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Synthesis of 4,6-Dimethyl-2-thioxo-1,2-dihydropyridine-3-carbonitrile by Condensation of Cyanothioacetamide with Acetaldehyde and 1-(Prop-1-en-2-yl)piperidine

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Abstract—Three-component condensation of cyanothioacetamide with acetaldehyde and 1-(prop-1-en-2-yl)-piperidine afforded 4,6-dimethyl-2-thioxo-1,2-dihydropyridine-3-carbonitrile which was alkylated with alkyl halides to obtain substituted 2-alkylsulfanyl-4,6-dimethylpyridine-3-carbonitriles, (3-amino-4,6-dimethylthieno-[2,3-*b*]pyridin-2-yl)(4-cyclohexylphenyl)methanone, and 2,2′-[ethane-1,2-diylbis(sulfanediyl)]bis(4,6-dimethylpyridine-3-carbonitrile).

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Alkyl-substituted 2-oxo(thioxo)-1,2-dihydropyridines are known as biologically active compounds possessing antitumor [1, 2], anti-HIV [3–5], and antidiabetic properties [6]. They are also used for the treatment of Parkinson's [7] and Alzheimer's diseases [8]. Therefore, development of new methods for the synthesis of 2-oxo(thioxo)-1,2-dihydropyridine derivatives and study of their chemical and biological properties are topical problems.

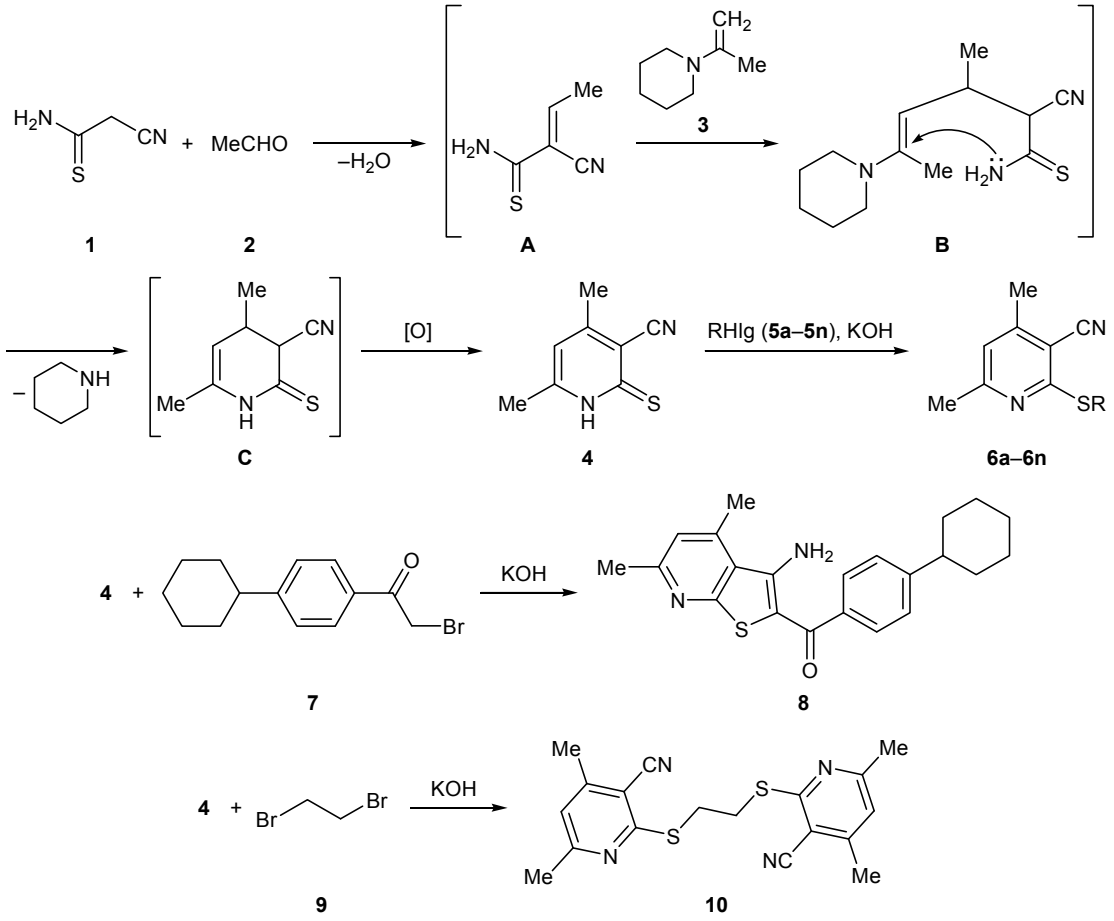
In continuation of our studies on the synthesis of alkyl-substituted pyridinechalcogenones starting from aliphatic aldehydes [9–12], in the present work we examined three-component condensation of cyanothioacetamide (**1**), acetaldehyde (**2**), and 1-(prop-1-en-2-yl)piperidine (**3**) in anhydrous ethanol at 20°C. As a result, we isolated 4,6-dimethyl-2-thioxo-1,2-dihydropyridine-3-carbonitrile (**4**) which was synthesized previously by condensation of acetylacetone with cyanothioacetamide [13], as well as by Michael-type reaction (exchange of methylene components) of 2-cyano-2-cyclohexylidenethioacetamide with acetylacetone [14].

