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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

**SYNTHESIS AND INVESTIGATION OF CETANE NUMBER
IMPROVING ADDITIVES BASED ON ORGANIC ACIDS**

Speciality: 3303.01 - “Chemical technology and engineering”

Field of science: Technical sciences

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The work was performed at the Institute of Petrochemical Processes named after Y.H. Mammadaliyev of the Ministry of Science and Education of the Republic of Azerbaijan at the «Technology of Chemical Reagents for the Oil and Gas Extraction and Processing Industries» laboratory

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GENERAL DESCRIPTION OF WORK

Relevance of the topic and the degree of its development.

Improving the quality of fuels to meet the environmental requirements of the modern transportation and industrial sectors is of vital importance. In diesel distillates, one of the main indicators reflecting combustion parameters and engine efficiency is the cetane number. A low cetane number in diesel distillate can lead to increased ignition delay, higher emissions resulting from incomplete combustion, and exacerbated engine acoustic-vibration problems. Therefore, the production and application of cetane-improving additives is a pressing issue from both an economic and environmental perspective.

Although traditional cetane-improving additives are effective, some of them are quite expensive and exert a negative impact on the environment. Furthermore, certain cetane improvers exhibit poor compatibility with other engine performance parameters. As a result, the demand for cost-effective, environmentally safe, and efficient cetane-improving additives remains consistently high¹.

Existing studies show that while certain additives synthesized from organic acids increase the cetane number of diesel distillate, both the detailed explanation of reaction mechanisms and the comparative impact of various metal centers and fraction selection on the physical-chemical properties of the distillate have not been sufficiently comprehensively investigated in this field. In particular, the preparation of various metal salts of natural petroleum acids (NPA) derived from natural petroleum acid fractions has been carried out in only a limited number of studies².

Object and subject of the research. The object and subject of

¹ Mohammed, A.S. A comprehensive review on the effect of ethers, antioxidants, and cetane improver additives on biodiesel-diesel blend in CI engine performance and emission characteristics / A.S.Mohammed, S.M.Atnaw, A.V.Ramaya [et al.] // –Journal of the Energy Institute:London,–2023. vol.108, –p. 1-21.

² Abbasov, V.M. Study on the interaction of amidoesters with various petroleum acid compositions as antistatic additives in diesel distillate / V.M.Abbasov, E.K.Hasanov, L.A.Mahmudova [et al.] // –Processes of Petrochemistry and Oil Refining:Baku,–2025. vol.26, №2, –p. 544-554.

the dissertation work aim to systematically study the synthesis of metal (Ni, Co, Zn, Mn, Fe) salts based on readily available NPA and their fractions, determine the properties of their compositions combined with the nitration product of diesel distillate, and evaluate their effects when added to diesel distillate on the cetane number, petroleum-collecting capacity from water surfaces, and performance as an antistatic additive. This approach can provide essential data for application in fuel technologies while aiding in the understanding of reaction mechanisms at the molecular level.

Aims and objectives of the research. The objectives and objectives of the research work are the synthesis of metal salts from different fractions of NPA, the extraction of the nitration product of diesel, the determination of their structural and physical properties, and the evaluation of the effect of diesel distillate on the electrical conductivity, the cetane number and efficiency of oil collection from the surface of water when added to diesel distillate in different concentrations.

To achieve this goal, the following actions have been taken:

- NPA is separated into fractions (200-280°C, 280-340°C, 340-410°C) by vacuum distillation at 2 mmHg, their corresponding metal salts (Ni, Co, Zn, Mn, Fe) are obtained by exchange reaction and the physico-chemical parameters of the fractions are determined;
- At the end of 40 days, the antistatic indicators of diesel distillate were determined by the composition of metal salts of NPA fractions and their composition with the nitration product of diesel distillate, as well as the addition of amidoamine synthesized on the basis of ethylenediamine to diesel distillate at dosages of 0.05-0.15%;
- The effect of the metal salts of NPA fractions, and their compositions with the nitration product of diesel distillate, on the capacity to collect Naftalan and Salyan oils from the sea and tap water surface was studied over a period of 104 hours;
- The efficacy of metal salts of fractions of NPA and their composition with the nitration product of diesel distillate has been determined as an agent that increases the cetane number by

adding them to 100 g diesel distillate at concentrations of 60 and 500 ppm;

- Based on the results obtained, a principled scheme of the process has been developed that can be established in production in order to organize the large-scale production of antistatic additives that increase the electrical conductivity of diesel distillate and the cetane number;

Research methods. In the research work, NPA, diesel distillate and nitric acid were taken as raw materials. Fractions of NPA separated by vacuum distillation at 2 mmHg (200-280°C, 280-340°C and 340-410°C) were used. In order to obtain functional additives on the basis of these fractions, NPA was first neutralized with sodium hydroxide and converted to the form of sodium salts, and at the next stage, the corresponding metal salts were synthesized on the basis of the exchange reaction. The reactions were carried out in the solvent medium at a temperature range of 50-60°C, after the process was completed, it was divided into mixed phases, and the final products were obtained by removing the solvent from the organic phase.

In the research work, diesel distillate and nitric acid were used as raw materials for the nitration reaction. The nitric acid filled in the flask is mixed and heated to a temperature of 70°C. After the calculated amount of diesel distillate is added, the mixture is intensively mixed at 70°C for about 1 hour. After the reaction is completed, the system is cooled to a temperature of $25 \pm 5^\circ\text{C}$ and the mixing is stopped. As a result of cooling, the mixture is divided into two phases. In the lower phase, the nitric acid that has not entered the reaction is collected, and in the upper phase, the product obtained as a result of nitration is collected. The lower phase is separated and transferred to a separate vessel and stored. The upper phase is accepted as a nitration product and directed to the next processing stage. In order to eliminate the remaining moisture in the nitro product obtained at the last stage, the product is dried with a drying agent or in vacuum conditions, and as a result, a stable nitrocompound mixture is obtained. By preparing compositions of nitrified diesel distillate with metal salts of NPA in a ratio of 1:1 mass ratio, the electrical conductivity of diesel distillate, the efficiency of the ability to collect oil from the surface of

the water and its effect on the cetane number were studied.

IR spectroscopy was used to determine the functional groups and structural characteristics of the samples. NMR spectroscopy was used to study the molecular structure more accurately.

The special electrical conductivity of the prepared compositions was determined in laboratory conditions by means of the ЭЛ-4М device in accordance with the requirements of ГОСТ 25950-83 and ГОСТ 33461-2015 standards.

Key findings submitted for defense.

