



Synthesis and structure of salts of 2-(((3-mercapto-5-methyl-4H-1,2,4-triazole-4-yl)imino)methyl)benzoic acid

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Modern pharmacy and medicine can offer a substantial number of highly effective synthetic medicines. A great portion of these medicines falls into the group of 1,2,4-triazole derivatives.

1,2,4-Triazoles are distinguished by important pharmacological properties, including antimicrobial, antifungal, anti-inflammatory, hypoglycemic, while also being capable of stimulating the central nervous system.

Undoubtedly, a constant increase of number of publications regarding the synthesis, characterization of biological, chemical, and physical properties of 1,2,4-triazole derivatives by both foreign and Ukrainian researchers promotes the search for promising compounds of this class of heterocyclic compounds globally.

The purpose of our work was to synthesize and characterize structures of salts of 2-(((3-mercapto-5-methyl-4H-1,2,4-triazole-4-yl)imino)methyl)benzoic acid, as potentially bioactive compounds.

Materials and methods. The physical-chemical characterization of the synthesized compounds was conducted according to the guidelines provided by the State Pharmacopoeia of Ukraine. The melting point was determined using MPA100 apparatus. Elemental analysis was held using Elementar Vario L.cube analyzer. ¹H NMR spectra were recorded using Varian Mercury VX-200 spectrometer (1H, 200 MHz). Chromatographic and mass-spectrometric data were obtained using Agilent 1260 Infinity LC system coupled with Agilent 6120 mass spectrometer.

Results and discussion. 2-(((3-Mercapto-5-methyl-4H-1,2,4-triazole-4-yl)imino)methyl)benzoic acid was used as the starting compound for the synthesis of the new structures. In order to afford the desired compounds, the starting material was subjected to salt formation reactions with ammonium hydroxide, sodium and potassium hydroxides, methylamine, ethylamine, dimethylamine, 1-propylamine, 2-propylamine, monoethanolamine, diethylamine, *tert*-butylamine, piperidine, piperazine, morpholine, 2-methylpiperidine, and 4-methylmorpholine whether in alcohol or water solutions.

Conclusions. The chemical experiments resulted in 16 new substances, salts of 2-(((3-mercapto-5-methyl-4H-1,2,4-triazole-4-yl)imino)methyl)benzoic acid. The chemical structure of the synthesized compounds was confirmed using instrumental methods, including ¹H NMR spectroscopy, LC-MS, and elemental analysis. The obtained salts will be used in further pharmacological research.

Синтез і структура солей 2-(((3-меркапто-5-метил-4H-1,2,4-тріазол-4-іл)іміно)метил)бензойної кислоти

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Сучасна фармація та медицина мають у своєму арсеналі багато високоефективних лікарських засобів синтетичного походження. Значна частина таких засобів – похідні 1,2,4-тріазолу.

Сполуки 1,2,4-тріазолу мають важливі фармакологічні властивості: протимікробну, протигрибкову, протизапальну, гіпоглікемічну, є стимуляторами ЦНС.

Безперечно, постійне збільшення кількості публікацій іноземних і вітчизняних авторів щодо методів синтезу, біологічних і фізико-хімічних властивостей похідних 1,2,4-тріазолу спонукає вчених різних країн займатися пошуком перспективних молекул серед цього класу гетероциклів.

Мета роботи – уперше здійснити синтез і підтвердити структуру солей 2-(((3-меркапто-5-метил-4H-1,2,4-тріазол-4-іл)іміно)метил)бензойної кислоти як майбутніх біологічно активних агентів.

Матеріали та методи. Дослідження фізико-хімічних параметрів речовин, які синтезували, виконали за методиками, які наведені в Державній Фармакопеї України. Температуру плавлення встановили за допомогою приладу MPA100. Елементний аналіз речовин здійснили, використовуючи аналізатор Elementar Vario L.cube. ¹H ЯМР-спектри сполук зняті на спектрометрі Varian Mercury VX-200 (1H, 200 MHz). Хромато-мас-спектральні дослідження виконали на газорідному хроматографі Agilent 1260 Infinity з мас-спектрометром Agilent 6120.

