ISSN 1070-3632, Russian Journal of General Chemistry, 2012, Vol. 82, No. 11, pp. 1793–1797. © Pleiades Publishing, Ltd., 2012.
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Synthesis and Properties of Aminomethoxyderivatives of 1-Ethylthiopentane

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Received May 31, 2011

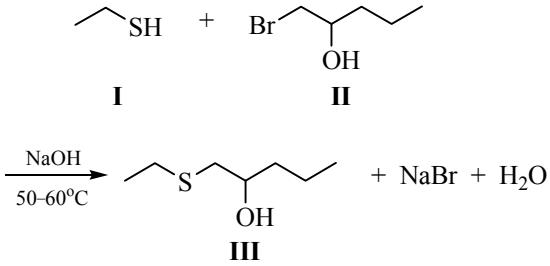
Abstract—Previously unknown aminomethoxy derivatives of 1-ethylthiopentane were synthesized by the three-component Mannich reaction of 1-ethylthiopentanol with formaldehyde in the presence of secondary amines and they were characterized. The structure of the compounds obtained was established by means of elemental analysis, IR and ^{1}H NMR spectroscopy, and mass spectrometry. The products synthesized were tested as antimicrobial additives to lubricants and antiseptic substances against bacteria and fungi. They are more effective antimicrobial agents than the preparations which are nowadays used in practice.

DOI: 10.1134/S1070363212110084

Organic substances simultaneously containing various functional groups and heteroatoms like nitrogen and sulfur are widely known as effective biologically active compounds and pharmaceutical preparations. They are also used as antioxidant, anticorrosion, and antimicrobial additives to fuels and oils [1–5].

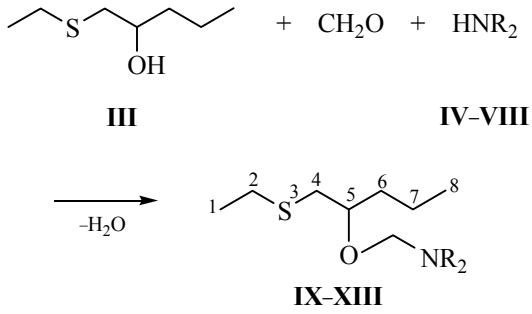
The development of modern technique is characterized by increased demands to the performance of machines, and hence, to the quality of lubricants. Therefore a great importance is acquired by the synthesis of new types of additives and studies of functional properties of new organic compounds [6, 7]. The problem of the targeted synthesis of such compounds used in various chemical areas is important not only for the development of the synthetic organic chemistry, but also for the studies of applied character. In connection with that the synthesis of new compounds of this type from the available raw material by improved procedures attracts the attention of researchers [8–10]. Mannich reaction is one of significant and promising methods of synthesis of sulfur- and nitrogen-containing organic compounds [11–14].

Continuing our studies in the field of synthesis of nitrogen-containing alkyl(sulfanyl)alkanes [15–22] we present the results of the synthesis and the investigation of properties of new aminomethoxy derivatives of 1-(ethylsulfanyl)pentane. With this purpose we have at first synthesized previously unknown sulfur-containing secondary aliphatic alcohol, 1-(ethylsulfanyl)pentan-2-ol **III** by the reaction of ethanethiol **I** with 1-bromopentan-2-ol **II** according to the following scheme.



The synthesis of alcohol **III** was carried out by the reaction of equimolar amounts of starting components **I** and **II** in alkaline medium (40% sodium hydroxide) at 45–50°C within 3–4 h. Yield of the target product **III** was 62%.

After that by the Mannich condensation of alcohol **III** with formaldehyde in the presence of aliphatic and heterocyclic amines (diethylamine, dibutylamine, piperidine, morpholine, and hexamethylenimine) **IV**-**VIII** new representatives of aminomethoxy derivatives of 1-(ethylsulfanyl)pentane **IX–XIII** were synthesized according to the following scheme.



IX–XIII

NR_2 = NEt_2 (**IV, IX**), NBu_2 (**V, X**), piperidine (**VI, XI**), morpholine (**VII, XII**), $N(CH_2)_6$ (**VIII, XIII**).

Synthesis of aminomethoxy derivatives of 1-(ethylsulfanyl)pentane was carried out at 40–55°C for 3–4 h at the equimolar ratio of starting components. Yield of the products **IX–XIII** was 67–74%.

Alcohol **III** and its amino derivatives **IX–XIII** are colorless liquids with the characteristic odor. They are insoluble in water but well soluble in organic solvents like ethanol, acetone, chloroform, benzene, carbon tetrachloride, etc.

The composition and structure of the compounds synthesized were confirmed by the elemental analysis data, IR and ^{1}H NMR spectroscopy, and also by mass spectrometry.

The purity of starting substances and the compounds obtained, and also the composition of reaction mixtures was controlled by GLC.

