining Complexes of Azerbaijan, and a principle technological scheme for organizing their production was developed.

The prepared conservation fluids are highly effective protective coatings that prevent corrosion of metals in various aggressive environments and can be successfully applied as a high-protection agent on the surface of the metal. This was also confirmed in the act issued for the tests conducted at "Baku Steel Company" LLC.

In addition, NPA Ca salt was synthesized based on NPA and calcium carbonate, and scientific research was conducted on the hydrophobicity of this powder by adding its composition with soapstock to stone dust in an amount of 2%.

Tests conducted at the "Azeryolemitadqiqatlayiha" Institute proved that this composition has a high hydrophobicity.

As a result of our research, a principle scheme has been developed to create a production scheme for the production of preservation liquids with different compositions and additives to road bitumen at the Experimental Industrial Plant of the National Petroleum Corporation of Azerbaijan (NPCI) in order to propose the production of corrosion protection coatings for metal equipment of the "Polad-3" brand and additives added to road bitumen. The proposed preservation liquids with different compositions can be used in various fields of industry, in the oil and gas production and processing sector for the preservation of military equipment and agricultural equipment, and the synthesized additives of various compositions can be used as additives to road bitumen in the

“Azeryolelmitadqiqatlayiha” project.

**Approval and application.** The main results of the dissertation work were discussed at the following scientific conferences: VIII Republican scientific conference of doctoral students, masters and young researchers "Actual problems of chemistry" dedicated to the 91st anniversary of the birth of Heydar Aliyev, (Baku, 2014); International conference "Caspian Sea, past, present, future", Dagestan State University, (Russia, 2014); Abstracts of reports of the Republican scientific and practical conference dedicated to the 100th anniversary of Academician S.S. Mehdiyev, (Baku, 2014); III International scientific conference of young researchers dedicated to the 92nd anniversary of the birth of Heydar Aliyev. Qafqaz University, (Baku, 2015); III International scientific and practical conference "Actual issues of natural and mathematical sciences in modern conditions of the country's development", (St. Petersburg, 2016); International youth forum Integration processes of the world science in the 21st century. (Ganja, 2016); Actual problems of modern natural sciences, (Ganja, 2017); International scientific-practical conference "Innovative prospects for the development of oil refining and petroleum chemistry", dedicated to the 11th anniversary of academician V.S. Aliyev, (Baku, 2018); XII International Scientific Conference "Current Problems of Chemistry" of doctoral students, masters and young researchers dedicated to the 95th anniversary of the birth of Heydar Aliyev. Baku State University, (Baku, 2018); The International Scientific Conference "Actual Problems of Modern Chemistry" Dedicated to the 90th Anniversary of the Academician Y.H. Mammadaliyev Institute of Petrochemical Processes, (Baku, 2019); International scientific conference on “Actual problems of modern natural and economic sciences” dedicated to the 100th anniversary of the birth of the general leader Heydar Aliyev, (Baku, 2023); World of conferences. IV International scientific conference, Scientific advances and innovative approaches, (Tokyo, 2023).

**Publication.** 26 scientific works have been published on the dissertation work, including 13 articles, 12 theses and 1 patent. The published works fully reflect the essence of the dissertation.



**Personal participation of the author.** The author personally performed the brief and correct formulation of the issues included in the dissertation, the implementation of the research, the generalization and interpretation of the experimental results obtained, and the conduct of the tests.

**Name of the organization where the dissertation work was performed.** The dissertation work was performed at the Institute of Petrochemical Processes named after academician Y.H. Mammadaliyev of the Ministry of Science and Education of the Republic of Azerbaijan.

**The total volume of the dissertation in characters, with the volume of the structural sections of the dissertation separately indicated.** The dissertation consists of an introduction, 6 chapters, conclusions and a list of 220 references. The dissertation consists of 42 figures, 26 graphics, 2 schemes, 50 tables and a total volume of 340 pages of printed material. The dissertation consists of 377756 characters (introduction 17203, chapter I 101075, chapter II 19514, chapter III 30955, chapter IV 94710, chapter V 88440, chapter VI 22040 and conclusion 3819 characters), excluding tables, figures, schemes and used literature.

The **introductory part** of the dissertation provides information on the relevance of the topic, the set goal, scientific novelty and practical significance of the work.

The **first chapter** consists of a literature review that includes information on additives for various purposes, corrosion problems, their solutions, atmospheric corrosion and its causes, the classification and history of preservation fluids, the technology for obtaining road bitumen and its quality requirements, and additives used to improve the quality indicators of road bitumen.

The **second chapter** is devoted to the study of the physicochemical properties of various reagents necessary for conducting research and confirmation of their structures by spectral analysis methods, research methods, testing methods of conservation fluids in various aggressive environments, the working principle of the apparatus used to determine the protective effect against corrosion, and information on analysis methods for determining the

quality indicators of bitumen.

**The third chapter** is devoted to the physicochemical properties of synthesized amides, imidazolines, nitro compounds, metal salts of natural petroleum acids and other components used for conducting research, the anticorrosion properties of oils of various compositions and their application to conservation fluids.

**The fourth chapter** is devoted to the analysis of the results of the corrosion protection effect of the preservation fluids prepared on the basis of synthesized amides and imidazolines on metal plates. The results of the studies were analyzed comparatively and it was determined that the corrosion inhibitory effect of the synthesized amides, various metal salts, and nitro compounds in combination with these compounds is higher than the corrosion inhibitory effect of these compounds separately.

**The fifth chapter** is devoted to the synthesis of sulfate derivatives of natural petroleum acids, distilled petroleum acids, and distillates of natural petroleum acids, and the application of carbonate mineral powders as hydrophilizers. This chapter shows the physical and mechanical characteristics of asphalt-concrete mixtures prepared with activated and non-activated mineral powder obtained from limestone rock and their results.

It also presents the study of amides and imidazolines synthesized with polyamines of natural petroleum acids, their various compositions with mineral oil distillates as additives to road bitumen.

**In the sixth chapter**, a technical and economic assessment of the process of producing a preservation fluid based on T-30 oil distillate, a generalized scheme of the principle of obtaining preservation fluids, a technical and economic assessment was carried out for the proposed production process in terms of the duration of action and raw material costs. The change in the mass of bitumen after adding an additive to road bitumen and bitumen is given in comparison.

## MAIN CONTENT OF THE WORK

### Synthesis of amides based on Berolamine-20 and NPA and study of their physicochemical properties

Berolamine-20 consists of a mixture of triethylenetetramine, tetraethylenepentamine and amino-ethylethanolamine. The synthesis of amides based on NPA and BA-20 was carried out as follows, taking them in different molar ratios (1:1, 2:1, 3:1, 4:1, 5:1 and 6:1): a pre-calculated amount of natural petroleum acid was poured into a three-necked flask equipped with a heating stirrer, thermometer and separatory funnel and heated to 80-100°C with stirring. Then, at this temperature, BA-20 intended for synthesis is added to the acid in the flask. The reaction temperature is gradually brought to 140°C and the synthesis is continued with intensive stirring for 3-3.5 hours. After that, when the reaction is complete, the heater is turned off, the reaction product is cooled to 30- 40°C while continuing to stir, and transferred from the reaction flask to another tightly closed container.

The synthesized amides were analyzed and their physicochemical properties are as follows (table 1).

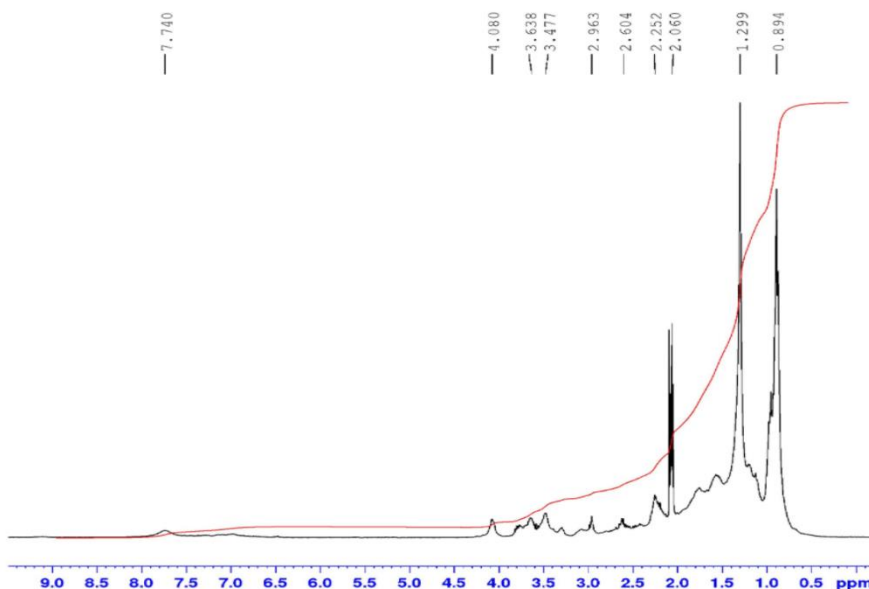
**Table 1.**

#### Physico-chemical properties of synthesized amides

№	Synthesized additives	Kinematic viscosity	Density 20°C, q/m <sup>3</sup>	Pour point, °C
		100°C		
1	Natural petroleum acid	41,696	0,984	-35
2	Berolamine BA-20	6,8622	1,071	-18
3	NPA: BA-20 1:1 mol ratio	104,235	1,061	+10
4	NPA: BA-20 2:1 mol ratio	102,021	1,058	+5
5	NPA: BA-20 3:1 mol ratio	99,307	1,052	+10
6	NPA: BA-20 4:1 mol ratio	98,285	1,049	+10
7	NPA: BA-20 5:1 mol ratio	70,085	1,047	+5
8	NPA: BA-20 6:1 mol ratio	61,091	1,029	+5

In the <sup>1</sup>H NMR (BRUKER-Fourier 300 MHs, Acetone-D<sub>6</sub>, δ, m.h.) spectrum of the sample (NPA.Ber. 6:1), the resonance signal of the methyl group was observed at 0.89 m.p., the methylene group (CH<sub>2</sub>) at 1.29 m.p., the hydrogen atoms belonging to the naphthene

rings at 1.5-2.1 m.p., the methylene groups of the CH<sub>2</sub>-N fragment at 2.10-4.08 m.p., the hydrogen atoms belonging to the aromatic nuclei at 6.5-7.5 m.p., and the NH group at 7.7 m.p. (Figure 1).



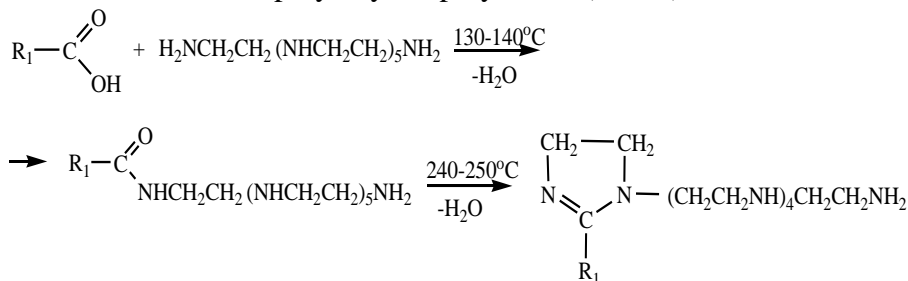
**Fig 1. <sup>1</sup>H NMR spectrum of amide obtained from the synthesis of berolamine-20 with natural petroleum acid.**

### **Synthesis of imidazolines and study of their physical and chemical properties**

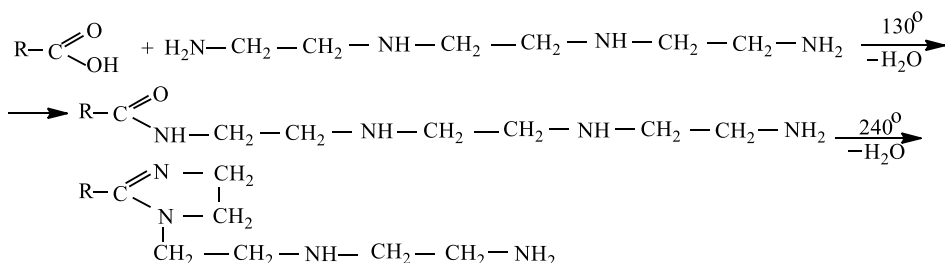
The reaction to obtain imidazolines from natural petroleum acids was carried out as follows: a pre-calculated amount of natural petroleum acid was poured into a reaction flask equipped with a heater, stirrer, thermometer and separatory funnel and heated to 80-100°C with stirring. At this temperature, the amine to be used (DETA, TETA, PEPA and Berolamine-20) was added dropwise to the acid. For amide synthesis, the temperature was raised to 140°C, and for imidazoline synthesis, it was raised to 240°C, and the reaction was carried out at this temperature with intensive stirring for 3-3.5 hours. After this period, heating was stopped, and stirring was continued until the temperature dropped to 50-60°C.

