REPUBLIC OF AZERBAIJAN

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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

SYNTHESIS OF NEW ORGANIC SUBSTANCES BASED ON THIOCARBON, THIOCYANATE ACIDS AND GLYCEROL DERIVATIVES AND RESEARCH AS AN ADDITIVE TO LUBRICANTS

Speciality: 2306.01-Organic chemistry
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Field of Science: Chemistry

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The work was performed at the laboratory "Organic compounds with tribological properties" of the Institute of Chemistry of Additives named after Academician A. Guliyev, Ministry of Science and Education of the Republic of Azerbaijan.

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INTRODUCTION

The current state of the problem and its relevance. One of the important theoretical and practical issues of organic chemistry is the synthesis of new substances of practical importance. The creation of high-quality lubricating oils in the field of petrochemistry also requires the development of more effective additives and multifunctional additive compositions.

Currently, in connection with the development of the field of mechanical engineering, great attention is paid to the synthesis and research of high-quality additives used in various transmissions. The synthesis of new types of additives, the study of the relationship between their functional effect and chemical structure are of great theoretical and practical importance.

Research conducted at the Institute of Additive Chemistry over the past 50 years has unequivocally proven that sulfur and nitrogen-containing substances are multifunctional additives[1]. Analysis of these studies shows that the possibilities for the synthesis, research and application of new organic compounds based on thiocarbonates, especially xanthogenates, are still quite wide and promising. At the same time, it is very important that approved additives are obtained from easily accessible and inexpensive starting compounds. Therefore, the presented work is very relevant for both organic chemistry and additive chemistry in terms of creating new tribologically (wear, shear properties) active compounds.

By the way, one of the important requirements for the production of additives is the cheapness and accessibility of the raw material base for their production. The cheapness and accessibility of the raw material base has significantly increased the production of glycerin, a by-product of the biodiesel production process, in recent years

Therefore, the preparation of the products of the mutual reaction of xanthogenates with glycerin derivatives and their study as

[1] Naghiyeva, E. Motor oils and additives to them / E.Naghiyeva. -Baku: – 2022. – 38 p.

additives can be considered an interesting, promising, and scientifically and practically important dissertation topic.

The object and subject of the research. The object of the study was monochloroacetic acid, allyl alcohol, salts of xanthate and trithiocarbonic acids, as well as 1,3-dioxolanes, their hydroxy- and chloromethyl esters, salts of xanthogenic acids, as well as sodium di-, trisulfides. New compounds were synthesized on the basis of these reactive substrates and nucleophilic-electrophilic reagents.

By studying some tribological properties of the compounds obtained as the subject of the study, marine gas turbine oils and lubricant compositions were created on the basis of benzylidene bisxanthate, which stands out for its activity among these compounds.

The purpose and objectives of the research. The main goal is to improve the synthesis method of little-studied dithiols and study the obtained substances as additives, including the development of methods for obtaining representatives of benzylidene bisxanthogenates unknown in the literature and the creation of lubricant compositions using these compounds, as well as the synthesis of new xanthogenates, trithiocarbonates, sulfides containing sulfur and nitrogen, as well as containing appropriate functional groups, and the study of their tribological properties.

To achieve this goal, the following issues are envisaged in the dissertation work:

– Synthesis of halogen-containing substances of interest as synthons for obtaining new nitrogen and sulfur-containing compounds that are valuable additives for oils;

– Synthesis of di- and trisulfides as anti-wear additives;

– Study of tribological properties of synthesized substances.

In addition to determining the chemical structure of the synthesized new substances, it is intended to study their useful properties, in particular, the lubricating effect.

Investigation methods.

Modern ¬ IR-, NMR-spectroscopy methods were widely used in the studies performed for the thesis work, and the obtained results were compared and their structures were confirmed.

The main provisions submitted to the defense:

– developed methods of organic synthesis for obtaining new organic reagents from the interaction of monochloroacetic acid and allyl alcohol with allyl esters of monochloroacetic acid and salts of xanthate and trithiocarbonic acids;

– determined ways of obtaining 1,3-dioxolanes from the co-condensation reaction of glycerin and ketones, as well as xanthate- and chloromethyl esters of 1,3-dioxolanes and salts of xanthogenic acids and sodium di-trisulfides;

– studied the tribological properties of the obtained substances and their dependence on the chemical structure, and also created marine gas turbine oils based on benzylidene bisxanthate.

Scientific novelty of the investigation

New dithiols and benzylidenebisxanthogenates, which are interesting research objects for delicate organic synthesis, have been synthesized, which have been little studied so far. Numerous experiments were carried out to calculate the MRD of dithiols, and then an atomic refraction of sulfur unknown in the literature was found, which is 8.60 units.

As a result of the studies, it was determined that during the reaction of benzaldehyde with xanthogenates, hemiacetal is obtained as an intermediate product, and then benzylidenebisxanthogenates. Two alternative methods for obtaining bis(2,2-dimethyl-4yl-carboxymethyl) sulfides have been developed, the yields of substances have been significantly increased by using a phase transfer catalyst (tetrabutylammonium bromide-[(C4H9)4N]Br).

When studying the tribological properties of the obtained substances, it was found that their high efficiency depends on the length and composition of the radicals.

Using more effective benzylidenebisxanthogenates, oil compositions for marine gas turbine engines have been developed.

As a result, 29 new substances were synthesized and the synthesized new substances were reflected in 8 patents.

Theoretical and practical significance of research. As a definite contribution to organic chemistry, a method for the synthesis of dithiols, which has been little studied so far, has been improved,

and a new method for the preparation of benzylidene bisxanthogenates has also been developed.

High-quality anti-skid additives have been synthesized based on cheap and readily available glycerin, which is of interest as an additive.

Lubricant compositions have been created using benzylidene bisxanthogenates for marine gas turbine oil.

Approbation and application of the work.

