REPUBLIC OF AZERBAIJAN

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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

SYNTHESIS OF FUNCTIONAL SUBSTITUTED ESTERS OF CYCLIC POLYOLS AND THEIR STUDY AS A BASE AND COMPONENT OF LUBRICANTİNG OILS

Speciality: 2314.01- Petrochemistry

Field of science: Chemistry

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The work was performed at the laboratory of «Synthetic oil» of the Institute of Petrochemical Processes named after acad. Y.H. Mammadaliyev of the Ministry of Science and Education Republic of Azerbaijan.

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GENERAL CHARACTERISTICS OF WORK

Relevance of the work and degree of development. Currently, the rapid development of technology imposes strict requirements on the quality of used lubricants and fuels. The required lubricants should have high thermal and thermooxidation stability, high lubricity, appropriate viscosity-temperature properties in the temperature range of 250°C and minus 60°C, and low freezing point. Since currently used mineral and synthetic oils do not meet these indicators, purposeful synthesis of new compounds containing various functional groups, active centers and cyclic fragments and their research as the basis and component of lubricants is of scientific and practical importance.

The analysis of literature materials shows that among the studied compounds such as synthetic lubricants, ester-type compounds prevail. Currently used ester-type lubricants based on aliphatic polyols and polycarbonic acids and their compositions do not meet the ever-increasing demand of the industry. These esters lose their quality in a wide range of temperatures and do not withstand harsh conditions. This, in turn, leads to premature failure of machines and mechanisms, unwanted material losses.[1,2]

From this point of view, in order to create new lubricants with high operating properties that meet the requirements of modern and prospective technology, the synthesis of esters of cyclic neopolyols with different structures and their research as the basis and component of lubricants are of some relevance.

Object and subject of work. The object of the research is symmetric, asymmetric, complex, propylene oxide fragmented, nitrogen-containing esters synthesized on the basis of cyclic

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[1] Мамедьяров, М.А. Синтетические смазочные масла (структура и свойства) / М.А. Мамедьяров, Ф.Х. Алиева, Г.Н. Гурбанов, Москва; Научный мир, -2017. -335 с.

[2] Цветков, О.Н. Смазочные масла – основа технического прогресса / О.Н. Цветков // Мир нефтепродуктов, -2008. №2, -с.23-27.

neopolyols. The subject of the research is the study of the application of synthesized esters as the basis and component of lubricating oils, as additives that improve the operational properties of other oils, as complex additives to diesel fuels, as bactericide-inhibitor, fungicidal substances, and as corrosion inhibitors for conservation liquids.

The purpose and objectives of the dissertation work. The presented thesis is the synthesis of some symmetric, unsymmetrical, complex, nitrogen-containing esters of cyclic neopolyols - 2,2,5,5-tetramethylolcyclopentanol (TMCP) and 2,2,6,6-tetramethylolcyclohexanol (TMCH), dedicated to the study of their properties and application areas.

The following issues were resolved in the dissertation work:

- finding the optimal condition for the synthesis of cyclic neopolyols by aldol condensation of cyclic ketones - cyclopentanone and cyclohexanone with formaldehyde;
- synthesis of some symmetrical, unsymmetrical, complex, containing propylene oxide fragments and nitrogen esters of TMCP and TMCH, study of their properties;
- creation of new oil compositions with mineral oils, synthetic industrial oils based on TMCP and TMCH esters and their research
- improving the quality of diesel fuels by adding some esters of cyclic neopolyols;
- Testing of some esters of TMTP and TMTH as a new corrosion inhibitor to conservation liquids, studying bactericidal-inhibitory and fungicidal properties;
- optimization of the process of obtaining complex esters of TMCP with C_5-C_9 monocarboxylic acids.

Research methods. Modern physico-chemical analysis methods (IR- and NMR- spectroscopy, determination of acid number, etc.) were used during the identification and determination of the characteristics of the products obtained in the research work.

The main provisions put forward for defense.

- some symmetric and unsymmetrical esters of cyclic neopolyols - TMCP and TMCH with C_5-C_9 monocarboxylic acids were synthesized and their properties were studied;

- complex esters of TMCP and TMCH were synthesized, their physico-chemical and operational properties were studied;
- esters of TMCP and TMCH with propylene oxide fragment were synthesized and studied;
- nitrogen-containing esters of cyclic neopolyols were synthesized and their properties were studied;
- new oil compositions were created and studied with the addition of esters;
- taking into consideration that the symmetrical esters of TMCP have high indicators, the process of obtaining pentaester with caproic acid was optimized;
- the relationship between the structure of synthesized esters and the operating properties of oils was investigated in the research work.

Scientific novelty of the dissertation.

- some symmetrical, unsymmetrical, complex, propylene oxide fragmented and nitrogen-containing esters of TMCP and TMCH were synthesized, their physico-chemical, viscosity-temperature, thermal oxidation stability and lubrication properties were studied;
- synthesized esters as the basis and component of new lubricants, as additives that improve the operating properties of other oils, as complex additives to diesel fuels, and some esters, including synthesized nitrogen-retaining esters, were first used as anti-corrosion bactericide-inhibitors, fungicidal substances and has been tested as a corrosion inhibitor to conservation liquids.

Theoretical and practical value of the work. As the synthesized esters of TMCP and TMCH contain cyclic rings, quadruple C atoms without H atom in the β position, and polar ester groups, as well as nitrogen atom and propylene oxide fragment, they have high thermal oxidation stabilities, high (250^{o}C ≥) and low (minus 60^{o}C) have standard viscosity-temperature indicators, as well as low freezing point. The structure-property dependencies of the synthesized esters have been determined, which allows us to predict their properties in advance when the structure of the esters is known, thereby determining whether the synthesis is selective and economically efficient. Esters containing all these properties can be recommended as the basis and component of lubricanting oils that meet the requirements of new, modern and promising technology.

The addition of TMCP and TMCH esters to some mineral and synthetic lubricanting oils has been found to significantly improve their quality and is recommended as an additive to these oils.

By adding 0.004% of some symmetric and unsymmetrical esters to diesel fuel, high indicators were obtained: the thermal oxidation stability and flash point of the fuel increases, the amount of sediment decreases, and the freezing temperature decreases.

The anti-corrosion bactericidal-inhibitory and fungicidal properties nitrogenous esters, including some of the other group esters of cyclic polyols were tested and their use in production was recommended. Also, these esters were tested as corrosion inhibitors for conservation liquids and satisfactory results were obtained.

Personal participation of the author. The results reflected in the dissertation were obtained by the author. With the participation of the author, the issues were set, experiments and tests were carried out, results were systematized and summarized.

Approbation and application of the work. The main provisions and results of the dissertation were reported and discussed at the following republican and international scientific and technical conferences (10 theses):

International Scientific Conference dedicated to the 90th anniversary of the Institute of Petrochemical Processes (IPCP, Baku, 2019); "Actual problems of modern natural and economic sciences" International Scientific Conference dedicated to the 96th anniversary of the birth of national leader Heydar Aliyev (Ganja State University, Ganja, 2019); 32. Online National Chemistry Congress (Turkey, 2020); "Current problems of modern natural sciences" International Scientific Conference dedicated to the 97th anniversary of the birth of national leader Heydar Aliyev (Ganja State University, Ganja, 2020); Usbik 2021 online international congress on natural sciences (Turkey, 2021); Republican Scientific Conference dedicated to the 90th anniversary of Academician Nadir Mir-Ibrahim oglu Seyidov (IPCP, Baku, 2022); Republican Scientific Conference dedicated to the 110th anniversary of academician Ali Musa oglu Guliyev on "Organic substances and composition materials of various purposes" (ICA, Baku, 2022); "Current problems of modern natural and economic sciences" International Scientific Conference dedicated to the 100th anniversary of the birth of national leader Heydar Aliyev (Ganja State University, Ganja, 2023).