The preparation of imidazolines based on the interaction of NPA with polyamines proceeds according to the scheme shown below:

1 Scheme for the preparation of imidazoline from the reaction of NPA with polyethylenepolyamine (PEPA):



2. Scheme for the preparation of imidazoline from the reaction of NPA with triethylenetetraamine (TETA):



**Results of the study of compositions of amides synthesized on the basis of Berolamine-20, prepared together with metal salts of NPA as additives to preservation fluids.**

In this section, preservation fluids were prepared by adding additive amides and various metal salts of natural petroleum acid to T-30, T-22, T-46 and T-1500 oil distillates, separately and in the form of a two-component composition, and the results were studied.

In recent years, continuous research has been conducted in this area at the IPCP and certain results have been obtained. Here, many oil compositions have been tested as solvent media. The oil distillates used in the study (T-22, T-30, T-46, T-1500, AK-15) were used.

It should also be noted that compared to other oil distillates, T-30 oil distillate was widely used in the implementation of the dissertation work, as it was more favorable in many respects.

In the preparation of preservation liquids, mainly high-molecular polymers were used as components.

Amides synthesized on the basis of NPA and Berolamine were named with the following codes.

1. “ABA-16” - amide synthesized from natural petroleum acid and Berolamine-20 in a 6:1 molar ratio

2. “ABA-15” - amide synthesized from natural petroleum acid and Berolamine-20 in a 5:1 molar ratio

3. “ABA-14” - amide synthesized from natural petroleum acid and Berolamine-20 in a 4:1 molar ratio

4. “ABA-13” - amide synthesized from natural petroleum acid and Berolamine-20 in a 3:1 molar ratio

5. “ABA-12” - amide synthesized from natural petroleum acid and Berolamine-20 in a 2:1 molar ratio

6. “ABA-11” - amide synthesized from natural petroleum acid and Berolamine-20 in a 1:1 molar ratio

The results of the tests of synthesized amides as preservation fluids in T-30 oil distillate medium are given in Table 2.

As can be seen from Table 2, the best results in different media were obtained by taking 90% T-30 turbine oil distillate and 10% synthesized amides (NPA: BA-20 in 6:1, 5:1 and 4:1 mole ratios). Thus, these samples (sample No. 3) with 5, 7 and 10% showed results in the “G-4” thermohumidity chamber for 102, 144, 177 days, in sea water for 43, 51, 69 days, and in 0.001% H<sub>2</sub>SO<sub>4</sub> medium for 42, 50, 68 days. In another sample (sample No. 4), the corrosion protection effect of the “Steel-3” brand metal plates of the conservation fluids prepared on the basis of amides synthesized in the molar ratios of NPA: BA-20 3:1, 2:1 and 1:1 was low (table 2, samples No. 5, 6 and 7).

When obtaining salts of natural petroleum acids with transition elements, a certain amount of water of hydration remains in the salt, which worsens its solubility in oil and, therefore, reduces its protective effect as a corrosion inhibitor. On the other hand, the water ligand enters the coordination system of the metal ion and weakens its connection with the metal surface.

**Table 2.**

**Test results of compositions prepared with metal salts of NPA of synthesized amides in T-30 oil distillate medium as preservation fluids**

№	Composition of samples	Total amount of inhibitor, %	Corrosion protection period, days		
			In the hydrochamber «Q-4»	In sea water	In 0,001% solution of H <sub>2</sub> SO <sub>4</sub>
1	T-30 oil distillate	100	34	15	9
2	T-30 oil distillate + “ABA-16”	5	95	36	37
		7	122	47	48
		10	132	57	56
3	T-30 oil distillate + “ABA-15”	5	102	43	42
		7	144	51	50
		10	177	69	68
4	T-30 oil distillate + “ABA-14”	5	105	46	46
		7	154	62	60
		10	185	81	80
5	T-30 oil distillate + “ABA-13”	5	82	30	31
		7	105	40	40
		10	115	47	46
6	T-30 oil distillate + “ABA-12”	5	70	27	25
		7	95	35	34
		10	110	42	41
7	T-30 oil distillate + “ABA-11”	5	65	25	24
		7	82	32	31
		10	97	40	38

Therefore, since the corrosion protection effect of metal salts and amides of natural petroleum acids (Steel-3) separately on metal plates is low, their combined compositions with different components were prepared and tested as preservation fluids.

The dependence of the test results of preservation fluids on the concentration of the inhibitor is expressed graphically below.

As can be seen from the studies conducted, the corrosion protection effect of metal plates of preservation fluids prepared on the basis of amides synthesized in the molar ratio of NPA:BA-20

(1:1 ÷ 3:1) is low and does not dissolve well in mineral oils. The corrosion protection effect of metal plates of preservation fluids prepared on the basis of amides synthesized in the molar ratio of NPA:BA-20 (4:1 ÷ 6:1) is high and since they dissolve well in mineral oils, these amides were used in the preparation of preservation fluids.

**Results of the study of preservation fluids prepared as additives based on amides synthesized on the basis of berolamine-20, metal salts of NPA and a nitro compound.**

After the addition of metal salts and amides of natural petroleum acids as components to mineral oils separately, complexes of this component with a nitro compound were prepared and added to mineral oils as inhibitors and tested as preservation fluids.

In order to add inhibitors to T-30, T-22, T-46 and T-1500 oil distillates, preservation fluids were prepared and tested in the form of compositions consisting of amides, various metal salts of natural petroleum acid, and components of a nitro compound (in a 1:1:1 molar ratio).

Thus, in this section, 15 compositions were prepared as follows, consisting of three components:

“CoABA16N” - 5.7 and 10% solution of amide (NPA:Berolamine 6:1 molar ratio) + NPA Co-salt + nitro compound composition;  
“CoABA15N” - 5.7 and 10% solution of amide (NPA:Berolamin 5:1 molar ratio) + NPA Co-salt + nitro compound composition;  
“CoABA14N” - 5.7 and 10% solution of amide (NPA:Berolamin 4:1 molar ratio) + NPA Co-salt + nitro compound composition;  
“NiABA16N” - 5.7 and 10% solution of amide (NPA:Berolamine 6:1 molar ratio) + NPA Ni-salt + nitro compound composition;  
“NiABA15N” - 5.7 and 10% solution of amide (NPA:Berolamin 5:1 molar ratio) + NPA Ni-salt + nitro compound composition;  
“NiABA14N” - 5.7 and 10% solution of amide (NPA:Berolamin 4:1 molar ratio) + NPA Ni-salt + nitro compound composition;  
“MnABA16N” - 5.7 and 10% solution of amide (NPA:Berolamine 6:1 molar ratio) + NPA Mn-salt + nitro compound composition;  
“MnABA15N” - 5.7 and 10% solution of amide (NPA:Berolamine



5:1 molar ratio) + NPA Mn-salt + nitro compound composition;  
 “MnABA14N” - 5.7 and 10% solution of amide (NPA:Berolamine  
 4:1 molar ratio) + NPA Mn-salt + nitro compound composition;  
 “ZnABA16N” - 5.7 and 10% solution of amide (NPA:Berolamine  
 6:1 molar ratio) + NPA Zn-salt + nitro compound composition;  
 “ZnABA15N” - 5.7 and 10% solution of amide (NPA:Berolamine  
 5:1 molar ratio) + NPA Zn-salt + nitro compound composition;  
 “ZnABA14N” - 5.7 and 10% solution of amide (NPA:Berolamine  
 4:1 molar ratio) + NPA Zn-salt + nitro compound composition;  
 “BaABA16N” - 5.7 and 10% solution of amide (NPA:Berolamine  
 6:1 molar ratio) + NPA Ba-salt + nitro compound composition;  
 “BaABA15N” - 5.7 and 10% solution of amide (NPA:Berolamine  
 5:1 molar ratio) + NPA Ba-salt + nitro compound composition;  
 “BaABA14N” - 5.7 and 10% solution of amide (NPA:Berolamine  
 4:1 molar ratio) + NPA Zn-salt + nitro compound composition.

Preservation fluids were prepared and tested by adding amides synthesized on the basis of natural petroleum acid, metal salts of natural petroleum acid and a nitro compound synthesized on the basis of  $\alpha$ -olefin as inhibitors to T-30 oil distillate. The results of the corrosion protection effect of these prepared preservation fluids on “Steel-3” brand metal plates in all three environments were studied. It was found that adding the synthesized inhibitors not separately, but in a joint composition to T-30 oil distillate led to a further increase in the protective effect of the prepared preservation fluids.

Accordingly, metal salts of synthesized natural petroleum acid, amides, and nitro compound in a composition of 10% were added to T-30 turbine oil and tested as preservation fluids and are given in Table 3 below.

As can be seen from Table 3, the protective effects of preservation fluids prepared based on the composition of metal salts of synthesized natural petroleum acid, amides, and a nitro compound product obtained from tetradecene-1, an  $\alpha$ -olefin, showed higher results.

**Table 3.**

**Test results of preservation fluids prepared by adding a composition of metal salts, amides, and nitro compounds of natural petroleum acid to T-30 oil distillate**

№	Compositions solution in T-30 oil distillate			Corrosion protection period, days		
	Composition	Amount of components in solution, %				
			Inhibitor	Solution	In the hydrochamber «Q-4»	In sea water
1	2	3	4	5	6	7
1	T-30 turbine oil distillate	0	100	34	15	9
	T-30 oil distillate + C14-based nitro derivative	10	10	108	47	28
2	T-30 turbine oil distillate + “CoABA16N”	1,67 1,67 1,67	5	172	72	70
		2,33 2,33 2,33	7	185	77	74
		3,33 3,33 3,33	10	202	85	85
3	T-30 turbine oil distillate + “CoABA15N”	1,67 1,67 1,67	5	185	64	61
		2,33 2,33 2,33	7	198	90	87
		3,33 3,33 3,33	10	220	102	101
4	T-30 turbine oil distillate + “CoABA14N”	1,67 1,67 1,67	5	190	78	76
		2,33 2,33 2,33	7	207	98	96
		3,33 3,33 3,33	10	235	115	114

Continuation of the table 3.

