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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

SYNTHESIS AND STUDY OF FUNCTIONAL PROPERTIES OF NEW ORGANIC COMPOUNDS CONTAINING RHODANIDE GROUP

Speciality: 2314.01 - Petroleum chemistry

Field of Science: Chemistry

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INTRODUCTION

The relevance of the topic and the degree of development. It is well established that lubricants and other petroleum-based products, regardless of climatic conditions, are exposed to various external influences during long-term storage, transportation, and operational use. These factors lead to the formation of acidic by-products, which adversely affect the initial physicochemical and performance properties of petroleum products. Such substances may also interact with the metallic surfaces of machinery and mechanisms, accelerating corrosion and wear. In engine systems, they contribute to the formation of resinous substances and deposits in motor oils, leading to carbon buildup on pistons and injector nozzles. These undesirable processes ultimately disrupt the normal functioning of engines and significantly reduce their service life.[1]

One of the most effective and economically viable methods for protecting petroleum and petroleum-derived products is the incorporation of antioxidant additives.

This fundamental and innovative research addresses critical issues such as oxidation, microdamage, and pathological processes in living organisms, which hinder the advancement of technology, medicine, and related fields. The risk of such degradation processes is particularly high in environments where living microorganisms are present. In this context, the synthesis of novel compounds with combined antioxidant and physiological activity has become increasingly relevant. Therefore, expanding scientific research into the development and application of refined methods for organic synthesis is of particular importance. From this perspective, the synthesis of new derivatives of rhodanines and the investigation of the relationship between their molecular structure and biological activity form the core of the presented research. These studies hold substantial theoretical and practical significance in the field of

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[1] Farzaliev, V.M. Inhibited oxidation of hydrocarbons in the presence of nitrogen, phosporus, selenium, sulfur-containing heterocyclic compounds / V.Farzaliyev, I.Rzayeva, A.Sujayev at all // Iranian Journal of Chemistry and Chemical Engineering, – 2023. Vol. 42, Issue 4, – p. 1154-1163.

modern organic chemistry and its applications.

Analysis indicates that addressing challenges such as environmental safety and the protection of living organisms' health requires a multidisciplinary approach. In this context, while the oil industry demands high-quality, novel antioxidants, the pharmaceutical sector simultaneously requires antimicrobial agents, therapeutic compounds, and biological additives. Therefore, it is considered essential to expand research in the synthesis and application of next-generation antioxidants and antimicrobial agents with multifunctional properties relevant to various complex domains.

The object and subject of the research. The object of the research includes rhodanides, alkyl- and arylbromides, chloramine-B, aziridines, salicylaldehyde, and other related compounds. Utilizing these reactive synthons, a series of multifunctional compounds were synthesized through refined organic synthesis methods and supported by computer-assisted quantum chemical calculations. These novel compounds exhibit both antioxidant and biocidal additive properties capable of preventing hydrocarbon oxidation and microbiological degradation, as well as physiological activity against certain pathological processes in living organisms.

The subject of the research encompasses the study of these multifunctional compounds as antioxidants, antiwear and antifriction additives for lubricants, and combined-action biocidal and antioxidant additives for lubricant-cooling fluids (LCFs). Additionally, the study investigates the structure–activity relationships (SAR) governing their enzyme/isoenzyme inhibitory effects on pathological processes in biological systems, exploring how these effects evolve from simple to complex depending on molecular structure.

The purpose and objectives of the research. The main purpose of the study is the targeted synthesis of alkyl(aryl)rhodanines and novel heterocyclic derivatives containing the rhodanine (thiocyanate) group, which inhibit oxidation—the key operational degradation process in lubricants. The research also aims to identify the potential applications of these compounds as multifunctional antioxidant and biocidal additives in lubricants and LCFs, as well as investigate their potential inhibitory effects against enzymes/isoenzymes involved in certain pathological states, using molecular docking methods.

The following research objectives were pursued to achieve these goals:

- Development of a targeted synthesis method for alkyl- and arylrhodanines using a phase-transfer catalyst;
- Synthesis of novel organic compounds containing the rhodanine group;
- One-step three-component synthesis of hydroxybenzamides based on rhodanines;
- Theoretical and experimental study of the reaction mechanisms of certain rhodanine-containing heterocyclic compounds using Density Functional Theory (DFT);
- Investigation of hydroxybenzamides and heterocyclic compounds derived from rhodanines as antioxidant additives capable of inhibiting hydrocarbon oxidation;
- Evaluation of N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanato)-4,6-dimethyl]piperidine as a multifunctional additive for synthetic lubricants;
- Determination of the antimicrobial properties of alkyl- and arylrhodanines in LCFs;
- Study of hydroxybenzamide derivatives of rhodanines as biocidal additives preventing microbiological degradation in LCFs;
- Synthesis and investigation of 1-(2-hydroxyphenyl)-2-((4-thiocyanatophenyl)amino)ethanone as a polyethylene stabilizer;
- Investigation of the biological activity of selected thiocyanate-containing heterocyclic compounds.

Investigation methods. The conducted research employed refined organic synthesis techniques alongside modern computational modeling tools. One-step two- and three-component reactions were performed using appropriate active synthons. Structural elucidation of the synthesized compounds was carried out through advanced physicochemical analysis methods, including IR spectroscopy, elemental analysis, ^{1}H and ^{13}C NMR spectroscopy. Thin-layer chromatography (TLC) was used for kinetic monitoring of the reactions. All computational chemistry calculations were conducted using the Gaussian 16 software package.

In the study of functional properties, the antioxidant performance of the synthesized compounds was investigated using model hydrocarbon oxidation reactions, zone diffusion methods, and thermoanalytical techniques. The antioxidant activity was assessed via oxidation reactions in the presence of aluminum, steel, and copper plates, while antiwear properties were evaluated using a "Four-Ball Friction Tester." All materials and methodologies were appropriately aligned with the aims, objectives, and outcomes of the dissertation.

The main provisions submitted to the defense:

- Novel synthesis protocols for multifunctional antioxidants with enhanced performance characteristics have been developed to improve the operational properties of lubricants.
- New rhodanine derivatives containing specific functional groups have been synthesized, and their functional properties have been determined. The structure–property relationships and, in some cases, the theoretical basis of their functional mechanisms have been established.
- A multifunctional additive for synthetic lubricants used in gas turbine engines in aviation has been synthesized and shown to improve lubricant performance.
- A novel multidisciplinary research direction has been identified in bioorganic synthesis by studying the physiological activity of newly synthesized heterocyclic thiocyanate-containing compounds intended for use as additives.

Scientific novelty of the investigation:

- For the first time, targeted synthesis methods for alkyl, aryl, and new heterocyclic rhodanine derivatives with combined antioxidant properties were developed via phase-transfer catalysis, achieving yields of up to 92%.
- Density Functional Theory (DFT) calculations revealed the probable mechanisms of heterocyclic compound formation containing rhodanine groups, and theoretical predictions were validated by experimental data.
- A novel, efficient, catalyst-free one-pot three-component synthesis

method was developed for hydroxybenzamides bearing hydroxyl groups, structurally distinct from known analogs.
- Rhodanines and their transformation products, hydroxybenzamides, were found to possess both bactericidal and fungicidal activity when used as biocidal additives in emulsion-based lubricant-cooling fluids (LCFs), effectively preventing microbiological degradation.
- A new compound – 1-(2-hydroxyphenyl)-2-[(4-thiocyanatophenyl)amino]ethenone – was synthesized from the reaction of 3-(dimethylamino)-1-(2-hydroxyphenyl)propan-1-one hydrochloride with 4-thiocyanatoaniline and proposed as a stabilizer for polyethylene. Its high photostabilizing efficiency was attributed to strong intramolecular hydrogen bonding (OH···O=C) between the phenolic hydroxyl and the carbonyl oxygen, forming a chelate-type interaction.
- Molecular docking studies of the synthesized compounds and their derivatives revealed inhibitory activity against carbonic anhydrase isoenzymes (CA I, II), acetylcholinesterase (AChE), and α-glycosidase (α-Gly). Some newly identified inhibitors exhibited potential to dilate retinal capillaries and prevent microvascular blockages, suggesting therapeutic relevance to vascular disorders.