1. Synthesis of various metal salts on the basis of fractions of NPA separated in different temperature ranges and study of their physico-chemical properties;
2. Determination of structural properties of synthesized metal salts by IR and NMR spectroscopy methods;
3. Acquisition of the nitration product of diesel distillate and preparation of its compositions of NPA with metal salts and study of the mechanism of action and effectiveness as an antistatic additive in diesel distillate;
4. Assessment of the effect of synthesized reagents and compositions on the cetane number and other important indicators of diesel distillate;
5. Experimental determination of the ability of the prepared compositions to accumulate various oils from the surface of the water.

Scientific novelty of the research.

1. On the basis of different fractions at 2 mmHg of NPA (200-280°C; 280-340°C; 340-410°C), Ni, Co, Zn, Mn, Fe salts and the nitration product of diesel distillate were synthesized. It was determined that the increase in various parameters of diesel distillate was determined by the addition of these salts separately and together with the nitration product of diesel distillate to diesel distillate;
2. For the first time, it has been found that the nitration product of diesel distillate and the composition of various fractions of NPA with metal salts have the ability to collect Salyan and Naftalan oil from the surface of seawater with high efficiency;

3. It has been determined that the combination of metal salts of different fractions of NPA and diesel distillate with the nitration product increases the cetane number of diesel distillate;
4. For the first time, the electrical conductivity of diesel distillate was studied by adding metal salts of various fractions of NPA, the nitration product of diesel distillate and the compound of diesel distillate together with amidoamine synthesized on the basis of ethylenediamine to diesel distillate as an antistatic additive;
5. For the first time, a new functional solvent consisting of solutions in isopropyl alcohol of the composition of metal salts and nitration products of NPA has been developed, and it has been determined that it increases the ability to collect oil from the surface of water;

Theoretical and practical significance of the research. The theoretical significance of the work lies in the fact that the structural, physical and functional properties of metal salts obtained from different fractions of NPA, as well as the compositions of diesel distillate prepared with the nitration product have been systematically studied. The constituent properties and functional properties of these compounds have been determined by means of IR and NMR spectroscopy, as well as other methods of physico-chemical analysis.

The practical significance of the study is the discovery of the possibilities of application of metal salts obtained on the basis of different fractions of NPA and their compositions with nitroized diesel distillate as an antistatic and an additive that increases the cetane number for diesel distillate. It has been determined that the addition of these substances to diesel distillate in different concentrations increases its electrical conductivity, increases the cetane number and has a positive effect on a number of important operational indicators. This is of great practical importance in terms of improving the quality of diesel distillates and increasing the safety of operation.

The results obtained are of practical importance for the development of new functional additives based on local raw materials in the oil refining and chemical industry. From this point of view, the results of the research serve to make efficient use of local raw material

resources, reduce dependence on imports and develop technologies for the purchase of additives.

Approbation and application. 12 scientific works, including 7 articles and 5 report theses, were published on the dissertation. The published works fully reflect the essence of the dissertation.

The main results of the dissertation were discussed at the following scientific conferences: Republican scientific-practical conference on "Protection of the ecosystem in the efficient use of natural resources and wastes" (Ganja, 2025); International Scientific Conference on "Global Challenges in the Field of Natural and Social Sciences" dedicated to the 102nd anniversary of the birth of the National Leader Heydar Aliyev, (Ganja, 2025); 1st International Conference on "Modern Challenges of Chemistry" dedicated to the 103rd anniversary of the birth of the National Leader Heydar Aliyev, (Ganja, 2026); ISPEC 21. International Conference on Engineering & Natural Sciences (Izmir, Turkey, 2026) and VII International Scientific Conference "Innovative scientific forum" (Helsinki, Finland, 2026).

The name of the organization where the dissertation was completed. The dissertation work was carried out in the laboratory "Technology of chemical reagents for oil and gas extraction and refining industries" of the Institute of Petrochemical Processes named after academician Y.H. Mammadaliyev of the Ministry of Science and Education of the Republic of Azerbaijan.

Author's personal participation. It is the responsibility of the claimant to determine the goals and directions of the work, to conduct experiments, as well as to analyze and generalize the results obtained.

Scope of the dissertation with an indication of the volume of individual structural sections and the total volume of the work.

The dissertation consists of an introduction, 6 chapters, conclusions, and a list of 170 references. The dissertation consists of 12 images, 25 graphs, 4 schemes, 49 tables and a total volume of 173 pages of printed materials. The dissertation consists of 196762 marks, except for tables, pictures, schemas and references (Introduction 13358, Chapter I 33030, Chapter II 13890, Chapter III 32760, Chapter IV 27135, Chapter V 37825, Chapter VI 37070 and Conclusion 1694 marks).

In the introductory part, information is given about the relevance of the topic, the purpose of the dissertation, the scientific novelty and practical significance of the work.

The first chapter is devoted to the literature review. In this chapter, the physico-chemical properties of diesel distillate, the essence of the cetane number, the general classification of additives that increase the cetane number, the effect of the cetane number on the operation of diesel engines, the cetane-enhancing additives synthesized on the basis of organic acids, modern environmental and technological requirements, as well as the current state of the problem and the justification of the research direction are given.

The second chapter is devoted to research objects, materials and methods. In this chapter, the study of the effect of antistatic additives on special electrical conductivity in diesel distillate and the methods of measuring conductivity, methods and methods for determining the cetane number in diesel distillate, as well as the calculation of the cetane number in diesel distillate are explained.

The third chapter is devoted to the synthesis of various metal salts and other used components of NPA, their physico-chemical properties. In this chapter, various fractions of NPA and their physico-chemical properties, synthesis of various metal salts, extraction of nitroized diesel distillate, preparation of its compositions with metal salts, physicochemical properties of solutions of these compositions in isopropyl alcohol, as well as IR and NMR spectra of synthesized samples are given.

The third chapter is devoted to fractions of NPA and their physicochemical properties, the synthesis of various metal salts, the preparation of nitrated diesel distillate, and the formulation of its compositions with metal salts. It also covers the physicochemical properties of solutions of these compositions in isopropyl alcohol, as well as the IR and NMR spectra of the synthesized samples.

The fourth chapter is devoted to the study of metal salts of NPA in diesel distillate. In this chapter, the effect of metal salts synthesized on the basis of the total fraction of NPA, as well as the fractions of 200-280°C, 280-340°C and 340-410°C and their compositions with diesel nitration product as an antistatic additive was

investigated, and their effect on the electrical conductivity of diesel was analyzed in a comparative manner.

