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**ORIGINAL ARTICLE / ÖZGÜN MAKALE**

# SYNTHESIS AND ANTIRADICAL ACTIVITY OF ALKYL DERIVATIVES OF 5-(5-METHYL-1*H*-PYRAZOL-3-YL)-4-PHENYL-4*H*-1,2,4-TRIAZOLE-3-THIOL

## *5-(5-METİL-1H-PİRAZOL-3-İL)-4-FENİL-4H-1,2,4-TRİAZOL-3-TİYOL ALKİL TÜREVLERİNİN SENTEZ VE ANTİRADİKAL AKTİVİTELERİ*

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## ABSTRACT

**Objective:** *The aim of the work was to develop effective methods for the synthesis of biologically active heterocyclic systems containing 1,2,4-triazole and pyrazole.*

**Material and Method:** *Chemical structures of synthesized compounds were characterized with elemental analysis, IR, $^{1}$H-NMR, LC-MS techniques. The antiradical activity was evaluated using the 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging activity assay.*

**Result and Discussion:** *Developed optimal methods of obtaining of alkylderivatives of 5-(5-methyl-1H-pyrazol-3-yl)-4-phenyl-4H-1,2,4-triazole-3-thiol. A number of synthesized compounds showed moderate antiradical activity.*

**Keywords:** *antiradical activity, pyrazole, synthesis,1,2,4-triazoles*

## ÖZ

**Amaç:** *Çalışmanın amacı biyolojik olarak aktif 1,2,4-triazol ve pirazol içeren heterosiklik sistemlerin sentezi için etkili yöntemler geliştirmektir.*

**Gereç ve Yöntem:** *Sentezlenen bileşiklerin kimyasal yapıları elemantal analiz, IR, $^{1}$H-NMR, LC-MS teknikleri ile karakterize edildi. Antiradikal aktivite 2,2-difenil-1-pikrilhidrazil (DPPH) radikal süpürücü aktivite deneyi kullanılarak değerlendirildi.*

**Sonuç ve Tartışma:** *5-(5-metil-1H-pirazol-3-il)-4-fenil-4H-1,2,4-triazol-3-tiyol alkil türevlerinin elde edilmesi için optimum yöntemler geliştirildi. Bir dizi sentezlenmiş bileşik, orta derecede antiradikal aktivite göstermiştir.*

**Anahtar Kelimeler:** *antiradikal aktivite, pirazol, sentez, 1,2,4-triazol*

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## INTRODUCTION

Chemistry of pyrazole and 1,2,4-triazole derivatives is of considerable interest in scientific circles [1, 2, 3]. The attractiveness of these heterocycles is associated with great opportunities in their structural modification and their practical use as medicines [1, 3, 4]. Currently, there are known methods of synthesis that allow you to combine these two synthons into one molecule. Despite this, some aspects of the transformations of these molecules and the study of their properties are not fully exhausted. Thus, the search for optimal ways of synthesis of substances that combine in their structure fragments of 1,2,4-triazole and pyrazole, as well as the study of the properties of their derivatives is relevant and practically significant.

The aim of the present work was to synthesize of alkylderivatives of 5-(5-methyl-1*H*-pyrazol-3-yl)-4-phenyl-4*H*-1,2,4-triazole-3-thiol and to study the antioxidant activity of the obtained substances.

## MATERIAL AND METHOD

*Chemistry*

During the study as synthones for the preparation of a new series of compounds, pyrazole was selected. It is important to note that due to the variety and power of pharmacological effects that appear, this structure is a worthy place among heterocyclic compounds [1, 3, 5 - 7, 14]. 5-(5-Methyl-1*H*-pyrazole-3-yl)-4-phenyl-4*H*-1,2,4-triazole-3-thiol was obtained from diethyloxalate, acetone and sodium methanoate through a series of successive stages [8].