Theoretical and practical significance of research. The research established structure–property relationships for rhodanine derivatives and elucidated the mechanisms underlying their functional behavior. The antioxidants derived from rhodanines demonstrated synergistic effects when used in additive packages, resulting in at least a twofold improvement in lubricant performance compared to conventional antioxidants. This provided the scientific basis for the development of novel multifunctional antioxidant additives essential for enhancing the oxidative stability of lubricants.
The compound N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanato)-4,6-dimethyl]piperidine, containing a rhodanine moiety, was thoroughly evaluated as a multifunctional additive in synthetic lubricants for gas turbine engines. Testing revealed that adding this compound significantly improves lubricant quality, extends service life, and offers notable economic advantages. As a result, it is recommended for use as an antioxidant and antiwear

additive in high-performance synthetic lubricants. The applied scientific findings from this study were recognized and protected by the Intellectual Property Agency of the Republic of Azerbaijan with Patent No. İ20220100.

Approbation and application of the work. The key outcomes of the research were presented at the following national and international conferences and symposia:

II International scientific conference of young researchers dedicated to the 95th anniversary of National leader Heydar Aliyev (Baku, 2018); International scientific conference on “Current issues of modern natural and economic sciences” (Ganja, 2018); IV International scientific conference of young researchers dedicated to the 97th anniversary of Heydar Aliyev (Baku, 2020); XIV International scientific conference “Current problems of chemistry” (Baku, 2021); 1st International congress on natural sciences (ICNAS 2021, Erzurum, Turkey); Republican scientific conference “Organic compounds and composite materials for various purposes” dedicated to the 110th anniversary of academician Ali Guliyev (Baku, 2022); International scientific conference dedicated to the 60th anniversary of polymer and organic compound technologies (Baku, 2024); II International conference of young researchers dedicated to the 101st anniversary of Heydar Aliyev (Baku, 2024)

Published scientific works. On the topic of the dissertation, 16 scientific papers were published, including 6 articles, 2 patents and several conference materials. Three of the articles have been published in high-impact international journals indexed in Web of Science and Scopus and recommended by the Supreme Attestation Commission under the President of the Republic of Azerbaijan.

The name of the institution where the dissertation work was performed. The presented dissertation work was completed in the laboratory "Physiologically active organic compounds" of the Institute of Chemistry of Additives named after Academician Ali Guliyev of the Academy of Sciences of the Republic of Azerbaijan.

Personal involvement of the author. The author was directly involved in the formulation of research objectives, experimental work, data analysis, and the generalization of results.

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, 3 chapters, a conclusion, a bibliography of 183 sources, and appendices. The dissertation consists of 161 pages, including 15 tables, 32 figures, 63 schemes. In addition, the introduction consists of 14166 characters, Chapter I – 33645, Chapter II – 21656, Chapter III – 96290 and the conclusion – 5245 characters, which in total is 171002 characters.

The **introduction** explains the relevance of the scientific research, its main objective, the fundamental and applied scientific innovations achieved, and the theoretical-practical significance of these innovations.

The **first chapter** provides a comparative analysis and generalization of the most recent literature data on thiocyanates, their synthesis, various transformations—mainly cyclization and heterocyclization—and their fields of application, related to the dissertation topic.

The **second chapter** covers to the experimental section, which includes both the synthesis of new substances and their synthesis methods, details of computational chemical calculations, the conditions for conducting reactions of compounds containing rhodanide (thiocyanate) with cumylperoxyde radicals and cumyl hydroperoxide, the investigation of their fungicidal properties in lubricant-cooling fluids, enzyme analyses, and molecular docking studies.

The **third chapter** discusses the detailed research results related to: the targeted synthesis of alkyl- and arylrhodanides via phase-transfer catalysis; the preparation of new organic compounds containing a thiocyanate group; the synthesis of new compounds containing a thiourea fragment based on rhodanides through three-component condensation; the simulation of the theoretical-experimental mechanism of synthesis reactions for some thiocyanate-containing heterocyclic compounds using DFT calculations; antioxidant additives against the oxidation of hydrocarbons containing rhodanide (thiocyanate), including the study of the more effective compound N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-

(thiocyanato)-4,6-dimethyl]-piperidine as a multifunctional additive for synthetic lubricating oils; the investigation of substances containing a thiocyanate group in the molecule and their derivatives as additives preventing the microbiological degradation of lubricant-cooling fluids; the synthesis and study of 1-(2-Hydroxyphenyl)-2-((4-thiocyanatophenyl)amino)ethanone as a polyethylene stabilizer; and finally, the study of the biological activity of some thiocyanate-containing heterocyclic compounds.

At the end of the dissertation, the conclusions reflecting the conducted research, a list of referenced literature, and appendices are included.

THE MAIN CONTENT OF THE WORK

1. Discussion of results

1.1. Targeted synthesis and study of alkyl- and arylthiocyanates via phase-transfer catalysis.

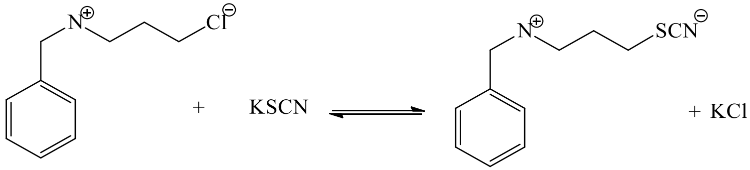
Thiocyanates (I–VI) were synthesized by optimizing the reaction of potassium thiocyanate with alkyl bromides (Scheme 1):

$$RBr + KSCN \longrightarrow \underset{(I\text{-}VI)}{RSCN} + KBr$$

R=CH_3 (I), C_4H_9 (II), C_5H_{11} (III), C_6H_5 (IV), CH_3CH-$COOH$ (V), C_6H_5-CH_2 (VI).

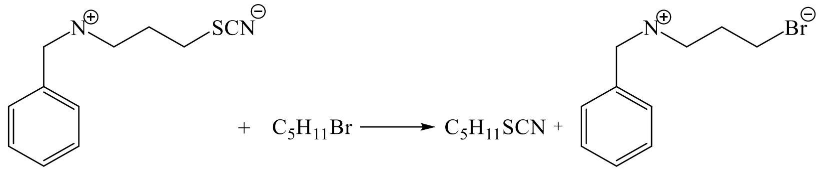
Scheme 1. Synthesis of alkyl(aryl)rhodanides.

For the first time, phase-transfer catalysis was employed in this reaction, enabling a significant increase in the yield of the target compounds from the reported 50–60% to up to 92%. The underlying principle of this method can be explained as follows: one of the immiscible phases (aqueous phase) contains KSCN, which acts as a nucleophile, while the organic phase contains the substrate (e.g., $C_5H_{11}Br$). Since water and organic solvents are immiscible, no reaction occurs initially. However, upon the addition of a catalytic amount of a quaternary ammonium salt, such as $C_6H_5CH_2N(CH_3)_3Cl$, which is soluble in both phases, in this case equilibrium is established in the aqueous phase (Scheme 2):



Scheme 2. Equilibrium in the Aqueous Phase.