16 scientific articles on the results of the dissertation work (2 of them by a single author) were published in name of "Processes of petrochemistry and oil refining", "Oil refining and oil chemistry", "Azerbaijan Oil Industry", "World of oil products", "Proceedings of the Azerbaijan National Academy of Sciences", "Azerbaijan Chemical Journal", "Technologies of oil and gas" journals.

1 Azerbaijan Patent has been published.

Place of the dissertation work The work was carried out in the laboratory “Synthetic oils” of the Institute of Petrochemical Processes named after Academician Yu.H Mammadaliyev Ministry of Science and Education of Azerbaijan.

The total volume of the dissertation indicating the volume of structural sections. Dissertation work is 170 pages long, consists of introduction, 4 chapters, conclusions, bibliography, appendix and abbreviations. The work includes 53 tables and 21 pictures. The structure of the dissertation - introduction 10849, the first chapter 60051, the second chapter 42404, the third chapter 48364, the fourth chapter 14415, and the results consist of 3660 marks. The dissertation has a total volume of 179743 characters (excluding the table of contents, tables, pictures and bibliography).

In the introduction, the relevance, purpose, scientific novelty, theoretical and practical importance of the researches are given and justified.

In the first chapter, the existing esters of aliphatic and cyclic alcohols, their research as lubricants, components and additives to other oils and fuels, literature research on the latest situation and perspective of creating new oil compositions based on industrial lubricants were discussed and the issue was justified.

In the second chapter, the synthesis of primary neopolyols - TMCP and TMCH with C_5-C_9 monocarboxylic acids, some symmetric and unsymmetrical esters, complex esters with dicarboxylic acids, the esters containing a nitrogen atom and a propylene oxide fragment, and their physicochemical properties are given. In this chapter, taking into account that synthesizing ester of TMCP with C_5-C_9 monocarboxylic acids have higher indicators, the process of obtaining pentacaproate ester of TMCP was also optimized. Here, in addition to the obtained esters, the creation of new oil compositions in order to further improve the quality of mineral and synthetic oils was reflected.

In the third chapter, the viscosity-temperature, thermal oxidation stability and lubrication properties of synthesized esters of TMCP and TMCH with different functional composition were studied. Esters were compared among themselves, the dependence between the structure of esters and operational properties was systematically studied, and certain results were obtained. In order to improve the quality of mineral and synthetic industrial oils with the addition of esters, new oil compositions have been developed and studied.This chapter also tested some esters of neopolyols as new corrosion inhibitors for conservation liquids. Esters have been determined to have high bactericidal-inhibitory, fungicidal properties against corrosion. Certain positive results have been achieved in the field of improving the quality of diesel by adding them to diesel fuels.

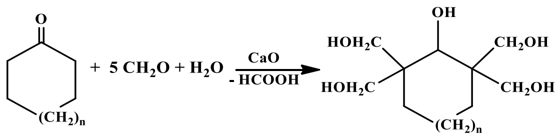
In the fourth chapter, the dependence of the viscosity-temperature and thermal oxidation properties of esters of cyclic neopolyols on their structure, as well as the dynamics of the effect of esters of different structures on the properties of diesel fuels were investigated.In conclusion, recommendations on practical application methods of esters of TMCP and TMCH are given.

At the end of the dissertation work, the results of the conducted research, a list of literature, appendix and abbreviations are given.

MAIN CONTENT OF THE WORK

Synthesis of cyclic neopolyols

Synthesis of primary raw materials cyclic neopolyols - 2,2,5,5-tetramethylolcyclopentanol (TMCP) and 2,2,6,6-tetramethylolcyclohexanol (TMCH) according to the known method according to the Cannizzaro reaction of cyclic ketones (cyclopentanone and cyclohexanone) in an alkaline medium (in the presence of CaO) with formaldehyde is carried out by aldol condensation. We have optimized the reaction and determined that the maximum yield of the final product (81%) is obtained when the ketone:formaldehyde:CaO molar ratio is 1:5.5:0.6, the temperature is 21°C and the pH of the medium is 10.8.



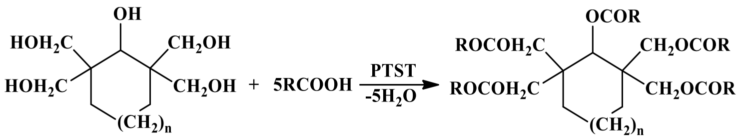
n=0, 1

Synthesized TMCP and TMCH were white crystalline substances with melting points of 132-133°C and 129-130°C, respectively.

Synthesis of symmetrical and unsymmetrical esters of cyclic neopolyols

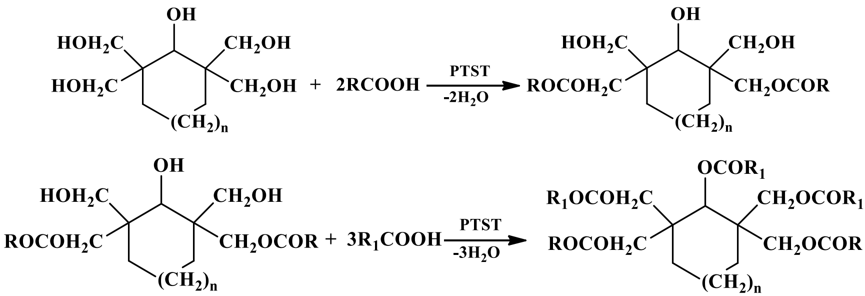
Synthesis of symmetrical esters of TMCP and TMCH with C_5-C_9 monocarboxylic acids was carried out. 1:6 molar ratio of polyol to acid (acid is removed so that the esterification is complete, and the excess acid is removed at the end), para-toluenesulfonic acid (PTSA) in the amount of 1% of the components taken as a catalyst and toluene as an azeotropic agent for 5 hours the reaction is continued until the complete separation of water at a temperature of 110-120°C.

After the end of the reaction, the obtained mass is neutralized with 5% NaOH solution, then washed with water until a neutral medium is obtained, dried over anhydrous Na_2SO_4 and distilled under vacuum. The probable scheme of the reaction is as follows:



R= C_5H_{11}- ÷ C_9H_{19}-; n= 0 (ester 1-5), n=1 (ester 10-14)

The synthesis of unsymmetrical esters of cyclic neopolyols is carried out in 2 stages: in the first stage, in order to obtain diester of the polyol with C_6-C_9 monocarboxylic acids, PTSA is used as a catalyst (in the amount of 1% of the components taken) and toluene is taken as an azeotropic agent for 5 hours at a temperature of 110-120°C the reaction is continued until separation of water is complete. In stage II, the reaction is continued by adding caproic acid to fully esterify the free hydroxyl groups. After the end of the reaction, the obtained mass is neutralized with 5% NaOH solution, then washed with water until a neutral medium is obtained, dried over anhydrous Na_2SO_4 and distilled under vacuum. The probable general scheme of the reaction is as follows:



R= C_6H_{13}- ÷ C_9H_{19}-; R_1= C_5H_{11}-; n=0 (ester 6-9), n=1 (ester 15-18)

Identification of the synthesized esters was carried out by IR- and NMR- spectroscopy methods, determination of acid number.