1	2	3	4	5	6	7
5	T-30 turbine oil distillate + “NiABA16N”	1,67 1,67 1,67	5	125	51	48
		2,33 2,33 2,33	7	131	55	53
		3,33 3,33 3,33	10	145	58	56
6	T-30 turbine oil distillate + “NiABA15N”	1,67 1,67 1,67	5	70	39	37
		2,33 2,33 2,33	7	92	44	41
		3,33 3,33 3,33	10	107	48	47
7	T-30 turbine oil distillate + “NiABA14N”	1,67 1,67 1,67	5	127	47	45
		2,33 2,33 2,33	7	140	57	54
		3,33 3,33 3,33	10	165	75	73
8	T-30 turbine oil distillate + “MnABA16N”	1,67 1,67 1,67	5	101	42	40
		2,33 2,33 2,33	7	110	47	45
		3,33 3,33 3,33	10	122	55	54
9	T-30 turbine oil distillate + “MnABA15N”	1,67 1,67 1,67	5	107	39	38
		2,33 2,33 2,33	7	115	51	50
		3,33 3,33 3,33	10	135	58	58

Continuation of the table 3.

1	2	3	4	5	6	7
10	T-30 turbine oil distillate + “MnABA14N”	1,67 1,67 1,67	5	124	45	41
		2,33 2,33 2,33	7	137	56	55
		3,33 3,33 3,33	10	148	72	71
11	T-30 turbine oil distillate + “ZnABA16N”	1,67 1,67 1,67	5	121	49	46
		2,33 2,33 2,33	7	138	58	57
		3,33 3,33 3,33	10	150	78	77
12	T-30 turbine oil distillate + “ZnABA15N”	1,67 1,67 1,67	5	151	68	64
		2,33 2,33 2,33	7	162	75	72
		3,33 3,33 3,33	10	181	83	82
13	T-30 turbine oil distillate + “ZnABA14N”	1,67 1,67 1,67	5	164	86	84
		2,33 2,33 2,33	7	177	81	80
		3,33 3,33 3,33	10	191	90	88
14	T-30 turbine oil distillate + “BaABA16N”	1,67 1,67 1,67	5	145	52	48
		2,33 2,33 2,33	7	171	78	76
		3,33 3,33 3,33	10	186	85	82

Continuation of the table 3.

1	2	3	4	5	6	7
15	T-30 turbine oil distillate + “BaABA15N”	1,67	5	177	75	71
		1,67				
		1,67				
		2,33	7	182	84	82
		2,33				
		2,33				
16	T-30 turbine oil distillate + “BaABA14N”	3,33	10	201	90	87
		3,33				
		3,33				
		1,67	5	182	84	82
		1,67				
		1,67				
16	T-30 turbine oil distillate + “BaABA14N”	2,33	7	190	88	85
		2,33				
		2,33				
		3,33	10	212	95	91
		3,33				
		3,33				

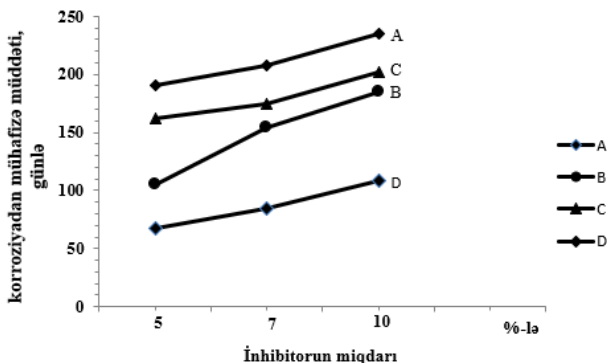
*Note:* A nitro derivative based on C<sub>14</sub> (tetradecene-1), an  $\alpha$ -olefin, was used as the nitrocompound product.

Thus, when these inhibitors were added separately to T-30 turbine oil in an amount of 10%, the corrosion protection effects of “polad-3” brand metal plates in different environments (“Q-4” thermo-humidity chamber, sea water and 0.001% H<sub>2</sub>SO<sub>4</sub> environment) were 135, 72, 70 days, 185, 81 and 80 days, while the composition of these inhibitors together with the nitro compound, taken in the same amount, showed results of 235, 115 and 114 days (table 3, sample No. 4).

If we look at Fig. 2, we can see this more clearly.

As can be seen from Fig. 2, the corrosion protection effect of the “Steel-3” brand metal plates of the preservation fluids prepared by adding the synthesized inhibitors separately to the T-30 oil distillate showed a lower result than the corrosion protection effect of the preservation fluids prepared based on the addition of these inhibitors in the form of a joint composition. Thus, the corrosion protection effect of the preservation fluids prepared based on the addition of 10% of the nitro compound product obtained on the basis of  $\alpha$ -olefin C<sub>14</sub> (teradecene-1) to the T-30 oil distillate in the “Q-4” thermohumidity chamber was 108 days (Fig. 2, curve D), while the

corrosion protection effect of the metal plates in the T-30 oil distillate with the same percentage of amide (NPA:BA-20 in a 4:1 mol ratio) was 185 days (Fig. 2, curve B).



**Fig. 2. Test results of preservation fluids prepared based on the addition of synthesized amides, natural petroleum acid Co salt, and nitro compound product separately and in a combined composition to T-30 oil distillate.**

D – C<sub>14</sub>-based nitro derivative + T-30 oil distillate

B – amide (NPA: BA-20 4:1 molar ratio) + T-30 oil distillate

C – NPA Co salt + T-30 oil distillate

A – amide (NPA: BA-20 4:1 molar ratio) + NPA Co salt + C<sub>14</sub>-based nitro derivative + T-30 oil distillate

However, the corrosion protection effect of preservation fluids prepared based on the combined composition of these inhibitors showed results of 202 (Fig. 2, curve C) and 235 days (Fig. 2, curve A).

The conducted studies show that it is more expedient to prepare synthesized inhibitors in the form of compositions. The high corrosion protection effect of preservation fluids prepared on the basis of these compositions on metal plates is explained by the fact that the compound can form a strong chemisorption bond by good physical adsorption on the metal surface due to the unused electron pair in the nitrogen atoms of the inhibitor molecule or partial protonation of the nitrogen atom. Accordingly, these preservation fluids create a coating-forming layer on the metal surface, which prevents water vapor from entering the metal surface.

The reason for using the nitration product of  $\alpha$ -olefin

(tetradecene-1) in the preparation of preservation fluids, along with metal salts and amides of synthesized natural petroleum acids, is that this nitro compound, being a stronger ligand than water, removes water molecules entering the metal surface. This is explained by the fact that these compositions are better chemisorbed on the metal surface and create a strong protective layer against corrosion.

The reason for the high corrosion protection effect of compositions prepared on the basis of synthesized nitro compounds, such as preservation fluids, on metal plates is the stability of the composition of these compounds. The reason for the stability of the chemical composition is that it has a linear structure and contains functional OH and NO<sub>2</sub> groups located in the  $\alpha$ -position from each other in its molecule. It should be noted that the functional groups in the composition of the nitro compound added to the mineral oil simultaneously slow down the corrosion processes at both the anode and cathode. Thus, one of these groups is of the anode type (NO<sub>2</sub><sup>-</sup> electron acceptor), and the other is of the cathode type (OH-electron donor), forming an adsorption film on the metal surface, making the surface hydrophobic and making it difficult to form a water film on the metal surface.

The test results of preservation fluids prepared by adding metal salts of natural petroleum acids, nitro compound and amide composition to T-22 oil distillate are shown below.

1. T-22 oil distillate 90% + “CoABA16N” 10% solution was kept in the “Q-4” thermohumidity chamber for 194 days, in seawater for 74 days, and in 0.001% H<sub>2</sub>SO<sub>4</sub> solution for 71 days.
2. T-22 oil distillate 90% + 10% solution of “CoABA15N” was kept in the “Q-4” thermohumidity chamber for 210 days, in seawater for 80 days, and in 0.001% H<sub>2</sub>SO<sub>4</sub> solution for 78 days.
3. T-22 oil distillate 90% + 10% solution of “CoABA14N” was kept in the “Q-4” thermohumidity chamber for 224 days, in seawater for 101 days, and in 0.001% H<sub>2</sub>SO<sub>4</sub> solution for 100 days.
4. T-22 oil distillate 90% + 10% solution of “NiABA16N” was kept in the “Q4” thermohumidity chamber for 132 days, in seawater for 50 days, and in 0.001% H<sub>2</sub>SO<sub>4</sub> solution for 47 days.
5. T-22 oil distillate 90% + 10% “NiABA15N” solution was kept in

the “Q-4” thermohumidity chamber for 96 days, in seawater for 41 days, and in 0.001%  $\text{H}_2\text{SO}_4$  solution for 41 days.

6. T-22 oil distillate 90% + 10% solution of “NiABA14N” was kept in the “Q-4” thermohumidity chamber for 151 days, in seawater for 70 days, and in 0.001%  $\text{H}_2\text{SO}_4$  solution for 68 days.

7. T-22 oil distillate 90% + 10% solution of “MnABA16N” was kept in the “Q-4” thermohumidity chamber for 111 days, in seawater for 49 days, and in 0.001%  $\text{H}_2\text{SO}_4$  solution for 48 days.

8. T-22 oil distillate 90% + 10% solution of “MnABA15N” was kept in the “Q-4” thermohumidity chamber for 122 days, in seawater for 51 days, and in 0.001%  $\text{H}_2\text{SO}_4$  solution for 50 days.

9. T-22 oil distillate 90% + 10% solution of “MnABA14N” was kept in the “Q-4” thermohumidity chamber for 133 days, in seawater for 66 days, and in 0.001%  $\text{H}_2\text{SO}_4$  solution for 63 days.

10. T-22 oil distillate 90% + 10% solution of “ZnABA16N” was kept in the “Q-4” thermohumidity chamber for 138 days, in seawater for 71 days, and in 0.001%  $\text{H}_2\text{SO}_4$  solution for 70 days.

11. T-22 oil distillate 90% + 10% solution of “ZnABA15N” was kept in the “Q-4” thermohumidity chamber for 173 days, in seawater for 78 days, and in 0.001%  $\text{H}_2\text{SO}_4$  solution for 77 days.

12. T-22 oil distillate 90% + 10% solution of “ZnABA14N” was stored in the “Q-4” thermohumidity chamber for 180 days, in seawater for 83 days, and in 0.001%  $\text{H}_2\text{SO}_4$  solution for 83 days.

13. T-22 oil distillate 90% + 10% solution of “BaABA16N” was stored in the “Q-4” thermohumidity chamber for 174 days, in seawater for 81 days, and in 0.001%  $\text{H}_2\text{SO}_4$  solution for 77 days.

14. T-22 oil distillate 90% + 10% solution of “BaABA15N” was stored in the “Q-4” thermohumidity chamber for 189 days, in seawater for 83 days, and in 0.001%  $\text{H}_2\text{SO}_4$  solution for 82 days.

15. T-22 oil distillate 90% + 10% “BaABA14N” solution was kept in the “Q-4” thermohumidity chamber for 204 days, in seawater for 91 days, and in 0.001%  $\text{H}_2\text{SO}_4$  solution for 91 days.

Preservation fluids prepared by adding 10% of the composition of synthesized cobalt salt of natural petroleum acid, amide and nitro compound to T-22 oil distillate have higher preservation capacity (inhibitor, “CoABA14N”). Thus, preservation fluids prepared by



adding 10% of this inhibitor to T-22 oil distillate showed results for 224, 101 and 100 days in the most aggressive “Q-4” thermo-humidity chamber, in seawater and in 0.001% H<sub>2</sub>SO<sub>4</sub> environment, respectively

. As a result of the conducted studies, it was determined that in mineral oils used as solvents, a synergistic effect is created between the cobalt salt of natural petroleum acid, amide (in a 4:1 mol ratio) and nitro compound, which in turn leads to a more than 2-fold increase in the corrosion protection effect of metals.

In addition, the study of various oils used in the preparation of preservation fluids showed that the test results of preservation fluids prepared on the basis of T-30 oil distillate are higher in all 3 environments, and the preparation of preservation fluids based on T-30 oil distillate is considered more appropriate.