The fifth chapter is devoted to the study of the ability of synthesized reagents to accumulate oil from the surface of the water. In this chapter, the effect of the compositions of the diesel nitration product on the basis of the total fraction of NPA, metal salts synthesized on the fractions of 200-280°C, 280-340°C and 340-410°C on the ability to accumulate Salyan and Naftalan oils from the water surface is studied and the results obtained are presented in a comparative manner.

The sixth chapter is devoted to the study of the effect of synthesized additives on the cetane number and their technical and economic assessment. In this chapter, the total fraction of NPA, as well as the properties of metal salts synthesized on the basis of fractions of 200-280°C, 280-340°C and 340-410°C and their compositions with the nitration product of diesel distillate as additives that increase the cetane number are studied. In addition, the generalized principled technological scheme of the processes, the results of the technical and economic assessment of the process of obtaining iron salt of NPA and the material balance of the product of the extraction of nitrocompound with salts of NPA were presented.

At the end of the dissertation, the main results of the research, the list of references, additions and abbreviations are given.

THE MAIN CONTENT OF THE WORK

1. Synthesis and Physicochemical Characterization of Metal Salts of Natural Petroleum Acids and Associated Components

Distillation of NPA under a vacuum of 2 mmHg yielded three fractions corresponding to boiling ranges of 200–280°C, 280–340°C, and 340–410°C (Table 1). As the distillation range increases, density (from 930 to 1050 kg/m³) and refractive index (from 1.470 to 1.500) increase consistently due to rising molecular mass.

The closeness of acid numbers in the 280–340°C and 340–410°C fractions indicates stabilization of carboxyl group content per unit mass. This value determines alkali calculations required for metal salt synthesis.

Table 1.

Properties of NPA fractions at 2 mmHg

Indicator	200–280°C fraction	280–340°C fraction	340–410°C fraction
Density (20°C), kg/m ³	930 – 970	970 – 1010	1000 – 1050
Acid number, mg KOH/g	200.64	271.62	277.67
Refractive index (n_D^{20})	1.470	1.485	1.500
Pour point, °C	minus 10	minus 15	minus 5
Color	Light yellow	Yellow- brown	Dark brown

There is no monotonous dependence on freezing temperature. Thus, minus 10°C in the fraction of 200–280°C, negative 15°C at 280–340°C and negative 5°C at 340–410°C. The fact that the medium fraction (280–340°C) has a lower freezing temperature confirms that it shows better fluidity from a rheological point of view and has a technological advantage in the synthesis of additives.

As the fraction becomes heavier, the darkening of the color from

light yellow to dark brown reflects an increase in the proportion of coloring high-boiling components and oxidized structures in the fraction. This visual criterion is important for operational control and raw material detection during synthesis.

The process of obtaining metal salts of NPA is carried out by neutralizing the solution of NPA and the interaction of sodium salt, which is the reaction product, with the solution of the corresponding metal salt.

To carry out the synthesis, a 1000 ml flask with three throats is used. The flask is filled with NPA, a 20% alkaline solution is added to it and mixed.

After 30 minutes, 300 ml of solvent and a water solution of metal salt are gradually added to the sodium salt of NPA, which is the reaction product. During this time, the temperature is regulated by a heater in the range of 50-60°C.

The physico-chemical properties of samples taken on the basis of various metal salts of NPA are presented in Table 2. Here both the methodological basis of analytical measurements and the direction of the observed changes depending on the nature of the metal ion are clearly expressed.

The average molecular weight of NPA was 503 g/mol for Zn salt, 497 g/mol for Co salt, 493 g/mol for Mn salt, 496 g/mol for Ni salt, and 494 g/mol for Fe salt.

In Table 2, the density of NPA at a temperature of 20°C for Zn and Co salts was 1050 kg/m³. For the Mn and Ni salts of NPA, it was 1100 kg/m³. The density of the Fe salt of NPA was 1150 kg/m³.

Diesel distillate was used as a raw material for the synthesis of nitrified diesel distillate. To shorten the induction phase of the reaction, NaNO₂ is added to nitric acid to form nitrogen-dioxide (NO₂). The mixture is then heated to 70°C and drops of diesel distillate are added to it. The heat balance is regulated in the range of 70±9°C, as temperatures above 90°C increase the danger of the process and harmful NO and NO₂ gases are released during the reaction.

Table 2.

**Physico-chemical properties of various metal salts of
natural petroleum acid**

Parameters	Name of the device	Method	The name of the example				
			Zn-salt of NPA	Co-salt of NPA	Mn-salt of NPA	Ni-salt of NPA	Fe-salt of NPA
Average molecular weight, g/mol	Mass Spectrometry	-	503	497	493	496	494
Density, kg/m ³ 20 °C	DMA 4500 M	ASTM D5002	1050	1050	1100	1100	1150
Refractive index, 20 °C	Abbemat 500	-	1.4970	1.4962	1.5002	1.5025	1.5072
Colour	-	-	Brown-dark green	dark purple	dark red	dark green	dark black

After the reaction is completed and the system is cooled to $25 \pm 5^\circ\text{C}$, the product is divided into two phases. These are the lower phase, which consists of nitric acid, which has not entered the reaction but can be regenerated later, and the upper phase, which consists of nitrocompounds. The upper phase consisting of nitrocompounds is obtained as a finished nitro product after being washed and rinsed with distillation water and cleaned of moisture by means of a drying agent (or vacuum). As a result, a stable mixture of nitrocompounds, which is used as a nitration product of diesel distillate, is synthesized with a yield of about 78% by mass of the raw material. The resulting gases are neutralized by means of a neutralization system.

The physico-chemical parameters of hydropurified diesel distillate (Euro-3) (Table 3) reflect its adaptive properties in the engine, rheological behavior and low temperature indicators. The density of the distillate at 20°C is 854 kg/m^3 , the 90% distillation is 325°C and the boiling end is 338°C , which indicates that it is located in the heavy distillate range, and $5,64 \cdot 10^{-6} \text{ m}^2/\text{s}$ its kinematic viscosity determines the drip character in the blasting system.

Table 3.