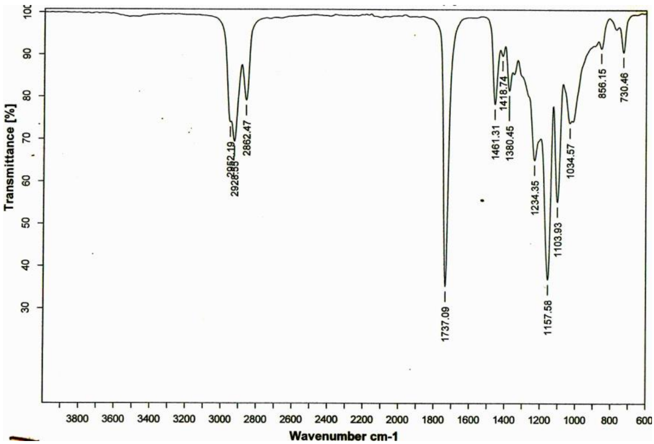


Fig. 1. IR spectrum of unsymmetrical ester of TMCH with caproic and enanthic acids (ester 15)

In the IR-spectrum of ester, 730, 1380, 1417, 1461 cm^{-1} strain and 2862, 2928, 2952 cm^{-1} valence oscillations are C-H bonds in CH_3 and CH_2 groups, and 1103, 1157, 1737 cm^{-1} valence oscillations prove that the ester has C-O and C=O connection.

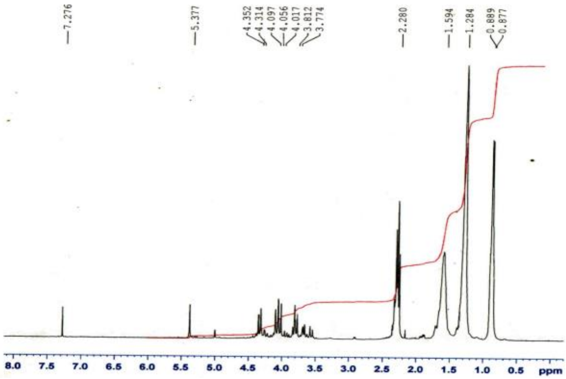


Fig. 2. ^{1}H NMR spectrum of unsymmetrical ester of TMCH with caproic and enanthic acids (ester 15)

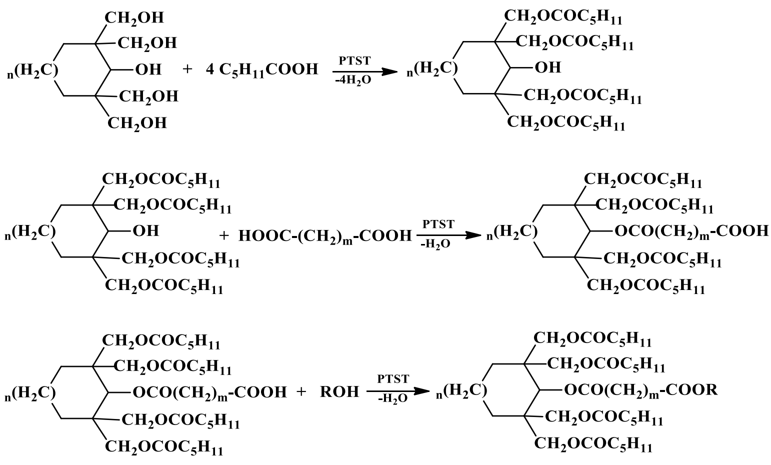
In the ^{1}H NMR spectrum of ester (BRUKER-Fourier 300 MHs, $CDCl_3$, δ, m.h.): 0.88 (15H, CH_3), 1.28 (26H, CH_2), 1.59 (14H, CH_2), 2.27 (10H, CH_2CO), 3.77-4.35 (8H, OCH_2), 5.37 (1H, OCH).

In the ^{13}C NMR spectrum of ester (BRUKER-Fourier 75 MHs, CDCl3, δ, m.h.): 13.86, 13.98 (CH_3), 27.92, 28.83 (C-CH_2), 22.27, 22.45, 24.44, 24.51, 24.73, 24.80, 28.79, 31.26 , 31.40, 33.47, 34.12, 38.31, 42.29 (CH_2), 62.42, 66.09, 66.38, 69.72 (OCH_2), 72.80 (OCH), 171.96, 173.22, 173.37, 173.53 (C=O).

Physico-chemical properties of esters have been studied. They are colorless liquids with a high boiling point, the yield of esters is 72-90%.

Synthesis of complex esters of cyclic neopolyols

Synthesis of complex esters was carried out in three stages, in the presence of PTSA catalyst, taking p-xylene as an azeotropic agent at a temperature of 140-150°C. In stage I, the tetraester of polyol is synthesized with kapronic acid, in stage II, the free hydroxyl groups are esterified with dicarboxylic acid, and in stage III, the free carboxyl group is esterified with alcohol. After the reaction, the esterified is washed with 5% NaOH solution and water until neutral. After removal of p-xylene and light volatile products of the reaction, the esters are obtained.



m=3,5,7; R=$-CH_3$, $-C_6H_{13}$, $-C_8H_{17}$; n=0 (ester 19-27), n=1 (ester 28-36)

The structures of complex esters were confirmed and their physical and chemical properties were studied.

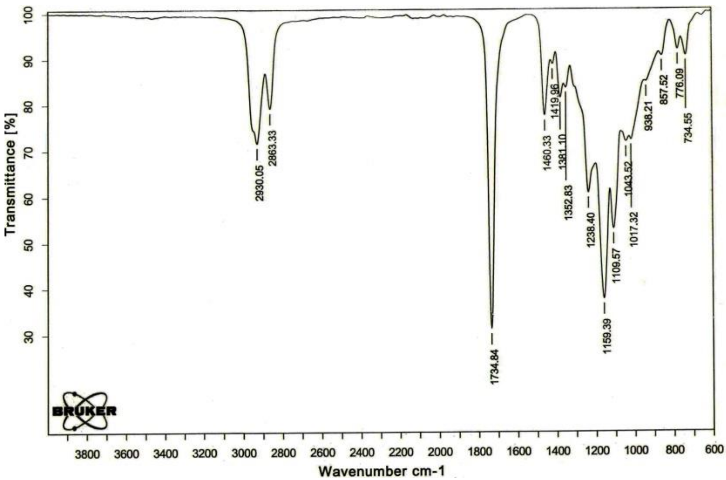


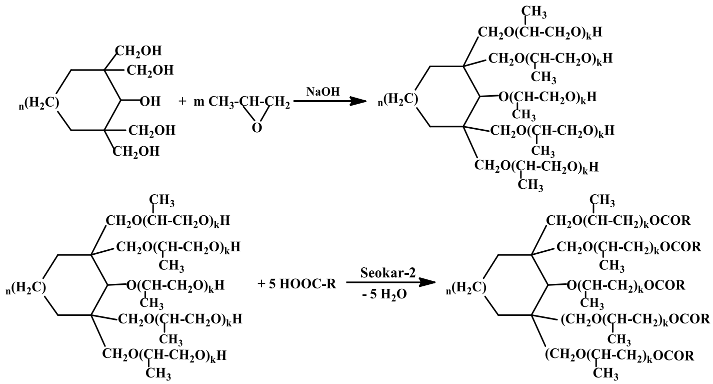
Fig. 3. IR-spectrum of the complex ester of TMCP with caproic, glutaric acids and hexyl alcohol (ester 20)

In the IR spectrum of complex ester, 734, 1381, 1419, 1460 cm^{-1} strain and 2863, 2930 cm^{-1} valence oscillations are C-H bonds in CH_3 and CH_2 groups, and 1109, 1159 and 1734 cm^{-1} valence oscillations are C-O and C=O of ester characterizes their relationships.

