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ABSTRACT

of the dissertation for the degree of
Doctor of Philosophy

SYNTHESIS AND STUDY OF BIOLOGICAL ACTIVITY OF DITHIOCARBAMATE DERIVATIVES

Speciality:	3400.02 – Pharmaceutical chemistry, pharmacognosy
Field of science:	Pharmacy
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GENERAL CHARACTERISTICS OF THE WORK

Relevance of the topic and degree of elaboration

One of the most important challenges facing pharmaceutical science, including pharmaceutical chemistry, is the search for highly reactive compounds for the development of new biologically active substances.

The discovery of new medicinal substances is conducted through rational searches, chemical modification of the structure of known synthetic or natural compounds, synthesis of biologically and physiologically active substances, inclusion of pharmacophores into the molecular structure of new organic compounds, molecular modeling, biotechnology, and other advanced methods.[1]

The purposeful synthesis of substances with diverse biological or pharmacological activities is an area of significant interest nowadays. Within this context, the development of new substances with antifungal activity is particularly relevant. [2] The widespread occurrence of fungal infections, the growing resistance to existing antifungal drugs, and the emergence of previously non-pathogenic fungi as clinical threats necessitate the search for new antifungal agents. Most antifungal medications used in clinical practice, especially polyenes, azoles, and allylamines, suffer from issues of insufficient selectivity and high toxicity.[3]

The search for new pharmacologically active substances with lower toxicity and fewer side effects, effective against fungal infections, and capable of overcoming resistance to currently used

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[1]Andrew, F.P., Ajibade, P.A. Synthesis, characterization and anticancer studies of bis (1-phenylpiperazine dithiocarbamato) Cu(II), Zn(II) and Pt(II) complexes: crystal structures of 1-phenylpiperazine dithiocarbamato-S,S′ zinc(II) and Pt(II) //Journal of Molecular Structure, -2018, 1170, -p. 24–29.

[2] Donnici, C.L., Nogueira, L.J., Araujo, M.H. In Vitro Studies of the Activity of Dithiocarbamate Organoruthenium Complexes against Clinically Relevant Fungal Pathogens // Molecules, -2014, 19 (4), -p. 5402-5420.

[3] Ayodele, T.O., Peter, A.A. Dithiocarbamates: Challenges, Control, and Approaches to Excellent Yield, Characterization, and Their Biological Applications // Bioinorganic Chemistry and Applications, -2019, 15 pages.

antifungal drugs, is both important and relevant for the treatment of living organisms.

One of the promising classes of compounds in terms of antifungal activity are dithiocarbamates. Dithiocarbamates, derivatives of dimethyl dithiocarbamate and ethylene-bis-dithiocarbamic acid, are among the earliest groups of organic compounds known to have fungicidal effects on plants.[4]

Nickel dithiocarbamate derivatives were synthesized, considering the complex formation properties of dithiocarbamates. The antifungal and antibacterial properties of these compounds were tested, and the results demonstrated that they exhibit higher activity than existing antibiotics.[5]

Examples of synthesized dithiocarbamate derivatives include monosubstituted piperazine carbodithioates, with 42 derivatives of this compound having been synthesized. Most of these synthesized substances demonstrated significant antifungal, spermicidal, and antitrichomonal activity.[6]

Therefore, considering the above, the synthesis of new dithiocarbamate derivatives is both important and relevant.

The object and subject of the research

The object of the research is dithiocarbamate salts, 4-morpholinaniline, 4-piperidinaniline, N-methylpiperazine aniline, and 15 newly synthesized substances, which serve as the starting materials in the synthesis of dithiocarbamate derivatives. The identification of these 15 new substances, along with their physicochemical properties,

[4] Kakitani, A. A rapid and sensitive analysis of dithiocarbamate fungicides using modified QuEChERS method and liquid chromatography-tandem mass spectrometry / Ayano Kakitani, Toshiaki Yoshioka, Yasushi Nagatomi [et al.] // Journal of Pesticide Science, -2017, 42 (4), -p. 145–150.

[5] Al-Mukhtar, S., Aghwan, M.T. Synthesis and characterization of 3-methoxypropyldithiocarbamate complexes with Iron (II), Cobalt (II), Nickel (II), Copper (II) and Zinc (II) and their adducts with nitrogenbased ligands // Rafidain Journal of Science, -2013, 24, pp. 50–59.

[6] Jangir, S., Bala, V., Lal, N.A unique dithiocarbamate chemistry during design & synthesis of novel sperm-immobilizing agents // Organic & Biomolecular Chemistry, -2014, 12, -p. 3090–3099.

was conducted using IR, MS, and NMR spectroscopy. Additionally, the biological activities of these new substances were studied[7].

Research goals and objectives

The aim of this research is the synthesis of new dithiocarbamate derivatives, identification of the synthesized compounds, and evaluation of their biological activity.

The following tasks are planned to achieve the set goal:

- preparation of a comprehensive literature review on the physical-chemical properties and biological activities of dithiocarbamate derivatives;
- synthesis of piperidine dithiocarbamate derivatives;
- synthesis of piperazine dithiocarbamate derivatives;
- synthesis of morpholine dithiocarbamate derivatives;
- studying and identifying the structural features of the newly synthesized substances;
- study of biological activity of the synthesized dithiocarbamate derivatives.

Main provisions for the defense

- The development of optimal conditions for synthesis has proven favorable for obtaining new dithiocarbamate derivatives;
- The synthesis conditions developed for the synthesis of piperidine dithiocarbamate, piperazine dithiocarbamate and morpholine dithiocarbamate derivatives provide the basis for obtaining 15 new substances, respectively;
- The study of the biological activity of 15 newly synthesized dithiocarbamate derivatives suggests promising potential for developing a new antifungal agent;
- Investigations of the biological activity of newly synthesized dithiocarbamate derivatives using in silico methods with the PASS program indicate that these compounds are promising for the development of future medicinal substances for the

[7] Suleymanov, T.A., Shukurov, Ch.Y. Synthesis and study of biological activity of dithiocarbamate derivatives // Journal of Medicine and Science, -2018, No.3 (13), - p. 37-41.

treatment of various diseases.

