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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

STUDY OF ORGANOSULFUR COMPOUNDS DERIVED ON THE BASİS OF CHLORACETAMIDE AS ADDITIVES FOR LUBRICATING OILS

Speciality: 2314.01– Pertochemistry

Field of science: Chemistry

Applicant**:** **Irada Balay Hajiyeva**

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The work was performed at the Institute of Chemistry of Additives after Academician Ali Guliyev and Azerbaijan University of Architecture and Construction of Ministry of Science and Education of the Republic of Azerbaijan.

Scientific supervisors: Doctor of Sciences in Chemistry, Professor **Nazim Pirmammad Mustafayev**

Doctor of Sciences in Chemistry, Professor **Ali Binnat Aliyev**

Official opponents: Doctor of Sciences in Chemistry, Associate Professor **Fatmakhanum Kheybar Aliyeva**

Doctor of Sciences in Chemistry, Professor **Qasim Zulfali Huseynov**

Doctor of Sciences in Chemistry, Professor **Akbar Ali Agayev**

Dissertation council ED 1.16 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at the Institute of Petrochemical Processes named after academician Y.H. Mammadaliyev of the Ministry of Science and Education

Chairman of the Dissertation Council: Doctor of Sciences in Chemistry, Academician **Vagif Maharram Abbasov**

Scientific Secretary of the Dissertation Council: Doctor of Sciences in Chemistry, Associate Professor **Lala Mahammad Afandiyeva**

Chairman of the Scientific seminar: Doctor of Sciences in Chemistry, Associate Professor **Fuzuli Akbar Nasirov**

GENERAL CHARACTERISTICS OF THE RESEARCH

The relevance of the topic and the degree of development. Technical progress is related to the tightening of the working parameters of machines and mechanisms, their ability to ensure reliable, long-term and economical work, which, in turn, leads to the aggravation of the working conditions of the used lubricants and the requirements for the quality of these oils causes an increase.[1]

The synthesis of new types of additives, the study of the dependence between their functional effect and chemical structure are of great theoretical and practical importance in creating more effective lubricants that meet new requirements.

From the analysis of the functional properties of sulfur and nitrogen compounds, which are more studied and used as additives, it appears that it is more typical for these compounds to improve the lubricity, anti-corrosion and anti-oxidation properties of oils. In addition, research conducted at the Institute of Chemistry of Additives named after academician A.M. Guliyev shows that it is possible to further increase the effect of sulfur compounds, especially thiocarbonate and thiophosphates, by introducing various functional groups into their molecule.[2]

Due to their difficult solubility in oils, amides, as well as sulfur compounds with an amide group in their molecule, have not been practically studied as additives. Therefore, purposeful synthesis of thiocarbonic acid derivatives with N-substituted amide group in the molecule, research as multifunctional additives, study of the dependence between their chemical structures and functional properties, creation of new types of additives and effective lubricant compositions with their application is an urgent research work.

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[1] Аббасов, В.М. Сборник научно-исследовательских работ 2016-2018 г.г. в области разработки смазочных масел. / В.М.Аббасов, С.Е.Абдуллаев, Ф.И.Самедова, Р.З.Гасанова, Н.Д.Набиева. Баку: - «Муаллим», -2018, - 142 с.

[2] Kerimov, A.Kh., Synthesis of some ethers of diethyldithiocarbamic acid and investigation of their influence on anti-wear and extreme pressure properties of lubricating oils / Kh.A.Mamedova, E.S.Dzhafarova, I.A.Khudiyeva // Journal of Chemical problems, Baku:- 2022, № 3(20), - pp223.

The object and subject of the research. The synthesis of new substances containing xanthogenate or tritiocarbonate groups and their study as a multifunctional additive. The dependence of newly synthesized substances on their chemical structure with lubricity, anti-corrosion, anti-oxidation and biocide effect has been studied.At the same time, new substances have also been studied in the composition of synthetic oil, which have a fairly lethal effect on the mushroom mixture. For this purpose, lubricant compositions were created using allylcarbamoylmethylhexyltrithiocarbonate (AKHTK), the lubricity properties of which are quite high in the synthesized substances, and the synthesis route is relatively simple. The new substance AKHTK additive not only has a fairly high lubricity effect, but also has an anti-corrosion effect.

The purpose and objectives of the work. The main goal is the synthesis of new substances that have xanthogenate or trithiocarbonate groups as well as amide groups in the molecule and are soluble in mineral oils; study of the obtained substances as multifunctional additives; it is the study of the dependence between their lubricity, corrosion and oxidation resistance, as well as their biocidal effect and their chemical structure.

In order to achieve this goal, the solution of the following issues is envisaged in the dissertation work:

- synthesis of chloracetamide derivatives that can provide oil solubility, in other words, synthesis of one or two NH_3 hydrogens;
- synthesis of substances with thiocarbonate and amide groups in the molecule by reacting N-mono- and N,N-disubstituted chloracetamides with salts of xanthogenate and trithiocarbonate acids;
- research of new synthesized compounds as additives against corrosion and oxidation, against corrosion and oxidation, as well as against bacteria and fungi, according to their oil solubility and chemical composition;
- investigating the possibilities of using the substances whose properties have been studied in accordance with the functional quality indicators in the composition of lubricant compositions.

Research methods: Modern İQ-, $^{1}H^{13}C$ NMR-, UB-

spectroscopy methods were widely used in the research carried out for the dissertation work, and the obtained results were compared and confirmed.

Main clauses defended:

N-substituted chloroacetamides, which play a coordinating role in obtaining sulfur derivatives of acetamide, were synthesized;

Substances with thiocarbonate and amide groups in the molecule were obtained from the interaction of chloracetamide and N-substituted chloracetamides with salts of xanthogenate and trithiocarbonate acids;

The dependence of the tribological properties of the obtained substances on their chemical structure was studied;

Among the representatives of synthesized substances with different content and structure, those with biocidal effect were studied;

Lubricant compositions have been created.

Scientific novelty of the research. A new class of compounds that are an interesting research object for additive chemistry - substances with thiocarbonate and amide groups in their molecule - have been synthesized and identified. The limits of the amidomethylation reaction have been further expanded by using S-carbamoylmethylxanthogenates as amides, as well as their N-methylol derivatives, while developing alternative methods of obtaining new substances.

The study of the obtained substances as additives in mineral and synthetic oils showed that the inclusion of N-substituted amide groups in the molecule of thiocarbonate esters, known for their anti-aging and anti-scratching effect, not only improves their lubricating properties, but also turns the substance into a multifunctional additive. The dependence of the additive against lubricity, corrosion and oxidation, as well as the biocidal properties on the chemical structure factors - the number and location of sulfur atoms in the molecule, the number and nature of the substituents on the nitrogen atom, the length of the alkyl radical, was determined, which allows further research to be carried out more appropriately.