Complex esters have a high refractive index and density, their yield is 70-85%.

Synthesis of esters of cyclic neopolyols containing the propylene oxide fragment

The synthesis of complex esters of TMCP and TMCH containing the propylene oxide fragment was carried out in 2 stages: in the first stage, oxypropylation of the polyol was carried out in an autoclave at a temperature of 150-160^{o}C for 5 hours, in the ratio of polyol:propylene oxide 1:6 and 1:10 and in the presence of the NaOH catalyst taken in the amount of 1% of the total mass of the components included in the reaction. The obtained oxypropylated product was subjected to vacuum distillation to separate into fractions. In the second stage, the obtained oxypropylating ether of polyol were fully esterified with C_4-C_8 monocarbon acids at 140-150^oC, in the presence of Seokar-2 in the amount of 1% of the total mass as a catalyst, and p-xylene as an azeotropic agent for 6-8 hours.



m=6-10; k=1-2; R= C_4H_9-÷ C_8H_{17}-; n=0 (ester 37-41), n=1 (ester 42-46)

The esters obtained are highly viscous products with high boiling points.

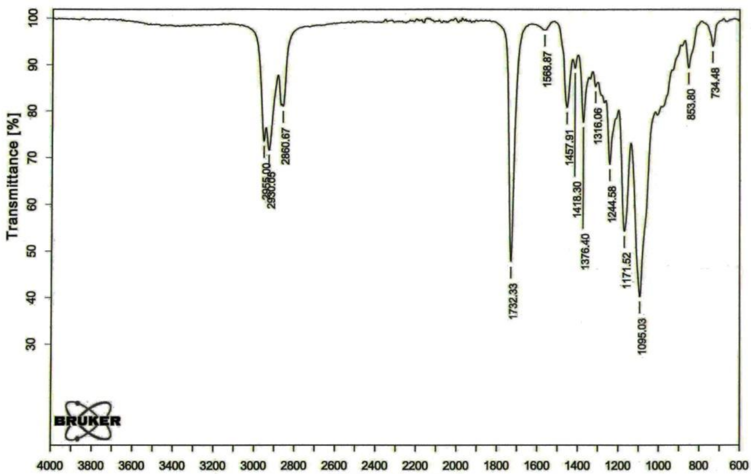


Fig. 4. IR spectrum of the pentacaproate ester of TMCH containing the propylene oxide fragment (ester 43)

In the IR spectrum of ester, 734, 1376, 1418, 1457 cm^{-1} strain and 2860, 2930, 2955 cm^{-1} valence oscillations of C-H bonds in CH_3 and CH_2 groups, 1095 cm^{-1} valence oscillations of C-O bond of ether, 1171 and 1732 cm^{-1} valence oscillations characterize the C-O and C=O bonds of ester, respectively.

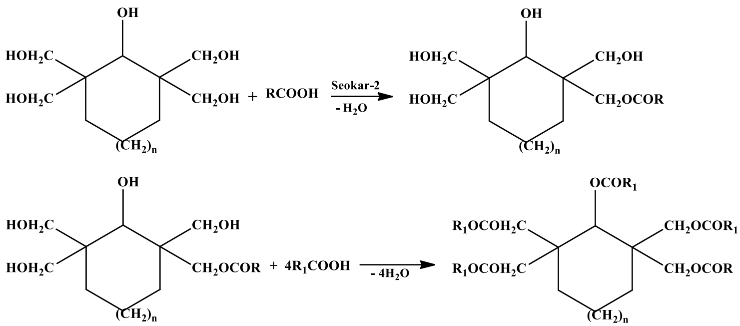
The yield of esters is 60-78%, they are high molecular compounds with a high refractive index.

Synthesis of nitrogen-containing esters of cyclic neopolyols

Since the possibility of using N-containing esters of TMCP and TMCH as lubricants is of scientific interest, their esters with 2-aminopropionic, 2-aminoisovaleric and caproic acids were synthesized.

Obtaining of esters was carried out in 2 stages: in the first stage, taking polyols with amino acids in a 1:1 mol ratio, p-xylene as an azeotrope agent, and Seokar-2 up to 1% of the reacting mixture as a catalyst, at a temperature of 140-150°C for about 5 hours the reaction is continued until the end of water separation. In the next step, free hydroxyl groups are completely esterified with caproic acid (1:4 mole ratio).

The probable general synthesis scheme of esters can be shown as follows:



R= $-CH(NH_2)-CH_3$, $-CH(NH_2)-CH(CH_3)-CH_3$; $R_1=C_5H_{11}$;

n=0 (efir 47-48); n=1 (efir 49-50)

Nitrogen-containing esters are viscous, lubricant-type compounds. The yield of esters is 70-80%.

The structure of the synthesized esters was confirmed.

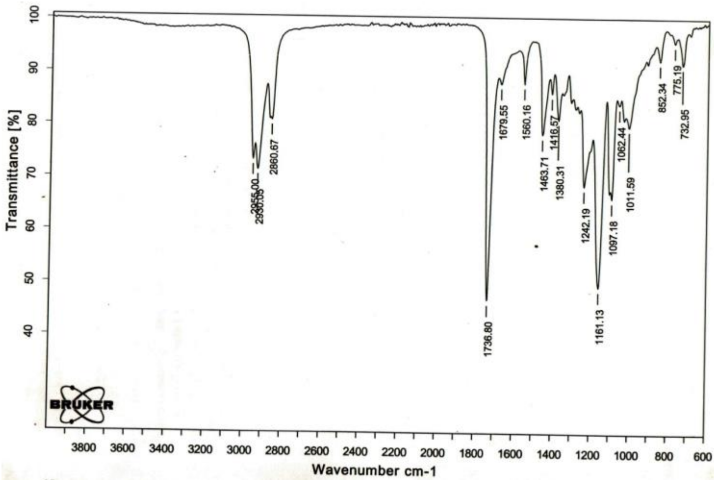


Fig.5. IR spectrum of the ester of TMCH with 2-aminoisovaleric and caproic acids (ester 50)

In the IR-spectrum of ester, deformations at 1380, 1416, 1463 cm^{-1} and valence oscillations at 2860, 2930, 2955 cm^{-1} indicate C-H bonds in CH_3 and CH_2 groups. The deformation absorption band characteristic of the H-N group is observed at 1560 cm^{-1}, and the absorption band characteristic of the valence oscillations corresponding to the C-N group is observed at 1242 cm^{-1}. The absorption bands at 1736 and 1161 cm^{-1} correspond to the C=O and C–O group, respectively, of the ester.

Study of operational properties of esters of cyclic neopolyols

Viscosity-temperature, thermal oxidation stabilities and lubrication properties of symmetrical and unsymmetrical esters of TMCP and TMCH were studied.

Table 1 shows the viscosity-temperature properties of esters.

As can be seen from table 1 symmetrical and unsymmetrical esters have a viscosity of 7.90-10.71 mm^2/s at 100°C, a flash point of 281-332°C and a viscosity index of 124-138 units, and a freezing point of minus 38°C - minus 58°C. The highest indicator for flash point, freezing temperature and viscosity index belongs to the unsymmetrical ester of TMCP.