The obtaining salt (I) transfers to the organic phase, where the reaction proceeds (Scheme 3):



Scheme 3. Phase Transfer in the Formed Salt.

The resulting pentylthiocyanate remains in the organic phase, while the salt transfers back to the aqueous phase. This process repeats, gradually replacing Br^{-} ions with CNS^{-} ions in solution. Thus, under mild conditions (40–70°C, 1–4 h), pentylthiocyanate was obtained in yields as high as 90%.

Furthermore, by substituting potassium thiocyanate with ammonium thiocyanate, the desired compounds were synthesized under even milder conditions (shorter reaction time and lower temperature), also in high yields, using phase-transfer catalysis.

1.2. Synthesis of novel organic compounds containing the thiocyanate group.

As a new class of heterocyclic thiocyanate derivatives, 1-(phenylsulfonyl)-2-(thiocyanatomethyl)aziridine (VII) was obtained via the reaction of potassium thiocyanate with 2-(chloromethyl)-1-(phenylsulfonyl)aziridine.

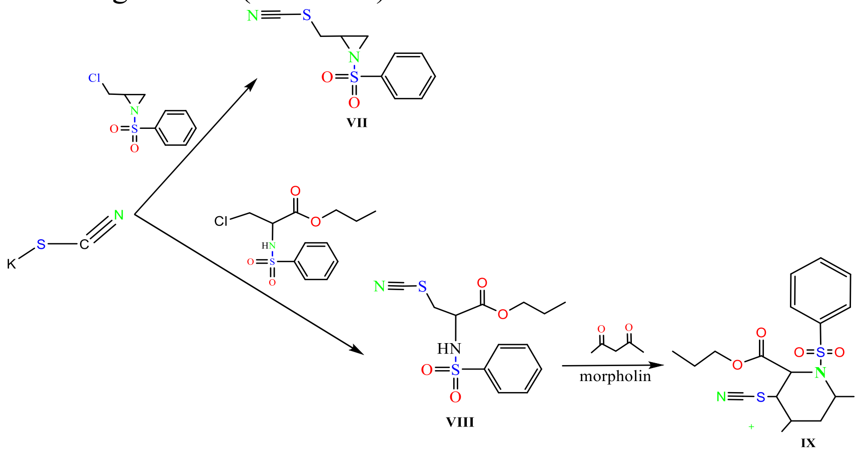
The bond angles in aziridines are approximately 60°, significantly smaller than the normal hydrocarbon bond angle of 109.5°, resulting in angle strain, as observed in cyclopropane and ethylene oxide. This so called “banana bond” model explains bond formation in aziridines.

The ring strain in aziridine also increases the energy barrier for nitrogen inversion. This barrier allows for the isolation of cis- and trans-isomers of 1-(phenylsulfonyl)-2-(thiocyanatomethyl)aziridine (VII).

Additionally, compound VIII was synthesized via the reaction of potassium thiocyanate with propyl-3-chloro-2-(phenylsulfonamide)propionate.

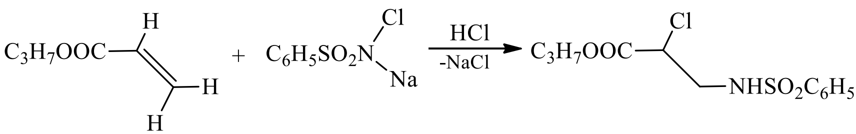
In the next step, N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanate)-4,6-dimethyl]piperidine (IX) was synthesized by the reaction propyl-2-(phenylsulfonamide)-3-(thiocyanate)propionate with acetylacetone (pentane-2,4-dione).

The reaction proceeds in aqueous medium according to the following scheme (Scheme 4).



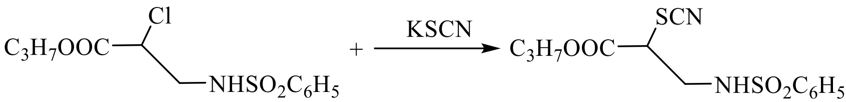
Scheme 4. Synthesis of Heterocyclic Compounds Containing the Thiocyanate Group.

The synthesis of propyl-3-chloro-2-(phenylsulfonamide)propionate, which is used as a starting material for the synthesis of N-[1(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanato)-4,6-dimethyl]-piperidine as follows in Scheme 5:



Scheme 5. Synthesis of Propyl-3-chloro-2-(phenylsulfonamide)propionate.

This intermediate was further used for the synthesis of various derivatives. The presence of an electron-withdrawing sulfonamide fragment facilitates the substitution of the chlorine atom by nucleophiles to form propyl-2-(phenylsulfonamide)-3-(thiocyanate)propionate (Scheme 6):



Scheme 6. Synthesis of Propyl-2-(phenylsulfonamide)-3-(thiocyanate)propionate.

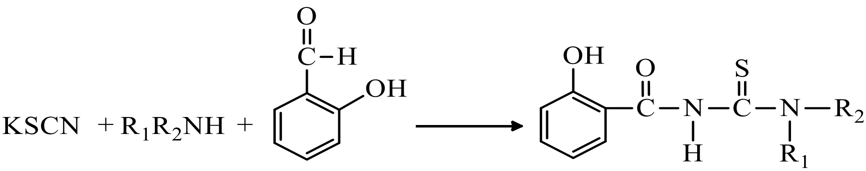
It should be noted that, the reaction between chloramine-B and allyl chloride was studied under various conditions, and the synthesized derivatives 1-phenylsulfonamide-N-2,3-dichloropropane and 1-phenylsulfonamide-N-2-iodo-3-chloropropane were used in further transformations.

The novelty of the synthesized compounds was confirmed using the SciFinder global search tool, and their structures were verified through modern physicochemical techniques (IR, ^{1}H, ^{13}C NMR, elemental analysis).

The specific optical rotation of optically active compounds was measured using an AUTOPOL-III polarimeter.

1.3. Synthesis of hydroxybenzamides based on thiocyanates via three-component condensation.

The synthesis of hydroxybenzamides containing various functional groups, especially a thiourea fragment, in a molecule with multifunctional activity based on rhodanides was carried out in a single-stage three-component reaction according to the following scheme (Scheme 7):



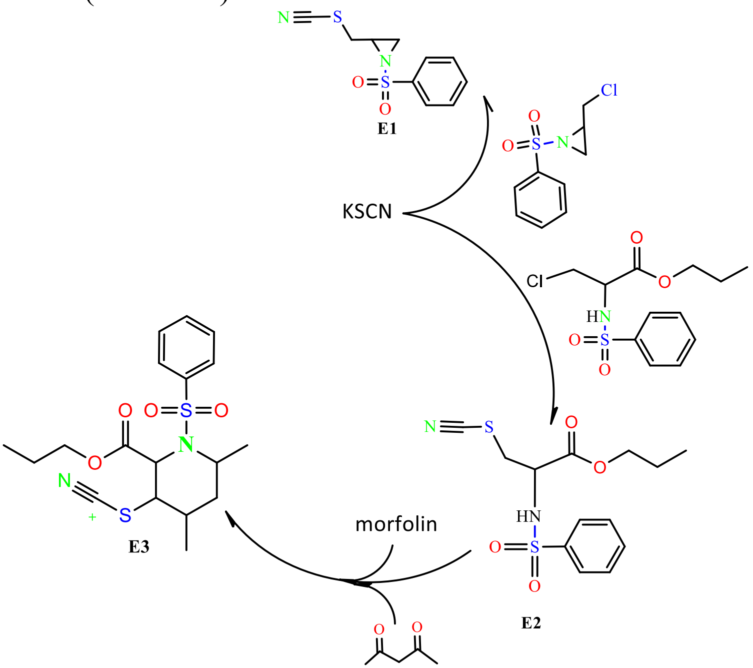
R_1=H; R_2 = C_2H_5 (X), - $C(CH_3)_3$ (XI); - C_4H_9 (XII);
R_1=R_2 = - $(C_4H_9)_2$ (XIII); - $(C_4H_8NH_2)_2$ (XIV)

Scheme 7. Synthesis of Hydroxybenzamides Based on Thiocyanates via Three-Component Condensation.