The results of the dissertation work were presented and discussed at the following national and international conferences:

International Youth Forum/"International Processes of the World Science in the 21st Century", (Ganja, 2016), International Scientific Conference "Functional Monomers and Polymer Materials with Special Properties: Problems, Prospects and Practical Views" dedicated to the 55th anniversary of the establishment of Sumgayit State University, (Sumgayit State University 2017), "Actual Problems of Modern Natural and Economic Sciences. International Scientific Conference", (Ganja, 2018), XII International Scientific Conference "Actual Problems of Chemistry", (Baku, 2019), International Scientific and Practical Conference "Prospects of Innovative Development of Oil Refining and Petrochemistry" dedicated to the 110th anniversary of Academician V. Aliyev, (Baku, 2018), I Republican Student Scientific Conference on "Prospects in Chemistry and Chemical Engineering" dedicated to the 96th anniversary of the birth of the national leader Heydar Aliyev, (Baku, 2019), XIII International Scientific Conference of PhD students, masters and young researchers on "Actual Problems of Chemistry" dedicated to the 96th anniversary of the birth of the national leader Heydar Aliyev, (Baku, 2019), "The 1st International Conference on Air-Land-Sea Interaction", (Baku, 2019), VII International Scientific and Technical Conference "Alternative Sources of Raw Materials and Fuels", АИСТ-2019, (Minsk, 2019), "6Th International Symposium on Polymers and Advanced Materials, Batumi Shota Rustaveli State University, (Batumi, 2019), "2nd International Environmental Chemistry Congress (EnviroChem), (Antalya 2019), "Second International Scientific Conference of Young Scientists and

Specialists", (Baku, 2020), XXXIII International Scientific and Technical Conference, dedicated to the memory of the academician AN RB D.L. Rakhmankulova, г. Ufa, (Ufa, 2019), Republican scientific conference on "Modern problems of chemistry", (Sumgait, 2021), "3. International Baku Scientific Research Congress, Baku Eurasian University", (Baku, 2021), VIII International Scientific and Technical Conference "Alternative Sources of Raw Materials and Fuel", (AIST-2021) (MINSK, 2021), "II International Khazarian Scientific Research Congress", (Baku, 2021), "Dedicated to the 90th anniversary of the distinguished scientist Nadir Seyidov Republican scientific conference on "Catalysts, olefins and beeswax", (Baku, 2022), "AHI EVRAN 3rd International Scientific Research Congress", (Baku, 2023), Republican scientific conference "Modern approaches in chemistry and chemical technology" dedicated to the 80th anniversary of the Department of Petroleum Chemistry and Chemical Technology, (Baku, 2023), "9 Internationals to the 100th Anniversary of the Republic" Ankara scientific research congress", (Ankara, 2023)

Published scientific works. On the topic of the dissertation, 43 scientific papers were published, including 13 articles and 22 conference materials and theses, 8 patents were received.

Personal involvement of the author. The main ideas of the dissertation, including the synthesis of new sulfur-nitrogen-containing substances, conducting experiments to create oils based on them, making experimental samples, patenting the practical results obtained, as well as writing articles and conference abstracts, were implemented by the applicant.

The name of the institution where the dissertation work was performed. The presented dissertation work was completed in the laboratory "Organic Compounds with Tribological Properties" of the Institute of Chemistry of Additives named after Academician Ali Guliyev of the Academy of Sciences of the Republic of Azerbaijan.

The total volume of the dissertation with a sign indicating the volume of the structural sections of the dissertation separately. The dissertation consists of an introduction, 5 chapters, a conclusion, a bibliography of 220 sources, and appendices. The

dissertation consists of 166 pages, including 27 tables, 8 figures, and a 20-page appendix to the dissertation. In addition, the introduction consists of 10396 characters, Chapter I – 33360, Chapter II – 44393, Chapter III – 38673, Chapter IV – 30723 and the conclusion – 3026 characters, which in total is 160571 characters.

Introduction. The introduction explains and justifies the relevance, purpose, scientific novelty and practical significance of the work.

In the first chapter, The information is posted on the synthesis and study of derivatives of glycerol, xanthogen, dithiocarbamic acid, disulfides, their properties as additives and biologically active substances, as well as literary information on transmission oils.

In the second chapter, dithiols, benzylidenebisxanthates, reaction of chloroacetic acid with allyl ether with di- and trithiocarboxylic acids, reaction of glycerol with monochloroacetic acid, synthesis of diethyldithiocarbamic acid derivatives containing functional groups, synthesis of thiocyanate derivatives, synthesis of 1,3-dioxolanes containing a xanthogen fragment, xanthogenatomethyl The synthesis of 1,3-dioxolanes, the synthesis of sulfides consisting of 1,3-dioxolane fragments are discussed.

In the third chapter, Experiments on the synthesis of dithiols, benzylidenebisxanthates, allyl esters of sodium and trithiocarboxylic acids, glycerol, xanthogen, derivatives of dithiocarbamic acids, and disulfides are reflected.

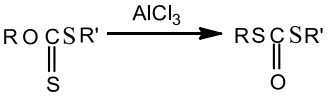
In the fourth chapter The article presents materials on the use of dithiols, benzylidene bisoxanthates, allyl esters of sodium and trithiocarboxylic acids, glycerol, xanthogen, derivatives of dithiocarbamic acid, and disulfides in various oils.

THE MAIN CONTENT OF THE WORK

Synthesis of sulfur- and nitrogen-containing substances.

Organic compounds in which the bond between carbon and sulfur is C−S are considered sulfur-containing compounds. This class of compounds also includes compounds in which the sulfur atom is bonded to the carbon atom via oxygen (O) or nitrogen (N). Since sulfur is an analogue of oxygen, there are a large number of sulfur-containing compounds. Compounds of this type include thioalcohols R−S−H, thioethers R−S−R, corresponding to simple ethers, etc. These compounds include, in particular, dithiols, xanthate, esters of trithiocarbonic and diethyldithiocarbamic acids, sulfides, and other compounds. It should be noted that it is sulfur-containing compounds that are found in high concentrations in petroleum products. We were interested in both the synthesis of new representatives of these substances and their study as additives. A number of dithiols have been synthesized, which are little known in the literature and are mainly obtained as a result of the reaction of xanthate conversion. The method of converting xanthate esters into dithiol carboxylic acid esters proposed by Japanese scientists[2] was improved by the works of the Institute of Additive Chemistry.

In continuation of the works , new representatives of the compounds presented in this dissertation were synthesized:



$R = C_4H_9, C_2H_5$; $R' - CH_3, C_2H_5$

Scheme 1. Conversion of xanthate esters into dithiolcarboxylic acid esters.

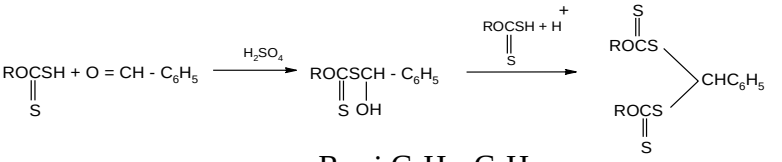
[2] Mustafaeva, G. Synthesis of some new representatives of dithiol derivatives and study of their tribological properties // 9th International Ankara Research Congress dedicated to the 100th anniversary of the Republic of Turkey.-Ankara:-2023,-.p.870

The conversion is carried out by adding $AlCl_3$ as a catalyst directly to the xanthate acid esters without using a solvent, while the reaction mixture is stirred at a temperature of 50-55°.

Synthesis of benzylidene bisxanthates.

In order to identify new highly effective additives based on benzylidene bisxanthate and xanthic acid, benzylidene bisxanthate was synthesized.

The reaction scheme of the obtained substances is as follows:



R = i C_3H_7; C_4H_9

Scheme 2. Synthesis of benzylidenebisxanthates.