Table 1

Viscosity-temperature properties of symmetric and unsymmetric esters

	№	R-acid radicals	Viscosity, mm²/s			Viscosity index	Temperature, °C	
			100°C	40°C	-30°C		flash	freezing
Esters of TMCH	1	C_5H_{11}-	7.90	34.2	26346	125	290	-53
	2	C_6H_{13}-	8.38	35.1	25042	132	299	-51
	3	C_7H_{15}-	9.12	38.26	27012	130	311	-49
	4	C_8H_{17}-	9.86	45.23	28060	126	320	-46
	5	C_9H_{19}-	10.25	47.12	30026	130	332	-45
	6	C_5H_{11}-, C_6H_{13}-	8.12	31.61	25216	134	295	-52
	7	C_5H_{11}-, C_7H_{15}-	8.61	36.28	26763	135	309	-50
	8	C_5H_{11}-, C_8H_{17}-	8.95	38.16	27021	138	332	-50
	9	C_5H_{11}-, C_9H_{19}-	9.20	41.66	28013	132	325	-58
Esters of TMCH	10	C_5H_{11}-	10.28	42.23	26500	125	281	-47
	11	C_6H_{13}-	10.35	42.41	27200	128	286	-45
	12	C_7H_{15}-	10.58	44.82	24600	124	298	-40
	13	C_8H_{17}-	10,62	45.13	24200	126	305	-38
	14	C_9H_{19}-	10.71	47.11	25300	130	315	-36
	15	C_5H_{11}-, C_6H_{13}-	10.42	43.28	25100	132	285	-45
	16	C_5H_{11}-,C_7H_{15}-	10.47	44.51	24300	135	291	-44
	17	C_5H_{11}-, C_8H_{17}-	10.60	45.16	23800	131	306	-42
	18	C_5H_{11}-, C_9H_{19}-	10.64	45.28	23600	133	310	-40
	Esters of PET with C_5-C_9 fr. of mixture of fatty acids		4.50	14.90	9260*	130	246	-58

* at minus 40°C

A comparison of the esters' performance with pentaerythritol (PET) esters shows that they have superior properties.

The thermal oxidation stabilities of esters were studied and it was determined that these esters are quite stable during oxidation. Esters have low volatility (0.010-0.060% by mass) and the amount of precipitate insoluble in isooctane is 0.002-0.015% by mass. Aluminum AK-4 (0.009-0.24 mg/cm^2) and steel ШХ-15 (0.016-0.040 mg/cm^2) electrodes were slightly corroded. After oxidation, the acid number of esters is 0.15-1.60 mgKOH/g. When comparing the operating properties of esters, it was found that unsymmetrical esters have better indicators than symmetrical ones, which is explained by their molecular structure, that is, the composition of ester groups and the location conformation. The indicators of esters were compared with PET esters (volatility - 1.2% mass, amount of sediment - 0.021% mass, acid value - 1.89 mgKOH/g) and it was clear that esters have certain advantages due to thermal oxidation properties.

The study of the lubricating properties of esters shows that they have a good lubricating ability: when the amount of critical load changes in the range of Pcr,H=690-840, the diameter of the wear is Dw=0.55-0.80 mm. When compared with the lubricating properties of PET esters (Pcr,H=500, Dw= 0.79 mm), it was found that these esters are superior. High lubricity is explained by the chemical structure of esters, that is, the presence of a large number of polar ester groups and cyclic fragments in the molecule plays an important role in creating a strong oil layer between rubbing surfaces. Unsymmetrical esters have better performance than symmetrical ones, which is explained by the spatial structure of their molecule, as well as the composition and location of ester groups. It is also known that unsymmetrical esters form stronger donor-acceptor bonds with rubbing surfaces and thus represent an additional polar center that improves the anti-aging properties of oils.

Thus, symmetric and unsymmetrical esters have good performance properties compared to pentaerythritol esters, and they can be proposed as a base and component of new lubricants.

The operating properties of the complex and propylene oxide containing esters synthesized on the basis of TMCP and TMCH were also studied.

Viscosity-temperature properties of complex esters are given in table 2.

As can be seen from the table 2, complex esters have high viscosity at 100°C (14.12-23.61 mm^2/s), viscosity index (123-142 units) and flash point (281-355°C), relatively low freezing temperature (minus 30°C÷ minus 43°C).

Table 2

Viscosity-temperature properties of complex esters

№	Composition of esters	Viscosity, mm²/s,		Viscosity index	Temperature, °C	
		40°C	100°C		flash	freezing
1	**2**	**3**	**4**	**5**	**6**	**7**
19	TMCP, glutar, caproic acids,methanol (1:1:4:1)	43.16	14.12	130	281	-39
20	TMCP, glutar, caproic acids, hexanol (1:1:4:1)	51.72	16.48	133	296	-36
21	TMCP, glutar, caproic acids, 2-ethylhexanol (1:1:4:1)	57.61	18.63	142	312	-41
22	TMCP, pimeline, caproic acids, methanol (1:1:4:1)	49.12	15.70	129	290	-38
23	TMCP, pimeline, caproic acids, hexanol (1:1:4:1)	56.23	17.90	136	320	-37
24	TMCP, pimeline, caproic acids, 2-ethylhexanol (1:1:4:1)	60.49	19.57	138	328	-43
25	TMCP, azelain, capron, acids, methanol (1:1:4:1)	53.18	17.12	140	301	-36
26	TMCP, azelain, capron, acids, hexanol (1:1:4:1)	59.35	19.56	132	330	-35
27	TMCP, azelain, capron, acids, 2-ethylhexanol (1:1:4:1)	71.11	22.90	135	337	-39
28	TMCH, glutar, caproic acids, methanol (1:1:4:1)	61.72	15.28	136	296	-36
29	TMCH, glutar, caproic acids, hexanol (1:1:4:1)	74.68	18.26	132	312	-33
30	TMCH, glutar, caproic acids, 2-ethylhexanol (1:1:4:1)	80.12	19.18	140	321	-38

Continuation of Table 2

1	2	3	4	5	6	7
31	TMCH, pimeline, caproic acids, methanol (1:1:4:1)	65.52	16.33	128	328	-33
32	TMCH, pimeline, caproic acids, hexanol (1:1:4:1)	71.63	17.98	125	334	-31
33	TMCH, pimeline, caproic acids, 2-ethylhexanol (1:1:4:1)	81.39	20.01	133	341	-30
34	TMCH, azelain, capron, acids, methanol (1:1:4:1)	69.22	17.48	123	346	-34
35	TMCH, azelain, capron, acids, hexanol (1:1:4:1)	79.48	19.56	131	349	-32
36	TMCH, azelain, capron, acids, 2-ethylhexanol (1:1:4:1)	94.91	23.61	135	355	-36

The comparison of esters among themselves shows that the highest indicator in terms of viscosity index and freezing temperature belongs to esters of TMCP, and in terms of flash point to ester of TMCH.

Thermal oxidation stabilities of complex esters were studied and it was determined that esters have a low acid number after oxidation (2.23-3.26 mgKOH/g), a precipitate insoluble in isooctane (0.091-0.20% mass), volatility (0.361-0.83%) and mild corrosion (AK-4 - 0.072-0.14 mg/cm^2, ШХ-15- 0.086-0.20 mg/cm^2) were observed.

The study of the lubricating properties of complex esters shows that they have high anti-wear and anti-friction properties: when the critical load is Pcr,H=820-980, the diameter of the wear circle is Dw=0.40-0.60mm. These high molecular weight esters withstand harsh conditions and form a strong and durable oil film between rubbing surfaces due to the presence of many polar ester groups.