The study of physico-chemical properties of the obtained compounds was carried out using methods listed in the State Pharmacopoeia of Ukraine. Melting points were determined in open capillary tubes in a "Stanford Research Systems Melting Point Apparatus 100" (SRS, USA). The elemental analysis (C, H, N) were performed using the "Elementar vario EL cube" analyzer (Elementar Analysensysteme, Germany). IR spectra (4000 – 400 $cm^{-1}$) were taken off the module ALPHA-T of Bruker ALPHA FT-IR spectrometer (Bruker optics, Germany). $^1H$ NMR spectra (400 MHz) were recorded at "Varian-Mercury 400" spectrometer with $SiMe_4$ as internal standard in DMSO-$d_6$ (dimethyl sulfoxide-$d_6$) solution. Chromatography-mass spectral studies were conducted on the instrument "Agilent 1260 Infinity HPLC" equipped with a mass spectrometer "Agilent 6120" (method of ionization – electrospray (ESI)).

***Methyl 5-methylpyrazole-3-carboxylate (4).*** Hydrazine hydrate (10 g, 0.2 mol) in ethanol (25 ml) is gradually added with stirring and cooling to a solution of methyl 2,4-dioxopentanoate (**3**) (0.2 mol) in alcohol. After 1 hour of heating, the alcohol was discarded. The resulting compound (99%) was recrystallized from aqueous ethanol in the form of a needle crystal with a melting point of 82 °C (**Fig. 1**).

***5-Methylpyrazole-3-carbohydrazide (5).*** Methyl 5-methylpyrazole-3-carboxylate (58.4 g) and hydrazine hydrate (25 g) were heated for 8 hours. The reaction product was obtained in the form of prismatic crystals, which was recrystallized from water. The precipitate was dried at 100 °C (**Fig. 1**). Yield: 90%; m. p.: 153 – 154 °C; IR (ν, $cm^{-1}$): 3409-3235 (NH, $NH_2$), 1627 (C=O); $^1$H NMR ($\delta$, ppm): 2.23 (s, 3H, $CH_3$), 4.34 (s, 2H, $NH_2$), 6.38 (s, 1H, pyrazole, =CH-), 9.18 (s, 1H, CONH), 13.52 (s, 1H, pyrazole, NH). Anal. calcd. (%) for $C_5H_8N_4O$: C, 42.85; H, 5.75; N, 39.98. Found: C, 42.74; H, 4.82; N, 40.08.

***2-(5-Methyl-1H-pyrazole-3-carbonyl)-N-phenylhydrazine-1-carbothioamide (6).*** 0.05 mol of 5-methylpyrazole-3-carbohydrazide, 150 ml of dioxane and 60 ml of water are heated to dissolve the starting material. To the solution obtained add an equivalent amount of phenyl isothiocyanate, boil for 1 hour, cool, add 100 ml of water, the precipitate is filtered off, washed with water, propan-2-ol and crystallized from DMF (**Fig. 1**). Yield: 71%; m. p.: 263 - 265 ºC; IR (ν, $cm^{-1}$): 3231 (NH), 1649 (C=O); $^1$H NMR ($\delta$, ppm): 2.26 (s, 3H, $CH_3$), 6.45 (s, 1H, pyrazole, =CH-), 7.37-7.41 (dd, $J$ = 6.65, 2.86 Hz, 2H, $C_6H_5$), 7.53-7.60 (m, 3H, $C_6H_5$), 8.54 (s, 1H, CONH), 9.63 (s, 1H, NHCS), 13.28 (s, pyrazole, NH). Anal. calcd. (%) for $C_{12}H_{13}N_5OS$: C, 52.35; H, 4.76; N, 25.44; S, 11.64. Found: C, 52.47; H, 4.75; N, 25.37; S, 11.67.