The reaction of formaldehyde with xanthates is known; this reaction was carried out in 1964 by Tamm Byron and Tryon Sager[3]. However, the reaction of xanthates with other aldehydes has not been found in the literature. Perhaps due to the instability of xanthates, on the one hand, and their relatively high acidity, on the other, their reactions with benzaldehyde have not been studied. In general, the nature of the reaction leads to the formation of an intermediate hemiacetal.

Synthesis of xanthoacetic acid derivatives.

Among organic compounds, alkenes are chemically active due to the presence of a double bond in their molecule.

Such compounds have a paired electron, so they easily react with particles with unpaired electrons, including metals, which is important for additives.

Therefore, we are interested in substances with double bonds, including those with allyl fragments. Allyl bonds are approximately 15% weaker than bonds in a normal carbon center. and, therefore, are less active. Many substituents can be attached to the allyl group,

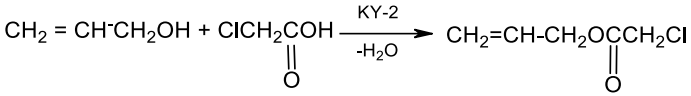
[3] Thumm, B. A. Reaction of formaldehyde with methyl and ethyl xanthates. Organic chemistry. // Tamm Byron Ashley, Tryon Sager. –1964. –V. 29. –№ 10. –P. 2999-3002.

forming stable compounds. Some of the most important and widely used substances are allyl alcohol and allyl chloride.

These compounds were used by the author to synthesize new allyl compounds.

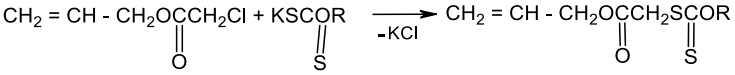
For the synthesis of allyl esters of xanthic acid, industrial compounds were used as starting materials - monochloroacetic acid and allyl alcohol.

Allyl ester of monochloroacetic acid was synthesized by a known method based on monochloroacetic acid and allyl alcohol:



Scheme 3. Synthesis of allyl ester of monochloroacetic acid.

Subsequently, a series of allyl esters of alkylxanthate acids were obtained by the interaction of allyl ester of monochloroacetic acid with the potassium salt of alkylxanthate:



R –= C_2H_5; i-C_3H_7;C_4H_9; i-C_4H_9; C_5H_{11}

Scheme 4. Preparation of allyl esters of alkylxanthogenic acid.

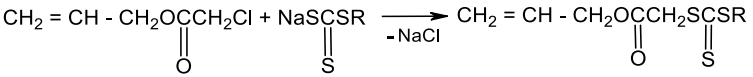
The initial reactions of synthesizing allyl esters of xanthic acid were carried out in a benzene solution, but the yield of the final products did not exceed 50%.

The synthesis of allyl esters of xanthic acid was carried out by mixing allyl ester of monochloroacetic acid with alkyl xanthate in dimethylformamide at a temperature of 80 ° C for 6 hours. Finally, the organic layer was washed with water, dried over Na2SO4 and evaporated in a vacuum. The obtained allyl esters of alkyl xanthate of acetic acid are light yellow liquids, soluble in organic solvents and lubricants, but insoluble in water.

Studies have shown that the solvent has a significant effect on the course of the reaction and the yield of the product.

One of the most important physical properties of solvents is their dielectric constant. It is known that the higher the dielectric constant, the easier it is for molecules to dissociate, and polar solvents facilitate the reaction, including heterolytic decomposition.

As a result of the interaction of allyl ether of monochloroacetic acid with sodium trithiocarbonate, allyl esters of trithiocarbonic acid were synthesized:



$R=C_4H_9; C_5H_{11}$

Scheme 5. Synthesis of allyl esters of trithiocarbonic acid.

The reactions were carried out in dimethylformamide at 80°C for 6 hours:

It should be noted that to study the yield of substances, the reactions were carried out in both protic and aprotic solvents and it was found that the highest yield of the reaction was obtained in dimethylformamide, a very effective protic solvent.

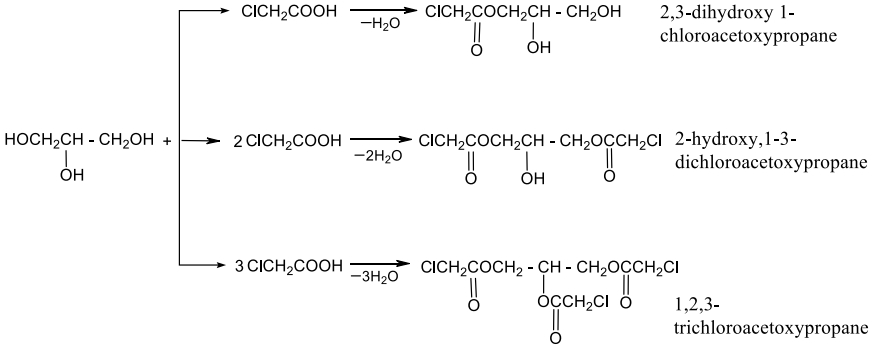
The structure of the synthesized esters was confirmed by the results of IR and NMR 1H spectral analysis, and the composition was confirmed by elemental determination and study of some physicochemical properties.

A series of acetoxypropanes obtained from glycerol.

It is very important to use available and inexpensive starting reagents for the synthesis of additives, in this regard, glycerol, which is a large by-product in the production of biodiesel fuel, was used as the starting reagent.

It is known that one of the important synthons for the synthesis of new compounds are chlorine-containing substances. Simple and inexpensive glycerol and monochloroacetic acid were used for the synthesis of chlorine-containing synthons. Monochloroacetic acid has a very high reactivity, so it is widely used in organic synthesis, including the synthesis of substances with anti-slip properties. As a result of the reactions, mono-, di- and triglycerides were obtained depending on the ratio of the monochloroacetic acid taken (1:1; 1:2; 1:3), the structure of the

substances is proven by NMR spectra.



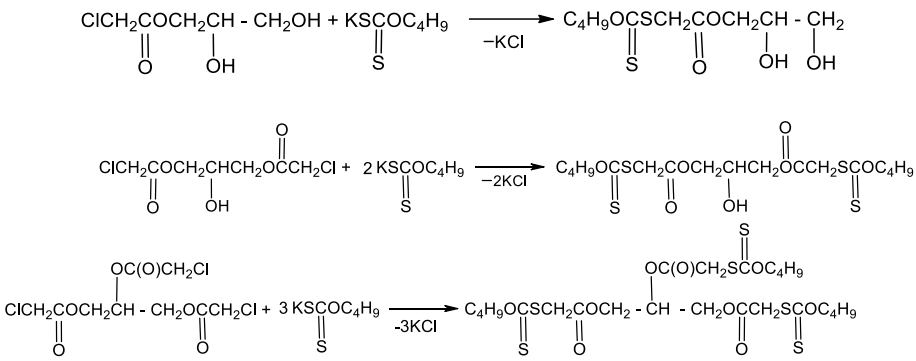
Scheme 6. Preparation of mono-, di- and triglycerides.