**Physico-chemical parameters of hydropurified diesel distillate
(Euro-3)**

Parameter		Value
Density, at 20°C, kg/m ³		854
Kinematic viscosity, at 20°C, m ² /s		5,64·10 ⁻⁶
Aromatic hydrocarbon content, %		14
Flash point (closed cup), °C		77
Total sulfur content, %		0.0314
Cloud point, °C		minus 6
Pour point, °C		minus 19
Composition of the fraction	5% recovery temperature, °C	202
	10% recovery temperature, °C	212
	20% recovery temperature, °C	222
	40% recovery temperature, °C	256
	60% recovery temperature, °C	267
	70% recovery temperature, °C	283
	80% recovery temperature, °C	300
	90% recovery temperature, °C	325
Final boiling point, °C		338
Carbon residue, %		0.003
Ash content, %		0.005
Acidity, mg KOH/100 cm ³		1.5
Cetane number, units		49.26

Although the 14% aromatic hydrocarbons in its composition limit the ignition quality, the cetane number being at the level of 49.26 units demonstrates the positive impact of hydrotreating. The flash point of 77°C, which reduces the risk of volatility, total sulfur of 0.0314%, coking of 0.003%, and ash content of 0.005% confirm the storage safety of the distillate and its low tendency to form deposits in injectors. The cloud point (minus 6°C) and pour point (minus 19°C) indicate the controllable nature of paraffin crystallization despite the heavy fraction, while the acidity value of 1.5 mg KOH/100 cm³ highlights the necessity of monitoring corrosion risk. Overall, these parameters provide a reliable basis for applying metal salt-based additives that increase the cetane number without deteriorating the properties of the distillate, as well as for evaluating their impact on rheology, low-temperature stability, and ash content.

Figure 1 presents the IR spectrum of the nitration product of diesel distillate. Analysis of the spectrum shows that nitro groups have been incorporated into the initial hydrocarbon-containing system, while aromatic fragments and oxygen-containing functional groups are also present in the sample.

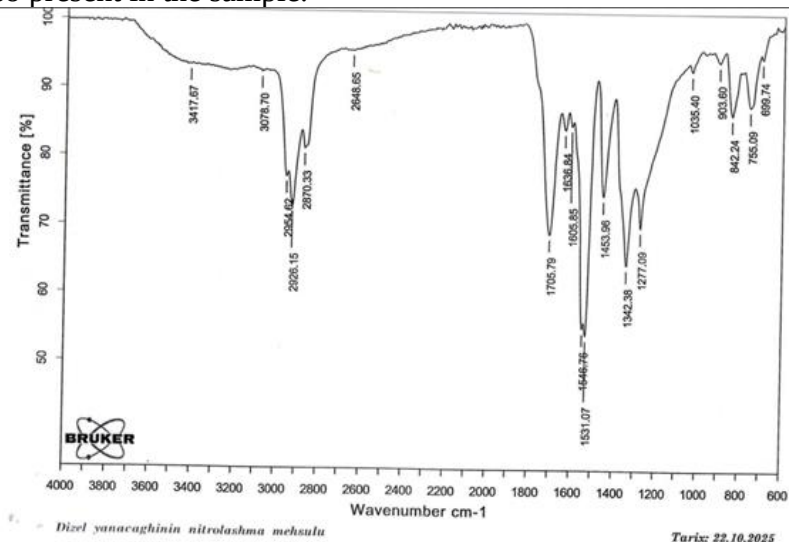


Figure 1. IR spectrum of the nitration product of diesel distillate.

The following absorption bands are observed in the IR spectrum:

- deformation (1453 cm^{-1}) and stretching ($2870, 2926, 2954 \text{ cm}^{-1}$) vibrations of the C-H bond belonging to CH_3 and CH_2 groups;
- deformation vibrations belonging to the C-H bond of the benzene ring ($699, 755, 842 \text{ cm}^{-1}$);
- stretching vibrations belonging to the C-C bond of the benzene ring (1605 cm^{-1});
- stretching vibrations belonging to the $-\text{NO}_2$ group (1636 cm^{-1});
- stretching vibrations belonging to the C- NO_2 group ($1342, 1531, 1546 \text{ cm}^{-1}$).

Figure 2 displays the ^1H NMR spectrum of the nitration product of diesel distillate, showing the distribution of hydrogen atoms across aliphatic, petroleum, aromatic, and olefinic structural units.

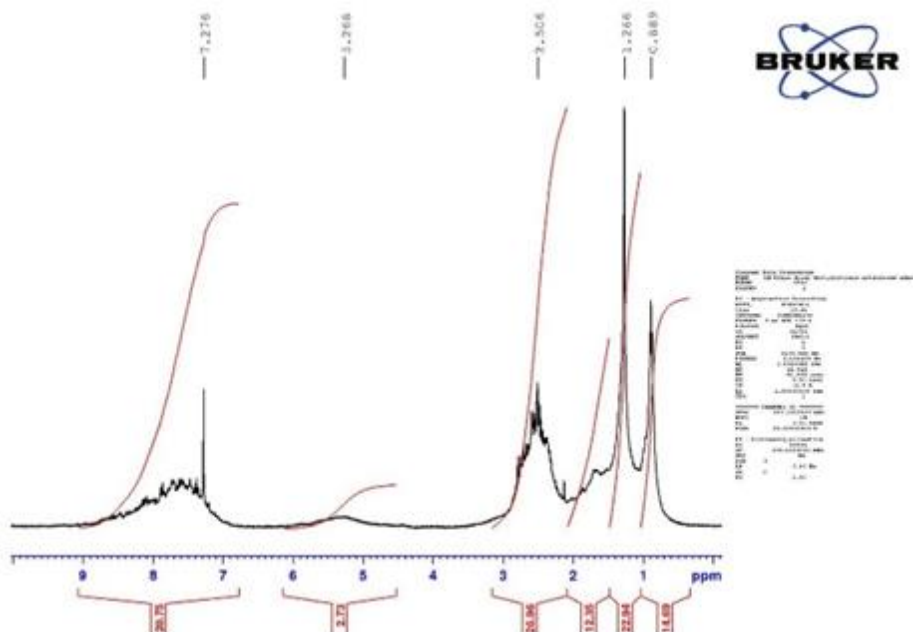


Figure 2. ^1H NMR spectrum of the nitration product of diesel distillate.

In the ^1H NMR spectrum (BRUKER-Fourier 300.18 MHz, CDCl_3 , δ , ppm) of the nitration product of diesel distillate, the resonance signal recorded at 0.70-1.00 ppm corresponds to the CH_3 group, 1.00-1.50 ppm to the CH_2 group, 1.50-2.10 ppm to naphthenic rings, 2.10-3.10 ppm to CH_2 groups located in the α -position relative to the aromatic ring, 4.80-6.10 ppm to olefinic hydrogens, and the resonance signal observed at 6.90-9.00 ppm corresponds to protons belonging to the aromatic ring.