The testing process was carried out in the experimental chamber called "Corrosionbox-1000E", one of the modern technological devices of the recent period, within the framework of existing standards. The tests were carried out in the experimental chamber under the specified parameters in two phases: the condensation phase and the environmental phase. In order to perform continuous testing in the experimental chamber, the standard parameters are adjusted using electronic devices.

Experimental research was carried out on the preservation fluids prepared by adding compositions based on synthesized amides and metal salts of natural petroleum acid to various oil distillates (T-22, T-30, T-46, T-1500) in different concentrations (5, 7 and 10%) in the "Corrosionbox-1000E" apparatus.

First, the results of tests on the compositions of amides synthesized in T-30 oil distillate medium and various metal salts of natural petroleum acid as preservation fluids were carried out in the "Corrosionbox-1000E" apparatus. The tests conducted are given in Table 6.

The results of the tests on the preservation fluids prepared on the basis of the composition of additives synthesized in T-30 oil distillate medium in the "Corrosionbox-1000E" apparatus in both environments (condensation phase and atmospheric or environmental

phase) showed that the preservation fluids prepared on the basis of the composition of various metal salts of natural petroleum acid with the synthesized amide of natural petroleum acid in a 4:1 molar ratio with Berolamine-20 showed higher results (Table 4).

**Table 4.**  
**Test results of compositions based on synthesized amides and metal salts of NPA as preservation fluids in the "Corrosionbox-1000E" apparatus**

№	Compositions solution in T-30 oil distillate			Corrosion protection period, days	
	Composition	Amount of components in solution, %		Condensation phase	Atmospheric phase
		Inhibitor	Solution		
1	"CoABA16"	5 5	10	268	312
2	"CoABA15"	5 5	10	292	345
3	"CoABA14"	5 5	10	310	365
4	"NiABA16"	5 5	10	251	296
5	"NiABA15"	5 5	10	271	325
6	"NiABA14"	5 5	10	287	335
7	"MnABA16"	5 5	10	205	262
8	"MnABA15"	5 5	10	258	302
9	"MnABA14"	5 5	10	270	322
10	"ZnABA16"	5 5	10	242	284
11	"ZnABA15"	5 5	10	265	317
12	"ZnABA14"	5 5	10	286	332
13	"BaABA16"	5 5	10	245	291
14	"BaABA15"	5 5	10	272	328
15	"BaABA14"	5 5	10	282	333

As in the previous tests of preservation fluids prepared on the basis of various metal salts of natural petroleum acid, in the

"Corrosionbox-1000E" apparatus, higher results were observed in preservation fluids prepared on the basis of the composition of the Co salt of natural petroleum acid among these salts.

As can be seen from Table 4, the highest results were obtained for preservation fluids prepared on the basis of the composition of NPA; Berolamine -20 in a 4:1 mol ratio and 10% of the Co salt of natural petroleum acid in T-30 oil distillate medium. The test results in both environments (condensation phase and atmospheric or environmental phase) were 310, 365 days consecutively (Table 4, sample No. 3).

### **Results of the study of compositions of imidazolines synthesized on the basis of Berolamine-20 and NPA with metal salts as additives to preservation fluids.**

In our subsequent research, imidazolines were also used as inhibitors in the preparation of preservation fluids. The synthesis of imidazolines was carried out by taking NPA with Berolamine-20 (BA-20) in different molar ratios (1:1, 2:1, 3:1 and 4:1).

The preservation fluids prepared with various oil distillates of the synthesized imidazolines are as follows:

1. "IBA11" - imidazoline synthesized on the basis of natural petroleum acid and Berolamine-20 in a 1:1 molar ratio
2. "IBA12" - imidazoline synthesized on the basis of natural petroleum acid and Berolamine-20 in a 2:1 molar ratio
3. "IBA13" - imidazoline synthesized on the basis of natural petroleum acid and Berolamine-20 in a 3:1 molar ratio
4. "IBA14" - imidazoline synthesized on the basis of natural petroleum acid and Berolamine-20 in a 4:1 molar ratio

Tests of the prepared preservation liquids were carried out in various environments in the "Q-4" thermo-humidity chamber, in sea water and in a 0.001% H<sub>2</sub>SO<sub>4</sub> environment.

Preservation liquids were prepared by adding inhibitors prepared based on synthesized imidazolines to various oil distillates (T-22, T-30, T-46 and T-1500) at different concentrations of 5, 7 and 10%, and the test results are given in Table 5.

**Table 5.**  
**Test results of preservation fluids prepared by adding**  
**synthesized imidazolines as inhibitors to T-22, T-30, T-46 and**  
**T-1500 oil distillates**

№	Compositions solution in T-30(I), T-22 (II), T-46(III) and T-1500(IV) oil distillates		Corrosion protection period, days		
	Composition	Amount of inhibitor, %	In the hydrochamber «Q-4»	In sea water	In 0,001% solution of H <sub>2</sub> SO <sub>4</sub>
1	2	3	4	5	6
I	T-30 oil distillate + “IBA11”	5	62	21	19
		7	65	25	22
		10	72	31	29
I	T-30 oil distillate + “IBA12”	5	64	22	20
		7	68	27	26
		10	74	34	31
I	T-30 oil distillate + “IBA13”	5	63	24	23
		7	70	28	27
		10	79	36	33
I.	T-30 oil distillate + “IBA14”	5	67	27	24
		7	80	36	34
		10	90	43	42
II	T-22 oil distillate + “IBA11”	5	40	17	14
		7	61	21	20
		10	71	28	26
II	T-22 oil distillate + “IBA12”	5	51	19	17
		7	63	23	20
		10	72	28	26
II	T-22 oil distillate + “IBA13”	5	64	22	21
		7	67	26	25
		10	71	32	30
II	T-22 oil distillate + “IBA14”	5	63	24	22
		7	70	28	26
		10	77	35	34
III	T-46 oil distillate + “IBA11”	5	34	14	13
		7	45	17	15
		10	63	23	22
III	T-46 oil distillate + “IBA12”	5	44	17	16
		7	50	19	15
		10	64	25	29
III	T-46 oil distillate + “IBA13”	5	60	21	20
		7	62	23	21
		10	65	27	26

Continuation of the table

1	2	3	4	5	6
III	T-46 oil distillate +“iBA14”	5	64	24	22
		7	66	26	24
		10	70	32	30
IV	T-1500 oil distillate +“iBA11”	5	38	11	10
		7	41	14	12
		10	46	19	18
IV	T-1500 oil distillate +“iBA12”	5	40	14	12
		7	43	16	15
		10	51	21	20
IV	T-1500 oil distillate +“iBA13”	5	44	17	15
		7	47	19	18
		10	63	23	21
IV	T-1500 oil distillate +“iBA14”	5	47	18	17
		7	50	21	19
		10	67	25	24

As can be seen from Table 5, the results of the tests of preservation fluids prepared on the basis of synthesized imidazolines and oils of various compositions in various mole ratios of NPA:Berolamine (1;1, 2;1, 3;1 and 4;1) in the “Q-4” thermo-humidity chamber, in seawater and in 0.001% H<sub>2</sub>SO<sub>4</sub> solution showed that the highest result was obtained in preservation fluids prepared with the addition of 10% of synthesized imidazoline in T-30 oil distillate in a molar ratio of NPA:Berolamine 4:1. Thus, the results in the “Q-4” thermo-humidity chamber, in seawater and in 0.001% H<sub>2</sub>SO<sub>4</sub> solution were 90, 43 and 42 days, respectively.

In this section, preservation fluids containing imidazolines and various metal salts of natural petroleum acid, two-component compositions were prepared and tested to be added as additives to T-30, T-22, T-46 and T-1500 oil distillates, and the results were studied.

The following 20 compositions of two components consisting of synthesized imidazolines, metal salts of natural petroleum acid and two-component compositions were prepared:

“CoİBA11” - 10% solution of composition imidazoline (NPA:Berolamine in a 1:1 molar ratio) + Co-salt of natural petroleum acid;

“CoIBA12” - 10% solution of composition imidazoline (NPA:Berolamine in a 2:1 molar ratio) + Co-salt of natural petroleum acid;

“CoIBA13” - 10% solution of composition imidazoline (NPA:Berolamine in a 3:1 molar ratio) + Co-salt of natural petroleum acid;

“CoIBA14” - 10% solution of composition imidazoline (NPA:Berolamine in a 4:1 molar ratio) + Co-salt of natural petroleum acid;

“NiIBA11” - 10% solution of composition imidazoline (NPA:Berolamine in a 1:1 molar ratio) + Ni-salt of natural petroleum acid;

“NiIBA12” - 10% solution of composition imidazoline (NPA:Berolamine in a 2:1 molar ratio) + Ni-salt of natural petroleum acid;

“NiIBA13” - 10% solution of composition imidazoline (NPA:Berolamine in a 3:1 molar ratio) + Ni-salt of natural petroleum acid;

“NiIBA14” - 10% solution of composition imidazoline (NPA:Berolamine in a 4:1 molar ratio) + Ni-salt of natural petroleum acid;

“MnIBA11” - 10% solution of composition imidazoline (NPA:Berolamine in a 1:1 molar ratio) + Mn-salt of natural petroleum acid;

“MnIBA12” - 10% solution of composition imidazoline (NPA:Berolamine in a 2:1 molar ratio) + Mn-salt of natural petroleum acid;

“MnIBA13” - 10% solution of composition imidazoline (NPA:Berolamine in a 3:1 molar ratio) + Mn-salt of natural petroleum acid;

“MnIBA14” - 10% solution of composition imidazoline (NPA:Berolamine in a 4:1 molar ratio) + Mn-salt of natural petroleum acid;

“ZnIBA11” - 10% solution of composition imidazoline (NPA:Berolamine in a 1:1 molar ratio) + Zn-salt of natural petroleum acid;

“ZnĪBA12”- 10% solution of composition imidazoline (NPA:Berolamine in a 2:1 molar ratio) + Zn-salt of natural petroleum acid;

“ZnĪBA13” - 10% solution of composition imidazoline (NPA:Berolamine in a 3:1 molar ratio) + Zn-salt of natural petroleum acid;

“ZnĪBA14” - 10% solution of composition imidazoline (NPA:Berolamine in a 4:1 molar ratio) + Zn-salt of natural petroleum acid;

“BaĪBA11”- 10% solution of composition imidazoline (NPA:Berolamine in a 1:1 molar ratio) + Ba-salt of natural petroleum acid;

“BaĪBA12 - 10% solution of composition imidazoline (NPA:Berolamine in a 2:1 molar ratio) + Ba-salt of natural petroleum acid;

“BaĪBA13 - 10% solution of composition imidazoline (NPA:Berolamine in a 3:1 molar ratio) + Ba-salt of natural petroleum acid;

“BaĪBA14- 10% solution of composition imidazoline (NPA:Berolamine in a 4:1 molar ratio) + Ba-salt of natural petroleum acid.

Research work was carried out on preservation fluids prepared on the basis of synthesized imidazolines and metal salts of natural petroleum acid with the addition of turbine oil distillate (T-30) in various concentrations (5, 7 and 10%).

As in the preservation fluids prepared on the basis of the composition of synthesized amides with various metal salts of natural petroleum acid, the highest results in preservation fluids prepared on the basis of imidazolines were observed in the NPA:Berolamine 4:1 mol ratio and in the composition of the Co salt of natural petroleum acid. Thus, with the addition of these additives, “Q-4” showed results of 121, 51 and 50 days in the thermohumidity chamber, in sea water and in a 0.001% H<sub>2</sub>SO<sub>4</sub> solution, respectively.

**Results of the study of compositions prepared with amides synthesized on the basis of Berolamine-20 together with other components as additives to conservation lubricants.**

In the modern era of rapid development of technology, one of the conditions for the delivery of technological devices and metal equipment with a wide range of applications to the consumer is their protection from atmospheric corrosion. One of the most effective methods for protection is the application of conservation lubricants. While conservation fluids are mainly used to protect metal-containing parts, spare parts and interior parts of cars from corrosion, conservation lubricants are mainly used to protect devices and equipment during storage and delivery.