Theoretical and practical significance of research. In addition to being multifunctional, additives that improve individual properties

more effectively and can be promising components for different lubricant compositions (S(N-acetylcarbamoyl)methyl-O-alkylxanthogenates for complex ester-type synthetic oils, N-benzylcarbamoylmethyl esters of trithiocarbonic acids for motor oils, S-(N-methylolcarbamoyl)methyl-O-alkylxanthogenates) for lubricant-coolant fluids.

The study of N-allylcarbamoylmethylhexyltrithiocarbonate with high lubricating properties and relatively simple synthesis in I-40A, AK-15, K-19 oils and the developed compounds of these oils showed that it is possible to create economically important added transmission (type $Та_{п}$-15B) and industrial oils (types ИТД-68, ИТД-100) based on Baku base oils.

Author's personal contribution. Setting and solving issues related to the main ideas in the dissertation, experimenting, analyzing and systematizing the obtained results, written articles were carried out with the applicant's participation, each stage related to the dissertation preparation was performed by the applicant personally.

Approbation and implementation of the work. The scientific results of the dissertation work were reported and discussed at the following national and international conferences: "Synthesis of xanthogenates with an amide group in the molecule and research as an additive to lubricants" scientific works of Azerbaijan University of Civil Engineers (Baku-1997), "S-methyl of xanthogenic acids Synthesis and research of its derivatives as additives to lubricants" Proceedings of the 5th republican scientific conference of graduate students and young researchers (Baku-1999), "Synthesis and study of amide group xanthogenates as biocide additives" Proceedings of the 6th republican scientific conference of graduate students and young researchers (Baku-2000), "Lubricating efficiency of new derivatives of thiougolic acids" IV Baku International Mamedaliyev Conference on Petroleum Chemistry (Baku-2000), "A new additive against corrosion for synthetic and mineral oils" Proceedings of the VII republican scientific conference of graduate students and young researchers (Baku-2001), "The use of thiocarbonate acids Lubrication of new derivatives properties" Proceedings of the II International Scientific Conference on Fine Organic Synthesis and

Catalysis (Baku-2002), "Research of New Derivatives of Thiocarbonate Acids as Additives to Synthetic Oils" Proceedings of the Eighth Republican Scientific Conference of Graduate Students and Young Researchers (Baku-2002), "Research of New Derivatives of Thiocarbonate in Lubricating Oils research as an additive" materials of the scientific conference of professors and teaching staff and graduate students of AzMIU (Baku-2007), "Research and reactions of aminolysis products of alkyl chloroacetates" materials of the republican scientific conference dedicated to the 100th anniversary of H.K. Efendiyev, corresponding member of ANAS (Baku-2007), "Synthesis of N-substituted carbamoylmethyl esters of xanthogenous acids and research as additives" Materials of the republican scientific conference dedicated to the 85th anniversary of the national leader H. Aliyev (Baku-2008), "Synthesis of N-substituted carbamoylmethyl esters of thiocarbonate acids and their lubricating properties" commemorating the 100th anniversary of academician A.M. Guliyev "Synthesis of 2,2-dimethyl-4-methyl-oxymethyl-1,3-dioxolane-4-carbomoylmethyl butylxantho-genate and research in synthetic and semi-synthetic oil as an anti-scratch additive" (Turkey-2020), "S-(N-acetylcarbamoyl) methyl-O-butylxanthogenate as a stripping additive for synthetic essential oils" (State Agency for Standardization, Metrology and Patent of the Republic of Azerbaijan I 2003 0106) was published.

The name of the institution where the dissertation work was performed. The submitted dissertation was performed at the Institute of Chemical Additives, named after academician A.M.Guliyev and at Azerbaijan University of Architechture and Construction. State registration No.0106 Az 0073, No.0101 Az 00109. This research is a part of the scientific research works carried out on the topic "Creation of additives and additive compositions that provide high lubricity and based on them, development of transmission and hydraulic oils that meet modern requirements."

Publishing. The results of the dissertation were reflected in 21 scientific works. Among them, 8 articles (2 of them are included in AAK of Russia and 1 into AAK of Kazakhstan), 12 theses were published, and 1 patent was obtained.

The scope and structure of the dissertation work. The dissertation consists of an introduction, 4 chapters (188177 symbols), conclusions, reference list with 175 sources of literature and appendices, 50% of which contains the literature of the last 10 years. The text is given in 178 pages, including 18 tables and 10 figures.

In the introduction (9999 symbols), the relevance of the problems facing us in the modern era, their solution, the purpose of the work, scientific innovation, and practical importance are shown and justified.

The first chapter (64433 symbols) characterizes the functional properties of thiocarbonic acid derivatives and amides as additives and characterizes the synthesis and study of compounds containing xanthogenate, trithiocarbonate and amide groups with different effects in the molecule in order to obtain multifunctional additives as a purposeful research direction.

The second chapter (26310 symbols) is devoted to the synthesis and study of the structures of key compounds that are expected to be useful as auxiliaries and additives.

The third chapter (50026 symbols) discusses the results of determining the lubricity, anti-corrosion and anti-oxidation and biocidal properties of the new substances, as well as evaluates the lubricant compositions developed using them.

The fourth chapter (37409 symbols) describes the preparation of starting substances for research, synthesis conditions of intermediate and main substances, research and testing methods.

MAIN CONTENT OF THE WORK

The aim of the dissertation work is the synthesis of new substances that have xanthogenate or trithiocarbonate groups as well as amide groups in the molecule and are soluble in mineral oils; study of the obtained substances as multifunctional additives; it consists of the study of the dependence between their lubricity, anti-corrosion, anti-oxidation and biocide effect and their chemical structure.

To achieve this goal, chloracetamide was selected as the central compound of synthetic research. Due to the reactions specific to the amino group of acetamide, various functional groups are added to the molecule, At the same time, by replacing one of the methyl hydrogens with a chlorine atom, it was intended to obtain the methyl esters of xanthogen and trithiocarbonic acids.

To obtain chloroacetamide: first, the complex ester of chloroacetic acid was synthesized, and then the obtained ester was subjected to ammonolysis by a known method.

$ClCH_2COOR + NH_3 \rightarrow ClCH_2CONH_2 + ROH$

Here, $R=CH_3, C_2H_5$

In order to perform the additive function, it is important that the substance is soluble in mineral oil. After the first studies, the necessity of synthesizing N-substituted derivatives of chloracetamide as intermediates appeared due to the difficult solubility of amide derivatives in oil without replacement of hydrogen atoms. In this regard, a number of new N-substituted precursors were obtained. When alkylchloroacetates react with allyl- and benzylamides at low temperatures, substitution occurs predominantly near the carbonyl group.

$$ClCH_2COOR + R'NH_2 \rightarrow \overset{\bullet}{ClCH_2CONHR'} \overset{HCl}{+} ROH + R'NHCH_2CONHR'$$

$R = CH_3, C_2H_5$; $R' = CH_2 = CHCH_2$, $C_6H_5CH_2$

It should be noted that both single and double amines interact more actively with methylchloroacetate than with ethylchloroacetate.