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Результати. Як вихідні речовини для синтезу нових структур використали 2-(((3-меркапто-5-метил-4*H*-1,2,4-тріазол-4-іл)іміно)метил)бензойну кислоту. Поставили реакції солеутворення вихідної сполуки з гідроксидом амоніаку, гідроксидами натрію та калію, метиламіном, етиламіном, диметиламіном, 1-пропіламіном, 2-пропіламіном, моноетаноламіном, діетиламіном, трет-бутиламіном, піперидином, піперазином, морфоліном, 2-метилпіперидин, 4-метил морфоліном у спиртових або водних середовищах.

Висновки. У результаті дослідження хімічним шляхом отримали 16 нових речовин, солей 2-(((3-меркапто-5-метил-4*H*-1,2,4-тріазол-4-іл)іміно)метил)бензойної кислоти. Хімічна структура синтезованих сполук підтверджена комплексом сучасних методів аналізу: ¹H ЯМР-спектроскопії, LS/MS та елементного аналізу. Синтезовані солі надалі будуть використані у фармакологічних дослідженнях.

Ключові слова: синтез, 1,2,4-тріазолі, фізико-хімічні властивості.

Актуальні питання фармацевтичної і медичної науки та практики. 2020. Т. 13, № 1(32). С. 17–22

Синтез и структура 2-(((3-меркапто-5-метил-4*H*-1,2,4-тріазол-4-іл)іміно)метил)бензойної кислоти

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Современная фармация и медицина имеют в своем арсенале достаточно много высокоэффективных лекарственных средств синтетического происхождения. Значительная часть таких средств – производные 1,2,4-тріазола.

Соединения 1,2,4-тріазола отличаются важными фармакологическими свойствами: противомикробной, противогрибковой, противовоспалительной, гипогликемической активностью, являются стимуляторами ЦНС.

Бесспорно, постоянный рост количества публикаций иностранных и отечественных авторов относительно методов синтеза, биологических и физико-химических свойств производных 1,2,4-тріазола побуждает ученых разных стран заниматься поиском перспективных молекул среди данного класса гетероциклов.

Цель работы – впервые провести синтез и подтвердить структуру солей 2-(((3-меркапто-5-метил-4*H*-1,2,4-тріазол-4-іл)іміно)метил)бензойної кислоти как будущих биологически активных агентов.

Материалы и методы. Исследование физико-химических параметров синтезированных веществ выполнено согласно методикам, которые приведены в Государственной Фармакопее Украины. Температура плавления установлена с помощью прибора МРА100. Элементный анализ веществ проведен с использованием анализатора Elementar Vario L.cube. ¹H ЯМР-спектры соединений сняты на спектрометре Varian Mercury VX-200 (1H, 200 MHz). Хромато-масс-спектральные исследования проводили на газожидкостном хроматографе Agilent 1260 Infinity с масс-спектрометром Agilent 6120.

Результаты. Как исходное вещество для синтеза новых структур использована 2-(((3-меркапто-5-метил-4*H*-1,2,4-тріазол-4-іл)іміно)метил)бензойная кислота. Поставлены реакции солеобразования исходного соединения с гидроксидом аммония, гидроксидами натрия и калия, метиламином, этиламин, диметиламином, 1-пропиламино, 2-пропиламин, моноэтанолламин, диетиламином, трет-бутиламином, пиперидином, пиперазином, морфоліном, 2-метилпиперидин 4-метил морфоліном в спиртовых или водных средах.

Выводы. В результате исследования химическим путем получены 16 новых веществ, солей 2-(((3-меркапто-5-метил-4*H*-1,2,4-тріазол-4-іл)іміно)метил)бензойної кислоти. Химическая структура синтезированных соединений подтверждена комплексом современных методов анализа: ¹H ЯМР-спектроскопии, LS/MS и элементного анализа. Синтезированные соли будут использованы в дальнейших фармакологических исследованиях.

Ключевые слова: синтез, 1,2,4-тріазол, физико-химические свойства.

Актуальные вопросы фармацевтической и медицинской науки и практики. 2020. Т. 13, № 1(32). С. 17–22

Nowadays pharmaceutical and medical developments have made available numerous highly effective synthetic medicinal products. Many of such molecules contain 1,2,4-triazole moiety [1–4].