It is reasonable to presume intermediate formation of Knoevenagel condensation product **A** which reacts with enamine **3** according to Stork [15] to give adduct **B**. Intramolecular transamination [16] of the latter leads to closure of tetrahydropyridine ring (intermediate **C**), and dehydrogenation of **C** (presumably, by the action of atmospheric oxygen) yields substituted 1,2-dihydropyridine **4** (Scheme 1). The structure of **4** was confirmed by its chemical transformations. The alkylation of **4** with alkyl halides **5a**–**5n** in DMF in the presence of aqueous potassium hydroxide gave sulfides **6a**–**6n**, which is consistent with the general behavior of 2-thioxo-3-cyanopyridines [17–19].

When α-bromo ketone **7** was used as alkylating agent, we isolated 2-(4-cyclohexylbenzoyl)-4,6-dimethylthieno[2,3-*b*]pyridin-3-amine (**8**) as a result of facile intramolecular cyclization of intermediate **6**, which is typical of substituted pyridines with vicinal alkylsulfanyl and cyano groups [20–22]. Thienopyridine **8** may be promising for the design of antistress [23] and antibacterial agents [24]. By reaction of 2 equiv of **4** with 1,2-dibromoethane (**9**) we obtained 2,2′-[ethane-1,2-diylbis(sulfanediyl)]bis(4,6-dimethylpyridine-3-carbonitrile) (**10**).

The structure of **6a**–**6n**, **8**, and **10** was confirmed by spectral methods. Their ^{1}H NMR spectra contained singlets from methyl protons (see Experimental) and a signal from 5-H in the pyridine ring at δ 6.81–7.12 ppm. The SCH_2 protons of **6a**–**6n** resonated in the ^{1}H NMR spectra as a singlet at δ 4.01–4.76 ppm, which was not observed in the spectrum of **8**. Instead, the latter displayed a broadened singlet at δ 7.99 ppm due to protons of the amino group, which is typical of structurally related systems [25–27]. In the IR spectra

Scheme 1.



5, **6**, Hlg = Br, R = $BuOC(O)CH_2$ (**a**), 2-(2-oxo-2*H*-chromen-3-yl)-2-oxoethyl (**b**), 3,4-$(HO)_2C_6H_3C(O)CH_2$ (**c**), 4-$FC_6H_4C(O)CH_2$ (**d**), 2-oxo-2-(thiophen-2-yl)ethyl (**e**), $PhC(O)CH_2$ (**f**), $PhC(O)CHCHMe_2$ (**g**), 2-$MeC_6H_4CH_2$ (**h**), $HC{\equiv}CCH_2$ (**i**), 2-$ClC_6H_4CH_2$ (**j**); Hlg = Cl, $(Me)_2CHOC(O)CH_2$ (**k**), $PrOC(O)CH_2$ (**l**), $PhCH_2$ (**m**), $PhCHC(O)NH_2$ (**n**).

of **6a**–**6n** and **10** we observed a characteristic absorption band at 2214–2225 cm^{-1} due to stretching vibrations of the conjugated cyano group and carbonyl stretching band at 1695–1714 cm^{-1}.