Scientific novelty of the research

For the first time, piperidine dithiocarbamate derivatives 5 substances, piperazine dithiocarbamate derivatives 5 substances, morpholine dithiocarbamate derivatives 5 substances, a total of 15 new substances were synthesized[8]. These compounds were characterized based on their distinctive physicochemical properties and analyzed using UV, IR, MS, and NMR spectroscopy[9].

For the first time, the biological activity of newly synthesized substances was studied, and the following compounds were found to have high antifungal activity: 2-((4-(4-methylpiperazine-1-yl) phenyl) amino)-2-oxoethyl 4-(4-methylbenzyl) piperazine-1-carbodithioate, 2-((4-(4-methylpiperazine-1-yl) phenyl) amino)-2-oxoethyl 4-ethylpiperazine-1-carbodithioate, 2-((4-(piperidine-1-yl) phenyl) amino) -2-oxoethyl 4-benzylpiperazine-1-carbodithioate, 2-((4-(piperidine-1-yl) phenyl) amino)-2-oxoethyl-4-(4-methoxybenzyl) piperazine-1-carbodithioate, 2-((4-morpholinophenyl) amino) - 2-oxoethyl 4-(4-methoxybenzyl) piperazine-1-carbodithioate and 2- ((4-morpholinophenyl) amino)-2-oxoethyl 4-ethylpiperazine-1-carbodithioate.

A Eurasian patent on the synthesis and determination of the biological activity of 2-((4-(4-methylpiperazin-1-yl)phenyl)amino)-2-oxoethyl 4-ethylpiperazine-1-carbodithioate, a dithiocarbamate derivative, was received (No. 025187, 30.09.2016)[10].

The compound 2-((4-Morpholinophenyl)amino)-2-oxoethyl 4-(4-methylbenzyl)piperazine-1-carbodithioate, synthesized for the first time, was registered as a new substance in the American Chemical

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[8] Shukurov, Ch.Y. Chemical structure and biological activity of dithiocarbamate derivatives // Modern Achievements of Azerbaijan Medicine scientific and practical journal, -2020, №1, -p. 44-48.

[9] Suleymanov, T.A., Yusuf Ozkay, Shukurov, Ch.Y. Synthesis and antimicrobial activity of 2-((4-morpholinphenyl)amine)-2-oxoethyl piperazine-1-carbodithioate derivatives // Azerbaijan Journal of Pharmacy and Pharmacotherapy, -2015, No.1, -p.11-16.

[10] Suleymanov, T.A., Shukyurov Ch.Ya. 2-((4-(4-methylpiperazine-1-yl)phenyl)amino)-2-oxoethyl 4-ethylpiperazine-1-carbodithioate, Eurasian patent No. 025187, -2016.

Society Chemical Abstracts Service (CAS) on March 1, 2017, with the registry number (RN) 2055204-10-7.

The theoretical and practical significance of the research

For the first time, 15 new dithiocarbamate derivatives were synthesized, and their biological activity was studied. The results of this research, including the development of optimal conditions for the synthesis of piperidine, piperazine, and morpholine derivatives of dithiocarbamates, provide a theoretical foundation for the synthesis of additional dithiocarbamate derivatives. The synthesized substances 2-((4-(4-methylpiperazine-1-yl)phenyl)amino)-2-oxoethyl-4-(4-methylbenzyl)piperazine-1-carbodithioate, 2-((4-(4-methylpiperazine-1-yl))phenyl)amino)-2-oxoethyl-4-ethylpiperazine-1-carbodithioate, 2-((4-(piperidin-1yl)phenyl)amino)-2-oxoethyl-4-benzyl piperazine-1-carbodithioate, 2-((4-(piperidin-1-yl) phenyl) amino)-2-oxoethyl4-(4-methoxybenzyl) piperazine-1-carbodithioate, 2-((4-morpholinophenyl)amino)-2-oxoethyl 4-(4-methoxybenzyl) piperazine- 1-carbodithioate and 2-((4morpholinophenyl)amino)-2-oxoethyl-4-ethylpiperazine-1-carbodithioate were found to have high antifungal activity. This confirms the practical importance of these compounds in the development of new antifungal drugs.

Personal involvement of the author

All the results reflected in the dissertation were obtained by the author's direct participation in the studies. Setting the issues, conducting experiments and preliminary tests, systematization, research and generalization of obtained experimental and scientific results were carried out with the participation of the author.

Approbation and application

The results obtained during the dissertation work were presented at several scientific-practical conferences, including the "Actual Problems of Medicine" conference dedicated to the 92nd anniversary of the national leader Heydar Aliyev's birth (2015), the "Actual Problems of Medicine" conference dedicated to the 25th anniversary of Azerbaijan's state independence (2017), and the IV International Scientific and Practical Conference "Medical Drugs for Humans: Modern Issues of Pharmacotherapy and Prescription of Medicine" (2020, Ukraine).

The research findings are incorporated into the teaching curriculum in the Bachelor's degree course on "Dithiocarbamic Acid Derivatives" in Pharmaceutical Chemistry at the Faculty of Pharmacy of Azerbaijan Medical University, as well as in the Master's degree course on "Physico-Chemical and Chemical Methods in Pharmaceutical Analysis."

Approval and application 18 scientific works have been published, including 1 Eurasia patent, 9 papers and abstracts of reports at 8 international and republic conferences. 1 substance has been assigned registration number (RN): 2055204-10-7 as a new substance by CAS (Chemical Abstracts Service).

Name of the organization where the dissertation work is performed

The dissertation work was performed at the Department of Pharmaceutical Chemistry of the Azerbaijan Medical University.