Currently, the protection of equipment from atmospheric corrosion during operation and idle periods is one of the most urgent problems in our Republic. To overcome this problem, it is important to create new conservation fluids and lubricants.

The formation of synergism effect and increase of coating forming ability in compounds used for creation of new high-performance lubricant compositions largely depend on functional groups and heteroatoms in these compounds, as well as on their intermolecular interaction. Inhibitory properties of these compounds depend on adsorption and chemisorption processes on the protected surface, electron density in the adsorption center of functional groups and strong bond of protective film formed on the surface with metal. Therefore, in order to protect metals from atmospheric corrosion more effectively, these properties were taken into account in the preparation of conservation lubricants, as in conservation liquids.

To prepare conservation lubricants, 10% (mass) paraffin was added to a three-necked flask containing a pre-calculated amount of conservation liquid and mixed by heating at 100°C for 60 minutes. The prepared conservation lubricants were tested on the surface of previously polished and cleaned metal plates by 2 methods (rubbing and liquid method). The preservation lubricants were applied to the surface of the metal plates by rubbing them in a thickness of 1-2 mm. With the liquid method, the preservation lubricants were heated to 100°C and completely melted, and then the metal plates were



immersed in these preservation lubricants for 5 minutes, then removed and experimental research was conducted in various environments.

For many years, Azerbaijani scientists have developed a technology for the production of a number of valuable lubricants based on oil distillates obtained from Baku oils and created effective additive compositions suitable for these oils. In this direction, researchers have developed optimal ways for the targeted synthesis of chemicals that improve the quality indicators of lubricants and extend their service life. However, the creation of lubricants with high performance properties based on corrosion inhibitors with multifunctional compositions remains relevant.

First, we prepared and tested conservation lubricants with the addition of 10, 15% of inhibitor-free paraffin to T-30 and T-22 oil distillates, which are used as solvents in the preparation of conservation lubricants (table 6).

**Table 6.**

**Test results of conservation lubricants based on T-30 and T-22 oil distillates and paraffin**

№	Cod of samples	Amount of paraffin, %	Corrosion protection period, days			
			Condensation phase		Atmospheric phase	
			Liquid method	Rubbing method	Liquid method	Rubbing method
1	T-30 oil distillate	10	55	62	70	82
		15	70	77	85	94
2	T-22 oil distillate	10	47	56	64	75
		15	63	66	75	85

The paraffin used is solid paraffin, which corresponds to the “C” brand given in GOST 23683-89 and is used for technical purposes in industry. The prepared lubricant compositions were tested in the “CORROSIONBOX-1000E” experimental chamber by two methods (friction and liquid method).

As can be seen from Table 6, the corrosion protection effects of metal plates in both oil distillates are low. Therefore, the addition of highly effective corrosion inhibitors to the oil distillates used as solvents is mandatory.

In our previous research, we prepared and tested preservation fluids by adding the compositions to T-30 oil distillate in amounts of 5, 7 and 10% (Table 2). In the research, we found that the preparation of preservation fluids based on these compositions has a corrosion protection effect on metal plates in all three environments. Therefore, we decided to develop and test conservation lubricants based on these compositions.

Accordingly, conservation lubricants were prepared and tested by adding metal salts and amides of natural petroleum acid, nitro derivatives and paraffin as corrosion inhibitors to various brands of oils produced at the H. Aliyev Oil Refinery.

In the preparation of conservation lubricants, Co and Ba salts of natural petroleum acid, which showed the highest results as conservation fluids, were used. Conservation lubricants were prepared by taking T-30 turbine oil as a solvent, metal salts of natural petroleum acid, amide in an amount of 10% and paraffin in an amount of 5, 10, 15%.

The coded names of conservation lubricants prepared on the basis of a binary component of the composition of synthesized amides and metal salts are as follows:

“CoABAS16” – 10% solution composition of amide synthesized in a 6:1 molar ratio based on natural petroleum acid and Berolamine-20 + Co-salt of natural petroleum acid;

“CoABAS15” – 10% solution composition of amide synthesized in a 5:1 molar ratio based on natural petroleum acid and Berolamine-20 + Co-salt of natural petroleum acid;

“CoABAS14” – 10% solution of composition of amide synthesized in a 4:1 molar ratio based on natural petroleum acid and Berolamine-20 + Co-salt of natural petroleum acid;

“BaABAS16” – 10% solution composition of amide synthesized in a 6:1 molar ratio based on natural petroleum acid and Berolamine-20 + Ba-salt of natural petroleum acid;

“BaABAS15” – 10% solution composition of amide synthesized in a 5:1 molar ratio based on natural petroleum acid and Berolamine-20 + Ba-salt of natural petroleum acid;

“BaABAS14” – 10% solution composition of amide

synthesized in a 4:1 molar ratio based on natural petroleum acid and Berolamine-20 + Ba-salt of natural petroleum acid.

The prepared conservation lubricants were tested in the "Corrosionbox-1000E" apparatus. It was determined that the conservation lubricants prepared by taking the synthesized amide (NPA:BA-20, 4:1 mol ratio), natural petroleum acid with Co salt in a 10% amount, and paraffin in an amount of 5, 10 and 15%, had a higher corrosion protection effect on metal plates of the "Steel-3" brand. Thus, the test results of these conservation lubricants in the condensation phase showed results of 302, 318 and 332 days.

If we compare the experimental and research studies of the prepared conservation lubricants, we will see that the conservation lubricants prepared on the basis of T-30 oil distillate showed a higher corrosion protection effect on metal plates. For example, in conservation lubricants prepared on the basis of T-22 oil distillate, the conservation lubricant prepared with the addition of 15% paraffin to its composition, which showed the highest results, showed a result of 323 days in the condensation phase, while the conservation lubricants prepared on the basis of T-30 oil distillate of these compositions showed a higher result of 350 days.

As a result of the study of conservation fluids, we found that the protective effect of conservation fluids prepared on the basis of the composition of synthesized metal salts and amides of natural petroleum acid with a nitro compound product was higher. Based on these studies, we decided to prepare conservation lubricants based on three components. In the preparation of conservation lubricants, Co and Ba salts of natural petroleum acid, synthesized amides and nitro compound product were added to T-30 and T-22 oil distillates in an amount of 10%, and paraffin content was 5, 10 and 15%, and conservation lubricants were prepared and tested.

The coded names of conservation lubricants prepared on the basis of the ternary component composition of synthesized amides, metal salts and nitro compound product are as follows:

"CoABAS16N" – 5, 10, 15% solution of composition of amide synthesized in a 6:1 molar ratio based on natural petroleum acid and Berolamine-20 + Co-salt of natural petroleum acid + nitro compound

“CoABAS15N” – 5, 10, 15% solution of composition of amide synthesized in a 5:1 molar ratio based on natural petroleum acid and Berolamine-20 + Co-salt of natural petroleum acid + nitro compound

“CoABAS14N” – 5, 10, 15% solution of composition of amide synthesized in a 4:1 molar ratio based on natural petroleum acid and Berolamine-20 + Co-salt of natural petroleum acid + nitro compound

“BaABAS16N” – 5, 10, 15% solution of composition of amide synthesized in a 6:1 molar ratio based on natural petroleum acid and Berolamine-20 + Ba-salt of natural petroleum acid + nitro compound

“BaABAS15N” – 5, 10, 15% solution of composition of amide synthesized in a 5:1 molar ratio based on natural petroleum acid and Berolamine-20 + Ba-salt of natural petroleum acid + nitro compound

“BaABAS14N” – 5, 10, 15% solution of composition of amide synthesized in a 4:1 molar ratio based on natural petroleum acid and Berolamine-20 + Ba-salt of natural petroleum acid + nitro compound

The results of the tests of the prepared conservation lubricants in the condensation and atmospheric phases were studied in the apparatus "Corrosionbox-1000E". The lubricant obtained by adding 15% paraffin to these compositions (Co salt of natural petroleum acid + amide (4:1) and nitro compound product -10%) showed results of 360 days in the condensation phase by the liquid method, 377 days by the rubbing method, and 455 days by the liquid method in the atmospheric phase, and 467 days by the rubbing method.

The preparation of conservation lubricants based on the compositions that showed the highest results and their test results showed that it is advisable to prepare conservation lubricants based on these compositions.

Looking at the indicators obtained as a result of the tests, it can be seen that in all three cases, very high results were achieved in the new compositions prepared as preservation fluids in the “Q-4” hydrochamber. Even at a 5% concentration of the inhibitor, the results obtained met the requirements. As in the preservation fluids, the change in the molar ratio of petroleum acid to polyethylenepolyamine, berolamine in the composition of the amides used in the lubricants had an effect on the results. In these studies, higher results were achieved based on the triple component of NPA

to PEPA, Berolamine-20 in different molar ratios, metal salts of natural petroleum acid and the nitro compound product. In this case, the results of the studies in all three environments fully met the requirements.

The high results of the obtained preservation fluids and lubricants can also be seen from the comparative analysis of their foreign analogues (Table 7).

**Table 7.**

**Comparative test results of purchased conservation fluids and lubricants with known foreign analogues**

№	Konservasiya mayeləri və sürtkülərinin şərti adları		Corrosion protection in various harsh conditions	
			«Г-4» hydrocamera	Sea water
1.	Analog	Mayakor	100	90
2.		K-17	70	30
3.		Kormin	200	105
4.		NQ-203R	100	44
5.		Mifol KM	70	30
Konservasiya mayeləri				
6.	New ingredients	T-22 oil distillate+ «CoABA14N»	224	101
7.		T-30 oil distillate + «CoABA14N»	235	115
		Conservation lubricants	«Corrosionbox- in the apparatus 1000E»	
			Condensation phase	Atmospheric phase
8.		«CoABAS14»	350	390
9.		«CoABASN14N»	377	467

As can be seen from Table 7, the results of the studies are much higher than those of the corresponding analogues. Thus, while the highest result of the analogue conservation fluid called "Kormin" in the "G-4" hydrochamber was 200 days, the conservation fluid obtained as a result of our research showed a corrosion protection effect for 235 days. In the conservation lubricant, this result was 377 days in the higher condensation phase and 467 days in the atmospheric phase.

## **Study of amides synthesized with NPA and Berolamine-20 and mineral oil distillates as an additive to road bitumen**

In order to improve the quality indicators of road bitumen produced at Baku “Azerneftlyagh” Oil Refinery, amides with Berolamine-20 (BA-20) and polyethylene polyamine were synthesized in laboratory conditions based on natural petroleum acids, cubic residue of distillates of natural petroleum acids and distilled natural petroleum acids and were applied by adding them as an additive to road bitumen imported from “Azerneftlyagh” Oil Refinery.

For this purpose, natural petroleum acids, cubic residue of distillates of natural petroleum acids, and amides of distilled natural petroleum acids with Berolamine-20 (BA-20) and polyethylenepolyamine were synthesized in laboratory conditions and added as additives to road bitumen in various concentrations (0.4, 0.6% and 1%), and the quality indicators of bitumen were checked.

Addition of synthesized amides to road bitumen in an amount of 0.4% is given in Table 8.

**Table 8.**

**Quality indicators of road bitumen with the addition of 0.4% synthesized amides**

N	Addition of synthesized additives to road bitumen, amides	Softening temperature, °C	Needle insertion depth at 25°C	Stretch, cm	Brittleness temperature, °C	Adhesion
				25°C		With
1	Road bitumen	48	48	75	-18	3
2	“ABA16”	45	55	62,59	-26	1
3	“ABA15”	48	53	63	-24	1
4	“ABA14”	47,6	54	96,59	-25	1
5	“ABA13”	48	52	80	-24	2
6	“ABA12”	48,2	52	62,5	-23	1
7	“ABA11”	49,2	51	50	-19	1

Testing the quality indicators of amides synthesized on the basis of berolamine (BA-20) as additives added to road bitumen in amounts of 0.4 and 1% showed that these additives have a positive effect on the quality of road bitumen, including significantly improving adhesion.