EXPERIMENTAL

The IR spectra were recorded on an IKS-40 spectrometer from samples dispersed in mineral oil. The ^1H NMR spectra were measured on a Varian Mercury-400 spectrometer at 400.397 MHz from solutions in DMSO-d_6 containing tetramethylsilane as internal standard. The mass spectra (electron impact, 70 eV) were obtained on an Agilent GC/MS instrument; samples were introduced in a CF_3COOH matrix. The elemental compositions were determined on a Perkin Elmer CHN analyzer. The melting points were measured on a Kofler hot stage. The progress of reactions and the purity of the isolated compounds were monitored by TLC on Silufol-254 plates using acetone–hexane (3 : 5) as eluent; spots were visualized by treatment with iodine vapor and under UV light.

4,6-Dimethyl-2-thioxo-1,2-dihydropyridine-3-carbonitrile (4). A mixture of 0.6 mL (10 mmol) of freshly distilled acetaldehyde (**2**), 1.0 g (10 mmol) of cyanothioacetamide (**1**), and a drop of *N*-methylmorpholine in 20 mL of anhydrous ethanol was stirred for 20 min at 20°C, 1.3 g (10 mmol) of enamine **3** was added, and the mixture was stirred for 1 h and left to stand for 24 h. The mixture was then treated with 10% aqueous HCl until pH 5 and kept for 5 h, and the precipitate was filtered off and washed with ethanol and hexane. Yield 1.1 g (70%), yellow crystals, mp 263–265°C (from EtOH); published data [13]: mp 264°C.

Alkylation of compound 4 (*general procedure*). To a mixture of 1.64 g (10 mmol) of compound **4** and 15 mL of DMF we added under stirring in succession

5.6 mL (10 mmol) of 10% aqueous KOH and 10 mmol of alkylating agent **5a**–**5n** or **7**. The mixture was stirred for 4 h and diluted with an equal volume of water, and the precipitate of **6a**–**6n** or **8** was filtered off, and washed with water, ethanol, and hexane.

Butyl 2-(3-cyano-4,6-dimethylpyridin-2-ylsulfanyl)acetate (6a). Yield 2.2 g (79%), mp 56°C (from MeOH). IR spectrum, ν, cm^{-1}: 2225 (C≡N), 1695 (C=O). ^{1}H NMR spectrum, δ, ppm: 0.84 t (3H, Me, J = 6.2 Hz), 1.09–1.68 m (4H, CH_2), 2.42 s (6H, Me), 4.01 s (2H, SCH_2), 4.07 t (2H, OCH_2, J = 6.6 Hz), 7.12 s (1H, 5-H). Mass spectrum: *m*/*z* 279 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 60.28; H 6.41; N 9.95. $C_{14}H_{18}N_2O_2S$. Calculated, %: C 60.41; H 6.52; N 10.06. *M* 278.375.

4,6-Dimethyl-2-{[2-oxo-2-(2-oxo-3,4-dihydro-2*H*-1-benzopyran-3-yl)ethyl]sulfanyl}pyridine-3-carbonitrile (6b). Yield 2.5 g (72%), mp 212–214°C (from AcOH). IR spectrum, ν, cm^{-1}: 2219, 1703, 1699 (C=O). ^{1}H NMR spectrum, δ, ppm: 2.27 s (3H, Me), 2.38 s (3H, Me), 4.76 s (2H, CH_2), 7.05 s (1H, 5-H), 7.31–7.58 m (2H, H_{arom}), 7.78 d (1H, H_{arom}, J = 7.5 Hz), 7.98 d (1H, H_{arom}, J = 8.0 Hz), 8.72 s (1H, 4′-H). Mass spectrum: *m*/*z* 351 (I_{rel} 100) $[M + 1]^+$. Found, %: C 65.02; H 3.89; N 7.88. $C_{19}H_{14}N_2O_3S$. Calculated, %: C 65.13; H 4.03; N 8.00. *M* 350.399.