N,N-dialkylchloroacetamides were obtained by the known Schotten-Baumann method.

$$ClCH_2COCl + R_2NH \xrightarrow[-\ NaCl,\ -\ H_2O]{NaOH} ClCH_2CONR_2$$

$R=C_2H_5, C_4H_9$

In order to study the relationship between structure and functional activity, one of the amino hydrogens of chloracetamide was replaced by a second acyl group.

Acetic anhydride was used as an acylating reagent to obtain N-acetylchloroacetamide.

$\xrightarrow{H_2SO_4,\ 120^oC}$

$ClCH_2CONH_2 + (CH_3CO)_2O \rightarrow ClCH_2CONHCOCH_3 + CH_3COOH$

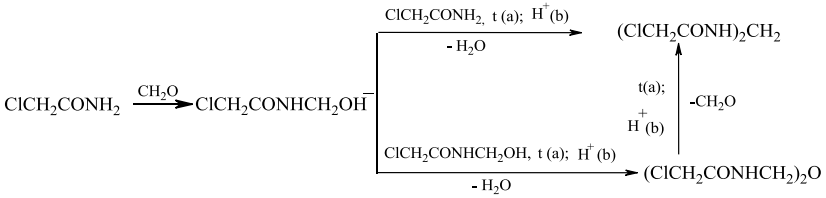
N-acetylchloroacetamide, soluble in water and ethyl alcohol, is a crystalline substance that melts at 110^0C.

Investigation of the condensation reactions of chloroacetamide in the presence of formaldehyde

Although N-Methylolchloroacetamide was one of the first methylolamides synthesized earlier, it was studied as an amidomethylating reagent for the amidomethylation of aromatic compounds by forming more carbon-carbon bonds. Compounds with simple ether and thioether fragments that N-Methylolchloroacetamide can form with alcohols and mercaptans are not known in the literature. Therefore, the synthetic research conducted on the basis of chloracetamide was of interest not only with its final products - sulfur derivatives, but also with its intermediate products - N-Alkoxymethyl and N-alkylthiomethyl derivatives.

Our experiments showed that the condensation of chloroacetamide with formaldehyde takes place in acidic, alkaline, and neutral environments, and depending on the selected conditions, three different products, either pure or mixed: N-methylolchloroacetamide, N,N'-oxydimethylenebischloroacetamide and N,N'- methylenebischloroacetamide is obtained. Factors affecting the composition of the reaction product are: the mole ratio of the components taken, whether or not a catalyst is used and its nature, temperature and reaction time.

The obtained results can be visually described by the following scheme:



The purpose of studying the interaction of chloracetamide with formaldehyde was to obtain a coupling intermediate to obtain oil-soluble sulfur derivatives of acetamide. Highly polar N-

methylolchloroacetamide is insoluble in oil. Substances obtained by replacing the chlorine atom in the molecule with derivatives of thiocarbonic acids were also less likely to be soluble in oil. But at the same time, replacement of hydroxyl hydrogen in the molecule with alkyl radicals gave grounds for such a possibility. Therefore, the study of the interaction of N-methylolchloroacetamide with the corresponding alcohols and mercaptans in order to obtain alkoxy- and alkylthiomethyl ethers was a logical continuation of the research within the framework of this dissertation.

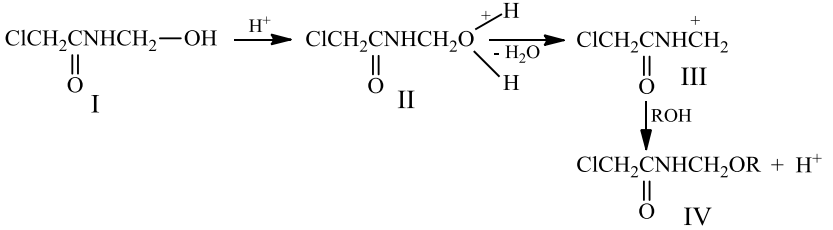
The synthesis scheme with the participation of solid sulfate or hydrochloric acid is as follows:

$$ClCH_2CONH_2 + CH_2O + ROH \xrightarrow[-H_2O]{H^+} ClCH_2CONHCH_2OR \quad \text{Method A}$$

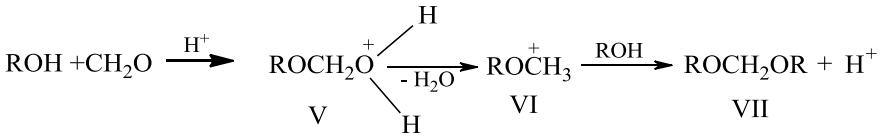
$$R=C_3H_7,\ C_4H_9,\ C_6H_{13}$$

Studies have shown that N-Alkoxymethylchloroacetamides are not the only products of the reaction of chloroacetamide, formaldehyde and alcohols in the presence of acid catalysts at 60-65°C. Thus, in addition to the main substance, the reaction products contain a certain amount of dialkyl methanes (up to 15%) and N-methylolchloroacetamide (10-16%).

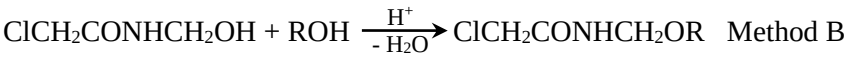
The synthesis scheme of the reaction is as follows:



The oxonium compound formed at the expense of a proton loses a water molecule, carbcation III, which, in turn, interacts with a nucleophilic alcohol molecule to form the final product - N-Alkoxymethylchloroacetamide. Under the conditions of the reaction, formaldehyde quite easily causes the formation of first carbocation VI, and then dialkyloxymethane, directly reacting with an alcohol molecule.

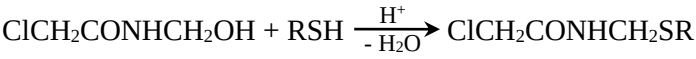


The study of the reaction of N-Methylolchloroacetamide with alcohols in an acidic medium showed that this method results in the production of N-Alkoxymethylchloroacetamides. In addition, although the method is two-stage, it is more convenient in terms of the difficulty of obtaining the dialkyloxymethanes and the slightly higher yield of the main product:



R=C_3H_7, C_4H_9, C_5H_{11}, i –C_5H_{11}, C_6H_{13}

N-alkylthiomethylchloroacetamides were synthesized using mercaptans instead of alcohol under similar conditions but in a different ratio of reagents:



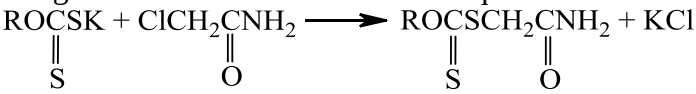
R=$(CH_3)_2CHCH_2$, C_6H_{13}

The structure of the obtained primary substances was proved by their determined physicochemical and IR, H^1 and ^{13}C NMR spectral characteristics.