The physicochemical properties of the reaction products were studied.

In order to increase the yields of mono-, di- and trichloroacetylglycerides, numerous experiments were conducted using benzene and toluene as solvents, as well as various catalysts, such as benzene-, p-toluenesulfonic acids and acidic cation exchange resin KU-2.

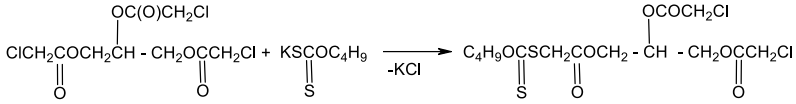
Benzene- and p-toluenesulfonic acids are catalysts of similar purpose, used in the etherification of carboxylic acids, re-etherification of complex esters. Cation exchanger KU-2 is a multifunctional strong acid resin used as a catalyst for organic chemical reactions. It is a high-molecular polymer compound (a copolymer of styrene and divinylbenzene).

The choice of catalyst in chemical processes is mainly determined empirically. For us, after numerous experiments, the catalyst of p-toluenesulfonic acid ($TsOH \cdot H_2O$) was given greater preference. Using the obtained mono-, di- and triacylglycerides and potassium butylxanthate, mono-, di- and tributylxanthogenatoacetyloxymethyl propanes were synthesized, and the structure of the substances was proven by NMR spectra, which is reflected in the patent**.**



Scheme 7. Synthesis of mono-, di- and tributylxanthogenatoacetyloxymethylpropanes.

These reactions are exothermic, carried out in acetone as a solvent at a temperature of 50°C for 6 hours, the yield of substances is 70-75%. It is known that substances containing both chlorine atoms and sulfur atoms have very high anti-slip properties as additives, so chlorine-containing substances were also of interest to us.



Scheme 8. Synthesis of new representatives of chlorine-containing substances.

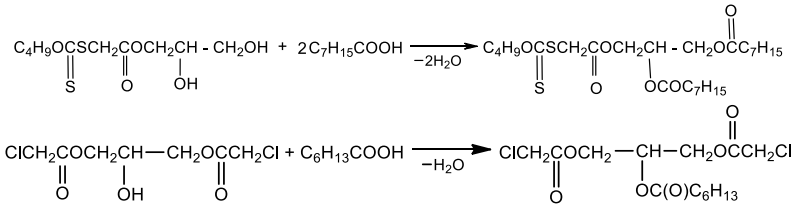
The reaction is carried out in acetone at a temperature of 50-55°C for 5 hours.

To synthesize this compound, several experimental variants were carried out using various solvents (acetone, isopropyl alcohol) and optimal conditions were determined. The reactions were carried out at 20°C, 50°C and 80°C, respectively, with an experiment duration of 3, 7 and 8 hours.

These studies were carried out in order to increase the yield of the final product, α-xanthogenatoacetyl-β–γ-di(chloroacetyl)triglyceride.

Recent studies in the field of lubricant additives have shown that substances with enanthate and caprylyl fragments significantly improve sliding properties. On this basis, using caprylic and enanthic

acids, both chlorine-containing and chlorine-free butylxanthogenatomethylacetyloxymethylpropanes were synthesized:

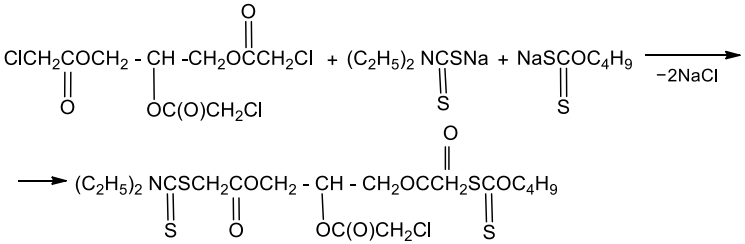


Scheme 9. Synthesis of butylxanthogenatomethylacetyloxymethylpropanes that do not contain chlorine.

These reactions were continued in a flask equipped with a Dean-Stark apparatus until the water completely disappeared. The substances were purified by liquid column chromatography. The structure of the compounds was proven by elemental analysis, determination of some physicochemical parameters and IR spectra and is reflected in the patent.

Synthesis of substances with mixed fragments.

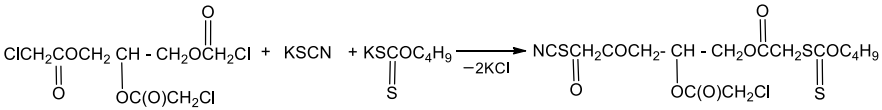
Based on 1,2,3-trichloroacetoxypropane, mixed ethers with fragments of S-butylxanthate and N-diethyldithiocarbamate were obtained, the structure of which was confirmed by the NMR spectrum and reflected in the patent.



Scheme 10. Preparation of mixed esters with fragments of S-butyl xanthate and N-diethyldithiocarbamate.

The reaction is exothermic, carried out in isopropyl alcohol, N-diethyldithiocarbamate and S-alkyl xanthate are introduced into the reaction zone as initial reagents at intervals of 3 hours.

At the same time, similar reactions of 1,2,3-trichloroacetoxypropane based on thiocyanate and butyl xanthate were carried out, and the structure was confirmed by NMR spectrum.



Scheme 11. Synthesis reactions of 1,2,3-trichloroacetoxypropane based on thiocyanate and butyl xanthate.

This reaction is also exothermic and occurs at a temperature of 50–55°C for 5–6 hours. The yield was 70–80%.

The structure of both synthesized compounds was proven by studying their elemental composition, physicochemical properties, IR and NMR spectroscopy and is reflected in the patent.

The physicochemical properties of mixed fragmented substances were also studied.

Synthesis of substances containing 1,3-dioxolane.

One of the interesting groups of synthesized compounds are 2,2-dimethyl-4-xanthogenatomethyloxymethyl-1,3-dioxolanes,

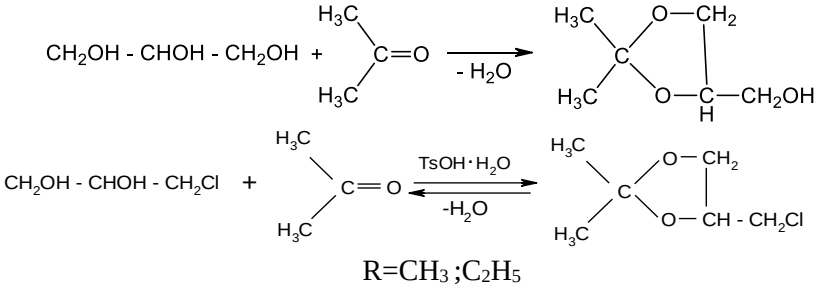
which are obtained as a result of the interaction of 2,2-dimethyl-4-hydroxymethyl-1,3-dioxolane and hydroxymethyl esters of alkylxanthic acids, which are obtained on the basis of glycerol.