As demonstrated by the increasing number of scientific publications regarding the 1,2,4-triazole moiety, this research direction holds significant promise [5–7]. This class of organic compounds has sparked interest among researchers working in the fields of medicine, pharmaceuticals, and veterinary practice [2,6], while it is attractive for specialists working in agriculture [8]. Apart from that, 1,2,4-triazole derivatives are used as dyes, antioxidant agents, some of them have applications as corrosion inhibitors, and others are utilized as pesticides [4].

1,2,4-Triazole derivatives possess a range of useful pharmacological properties, such as antimicrobial, antifungal, anti-inflammatory, hypoglycemic, and act as central nervous system (CNS) stimulants [2,4,9].

The number of publications devoted to synthesis as well as biological and physical-chemical characterization of 1,2,4-triazoles by foreign and Ukrainian researchers is growing, which promotes the search for promising heterocyclic compounds, including 1,2,4-triazoles [10–13].

Aim

The purpose of our work was to synthesize and characterize structures of salts of 2-(((3-mercapto-5-methyl-4*H*-1,2,4-triazole-4-yl)imino)methyl)benzoic acid, as potentially bioactive compounds.

Materials and methods

The chemical reagents were obtained from “Ukrorgsyntez Ltd (UOS)” with all quality documentation. The physical-chemical characterization of the synthesized compounds was conducted

according to the guidelines provided by the State Pharmacopoeia of Ukraine (SPU). The melting point was determined using MPA100 apparatus (Automated melting point system, USA). Elemental analysis was held using Elementar Vario L.cube analyzer (Germany). ^1H NMR spectra were recorded using Varian Mercury VX-200 spectrometer (1H, 200 MHz) (USA) with dimethyl sulfoxide- d_6 as the solvent and tetramethylsilane as the internal standard; spectra were interpreted using SpinWorks software. Chromatographic and mass-spectrometric data were obtained using Agilent 1260 Infinity LC system (USA) coupled with Agilent 6120 mass spectrometer equipped with an electrospray ionization source (ESI) [14–16].

Results

As the starting material 2-(((3-mercapto-5-methyl-4H-1,2,4-triazole-4-yl)imino)methyl)benzoic acid **1.1** (Fig. 1) was used. This compound was synthesized by us [3].

Compound **1.1** was reacted with ammonium hydroxide, sodium and potassium hydroxides, methylamine, ethylamine, dimethylamine, *n*-propylamine, *i*-propylamine, monoethanolamine, diethylamine, *tert*-butylamine, piperidine, piperazine, morpholine, 2-methylpiperidine, and 4-methylmorpholine whether in alcohol or water solutions to obtain the corresponding salts **2.1–2.16** (Fig. 2).

Experimental

Ammonium salt of 2-(((3-mercapto-5-methyl-4H-1,2,4-triazole-4-yl)imino)methyl)benzoic acid (2.1). 40 mL of 25 % ammonia in water was added to 0.01 moles of 2-(((3-mercapto-5-methyl-4H-1,2,4-triazole-4-yl)imino)methyl)benzoic acid (**1.1**), then the mixture was stirred until the substance was dissolved. The obtained solution was filtered and evaporated. The compound was obtained in a form of green crystals. For analytical purposes, the compound was recrystallized from *n*-butanol.

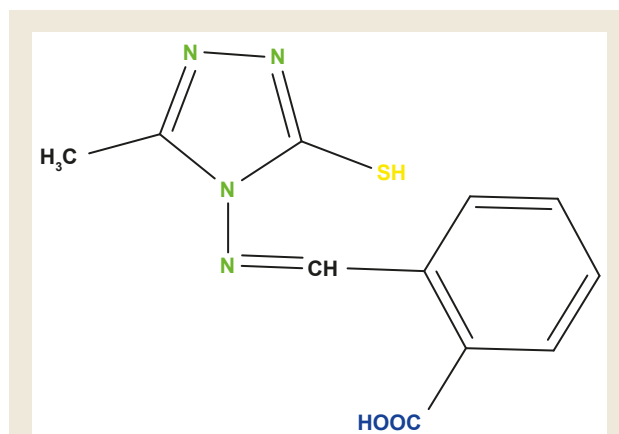


Fig. 1. Structure of 2-(((3-mercapto-5-methyl-4H-1,2,4-triazole-4-yl)imino)methyl)benzoic acid (**1.1**) used as the starting material.