The structures of the synthesized hydroxybenzamides (X–XIV) were confirmed by IR spectroscopy, and their functional groups were clearly identified by characteristic absorption frequencies.

1.4. Dft study of the synthesis of heterocyclic compounds containing the thiocyanate group.

The aim of this study was to investigate the bioactivity of synthesized heterocyclic compounds—namely, 1-(phenylsulfonyl)-2-(thiocyanatomethyl)aziridine (VII), propyl-2-(phenylsulfonamide)-3-(thiocyanate)propionate (VIII), and N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanate)-4,6-dimethyl]piperidine (IX)—towards key enzymes and to determine the free energy profile of the E1 reaction (Scheme 8):



Scheme 8. Calculation of the reaction mechanism for the synthesis of heterocyclic derivatives of rhodanides.

Since the formation pathway of propyl-2-(phenylsulfonamide)-3-(thiocyanato)propionate (VIII) is the same as that of (1-

(phenylsulfonyl)-2-(thiocyanatomethyl)aziridine (VII), which is the displacement of chloride by cyanide, the related mechanism is not considered here to avoid repetition. The reaction of propyl-2-(phenylsulfonamide)-3-(thiocyanate)propionate (VIII) to form N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanate)-4,6-dimethyl]-piperidine (IX) based on acetylacetone is a multistep mechanism that leads to the cleavage of bonds at the carbonyl and the formation of C-N, C-C bonds.

Computational chemical calculations using DFT were performed for nucleophilic substitution reactions between starting substrates and KSCN, resulting in the formation of (1-(Phenylsulfonyl)-2-(thiocyanatomethyl)aziridine (VII), propyl-2-(phenylsulfonamide)-3-(thiocyanato)propionate (VIII).

Figure 1 shows the reaction pathway for the formation of 1-(phenylsulfonyl)-2-(thiocyanatomethyl)aziridine (VII):

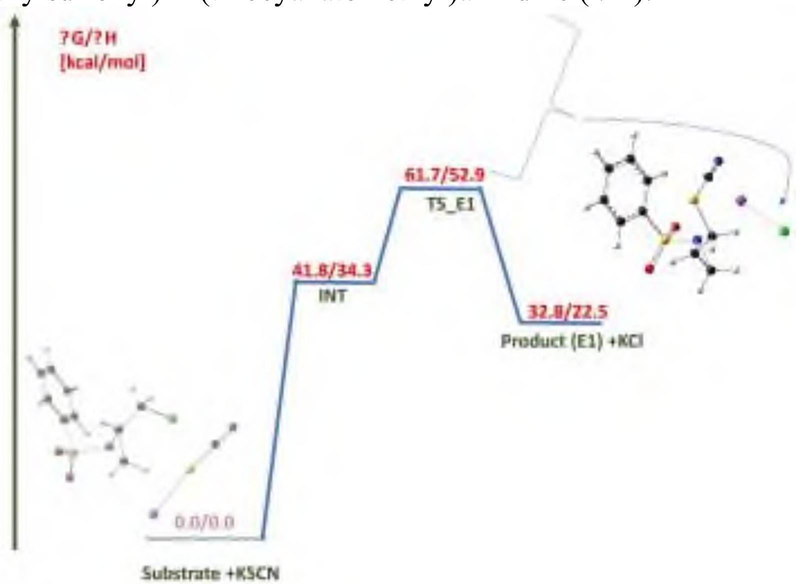


Figure 1. Free energy profile for the formation reaction of (1-(phenylsulfonyl)-2-(thiocyanatomethyl)aziridine (VII). (black: C, gray: H, green: Cl, blue: N, yellow: S, red: O, purple: K)

The overall reaction was found to be endergonic, with a free energy change of 32.8 kcal/mol. The attack of thiocyanate on the electrophilic carbon is possible through an energy barrier of 61.7 kcal/mol (TS_VII). The bond length between the sulfur and electrophilic carbon of thiocyanate is calculated to be 2.58 Å. The C-Cl bond extends to 2.39 Å and Cl makes an electrostatic interaction with potassium at a distance of 4.5 Å. As can be seen from the TS_VII reaction profile, it gives the formation of product (VII) and the release of KCl. The formation of propyl-2-(phenylsulfonamide)-3-(thiocyanato)propionate (VIII) was also calculated using the same method. It is observed that, in contrast to the formation of (1-(phenylsulfonyl)-2-(thiocyanatomethyl)aziridine (VII), the formation of (VIII) is exergonic with an energy of 62.8 kcal/mol. (Figure 2):

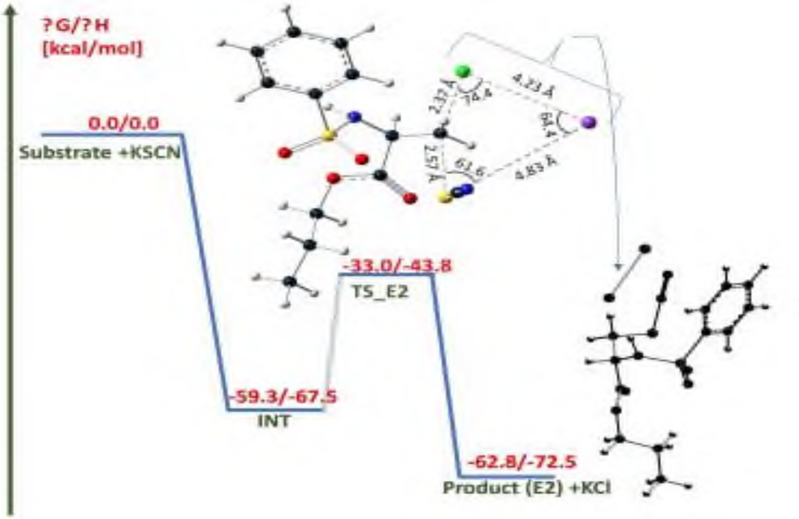


Figure 2. Free Energy Profile of the Reaction Leading to Compound VIII (black: C, gray: H, green: Cl, blue: N, yellow: S, red: O, purple: K)

This contrast between similar reaction pathways shows further exploration into the differing spontaneous behaviors.

Mulliken charge analysis shows that the electrophilic carbon (attached to Cl) in TS_VII and TS_VIII exhibits markedly different charges: 0.09 (VII pathway) vs. 0.54 (VIII pathway). This higher electronegativity in VIII is attributed to the presence of two strongly electron-withdrawing groups near the reactive center, disrupting electron distribution. Analysis of the TS_ VII and TS_VIII connections and their angular active points show that they have similar values.

2. Investigation of the functional properties of the synthesized compounds

2.1. Study of the synthesized compounds as antioxidant additives preventing hydrocarbon oxidation. The antioxidant properties of the newly synthesized heterocyclic compounds containing the thiocyanate group (VII–IX) were studied using model reactions.