At present, sufficient data have been accumulated to allow us to assert that the alkylation of alkali metal xanthates is the main method for synthesizing xanthate esters.This method has produced a wide range of ether derivatives of xanthate acids, distinguished by a variety of S-substituents. Xanthates can be obtained by the interaction of alkali metal xanthates with organohalogen compounds. However, as a result of scientific research, it was discovered that various esters of xanthogenic acids can be obtained using hydroxymethyl esters obtained by condensation of xanthogenic acids with formaldehyde as the starting reagent.

The heterolytic alcohol (1,3-dioxolane) obtained by acetylation of glycerol was subjected to xanthogenomethylation with

hydroxymethyl esters of alkylxanthogenic acids. The first information about hydroxymethyl esters of xanthogenic acids was expressed by Alles (1938), who reported the formation of unstable compounds during the interaction of cellulose xanthate with aldehydes.

It is known that 1,3-dioxolane derivatives are currently used in many areas of industry, but there is practically no information in the literature about their use as additives to lubricants. The starting materials, 2,2-dialkyl-4-hydroxy-, chloromethyl-1,3-dioxolanes and 2,2-dimethyl-4-chlorocarboxymethyl-1,3-dioxolane, were prepared according to the schemes given below:



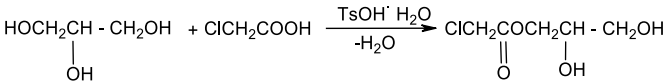
R=CH_3 ;C_2H_5

Scheme 12. Synthesis reactions of dioxalanes.

2,2-dimethyl-4-chloromethylcarboxymethyl-1,3-dioxolane as the starting material was obtained by two methods. The materials obtained by both methods are 2-step.

Method I.

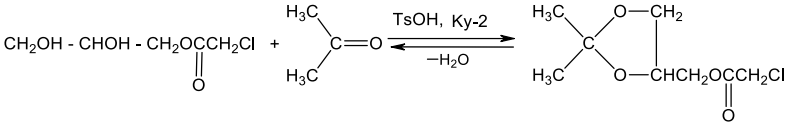
Chloromethylcarboxymethyl propanediol is obtained from the condensation reaction of glycerol and monochloroacetic acid:



Scheme 13. Preparation of chloromethylcarboxymethyl propanediol.

The reaction is carried out at 80°C in the presence of a TsOH•H_2O catalyst. Then, 2,2-dimethyl-4-

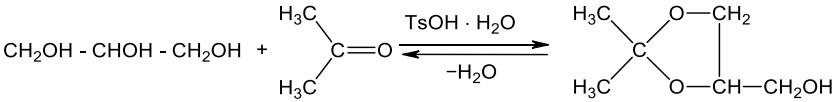
chloromethylcarboxymethyl-1,3-dioxolane is obtained by interaction with acetone:



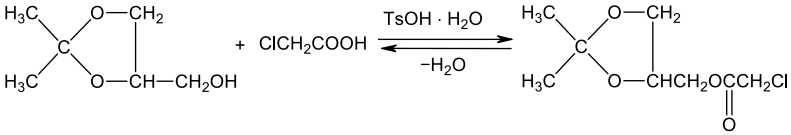
Scheme 14. Preparation of 2,2-dimethyl-4-chloromethylcarboxymethyl-1,3-dioxolane.

Method II.

First, 2,2-dimethyl-4-hydroxymethyl-1,3-dioxolane is obtained by condensation. Then, 2,2-dimethyl-4chloromethylcarboxymethyl-1,3-dioxolane is obtained by reaction with monochloroacetic acid:



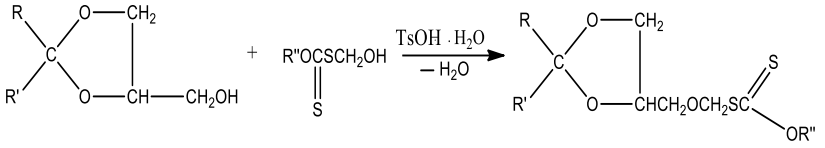
Scheme 15. Preparation of 2,2-dimethyl-4-hydroxymethyl-1,3-dioxolane.



Scheme 16. Preparation of 2,2-dimethyl-4chloromethylcarboxymethyl-1,3-dioxolane.

2,2-Dimethyl-4-hydroxymethyl-1,3-dioxolane and xanthogenatomethylated 1-3-dioxolanes were synthesized by condensation on the basis of hydroxymethyl esters of xanthogenates.

Xanthogenatomethylated 1,3-dioxolanes were synthesized according to the following scheme:

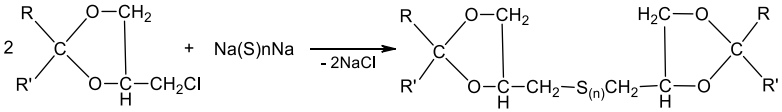


R=CH_3 , R/-CH_3, R//-C_2H_5, C_4H_9

Scheme 17. Synthesis of xanthogenatomethylated 1,3-dioxolanes.

2,2-Dialkyl-4-hydroxymethyl-1,3-dioxolane and hydroxymethyl ether of alkylxanthate were mixed first at room temperature for 10 min and then at 35˚C for 5 h. Benzene was used as the solvent and TsOH•H_2O as the catalyst.

Using 2,2-dimethyl-4-chloromethyl-1,3-dioxolane, mono-, di- and trisulfides were obtained:



R= CH_3, C_2H_5, R/= CH_3, C_2H_5 ; n=1, 2, 3

Scheme 18. Preparation of mono-, di- and trisulfides.

It should be noted that during the synthesis of di- and trisulfides containing a 1,3-dioxalane fragment, the initial sodium di- and trisulfides are not separated from the reaction mass in individual form, i.e., 2,2-dimethyl-4-chloromethyl-1,3-dioxolane is added directly to the reaction flask to the synthesized sodium di- and trisulfides.

Study of functional properties

Lubricity properties.

One of the most important issues of this work was the study of the lubricating properties of the synthesized substances in various oils. Various base oils were used in the studies:

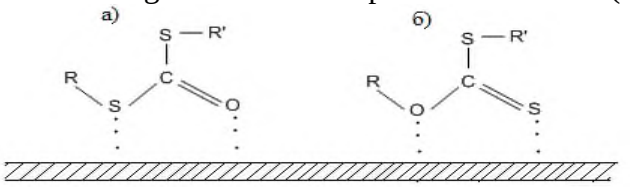
- АК-15, МС-20, Baku base oils;
- Т-1500, *И*-40А Russian base oil;
- Vaseline oil Belarusian base oil.