From the given indicators it is clear that as a result of the test conducted according to GOST 11508-74, the proposed amide in the amount of 0.4 and 1% provides good adhesion of bitumen with river gravel. This adhesion is measured by 1 point. Bitumen, however, exhibits low thermostability in the same measurements, its adhesion is equal to 3 points (Fig. 3, 4).



**Fig. 3. Test result of road bitumen according to GOST 11508 adhesion 3 points.**



**Fig. 4. Test result with the addition of 0.4% of the synthesized additive to road bitumen adhesion 1 point.**

As can be seen, some of the amides synthesized in different molar ratios of natural petroleum acids and berolamine have a positive effect on the quality indicators of bitumen. For example, when NPA:Berolamine (in molar ratios of 6:1, 5:1, 4:1, 2:1 and 1:1) was added to bitumen in an amount of 0.4 and 1%, the adhesion of bitumen changed from 3 points to 1 point. This means that the adhesion of bitumen improves after the addition of the amide and significantly increases its thermostability. In particular, the amide synthesized in a molar ratio of NPA:Berolamine (BA-20) of this amide has a positive effect on the quality of bitumen and leads to a further improvement in the quality indicators of bitumen.

The amide synthesized by taking natural petroleum acid and Berolamine (BA-20) in a molar ratio of 4:1 was added to bitumen as the “V-2” additive and was tested in the laboratory at the “Azeryol Scientific Research Project” Institute. The results of laboratory tests of BNB 50/70 bitumen and bitumen with 0.4% “V-2” adhesion additive were studied. It was found that all indicators improved due to the addition of 0.4% “V-2” adhesion additive to bitumen.

In addition, since it is economically efficient, research was conducted by adding additives to synthesized amides using various oil distillates and adding them to road bitumen. Research was conducted by adding additives to road bitumen in different percentages (5, 10 and 15%) of synthesized amides with AK-15 oil distillates in amounts of 0.4 and 0.6%.

1. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA16” 95% + AK-15 oil 5% to road bitumen:

Additive in the amount of 0.4 %	Additive in the amount of 0.6%
$T_{\text{soft}} = 46,2^{\circ}\text{C}$	$T_{\text{soft}} = 46^{\circ}\text{C}$
$ID_{25} = 48 \times 0,1 \text{ mm}$	$ID_{25} = 49 \times 0,1 \text{ mm}$
$D_{25} = 62 \text{ sm}$	$D_{25} = 65 \text{ sm}$
$T_{\text{br}} = -17^{\circ}\text{C}$	$T_{\text{br}} = -18^{\circ}\text{C}$
Adhesion = 2	Adhesion = 2

2. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA16” 90% + AK-15 oil 10% to road bitumen:

Additive in the amount of 0.4 %	Additive in the amount of 0.6%
$T_{\text{soft}} = 46,2^{\circ}\text{C}$	$T_{\text{soft}} = 46,5^{\circ}\text{C}$
$ID_{25} = 52 \times 0,1 \text{ mm}$	$ID_{25} = 52 \times 0,1 \text{ mm}$
$D_{25} = 72,4 \text{ cm}$	$D_{25} = 73 \text{ cm}$
$T_{\text{br}} = -18^{\circ}\text{C}$	$T_{\text{br}} = -23^{\circ}\text{C}$
Adhesion = 1	Adhesion = 1

3. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA16” 85% + AK-15 oil 15% to road bitumen:

Additive in the amount of 0.4 %	Additive in the amount of 0.6%
$T_{\text{soft}} = 46,1^{\circ}\text{C}$	$T_{\text{soft}} = 46,6^{\circ}\text{C}$
$ID_{25} = 54 \times 0,1 \text{ mm}$	$ID_{25} = 54 \times 0,1 \text{ mm}$
$D_{25} = 80 \text{ cm}$	$D_{25} = 81 \text{ cm}$
$T_{\text{br}} = -21^{\circ}\text{C}$	$T_{\text{br}} = -23^{\circ}\text{C}$
Adhesion = 2	Adhesion = 1

4. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA16” 95% + AK-15 oil 5% to road bitumen:

Additive in the amount of 0.4 %	Additive in the amount of 0.6%
$T_{\text{soft}} = 46,4^{\circ}\text{C}$	$T_{\text{soft}} = 47^{\circ}\text{C}$



$\dot{ID}_{25} = 48 \times 0,1 \text{ mm}$

$D_{25} = 87,6 \text{ cm}$

$T_{br} = -24 \text{ }^{\circ}\text{C}$

Adhesion = 2

$\dot{ID}_{25} = 49 \times 0,1 \text{ mm}$

$D_{25} = 80 \text{ cm}$

$T_{br} = -19 \text{ }^{\circ}\text{C}$

Adhesion = 2

5. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA15” 90% + AK-15 oil 10% to road bitumen:

Additive in the amount of 0.4 %

$T_{soft} = 46,2^{\circ}\text{C}$

$\dot{ID}_{25} = 51 \times 0,1 \text{ mm}$

$D_{25} = 88 \text{ cm}$

$T_{br} = -22 \text{ }^{\circ}\text{C}$

Adhesion = 1

Additive in the amount of 0.6%

$T_{soft} = 46,4^{\circ}\text{C}$

$\dot{ID}_{25} = 51 \times 0,1 \text{ mm}$

$D_{25} = 81,9 \text{ cm}$

$T_{br} = -24 \text{ }^{\circ}\text{C}$

Adhesion = 1

6. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA15” 85% + AK-15 oil 15% to road bitumen:

Additive in the amount of 0.4 %

$T_{soft} = 45,9^{\circ}\text{C}$

$\dot{ID}_{25} = 52 \times 0,1 \text{ mm}$

$D_{25} = 89,91 \text{ cm}$

$T_{br} = -25 \text{ }^{\circ}\text{C}$

Adhesion = 2

Additive in the amount of 0.6%

$T_{soft} = 46,5^{\circ}\text{C}$

$\dot{ID}_{25} = 50 \times 0,1 \text{ mm}$

$D_{25} = 83,18 \text{ cm}$

$T_{br} = -24 \text{ }^{\circ}\text{C}$

Adhesion = 2

7. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA14” 95% + AK-15 oil 5% to road bitumen:

Additive in the amount of 0.4 %

$T_{soft} = 48^{\circ}\text{C}$

$\dot{ID}_{25} = 49 \times 0,1 \text{ mm}$

$D_{25} = 88,85 \text{ cm}$

$T_{br} = -20 \text{ }^{\circ}\text{C}$

Adhesion = 1

Additive in the amount of 0.6%

$T_{soft} = 47,5^{\circ}\text{C}$

$\dot{ID}_{25} = 51 \times 0,1 \text{ mm}$

$D_{25} = 90 \text{ cm}$

$T_{br} = -21 \text{ }^{\circ}\text{C}$

Adhesion = 1

8. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA14” 90% + AK-15 oil 10% to road bitumen:

Additive in the amount of 0.4 %  
0.6%

$T_{soft} = 47,8^{\circ}\text{C}$

$\dot{ID}_{25} = 51 \times 0,1 \text{ mm}$

Additive in the amount of

$T_{soft} = 46^{\circ}\text{C}$

$\dot{ID}_{25} = 51 \times 0,1 \text{ mm}$

D<sub>25</sub> = 94 cm

T<sub>bt</sub> = -23 °C

Adhesion = 1

D<sub>25</sub> = 95cm

T<sub>br</sub> = -26 °C

Adhesion= 1

9. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA14” 85% + AK-15 oil 15% to road bitumen:

Additive in the amount of 0.4 %  
0.6%

T<sub>soft</sub>= 45,6°C

İD<sub>25</sub>= 51x0,1mm

D<sub>25</sub> = 98 cm

T<sub>br</sub> = -27 °C

Adhesion = 1

Additive in the amount of

T<sub>soft</sub>= 45,6°C

İD<sub>25</sub>= 51x0,1mm

D<sub>25</sub> = 98 cm

T<sub>br</sub> = -26 °C

Adhesion = 1

10. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA13” 95% + AK-15 oil 5% to road bitumen:

Additive in the amount of 0.4 %

T<sub>soft</sub>= 48°C

İD<sub>25</sub>= 49x0,1mm

D<sub>25</sub> = 64,76 cm

T<sub>br</sub> = -19 °C

Adhesion = 2

Additive in the amount of 0.6%

T<sub>soft</sub>= 47,6°C

İD<sub>25</sub>= 50x0,1mm

D<sub>25</sub> = 67cm

T<sub>br</sub> = -21 °C

Adhesion = 2

11. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA13” 90% + AK-15 oil 10% to road bitumen:

Additive in the amount of 0.4 %

T<sub>soft</sub>= 47,8°C

İD<sub>25</sub>= 51x0,1mm

D<sub>25</sub> = 66,7cm

T<sub>br</sub> = -20 °C

Adhesion = 1

Additive in the amount of 0.6%

T<sub>soft</sub>= 47,5°C

İD<sub>25</sub>= 51x0,1mm

D<sub>25</sub> = 70 cm

T<sub>br</sub> = -19 °C

Adhesion = 1

12. Quality indicators of bitumen with the addition of “ABA13” 85% + AK-15 oil 15% additive in the amount of 0.4% and 0.6% to road bitumen:

Additive in the amount of 0.4 %

T<sub>soft</sub>= 47,5°C

İD<sub>25</sub>= 51x0,1mm

D<sub>25</sub> = 68,6 cm

Additive in the amount of 0.6%

T<sub>soft</sub>= 47,2°C

İD<sub>25</sub>= 50x0,1mm

D<sub>25</sub> = 75cm

$T_{br} = -19\text{ }^{\circ}\text{C}$

Adhesion = 2

$T_{br} = -19\text{ }^{\circ}\text{C}$

Adhesion = 2

13. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA12” 95% + AK-15 oil 5% to road bitumen:

Additive in the amount of 0.4 %

$T_{soft} = 47,2^{\circ}\text{C}$

$\dot{ID}_{25} = 49 \times 0,1 \text{ mm}$

$D_{25} = 80 \text{ cm}$

$T_{br} = -22\text{ }^{\circ}\text{C}$

Adhesion = 2

Additive in the amount of 0.6%

$T_{soft} = 47^{\circ}\text{C}$

$\dot{ID}_{25} = 49 \times 0,1 \text{ mm}$

$D_{25} = 58 \text{ cm}$

$T_{br} = -20\text{ }^{\circ}\text{C}$

Adhesion = 2

14. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA12” 90% + AK-15 oil 10% to road bitumen:

Additive in the amount of 0.4%

$T_{soft} = 46,6^{\circ}\text{C}$

$\dot{ID}_{25} = 51 \times 0,1 \text{ mm}$

$D_{25} = 85 \text{ cm}$

$T_{br} = -23\text{ }^{\circ}\text{C}$

Adhesion = 2

Additive in the amount of 0.6%

$T_{soft} = 46,7^{\circ}\text{C}$

$\dot{ID}_{25} = 51 \times 0,1 \text{ mm}$

$D_{25} = 71,33 \text{ cm}$

$T_{br} = -21\text{ }^{\circ}\text{C}$

Adhesion = 2

15. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA12” 85% + AK-15 oil 15% to road bitumen:

Additive in the amount of 0.4 %

$T_{soft} = 46^{\circ}\text{C}$

$\dot{ID}_{25} = 52 \times 0,1 \text{ mm}$

$D_{25} = 84 \text{ cm}$

$T_{br} = -24\text{ }^{\circ}\text{C}$

Adhesion = 2

Additive in the amount of 0.6%

$T_{soft} = 46,7^{\circ}\text{C}$

$\dot{ID}_{25} = 52 \times 0,1 \text{ mm}$

$D_{25} = 77,32 \text{ cm}$

$T_{br} = -22\text{ }^{\circ}\text{C}$

Adhesion = 2

16. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA11” 95% + AK-15 oil 5% to road bitumen:

Additive in the amount of 0.4 %

$T_{soft} = 47^{\circ}\text{C}$

$\dot{ID}_{25} = 48 \times 0,1 \text{ mm}$

$D_{25} = 77 \text{ cm}$

$T_{br} = -19\text{ }^{\circ}\text{C}$

Adhesion = 2

Additive in the amount of 0.6%

$T_{soft} = 46,8^{\circ}\text{C}$

$\dot{ID}_{25} = 49 \times 0,1 \text{ mm}$

$D_{25} = 82 \text{ cm}$

$T_{br} = -21\text{ }^{\circ}\text{C}$

Adhesion = 2

17. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA11” 90% + AK-15 oil 10% to road bitumen:

Additive in the amount of 0.4 %	Additive in the amount of 0.6%
$T_{\text{soft}} = 46,4^{\circ}\text{C}$	$T_{\text{soft}} = 46,2^{\circ}\text{C}$
$ID_{25} = 51 \times 0,1 \text{ mm}$	$ID_{25} = 51 \times 0,1 \text{ mm}$
$D_{25} = 74 \text{ cm}$	$D_{25} = 84 \text{ cm}$
$T_{\text{br}} = -20^{\circ}\text{C}$	$T_{\text{br}} = -25^{\circ}\text{C}$
Adhesion = 2	Adhesion = 2

18. Quality indicators of bitumen with the addition of 0.4% and 0.6% of the additive “ABA11” 85% + AK-15 oil 15% to road bitumen:

Additive in the amount of 0.4 %	Additive in the amount of 0.6%
$T_{\text{soft}} = 46,2^{\circ}\text{C}$	$T_{\text{soft}} = 46^{\circ}\text{C}$
$ID_{25} = 52 \times 0,1 \text{ mm}$	$ID_{25} = 50 \times 0,1 \text{ mm}$
$D_{25} = 78 \text{ cm}$	$D_{25} = 86 \text{ cm}$
$T_{\text{br}} = -20^{\circ}\text{C}$	$T_{\text{br}} = -25^{\circ}\text{C}$
Adhesion = 2	Adhesion = 2

NPA:Berolamine-20 4:1 mol ratio of synthesized amides in an amount of 90% and AK-15 oil distillate in an amount of 10% and 15%, the additives added to the road bitumen further improve the quality indicators of the bitumen and bring its adhesion to 1 point. Thus, the quality indicators of the road bitumen without additives were, softening temperature  $48^{\circ}\text{C}$ , needle penetration depth  $48^{\circ}\text{C}$ , tensile strength 75cm, brittleness temperature  $-18^{\circ}\text{C}$ , and adhesion 3 points, while after the addition of the additive in an amount of 0.4%, the quality indicators of the bitumen increased further, softening temperature  $45.6^{\circ}\text{C}$ , needle penetration depth  $51^{\circ}\text{C}$ , tensile strength 98cm, brittleness temperature  $-27^{\circ}\text{C}$ , and adhesion reached 1 point.

Testing the quality indicators by adding amides and AK-15 oil distillates as additives to road bitumen in amounts of 0.4 and 0.6% showed that these additives have a positive effect on the quality of road bitumen. They significantly improve adhesion.

From the given indicators it is clear that as a result of the tests conducted according to GOST 11508, the proposed additives prepared by taking amides NPA:Berolamine (BA-20) in 6:1, 5:1, 4:1 and 3:1 mole ratios of 90% and AK-15 oil in 10% amount provide

good adhesion of bitumen with river gravel in the amount of 0.4 and 0.6%. In addition, the quality indicators of road bitumen based on the synthesized additives showed that the amide with NPA:Berolamine (BA-20) in 4:1 mole ratio has higher adhesion properties than AK-15 oil. (additive "ABA14" + AK-15 oil). This adhesion is measured by 1 point. Bitumen, however, exhibits low thermostability in the same dimensions, its adhesion (i.e. adhesion) is equal to 3 points.

Thus, it is recommended to use NPA:Berolamine (BA-20) in a 4:1 mol ratio and AK-15 oil, which are better additives than synthesized amide compounds, as an additive to bitumen.

Laboratory tests of asphalt concrete samples prepared with BNB 50/70 brand control bitumen and activated mineral powder, as well as V-2 adhesive bitumen and activated mineral powder provided by IPCP, were conducted at the “Azerelimitedqiqatlayiha” Institute. The test results are shown in Table 9 below.

**Table 9.**  
**Amount of materials in the asphalt-concrete mixture**

Materials	Control sample, %	Example (with V-2 adhesion additives), %
1. Crushed stone, fr.5-20 mm	51.0	51.0
2. Sand fr. 0-5 mm	9.0	9.0
3. Stonemasonry (otsev),fr 0-5mm	28.5	28.5
4. Mineral powder (IPCP)	6.0	6.0
5. Bitumen BNB 50/70	5.5	5.1+0.4 V- adhesion additives

When comparing the test results of asphalt concrete samples prepared with control bitumen and V-2 adhesion additive in Table 10, it is clear that the physical and mechanical indicators of the samples activated with adhesion additive and mineral powder are superior to those of the control sample.

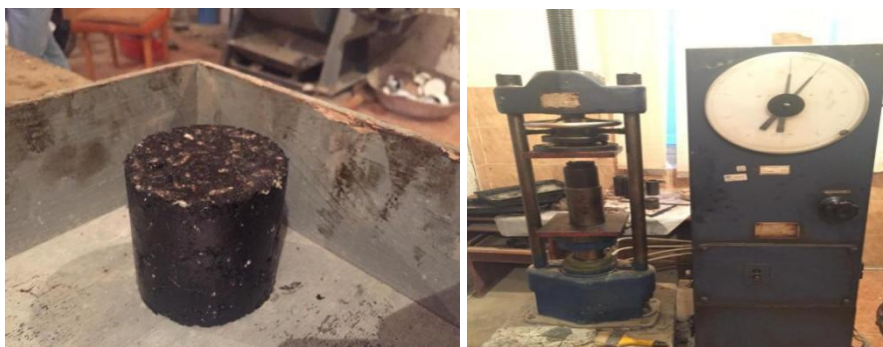
The activated mineral powder produced by IPCP passes 32% through a 0.071 mm sieve. It is recommended to conduct tests in production conditions to determine the effect of the V-2 additive added to bitumen and activated mineral powder (IPCP) on asphalt concrete.

**Table 10.**

**Physical and mechanical properties of samples molded from asphalt-concrete mixture**

Required and actual indicators	Medium density, q/cm <sup>3</sup>	Water saturation, %	Compressive strength limit, Mpa			Water resistance coefficient
			R <sup>vak</sup> <sub>20°C</sub>	R <sub>20°C</sub>	R <sub>50°C</sub>	
GOST9128-2013 requirements	2.20-2.40	2.0-5.0	-	>2.5	>1.1	>0.85
Control a/b example	2.32	3.34	2.64	3.1	1.1	0.85
V-2 adhesion additive a/b sample	2.32	3.25	2.67	3.1	1.1	0.86

Using the activator we synthesized and the control sample, asphalt concrete samples were prepared by adding a certain percentage of mineral powder to the “Azvirt” LLC and tested in laboratory conditions (Fig. 5).



**Fig. 5. Laboratory tests of asphalt concrete samples prepared with “V-2” adhesion additive at “Azvirt” LLC.**

In the first sample (1) - mineral powder was activated using distilled residue of petroleum acid.

In the second sample (2) - it was activated using the activator proposed by the Institute of Petrochemical Processes.

As can be seen from the samples, the activator of sample-2 has a good effect on the quality of bitumen and significantly improves the hydrophobicity of the mineral powder contained in the asphalt concrete coating. As can be seen from Table 10, the water resistance

coefficient of sample-1 was 0.89 in the control sample, and increased to 0.90 in sample-2.

We applied the composition of soapstock and natural petroleum acid brought from the Shirvan region as a hydrophilizer to  $\text{CaCO}_3$  mineral powders brought from the Garadagh cement plant.

Research was conducted by taking a composition prepared from 3% soapstock brought from Shirvan and 2% natural petroleum acid, heating it at 50-60°C, and adding it to a certain amount of mineral powders.

Tests of samples were carried out by adding 5% of this composition to activated mineral powder.

Results of asphalt-concrete mixtures prepared with activated and non-activated mineral powder obtained from limestone rock:

### 1. Amount of materials in the asphalt-concrete mixture:

Materials	Composition of the mixture prepared with BNB 50/70 viscous petroleum road bitumen produced at the Baku Oil Refinery named after H. Aliyev, %
1. Crushed stone, fr.5-20 mm	55,0
2. Sand fr. 0-5 mm	24,5
3. Stonemasonry (otsev), fr 0-5 mm	8,0
4. Mineral powder (IPCP)	7.0
5. Bitumen BNB 50/70	5.5

### 2. Physical and mechanical characteristics of samples molded from asphalt-concrete mixture:

Nümunələrin adları	Medium density, $\text{q/cm}^3$	Water saturation, %	Compressive strength limit, Mpa			Water resistance coefficient
			$R^{\text{vak}}_{20^\circ\text{C}}$	$R_{20^\circ\text{C}}$	$R_{50^\circ\text{C}}$	
GOST9128-2013 requirements	2.20-2.40	2.0-5.0	-	>2.5	>1.1	>0.85
Mineral powder (activated)	2.33	3.44	4,9	5,4	1.2	0.91
Mineral powder (not activated)	2.33	3.61	4,8	5,3	0,9	0.90

If we look at the above, we see that the physical and mechanical indicators of the samples molded from the mixture prepared with activated mineral powder have advantages over the samples prepared with non-activated powder.

As can be seen from the samples, activated mineral powder has a good effect on the quality of bitumen and significantly improves the hydrophobicity of the mineral powder contained in the asphalt concrete coating. As can be seen, the water resistance coefficient of non-activated mineral powder was 0.90, while the water resistance coefficient of activated mineral powder increased to 0.91. Based on the results obtained, it can be said that all physical and mechanical indicators of the mineral powder-2 and powder-3 samples are above the norm, and these mineral powders can be recommended for use in the preparation of asphalt concrete coatings.

### **Generalized principle technological scheme of processes**

Based on the positive results obtained, it is appropriate to organize large-scale production of preservation fluids and lubricants. This chapter provides a principle scheme of the process that can be established in production.

Natural petroleum acids No. 1 and 40% NaOH solution are fed from capacity No. 2 through pumps (n-1, n-2) in a pre-calculated amount to the reactor (R-1) and mixed with a mixer and heated to 40-50°C. After neutralization of natural petroleum acids, mixing is continued for 1-1.5 hours while maintaining the temperature. Then solvent No. 8 and a solution of salts in water are fed from capacity No. 3 through pumps (n-5, n-3) in a determined amount to the reactor (R-1) and mixing is continued for another 2 hours. After the stirring is stopped and the obtained product is cooled, the water-soluble white mineral part in the lower part of the reaction mixture is separated, and the dark hydrocarbon phase in the upper layer, i.e. the naphthenate salt taken for the reaction, is pumped with the same amount of mineral oil from tank No. 10 using a pump (n-4) and collected in tank No. 4. After this process, it is transferred from tank No. 4 to apparatus No. 5 to remove the solvent from the metal salt of the natural petroleum acids obtained.