The autoxidation of cumene in the presence of these compounds (VII–IX) was examined at 110°C (Figure 3), and it was observed that they inhibit the oxidation process, which is seen from the duration of the induction period (τ).

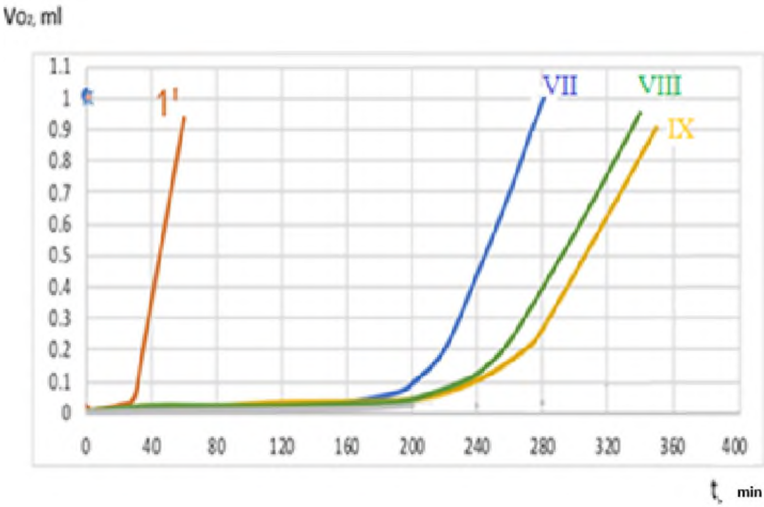


Figure 3. Kinetic curves of cumene autoxidation in the presence of compounds (VII–IX): T = 110°C; I_1[InH] = 0; [InH] = 5×10^{-4} mol•l^{-1}.

Figure 4 shows the kinetic curves of cumene oxidation initiated by azo-isobutyronitrile (AIBN) in the presence of the studied antioxidants (VII–IX).

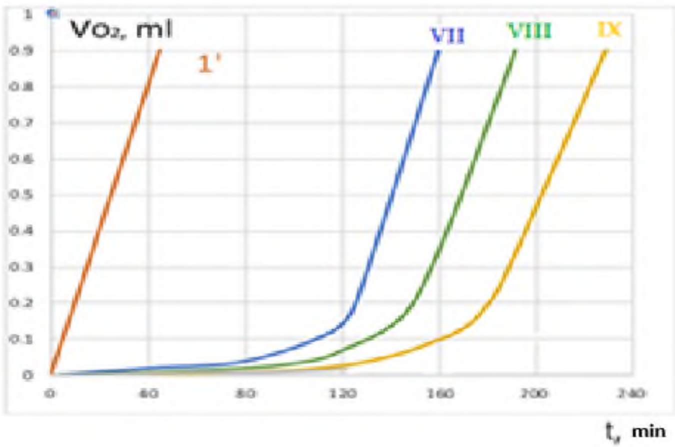


Figure 4. Kinetic curves of AIBN-initiated cumene oxidation in the presence of compounds (VII–IX): T = 60°C; I_1[InH] = 0; [AIBN] = 2×10^{-2} mol•l^{-1}.

From the kinetic curves of cumene oxidation, it is seen that the oxidation rate of cumene after the induction period in the presence of compounds (VII–IX) is lower than that of pure cumene. This confirms that the products formed by reaction with cumylperoxide radicals exhibit inhibitory activity, indicating secondary antioxidant properties.

Additionally, the thiocyanate derivatives (VII–IX) catalytically decompose cumylhydroperoxide (CHP), which forms during oxidation.

The kinetic curve of CHP decomposition in the presence of compound (IX) is shown in Figure 5.

Kinetic parameters characterizing the antioxidant behavior of the thiocyanate derivatives (VII–IX) compared with Ionol are presented in Table 1. As seen Table 1, while the stoichiometric coefficient (f) of Ionol is approximately 2, the values for the new thiocyanate derivatives vary between 3.18 and 6.34. Similarly, the rate constant K_7 ranges from 2.70 to 4.05×10^4 l/(mol•s), showing significantly higher efficiency than Ionol.

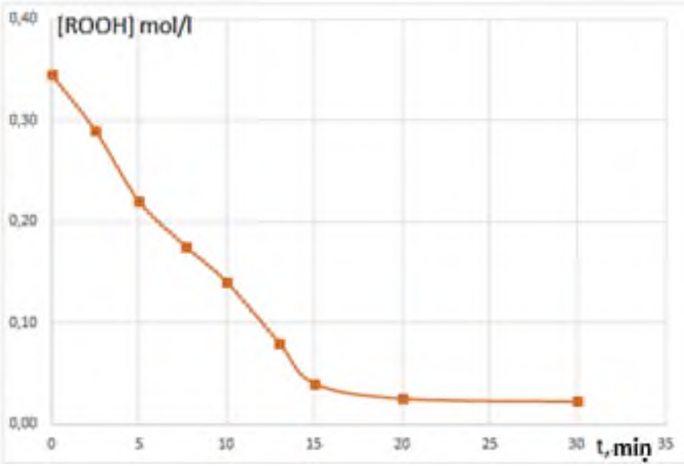
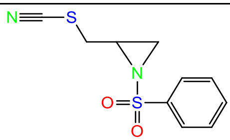
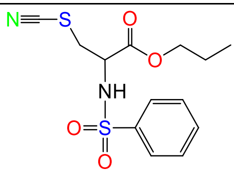
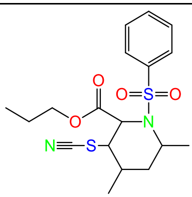


Figure 5. Kinetic curve of CHP decomposition in the presence of compound (IX): T = 110°C; [InH] = 5×10^{-4} mol•l^{-1}; [ROOH] = 0.38 mol•l^{-1}.

Table 1.

Kinetic parameters characterizing the reaction of thiocyanate derivatives (VII–IX) with cumylhydroperoxide and its decomposition

№	Formula of compound	T=60°C		T=110°C		τ, min
		f	$k_7\cdot10^{-4}$ l/(mol·s)	υ	$k_1\cdot$ mol$^{-1}\cdot$s^{-1}	
1	2	3	4	5	6	7
VII		3.18	2.70	6000	5.00	160
VIII		5.76	3.20	8000	6.00	190
IX		6.34	4.05	15000	8.05	240

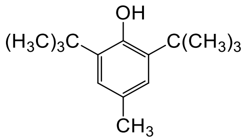
1	2	3	4	5	6	7
Ionol		2.00	2.10	-	-	150

Table 1 shows the number of CHP molecules decomposed by the action of one molecule of antioxidant and its transformation products and is shown the rate constant K of the decomposition reaction with the catalytic factor (υ). It is known that one molecule of cyanate decomposes two molecules of cumylhydroperoxide. However, when a thiocyanate group is included in the molecule, the decomposition of CHP increases a thousand times. The catalytic factor ranges from 6000 to 15000, with compound (IX) showing the highest value of 15000. The rate constant of catalytic decomposition varies from 5.00 to 8.05 $l \cdot mol^{-1} \cdot s^{-1}$. These results demonstrate that the investigated compounds belong to the class of multifunctional antioxidants: they not only break oxidation chains by reacting with cumylperoxide radicals but also catalytically decompose cumylhydroperoxide into molecular products.

2.2. Study of N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanato)-4,6-dimethyl]-piperidine as a multifunctional additive in synthetic lubricating oils.