The purpose of obtaining N-substituted chloracetamides was the synthesis of new substances with both thiocarbonate and amide groups in the molecule by reacting them with salts of thiocarbonic acids and their research as additives.

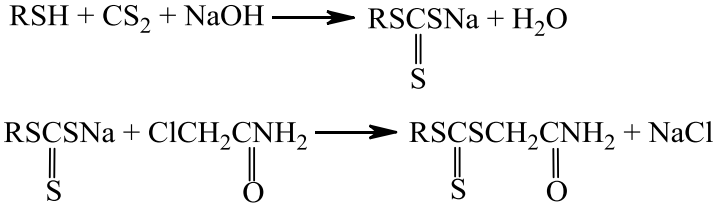
N-carbamoylmethylalkylxanthogenates and trithio-carbonates

The intended substances were obtained by reacting potassium alkylxanthogenates with chloracetamide in aqueous medium:



R=C_2H_5, $(CH_3)_2CH$, C_4H_9, C_6H_{13}, C_9H_{19}

Since alkyltrithiocarbonates are not commercially available as ready reagents, the purchase of N-carbamoylmethylalkyl-trithiocarbonates was performed in two stages. In the first stage, sodium alkyltrithiocarbonate was obtained by a known method using mercaptan, sodium hydroxide and carbon sulfide, and in the second stage, the latter was exposed to chloracetamide without leaving the medium from which it was obtained (dimethylformamide):



R= $(CH_3)_2CHCH_2$, C_5H_{11}

The yield and physicochemical parameters of carbamoylmethyl esters of synthesized alkylxanthogenate and trithiocarbonate acids are given in table 1.

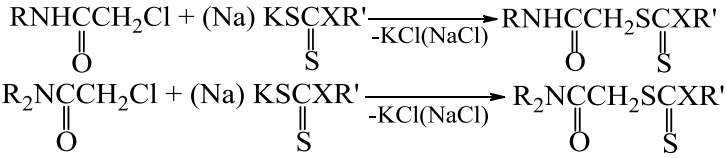
Table 1.

Yield and physicochemical parameters of carbamoylmethyl esters of alkylxanthogenic and trithiocarbonic acids

$RXCSCH_2CNH_2$ (S, O)

R	X	Yield ,%	Melting tempera-ture, °C	Found, %		Formula	Calculated, %	
				N	S		N	S
C_2H_5	O	94	113	7.53	35.38	$C_5H_9O_2NS_2$	7.81	35.77
$(CH_2)_3CH$	O	94	67	6.98	33.11	$C_6H_{11}O_2NS_2$	7.25	33.17
C_4H_9	O	96	94	6.49	30.62	$C_7H_{13}O_2NS_2$	6.76	30.93
C_6H_{13}	O	96	85	5.78	27.16	$C_9H_{17}O_2NS_2$	5.95	27.34
C_9H_{19}	O	96	80	4.92	22.87	$C_{12}H_{23}O_2NS_2$	5.05	23.11
$(CH_3)_2CHCH_2$	S	76	120	6.08	42.83	$C_7H_{13}ONS_3$	6.27	43.06
C_5H_{11}	S	80	102	5.72	40.25	$C_8H_{15}ONS_3$	5.90	40.51

N-mono- and N,N-disubstituted carbamoylmethyl esters of alkylxanthogenic and trithiocarbonic acids were obtained according to the following scheme:



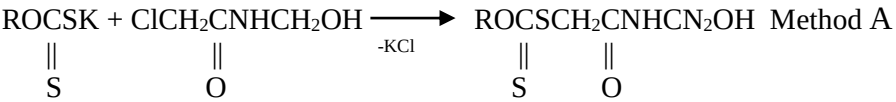
R= C_2H_5, X=O

Some of the synthesized substances are easily crystallized substances from ethyl and isopropyl alcohols.

Some N-allyl- and N,N-dialkyl derivatives are liquid, more soluble in mineral oils than crystalline substances.

S-(N-methylolcarbamoyl)methyl esters of alkylxanthogenic acids were obtained by two methods.

One of the methods is the reaction of potassium alkylxanthogenates with N-methylolchloroacetamide, corresponding to the classical synthesis option we used in the previous sections to obtain xanthogenic acid esters:

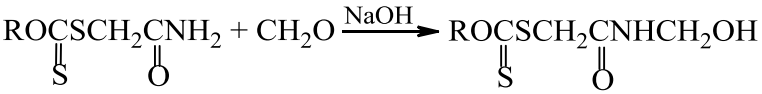


R= C_2H_5, $(CH_3)_2CH$, C_4H_9, C_6H_{13}, C_9H_{19}

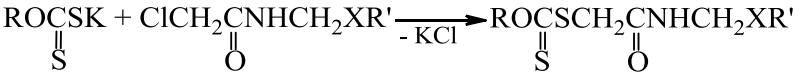
The reaction is carried out in an aqueous medium at 60-70°C in an equimolar ratio of components, and easily crystallized substances are obtained from ethyl and isopropyl alcohols with a high yield (≥ 90%).

An alternative method of obtaining O-Alkyl-S-(N-methylolcarbamoyl)methylxanthogenates is the reaction of these substances with S-carbamoylmethylalkylxanthogenates and formaldehyde:

Method B

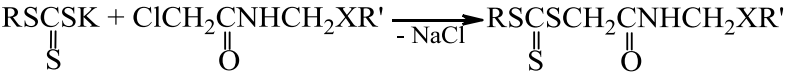


S-(N-Alkoxymethylcarbamoyl)- and -(N-alkylthiomethyl-carbamoyl)methyl esters of alkylxanthogenic and -trithiocarbonic acids were obtained from corresponding thiocarbonate salts and alkoxy(alkylthio)methylchloracetamides by substitution reactions:



R= C_2H_5, X=O, R' = C_4H_9

Method A

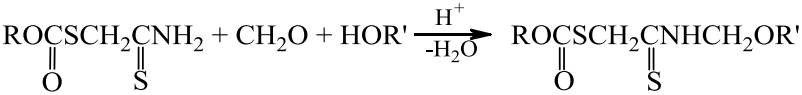


R= C_6H_{13}, X=S, R' = C_4H_9

Reactions of N-Alkoxymethylchloroacetamides with salts of thiocarbonic acids with thermal output were carried out in aqueous medium (with xanthogenates) and dimethylformamide (with trithiocarbonates), and were completed at 60-65°C for 2 hours.