The expelled solvent passes through condenser (cooler) No. 6 and is collected in container No. 7, then for reuse it is collected in container No. 8 through pump (n-5) and fed to reactor (R-1). The metal salt of natural petroleum acids expelled from the solvent is collected in container No. 9 and fed to reactor (R-4) through pump

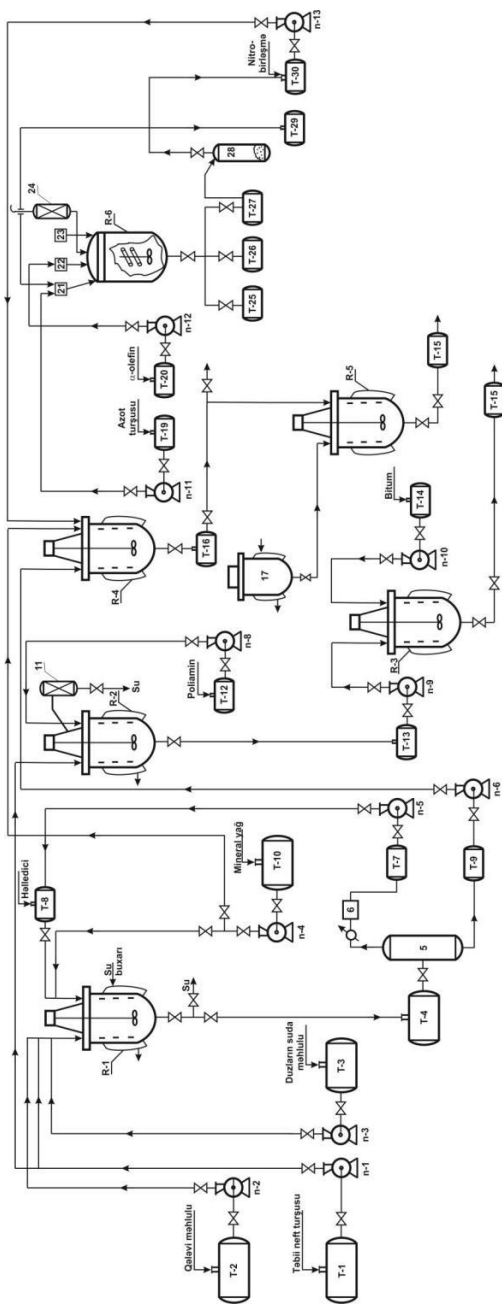


(n-6). A certain amount of natural petroleum acid is fed from container No. 1 to reactor (R-2) through pump (n-1) and heated to 80-100°C by mixing with a mixer.

Then, polyamine is gradually added to it from container No. 12 through pump (n-8) to bring the reaction temperature to 140°C. Stirring is continued for 3-3.5 hours while maintaining the temperature constant. During the reaction, the water phase is collected in container No. 11 and separated. The obtained additive is fed from tank No. 13 to the reactor (R-3) through a pump (n-9). Then, a certain amount of road bitumen is added from tank No. 14 to the reactor (R-3) through a pump (n-10), mixed with a mixer and heated to 80-100°C, and the mixing is continued for 1 hour. The road bitumen prepared by adding the additive is collected in tank No. 15 and put into use. The process of obtaining a nitro product from  $\alpha$ -olefin (tetradecene-1) and 61% nitric acid, which are used as raw materials, is carried out in reactor No. R-6.

Nitric acid and  $\alpha$ -olefin are collected in separate containers 19 and 20, from where they are fed to measuring containers 21 and 22 located above the reactor (R-6) by a vacuum pump. A specified amount of 57-61% nitric acid is charged to the reactor (R-6) from measuring container 21. 0.01 mol of  $\text{NaNO}_2$  is added to the nitric acid from container 23 so that  $\text{NO}_2$  is formed in the acid before adding olefin to the acid. In this case, the induction stage of the nitration process is eliminated, the conditions for additional processes such as auto-oxidation are reduced, and the reaction is facilitated.  $\text{NO}_2$  remains dissolved in the diluted nitric acid obtained after the initial process, and when used after mixing it with solid nitric acid and bringing it to 61%, there is no need to use  $\text{NO}_2$ . The nitric acid charged into the reactor is heated to 70°C and, with stirring, olefin is fed to it in portions from container No. 22 in such a way that the temperature does not rise above  $75 \pm 5^\circ\text{C}$ , since nitration at temperatures above 90°C is very dangerous.

Therefore, the reactor jacket is fed with boiling water, and cold water is fed to the serpentine tube inside, which allows maintaining the intended temperature regime in the reactor.



**Scheme. The principle scheme of the production technology of preservation liquids and lubricants, including additives to road bitumen:**

1- capacity for natural petroleum acid; 2- capacity for alkaline solution; 3- capacity for aqueous solution of salts; 4- capacity for the obtained inhibitor; 5- apparatus for removing the solvent from the obtained inhibitor; 6- condenser (cooler); 7- capacity for the removed solvent; 8- capacity for the solvent; 9- capacity for the inhibitor after removing the solvent; 10- capacity for mineral oil (I); 11- capacity for collecting the water phase separated during the reaction; 12- capacity for polyamine; 13- capacity for the obtained additive; 14- capacity for bitumen; 15- road bitumen prepared with the addition of additives; 16- capacity for the prepared preservation liquid; 17- apparatus for melting solid paraffin; 18- capacity for the prepared preservation lubricant; 19- capacity for nitric acid; 20- capacity for olefins; 21, 22- measuring capacity regulating the intermittent supply of nitric acid and  $\alpha$ -olefin to the reactor; 23- capacity for supplying water to the reactor; 24- apparatus for neutralizing the gas phase; 25, 26- capacity for collecting spent nitric acid; 27- capacity for collecting the nitro compound; 28- apparatus for drying the nitro compound; 29- capacity for collecting the neutralized gas phase; 30- capacity for collecting the dried nitro compound; R-1; R-2; R-3; R-4; R-5; R-6 – reactors;

The calculated amount of olefin is fed into the reactor and stirred at 70°C for one hour, then the reactor is cooled to 25±5°C and the mixer is stopped. At this time, the reaction mixture is separated into two phases, the lower part contains unreacted nitric acid, and the upper part contains the nitration product. The lower layer is sent to the intermediate storage tank No. 26 through the discharge valve. The nitration product remaining in the reactor is washed by adding a calculated amount of water from the water tank No. 24 within 15-20 minutes and kept for 30 minutes with the mixer stopped.

After the storage period is over, the washing water is collected in the tank No. 26, and the nitration product obtained in the upper phase is collected in the tank No. 25. In this process, the apparatus No. 24 is used to neutralize the gas phase and the neutralized gas phase is collected in the tank No. 29. Then, the obtained nitro product is sent to the apparatus No. 28 for drying, and after drying, it is collected in the tank No. 30.

After this process, to prepare multicomponent conservation fluids, amide obtained from tank No. 13 through pump (n-9), metal salts of natural petroleum acid from tank No. 9 through pump (n-6), nitro compound product from tank No. 30 through vacuum pump (n-13) are added in a calculated amount to the R-4 reactor and mixed with a mixer, heated to a temperature of 50-60°C, and mineral oil (I) is added from tank No. 10 through pump (n-4), and mixing continues at this temperature for 30 minutes. Then, the prepared multicomponent conservation fluid is collected in tank No. 16.

After the conservation fluid is prepared, one part is used as a conservation fluid, and the other part is fed to the reactor (R-5) to prepare conservation lubricants. Solid paraffin is heated with steam in reactor No. 17 and fed to the R-5 reactor in a calculated amount and mixed for 1 hour by heating to 70-80°C. Then, the prepared conservation lubricant is collected in container No. 18 and put into use.

## CONCLUSION

1. The compositions of amides and imidazolines synthesized on the basis of NPA and Berolamine-20, metal salts of NPA with nitro compounds of various compositions were added to T-22, T-30, T-46 and T-1500 oil distillates in amounts of 5, 7 and 10% as corrosion inhibitor components, and conservation fluids were prepared, and tests were conducted in the “Q-4” hydrochamber, in 0.001%  $\text{H}_2\text{SO}_4$  solution and seawater environments. The results obtained were recommended for application in industry based on the indicators of the tests conducted at “Baku Steel Company” LLC (the Act was submitted) [2, 3, 16, 22, 24, 26].

2. The conservation fluids prepared with the participation of amides and imidazolines obtained from the interaction of NPA and Berolamine-20 were tested in 2 phases, namely condensation and atmospheric phases, in the “CORROSIONBOX-1000E” experimental chamber. It was determined that when a composition containing a synthesized amide + Co salt of NPA + nitro compound in a 1:4 molar ratio of BA-20:TNT was added as a corrosion inhibitor component, the protection period was 327 days in the condensation phase and 422 days in the atmospheric phase [6, 13, 14, 15, 18, 21, 25].

3. Conservation fluids prepared on the basis of amides synthesized by reacting NPA with Berolamine-20 in different molar ratios (1÷6) were tested as conservation lubricants by adding paraffin. The highest results were obtained in compositions containing 10% (amide and NPA Co salt + nitro compound) + 15% paraffin. Thus, by adding this composition to T-30 oil distillate, a result of 377 days was obtained in the condensation phase by the liquid method, and 467 days in the atmospheric phase [5].

4. By adding amides and imidazolines of NPA synthesized with Berolamine-20 in different molar ratios (1÷6) to road bitumen in an amount of 0.4 and 0.6%, the quality indicators of bitumen were improved and high results were obtained. In particular, while the adhesion of bitumen itself was 3 points, the adhesion of the additive in an amount of 0.4% reached 1 point [4, 10, 19, 23].

5. The quality indicators and adhesion of bitumen were tested by adding additives prepared by adding AK-15 and Aftol oil (in amounts of 5, 10 and 15%) to the amides synthesized with NPA Berolamine-20 in different molar ratios to road bitumen. It was found that these oils added to the prepared additives have a positive effect on improving the quality indicators of bitumen and achieving high adhesion. I should also note that the use of these oils in the preparation of the additive is also considered economically advantageous [19, 20].

6. The quality indicators and adhesion of bitumen were tested by adding amides synthesized on the basis of distilled natural petroleum acids in different molar ratios together with mineral oil distillates (in amounts of 5, 10 and 15%) as additives to bitumen. It was found that the addition of 10% of oil distillate to the synthesized amide has a positive effect. Thus, the quality indicators of bitumen were Tsoft.- 46.4°C, needle penetration depth 51 cm, tensile strength 90.15 cm, brittleness temperature -27°C, and adhesion 1 point [1, 19].

7. Salts of sulfate derivatives of plant fatty acids were synthesized and their application as hydrophobizers to mineral powders was tested. For this purpose, a sulfate derivative of sunflower fatty acid imported from Azersun was synthesized and its Ca salt was obtained. Then, this product was applied as a hydrophobizer to mineral powders.

In addition, research was also conducted by adding a mixture of Azerbaijani oil, namely the residue above 350°C, to it and adding it to mineral powders.

As a result of the research, it was determined that the substances we synthesized increase the hydrophobicity of mineral powder, which has a positive effect on improving the quality of bitumen. "Tests of asphalt concrete samples prepared with BNB 50/70 brand control bitumen activated mineral powder at the Azerelmi-Tedgikhtlayihe Institute, as well as with "V-2" adhesion additive bitumen and mineral powder presented by ARETN's IPCP, show that the physical and mechanical indicators of the samples with adhesion additive and activated mineral powder are superior to the

indicators of the control sample. Alınmış nəticələrə əsaslanaraq yaradılmış optimal tərkibli konservasiya mayelərinin və yol bitumuna əlavə olunan aşqarların sənayedə tətbiqi üçün ümumi sxem tərtib olunmuşdur[9, 17].

8. Based on the results obtained, a general scheme has been developed for the industrial application of optimally composed conservation fluids and additives added to road bitumen [7, 8, 11, 12].

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