***5-(5-Methyl-1H-pyrazole-3-yl)-4-phenyl-4H-1,2,4-triazole-3-thiol (7).*** A mixture of 0.01 mol of 2-(5-methyl-1*H*-pyrazole-3-carbonyl)-*N*-hydrazinocarbothioamide, 0.011 mol of sodium hydroxide and 50 ml of water is heated to reflux for 2 hours, cooled and add 2 ml of concentrated acetic acid. The resulting precipitate is filtered off, washed with water. Recrystallized from DMF (**Fig. 1**). Yield: 84 %; m. p.: 274 - 276 ºC; IR (ν, $cm^{-1}$):

***Alkylation.*** 0.05 mol 5-(5-methyl-1*H*-pyrazole-3-yl)-4-phenyl-4*H*-1,2,4-triazole-3-thiol (**7**) is added to a solution of 0.05 mol of sodium hydroxide in 30-40 ml of water, reaching complete dissolution. Subsequently, a solution of 0.055 mol of the alkylating part in methanol or propan-2-ol is added (**Fig. 1**). The obtained precipitate was filtered, washed by water, dried and recrystallized with ethanol to give white crystalline compounds (**8.1-8.10**).

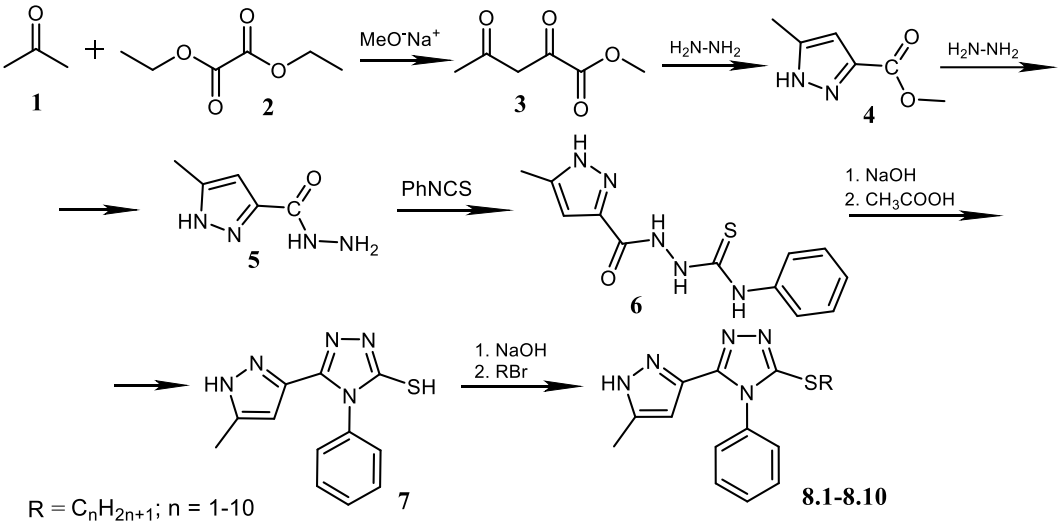


**Figure 1.** The synthetic route of title compounds

*Antiradical activity*

To assess the antiradical activity of the synthesized samples in the test with radical DPPH, a 0.1 *mM* DPPH solution in methanol was used [9, 12, 13]. To detect compounds with a marked antiradical activity of up to 2 ml of 0.1 *mM* DPPH solution in methanol, 2 ml of 2 *mM* solution was added to the DMSO of the test compound. Incubated for 30 minutes at 25 °C and optical density ($A_D$) was determined. In parallel, the optical density of the solution was determined, which consisted of 2 ml of 0.2 *mM* solution of the test compound in DMSO and 2 ml of 0.1 *mM* DPPH solution. The optical density of 2 ml of 0.1 mM methanol DPPH solution in DMSO was also determined. Ascorbic acid was used as a comparison drug. Antiradical activity was calculated by the formula:

$$\text{APA\%} = \frac{A_{DPPH} - A_D}{A_{DPPH}}$$

Weighing of the synthesized compounds and reagents was performed using electronic scales "ANG200" (Axis, Gdansk, Poland). The optical density was measured using a spectrophotometer "ULAB108UV" (Ulab, Shanghai, China).

## RESULT AND DISCUSSION

The reaction route for the synthesis of the newly synthesized compounds has been described in Figure 1.