One of the methods is condensing an acetamide derivative in which the chlorine atom in the molecule is replaced by a xanthogenate group with formaldehyde and alcohols in the presence of acid catalyst:

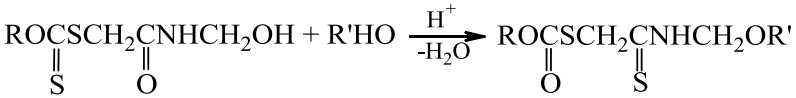
Method B



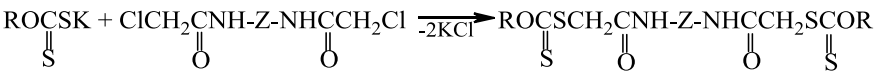
Reaction components 1:1. It is carried out at 60-70°C at a pH of 3-4 in a mol ratio of 2:2. As in the analogous reaction with chloroacetamide, in this reaction the alkoxymethylated amide is not the only product of the reaction.

In addition to the main substance, extraneous substances - O-alkyl-S-(N-methylolcarbamoyl)methylxanthogenate and dialkoxymethane are obtained. The presence of extraneous substances in the reaction mixture does not prevent the separation of S-(N-alkoxycarbamoyl)methylxanthogenates in pure form, because the components of the mixture, which differ in polarity, are very different from each other in their solubility properties in solvents.

Another alternative method developed to obtain O-Alkyl-S-(N-Alkoxymethylcarbamoyl)methylxanthogenates is the condensation of S-(N-Alkoxycarbamoyl)methylxanthogenates with alcohols in the presence of an acid catalyst:



N,N'-Oxydimethylene- and N,N'-methylenebisalkylxantho-genatoacetamides were obtained from the reaction of potassium alkylxanthogenates and the corresponding bischloroacetamides taken in a 2:1 mol ratio:

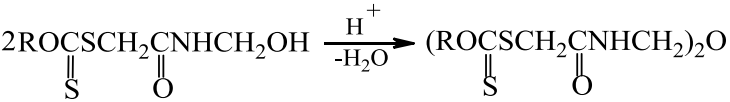


Here Z = CH_2OCH_2, R = C_2H_5, C_4H_9, C_6H_{13};

Z = CH_2, R = C_2H_5, C_4H_9, C_6H_{13}

Unlike the previous reactions, these reactions with potassium alkylxanthogenates were performed in dimethylformamide solution rather than in aqueous medium. Since methylene- and oxymethylenechloroacetamides, which have a high melting point, are also poorly soluble in water, their solubility in water during the reaction is very low, which leads to a very long reaction time. Since dimethylformamide is a suitable solvent for all components of the reaction, it was more convenient to use it.

One method of obtaining N,N'-Oxydimethylenebis-alkylxanthogenatoacetamides is self-condensation of O-alkyl-S-(N-methylolcarbamoyl)methylxanthogenates in a weakly acidic environment (pH=5-6):



It is possible to achieve a high yield of substances by removing the water obtained from the reaction from the environment (for example, through a Dean-Stark water trap).

N,N'-Methylenebisalkylxanthogenatoacetamides were obtained from the condensation of O-alkyl-S-carbomoylmethylxanthogenates and paraformaldehyde in the presence of an acid catalyst (solid chloride or sulfuric acid) taken in a mol ratio of 0,3:0,16:

The new synthesized compounds were investigated as additives against corrosion and oxidation, as well as against bacteria and fungi, in accordance with their oil solubility and chemical structure.Tests of substances soluble in mineral oils were carried out at the same molar concentration in I-40A industrial oil.

In some cases, T-46, M-15 oils were also used in order to obtain additional information. Since the N-allyl and N-benzylcarbamoylmethyl esters of thiocarbonic acids with small alkyl radical (K=C_2H_5; $(CH_3)_2CH$, C_4H_9) have very limited solubility in mineral oils, only the derivatives of hexylxanthogenic and hexyltrithiocarbonic acids with good solubility have been studied. The obtained results are presented in table 2.

Table 2

Lubricating properties of N-allyl- and N-benzylcarbamoylmethyl esters of xanthogenate and trithiocarbonic acids

No	The tested substance The chemical formula of the additive	The viscosity of the additive in the corresponding oil		Lubricating properties			
		T-46 %	İ-40A 10 mmol %	I_s, N	P_b, N	P_q, N	D_y, mm
1	2	3	4	5	6	7	8
1.	T-46 oil	-	-	265	490	1235	0,92
2.	İ-40A oil	-	-	262	490	1235	0,92
3.	$C_6H_{13}OCSCH_2CNHCH_2CH=CH_2$ (S, O)	1 3 -	- - 2,68	372 441 434	784 921 921	1960 2323 2323	0,50 0,52 0,52
4.	$C_6H_{13}OCSCH_2CNHCH_2C_6H_5$ (S, O)	1 3 -	- - 3,17	343 412 418	696 872 872	1842 2323 2323	0,50 0,55 0,56
5.	$C_6H_{13}SCSCH_2CNHCH_2CH=CH_2$ (S, O)	1 3 -	- - 2,83	412 490 485	784 980 980	2323 3283 3087	0,40 0,48 0,47
6.	$C_6H_{13}SCSCH_2CNHCH_2C_6H_5$ (S, O)	1 3 -	- - 3,30	400 470 478	696 872 921	2068 2930 2930	0,46 0,52 0,54
7.	$C_4H_9OCSC_8H_{17}$ (S)	1	-	323	617	1568	0,89
8.	$C_4H_9SCSC_9H_{19}$ (S)	1	-	364	917	1744	0,93

As can be seen from the table:

- at a concentration of 1%, these substances significantly improve all lubricity indicators of mineral oil;

– the tested substances exhibit superior lubricating properties compared to the thiocarbonates without an amide group in the molecule;

- when comparing allyl and benzyl derivatives of acids of the same name, it appears that allyl derivatives have a superior lubricating effect.

The results of the investigation of the lubricating properties of N,N-dialkylcarbamoylmethyl esters of xanthogenic and trithiocarbonic acids in the composition of I-40A and M-15 oils are indicated in table 3.