2-{[2-(3,4-Dihydroxyphenyl)-2-oxoethyl]sulfanyl}-4,6-dimethylpyridine-3-carbonitrile (6c). Yield 2.5 g (80%), mp 109–111°C (from EtOH). IR spectrum, ν, cm^{-1}: 3460, 3424 (OH); 2222 (C≡N); 1701 (C=O). ^{1}H NMR spectrum, δ, ppm: 2.32 s (3H, Me), 2.41 s (3H, Me), 4.66 s (2H, CH_2), 6.81 d (1H, H_{arom}, J = 7.9 Hz), 6.99 s (1H, 5-H), 7.40 s (1H, H_{arom}), 7.49 d (1H, H_{arom}, J = 7.9 Hz), 9.09 br.s (1H, OH), 9.61 br.s (1H, OH). Mass spectrum: *m*/*z* 315 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 60.98; H 3.33; N 8.80. $C_{16}H_{14}N_2O_3S$. Calculated, %: C 61.13; H 4.49; N 8.91. *M* 314.363.

2-{[2-(4-Fluorophenyl)-2-oxoethyl]sulfanyl}-4,6-dimethylpyridine-3-carbonitrile (6d). Yield 2.2 g (72%), mp 113–115°C (from AcOH). IR spectrum, ν, cm^{-1}: 2221 (C≡N), 1699 (C=O). ^{1}H NMR spectrum, δ, ppm: 2.23 s (3H, Me), 2.41 s (3H, Me), 4.72 s (2H, CH_2), 6.99 s (1H, 5-H), 7.22–7.43 m (2H, H_{arom}), 8.09–8.21 m (2H, H_{arom}). Mass spectrum: *m*/*z* 301 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 63.87; H 4.21; N 9.16. $C_{16}H_{13}FN_2OS$. Calculated, %: C 63.98; H 4.36; N 9.33. *M* 300.354.

4,6-Dimethyl-2-{[2-oxo-2-(thiophen-2-yl)ethyl]sulfanyl}pyridine-3-carbonitrile (6e). Yield 2.4 g (84%), mp 115–116°C (from AcOH). IR spectrum, ν, cm^{-1}: 2214 (C≡N), 1700 (C=O). ^{1}H NMR spectrum, δ, ppm: 2.15 s (3H, Me), 2.38 s (3H, Me), 4.74 s (2H, CH_2), 7.03 s (1H, 5-H), 7.29 d.d (1H, 4′-H, J = 5.2 Hz), 8.04 d (1H, 3′-H, J = 4.6 Hz), 8.18 d (1H, 5′-H, J = 3.7 Hz). Mass spectrum: *m*/*z* 289 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 58.22; H 4.06; N 9.67. $C_{14}H_{12}N_2OS_2$. Calculated, %: C 58.31; H 4.19; N 9.71. *M* 288.392.

4,6-Dimethyl-2-[(2-oxo-2-phenylethyl)sulfanyl]-pyridine-3-carbonitrile (6f). Yield 2.2 g (78%), mp 118–120°C (from AcOH). IR spectrum, ν, cm^{-1}: 2218 (C≡N), 1705 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.19 s (3H, Me), 1.33 s (3H, Me), 4.62 s (2H, CH_2), 6.81 s (1H, 5-H), 7.41 t (2H, Ph, J = 7.0 Hz), 7.52 t (1H, Ph, J = 7.0 Hz), 7.91 d (2H, Ph, J = 7.03 Hz). Mass spectrum: *m*/*z* 283 (I_{rel} 100) $[M + 1]^+$. Found, %: C 67.94; H 4.88; N 9.80. $C_{16}H_{14}N_2OS$. Calculated, %: C 68.06; H 5.00; N 9.92. *M* 282.364.