Sodium and potassium salts of 2-(((3-mercapto-5-methyl-4H-1,2,4-triazole-4-yl)imino)methyl)benzoic acid (2.2, 2.3). 0.01 moles of 2-(((3-mercapto-5-methyl-4H-1,2,4-triazole-4-yl)imino)methyl)benzoic acid (**1.1**) were dissolved in 50 mL of water, added 0.01 moles of whether sodium hydroxide or potassium hydroxide. The mixture was stirred with heating until substance dissolution. The obtained solutions were filtered and evaporated. For analytical purposes, the compounds were recrystallized from *n*-butanol (**2.2**) and methanol (**2.3**).

Methylammonium, ethylammonium, dimethylammonium, *n*-propylammonium, *i*-propylammonium, monoethanolammonium, diethylammonium, *tert*-butylammonium, piperidinium, piperazinium, morpholinium, 2-methylpiperidinium, and 4-methylmorpholinium salts of 2-(((3-mercapto-5-methyl-4H-1,2,4-triazole-4-yl)imino)methyl)benzoic acid (2.4–2.16). 0.01 moles of 2-(((3-mercapto-5-methyl-4H-1,2,4-triazole-4-yl)imino)methyl)benzoic acid (**1.1**) were dissolved in 30 mL of water *i*-propanol added 0.01 moles of methylamine, ethylamine, dimethylamine, *n*-propylamine, *i*-propyl-

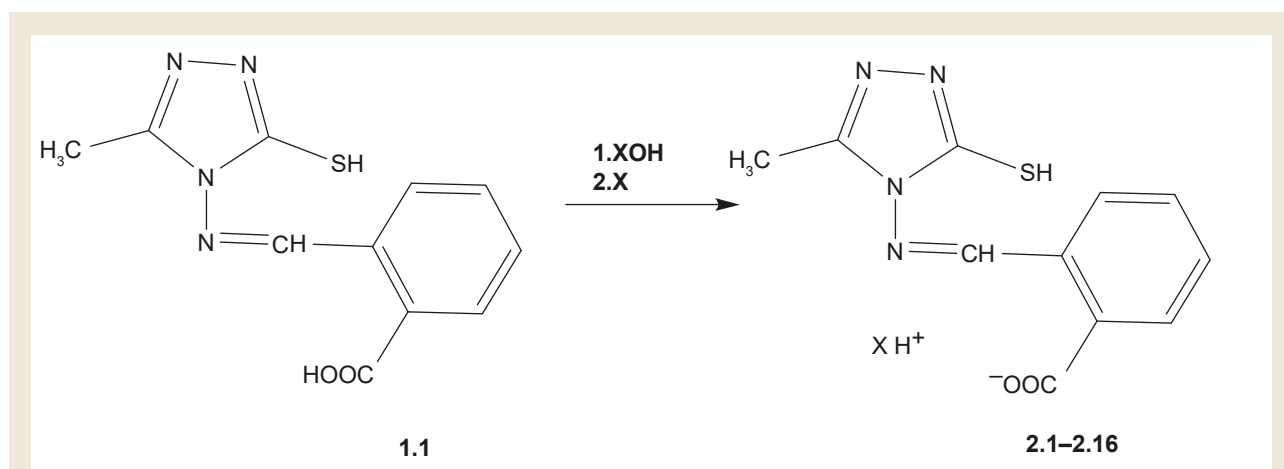
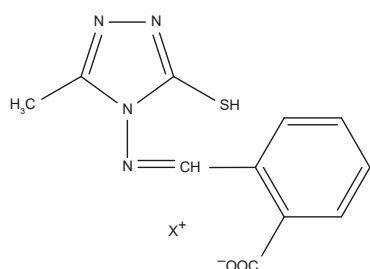


Fig. 2. Scheme of the synthesis of salts of 2-(((3-mercapto-5-methyl-4H-1,2,4-triazole-4-yl)imino)methyl)benzoic acid (**2.1–2.16**).