2. Investigation as an antistatic additive of metal salts synthesized on the basis of various fractions of natural petroleum acid by adding them to diesel distillate in various percentage concentrations

The electrical conductivity of diesel distillate was tested using an ЭЛ-4М device by adding compositions (1:1 mass ratio) of Ni, Co,

Zn, Mn, and Fe salts of NPAs, including fractions of these acids separated under vacuum conditions (200-280°C, 280-340°C, and 340-410°C), with the nitration product of diesel distillate to diesel distillate in amounts of 0.05%, 0.1%, and 0.15%. The results of the tests are shown in Table 4.

Table 4.

Electrical conductivity of diesel distillate with the addition of Ni, Co, Zn, Mn, and Fe salts of natural petroleum acid to diesel distillate in amounts of 0.05%, 0.1%, and 0.15%

Sample Name	Concentration, %	Electrical conductivity, pSm/m (without additive 52 pSm/m)				
		After 1 day	After 10 days	After 20 days	After 30 days	After 40 days
NPA Ni salt	0.05	131	326	332	364	382
	0.1	681	794	975	980	1040
	0.15	775	870	1001	1087	1096
NPA Co salt	0.05	95	678	619	837	870
	0.1	1610	1580	1560	1540	1505
	0.15	1702	1595	1605	1670	1565
NPA Zn salt	0.05	29	36	38	65	72
	0.1	154	249	361	425	502
	0.15	270	370	420	476	602
NPA Mn salt	0.05	214	591	687	776	820
	0.1	1080	1073	1020	1010	1001
	0.15	1145	1102	1075	1087	1132
NPA Fe salt	0.05	1390	1450	1330	1300	1260
	0.1	1710	1220	1070	1052	1035
	0.15	1950	1270	1160	1120	1175

Increasing electrical conductivity in diesel distillate and its stability over time depends on the type and amount of metal salt. The effect provided by the metal salts of NPA in the distillate environment is not limited only to combustion performance; parameters such as electrical conductivity also enable the evaluation of the system's colloidal stability, the formation of its dispersed structure, and its changes during storage. When adding the Co salt of NPA to diesel distillate in an amount of 0.15%, the electrical conductivity of the diesel distillate increased further, reaching 1565 pSm/m after 40 days.

Testing of the electrical conductivity of diesel distillate was

conducted by adding compositions of various metal salts synthesized on the basis of the 340-410°C fraction of NPA with the nitration product of diesel distillate to diesel distillate in 0.05; 0.1 and 0.15%, and the obtained results are presented in Table 5.

Table 5.

Electrical conductivity of diesel distillate with the addition of compositions of Ni, Co, Zn, Mn, and Fe salts of the 340-410 °C fraction at 2 mmHg of natural petroleum acid with the nitration product of diesel distillate (at 1:1 mass ratio) to diesel distillate in amounts of 0.05%, 0.1%, and 0.15%

Sample Name	Concentration, %	Electrical conductivity, pSm/m (without additive 52 pSm/m)				
		After 1 day	After 10 days	After 20 days	After 30 days	After 40 days
NPA Ni salt	0.05	272	390	442	460	462
	0.1	282	397	455	461	461
	0.15	295	425	452	487	490
NPA Co salt	0.05	715	780	867	965	968
	0.1	735	782	875	980	985
	0.15	747	768	920	962	965
NPA Zn salt	0.05	185	187	190	191	191
	0.1	191	195	196	198	198
	0.15	225	237	245	245	246
NPA Mn salt	0.05	682	735	852	915	957
	0.1	710	748	867	935	937
	0.15	810	817	912	985	1000
NPA Fe salt	0.05	1075	1410	1462	1467	1467
	0.1	1337	1436	1482	1510	1515
	0.15	1487	1560	1572	1585	1687

It comprehensively reflects the range of variation in the electrical conductivity of diesel from day 1 to day 40 as a result of adding compositions of Ni, Co, Zn, Mn, and Fe salts synthesized on the basis of the 340-410°C fraction of NPA with the nitration product of diesel distillate to diesel distillate in amounts of 0.05%, 0.1%, and 0.15%.

The Fe salt of NPA possesses the highest conductivity capacity, yielding superior results both in the initial stage and in the final stage. Thus, when added at 0.05%, it shows an increase from 1075 pSm/m

to 1467 pSm/m, and at 0.1%, an increase from 1337 pSm/m to 1515 pSm/m. When this additive is added at 0.15%, the electrical conductivity rises from 1487 pSm/m to 1687 pSm/m, which is one of the highest final values among the presented samples. This level indicates that iron-centered salts create a stronger ionic conductivity background within the diesel distillate, and as the concentration increases, this background is strengthened even further.

The Co salt of NPA also exhibits high electrical conductivity. In particular, the time factor enhances the effectiveness of this additive. When added at 0.05%, the value rises from 715 pSm/m to 968 pSm/m. When added at 0.1%, an increase from 735 pSm/m to 985 pSm/m is observed. When added at 0.15%, although the initial value is 747 pSm/m, it rises to 920 pSm/m by day 20 and reaches the level of 965 pSm/m by day 40.

Overall, comparative analysis of the salts obtained on the basis of the 340-410°C fraction shows that the Fe and Co salts of NPA have high efficiency in terms of increasing electrical conductivity.

In Table 6, among the samples, the results obtained by adding the composition of the amidoamine synthesized on the basis of NPA and ethylenediamine (EDA) with the Mn and Fe salts synthesized on the basis of the 340-410 °C fraction of NPA to diesel distillate in amounts of 0.05%, 0.1%, and 0.15% are the highest. The general pattern observed for all samples is that electrical conductivity increases over time.

As an antistatic additive, the composition of the amidoamine synthesized on the basis of NPA and ethylenediamine with the Fe salt synthesized on the basis of the 340-410 °C fraction of NPA increased the efficiency of the electrical conductivity of diesel distillate more compared to other metal salts. As seen in Table 6, upon adding 0.15% of this additive to diesel distillate, the electrical conductivity increased up to 1892 pSm/m over 40 days.

Table 6.