The scope and structure of the dissertation

The total volume of the dissertation, indicating the volume of the structural sections separately. The dissertation consists of 200 pages of introduction (12910 characters), 5 chapters (chapter I – 59433 characters, chapter II – 14428 characters, chapter III – 24691 characters, chapter IV – 40937 characters, chapter V – 24691 characters), final part (18280 characters), conclusions (3876 marks), practical recommendations (626 characters), 189 literature sources, including descriptions of abbreviation the total volume consists of 198817 characters (excluding pictures, tables, graphs, appendices and bibliography), The thesis includes 40 tables and 57 pictures.

The dissertation work is relevant to the direction of the scientific work carried out in the department (letter of the Council for Coordination of Scientific Research of the Republic of Azerbaijan, Problem Council for Biological, Agrarian and Medical Sciences No. 93/21, dated 21.06.2021).

MATERIALS AND METHODS OF RESEARCH

For the synthesis of dithiocarbamate derivatives, 1-Methylpiperazine: Merck, 1-Ethylpiperazine: Merck, 1-(4-Methylbenzyl)piperazine: Sigma-Aldrich, 1-Benzylpiperazine: Sigma-Aldrich, 1-(4-Methoxybenzyl)piperazine: Sigma-Aldrich, Carbon disulfide: Merck, Sodium hydroxide: Merck, Ethanol: Technical, Morpholine aniline: Acros Organics, 4-Piperidineaniline: Maybridge, N-methylpiperazineaniline: Maybridge, Chloroacetyl chloride: Merck, Tetrahydrofuran: Merck, Triethylamine: Merck and etc. reagents were used.

Magnetic heating stirrer: Heidolph, MR 3003, Electronic scale: Schimadzu, Libror EB-330 HU, Ultraviolet lamp: Camag, Cabinet, Infrared spectrophotometer: Shimadzu, 8400 FTIR, Nuclear magnetic resonance spectroscopy: Bruker, UltraShield 500 MHz equipment were used.

The melting temperature of the substances was determined by the Electrothermal 9100 digital device.

Fungal strains of *Candida albicans*, *Candida crusei*, *Candida parapsilosis* and *Candida glabrata*, which are pathogenic for the human body, were used for the purpose of studying *in vitro* antifungal activity.

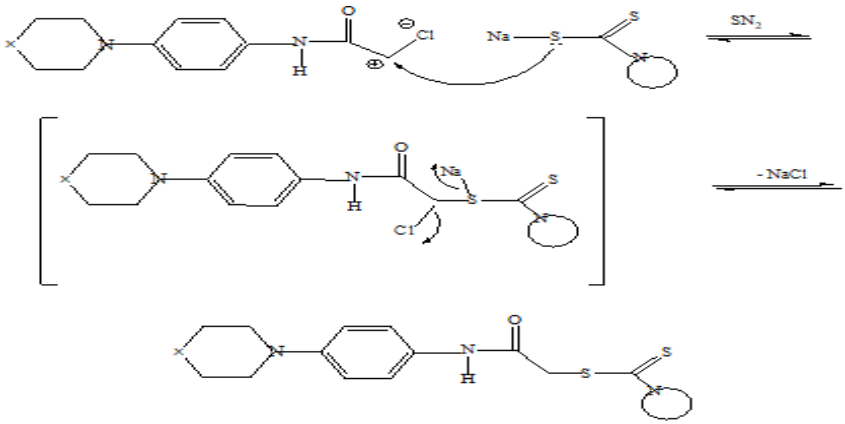
MAIN CONTENTS OF THE WORK

The synthesis process of dithiocarbamate derivatives was carried out in 3 stages. In the first stage, the reactions for obtaining dithiocarbamate salts were carried out.

In the second stage, 4-piperidine aniline, morpholine aniline and N-methyl piperazine aniline are acetylated with chloroacetyl chloride.

In the third stage, 15 new substances were synthesized from the reaction of 2-chloro-N-[4-(4-methyl-1-piperazinyl) phenyl] acetamide, 2-chloro-N-[4-(4-morpholinyl)phenyl] acetamide, and 2-chloro-N-[4-(1-piperidinyl)phenyl]acetamide compounds with each of 5 sodium dithiocarbamate salts (Figure 1). Synthesis reactions were

carried out at room temperature with stirring on a magnetic stirrer at 300 cycles per minute[11] [12]. The obtained dithiocarbamate sodium salt (5 mmol) was dissolved in acetone and the acetylation product (5 mmol) ((separately 2-chloro-N-[4-(4-methyl-1-piperazinyl) phenyl] acetamide, 2-chloro-N[4-(4-morpholinyl)phenyl]acetamide and 2-chloro-N-[4- (1piperidinyl)phenyl]acetamide substances) was added. The end of the reaction is determined by thin-layer chromatography.



X = CH_2; O; N-CH_3

Figure 1. The mechanism of obtaining final products

Identification of 15 new substances obtained as a result of research was carried out.

Substance E1

Melting point: 222^0C

Element analysis: Calculated (%); C, 55.85; H, 6.91; N, 13.71; O 7.83; S, 15.70.

[11] Priya, M.G., Panneerselvam, P., Karikalan, M. Synthesis, characterization and antibacterial, antifungal activities of Schiff bases of 4-(2-aminphenyl) morpholines // International Journal of Pharma and Bio Sciences, -2011, 2, -p. 267-272.

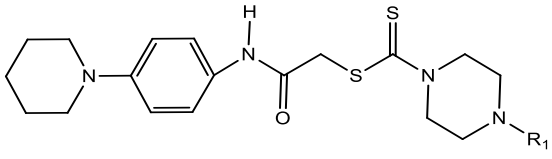
[12] Quiroga D., Becerra L.D., Coy-Barrera E. Ultrasound-Assisted Synthesis, Antifungal Activity against *Fusarium oxysporum* and Three-Dimensional Quantitative Structure–Activity Relationship of *N*, *S*-Dialkyl Dithiocarbamates Derived from 2-Amino Acids // ACS Omega, -2019, 4, -p. 13710−13720.