Comparative lubricating properties of derivatives of dithiol and

xanthate acids.

Since the structures of dithiols and xanthogenates are very similar, these substances were studied in highly refined vaseline oil to investigate the difference in their skimming and wear properties.

The comparative tribological properties of various esters of dithiols and xanthogen acids were studied. The mechanism of action of dithiols and xanthogenates can be explained as follows (Figure 1):



a) Dithiols; b) Xanthogenates

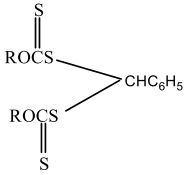
Figure 1. **Mechanism of action of dithiols and xanthogenates.**

According to the structure of the substances, both substances can be adsorbed on the metal surface only with two heteroatoms. Since the carbonyl group in dithiols is very polar, this group and the sulfur atom are adsorbed on the metal surface and form a protective layer. However, it is observed that the anti-skid properties of xanthates, although small, prevail, because thiol sulfur (C=S) is more active than other elements in the formation of a protective layer (this is known from the mechanisms of sulfur-containing substances given in the literature and from our own work experience).

Lubricating properties of benzylidenebisxanthates.

Table 1 shows the anti-skid properties of benzylidenebisxanthates, 2-butylene-1,4-bisbutylxanthate taken for comparison, and ethylene-bisisopropylxanthate (ЛЗ-23К), which is known to be anti-skid:

Table 1.
Etching and peeling properties of benzylidenebisanthogenates.



Compounds	Concentration of samples in oil, mmol	Anti-seize and anti-wear properties ГОСТ 9490-75			
		Anti-seize index İs, N	Critical load P_b, N	Welding load P_q, N	Wear diameter Dy, mm
АК-15	–	265	790	1590	0.72
R i-C_3H_7	20	612	1260	3550	0.68
C_4H_9	20	589	1235	3550	0.68
$(C_4H_9OCSCH_2CH_2)_2$ S	20	568	1098	3960	0.80*
$(i\text{-}C_3H_7OCSCH_2)_2$ S	20	520	1098	3096	0.75

*Researches were conducted at the Institute of Chemistry of Additives named after Academician A. Guliyev, Ministry of Science and Education of the Republic of Azerbaijan.

As can be seen from the table, benzylidene bisxanthogenates have anti-scratch properties, and according to some test indicators, they exceed those for comparison. This can be explained by the mechanism of action of benzylidene bisxanthogenates on the metal surface.

Due to the very low thermal stability of benzyl fragment substances, they quickly decompose under the influence of pressure and temperature, and the decomposition products react with the metal surface, resulting in the formation of a protective layer

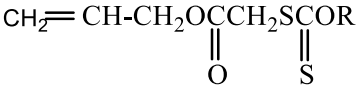
consisting mainly of sulfides

Lubricating properties of allyl xanthogenates.

To study the stripping properties of S-alkylxanthogenates, samples were prepared in petroleum jelly and MC-20 oil (Table 2):

Table 2

Lubricating properties of allyl xanthogenates in petroleum jelly.



Test samples	The concentration of additives in the oil		Anti-seize properties ГОСТ 9490-75		
	mmol	%	Anti-seize index İs, N	Critical load P_b, N	Welding load, P_q, N
Vazelin oil	–	–	10	549	1382
R					
C_2H_5	20	4.4	80	872	2604
i-C_3H_7	20	4.68	92	921	2764
C_4H_9	20	4.96	32	872	2323
i-C_4H_9	20	4.96	35	872	2340
C_5H_{11}	20	5.20	15	784	2067

The regularity obtained in Vaseline oil was confirmed by the results obtained in MC-20 mineral oil. Allyl esters of xanthogenates synthesized have high anti-scratching properties. Anti-scratch properties decrease as the radical of the substance increases.

Corrosion properties of allyl esters of alkylxanthogens were also studied.

The study of anti-corrosion properties of the samples prepared in TB-20 transmission oil (GOST 2917-76) at a concentration of 5% at 120°C for 3 hours shows that all the samples prepared with additives are not subject to corrosion, which is

confirmed by the color of the test plates taken as standards with copper plates. comparison proves.

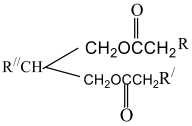
The determination of anticorrosion properties of allyl esters of alkylxanthogenate acids in TB-20 oil according to GOST 2917-76 shows that these compounds have effective anticorrosion properties, that is, the substances practically do not change the color of copper plates after tests, and their corrosion degree measured by honey corresponds to 1a. (the corrosion standard for transmission oils is 2c points).

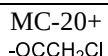
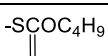
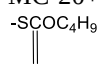
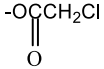
Lubricating properties of glycerol derivatives.

The anti-scratch and anti-corrosion properties of MC-20 oil are given in the table below:

Table 3

Lubrication properties of derivatives of acetoxypropanes obtained on the basis of glycerin in MC-20 oil



Compounds			The concentration of additives in the oil %	Lubricating properties according to ГОС9490-75		
				Anti-seize index İs, N	Critical load P_b, N	Welding load, P_q, N
MC-20				331	784	1097
R	$R^{/}$	$R^{//}$				
MC-20+ 			1	408	980	2500
–“–“–	–“–“–	–“–“–	3	620	1000	3550
–“–“–	–“–“–	–“–“–	5	686	980	3620
MC-20+ 			5	546	1260	2320

Continuation of table 3

1	2	3	4	5	6	7
MC-20+ 			5	627	1260	3550
			5	441	980	2763

As can be seen from the table, the substance containing 2 Cl(4) atoms surpasses the other substances in both the welding charge (Pq) and the shear index (Is). The percentage of chlorine atoms in this substance is 16.3%. The chlorine content in other substances is 6.47% (5) and 6.48% (6). The amount of chlorine in the substance has a significant effect on its anti-slip properties.

These substances are explained by the formation of a protective layer of bound sulfur sulfides and ferric chloride on the metal surface at high temperatures and pressures.

The anti-slip and anti-erosion properties of di- and tri(butylxanthogenatoacetyloxymethyl)propanes show that when comparing the slip indices (Is) and welding loads of additives taken in the same concentration, tri(butylxanthogenatoacyloxymethyl)propane surpasses these values. This can also be explained by the higher sulfur content. Based on both literature and our experience, it can be argued that the introduction of a xanthogen fragment into a molecule increases its tribological properties.

A substance with 3 xanthogen groups in a molecule is significantly superior to a substance with 2 xanthogen groups. The resulting di- and tri(butylxanthogenatoacetyloxymethyl)propanes are also more effective than the well-known anti-slip additive LZ-23K.

Organic thiocyanates are mainly used as insecticides, as well as for stabilizing lubricants and emulsifiers. We were interested in studying thiocyanates in other areas, especially as additives against

abrasion, corrosion, oxidation and wear.