**3-(5-Methyl-1*H*-pyrazol-3-yl)-5-(methylthio)-4-phenyl-4*H*-1,2,4-triazole (8.1).** Yield: 69 %; m. p.: 220 – 221 °C; IR (ν, $cm^{-1}$): 3229 (NH), 1607 (C=N); $^1$H NMR (*δ*, ppm): 2.32 (s, 3H, $CH_3$), 2.75 (s, 3H, $CH_3$), 6.46 (s, 1H, pyrazole, =CH-), 7.33-7.36 (dd, *J* = 6.68, 2.89 Hz, 2H, $C_6H_5$), 7.48-7.54 (m, 3H, $C_6H_5$), 11.73 (s, 1H, pyrazole, NH). Anal. calcd. (%) for $C_{13}H_{13}N_5S$: C, 57.54; H, 4.83; N, 25.81; S, 11.82. Found: C, 57.39; H, 4.82; N, 25.74; S, 11.85. ESI-MS: m/z= 272 [M+1], 274 [M+3].

**3-(Ethylthio)-5-(5-methyl-1*H*-pyrazol-3-yl)-4-phenyl-4*H*-1,2,4-triazole (8.2).** Yield: 82 %; m. p.: 212 – 214 °C; IR (ν, $cm^{-1}$): 3233 (NH), 1597 (C=N); $^1$H NMR (*δ*, ppm): 1.40-1.42 (t, *J* = 5.1 Hz, 3H, S-$CH_2$-C<u>*H*</u>$_3$), 2.35 (s, 3H, $CH_3$), 3.19-3.21 (m, *J* = 4.8 Hz, 2H, S-C<u>*H*</u>$_2$-$CH_3$), 6.45 (s, 1H, pyrazole, =CH-), 7.33-7.37 (dd, *J* = 6.64, 2.86 Hz, 2H, $C_6H_5$), 7.48-7.54 (m, 3H, $C_6H_5$), 11.71 (s, 1H, pyrazole, NH). Anal. calcd. (%) for $C_{14}H_{15}N_5S$: C, 58.93; H, 5.30; N, 24.54; S, 11.23. Found: C, 58.77; H, 5.29; N, 24.61; S, 11.20. ESI-MS: m/z= 286 [M+1], 288 [M+3].

**3-(5-Methyl-1*H*-pyrazol-3-yl)-4-phenyl-5-(propylthio)-4*H*-1,2,4-triazole (8.3).** Yield: 74 %; m. p.: 257 – 259 °C; IR (ν, $cm^{-1}$): 3219 (NH), 1615 (C=N); $^1$H NMR (*δ*, ppm): 1.07-1.09 (t, *J* = 5.3 Hz,3H, S-$(CH_2)_2$-C<u>*H*</u>$_3$), 1.73-1.76 (m, *J* = 8.2, 5.4 Hz, 2H, S-$CH_2$-C<u>*H*</u>$_2$-$CH_3$), 2.32 (s, 3H, $CH_3$), 3.14-3.17 (t, 2H, S-C<u>*H*</u>$_2$-$CH_2$-$CH_3$), 6.45 (s, 1H, pyrazole, =CH-), 7.33-7.37 (dd, *J* = 6.45, 2.71 Hz, 2H, $C_6H_5$), 7.47-7.54 (m, 3H, $C_6H_5$), 11.74 (s, 1H, pyrazole, NH). Anal. calcd. (%) for $C_{15}H_{17}N_5S$: C, 60.18; H, 5.72; N, 23.39; S, 10.71. Found: C, 60.03; H, 5.71; N, 23.45; S, 10.74. ESI- ESI-MS: m/z= 300 [M+1], 302 [M+3].