Electrical conductivity of diesel distillate with the addition of compositions of the NPA and EDA amidoamine with Mn and Fe salts synthesized on the basis of the 340-410 °C fraction at 2 mmHg of NPA to diesel distillate in amounts of 0.05%, 0.1%, and 0.15%

Sample Name	Concentration, %	Electrical conductivity, pSm/m (without additive 52 pSm/m)				
		After 1 day	After 10 days	After 20 days	After 30 days	After 40 days
NPA:EDA Amidoamine (1:1 molar ratio) + NPA Mn salt	0.05	1430	1433	1435	1437	1437
	0.1	1510	1512	1514	1516	1516
	0.15	1670	1672	1672	1674	1674
NPA:EDA Amidoamine (1:1 molar ratio) + NPA Fe salt	0.05	1612	1612	1613	1615	1615
	0.1	1790	1792	1792	1792	1795
	0.15	1885	1887	1890	1890	1892

Comparative analysis of the metal salts showed that among the Fe, Ni, Co, Zn, and Mn salts, the highest antistatic effect belonged to the composition of the NPA and EDA amidoamine with the Fe salt synthesized on the basis of the 340-410 °C fraction of NPA. The high efficiency of the Fe salt of NPA is based on the variable valency property of Fe ions and their ability to form coordination bonds. $\text{Fe}^{2+}/\text{Fe}^{3+}$ ions form stronger complexes with the functional groups of amidoamine molecules, thereby increasing ionic conductivity in the system and ensuring rapid dispersion of electrostatic charges. Furthermore, the high dispersibility of the Fe salt of NPA improves homogeneous distribution in the oil medium, enhancing the stability of the antistatic effect.

3. Investigation of the oil-collecting capacity of the synthesized reagents

The investigation of the oil-collecting capacity of the synthesized reagents is one of the main parameters evaluating their functional efficiency and application value in a real environment. An

oil film falling onto the water surface poses a serious threat to the ecosystem. Thus, as factors such as waves, wind, and temperature reduce the effectiveness of mechanical recovery means, the application of chemical reagents as surfactants and collectors becomes necessary. The efficiency of the reagents depends directly on the salinity of the medium, temperature, water hardness, and the fractional composition of the oil. At the same time, in accordance with environmental safety requirements, the toxicity and biodegradability of the reagent must also be taken into account.

The results of testing the oil-collecting capacity of the composition of the nitration product of diesel distillate with metal salts synthesized on the basis of the 200-280°C fraction of NPA on Naftalan oil in tap and sea water are presented in Table 7. Thus, the Fe salt of NPA (Sample-3) showed the highest efficiency in tap water, achieving a result of 89.02%. This indicator is one of the highest results among all tests. The fact that the Fe salt of NPA exhibits such high efficiency with Naftalan oil indicates that its form synthesized on the basis of the 200-280°C fraction strongly interacts with the naphthenic hydrocarbons of Naftalan oil. The decrease in the diameter of the oil slick from 9.1 cm to 0.9 cm after applying the reagent, and its remaining stable at the level of 1.0 cm over 104 hours, confirms the excellent compatibility of this reagent with Naftalan oil.

With Naftalan oil in tap water, the Mn salt of NPA (Sample-2) showed an efficiency of 77.66%, and the Zn salt of NPA (Sample-1) showed 70.84%. The result for the Ni salt of NPA (Sample-4) was 67.37%. Meanwhile, the Co salt of NPA (Sample-5) showed a lower result with an indicator of 63.74%.

The results with Naftalan oil in the sea water medium reveal a similar pattern. The Fe salt of NPA (Sample-3) showed a higher result with an indicator of 87.37%. The Mn salt of NPA (Sample-2) yielded 75.54%, and the Zn salt of NPA (Sample-1) yielded 71.29%. The indicator of 60.47% for the Co salt of NPA (Sample-5) is slightly higher than the result of 58.95% for the Ni salt of NPA (Sample-4).

Table 7.

Results of testing the Naftalan oil-collecting capacity of the composition of the nitration product of diesel distillate with metal salts synthesized on the basis of the 200-280°C fraction at 2 mmHg of NPA from tap and sea water

Specimen	1	2	3	4	5
In tap water					
Dish diameter, cm	9.8	9.6	9.3	9.8	9.3
Initial slick diameter, cm	9.6	9.4	9.1	9.5	9.1
Diameter after adding reagent, cm	2.7	2.0	0.9	3.0	3.2
Diameter after 24 hours, cm	2.8	2.1	1.0	3.0	3.2
Diameter after 56 hours, cm	2.8	2.1	1.0	3.1	3.3
Diameter after 104 hours, cm	2.8	2.1	1.0	3.1	3.3
Efficiency of the oil-collecting capacity of reagents on Naftalan oil, %	70.84	77.66	89.02	67.37	63.74
In sea water					
Dish diameter, cm	10.3	9.6	9.6	9.8	8.8
Initial slick diameter, cm	10.1	9.4	9.5	9.5	8.6
Diameter after adding reagent, cm	2.6	2.1	1.0	3.7	3.3
Diameter after 24 hours, cm	2.9	2.2	1.1	3.8	3.3
Diameter after 56 hours, cm	2.9	2.3	1.2	3.9	3.4
Diameter after 104 hours, cm	2.9	2.3	1.2	3.9	3.4
Efficiency of the oil-collecting capacity of reagents on Naftalan oil, %	71.29	75.54	87.37	58.95	60.47

Note: The tests were carried out by adding 2 mL of petroleum to 40 mL of water, followed by the addition of 2 g of a 5% solution of the sample

Sample 1. IPA-28.5 g + Nitration product of diesel distillate-0.75 g + Zn salt based on the 200-280 °C fraction of NPA-0.75 g

Sample 2. IPA-28.5 g + Nitration product of diesel distillate-0.75 g + Mn salt based on the 200-280 °C fraction of NPA-0.75 g

Sample 3. IPA-28.5 g + Nitration product of diesel distillate-0.75 g + Fe salt based on the 200-280 °C fraction of NPA-0.75 g

Sample 4. IPA-28.5 g + Nitration product of diesel distillate-0.75 g + Ni salt based on the 200-280 °C fraction of NPA-0.75 g

Sample 5. IPA-28.5 g + Nitration product of diesel distillate-0.75 g + Co salt based on the 200-280 °C fraction of NPA-0.75 g

The results of testing the Salyan oil-collecting capacity of the composition of the nitration product of diesel distillate with metal salts synthesized on the basis of the 340-410°C fraction of NPA from tap and sea water are presented in Table 8.

Table 8.