The lubricating properties of thiocyanates in the composition of SN-1200 oil were studied. Thiocyanates are produced in a concentration of 3% in oil, which is due to the fact that thiocyanates have limited solubility in mineral oils, probably due to the presence of a nitrogen atom in the thiocyanate molecule, but despite this, they have high anticorrosive activity. anti-slip properties. Thiocyanates do not have antirot properties.

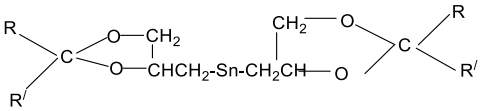
To improve the anticorrosive properties of thiocyanates, 1.8% of the DF-11 additive is introduced into the samples, due to which the diameter of the corrosion spot of the 2-hexylcarboxy-1,3-di(chloroacetoxy)propane sample decreases from 0.80 mm to 0.40 mm.

Lubricating properties of 1,3-dioxolanes.

Since chlorine-containing 1,3-dixolanes and sodium mono-, di- and trisulfides are relatively readily available starting compounds, the synthesis of mono-, di- and trisulfides on their basis was of interest. The interest in these compounds is caused, on the one hand, by the high anti-slip properties of sulfides, and on the other hand, by the fact that sulfides containing a 1,3-dioxolane fragment are new compounds. Therefore, it was very interesting to study them as an anti-slip additive. There is practically no information in the literature on the study of 1,3-dioxolanes as additives to lubricating oils. As can be seen from the results in the table, the results obtained with SN-1200 oil are higher.. A comparison of the anti-slip properties of 5% solutions shows the superiority of trisulfides. The high anti-shear properties of sulfides are due to the easy rupture of the -S-S- bond. Some researchers believe that the elongation of the sulfur atom chains in sulfides reduces the energy of the -S-S-S- bond, which leads to its easier rupture and faster formation of a protective layer, which increases anti-slip properties:

Table 4

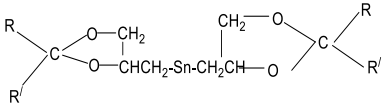
Tribological properties of 5% 1.3-Dioxolane sulfides in AK-15 oil



Test samples			Lubricating properties according to ГОС9490-75			
			Anti-seize index, $İ_s$, N	Critical load, P_b, N	Welding load P_q, N	Wear diameter, D_y,mm, 392N, 1hour
AK-15 oil			326	86	80	0.70
R	$R^/$	n	411	980	1586	0.60
CH_3	–	1				
CH_3	–	2	528	980	3980	0.80
CH_3	C_4H_9	2	450	980	3096	0.80
CH_3	–	3	714	1235	4410	0.85

Table 5

Tribological properties of 5% 1,3-Dioxolane sulfides in SN-1200 oil

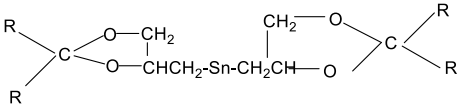


Test samples			Lubricating properties according to ГОСТ 9490-75			
			Anti-seize index, $İ_s$, N	Critical load, P_b, N	Welding load P_q, N	Wear diameter, D_y,mm, 392N, 1hour
SN-1200 oil			397	784	1235	0.80
R	R/	n	514	980	3920	0.73
CH_3	–	2				
CH_3	–	2	627	1235	4410	0.78

The lubricating properties of 1,3-di- and tri-1,3-dioxolane sulfides were studied in the composition of AK-15 and SN-1200 oils, the results are given in Table 6. As can be seen from the table, the results obtained with SN-1200 oil are higher. This is explained by the fact that SN-1200 oil, obtained from crude oil by vacuum distillation, has a higher quality than AK-15 oil:

Table 6

Composition of 1.3-Dioxolane sulfides in AK-15 oil with 1.5% ДФ-11 additive



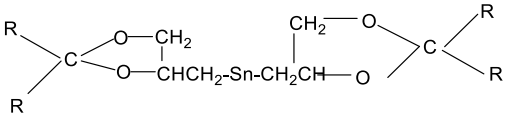
Test samples	Lubricating properties according to ГОСТ 9490-75			
	Anti-seize index, $İ_s$, N	Critical load, P_b, N	Welding load P_q, N	Wear diameter, D_y,mm, 392N, 1hour

Continuation of table 6

R	n				
CH_3	2	580	1235	3479	0.53
CH_3	3	–	–	–	0.80

Table 7

Corrosion properties of di- and trisulfides

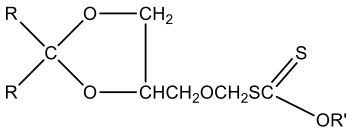


Compounds		Concen-tration %	Classifi-cation	Appoint-ment	description
R	n				
CH_3	2	5	2c	medium spotted	very colorful
CH_3+1,5%ДФ-11	2	5	1a	spotless	like a freshly polished strip
CH_3	3	5	4c	corrosion	dark black
CH_3+1,5%ДФ-11	3	5	4c	corrosion	dark black

Corrosivity of 1.3-dioxolane -di- and trisulfides for 3 hours was studied according to GOST 2917-76. As can be seen from the test results of Table No. 7, in order to improve the anti-corrosion effect of disulfides, it is possible to add DF-11 additive to the oil against oxidation and corrosion, but this situation is not observed with trisulfides.

Anti-scratch and anti-corrosion properties of xanthogenatomethylated 1,3-dioxolanes were studied in И-40а oil. Their research results and GOST norms of ИНСп-40, ИТД-32 oils are given in table 8 and 9.

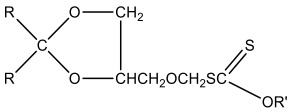
Table 8
Anti-seize and anti-corrosion properties of xanthogenatomethylated 1,3-dioxolanes in И-40А oil.



Test samples		The concentration of additives in the oil %	Concentration in DF-11, %	Lubricating properties according to ГОСТ 9490-75	
				Anti-seize index İs, N	Critical load, P_b, N
И-40А		–	–	303	588
R	R/				
CH_3	C_4H_9	1	–	343	686
—"	-	3	–	363	735
—"	-	5	–	430	833
C_2H_5	C_4H_9	1	–	391	617
"—	-	3	–	363	735
“—	-	5	–	426	784
CH_3	C_2H_5	1	–	346	686
—"—	-	3	–	363	784
CH_3	C_4H_9	5	1.5	454	980
ИНСп-40*			–	–	304
ИТД-32**			–	–	392

Note:* Characteristics of ИНСп-40oil
** Characteristics of ИТД-32 oil

Table 9
Anti-seize and anti-corrosion properties of xanthogenatomethylated 1,3-dioxolanes in И-40А oil.