4,6-Dimethyl-2-[(3-methyl-1-oxo-1-phenylbutan-2-yl)sulfanyl]pyridine-3-carbonitrile (6g). Yield 2.5 g (77%), mp 113–115°C (from AcOH). IR spectrum, ν, cm^{-1}: 2217 (C≡N), 1714 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.01 d and 1.05 d (3H each, **Me**$_2$CH, J = 6.4 Hz), 2.07 s (3H, Me), 2.25–2.31 m (1H, C**H**Me$_2$), 2.35 s (3H, Me), 3.69 d (1H, SCH, J = 6.0 Hz), 7.00 s (1H, 5-H), 7.55 t (2H, Ph, J = 7.6 Hz), 7.66 t (1H, Ph, J = 7.6 Hz), 8.10 d (2H, Ph, J = 7.6 Hz). Mass spectrum: *m*/*z* 325 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 70.19; H 6.02; N 8.48. $C_{19}H_{20}N_2OS$. Calculated, %: C 70.34; H 6.21; N 8.63. *M* 324.447.

4,6-Dimethyl-2-{[(2-methylphenyl)methyl]sulfanyl}pyridine-3-carbonitrile (6h). Yield 1.8 g (68%), mp 82–84°C (from EtOH). IR spectrum: ν 2217 cm^{-1} (C≡N). ^{1}H NMR spectrum, δ, ppm: 2.38 s (3H, Me), 2.55 s (3H, Me), 4.53 s (2H, CH_2), 7.06 s (1H, 5-H), 7.12–7.23 m (2H, H_{arom}), 7.40 t (2H, H_{arom}, J = 7.3 Hz). Mass spectrum: *m*/*z* 269 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 71.52; H 5.94; N 10.30. $C_{16}H_{16}N_2S$. Calculated, %: C 71.61; H 6.01; N 10.44. *M* 268.381.

4,6-Dimethyl-2-(prop-2-yn-1-ylsulfanyl)pyridine-3-carbonitrile (6i). Yield 2.4 g (70%), mp 87–89°C (from AcOH). IR spectrum, ν, cm^{-1}: 3310 (≡C–H), 2222 (C≡N), 2240 (C≡C). ^{1}H NMR spectrum, δ, ppm: 2.43 s (3H, Me), 2.51 s (3H, Me), 2.77 s (1H, ≡CH), 4.05 s (2H, CH_2), 7.08 s (1H, 5-H). Mass spectrum: *m*/*z* 203 (I_{rel} 100) $[M + 1]^+$. Found, %: C 65.21; H 4.80; N 13.69. $C_{11}H_{10}N_2S$. Calculated, %: C 65.32; H 4.98; N 13.85. *M* 202.28.

2-[(4-Chlorobenzyl)sulfanyl]-4,6-dimethylpyridine-3-carbonitrile (6j). Yield 2.1 g (74%), mp 129–131°C (from EtOH). IR spectrum: ν 2221 cm^{-1} (C≡N). ^1H NMR spectrum, δ, ppm: 2.38 s (3H, Me), 2.53 s (3H, Me), 4.50 s (2H, CH_2), 7.11 s (1H, 5-H), 7.32 d (2H, H_{arom}, J = 7.9 Hz), 7.49 d (2H, H_{arom}, J = 7.9 Hz). Mass spectrum, m/z 289 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 62.19; H 4.39; N 9.62. $C_{15}H_{13}ClN_2S$. Calculated, %: C 62.38; H 4.54; N 9.70. M 288.801.

Isopropyl 2-(3-cyano-4,6-dimethylpyridin-2-ylsulfanyl)acetate (6k). Yield 2.4 g (90%), mp 80–82°C (from *i*-PrOH). IR spectrum, ν, cm^{-1}: 2223 (C≡N), 1714 (C=O). ^1H NMR spectrum, δ, ppm: 1.19 d (6H, Me, J = 5.1 Hz), 2.41 s (3H, Me), 2.43 s (3H, Me), 4.03 s (2H, CH_2), 4.73–5.08 m (1H, C**H**Me_2), 7.10 s (1H, 5-H). Mass spectrum: m/z 265 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 58.97; H 5.95; N 10.49. $C_{13}H_{16}N_2O_2S$. Calculated, %: C 59.07; H 6.10; N 10.60. M 264.349.