**3-(Butylthio)-5-(5-methyl-1*H*-pyrazol-3-yl)-4-phenyl-4*H*-1,2,4-triazole (8.4).** Yield: 68 %; m. p.: 197 - 199 °C; IR (ν, $cm^{-1}$): 3225 (NH), 1592 (C=N); $^1$H NMR (*δ*, ppm): 0.93-0.95 (t, 3H, S-$(CH_2)_3$-C<u>*H*</u>$_3$), 1.36-1.39 (m, 2H, S-$(CH_2)_2$-C<u>*H*</u>$_2$-$CH_3$), 1.67-1.70 (m, *J* = 8.16, 7.27 Hz, 2H, S-$CH_2$-C<u>*H*</u>$_2$-$CH_2$-$CH_3$), 2.32 (s, 3H, $CH_3$), 3.16-3.19 (t, 2H, S-C<u>*H*</u>$_2$-$(CH_2)_2$-$CH_3$), 6.46 (s, 1H, pyrazole, =CH-), 7.33-7.37 (dd, *J* = 6.36, 2.83 Hz, 2H, $C_6H_5$), 7.46-7.53 (m, 3H, $C_6H_5$),11.75 (s, 1H, pyrazole, NH). Anal. calcd. (%) for $C_{16}H_{19}N_5S$: C, 61.32; H, 6.11; N, 22.35; S, 10.23. Found: C, 61.48; H, 6.12; N, 22.31; S, 10.21. ESI-MS: m/z= 314 [M+1], 316 [M+3].

**3-(5-Methyl-1*H*-pyrazol-3-yl)-5-(pentylthio)-4-phenyl-4*H*-1,2,4-triazole (8.5).** Yield: 77 %; m. p.: 168 – 169 °C; IR (ν, $cm^{-1}$): 3220 (NH), 1611 (C=N); $^1$H NMR (*δ*, ppm): 0.88-0.92 (t, 3H, S-$(CH_2)_4$-C<u>*H*</u>$_3$), 1.36-1.41 (m, 4H, S-$(CH_2)_2$-$(C$<u>*H*</u>$_2)_2$-$CH_3$), 1.66-1.69 (m, *J* = 7.92, 7.25 Hz, 2H, S-$CH_2$-C<u>*H*</u>$_2$-$(CH_2)_2$-$CH_3$), 2.33 (s, 3H, $CH_3$), 3.14-3.16 (t, *J* = 5.1 Hz, 2H, S-C<u>*H*</u>$_2$-$(CH_2)_3$-$CH_3$), 6.47 (s, 1H, pyrazole, =CH-), 7.33-7.37 (dd, *J* = 6.36, 2.59 Hz, 2H, $C_6H_5$), 7.48-7.53 (m, 3H, $C_6H_5$), 11.74 (s, 1H, pyrazole, NH). Anal. calcd. (%) for $C_{17}H_{21}N_5S$: C, 62.36; H, 6.46; N, 21.39; S, 9.79 Found: C, 62.52; H, 6.45; N, 21.35; S, 9.82. ESI-MS: m/z= 328 [M+1], 330 [M+3].

**3-(Hexylthio)-5-(5-methyl-1*H*-pyrazol-3-yl)-4-phenyl-4*H*-1,2,4-triazole (8.6).** Yield: 79 %; m. p.: 162 – 164 °C; IR (ν, $cm^{-1}$): 3237 (NH), 1618 (C=N); $^1$H NMR (*δ*, ppm): 0.87-0.90 (t, 3H, S-$(CH_2)_5$-C<u>*H*</u>$_3$), 1.37-1.42 (m, *J* = 4.8 Hz, 1.7, 1.3 Hz, 6H, S-$(CH_2)_2$-$(C$<u>*H*</u>$_2)_3$-$CH_3$), 1.72-1.75 (m, *J* = 7.8

Hz, 2H, S-$CH_2$-C$\underline{H_2}$-$(CH_2)_3$-$CH_3$), 2.33 (s, 3H, $CH_3$), 3.15-3.17 (t, *J* = 7.9 Hz, 2H, S-C$\underline{H_2}$-$(CH_2)_4$-$CH_3$), 6.47 (s, 1H, pyrazole, =CH-), 7.33-7.37 (dd, *J* = 6.68, 2.89 Hz, 2H, $C_6H_5$), 7.48-7.54 (m, 3H, $C_6H_5$), 11.73 (s, 1H, pyrazole, NH). Anal. calcd. (%) for $C_{18}H_{23}N_5S$: C, 63.31; H, 6.79; N, 20.51; S, 9.39. Found: C, 63.16; H, 6.78; N, 20.56; S, 9.41. ESI-MS: m/z= 342 [M+$H^+$], 344 [M+3].