Test samples		Lubricating properties according to ГОСТ 9490-75		Corrosion on copper plate in 3 hours GOST 2917-76, 3, point	Corrosion on DK-NAMI apparatus, at 1400C, for 20 hours, g/m2ГОСТ 11063-77
		Welding load P_c, N	Wear diameter, D_y,mm		
И-40А		980	0.84	2a	183
R	R/				
CH_3	C_4H_9	980	0.68	2a	57
—"	-	2450	0.72	2a	12
—"	-	3096	0.75	2a	
C_2H_5	C_4H_9	980	0.70	2a	68
"—	-	2852	0.72	2a	26
“—	-	3096	0.75	2a	9
CH_3	C_2H_5	1098	0.72	2a	60
— "— —	-	2852	0.72	2a	13
CH_3	C_4H_9	2450	0.45	2a	10
ИНСп-40*			1960		resistant
ИТД-32**			2450***	0.45	resistant

Note:* Characteristics of ИНСп-40oil
** Characteristics of ИТД-32 oil
*** Tests were conducted at AKI

As the length of the alkyl radical of the alkyloxy group increases, the anti-seize properties decrease relatively, which can probably be explained by the fact that shorter radical substances are more tightly adsorbed on the metal surface. At the same time, the anti-seize properties depend on the density of the substances, as the density increases, the anti-seize properties also increase. The study of eating properties shows that xanthogenatomethylated 1,3-dioxolanes do not have this property. The right way to improve edible properties is the use of edible additives.

Xanthogenatomethylated 1,3-dioxolanes as an additive in И-40 oil were compared with ИНСп-40 and ИТД-32 oils. According to the obtained main indicators, xanthogenatomethylated 1,3-dioxolanes can be used as high-quality components of ИНСп-40 and ИТД-32 oils.

Table 10

Properties and oil samples of marine gas turbine oils based on benzylidenebisanthogenates

Test samples	Lubricating properties according to ГОСТ 9490-75				Antioxidant properties Acidity, KOH/mg			sediment amount, %
	Anti-seize index, $İ_s$, N	Critical load P_b, N	Welding load P_q, N	Wear diameter, D_y,mm, 392N	before oxidation	after oxidation	change in acid numberΔ	
I. T-1500 (transformer oil)	245	490	1098	0.76	0.069	0.35	0.28	0.21
II. —"— + 1% (i-$C_3H_7O{\cdot}CS{\cdot}S)_2CHC_6H_5$	294	617	1568	0.65	0.12	0.48	0.36	0.20
III. —"— + 1% ($C_4H_9O{\cdot}CS{\cdot}S)_2CH\ C_6H_5$	290	588	1568	0.72	0.12	0.48	0.36	0.25
IV. —"— + 1% GQT oil (with sovol additive)	274	617	1303	0.75	0.04	0.65	0.61	0.20
V. —"— + 0.9%*, GQT oilТУ 38.40163	274	617	1382	0.50	0.35**	0.65	0.30	0.20
VI. (Standard for gas turbine oil ГОСТ 10289-79)	–	–	–	–	–	–	≤ 0.65	≤ 0.20

Marine gas turbine oils have been developed using synthesized benzylidene bisxanthate.

As can be seen from the table, lubricants developed on the basis of synthesized substances significantly improve both anti-scratch and anti-oxidation properties and fully meet the requirements of some operational indicators.

A comparison of the test results of prepared oils and GQT oil with technical conditions and with the norms for GQT oil (ГОСТ (10289-79)) is given in table 10.

From the comparison, it can be seen that the oils intended for the lubrication of ship-gas turbines differ from other oils with high stability against oxidation at high temperatures and operational properties.

Based on the results provided, a report was drawn up by the head of the department, Ahmadov T.Sh., and the engine tester, Yusifova R.N., as a result of the tests of the operational quality of the marine gas turbine oil (GTU), prepared on the basis of benzylidene bisxanthate obtained from the mutual synthesis of xanthates and benzylidene.

RESULTS

1. Benzylidenebisxanthogenates were synthesized on the basis of xanthogenic acids, and from their esters, dithiols, which have been little studied so far, were synthesized by transformation. It was found that benzylidenebisxanthogenates are synthesized by the formation of an intermediate hemiacetal, and dithiols, xanthogenic esters, since they are liquids, are synthesized in high yields without solvents and with AlCl3 taken in a catalytic amount [3; 40; 41; 42].

2. Allyl esters of xanthogenic acid were synthesized by the interaction of monochloroacetic acid and allyl alcohol. After conducting numerous experiments to increase the yield of the obtained substances, it was determined that carrying out the reaction in dimethylformamide, a polar aprotic solvent, leads to the highest yield of the intended substance [39; 43].

3. Using glycerol and monochloroacetic acid, which are affordable and inexpensive as starting materials, trichloroacetylglycerides were synthesized depending on the molar ratio of monochloroacetic acid. In order to obtain these substances in high yields, numerous experiments were conducted using benzene and toluene as solvents, as well as various catalysts, such as benzene-, p-toluenesulfonic acids, and acidic cation exchange resin KU-2, with the highest yield being obtained using toluene as the solvent and $TsOH•H_2O$ as the catalyst. In total, 29 new substances were synthesized using glycerol as the starting material. The composition and structure of all substances were confirmed by determining their physicochemical properties, IR, NMR spectral methods and were reflected in 8 patents. [4, 5, 7, 8, 9, 13, 18, 19, 20, 21, 22, 25, 26, 36, 37].

4. Anti-skid study of the synthesized substances shows that the various xanthogenates, trithiocarbonates, diethyldithiocarbonates, thiocyanates and mono-, di- and trisulfides obtained have high anti-skid properties, and at the same time it was determined that their tribological properties depend on the composition and structure of the substances [1, 2, 6, 10, 11, 15, 16, 17, 23, 27, 28, 29, 30, 31, 32, 33, 34, 35, 38].

5. Corrosion studies of synthesized di- and trisulfides

according to GOST showed that after adding 1.5% of the known oxidation and corrosion inhibitor DF-11 to the disulfide sample, lead plate in SN-1200 oil does not corrode, since without adding the DF-11 additive, the plate was classified as -2c, its appearance was multi-colored, after adding it, it was classified as -1a, its appearance was like a freshly polished plate [12, 14, 24].

6. A marine gas turbine oil was developed based on benzylidene bisxanthogenates, which have sufficiently high properties among the synthesized substances. This oil was tested together with two known marine gas turbine oils, and based on the results obtained, it can be stated that the proposed marine gas turbine oil is not inferior to the known oils and even surpasses them in some properties.

A report has been compiled as a result of testing the operational quality of marine gas turbine oil (GTU) prepared on the basis of benzylidene bisxanthate obtained from the mutual synthesis of xanthates and benzylidene.

THE MAIN RESULTS OF THE DISSERTATION WORK ARE REFLECTED IN THE FOLLOWING SCIENTIFIC WORKS.

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