The operational properties of complex esters were compared with individual analogs of cyclic neopolyols and PET esters and their significant advantage was determined.

The operating properties of complex esters of cyclic neopolyols with propylene oxide fragment were studied.

Table 3 shows the viscosity-temperature properties of esters

Table 3

Viscosity-temperature properties of esters

	№	R-acid radicals	Viscosity, mm^2/s		Viscosity index	Temperature, °C	
			40°C	100°C		flash	freezing
Esters of TMCP	37	C_4H_9-	92.48	23.50	145	305	-43
	38	C_5H_{11}-	130.95	28.06	133	320	-41
	39	C_6H_{13}-	136.80	30.12	140	330	-40
	40	C_7H_{15}-	150.12	33.72	135	350	-42
	41	C_8H_{17}-	160.43	40.22	150	380	-38
Esters of TMCH	42	C_4H_9-	130.00	28.42	136	306	-34
	43	C_5H_{11}-	141.16	30.71	139	315	-32
	44	C_6H_{13}-	145.28	31.93	137	322	-29
	45	C_7H_{15}-	150.12	33.55	140	330	-26
	46	C_8H_{17}-	156.71	35.48	143	341	-24

As can be seen from the table, esters have medium viscosity at 100°C (23.50-40.22 mm^2/s), low freezing point (minus 28 ÷ minus 43°C), high viscosity index (133-150 units) and flash point (305-380 °C).

The study of thermal oxidation stabilities shows that after oxidation, esters have a low acid value (2.50-3.28 mgKOH/g), corrosion (AK-4 - 0.06-0.16 mg/cm^2 and ШХ-15 - 0.07-0.17 mg/cm^2), a precipitate insoluble in isooctane (0.10-0.248% by mass) and volatility (0.30-0.50% by mass).

As for the lubricating properties of esters, they have satisfactory properties against corrosion and friction: Pcr,H=700-900, Dw=0.50-0.70 mm. This is explained by the fact that esters have a high molecular mass, multipolar ester groups and cyclic fragments. For this reason, they form a stable oil layer at high temperatures, thereby ensuring the reliability and durability of friction parts and mechanisms operating in a wide temperature range.

The fact that ester of oxypropylated TMCP and TMCH have high operational properties compared to pentacaproate ester and PET esters opens great opportunities for creating new promising lubricants based on them.

Testing of esters of cyclic neopolyols as bactericide-inhibitors, antifungals and corrosion inhibitors for conservation liquids

In order to investigate other areas of application of esters of cyclic polyol, the bactericidal-inhibitory, fungicidal properties of some of them were studied, and they were tested as corrosion inhibitors for conservation liquids.

Nitrogen-containing esters of TMCH (ester 49 and ester 50), symmetrical ester of TMCP with enanthic acid (ester 2), unsymmetrical esters of TMCH with caproic and enanthic, caproic and caprylic, caproic and pelargonic acids (ester 15, ester 16, ester 17, respectively), complex esters of TMCH with caproic, glutaric acids and hexyl alcohol (ester 29) were studied as a against the sulfate-reducing bacteria (SRB). The obtained results are listed in table 4 in comparison with industrial reagents (АМДОР-ИК-7 and АМДОР-ИК-10).

As can be seen from the table, esters showed higher results compared to industrial reagents at all concentrations. The highest results for nitrogen-containing esters were recorded especially at 200 mg/l concentration. The 10% complexes showed 94.4 and 95.1% bactericidal properties, reducing the number of bacteria from a million to 10^1, thereby significantly stopping the development of bacterial cells. As for other esters, the highest result belongs to complex ester (ester 29). The results of esters, not only at concentrations of 50 and 100 mg/l, but also at concentrations of 25 mg/l (77.1-99.6%), are higher than the results of industrial reagents.

Table 4
Bactericidal properties of esters

The name of the complexes	Concentration of the substance, C-mg/l	Bacterial count(cell count/ml)	Amount of H_2S, mg/l	Bactericidal effect, Z-%
1	**2**	**3**	**4**	**5**
5% ester 2	25	10^1	31.8	92
	50	10^1	15.7	95.8
	100	-	-	100
10% ester 2	25	10^1	2.6	99.3
	50	-	-	100
	100	-	-	100
5% ester 15	25	10^2	69.5	82
	50	10^1	23.1	94
	100	-	-	100
10% ester 15	25	10^2	58.1	84.7
	50	10^1	13	96.5
	100	-	-	100
5% ester 16	25	10^3	87.3	77.1
	50	10^1	40	89.5
	100	-	-	100
10% ester 16	25	10^2	80	79
	50	10^1	30.3	92
	100	-	-	100
5% ester 17	25	10^1	66.9	82.4
	50	10^1	37	90.3
	100	-	-	100
10% ester 17	25	10^1	48	87.4
	50	10^1	29	92.4
	100	-	-	100
5% ester 29	25	10^1	1.95	99.4
	50	-	-	100
	100	-	-	100
10% ester 29	25	10^1	1.23	99.6
	50	-	-	100
	100	-	-	100
5% ester 49	50	10^3	102	72.8
	100	10^2	54.9	85.3
	200	10^1	31.7	91.5

Continuation of Table 4

1	2	3	4	5
10% ester 49	50	10^2	62.5	83.3
	100	10^1	30	91
	200	10^1	18.2	95.1
5% ester 50	50	10^3	116	69
	100	10^2	70	81.3
	200	10^1	36	90.4
10% ester 50	50	10^2	81.3	78.3
	100	10^1	40.9	89
	200	10^1	21	94.4
Control-I amount of H_2S in medium without SRB	28 mg/l			
Control-II amount of H_2S in medium with SRB	382 mg/l			
Control-III number of bacteria in nutrient medium	10^8 cell count/ml			
АМДОР-ИК-7	50	-	-	60
	100	-	-	75
АМДОР-ИК-10	50	-	-	40
	100	-	-	80

Thus, all esters at a concentration of 100 mg/l, and ester 2 and ester 29 at a concentration of 50 mg/l showed 100% bactericidal properties and reduced the number of bacteria to zero.

In order to study the fungicidal properties of esters, 7 samples of esters: nitrogen-containing esters of TMCH (ester 49 and ester 50), symmetrical ester of TMCP with enanthic acid and symmetrical ester of TMCH with caproic acid (ester 2 and ester 10, respectively), symmetrical ester of TMCH with caproic and enanthic, caproic and caprylic, caproic and pelargonic acids (ester 15, ester 16, ester 17, respectively) against *Aspergillius niger* and *Penicillium digitatium* fungi were studied. According to the obtained results, only ester 49 had a strong effect against both fungi and prevented their development. Ester 50, ester 2, ester 15, ester 16 and ester 17 had a strong effect against Aspergillius niger fungus and stopped its development, and had a weak effect against Pencilium digitatium fungus. Ester 10, on the contrary, had a strong effect against the fungus Pencilium digitatium, and had a weak effect against the fungus Aspergillius niger, causing the development of the fungus.

3 and 10% compositions of some esters of TMCP and TMCH with T-30 turbine oil were prepared and tested as a corrosion inhibitors for conservation liquids. For this purpose, "Г-4" hydrocamera, sea water and 0.001% H_2SO_4 solution were selected as an aggressive environment.