As a result of studying the structure of the substance by using IR-spectroscopy, the N-H group in the absorption area of 3288 cm^{-1}, the aliphatic C-H groups according to the absorption areas of 2933-2848 cm^{-1}, the C=O group according to the absorption area of 1666 cm-1, the C=N and C=C groups according to their absorption areas of 1512-1417 cm^{-1} and 1,4-substituted benzene ring according to the absorption area of 806 cm^{-1} were determined

The molecular weight of the mentioned compound is 482, it was determined by means of MS-spectrometry.

As a result of the H^1 NMR-spectroscopy study of substance E1, the presence of 25 (H) aliphatic hydrogen protons at the 1.60 and 4.23 absorption areas was determined by the received multiplet signals. Aromatic hydrogen protons present in the structure of the substance were identified by obtaining doublet signals (2H) in the absorption areas at 6.86, 7.15, 7.21 and 7.39. The hydrogen proton attached to the nitrogen atom in the structure of the substance was identified by a singlet signal in the 10.01 (N-H) absorption region.

Consequently, according to its physicochemical properties, the results of IR-, NMR-spectroscopy, and MS-spectrometry, substance E1 was identified as 2-((4-(piperidine-1-yl)phenyl)amino)-2-oxoethyl4-(4-methylbenzyl)piperazine1-carbodithioate .



R1=-CH_2-C_6H_5-CH_3 - **E1**
R1=-C_6H_5 - **E2**
R1=-CH_2-C_6H_5-O-CH_3 - **E3**
R1=-CH_3 - **E4**
R1=-C_2H_5 - **E5**

Substance E2

Melting point: 188-190°C

Elemental analysis: Calculated (%): C, 64.07; H, 6.88; N, 11.95; O 3.41; S, 13.68

As a result of the research conducted by infrared spectrum analysis of E2 substance, N-H group at 3277 cm^{-1} absorption area, aliphatic C-H groups at 2933-2808 cm^{-1} absorption area, C=O group at 1658 cm^{-1} absorption area, C=N and C=C groups at absorption

range 1529-1429 cm^{-1} and 1,4-substituted benzene ring by absorption area at 856 cm^{-1} were determined.

The molecular weight of the mentioned substance was proved to be 467 by using of MS-spectrometry.

As a result of studying this compound by using of H^1 NMR-spectroscopy, the presence of 22 (H) aliphatic hydrogen protons in absorption fields 1.51 and 4.63 was determined by the received multiplet signals. Aromatic hydrogen protons present in the structure of the substance were identified by obtaining doublet signals (2H) in the absorption areas at 6.86, 7.27 and 7.39. The hydrogen proton attached to the nitrogen atom present in the structure of the compound was studied with a singlet signal in the 10.01 (N-H) absorption region.

Consequently, according to its physicochemical properties, the results of IR-, NMR-spectroscopy, and MS-spectrometry, substance E2 was identified as 2-((4-(piperidine-1-yl)phenyl)amino)-2-oxoethyl 4-benzylpiperazine-1-carbodithioate.

Substance E3

Melting point: 153-155°C

Elemental analysis: Calculated (%): C, 62.62; H, 6.87; N, 11.23; O 6.82; S, 12.86.

Based on the results of the IR-spectroscopy analysis of E3 substance, N-H group at 3277 cm^{-1} absorption area, aliphatic C-H groups at 2934-2808 cm^{-1} absorption areas, C=O group at 1659 cm^{-1} absorption area, C=N and C=C groups at 1530-1356 cm^{-1} absorption areas and 1,4-substituted benzene ring according to the absorption area of 856 cm^{-1} were determined.

The molecular weight of the mentioned compound is 498, it was determined by means of MS-spectrometry.

As a result of the investigation of the structure of the mentioned substance by means of H^1 NMR-spectroscopy, the presence of 25 (H) aliphatic hydrogen protons in the 2.5 and 4.63 absorption areas was determined by the received multiplet signals. Aromatic hydrogen protons present in the structure of the substance were identified by obtaining doublet signals (2H) in the absorption areas at 6.87, 6.91, 7.24 and 7.39. The hydrogen proton attached to the nitrogen atom present in the structure of the substance was studied with a singlet

signal in the 10.05 (N-H) absorption region.

Consequently, according to its physicochemical properties, the results of IR-, NMR-spectroscopy, and MS-spectrometry, substance E3 was identified as 2-((4-(piperidine-1-yl)phenyl)amino)-2-oxoethyl4-(4-methoxybenzyl)piperazine-1-carbodithioate.

Substance E4

Melting point: 135-138^{0}C

Elemental analysis: Calculated (%): C, 58.13; H, 7.19; N, 14.27; O 4.08; S, 16.34.

The study of the structure of this substance using IR-spectroscopy revealed in its structure the N-H group at the absorption area of 3277 cm-1, aliphatic C-H groups at the absorption area of 2929-2787 cm-1, the C=O group at the absorption area of 1658 cm-1, the C=N and C=C groups at the absorption areas of 1533-1469 cm-1 and 1,4-substituted benzene ring at the absorption area of 854 cm-1 were determined.

MS-spectrometry proved that the molecular weight of the mentioned substance is 392.

The study of substance E4 by means of H^1 NMR-spectroscopy determined the presence of 23 (H) aliphatic hydrogen protons in the absorption fields of 2.51 and 4.62 in the structure of the mentioned substance with multiplet signals. The hydrogen proton attached to the nitrogen atom in the structure of the substance was studied with a singlet signal in the 10.05 (N-H) absorption region.

Consequently, according to its physicochemical properties, the results of IR-, NMR-spectroscopy, and MS-spectrometry, substance E4 was identified as 2-((4-(piperidine-1-yl)phenyl)amino)-2-oxoethyl 4-methylpiperazine-1-carbodithioate.

Substance E5

Melting point: 173-175^{0}C

Elemental analysis: Calculated (%): C, 59.08; H, 7.44; N, 13.78; O 3.93; S, 15.77.

According to the investigation of this substance using infrared spectrum, in its structure N-H group according to absorption area of 3290 cm^{-1}, aliphatic C-H groups according to absorption area of 2926-2806 cm^{-1}, C=O group according to absorption area of 1664 cm^{-1}, C=N

and C=C groups according to absorption areas of 1519-1429 cm^{-1} and 1,4-substituted benzene ring according to the absorption area of 819 cm^{-1} were determined.