Results of testing the Salyan oil-collecting capacity of the composition of the nitration product of diesel distillate with metal salts synthesized on the basis of the 340-410°C fraction at 2 mmHg of NPA from tap and sea water

Specimen	1	2	3	4	5
In tap water					
Dish diameter, cm	8.8	10.3	9.4	9.6	9.3
Initial slick diameter, cm	8.5	10.1	9.2	9.4	9.1
Diameter after adding reagent, cm	2.8	1.1	0.8	3.0	1.9
Diameter after 24 hours, cm	2.9	1.2	0.8	3.2	2.0
Diameter after 56 hours, cm	2.9	1.3	0.9	3.2	2.0
Diameter after 104 hours, cm	2.9	1.3	0.9	3.2	2.0
Efficiency of the oil-collecting capacity of reagents on Salyan oil, %	65.89	87.13	90.22	65.96	78.03
In sea water					
Dish diameter, cm	9.4	10.1	9.4	9.6	9.1
Initial slick diameter, cm	9.1	10.0	9.1	9.3	9.0
Diameter after adding reagent, cm	2.9	1.2	0.9	3.2	1.9
Diameter after 24 hours, cm	2.9	1.4	1.0	3.3	2.1
Diameter after 56 hours, cm	3.0	1.4	1.2	3.5	2.1
Diameter after 104 hours, cm	3.0	1.4	1.2	3.5	2.1
Efficiency of the oil-collecting capacity of reagents on Salyan oil, %	67.04	86.00	86.81	62.37	76.67

Note: The tests were carried out by adding 2 mL of petroleum to 40 mL of water, followed by the addition of 2 g of a 5% solution of the sample.

Sample 1. IPA-28.5 g + Nitration product of diesel distillate-0.75 g + Zn salt based on the 340-410°C fraction of NPA-0.75 g

Sample 2. IPA-28.5 g + Nitration product of diesel distillate-0.75 g + Mn salt based on the 340-410°C fraction of NPA-0.75 g

Sample 3. IPA-28.5 g + Nitration product of diesel distillate-0.75 g + Fe salt based on the 340-410°C fraction of NPA-0.75 g

Sample 4. IPA-28.5 g + Nitration product of diesel distillate-0.75 g + Ni salt based on the 340-410°C fraction of NPA-0.75 g

Sample 5. IPA-28.5 g + Nitration product of diesel distillate-0.75 g + Co salt based on the 340-410°C fraction of NPA-0.75 g

In the conducted tests, the highest oil-collecting efficiency for Salyan oil from the surface of tap water was exhibited by the Fe salt of NPA (Sample-3), achieving a result of 90.22%. This value represents the highest recorded result for Salyan oil across all tests. The fact that the Fe salt of NPA demonstrated such a high performance in the 340-410°C fraction confirms that when synthesized with high-temperature fractions, this metal forms more effective surface-active structures.

Figure 3 illustrates the reduction of the oil slick over a specific period of time.



Figure 3. Oil-collecting capacity of the IPA + nitration product of diesel distillate + Fe salt of NPA composition for Salyan oil from the water surface

Studies conducted on the compositions of various metal salts of synthesized NPA show that the maximum oil-collecting capacity efficiency from the surface of both tap and sea water was recorded in the composition based on the Fe salt of NPA.

4. Investigation of the effect of additives on cetane number

The effect of additives prepared on the basis of metal salts of the 340-410°C fraction at 2 mmHg of NPA on the cetane number was investigated. Samples used in the study:

Sample-16: Zn salt of the 340-410°C fraction of NPA

Sample-17: Mn salt of the 340-410°C fraction of NPA

Sample-18: Ni salt of the 340-410°C fraction of NPA

Sample-19: Fe salt of the 340-410°C fraction of NPA

Sample-20: Co salt of the 340-410°C fraction of NPA

Table 9 presents the results of the effect of Samples 16 to 20, consisting of metal salts of NPA, on the cetane number of diesel distillate. The samples were tested by adding them to 100 g of diesel distillate at concentrations of 60 ppm and 500 ppm. An initial cetane number of 49.26 (without additive) was taken as the baseline for all tests.

At a concentration of 60 ppm, increase values among the samples constituted a range between 0.64 and 5.24 units. The highest increase at this concentration was recorded for the Fe salt of NPA (Sample-19) at 5.24 units. The Zn salt of NPA (Sample-16) showed an increase of 4.94 units, the Ni salt (Sample-18) 4.440 units, and the Co salt of NPA (Sample-20) 0.940 units. The lowest increase was determined for the Mn salt of NPA (Sample-17) at 0.640 units, with the final cetane number remaining at the level of 49.90 units. Furthermore, the variance among samples in this group was extremely high.

At a concentration of 500 ppm, it was observed that the increase values for all samples rose compared to the 60 ppm concentration. At this concentration, the highest increase was recorded for the Ni salt of NPA (Sample-18) at 7.34 units, with the final cetane number reaching 56.6 units.

Table 9.

Effect of using metal salts of the 340-410°C fraction at 2 mmHg of natural petroleum acid as diesel distillate additives on the cetane number

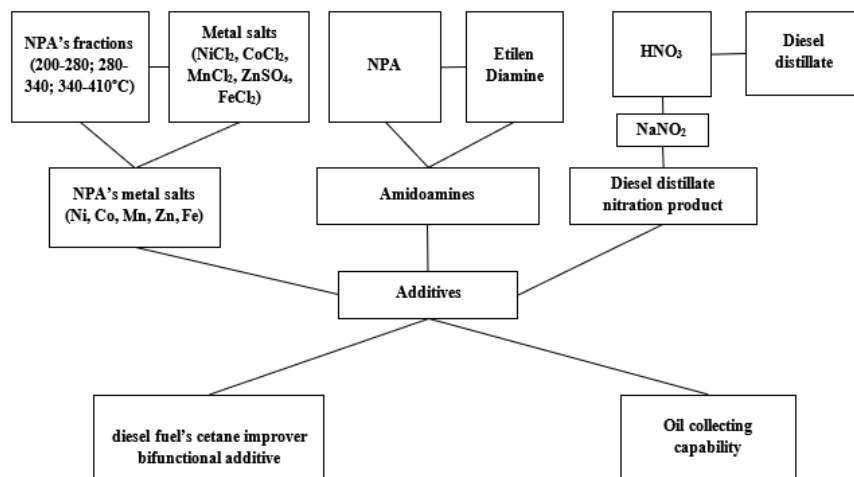
No.	Additive	Base Cetane Number, unit	Cetane Number with Additive, unit	Increase
			Dosage: 60 ppm	
1	Sample-16	49.26	54.2	4.94
2	Sample-17	49.26	49.9	0.64
3	Sample-18	49.26	53.7	4.44
4	Sample-19	49.26	54.5	5.24
5	Sample-20	49.26	50.2	0.94
			Dosage: 500 ppm	
1	Sample-16	49.26	56.5	7.24
2	Sample-17	49.26	50.6	1.34
3	Sample-18	49.26	56.6	7.34
4	Sample-19	49.26	56.4	7.14
5	Sample-20	49.26	51.7	2.44

The Zn salt of NPA (Sample-16) and Fe salt (Sample-19) showed close results with increases of 7.240 units and 7.140 units, respectively. Meanwhile, the Mn salt of NPA (Sample-17) demonstrated the lowest increase (1.340 units) at 500 ppm as well, with the final cetane number remaining at 50.60 units. Looking at the difference in increase between 60 ppm and 500 ppm, it was determined that this difference was 2.30 units for the Zn salt of NPA (Sample-16), 2.90 units for the Ni salt (Sample-18), and only 0.70 units for the Mn salt (Sample-17).