Table 3

Lubricating properties of N,N-dialkylcarbamoylmethyl esters of xanthogenic and trithiocarbonic acids

No.	The tested substance The chemical formula of the additive	The viscosity of the additive in the corresponding oil		Lubricating properties			
		İ-40A 10 mmol, %	M-15 20 mmol, %	I_s, N	P_b, N	P_q, N	D_y, mm
1.	İ-40A oil	-	-	262	490	1235	0,92
2.	M-15 oil	-	-	284	735	1470	0,68
3.	$C_2H_5OCSCH_2CN(C_4H_9)_2$ (S, O)	2.83 -	- 5,51	410 529	872 1166	2195 2764	0,66 0,71
4.	$C_4H_9OCSCH_2CN(C_2H_5)_2$ (S, O)	2.57 -	- 5.01	427 549	921 1166	2323 3087	0,63 0,68
5.	$C_4H_9SCSCH_2CN(C_2H_5)_2$ (S, O)	2.72	- 5.29	488 637	1039 1235	2930 3283	0,65 0,72
6.	$C_6H_{13}SCSCH_2CN(C_2H_5)_2$ (S, O)	2.96 -	- 5.79	472 617	980 1235	2764 3283	0,66 0,70
7.	$C_4H_9OCSC_8H_{17}$ (S)	-	4.99	470	980	2323	0.70
8.	$C_4H_9SCSC_9H_{19}$ (S)	-	5.53	549	1098	2930	0,73

From the tests carried out in I-40A oil, it can be seen that the N-mono- and N,N-disubstituted derivatives of xanthogenic and trithiocarbonic acids do not differ significantly from each other in

terms of stripping characteristics. However, the diameter of the corrosion mark (Dy) is higher in N,N-disubstituted derivatives.

As among the N-monosubstituted derivatives of xanthogenic and trithiocarbonic acids, among the N,N-disubstituted derivatives, trithiocarbonates are superior to xanthogenates in terms of lubricating properties. Synthesized derivatives of xanthogenic and trithiocarbonic acids are well soluble in mineral oils, so these substances are also prepared in high concentration (5-6%) M-15 oil. It can be seen from the table that doubling the thickness of the substances improves both their peeling indicators (Is, Pq) and the durability index of the protective layer (Pk).

Lubricating properties of N-Alkoxymethylcarbamoyl- and N-alkylthiomethylcarbamoylmethyl esters of xanthogenic and trithiocarbonic acids are given in table 4.

As can be seen from the table, Is and Pk indicators are not significantly different from those indicators of the given substances. The Dy indicators of N-Alkoxymethyl- and N,N-dialkylcarbamoylmethyl esters of the same named acids also have similar values. However, the welding load Pq index of N-Alkoxymethylcarbamoylmethyl esters is significantly smaller than other substances.

Table 4

Lubricating properties of N-Alkoxymethylcarbamoyl- and N-alkylthiomethylcarbamoylmethyl esters of xanthogenic and trithiocarbonic acids

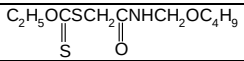
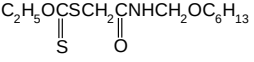
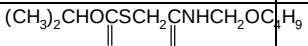
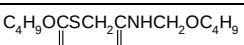
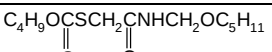
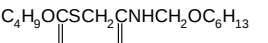
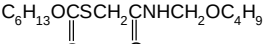
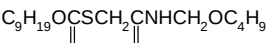
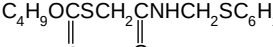
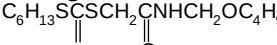
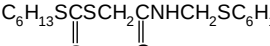
No.	The tested substance The chemical formula of the additive	Additive density in I-40A oil 10 mmol, in %	Lubricating properties			
			I_s, N	P_b, N	P_q, N	D_y, Mm
1.	İ-40A oil	-	262	490	1235	0,92
2.		2,58	451	1039	2195	0,66
3.		2,85	444	980	2195	0,64

Table 4 continuation

1	2	3	4	5	6	7
4.		2,72	445	980	2195	0,70
5.		2,85	440	980	2195	0,67
6.		2,98	425	921	2195	0,69
7.		3,11	400	872	2068	0,68
8.		3.11	398	872	2068	0,69
9.		3.51	377	784	1960	0,64
10.		3.26	485	1098	2450	0,66
11.		3.26	466	1098	2323	0,67
12.		3.68	524	1098	2764	0,66

There is another interesting correlation in Table 4. As the alkyl radical, either in the xanthogen fragment or in the N-alkoxymethyl group, increases, the value of the lubricity indicators of the substance decreases, and the change of the position of the alkyl radicals in the oxygen atoms does not affect the lubricity properties (7, 8). Lubricating properties increase when oxygen atoms are replaced by sulfur atoms in those substances (10 and 11). Replacing both oxygen atoms with sulfur atoms (supstance12) gives more effect.

Lubrication properties of mineral oil-insoluble N-acetylcarbamoylmethyl ethers from synthesized derivatives of xanthogenic acids were studied in the composition of pentaerythritol ether, which is the synthetic oil base of turbojet engines.

The study results are tabulated in comparison to the test results

of the synthetic oil itself, the tricresyl phosphate used in this oil, and the known additive proposed for use in table 5.

Table 5

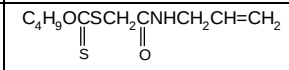
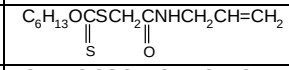
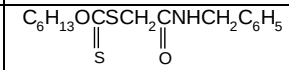
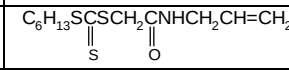
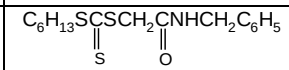
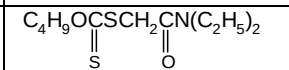
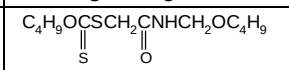
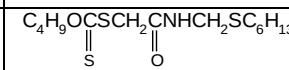
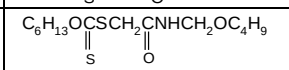
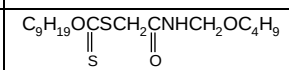
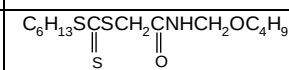
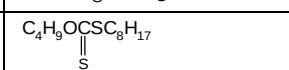
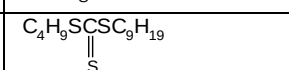
N-Acetylcarbamoylmethyl of xanthogenate acids lubricating properties of esters

No.	The tested substance The chemical formula of the additive	Density of substance of additive in synthetic oil (PEE), %	Lubricating properties				
			On OGD (ОПИ) GOST 9490-60	I_s, N	P_b, N	P_q, N	D_y, mm
1.	Synthetic ester oil (PEE)	-	-	314	617	1470	0,78
2.	$C_4H_9OCSCH_2CNHCCH_3$ (S, O, O)	1,53,0	51,6 64,0	510 627	1098 1382	1960 1960	0,72 0,76
3.	$C_6H_{13}OCSCH_2CNHCCH_3$ (S, O, O)	1,5	-	470	980	1842	0,77
4.	$C_9H_{19}OCSCH_2CNHCCH_3$ (S, O, O)	1,5	-	451	980	1744	0,80
5.	Tricresyl phosphate *	1,5 5,0	- 29,8	- -	588 696	- 1558	- -
6.	N-Allyl-5-hexyloxycarbonylmethyl-1,3-thiazolidin-4-one-2-thione *	1,5 5,0	47,3 48,5	- -	1098 1098	1960 1960	- -

As can be seen from the table, N-acetylcarbamoylxanthognaths taken at a concentration of 1.5% significantly improve other lubricity indicators without practically affecting the wear mark (Dy) indicators of synthetic oil. These substances can be promising components for lubricant compositions based on synthetic oils.