According to the research conducted by using of MS-spectrometry, the molecular weight of the substance was confirmed to be 406.

Based on the spectrum result obtained by using of H^1 NMR-spectroscopy of the substance in question, the presence of 25 (H) aliphatic hydrogen protons in absorption fields 2.37 and 4.20 was determined by the received multiplet signals. Aromatic hydrogen protons in the structure of the substance were identified by obtaining doublet signals (2H) in the 6.88 and 7.40 absorption fields. The hydrogen proton attached to the nitrogen atom present in the structure of the substance was studied with a singlet signal in the 10.01 (N-H) absorption region.

Consequently, according to its physicochemical properties, the results of IR-, NMR-spectroscopy, and MS-spectrometry, substance E5 was identified as 2-((4-(piperidine-1-yl)phenyl)amino)-2-oxoethyl 4-ethylpiperazine-1-carbodithioate.

Substance A1

Melting point: 161^0C

Element analysis: Calculated (%); C, 62.74; H, 7.09; N, 14.07; O, 3.21; S, 12.88.

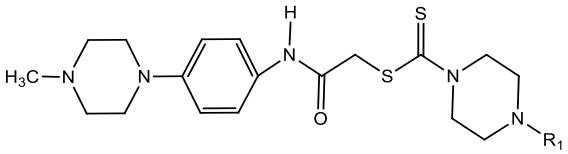
The structure of the synthesized substance was also studied by means of IR spectroscopy. According to the results, the presence of N-H group in the absorption area of 3282 cm^{-1} in the synthesized substance, aliphatic C-H groups according to the absorption areas of 2937-2796 cm^{-1}, C=O group according to the absorption area of 1666.5 cm^{-1}, C =N and C=C groups according to the absorption areas of 1589-1419 cm^{-1} and 1,4-substituted benzene ring to the absorption area at 852 cm^{-1} were determined.

According to H^1 NMR-spectroscopy, the molecular weight of the 2-((4-(4-Methylpiperazine-1-yl)phenyl)amino)-2-oxoethyl 4-(4-methylbenzyl)piperazine-1-carbodithioate substance was found to be 497.

The structure of the substance was studied by H^1 NMR

spectroscopy. In the obtained results, multiplet signals confirmed the presence of 26 (H) aliphatic hydrogen protons at the 2.20 and 4.20 absorption areas. Aromatic hydrogen protons present in the substance were determined by obtaining doublet signals (2H) in the absorption ranges at 6.88, 7.15, 7.21 and 7.41. The hydrogen proton attached to the nitrogen atom in the compound was identified by a singlet signal in the 10.01 (N-H) absorption region.

Thus, based on its physico-chemical properties, the results of IR, NMR-spectroscopy and MS-spectrometry, the structure of substance A1 was identified as 2-((4-(4-Methylpiperazine-1-yl)phenyl)amino)-2-oxoethyl 4-(4-methylbenzyl)piperazine-1-carbodithioate. The general formula of substances from the "A" series is as follows:



R1=-CH_2-C_6H_5-CH_3 - **A1**
R1=-C_6H_5 - **A2**
R1=-CH_2-C_6H_5-O-CH_3 - **A3**
R1=-CH_3 - **A4**
R1=-C_2H_5 - **A5**

Substance A2

Melting point: 219^0C

Element analysis: Calculated (%); C, 62.08; H, 6.88; N, 14.48; O, 3.31; S, 13.26.

The spectra of 2-((4-(4-methylpiperazine-1-yl)phenyl)amino)-2-oxoethyl 4-benzylpiperazine-1-carbodithioate substance were recorded by using of IR spectroscopy and its structure was confirmed. The presence of the N-H group in the absorption area of 3282 cm^{-1} in the spectrum, aliphatic C-H groups according to the absorption areas of 2937-2796 cm^{-1}, the C=O group according to the absorption area of 1647 cm^{-1}, C=N, and C=C groups according to the absorption areas of 1533-1417 cm^{-1} and the 1,4-substituted benzene ring according to the absorption area of 891 cm^{-1} were determined.

Based on the results of MS-spectrometry, the molecular weight of the mentioned substance was determined to be 483.

The structure of the substance was studied based on the number of hydrogen protons in the synthesized compound by using of H^1 NMR analysis. In the obtained results, multiplet signals confirmed the

presence of 23 (H) aliphatic hydrogen protons at the 2.22 and 4.20 absorption areas. Aromatic hydrogen protons present in the substance were determined by receiving doublet signals (2H) in the 6.87 absorption area and multiplet signals (9H) in the 7.34-7.36 absorption areas. The hydrogen proton attached to the nitrogen atom in the compound was identified by a singlet signal at the 10.01 (N-H) absorption region.

Thus, substance A2 was identified as 2-((4-(4-methylpiperazine-1-yl) phenyl) amino)-2-oxoethyl 4-benzylpiperazine-1-carbodithioate based on its physico-chemical properties, IR-, NMR-spectroscopy and MS-spectrometry results.

Substance A3

Melting point: 182^0C

Element analysis: Calculated (%); C, 60.79; H, 6.87; N, 13.63; O, 3.31; S, 13.26. The spectral analysis results are shown below.

According to the IR-spectroscopy results, N-H group in the absorption area of 3275 cm^{-1}, aliphatic C-H groups in the absorption areas of 2929-2794 cm^{-1}, C=O group in the absorption area of 1649 cm^{-1}, C=N and C=C groups in the absorption areas of 1531-1417 cm^{-1} and 1,4-substituted benzene ring in absorption area at 883 cm^{-1} were determined.

The molecular weight of substance A3 was determined to be 513 according to MS-spectrometry.