XOH: ammonium hydroxide, sodium hydroxide, potassium hydroxide; **X:** methylamine, ethylamine, dimethylamine, *n*-propylamine, *i*-propylamine, monoethanolamine, diethylamine, *tert*-butylamine, piperidine, piperazine, morpholine, 2-methylpiperidine, and 4-methylmorpholine.

Table 1. Properties and elemental composition of the synthesized compounds

No.	Kat ⁺	M.pt., °C	Chemical formula	Yield, %	Found %, Calculated %			
					C	H	N	S
2.1	NH ₄	78–80	C ₁₁ H ₁₃ N ₅ O ₂ S	82	47.30 47.18	4.69 4.68	25.07 25.05	11.48 4.51
2.2	Na	266–268	C ₁₁ H ₉ N ₄ NaO ₂ S	84	46.48 46.57	3.19 3.18	19.71 19.77	11.28 11.30
2.3	K	238–240	C ₁₁ H ₉ KN ₄ O ₂ S	85	43.98 44.05	3.02 3.03	18.65 18.69	10.67 10.67
2.4	CH ₃ NH ₃	147–149	C ₁₂ H ₁₅ N ₅ O ₂ S	82	49.30 49.36	4.83 4.82	23.96 23.91	10.97 10.99
2.5	C ₂ H ₅ NH ₃	85–87	C ₁₃ H ₁₇ N ₅ O ₂ S	80	50.80 50.83	5.57 5.57	22.78 22.80	10.43 10.44
2.6	(CH ₃) ₂ NH ₂	148–150	C ₁₃ H ₁₇ N ₅ O ₂ S	75	50.97 50.92	5.26 5.25	22.86 22.82	10.47 10.46
2.7	CH ₃ (CH ₂) ₂ NH ₃	155–157	C ₁₄ H ₁₉ N ₅ O ₂ S	85	52.32 52.44	5.96 5.97	21.79 21.84	9.98 9.99
2.8	(CH ₃) ₂ CHNH ₃	158–160	C ₁₄ H ₁₉ N ₅ O ₂ S	73	60.30 60.17	4.26 4.25	18.51 18.46	8.47 8.49
2.9	HO-(CH ₂) ₂ -NH ₃	172–174	C ₁₃ H ₁₇ N ₅ O ₃ S	77	48.28 48.26	5.30 5.29	21.66 21.67	9.92 9.93
2.10	(CH ₃ CH ₂) ₂ NH ₂	155–157	C ₁₅ H ₂₁ N ₅ O ₂ S	75	53.71 53.67	6.31 6.29	20.88 20.83	9.56 9.59
2.11	(CH ₃) ₃ CNH ₃	171–173	C ₁₄ H ₁₉ N ₅ O ₂ S	80	52.82 54.71	5.07 5.08	22.00 22.04	10.07 10.04
2.12	C ₅ H ₁₁ NH ₂	156–158	C ₁₆ H ₂₁ N ₅ O ₂ S	83	55.47 55.41	5.82 5.83	20.22 20.19	9.26 9.25
2.13	1/2 C ₄ H ₁₀ N ₂ H ₂	130–132	C ₂₆ H ₃₀ N ₁₀ O ₄ S ₂	81	52.16 52.21	4.56 4.96	24.33 24.32	9.28 9.30
2.14	C ₄ H ₉ ONH	163–165	C ₁₅ H ₁₉ N ₅ O ₃ S	82	51.56 51.60	5.48 5.47	20.04 20.06	9.18 9.16
2.15	C ₅ H ₁₀ NH-2-CH ₃	139–141	C ₁₇ H ₂₃ N ₅ O ₂ S	88	57.45 57.42	4.82 4.81	19.70 19.75	9.02 9.03
2.16	C ₄ H ₉ ONH-4-CH ₃	163–165	C ₁₆ H ₂₁ N ₅ O ₃ S	91	54.38 54.24	5.64 5.64	18.65 18.61	8.34 8.34