Table 5

Corrosion protection properties of compositions

Examples	Inhibitor concentration, %	Corrosion protection, days		
		«Г-4» Hydrocamera	Sea water	0.001% H_2SO_4 solution
Ester 16+Ester 50	3	167	28	26
	10	202	57	55
Ester 2	10	110	28	25
Ester 15	10	120	34	31
Ester 16	10	117	32	21
Ester 17	10	115	30	27
Ester 29	3	105	25	22
	10	162	70	62
T-30 oil	100	35	15	9
НГ-203А	-	83	16	-

As can be seen from the table, the highest results were observed in nitrogen-containing ester and 10% compositions. The comparison of the compositions with T-30 oil shows that the results of the compositions in all concentrations and in all three aggressive environments (especially in the "Г-4" hydrocamera) are much higher than the results of the oil itself. The test results were also compared with the НГ-203А inhibitor used in the industry. As can be seen from the table, the compositions prepared with the addition of esters in the "Г-4" hydrocamera and sea water have higher indicators compared to the industrial inhibitor.

Investigation of operational properties of new oil compositions based on cyclic neopolyol esters

Various esters of cyclic neopolyols have been used to improve the quality of a number of mineral oils (T-30, T-46) and synthetic industrial oils (AB, DOS, PET, PAO).

For this purpose, complex esters of TMCP (ester 22 and ester 24) are added to DOS oil, symmetrical and unsymmetrical esters of TMCP (ester 1 and ester 7) to PET oil, mixed esters of TMCP and TMCH to PAO and AB oils, symmetrical (ester 1 and ester 10) and unsymmetrical esters (ester 7 and ester 16) of TMCP and TMCH to T- 30 and T-46 oils, by adding in the amount of 5, 10, 20% of esters and new oil compositions were created, their viscosity-temperature, thermal oxidation stabilities and lubrication properties were studied.

Viscosity-temperature properties of the compositions are given in table 6 for comparison.

Table 6

Viscosity-temperature properties of compositions

Compositions	Viscosity, mm^2/s		Viscosity index	Temperature, °C	
	100°C	40°C		flash	freezing
1	**2**	**3**	**4**	**5**	**6**
DOS + 10% ester 22	5.46	17.19	132	237	-58
DOS + 20% ester 22	5.51	18.80	143	251	-56
DOS + 10% ester 24	5.42	18.09	136	253	-60
DOS + 20% ester 24	5.80	19.27	149	278	-50
DOS oil	3.2	8.89	130	216	-60
PET + 10% ester 1	4.78	4.96	128	210	-58
PET + 20% ester 1	4.96	15.21	130	235	-58
PET + 10% ester 7	4.91	5.10	132	223	-58
PET + 20% ester 7	5.18	17.42	136	258	-56
Б-3В industrial oil based on PET	4.70	13.85	130	210	-58
PAO + 10% [ester 10+PET ester]	10.01	8.23	130	255	-52
PAO + 20% [ester 10+PET ester]	10.22	40.11	132	281	-54
PAO + 10% [ester 1+PET ester]	9.38	6.21	130	263	-56

Continuation of Table 6

1	2	3	4	5	6
PAO + 20% [ester 1+PET efiri]	9.30	34.41	135	290	-58
AB + 20% [ester 10 + PET ester]	12.05	49.03	140	275	-52
AB + 20% [ester 1 + PET ester]	11.80	48.74	155	293	-58
T-30 + 20% ester 7	7.56	42.11	86	276	-48
T-30 + 10% ester 16	7.95	46.16	75	273	-43
T-30 oil	6.20	47.9	64	214	-34
T-46 + 10% ester 7	8.60	41.23	135	260	-45
T-46 + 20% ester 7	8.70	43.01	140	280	-48
T-46 oil	8.61	81.33	69	199	-30

As can be seen from the table, when comparing with the properties of oils, all the parameters of the compositions increased with the addition of esters: the viscosity index and flash point increased, and the freezing temperature decreased. The highest results were observed in 10 and 20% compositions

Table 7 shows the TOS indicators of the compositions. According to the obtained results, compared to the oils themselves, the acid number of the compositions, the amount of precipitation, volatility is much lower, slight corrosion was observed.

Table 7

Thermal oxidation stabilities of compositions

Conpisitions	Viscosity, 100°C, mm^2/s, (after oxid.)	Acid number, mgKOH /g	Amount of precipitate insoluble in isooctane, %	Corrosion, mg/cm^2		Volatility, %
				АК-4	ШХ-15	
1	**2**	**3**	**4**	**5**	**6**	**7**
DOS+20% ester 22	6.28	2.01	0.72	0.14	0.12	1.85
DOS+20% ester 24	6.33	2.90	1.04	0.15	0.11	2.03
DOS oil	4.95	6.42	1.41	0.24	0.21	2.1
PET+10% ester 1	6.56	1.15	0.0018	0.065	0.019	0.08
PET+20% ester 7	6.92	1.20	0.0019	0.075	0.023	0.082
PET oil	5.88	1.93	0.027	0.13	1.82	1.60
PAO+20%[ester+ PET ester]	13.48	1.10	0.02	0.10	0.55	0.96
PAO+20%[ester 1 + PET ester]	12.06	1.08	0.018	0.25	0.40	0.85

Continuation of Table 7

1	2	3	4	5	6	7
PAO oil	11.48	1.40	0.08	1.12	1.26	1.33
AB+20% [ester 10 + PET ester]	13.90	1.25	0.010	0.07	0.70	1.10
AB+20% [ester 1 + PET ester]	13.15	1.06	0.055	0.03	0.65	0.95
AB oil	14.60	1.38	0.03	0.18	0.93	1.40
T-30+20% ester 1	7.80	-	0.22	-	-	0.17
T-30+20% ester 10	8.12	-	0.30	-	-	0.16
T-30+20% ester 7	8.41	-	0.20	-	-	0.12
T-30+20% ester 16	9.14	-	0.40	-	-	0.16
T-30 oil	7.50	-	0.36	-	-	0.24
T-46+20% ester 1	9.26	-	0.38	-	-	0.00
T-46+20% ester 10	10.20	-	0.28	-	-	0.00
T-46+20% ester 7	9.70	-	0.32	-	-	0.00
T-46+20% ester16	10.60	-	0.43	-	-	0.00
T-46 oil	9.61	-	0.51	-	-	0.25

The TOS indicators of the compositions prepared with the addition of unsymmetrical esters are better than those of symmetrical esters. The positive effect of complex esters is explained by the presence of a cyclopentane ring, a large number of polar ester groups, oxidation-resistant 4-carbon atoms in the 2,5 position of the cyclic ring.

The lubricating properties of the compositions were studied and the results are given in table 8.

Table 8

Lubricating properties of compositions

Compositions	Amount of critical load, Pcr,H	The diameter of the wear circle, Dw, mm P=196H
DOS +20% ester 22	670	0.55
DOS + 20% ester 24	710	0.60
DOS	500	0.69
PET ester +20% ester 1	650	0.70
PET ester + 20% ester 7	650	0.55
PET oil	500	0.79

As can be seen from the table, the compositions have a high lubricating capacity. With the addition of esters, the diameter of the wear circle decreased. The highest results were observed in 20% compositions of unsymmetrical and complex esters of TMCP, which can be explained by the fact that esters have a high polarity and are better adsorbed on the metal surface and form a continuous layer between the rubbing surfaces.

Thus, as a conclusion of the study, it can be noted that the prepared oil compositions meet all the requirements of base oils currently applied in the industry and considered in the future, so they can be used in various areas of the industry.