Based on the results of H^1 NMR-spectroscopy conducted on the substance, multiplet signals confirmed the presence of 26 (H) aliphatic hydrogen protons in the 2.23 and 4.20 absorption fields. Aromatic hydrogen protons present in the substance were determined by receiving doublet signals (2H) at 6.87, 7.25 and 7.42 absorption areas and multiplet signals (4H) at 6.87-6.92 absorption areas. The hydrogen proton attached to the nitrogen atom in the compound was identified by a singlet signal in the 10.01 (N-H) absorption region.

Thus, substance A3 was identified as 2-((4(4-Methylpiperazine-1-yl)phenyl)amino)-2-oxoethyl 4-(4-methoxybenzyl)piperazine-1-carbodithioate based on its physico-chemical properties, IR-, NMR-spectroscopy and MS-spectrometry results.

Substance A4

Melting point: 196^0C

Element analysis: Calculated (%); C, 55.99; H, 7.17; N, 17.18; O, 3.93; S, 15.73.

The structure of the substance was studied using IR spectroscopy, according to the results, the N-H group at the absorption area of 3254 cm^{-1}, aliphatic C-H groups at the absorption area of 2918-2848 cm^{-1}, the C=O group at the absorption area of 1657 cm^{-1}, the C=N, and C=C groups according to the absorption areas of 1514-1415 $cm^{-1}$and 1,4-substituted benzene ring according to the absorption area of 820 cm^{-1} were determined.

The molecular weight of substance A4 was determined to be 407 according to MS-spectrometry.

According to the H^1 NMR spectra of this substance, multiplet signals confirmed the presence of 24 (H) aliphatic hydrogen protons in the 2.23 and 4.21 absorption fields. Aromatic hydrogen protons present in the substance were identified by obtaining doublet signals (2H) in the 6.88 and 7.41 absorption areas. The hydrogen proton attached to the nitrogen atom in the compound was identified by a singlet signal in the 10.01 (N-H) absorption region.

Consequently, substance A4 was identified as 4-methylpiperazine-1-carbodithioate based on its physico-chemical properties, IR-, NMR-spectroscopy and MS-spectrometry results.

Substance A5

Melting point: 184^0C

Element analysis: Calculated (%); C, 56.97; H, 7.41; N, 16.61; O, 3.79; S, 15.21.

According to the obtained IR-spectral results, the N-H group in the absorption area of 3264 cm^{-1}, the aliphatic C-H groups according to the absorption areas of 2974-2933 cm^{-1}, the C=O group according to the absorption area of 1658 cm cm^{-1}, C=N and C=C groups according to the absorption areas of 1508-1429 cm^{-1} and 1,4-substituted benzene ring according to absorption area of 825 cm^{-1} were determined.

Based on the results of MS-spectrometry, it was confirmed that the molecular weight of substance A5 is 421.

According to the H^1 NMR-spectroscopy results, the obtained multiplet signals confirmed the presence of 24 (H) aliphatic hydrogen protons in the 2.29 and 4.21 absorption areas. Aromatic hydrogen protons present in the substance were determined by obtaining doublet (2H) signals at the 6.88 and 7.42 absorption areas. The hydrogen proton attached to the nitrogen atom in the compound was identified by a singlet signal in the 10.03 (N-H) absorption region.

Thus, substance A5 was identified as 2-((4-(4-Methylpiperazine-1-yl)phenyl)amino)-2-oxoethyl 4-ethylpiperazine-1-carbodithioate based on its physico-chemical properties, IR-, NMR-spectroscopy and MS-spectrometry results.

Substance J1

Melting point: 145.5^0C

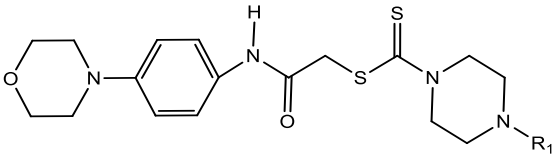
Element analysis: Calculated (%); C, 61.95; H, 6.65; N, 11.56; O, 6.60; S, 13.23.

According to the IR-spectral results of the synthesized substance, N-H group at 3292 cm^{-1} absorption area, aliphatic C-H groups at 2852-2767 cm^{-1} absorption area, C=O group at 1666.5 cm^{-1} absorption area, C=N and C=C groups at1514-1473 cm^{-1} absorption area and 1,4-substituted benzene ring according to the absorption area of 813 cm^{-1} were determined.

The molecular weight of substance J1 was determined to be 484 according to MS-spectrometry.

According to the results of NMR-spectroscopy of substance J1, multiplet signals confirmed the presence of 24 (H) aliphatic hydrogen protons in absorption areas 2.3 and 4.63. Aromatic hydrogen protons present in the substance were determined by obtaining doublet signals (2H) in the absorption areas at 6.89, 7.14, 7.20 and 7.43. The hydrogen proton attached to the nitrogen atom in the compound was identified by a singlet signal in the 10.04 (N-H) absorption region.

Thus, based on its physico-chemical properties, the results of IR, NMR-spectroscopy and MS-spectrometry, substance J1 was identified as 2-((4-Morpholinophenyl)amino)-2-oxoethyl 4-(4-methylbenzyl) piperazine-1-carbodithioate. The general formula of substances from the "**J**" series is as follows:



R1=-CH_2-C_6H_5-CH_3 - **J1**
R1=-C_6H_5 - **J2**
R1=-CH_2-C_6H_5-O-CH_3 - **J3**
R1=-CH_3 - **J4**
R1=-C_2H_5 - **J5**

Substance J2

Melting point: 148^0C

Element analysis: Calculated (%); C, 61.25; H, 6.42; N, 11.90; O, 6.80; S, 13.63.

According to the IR-spectroscopy results, the N-H group in the absorption area of 3292 cm^{-1}, the aliphatic C-H groups according to the absorption areas of 2852-2767 cm^{-1}, the C=O group according to the absorption area of 1666.5 cm^{-1}, and the C=N and C=C groups according to the absorption areas of 1514-1474 cm^{-1} and 1,4-substituted benzene ring according to the absorption area of 814 cm^{-1} were determined.

Based on the result of MS-spectrometry, the molecular weight of substance J2 was determined to be 470.