This section focuses on the synthesis and investigation of N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanat)-4,6-dimethyl]piperidine as a multifunctional additive for synthetic lubricating oils, particularly those used in aviation gas turbine engines. The antioxidant activity of N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanate)-4,6-dimethyl]piperidine was studied by the oxidation method (ГОСТ 23797-79) in the presence of aluminum, steel and copper plates by passing a dry air stream through oil samples at a temperature of 225°C for 25 hours.

As the base oil, pentaerythritol ester (PEE) derived from aliphatic acids (C_5–C_9) was used: $C(CH_2OCOR)_4$, R = C_5–C_9. For comparison, phenyl-α-naphthylamine and 2-mercaptobenzothiazole were tested.

Table 2.

Oxidation resistance of PEE at 225°C in the presence of N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanato)-4,6-dimethyl]-piperidine and known antioxidants

Samples	**After oxidation**						
	Acid number, *mg KOH/g*	**Precipitation,%**	**Kinematic viscosity, *mm^2/sec***		**Corrosion of metals, *g/m^2***		
			100°C	**40°C**	**Aluminum AK-4**	**Steel ШХ-4**	**Copper M-1**
Pentaerythritol ether (PEE*-without additive)	7,6	0,15	7,2	38000	+0,0023	+0,0091	-0,0082
PEE+0,5% phenyl-α-naphthylamine	4,8	0,2	6,45	28000	+0,0031	+0,0049	-0,0078
PEE+1% phenyl-α-naphthylamine	4,6	0,18	6,32	27740	+0,0027	+0,0045	-0,0077
PEE+0,5% 2-mercaptobenzothiazole	3,7	0,30	5,83	33800	+0,0025	+0,0067	-28
PEE+1% 2-mercaptobenzothiazole	4,5	0,32	6,10	33240	+0,0034	+0,0074	-50
PEE+ 0,5% I**	2,7	0,04	5,76	22820	0	0	0
PEE+ 1% I**	2,3	0,06	5,5	21960	0	0	0

Note: *Before oxidation, the acid number of the oil was 0.4 mg KOH/g; its viscosity was 5.1 mm²/s at 100 °C and 9850 mm²/s at -40 °C.
**N-[1-(Phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanato)-4,6-dimethyl]-piperidine.

Table 2 summarizes the results for base oil with and without the synthesized additive and standard antioxidants.

The results indicate that the synthesized compound at 0.5% and 1% concentration significantly improves oxidation resistance parameters such as acid number, viscosity, and corrosion compared to known antioxidants.

Unlike 2-Mercaptobenzothiazole, the formation of deposits in oil that is subject to oxidation in the presence of N-[1-(phenylsulfonyl)-

2-(propoxycarbonyl)-3-(thiocyanato)-4,6-dimethyl]-piperidine and the corrosion of the copper plate are rapidly prevented.

Table 3 shows that N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanato)-4,6-dimethyl]-piperidine improves anti-wear performance compared to 2-mercaptobenzothiazole. The anti-wear effect of the compound was determined on a "Four-ball friction machine" of the ChSHM type (GOST 9490-75). The evaluation indicator of the machine was determined by the diameter of the wear spot formed when the upper ball was subjected to a constant load of 40 kgf for 1 hour and 40 kgf for 4 hours.

As presented in table 3, test results of the effect of the proposed new compound and 2-mercaptobenzothiazole, taken as a standard, on the anti-wear properties of pentaerythritol ether (PEE).

It should be noted regarding the functional effect of N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanato)-4,6-dimethyl]-piperidine as a multifunctional additive in synthetic lubricating oils that well-known antioxidant additives such as phenyl-α-naphthylamine, phenothiazine, and diisooctyldiphenylamine, when added to synthetic oils, do not exhibit sufficient inhibitory properties at high temperatures, which leads to premature wear of engine components operating under severe conditions.

Table 3.

Test results on the effect of the new compound and 2-mercaptobenzothiazole on the anti-wear properties of pentaerythritol ether (pee)

Experimental samples	**Wear scar diameter (WSD), Dy, mm**	
	1 hour (40 *kgF*)	**4 hour (20 *kgF*)**
Pentaerythritol ether (PEE)	0,90	0,90
PEE + 0,5 % 2-mercaptobenztiazole	0,75	0,7
PEE + 1 % 2-mercaptobenztiazole	0,55	0,5
PEE + 0,5 % I*	0,60v	0,63
PEE + 1 % I*	0,5	0,47

Note: I* – N-[1-(Phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanato)-4,6-dimethyl]piperidine.

Except the other antioxidants mentioned above, the newly proposed compound as an additive effectively inhibits the oxidation of synthetic oil under severe temperature conditions, prevents the formation of deposits, reduces variations in the acid number of the oil, and also enhances its anti-wear properties.by adding the multifunctional additive-N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanato)-4,6-dimethyl]piperidine into expensive synthetic lubricants, it is possible to enhance their performance quality and extend service life, resulting in economic benefits.

The multifunctional additive N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanato)-4,6-dimethyl]-piperidine for synthetic lubricating oils is obtained on the basis of industrially produced products and can therefore be practically applied. By adding this newly synthesized compound to synthetic oil, it is possible to improve its quality, extend its service life, and thereby achieve economic efficiency.

Thus, the synthesized compound acts as a multifunctional additive that significantly improves the operational properties of lubricating oils. The presence of several interconnected functional groups in its structure, and the internal synergism whereby one functional group enhances the effect of another, scientifically substantiates the superior efficacy of the new compound—an outcome confirmed by experimental results.

Based on the conducted tests, N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanato)-4,6-dimethyl]piperidine is recommended as an antioxidant and anti-wear additive for synthetic lubricating oils.

2.3. Study of Rhodanines as Additives Preventing the Microbiological Degradation of Lubricating-Cooling Fluids.

Rhodanides were investigated as potential biocidal additives capable of preventing the microbiological deterioration of lubricating-cooling fluids (LCFs). The experimental samples were introduced into emulsion-based LCFs at varying mass percentages, while an LCF without any biocidal additive served as the control.

The antimicrobial efficiency of the investigated samples was assessed on the basis of the diameter of the microbial growth inhibition zone.

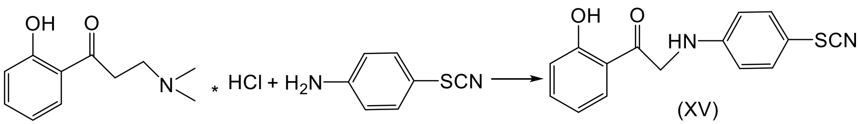
The results of the conducted tests revealed that the studied compounds exhibit dual functionality: they possess a certain degree of bactericidal activity, effectively suppressing bacterial growth, and at the same time, all compounds demonstrated pronounced fungicidal properties by completely inhibiting the growth of fungi.

The fungicidal performance of the examined compounds in LCFs was further evaluated in comparison with Azin-4, a biocidal additive developed at the Institute of Chemistry of Additives and currently applied in industrial practice. The findings demonstrated that all of the investigated compounds possess sufficiently high fungicidal activity, with the majority of them exceeding the efficiency of the industrially applied Azin-4.

In summary, the obtained results clearly indicate that the studied rhodanides can be effectively utilized as biocidal additives with strong fungicidal properties to protect lubricating-cooling fluids from microbiological degradation.