Propyl 2-(3-cyano-4,6-dimethylpyridin-2-ylsulfanyl)acetate (6l). Yield 1.8 g (70%), mp 53–54°C (from PrOH). IR spectrum, ν, cm^{-1}: 2227 (C≡N), 1709 (C=O). ^1H NMR spectrum, δ, ppm: 0.83 t (3H, Me, J = 6.2 Hz), 1.42–1.73 m (2H, CH_2), 2.41 s (3H, Me), 2.42 s (3H, Me), 4.02 t (2H, OCH_2, J = 4.7 Hz), 4.08 s (2H, SCH_2), 7.10 s (1H, 5-H). Mass spectrum: m/z 265 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 58.97; H 5.95; N 10.47. $C_{13}H_{16}N_2O_2S$. Calculated, %: C 59.07; H 6.10; N 10.60. M 264.349.

2-(Benzylsulfanyl)-4,6-dimethylpyridine-3-carbonitrile (6m). Yield 2.2 g (85%), mp 80–82°C (from EtOH). IR spectrum: ν 2215 cm^{-1} (C≡N). ^1H NMR spectrum, δ, ppm: 2.37 s (3H, Me), 2.53 s (3H, Me), 4.51 s (2H, SCH_2), 7.09 s (1H, 5-H), 7.21–7.49 m (5H, Ph). Mass spectrum: m/z 255 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 70.70; H 5.42; N 10.86. $C_{15}H_{14}N_2S$. Calculated, %: C 70.83; H 5.55; N 11.01. M 254.356.

2-[(3-Cyano-4,6-dimethylpyridin-2-yl)sulfanyl]-2-phenylacetamide (6n). Yield 2.4 g (81%), mp 174–176°C (from BuOH). IR spectrum, ν, cm^{-1}: 3355, 3290, 3199 (NH_2), 2216 (C≡N), 1669 (C=O). ^1H NMR spectrum, δ, ppm: 2.34 s (3H, Me), 2.47 s (3H, Me), 5.71 s (1H, SCH), 7.04 s (1H, 5-H), 7.11–7.45 m (3H, Ph, NH), 7.47–7.69 m (3H, Ph), 7.86 br.s (1H, NH). Mass spectrum: m/z 298 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 64.51; H 4.96; N 14.02. $C_{16}H_{15}N_3OS$. Calculated, %: C 64.62; H 5.08; N 14.13. M 297.379.

(3-Amino-4,6-dimethylthieno[2,3-*b*]pyridin-2-yl)(4-cyclohexylphenyl)methanone (8). Yield 1.8 g (71%), mp 171–173°C (from BuOH). IR spectrum, ν, cm^{-1}: 3322, 3290, 3198 (NH_2); 1711 (C=O); 1644 (δNH). ^1H NMR spectrum, δ, ppm: 1.11–1.52 m (6H, CH_2), 1.64–1.93 m (5H, CH_2CHCH_2), 2.51 s (3H, Me), 2.75 s (3H, Me), 7.08 s (1H, 5-H), 7.35 d (2H, H_{arom}, J = 8.0 Hz), 7.70 d (2H, H_{arom}, J = 8.0 Hz), 7.99 br.s (2H, NH_2). Mass spectrum: m/z 365 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 72.36; H 6.51; N 7.54. $C_{22}H_{24}N_2OS$. Calculated, %: C 72.49; H 6.64; N 7.69. M 364.512.

2,2′-[Ethane-1,2-diylbis(sulfanediyl)]bis(4,6-dimethylpyridine-3-carbonitrile) (10) was synthesized as described above for compounds **6** using 0.45 mL (5 mmol) of 1,2-dibromoethane (**9**) as alkylating agent. Yield 1.2 g (66%), mp 193–195°C (from BuOH). IR spectrum: ν 2225 cm^{-1} (C≡N). ^1H NMR spectrum, δ, ppm: 2.40 s (6H, Me), 2.44 s (6H, Me), 3.62 br.s (4H, CH_2), 7.06 s (2H, 5′-H). Mass spectrum: m/z 355 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 60.84; H 5.02; N 15.76. $C_{18}H_{18}N_4S_2$. Calculated, %: C 60.99; H 5.12; N 15.81. M 354.499.

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