amine, monoethanolamine, diethylamine, *tert*-butylamine, piperidine, piperazine, morpholine, 2-methylpiperidine, or 4-methylmorpholine (0.005 mol in case of piperazine). The mixture was heated until substance dissolution. The obtained solutions were filtered and left at room temperature for 48 hours, evaporated. Compounds were (2.4, 2.6, 2.8, 2.10, 2.15, 2.16), green (2.7, 2.14), brown (2.5, 2.9, 2.12), and red (2.11, 2.13). For analytical purposes, the compounds were

recrystallized *i*-propanol (2.4, 2.5, 2.7-2.10, 2.12-2.16) and *n*-butanol (2.6, 2.11).

Structure of the synthesized salts of 2-(((3-mercapto-5-methyl-4*H*-1,2,4-triazole-4-yl)imino)methyl)benzoic acid was confirmed by elemental analysis (Table 1), ¹H NMR spectroscopy, and LC-MS (Table 2). LC-MS chromatograms demonstrate the presence of individual peaks corresponding to the synthesized compounds (Table 2).

Table 2. Chemical shifts of protons in ¹H NMR spectra and LC-MS data for the synthesized compounds

No.	¹ H NMR DMSO-d ₆ , δ ppm	LC-MS, m/z [M+1]
2.1	δ 2.25 (3H, s), 7.43 (1H, ddd, J = 7.8, 7.3, 1.3 Hz), 7.76-7.86 (2H, 7.81 (ddd, J = 8.0, 7.3, 1.5 Hz), 7.81 (ddd, J = 8.0, 1.3, 0.5 Hz)), 8.22 (1H, ddd, J = 7.8, 1.5, 0.5 Hz), 9.32 (1H, s).	280.08
2.2	δ 2.20 (3H, s), 7.40 (1H, ddd, J = 7.8, 7.4, 1.3 Hz), 7.70-7.82 (2H, 7.77 (ddd, J = 8.0, 7.4, 1.4 Hz), 7.73 (ddd, J = 8.0, 1.3, 0.5 Hz)), 8.12 (1H, ddd, J = 7.8, 1.4, 0.5 Hz), 9.25 (1H, s).	285.27
2.3	δ 2.25 (3H, s), 7.43 (1H, ddd, J = 7.8, 7.3, 1.3 Hz), 7.76-7.86 (2H, 7.81 (ddd, J = 8.0, 7.3, 1.5 Hz), 7.80 (ddd, J = 8.0, 1.3, 0.5 Hz)), 8.23 (1H, ddd, J = 7.8, 1.5, 0.5 Hz), 9.32 (1H, s).	301.01
2.4	δ 2.25 (3H, s), 2.48 (3H, s), 7.42 (1H, ddd, J = 7.8, 7.4, 1.3 Hz), 7.71-7.83 (2H, 7.78 (ddd, J = 8.0, 7.4, 1.4 Hz), 7.74 (ddd, J = 8.0, 1.3, 0.5 Hz)), 8.17 (1H, ddd, J = 7.8, 1.4, 0.5 Hz), 9.31 (1H, s).	294.09
2.5	δ 1.25 (3H, t, J = 6.2 Hz), 2.28 (3H, s), 2.88 (2H, q, J = 6.2 Hz), 7.44 (1H, ddd, J = 7.5, 7.3, 1.3 Hz), 7.71-7.85 (2H, 7.79 (ddd, J = 8.1, 7.2, 1.3 Hz), 7.79 (ddd, J = 8.1, 1.2, 0.6 Hz)), 8.19 (1H, ddd, J = 7.6, 1.2, 0.4 Hz), 9.44 (1H, s).	308.11
2.6	δ 2.24 (3H, s), 2.60 (6H, s), 7.41 (1H, ddd, J = 7.6, 7.2, 1.6 Hz), 7.70-7.89 (2H, 7.74 (ddd, J = 8.1, 7.2, 1.1 Hz), 7.69 (ddd, J = 8.1, 1.2, 0.6 Hz)), 8.19 (1H, ddd, J = 7.6, 1.2, 0.6 Hz), 9.39 (1H, s).	308.11