Based on the NMR-spectroscopy of the substance, multiplet signals confirmed the presence of 24 (H) aliphatic hydrogen protons in the 2.26 and 4.37 absorption areas. Aromatic hydrogen protons present in the substance were determined by obtaining doublet signals in the absorption areas (2H) at 6.89, 7.15, 7.21 and 7.43. The hydrogen proton attached to the nitrogen atom in the compound was identified by a singlet signal in the 10.04 (N-H) absorption region.

Thus, based on its physicochemical properties, the results of IR-, NMR-spectroscopy and MS-spectrometry, substance J2 was identified as 2-((4-morpholinophenyl)amino)-2-oxoethyl4-benzylpiperazine-1-carbodithioate.

Substance J3

Melting point: 155^0C.

Elemental analysis: Calculated (%);C, 59.97; H, 6.44; N, 11.19; O 9.59; S, 12.81.

The IR-spectroscopy results of the mentioned substance show the N-H group in the absorption area of 3261 cm^{-1}, the aliphatic C-H

groups in the absorption areas of 3049-2960 cm^{-1}, the C=O group in the absorption area of 1660 cm^{-1}, and the C =N and C=C groups in the absorption areas of 1614-1433 cm-1 and 1,4-substituted benzene ring according to the absorption area of 821 cm-1 were determined.

The molecular weight of substance J3 was proved to be 500 by means of MS-spectrometry.

Referring to the H^1 NMR spectrum results, the multiplet signals obtained confirmed the presence of 23 (H) aliphatic hydrogen protons in the 2.51 and 4.63 absorption areas. Aromatic hydrogen protons present in the substance were identified by obtaining doublet signals (2H) in the absorption areas at 6.89, 6.91, 7.23 and 7.43. The hydrogen proton attached to the nitrogen atom in the compound was identified by a singlet signal in the 10.08 (N-H) absorption region.

Thus, based on its physicochemical properties, the results of IR-, NMR-spectroscopy and MS-spectrometry, substance J3 was identified as 2-((4-morpholinophenyl)amino)-2-oxoethyl 4-(4-methoxybenzyl)piperazine-1-carbodithioate.

Substance J4

Melting point: 130^0C

Elemental analysis: Calculated (%);C, 54.79; H, 6.64; N, 14.20; O, 8.11; S, 16.25.

According to the IR-spectral analysis, the N-H group in the absorption area of 3254 cm^{-1}, the aliphatic C-H groups according to the absorption areas of 2961-2851 cm^{-1}, the C=O group according to the absorption area of 1649 cm^{-1}, C= N and C=C groups according to the absorption areas of 1535-1415 $cm^{-1}$and 1,4-substituted benzene ring according to absorption area of 858 cm^{-1}were determined.

According to the results of the MS-spectrometry analysis, the molecular weight of the mentioned substance was determined to be 394.

Based on the results of H^1 NMR-spectra of substance J4, the presence of 21 (H) aliphatic hydrogen protons in absorption areas 2.51 and 4.63 was determined by the received multiplet signals. Aromatic hydrogen protons present in the substance were determined by obtaining doublet signals (2H) in the 6.90 and 7.43 absorption areas. The hydrogen proton attached to the nitrogen atom in the structure of

the substance was identified by a singlet signal in the 10.09 (N-H) absorption region.

Thus, based on its physicochemical properties, the results of IR, NMR-spectroscopy and MS-spectrometry, substance J4 was identified as 2-((4-morpholinophenyl)amino)-2-oxoethyl 4-methylpiperazine-1-carbodithioate.

Substance J5

Melting point: 222^0C

Element analysis: Calculated (%); C, 55.85; H, 6.91; N, 13.71; O, 7.83; S, 15.70.

As a result of the investigation of the structure of the substance by means of the IR spectrum, the N-H group in the absorption area of 3254 cm^{-1}, the aliphatic C-H groups according to the absorption areas of 2960-2852 cm^{-1}, the C=O group according to the absorption area of 1649 cm^{-1}, C=N, and C=C groups according to their absorption areas of 1535-1415 cm^{-1} and 1,4-substituted benzene ring according to the absorption area of 820 cm^{-1} were determined.

The molecular weight of the substance is 408. It was studied by means of MS-spectrometry.

The presence of 23 (H) aliphatic hydrogen protons in absorption fields 2.51 and 4.25 in the H^1 NMR-spectrum of substance J5 was determined by the obtained multiplet signals. Aromatic hydrogen protons present in the structure of the substance were determined by obtaining doublet signals (2H) in the absorption areas at 6.89 and 7.44. The hydrogen proton attached to the nitrogen atom in the structure of the substance was identified by a singlet signal in the 10.10 (N-H) absorption region.

Thus, based on its physico-chemical properties, IR-, NMR-spectroscopy and MS-spectrometry results, J5 substance was identified as 2-((4-morpholinophenyl)amino)-2-oxoethyl 4-ethylpiperazine-1-carbodithioate.

Consequently, as a result of research, 15 new substances related to dithiocarbamate derivatives were synthesized and their physicochemical properties were identified based on IR-, MS- and NMR-spectral methods.

Some of the synthesized substances were found to be non-toxic

up to a dose of 100 μg/ml.

During the study of antifungal activity, the newly synthesized substance A5 was proposed as an antifungal substance with the most active fungicidal effect. Also, E3 and E4 substances against Candida have also been considered as antifungal agents.

In silico studies have shown the prospect of using the synthesized substances in the treatment of cytostatic, antianginal, antibacterial, eczema, alopecia and many other diseases, along with antifungal activity.