2.4. Synthesis and Investigation of 1-(2-Hydroxyphenyl)-2-[(4-thiocyanatophenyl)amino]ethanone as a Polyethylene (PE) Stabilizer

This section presents the synthesis of 1-(2-hydroxyphenyl)-2-[(4-thiocyanatophenyl)amin]ethanone via the reaction of β-dimethylamino-2-hydroxy-5-methylpropiophenone hydrochloride with 4-thiocyanatoaniline, along with the results of studies on its thermal stability and stabilizing efficiency. The reaction between 4-thiocyanatoaniline and the hydrochloride salt of methylpropiophenone proceeds in an aqueous-alcoholic medium at 80°C, as illustrated in Scheme 9.



Scheme 9. Synthesis of 1-(2-hydroxyphenyl)-2-[(4-thiocyanatophenyl)amino]ethanone

Its stabilizing efficiency is influenced by substituents in the aniline fragment. In this respect, the compound can be distinguished by the effectiveness of its predominant mode of action.

Studies have shown that the compound possesses considerable light-stabilizing activity, which is most likely associated with the presence of a strong intramolecular chelate-type hydrogen bond (OH···O=C) formed between the hydroxyl proton of the phenol group and the oxygen atom of the carbonyl group in the molecule.

A similar regularity was also observed in the study of aged samples, where the difference between the thermoanalytical parameters of unstabilized polyethylene and polyethylene stabilized with the synthesized compound was even more pronounced. This finding confirms the efficiency of 1-(2-hydroxyphenyl)-2-[(4-thiocyanatophenyl)amin]ethanone as a stabilizer.

The investigated compounds also influence the rate of oxidation. Kinetic studies of the oxidation process demonstrated that, for unstabilized polyethylene, the decrease in oxygen pressure reached 10 mmHg after 15 minutes. In contrast, upon incorporation of the synthesized compound into polyethylene, the oxidation rate was significantly reduced. For instance, in the case of 1-(2-hydroxyphenyl)-2-[(4-thiocyanatophenyl)amino]ethanone, a measurable decrease in oxygen pressure was observed only after 140 minutes.

The investigation of the synthesized compounds as stabilizers against the "aging" of polyethylene revealed that 1-(2-hydroxyphenyl)-2-[(4-thiocyanatophenyl)amino]ethanone considerably prolongs the induction period of polyethylene oxidation.

2.5. Investigation of the Inhibitory Activities of New Heterocyclic Compounds Against Enzymes and Isoenzymes.

The inhibitory activity of compounds (VII–IX) against enzymes and isoenzymes was analyzed at Bartın University, Turkey. Among all the tested compounds (VII–IX), the derivative containing a propoxycarbonyl group, N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanato)-4,6-dimethyl]piperidine (IX), was identified as the most potent inhibitor of the hCA I isoform, exhibiting values of Ki = 4.08 ± 0.67 μM and IC_{50} = 3.93 μM with r^2 = 0.9723, in comparison with the standard compound acetazolamide (Ki = 57.64 ± 5.41 μM). It was also determined that the most active compound (IX) was the one showing the highest activity against AChE, according to Ki values (Ki = 52.07 ± 8.33 μM; IC50 = 71.52 μM).

2.6. Molecular Docking Studies.

To elucidate the possible inhibitory mechanisms of the most active compounds—identified through in-vitro experiments as potential drug-like candidates—molecular docking studies were performed. First, the crystal structures of the enzymes were analyzed to determine their catalytic active sites. The binding site with the highest SiteScore (greater than 0.8) was selected as the catalytic active site. These regions, illustrated in Figure 6, were represented as metal-binding sites, hydrophobic regions, and hydrophilic zones. Furthermore, these regions were used for evaluating the best-docked compounds.

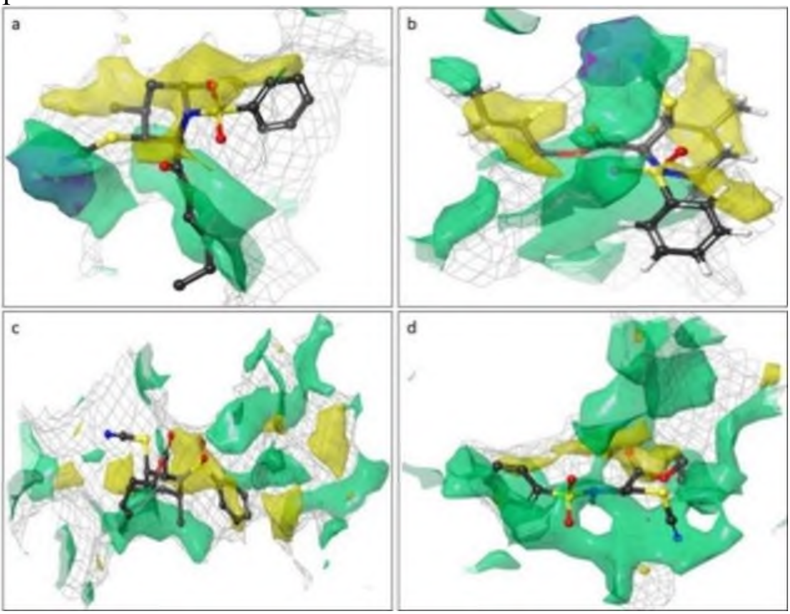


Figure 6. Catalytic active site of the enzyme and evaluation of docking results. a) IX-hCA I, b) IX-hCA II, c) IX-AChE and d) VII-α-glucosidase.

The docked compounds are represented as black stick models. The metallic region is shown as a solid purple surface, the hydrophobic region as a solid yellow surface, the hydrophilic region as a solid green surface, and the catalytic active sites are illustrated as gray surfaces.

Docking results revealed that the most active molecules showed binding affinities of −6.204 (hCA II), −4.423 (hCA I), −6.298 (α-glucosidase), and −6.623 kcal/mol (AChE). The thiocyanate group specifically inhibited hCA I and hCA II.

RESULTS

1. For the first time, purposeful synthetic methods for the reactions of potassium rhodanide and ammonium rhodanide with alkyl bromides in the presence of a phase-transfer catalyst have been developed. Under conditions of a 0.1:0.1 molar ratio of starting materials, a temperature of 65–80 °C, and a reaction time of 1.5–2 hours, the yields of the target alkyl- and arylrhodanides of various structures increased from the literature-reported 50–60% to as high as 87–92% [1, 2, 3].
2. Using the capabilities of fine organic synthesis and theoretical computational chemistry, three novel heterocyclic derivatives of rhodanides (thiocyanates) with high functional properties were obtained for the first time. The novelty of the synthesized compounds was verified through the international search programs *SciFinder* and *Reaxys*, while their structures were confirmed by modern physicochemical analysis methods [4, 7, 9].
3. The mechanisms of the synthesis reactions of the novel heterocyclic rhodanide derivatives were elucidated by DFT calculations. The results showed that two similar reactions possess distinct energy profiles: the formation of 2-(thiocyanato)-1-(phenylsulfonyl)aziridine is endergonic (+32.8 kcal/mol), whereas the synthesis of propyl-2-(phenylsulfonamide)-3-(thiocyanato)propionate is, in contrast, highly exergonic (–62.8 kcal/mol) [7, 8].
4. In model reactions examining the antioxidant properties of

rhodanide derivatives as inhibitors of hydrocarbon oxidation, it was determined that among them, 1-(2-hydroxyphenyl)-2-[(4-thiocyanatophenyl)amino]ethanone (XV) is the most effective inhibitor. Its kinetic parameters for decomposing cumyl hydroperoxide (f = 12.00; $K_7 \cdot 10^{-4}$ = 4.05 l/(mol·s); K = 18.05 $mol^{-1} \cdot s^{-1}$; ϑ = 35,000; τ = 340 min) significantly exceed those of ionol (f = 2.00; $K_7 \cdot 10^{-4}$ = 2.10 L/(mol·s); K = 0; ϑ = 0; τ = 150 min). This compound exhibits combined antioxidant activity by not only scavenging cumylperoxide radicals to terminate oxidation chains but also converting into substances that catalytically decompose cumylhydroperoxide and more effectively neutralize peroxide radicals [5, 6, 10].