Table 6

Corrosion of various carbamoylmethyl esters of xanthogenate and trithiocarbonic acids and anti-oxidation properties

No.	The tested substance The chemical formula of the additive	Density of the addtive in T-46 oil, %	Corrosion of lead plate, q/m^2	Sedimentation, (within 30 hours),%
1.	T-46A oil	-	178	2,45
2.		1	5,3	2,58
3.		1	8,2	2,51
4.		1	12	0,50
5.		1	10	1,98
6.		1	9,1	0,45
7.		1	6,2	0,96
8.		1,5 3,0	42 7,8	2,33 -
9.		1	5,4	1,68
10.		1,5 3,0	27 5,7	2,45 -
11.		1,5 3,0	69 10	2,78 -
12.		1	8,8	2,27
13.		1,5	36	2,23
14.		1,5	21	1,96
15.	Additive DF-11 (ДФ–11)	1,5	6,4	3,57

A group of synthesized carbamoylmethyl esters of xanthogen and trithiocarbonic acids were studied as anti-corrosion and anti-oxidation additives based on the experience of using many sulfur compounds as anti-oxidation additives.

As can be seen from Table 6, when the corrosion rate of T-46 oil is 178 g/m^2, the corrosion rate of synthesized substances drops significantly. As a result of the tests carried out by the appropriate standard methods, it was determined that S-N-(substituted carbamoyl)methyl esters of xanthogenic and trithiocarbonic acids in mineral oil have not only lubricating properties, but also substances that improve anti-corrosion and anti-oxidation properties.

It is known that many representatives of sulfur- and nitrogen-organic compounds have a biocidal effect. Therefore, the effect of the synthesized substances against bacteria and fungi as an additive in M-10 oil was studied.

Table 7

Antimicrobial properties of N-Alkoxymethylcarbamoylmethylxnathogenates in M-10 oils

No.	The chemical formula of the substance (number)	Density of substance in M-10 oil, %	Diameter of the zone of destruction of microorganisms, cm	
			bacteria mixture	mushroom mixture
1.	M-10 base oil	-	+	+
2.	$C_4H_9OCSCH_2CNHCH_2OC_4H_9$ (S, O)	1.0	+	2.9-3.0
		0.5	+	1.3-1.4
		0.25	+	0.9-1.0
3.	$C_4H_9OCSCH_2CNHCH_2OCH_2CH=CH_2$ (S, O)	1.0	+	1.8-2.0
		0.5	+	1.1-1.2
		0.25	+	0.6-0.8
4.	$C_9H_{19}OCSCH_2CNHCH_2OC_4H_9$ (S, O)	1.0	+	+
5.	ω- Nitrostyrene (standard)	1.0	+	1.5

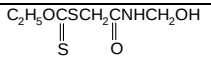
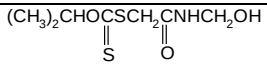
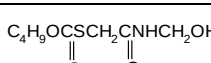
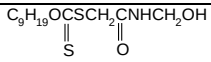
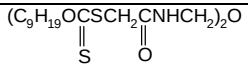
The obtained results show that N-Alkoxy- and N-allyloxymethylcarbamoyl-methylxanthogenates do not have bactericidal effect, but have strong fungicidal effect.

It is known that the presence of free water in the environment leads to intensive reproduction of microorganisms in the hydrocarbon environment. Since water is the main component in emulsion-type lubricating-cooling fluids (YSM), and the technical fluids prepared using them are affected by microorganisms. Therefore, the antimicrobial properties of the synthesized N-

methylolcarbamoylmethylxanthogenates were studied in the lubricating-cooling fluid composition (table 8).

Table 8

Lubricant-coolant of synthesized substances antimicrobial properties in liquid content

No.	The chemical formula of the substance (number)	Density of substance in YSM content, %	Diameter of the zone of destruction of microorganisms, cm		
			bacteria mixture	mushroom mixture	yeast fungus *Candida tropicalis*
1.	YSM	-	+++	+++	+++
2.		1.0	2.2-2.4	1.6-1.8	1.4-1.6
3.		1.0	2.5-2.5	2.2-2.4	1.4-1.6
4.		1.0	2.5-2.5	2.0-2.4	2.2-2.4
		0.5	1.6-1.8	1.6-1.8	1.8-2.0
		0.25	1.2-1.4	1.4-1.5	1.2-1.4
5.		1.0	1.6-1.8	2.6-2.8	2.4-2.6
6.		1.0	++	2.2-2.4	2.0-2.2

As can be seen from the results, N-methylolcarbamoylxanthogenates have a destructive effect on microorganisms, regardless of whether they are bacteria or fungi, of which the compounds with 3-4 carbon atoms in the alkyl radical of the xanthogenate fragment have a higher bactericidal effect, and the effect increases as the radical grows when the fungicidal effect occurs.

Among the investigated substances, N,N-oxydimethylenebisnonyl-xanthogenatoacetamide is also of interest. This substance retains both bactericidal and high fungicidal effect.

At the same time, one of the issues considered in the dissertation is the creation of lubricant compositions using new synthesized substances.

A relatively simple **allylcarbamoylmethylhexyltrithiocarbonate (ACNTC)** was used from a number of synthesized

substances with fairly high lubricity properties. It should be noted that currently the viscosity-temperature properties of domestic base oils do not meet modern requirements.

Table 9 shows the options of Tap-15V type transmission oil created on the basis of Baku base oils using ACNTC additive, which improves lubrication properties.

Table 9

Variants of TAп-15B type transmission oil created on the basis of Baku base oils

Indicators	Norm on ГОСТ 23652-79 for TAп-15B oil	Mixture of İ-40A and AK-15 oils (40:60)		Mixture of İ-40A and K-19 oils (50:50)		AK-15 + 0,5% Viscoplex 5-309 + 0,6% Viscoplex 2-670 + 0,003% ПМС-200A
		+ 0,5% Viscoplex 5-309 + 1,4% Viscoplex 2-670 + 0,003% ПМС-200A				
		A C N TC , %				
		2,5	3,0	3,5	3,0	3,0
Kinematic viscosity, mm^2/s At 40°C At 100°C	- 15±1	- -	154,6 14,66	- -	160,9 15,15	204,6 15,38
Viscosity index	-	-	93	-	94	68
Ignition temperature, °C	≥185	-	215	-	215	218
Freezing temperature, °C	≤ -20	-	-23	-	- 24	- 20
Corrosion of copper and steel plates, at 100°C, 3 hours	endures	-	endures	-	endures	endures
Lubrication properties in a four-wheeled machine:						
- creep index, N	≥ 490	470	510	539	519	524
- welding load, N	≥ 3283	3087	3283	3283	3283	3283
- indentation diameter, mm (392 N, 1 hour)	-	0,48	0,48	0,50	0,50	0,50

In order to determine the optimal amount of the additive to be added to the oil, its solutions in three different concentrations (2.5, 3.0 and 3.5%) were prepared in one of the selected compounds, and the lubrication properties were determined by adding anti-foam additive PMS-200A (0.003%) to the test samples.