2.7	δ 0.90 (3H, t, J = 7.3 Hz), 1.63 (2H, qt, J = 7.3, 6.5 Hz), 2.25 (3H, s), 2.95 (2H, t, J = 6.5 Hz), 7.42 (1H, ddd, J = 7.8, 7.4, 1.3 Hz), 7.71-7.83 (2H, 7.78 (ddd, J = 8.0, 7.4, 1.4 Hz), 7.74 (ddd, J = 8.0, 1.3, 0.5 Hz)), 8.17 (1H, ddd, J = 7.8, 1.4, 0.5 Hz), 9.31 (1H, s).	322.13
2.8	δ 1.25 (6H, d, J = 6.2 Hz), 2.21 (3H, s), 3.40 (1H, sept, J = 6.2 Hz), 7.46 (1H, ddd, J = 7.5, 7.3, 1.2 Hz), 7.78-7.86 (2H, 7.79 (ddd, J = 8.0, 7.3, 1.2 Hz), 7.75 (ddd, J = 8.0, 1.2, 0.6 Hz)), 8.19 (1H, ddd, J = 7.9, 1.4, 0.6 Hz), 9.35 (1H, s).	322.13
2.9	δ 2.21 (3H, s), 3.15 (2H, t, J = 5.5 Hz), 3.54 (2H, t, J = 5.5 Hz), 7.39 (1H, ddd, J = 7.6, 7.5, 1.2 Hz), 7.69-7.80 (2H, 7.75 (ddd, J = 8.0, 7.3, 1.2 Hz), 7.70 (ddd, J = 8.0, 1.2, 0.4 Hz)), 8.14 (1H, ddd, J = 7.6, 1.2, 0.3 Hz), 9.28 (1H, s).	324.11
2.10	δ 1.30 (6H, t, J = 6.2 Hz), 2.26 (3H, s), 3.06 (4H, q, J = 6.2 Hz), 7.44 (1H, ddd, J = 7.6, 7.5, 1.4 Hz), 7.76-7.85 (2H, 7.79 (ddd, J = 8.0, 7.3, 1.3 Hz), 7.76 (ddd, J = 8.0, 1.4, 0.6 Hz)), 8.19 (1H, ddd, J = 7.9, 1.5, 0.5 Hz), 9.35 (1H, s).	336.14
2.11	δ 1.21 (9H, s), 2.21 (3H, s), 7.44 (1H, ddd, J = 7.9, 7.3, 1.2 Hz), 7.74-7.863 (2H, 7.79 (ddd, J = 8.0, 7.1, 1.1 Hz), 7.70 (ddd, J = 8.0, 1.4, 0.6 Hz)), 8.19 (1H, ddd, J = 7.8, 1.3, 0.4 Hz), 9.36 (1H, s).	322.13
2.12	δ 1.53 (2H, dtt, J = 12.8, 3.3, 2.4 Hz), 1.71 (4H, dtdd, J = 13.5, 10.2, 3.3, 2.6 Hz), 2.30 (3H, s), 3.09 (4H, ddd, J = 14.6, 10.3, 2.7 Hz), 7.47 (1H, ddd, J = 7.5, 7.2, 1.1 Hz), 7.77-7.87 (2H, 7.82 (ddd, J = 8.0, 7.2, 1.2 Hz), 7.79 (ddd, J = 8.0, 1.1, 0.4 Hz)), 8.21 (1H, ddd, J = 7.4, 1.1, 0.5 Hz), 9.38 (1H, s).	348.14
2.13	δ 1.52 (2H, dtt, J = 12.8, 3.3, 2.4 Hz), 1.75 (4H, dtdd, J = 13.5, 10.2, 3.3, 2.6 Hz), 2.33 (6H, s), 3.09 (4H, ddd, J = 14.6, 10.3, 2.7 Hz), 7.35 (2H, ddd, J = 7.4, 7.2, 1.1 Hz), 7.66-7.71 (4H, 7.80 (ddd, J = 8.0, 7.1, 1.2 Hz), 7.71 (ddd, J = 8.0, 1.2, 0.6 Hz)), 8.22 (2H, ddd, J = 7.9, 1.3, 0.5 Hz), 9.25 (2H, s).	611.19
2.14	δ 2.23 (3H, s), 3.43 (4H, ddd, J = 15.5, 10.2, 2.5 Hz), 3.77 (4H, ddd, J = 16.2, 10.2, 2.5 Hz), 7.41 (1H, ddd, J = 7.8, 7.4, 1.3 Hz), 7.70-7.82 (2H, 7.75 (ddd, J = 8.0, 7.2, 1.3 Hz), 7.78 (ddd, J = 8.0, 1.4, 0.6 Hz)), 8.21 (1H, ddd, J = 7.9, 1.3, 0.5 Hz), 9.44 (1H, s).	350.12