According to Scheme 1, the process of preparing additives that increase the cetane number and electrical conductivity of diesel distillate consists of 5 stages:

1. Synthesis of various metal (Ni, Co, Mn, Zn, and Fe) salts of NPAs was carried out.
2. A nitro compound product was synthesized on the basis of diesel distillate and nitric acid (69%).
3. Compositions of the synthesized metal salts with the nitration product of diesel were prepared.
4. Amidoamines of NPA with ethylenediamine were synthesized.

- Compositions of the synthesized amidoamines with various metal salts of NPA were prepared and evaluated in diesel distillate as additives that improve both antistatic properties, cetane number, and electrical conductivity.



Scheme 1. Preparation scheme of synthesized metal salts and amidoamine based on local raw materials, and their compositions as antistatic additives

5. Generalized Principal Technological Scheme of the Processes

A principal process flow scheme has been developed for the large-scale production of antistatic additives that increase the electrical conductivity and cetane number of diesel distillate (Scheme 2). The nitration process is carried out in reactor No. 1 (R-1) based on 69% nitric acid and diesel distillate feedstocks. NaNO₂ is added to the acid to intensify the reaction, shorten the induction period, and prevent auto-oxidation, with the resulting NO₂ ensuring the stability of the process.

After heating the acid introduced into the reactor up to 70°C, diesel distillate is added in portions, and the thermal regime is maintained in the range of 70±9°C via a heating jacket and an internal

coil system. After the reaction is completed and the system is cooled to $25\pm 5^{\circ}\text{C}$, the product separates into two phases: unreacted nitric acid is discharged into intermediate tank No. 7 (T-7), while the upper phase is washed with water, settled, fed to dryer No. 10, and stored in tank No. 12 (T-12) as a finished nitro-product. Gases evolved during the process are neutralized in neutralizing system No. 6.

To obtain metal salts, NPAs added to reactor No. 2 (R-2) are neutralized with a 40% NaOH solution at $40\text{--}50^{\circ}\text{C}$. Subsequently, diesel distillate as a solvent and an aqueous solution of the corresponding metal salt are added to the system and mixed. The water-mineral layer separated upon cooling is removed, and the metal salts of NPAs dissolved in diesel distillate in the upper layer are collected in tank No. 17 (T-17).

In the final stage, the metal salt of natural petroleum acids dissolved in diesel distillate from tank No. 17 (T-17) and the nitrated diesel distillate from tank No. 12 (T-12) are introduced into reservoir Q1 equipped with a special stirrer and intensively mixed for 30 minutes. The resulting finished additive composition is collected in tank No. 18 (T-18) and supplied for application.

Technical and economic evaluation:

Industrial production of a cetane-boosting additive was evaluated, thoroughly comparing efficiency against an analog. Economic benefits stem from two key factors: lower material consumption (analog usage is 1.5 times higher) and lower production cost (2,978 vs 3,026 AZN/t).

Annual savings:

For 2.3M t of diesel fuel, 138 t of Fe salt (410,962 AZN) replaces 207 t of Zn salt (626,382 AZN), delivering 215,420 AZN/year in savings.

CONCLUSION

1. Various metal salts of natural petroleum acids, as antistatic additives in diesel distillate, significantly increase electrical conductivity, and this effect varies depending on the nature of the metal, fractional composition, concentration, and time factor. Overall, the Fe and Co salts of NPA exhibited a higher antistatic effect [1, 4, 5, 6, 7].
2. Among the metal salts synthesized on the basis of various fractions of NPA, the highest electrical conductivity was recorded at 1892 pSm/m on the fortieth day with the 0.15% addition of the Fe salt synthesized based on the 340-410 °C fraction at 2 mmHg of natural petroleum acid and the amidoamine synthesized based on natural petroleum acid and ethylenediamine. Based on the indicators of testing conducted at "AR ETN TSZ" LLC, the obtained results were recommended for industrial application (Act attached) [6, 7, 8].
3. It was determined that the oil-collecting capacity from the water surface of compositions prepared on the basis of the nitration product of diesel distillate and metal salts of natural petroleum acid increased [3, 10, 11].
4. The composition of the nitration product of diesel distillate with the Fe salt synthesized based on the 340-410°C fraction at 2 mmHg of natural petroleum acid showed the highest result, with an efficiency of 90.22% for collecting Salyan oil from the surface of tap water [3, 10, 11].
5. The composition of the nitration product of diesel distillate with the Fe salt synthesized based on the 200-280°C fraction at 2 mmHg of natural petroleum acid showed the highest result, with an efficiency of 89.02% for collecting Naftalan oil from the surface of tap water. Based on the indicators of testing conducted at "Inkor" LLC, the obtained results were recommended for industrial application (Act attached) [3, 10, 11].
6. The highest result for increasing the cetane number (an increase of 7.34) was obtained with the Ni salt of the 340-410°C fraction at 2 mmHg of natural petroleum acid. However, the Fe salt of

the 340-410°C fraction at 2 mmHg of natural petroleum acid also gave a very high result with an increase of 7.14 [2, 9, 12].

LIST OF PUBLISHED SCIENTIFIC WORKS ON THE TOPIC OF THE DISSERTATION:

1. Həsənov, E.K., Təmrazlı, T.T., Əlizadə, R.A., Əliyev, T.S. Təbii neft turşusunun müxtəlif duzlarının dizel distillatına əlavə olunması ilə elektrik keçiriciliyinin təyini // "Təbii ehtiyatlar və tullantıların səmərəli istifadəsində ekosistemin qorunması" mövzusunda respublika elmi-praktik konfransının materialları, – Gəncə, – 25 aprel, – 2025, – 15-17 s.
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A handwritten signature in blue ink, consisting of stylized, overlapping loops and strokes, likely representing the initials or name of the author.

The defense will be held on 30 October 2026 at 10:00 at the meeting of the Dissertation council FD 1.17 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at the Institute of Petrochemical Processes named after Y.H. Mammadaliyev of the Ministry of Science and Education of the Republic of Azerbaijan

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