Improvement of the quality of diesel fuels by addition of esters of cyclic neopolyols

In order to improve the quality of diesel fuels, symmetrical esters of TMCP and TMCH with caproic acid (ester 1 and 10), unsymmetrical esters with caproic and caprylic acids (ester 7 and 16) and complex esters with caproic, pimelic acids and hexyl alcohol (ester 23 and ester 32) were tested as antioxidant additives to improve thermal oxidation stability by adding 0.004% to diesel fuel.

According to the obtained results, the addition of esters had almost no effect on the fractional composition of diesel fuel, 96% was distilled in the temperature range of 350-362^{o}C, the density of all samples was 845-847 kg/m^3. The amount of sediment decreased from 8.6 to 0.6-3.6 mg/100 ml, the flash point in a closed furnace increased from 76^{o}C to 88^{o}C, and the freezing temperature decreased from minus 18.2^{o}C to minus 25.1^{o}C. The best indicators were observed in symmetrical and asymmetrical esters of TMCP, which is directly related to their structure. Considering the above, these esters can be recommended as complex additives to diesel fuels.

The structure-property dependence of the synthesized esters was also investigated in the research work, and certain results and regularities were obtained, which, when the structure of the esters is known, make it possible to predict their properties in advance, thereby determining whether the synthesis is selective and economically efficient.

CONCLUSION

1. Some symmetrical and unsymmetrical esters of 2,2,5,5-tetramethylolcyclopentanol (TMCP), 2,2,6,6-tetramethylol-cyclohexanol (TMCH) with C_5-C_9 aliphatic monocarbon acids were synthesized, their physico-chemical and viscosity-temperature, thermal oxidation stabilities (TOS), lubrication properties were studied. It has been found that these esters have good viscosity-temperature indicators, viscosity index, high flash point, low freezing temperature, high TOS and lubrication properties [2,3, 7, 13, 15].
2. Complex esters of TMCP and TMCH with caproic, glutaric, pimelic, azelaic acids, 2-ethylhexanol, hexanol and methanol were synthesized in 3 stages, their physico-chemical, viscosity-temperature, TOS properties were studied. Complex esters have a viscosity of 16.48-22.90 mm^2/s at 100^oC, a viscosity index of 130-140 units, a flash point of 300-337^oC, a freezing temperature of minus 38^oC-minus 41^oC. These esters are recommended as high temperature resistant special purpose oils and as components or additives to other oils to improve their operational properties [1, 4, 6, 11].
3. Esters of oxypropilated TMCP and TMCH with C_4-C_8 monocarboxylic acids were synthesized and their operational properties were studied. It was found that they have high thermal oxidation stability, viscosity-temperature indicators (viscosity at 100^oC - 23.50-40.22 mm^2/s, viscosity index - 133-150 units, flash point - 305-380^oC), lubrication properties (Pcr,H =700-900, Dw=0.55-0.70 mm) and have a satisfactory freezing temperature (minus 38^oC - minus 41^oC). Taking these into account, these esters can be recommended as components or additives to high-temperature resistant lubricants and to other oils that improve their operational properties [22-23, 25-26].
4. In order to improve the quality of mineral oils (T-30, T-46) and industrial synthetic oils (AB, DOS, PET, PAO) with the

addition of 5-20% of TMCP and TMCH esters, new oil compositions were developed and studied. As a result, a considerable increase in the operating properties of the mentioned oils was observed [8, 9, 15-20, 23, 24].

5. Compositions were prepared by adding 0.004% of some esters of cyclic neopolyols to diesel fuels and it was found that the thermal oxidation stability of diesel fuels increased, the amount of sediment decreased from 8.6 to 0.6-3.6 mg/100 ml, and the flash point increased from 76°C to 88°C. increased, and the freezing temperature decreased from minus 18.2°C to minus 25.1°C. For this reason, these esters are recommended as complex additives in diesel fuels [5, 10, 13].
6. Nitrogen-containing esters of cyclic neopolyols with caproic, 2-aminopropionic and 2-aminoisovaleric acids were synthesized and their properties were studied. Nitrogen esters, as well as other esters of cyclic neopolyols, for the first time, were tested as bactericidal-inhibitors against sulfate-reducing bacteria (SRB) (69-100% bactericidal properties), fungicidal properties were tested (in particular, the development of the Aspergillus niger fungus was completely stopped) and were found to have high performance. It is for this reason that they are superior to their counterparts, so they are recommended for use in industries. Also, some esters were tested as a corrosion inhibitor for conservation liquids and satisfactory results were obtained (11-62 days in 0.001% sulfuric acid solution, 15-70 days in seawater, and 85-202 days in "Г-4" hydrocamera).
7. Among the synthesized esters, systematic comparisons and studies were carried out between their structure and operational properties, and certain results and regularities were obtained, which, when the structure of esters is known, make predictions about their properties in advance, thereby making the conducted synthesis selective and economically efficient allows to determine [12].
8. Taking into account that the esters of TMTP have high

indicators, the optimal conditions for the process of obtaining the pentaester of TMCP with caproic acid were found (T=160°C, TMTP:CA mole ratio 1:6, t=5 hours, the amount of catalyst 1.5% by mass, yield 82.3%) and its use in industry was recommended [14].

The following scientific works on the dissertation materials have been published:

1. Mammadyarov, M.A., Gurbanov, H.N., Yusifova, L.M. Synthesis of complex oligoesters of 2,2,5,5-tetramethylolcyclopentanol and their study as high-temperature lubricants // Proceedings of the Azerbaijan National Academy of Sciences, -2019. №1, -p.48-52.
2. Mammadyarov, M.A., Gurbanov, H.N., Yusifova, L.M. Synthesis and study of 2,2,5,5-tetramethylolcyclopentanol esters as a base and component of lubricating oils // Oil and Gas Technologies, -2019. №3, -p. 22-26.
3. Mammadyarov, M.A., Gurbanov, H.N., Yusifova, L.M. Alkyl-substituted esters of cyclic polyols as a base and component of lubricating oils // World of Oil Products, -2019. №5, p. 27-31.
4. Mammadyarov, M.A., Gurbanov, H.N., Yusifova, L.M. Complex polyesters of cyclic polyols are promising lubricants // International scientific conference "Current problems of modern natural and economic sciences", dedicated to the 96th anniversary of the birth of national leader Heydar Aliyev, -Ganja: -May 3-4, -2019, -p. 272-275.
5. Gurbanov, H.N., Yusifova, L.M., Guluzade, F.A., Aliyeva, S.Q., Guliyeva, E.M. Symmetric and unsymmetric esters of cyclic polyols as multifunctional additives to diesel fuels // International Scientific Conference dedicated to the 90th anniversary of the Institute of Petrochemical Processes, - Baku: - October 2-4, - 2019, - p. 140.
6. Mammadyarov, M.A., Gurbanov, H.N., Yusifova, L.M.

Complex esters of 2,2,5,5-tetramethylolcyclopentanol as a basis and component of high-temperature lubricating oils // International scientific conference dedicated to the 90th anniversary of the Institute of Petroleum Processes, -Baku: - October 2-4, -2019, - p.141.

7. Mammadyarov, M.A., Gurbanov, H.N., Yusifova, L.M. Unsymmetrical esters of 2,2,6,6-tetramethylolcyclohexanol as promising lubricating oils //International scientific conference dedicated to the 90th anniversary of the Institute of Petrochemical Processes, -Baku: -October 2-4, 2019, pp.147.
8. Gurbanov, H.N., Mammadyarov, M.A., Yusifova, L.M. Creation and research of new oil compositions based on complex esters of cyclic polyatomic alcohols and industrial pentaerythritol esters // -Baku: Azerbaijan Oil Industry Journal, -2020. № 8, p. 53-57.
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