2.15	δ 1.29 (3H, d, J = 6.4 Hz), 1.45 (1H, dtt, J = 12.9, 3.3, 2.4 Hz), 1.53-1.76 (5H, 1.66 (dtd, J = 12.9, 3.3, 2.7, 2.4 Hz), 1.64 (dtt, J = 12.9, 10.2, 3.3 Hz), 1.62 (dtd, J = 12.9, 10.2, 3.3 Hz), 1.68 (dtd, J = 12.9, 3.3, 2.7, 2.4 Hz), 1.66 (dtd, J = 13.1, 10.2, 3.3, 2.6 Hz)), 2.36 (3H, s), 3.00 (1H, ddd, J = 14.5, 10.3, 2.7 Hz), 3.20 (1H, ddd, J = 14.5, 2.9, 2.6 Hz), 3.67 (1H, dqd, J = 10.3, 6.4, 2.7 Hz), 7.51 (1H, ddd, J = 6.8, 5.4, 1.1 Hz), 7.79-7.88 (2H, 7.80 (ddd, J = 8.0, 7.2, 1.2 Hz), 7.80 (ddd, J = 8.1, 1.2, 0.6 Hz)), 8.25 (1H, ddd, J = 7.9, 1.3, 0.4 Hz), 9.21 (1H, s).	362.16
2.16	δ 2.21 (3H, s), 2.97 (3H, s), 3.23-3.38 (4H, 3.31 (ddd, J = 15.5, 3.1, 2.5 Hz), 3.31 (ddd, J = 15.5, 10.2, 2.5 Hz)), 3.75-3.93 (4H, 3.82 (ddd, J = 14.4, 10.2, 2.5 Hz), 3.88 (ddd, J = 14.4, 3.1, 2.5 Hz)), 7.46 (1H, ddd, J = 7.6, 7.5, 1.4 Hz), 7.73-7.88 (2H, 7.79 (ddd, J = 8.0, 7.3, 1.5 Hz), 7.76 (ddd, J = 8.0, 1.2, 0.4 Hz)), 8.19 (1H, ddd, J = 7.9, 1.5, 0.5 Hz), 9.35 (1H, s).	364.14

Discussion

The elemental analysis confirmed the composition of the synthesized compounds (Table 1), ¹H NMR data indicated that theoretical structures are in agreement with the experimental results. ¹H NMR spectrum of potassium 2-(((3-mercapto-5-methyl-4H-1,2,4-triazole-4-yl)imino)methyl)benzoate (**2.3**) was characterized by a chemical shift in a strong field appearing as a triple-proton singlet of the methyl group attached to the ring of 1,2,4-triazole at 2.25 ppm. Protons of the phenyl substituent appear as two multiplets and one doublet at 7.43 ppm, 7.76–7.86 ppm, and 8.23 ppm, while

proton of imino group resonates as a single-proton singlet at 9.32 ppm.

Conclusions

Sixteen new compounds were obtained during the chemical synthesis, specifically, the salts of 2-(((3-mercapto-5-methyl-4H-1,2,4-triazole-4-yl)imino)methyl)benzoic acid. Structures of the synthesized compounds were confirmed by a set of instrumental methods, including ¹H NMR spectroscopy, LC-MS, and elemental analysis. For the obtained salts the pharmacological researches are planned to be evaluated

(antioxidant, antihypoxic, antimicrobial, cardio- and hepatoprotective actions).

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