**3-(Heptylthio)-5-(5-methyl-1*H*-pyrazol-3-yl)-4-phenyl-4*H*-1,2,4-triazole (8.7).** Yield: 68 %; m. p.: 161 – 163 °C; IR (ν, $cm^{-1}$): 3223 (NH), 1588 (C=N); $^1$H NMR (*δ*, ppm): 0.85-0.88 (t, 3H, S-$(CH_2)_6$-C$\underline{H_3}$), 1.33-1.40 (m, 8H, S-$(CH_2)_2$-(C$\underline{H_2}$)$_4$-$CH_3$), 1.70-1.73 (m, *J* = 8.2 Hz, 2H, S-C$\underline{H_2}$-$(CH_2)_5$-$CH_3$), 2.31 (s, 3H, $CH_3$), 3.13-3.15 (t, *J* = 7.9 Hz, 2H, S-C$\underline{H_2}$-$(CH_2)_5$-$CH_3$), 6.47 (s, 1H, pyrazole, =CH-), 7.34-7.38 (dd, *J* = 6.78, 2.95 Hz, 2H, $C_6H_5$), 7.47-7.54 (m, 3H, $C_6H_5$), 11.73 (s, 1H, pyrazole, NH). Anal. calcd. (%) for $C_{19}H_{25}N_5S$: C, 64.19; H, 7.09; N, 19.70; S, 9.02. Found: C, 64.33; H, 7.08; N, 19.65; S, 9.05. ESI-MS: m/z= 356 [M+$H^+$], 358 [M+3].

**3-(5-Methyl-1*H*-pyrazol-3-yl)-5-(octylthio)-4-phenyl-4*H*-1,2,4-triazole (8.8).** Yield: 76 %; m. p.: 156 – 158 °C; IR (ν, $cm^{-1}$): 3225 (NH), 1604 (C=N); $^1$H NMR (*δ*, ppm): 0.85-0.87 (t, 3H, S-$(CH_2)_7$-C$\underline{H_3}$), 1.32-1.38 (m, 10H, S-$(CH_2)_2$-(C$\underline{H_2}$)$_5$-$CH_3$), 1.68-1.71 (m, *J* = 8.0 Hz, 2H, S-$CH_2$-C$\underline{H_2}$-$(CH_2)_5$-$CH_3$), 2.31 (s, 3H, $CH_3$), 3.13-3.15 (t, *J* = 7.8 Hz, 2H, S-C$\underline{H_2}$-$(CH_2)_7$-$CH_3$), 6.46 (s, 1H, pyrazole, =CH-), 7.34-7.38 (dd, *J* = 6.68, 2.89 Hz, 2H, $C_6H_5$), 7.47-7.53 (m, 3H, $C_6H_5$), 11.73 (s, 1H, pyrazole, NH). Anal. calcd. (%) $C_{20}H_{27}N_5S$: C, 65.01; H, 7.37; N, 18.95; S, 8.68. Found: C, 64.83; H, 7.36; N, 19.00; S, 8.70. ESI-MS: m/z= 370 [M+$H^+$], 372 [M+3].