5. The synthesized compound N-[1-(phenylsulfonyl)-2-(propoxycarbonyl)-3-(thiocyanato)-4,6-dimethyl]piperidine (IX) was studied as a multifunctional additive that markedly improves the operational properties of synthetic lubricating oils. The presence of several interrelated functional groups within its structure provides internal synergism, whereby the activity of one group enhances that of another. Consequently, when IX was added to lubricating oil at concentrations of 0.5% and 1% and subjected to oxidation at 225 °C, the performance parameters—including corrosion resistance, acid number, and kinematic viscosity—were improved to a much greater extent compared to known antioxidant additives. Therefore, this compound is recommended as a multifunctional additive against oxidation and wear in synthetic lubricants [9].
6. Investigations into rhodanides and their transformation products—novel hydroxybenzamides—as additives (biocides) for preventing microbiological degradation of emulsion-based cutting fluids showed that rhodanides themselves exhibit both bactericidal and fungicidal activity, whereas hydroxybenzamides display only fungicidal properties. Among them, the most active compound, N-(tert-butylcarbamothioyl)-3-hydroxybenzamide, exhibited fungal inhibition zones of 2.8–3.0 cm at a 0.5% concentration. Incorporation of the hydroxyl group into the molecule did not diminish the activity of other functional groups; rather, through

internal synergism, it significantly enhanced antifungal efficacy in lubricating–cooling fluids compared to both the prototype (sodium pentachlorophenolate) and the control sample (bioside-free cutting fluid) [12, 13, 15].

7. The compound 1-(2-hydroxyphenyl)-2-[(4-thiocyanatophenyl)amino]ethanone, synthesized from the reaction of the hydrochloride salt of β-dimethylamino-2-oxo-5-methylpropiophenone with 4-thiocyanatoaniline, demonstrated high stabilizing properties when used as a polyethylene stabilizer. Thermoanalytical studies revealed that, due to the rhodanide group in the aniline fragment, the thermal stability of the compound lies within 165–213 °C, with half-decomposition (T_{50}%) in the range of 200–286 °C. When incorporated into polyethylene, the compound increased resistance to thermal influences as well as the onset temperature of thermal oxidation. The light-stabilizing activity of these compounds is primarily attributed to the presence of a strong intramolecular chelating hydrogen bond (OH···O=C) between the phenolic OH proton and the carbonyl oxygen. Compared to commercial stabilizers such as Benzon-OA and Benazol MBKh, the studied compounds demonstrated superior light-stabilizing efficiency [14].
8. In studies aimed at developing next-generation inhibitors of metabolically relevant enzymes associated with certain global diseases, heterocyclic thiocyanate-containing compounds were evaluated for their inhibitory activity against hCA I, hCA II, AChE, and α-glucosidase enzymes. The newly synthesized molecules exhibited effective inhibition of enzymatic activity at low micromolar concentrations. Molecular modeling showed that the most active molecules possessed binding affinities of 6.204, 4.423, 6.298, and 6.623 kcal/mol toward hCA II, hCA I, α-glucosidase, and AChE, respectively. The thiocyanate fragment of propyl-2-(phenylsulfonamide)-3-(thiocyanato)propionate specifically inhibited hCA I and hCA II. These findings further confirm the potential of CA inhibitors to dilate retinal capillaries and prevent capillary occlusion [11].

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

1. Mammadova, Z.T., Farzaliyev, V.M., Sujayev, A.R., Garibov, E.N., Nazarov, N.M., Najafova, R.A. Synthesis and properties of new rhodanide derivatives // II International Scientific Conference of Young Researchers dedicated to the 95th anniversary of National Leader Heydar Aliyev, – Baku: 27–28 april, -2018, -p. 148-149.
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3. Farzaliyev, V.M. Investigation of the functional properties of new alkyl and aryl derivatives of rhodanides / V.M. Farzaliyev, A.R. Sujayev, Z.T. Mammadova, N.M. Grigoryeva, E.N. Garibov, I.A. Rzayeva, A.A. Hasanli // Scientific Works of the National Aviation Academy, -Baku: -2019 No. 1, p. 44–49.
4. Mammadova, Z.T., Sujayev, A.R., Aliyeva, L.N. Synthesis and biological activity of novel aziridines // 4th International Scientific Conference of Young Researchers Dedicated to the 97th Anniversary of National Leader Heydar Aliyev, - Baku: -2020, - p. 236-237.
5. Farzaliyev, V.M., Israfilova, Z.T., Sujayev, A.R. Creating new and combined effect antioxidants / 1st International Congress on Natural Sciences (ICNAS 2021) Ataturk University, -Erzurum: –2021, – p. 106.
6. Israfilova, Z.T., Farzaliyev, V.M., Aliyeva, L.S., Garibov, E.N., Rzayeva, I.A. Creation of new antioxidants with combined effect improving the operational properties of lubricant materials // XIV International Scientific Conference “Actual Problems of Chemistry” Dedicated to the 98th Anniversary of National Leader Heydar Aliyev, for doctoral, master students and young researchers, -Baku State University, -Baku: 25–26 may, -2021, -

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7. Israfilova, Z.T. Synthesis and investigation of new heterocyclic derivatives of rhodanides // Journal of Young Researchers, - Baku: - 2021. Vol. 7, No.1. - p. 46–52.
8. Israfilova, Z.T., Abdullayev, Y.A., Sujayev, A.R. (2022). Investigation of the theoretical-experimental mechanism of the synthesis reaction of new rhodanide derivatives using DFT calculations // Republic Scientific Conference Dedicated to the 110th Anniversary of Ali Musa oglu Quliyev: "Organic Compounds and Composite Materials for Various Applications", - Baku: 29–30 may, - 2022, - p. 181
9. Farzaliyev, V.M. N-[1-(Phenylsulfonyl)-2-(propoxycarbonyl)-(thiocyanato)-4,6-dimethyl]-piperidine as a multifunctional additive for synthetic lubricating oils, Invention Patent No. i2022 0100, Republic of Azerbaijan / Israfilova Z.T., Sujayev A.R., Aliyeva L.N., Qadirov A.A.
10. Israfilova, Z.T. Study of some Heterocyclic Compounds Containing Rhodanide Group as Antioxidant Additives // – Baku: Herald of the Azerbaijan Engineering Academy, - 2022, Vol, 14, №4, - p. 83 – 91.
11. Israfilova, Z.T. Some thiocyanate containing heterocyclic compounds: Synthesis, bioactivity and molecular docking study / Zubeyda Israfilova, Parham Taslimi, Gulchin İlhami Vagif Farzaliyev, Afsun Sujayev, Muhammet Karaman, Saleh H.Alvasel // Chemistry Select Journal, – 2023. 8 (3), - e202203653
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16. Mammadova, S. Synthesis, biological evaluation and molecular docking of novel sulfonamide derivatives as dual inhibitors of carbonic anhydrase isoenzymes I/II and acetylcholinesterase / Sevgili Mammadova, Yeliz Demir, Zubeyda Israfilova, Lala Zeynalova, Nazar Nazarov, Afsun Sujayev, Nina Ladokhina, Dusan Dimic, Ilhami Gulchin // Journal of Biochemical and Molecular toxicology, -september 2025, № 9, Vol. 39, - e70452.

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