CONCLUSIONS

1. For the synthesis of dithiocarbamate derivatives, optimal conditions consisting of 3 stages have been developed with the synthesis of dithiocarbamate salts, acetylation of 4-piperidine aniline, morpholine aniline and N-methyl piperazine aniline compounds, and the reaction of acetylation products with dithiocarbamate salts, and as a result, 15 new substances have been synthesized.
2. 5 new substances with piperidine and dithiocarbamate fragments in their structure have been synthesized, and their physico-chemical properties were identified using IR-, NMR-spectroscopy and MS-spectrometry as 2-((4-(piperidin-1-yl)phenyl)amino)-2-oxoethyl 4-(4-methylbenzyl)piperazine-1-carbodithioate, 2-((4-(piperidin-1-yl)phenyl)amino)-2-oxoethyl 4-benzyl piperazine-1-carbodithioate, 2-((4-(piperidin-1-yl)phenyl)amino)-2-oxoethyl 4-(4-methoxybenzyl)piperazine-1-carbodithioate, 2-((4-(piperidin-1-yl)phenyl)amino)-2-oxoethyl 4-methylpiperazine-1-carbodithioate and 2-((4-(piperidin-1-yl)phenyl)amino)-2-oxoethyl 4-ethylpiperazine -1-carbodithioate.
3. 5 new substances with morpholine and dithiocarbamate fragments in their structure have been synthesized, and their physico-chemical properties were identified using IR-, NMR-spectroscopy and MS-spectrometry as 2-((4-morpholinophenyl)amino)-2-oxoethyl 4-(4-methylbenzyl)piperazine-1-carbodithioate, 2-((4-

morpholinophenyl)amino)-2-oxoethyl 4-benzylpiperazine-1-carbodithioate, 2 -((4-morpholinophenyl)amino)-2-oxoethyl 4-(4-methoxybenzyl)piperazine-1-carbodithioate, 2-((4-morpholinophenyl)amino)-2-oxoethyl 4-methylpiperazine-1-carbodithioate, 2- ((4-morpholinophenyl)amino)-2-oxoethyl 4-ethylpiperazine-1-carbodithioate. 2-((4-Morpholinophenyl) amino)-2-oxoethyl 4-(4-methylbenzyl) piperazine-1-carbodithioate compound was registered as a new substance by CAS (Chemical Abstracts Service) on 03.01.2017 (RN: 2055204-10 -7).

4. 5 new substances with methylpiperazine and dithiocarbamate fragments in their structure have been synthesized, and their physico-chemical properties were identified using IR-, NMR-spectroscopy and MS-spectrometry as2-((4-(4-methylpiperazin-1-yl)phenyl)amino)-2-oxoethyl 4-(4-methylbenzyl) piperazine-1-carbodithioate, 2-((4-(4 - methylpiperazine – 1 - yl)) phenyl)amino)-2-oxoethyl 4-benzylpiperazine-1-carbodithioate, 2-((4-(4-methylpiperazin-1-yl)phenyl)amino)-2-oxoethyl4-(4-methoxybenzyl)piperazine-1- carbodithioate, 2-((4-(4-methylpiperazin-1-yl)phenyl)amino)-2-oxoethyl 4-methylpiperazin-1-carbodithioate and 2-((4-(4-methylpiperazin-1-yl)phenyl) amino)-2-oxoethyl 4-ethylpiperazine-1-carbodithioate. For the first time, a Eurasian Patent was obtained for the synthesis and studying the results of antifungal activity of 2-((4-(4-methylpiperazin-1-yl)phenyl)amino)-2-oxoethyl 4-ethyl piperazine-1-carbodithioate substance (No. 025187, 30.09 .2016 year).
5. The biological activity of the synthesized compounds has been studied and as a result 2-((4-(4-methylpiperazin-1-yl) phenyl) amino)-2-oxoethyl4-(4-methylbenzyl) piperazine-1-carbodithioate, 2-((4-(4-Methylpiperazin-1-yl)phenyl)amino)-2-oxoethyl 4-ethylpiperazine-1-carbodithioate, 2-((4-(piperidin-1-yl) phenyl) amino)-2-oxoethyl4-benzylpiperazine-1-carbodithioate, 2-((4-(piperidin-1-yl) phenyl) amino)-2-oxoethyl4-(4-methoxybenzyl) piperazine-1-carbodithioate, 2-((4-morpholinophenyl) amino)-2-oxoethyl 4-(4-methoxybenzyl)

piperazine-1-carbodithioate and 2-((4-morpholinophenyl)amino)-2-oxoethyl 4-ethylpiperazine-1-carbodithioate substances have been determined to have high antifungal activity.

As a result of in silico studies, important directions of biological activity of the synthesized dithiocarbamate derivatives were determined through the PASS (Prediction of Activity Spectra of Substances) program. As a result, dithiocarbamate derivatives were found to have cytostatic, antianginal, antibacterial, cytochrome P450 inhibitor and other effects. It is relevant and promising to carry out comprehensive research in the direction of the creation of new medicinal substances for the indicated activities.

PRACTICAL RECOMMENDATIONS

1. The development of 3-step optimal conditions (Results section: paragraph 1) for the synthesis of dithiocarbamate derivatives can be successfully applied to the synthesis of other dithiocarbamate derivatives.
2. A comprehensive study of the synthesized dithiocarbamate derivatives for designing new drugs with cytostatic, antianginal, and antibacterial effects is promising.
3. The prognostic studies of the newly synthesized dithiocarbamate derivatives using the PASS program indicate that these compounds are promising in the search for new medical substances to be applied in the treatment of other diseases in the future.

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LİST OF ABBREVİATİONS AND SYMBOLS

AMU	– Azerbaijan Medical University
IR	– Infrared
UB	– Ultraviolet
TLC	– Thin layer chromatography
HPLC-MS	– High-performance liquid chromatography-mass spectrometry
HPLC-QQQ-MS/MS	– High Performance Liquid Chromatography - Triple Quadrupole-Mass Spectrometry/ Mass Spectrometry
LC-MS	– Liquid chromatography mass spectrometry
NMR	– Nuclear magnetic resonance
TMS	– Tetramethylsilane
THF	– Tetrahydrofuran
DMSO	– Dimethylsulfoxide
SH group	– Sulfhydryl group
SRN	– State Registration Number
CAS	– Chemical Abstracts Service
CLSI	– Clinical and Laboratory Standards Institute
FTIR	– Fourier Transform Infrared
PASS	– Prediction of Activity Spectra of Substances
RN	– Registration Number

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