**3-(5-Methyl-1*H*-pyrazol-3-yl)-5-(nonylthio)-4-phenyl-4*H*-1,2,4-triazole (8.9).** Yield: 71 %; m. p.: 150 – 152 °C; IR (ν, $cm^{-1}$): 3221 (NH), 1579 (C=N); $^1$H NMR (*δ*, ppm): 0.83-0.86 (t, 3H, S-$(CH_2)_8$-C$\underline{H_3}$), 1.32-1.38 (m, 12H, S-$(CH_2)_2$-(C$\underline{H_2}$)$_6$-$CH_3$), 1.68-1.71 (m, *J* = 8.2 Hz, 2H, S-$CH_2$-C$\underline{H_2}$-$(CH_2)_6$-$CH_3$), 2.32 (s, 3H, $CH_3$), 3.11-3.13 (t, *J* = 7.9 Hz, 2H, S-C$\underline{H_2}$-$(CH_2)_7$-$CH_3$), 6.46 (s, 1H, pyrazole, =CH-), 7.33-7.37 (dd, *J* = 6.62, 2.85 Hz, 2H, $C_6H_5$), 7.45-7.51 (m, 3H, $C_6H_5$), 11.72 (s, 1H, pyrazole, NH). Anal. calcd. (%) for $C_{21}H_{29}N_5S$: C, 65.76; H, 7.62; N, 18.26; S, 8.36. Found: C, 65.92; H, 7.63; N, 18.21; S, 8.33. ESI-MS: m/z= 384 [M+$H^+$], 386 [M+3].

**3-(Decylthio)-5-(5-methyl-1*H*-pyrazol-3-yl)-4-phenyl-4*H*-1,2,4-triazole (8.10).** Yield: 73 %; m. p.: 152 – 154 °C; IR (ν, $cm^{-1}$): 3238 (NH), 1603 (C=N); $^1$H NMR (*δ*, ppm): 0.83-0.85 (t, 3H, S-$(CH_2)_9$-C$\underline{H_3}$), 1.31-1.36 (m, 14H, S-$(CH_2)_2$-(C$\underline{H_2}$)$_7$-$CH_3$), 1.67-1.71 (m, *J* = 8.1 Hz, 2H, S-$CH_2$-C$\underline{H_2}$-$(CH_2)_7$-$CH_3$), 2.32 (s, 3H, $CH_3$), 3.10-3.13 (t, *J* = 7.7 Hz, 2H, S-C$\underline{H_2}$-$(CH_2)_8$-$CH_3$), 6.46 (s, 1H, pyrazole, =CH-), 7.33-7.36 (dd, *J* = 6.68, 2.89 Hz, 2H, $C_6H_5$), 7.49-7.54 (m, 3H, $C_6H_5$), 11.75 (s, 1H, pyrazole, NH). Anal. calcd. (%) for $C_{22}H_{31}N_5S$: C, 66.46; H, 7.86; N, 17.62; S, 8.06. Found: C, 66.27; H, 7.87; N, 17.57; S, 8.08. ESI-MS: m/z= 398 [M+$H^+$], 400 [M+3].

The sets of S-alkyl fragment proton signals are fixed in the expected magnetic field region, and their parameters correspond to the literature data [10]. For example, the signals of protons methyl group are shown at 2,75 ppm in the form of singlet (8.1). Increasing the length of the alkyl chain contributes

to the shift of proton signals in the direction of a stronger field (+I and +M-effects). Thus, the signals of the protons of the methyl fragment is gradually changed to 0.83 ppm; the signals of protons of methylene fragment are observed in the region of strong fields in the form of a triplet (3.10-3.19 ppm) or multiplets (1.31-1.42 ppm, 1.66-1.75 ppm). Signals in the form of doublets (7.33-7.38 ppm) and multiplets (7.45-7.54 ppm) are observed in the absorption region of aromatic protons. Hydrogen at $C_4$ atom of the pyrazole fragment resonates at 6.45-6.47 ppm. in the form of a singlet.

In the IR-spectrum of synthesized alkyl derivatives observe deformation vibrations of alkyl groups in ranges from 645 to 1300 $cm^{-1}$ and H-C-H fragment in a narrow area of frequency 1465 - 1370 $cm^{-1}$. For example, for $CH_3$-group δ-vibrations occupied area at 1367 - 1374 $cm^{-1}$. Valence vibrations of bonds of C-H alkyl groups form bands in area 3085 - 2830 $cm^{-1}$ [11].

In the mass spectrum, there is a peak of the molecular ion and peaks of fragment ions, which confirm this structure.