The experiment results show that ACNTC additive provides a certain level of lubricity properties for Tap-15V oil at a concentration of 3%.

Therefore, when using K-19 oil, the concentration of ACNTC additive was 3%. It can be seen from the table that all three oil samples fully meet the standard's lubricity, non-corrosion of metal plates and other requirements.

The fact that ACNTC additive has a sufficiently high lubricating effect, as well as an anti-corrosion effect, aroused interest in its use in the development of ITD-68 and ITD-100 brand oils, which are used to lubricate the gear transmissions of medium and heavy-duty industrial equipment.

The comparison of the test results of the prepared oils with the norms of quality indicators determined by the technical conditions (TU) of that series of oils is given in table 10.

Table 10

İndicators	For ИТД -68 oil the norm on TY 38.1011337-90	İ-40A oil	For ИТД -100 oil the norm on TY 38.1011337-90	Mixture of İ-40A and AK-15 oils (60:40)
		0,2% Viscoplex 5-309+ 0,3% Viscoplex 2-670+ 2,5% ACHTC + 0,003% ПМС-200A		0,4% Viscoplex 5-309+ 0,6% Viscoplex 2-670+ 2,5% ACHTC + 0,003% ПМС-200A
Kinematic viscosity, mm^2/s, at 40°C	61,2 -74,8	69,6	90-110	98,8
Ignition temperature,	≥ 200	208	≥ 210	216
Freezing temperature,	≤ -18	-23	≤ -18	- 27
Corrosion of copper and steel plates, at 100°C, 3 hours	endures	endures	endures	endures

Table 10 continuation

1	2	3	4	5
Ash content, %	≤ 0,3	0,003	≤ 0,3	0,003
Lubrication properties in a four-wheeled machine:				
- creep index, N	≥ 392	451	≥ 441	480
- welding load, N	-	2607	-	2764
- indentation diameter, mm (196 N, 4 hours)	≤ 0,45	0,42	≤ 0,45	0,43

From the comparison, it can be seen that both developed variants of ITD series oil fully meet the requirements of viscosity-temperature, corrosion and lubricity properties. Thus, significant transmission (Tap-15V type) and industrial oils (ITD-68, ITD-100) can be obtained using ACNTC additive.

CONCLUSİONS

1. N-substituted chloroacetamides, which play a coordinating role in obtaining sulfur derivatives of acetamide, were synthesized by various known methods. Physico-chemical constants of 16 compounds obtained, twelve of which were new, were determined, and their structures were confirmed by İQ, ^{1}H və ^{13}C NMR spectral methods [1,11,12,18].

2. 56 new multifunctional compounds with thiocarbonate and amide groups in the molecule were synthesized from the interaction of chloroacetamide and N-substituted chloroacetamides with salts of xanthogenate and trithiocarbonic acids. By condensing S-carbamoylmethylxanthogenates with formaldehyde and alcohols, the limits of the reaction were further extended, and thus an alternative method of obtaining C-(N- alkoxymethylcarbamoyl) methylxanthogenates was developed. The results of the study of substances obtained by alternative methods, both by spectral and traditional methods of analysis, unambiguously confirm their chemical structure [2,14].

3. The study of the synthesized substances as anti-aging and anti-scratching additives in mineral oils shows that it is possible to further increase their inherent lubricating properties by introducing an N-substituted carbamoyl group into the molecule of esters of xanthogenate and tritocarbonic acids. The test results also show that the lubricating effect of the new substances depends on their chemical structure, as well as on the viscosity of the mineral oil and the viscosity of the additive. Thus, the successive increase in the number of sulfur atoms does not include the thiocarbonyl group of the sulfur atom, which replaces the oxygen atom in the molecule. Joining the carbamoyl group, shortening the alkyl radicals while keeping the substance soluble in oil, increases the lubricating properties of the additive; The N-substituent can be arranged in the following order according to the increasing effect (I_z) of protecting the rubbing surface from disintegration [3,10,13,17]:

$$ROCH_2 < C_6H_5CH_2 < CH_2=CHCH_2 < RSCH_2$$

4. N-acetylcarbamoyl methylalkylxanthogenates studied in pentaerythritol ether, which is the synthetic oil base of turbojet engines, have been found to have high lubricity properties. N-acetylcarbamoylmethylbutylxanthogenate, whose 3% solution is superior to 5% solutions of known additives, was determined to be a promising component for lubricant compositions based on synthetic oils [5,6,9].

5. Anti-corrosion and oxidation research of the synthesized substances shows that they have an anti-corrosion effect in the mineral oil content; a number of derivatives of butylxanthogenic acid (N-allyl, N,N-diethyl-n-hexylthiomethylcarbamoylmethyl ethers) practically prevent corrosion of lead plates at a concentration of 1%; Replacing the oxygen atom in the N-monosubstituted carbamoyl group with sulfur increases the anti-corrosion effect; Although most of the studied substances cannot prevent oil oxidation, N-benzylcarbamoylmethyl esters of thiocarbonate acids have a high antioxidant effect [7,8].

6. The study of the representatives of the synthesized substances with different composition and structure, in which they

are dissolved in the respective oils and liquids, shows that many of them have a strong biocidal effect. Most of the tested substances have fungicidal effect, and some have bactericidal effect at the same time. The investigation of the relationship between the chemical structure of the substances and their biocidal effect shows that what type of effect they have (fungicidal or both fungicidal and bactericidal) and the degree of effectiveness depends mainly on N-substituents, and the length of the alkyl radical in the xanthogenate fragment affects the degree of effectiveness of the substance [4 ,15].

7. The study of N-allylcarbamoylmethyl hexyltrithiocarbonate, which has rather high lubricating properties among the synthesized substances, and the synthesis method is relatively simple, in the composition of Baku base oils showed that the additive has lubricating properties that can ensure the normal operation of gears. By using these additives, it has been shown to be useful in the production of agriculturally important transmission (type ТАп-15В) and industrial oils (ИТД-68, ИТД-100). Tests conducted with appropriate methods on the mineral oil content of S-(N-substituted carbamoyl) methyl esters of xanthogenate and trithiocarbonic acids are substances with a multifunctional chemical structure that improves not only the lubricating properties, but also the properties against corrosion and oxidation [16,19,20,21].

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Address: 30, Khojali avenue, Baku, AZ1025, e-mail: nkpi@ nkpi.az

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