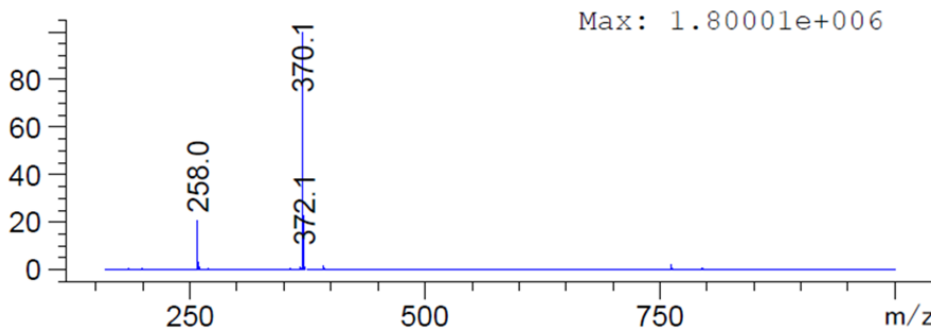


**Figure 2.** Mass fragmentation pattern of compound 8.8 (CID*, 200 eV)

*CID – Collision-induced dissociation

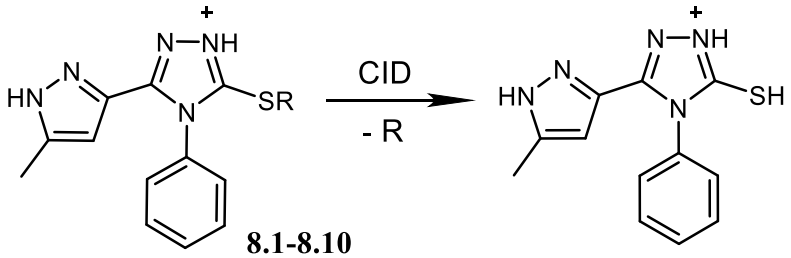


**Figure 3.** Molecular ion fragmentation scheme (CID, 200 eV)

A study of antiradical activity of synthesized substances was carried out to assess the prospects of this research direction. The decision to study this type of activity was made in connection with the detection of this type of activity in similar substances.

The synthesized compounds exhibit moderate antiradical activity, which in some of them approaches the reference product "Ascorbic acid". Modification of the alkyl fragment does not lead to a

significant change in antiradical activity. Importantly, reducing the concentration of the compounds also does not lead to a significant loss of antiradical activity. The highest loss of activity was observed for compound 8.10. It should also be noted that the appearance of the alkyl fragment in most cases leads to a decrease in activity (5% - 42%). This is due to the more pronounced acidic properties of the free thiol group compared to the alkylated one.

The data obtained confirmed the assumption of the presence of antiradical activity, but it was not expressed (**Table 1**).

**Table 1.** Antiradical activity of synthesized compounds

| № | Compound | 2 ml 2 mM compound, investigated + 2 ml 0.1 mm DPPH solution, I%; $1•10^{-3}$ | 2 ml 2 mM compound, investigated + 2 ml 0.1 mm DPPH solution, I%; $1•10^{-4}$ |
|---|---|---|---|
| 1 | Ascorbic acid | 92,42 | 79,85 |
| 2 | 7 | 76,15 | 65,13 |
| 3 | 8.1 | 72,31 | 55,04 |
| 4 | 8.2 | 60,68 | 51,54 |
| 5 | 8.3 | 59,15 | 52,31 |
| 6 | 8.4 | 43,93 | 42,31 |
| 7 | 8.5 | 78,12 | 72,82 |
| 8 | 8.6 | 63,16 | 55,64 |
| 9 | 8.7 | 84,10 | 74,27 |
| 10 | 8.8 | 63,42 | 63,93 |
| 11 | 8.9 | 78,80 | 69,06 |
| 12 | 8.10 | 50,09 | 10,43 |

A universal method for the preparation of 5-(5-methyl-1*H*-pyrazole-3-yl)-4-phenyl-4*H*-1,2,4-triazole-3-thiol and its alkylderivatives was developed. The structure and individuality of the synthesized compounds was confirmed by $^{1}$H NMR, IR and LC-MS spectra, elemental analysis. The biological potential of new compounds was ascertained.

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