In the IR spectrum of 1-(ethylsulfanyl)pentan-2-ol **III** broad absorption band is observed at 3500 cm^{-1} characteristic of hydroxy group [23], and in the IR spectra of compounds **IX–XII** this band is absent. Spectra of all substances synthesized contained the band in the range 730 cm^{-1} characteristic of C–S bond. In the IR spectra of compounds **IX–XIII** an absorption band at 1200–1010 cm^{-1} is observed. It is characteristic of the C–N bond vibrations. Besides the above-mentioned bands the IR spectra contained the bands at 2910–2895 and 2880–2810 cm^{-1} characteristic of C–H vibrations of the methyl and methylene groups respectively.

^{1}H NMR spectra of synthesized compounds **IX–XIII** also confirm their structures.

In the ^{1}H NMR spectrum of secondary alcohol **III** OCH group proton belonging to C^5 carbon atom adjacent to the hydroxy group appeatrs as a multiplet at 3.65 ppm. The protons of the methylene group located at C^4 atom neighboring to sulfur (SCH_2) give rise to a doublet of doublets at 2.75 ppm. The singlet of the hydroxy group proton is observed at 2.70 ppm. The protons of the methylene group at C^2 near the sulfur atom (SCH_2) give rise to a quartet at 2.5 ppm. The protons of the methylene group of the C^7 carbon atom appear as a multiplet in the range 1.5–1.7 ppm. The protons of the terminal methyl groups located at C^1 and C^8 atoms are observed as triplets at 1.3 and 0.9 ppm respectively.

In the ^{1}H NMR spectra of compounds **IX–XIII** the location of signals of protons linked to C^1-C^8 carbon atoms remains almost the same. The signals of protons of OCH_2N fragment in the spectra of these substances form a doublet of doublets at 4.2 ppm, and the proton of OCH fragment (C^5) is observed as a multiplet at 3.2 ppm. In the compound **IX** the protons of methyl groups from the diethylamine fragment give rise to a triplet at 1.2 ppm, while in the case of dibutyl fragment in compound **X** the triplet of the corresponding methyl group is observed at 0.9 ppm. The signals of protons of CH_2–N–CH_2 fragment in the compounds **IX–XIII** are observed at 2.4–2.6 ppm as a multiplet. In the spectrum of compound **XII** a multiplet observed at 3.7 ppm belongs to CH_2–O–CH_2 protons of the morpholine fragment.

In the mass spectra of electron impact ionization of compounds **III**, **IX–XIII** the peaks of the corresponding molecular ions and also the products of their fragmentation are observed.

Compounds **IX–XIII** were tested as additives in the Institute of Chemistry of Additives of the National Academy of Sciences of Azerbaijan. Experiments were carried out in the M-11 oil (GOST-9-052-75). Fungi (*Aspergillus niger, Candida tropicalis*) and bacteria (*Pseudomonas aeruginosa*) were used as test cultures.

Results of the tests are listed in the table.

As is seen from the table 2-(*N,N*-diethylaminomethoxy)-1-(ethylsulfanyl)pentane **IX**, 2-(*N,N*-dibutylaminomethoxy)-1-(ethylsulfanyl)pentane **X,** 2-piperidinomethoxy-1-(ethylsulfanyl)pentane **XI**, 2-morpholinomethoxy-1-(ethylsulfanyl)pentane **XII**, and 2-hexamethyleniminomethoxy-1-(ethylsulfanyl)pentane **XIII** exhibit bactericidal and fungicidal properties and effectively suppress the growth of microorganisms in M-11 oil in the concentration 0.25–0.5%. Compounds **IX** and **XIII** show higher efficiency than the rest substances and 8-oxyquinoline, the industrial additive

used as the reference substance. The rest substances show the effect close to that of 8-oxyquinoline.

Antimicrobial activity of compounds **IX–XIII** was also tested in comparison with such practically used substances as ethanol, phenol, chloramine, rivanol, and nitrofungine. Antimicrobial activity was studied by means of the serial dilution method. Gram-negative (*Escherichia coli, Bacillus pyocyaneus*), Gram-positive (*Staphylococcus* aureus), sporiferous (antracoid) bacteria, and yeast-like fungi (*Candida*) were used as test cultures. Obtained results showed that compounds **IX–XIII** exhibit more expressed antimicrobial activity than ethanol, phenol, rivanol, nitrofungin, and furacyllin. These substances may be recommended as antimicrobial preparations.

Hence, new representatives of aminomethoxy derivatives of 1-(ethylsylfanyl)pentane were synthesized and characterized as effective biologically active compounds.

EXPERIMENTAL

IR spectra were obtained on a UR-20 spectrometer in the range 4000-400 cm^{-1}. ^1H NMR spectra were taken on a Bruker WR-400 (400 MHz) spectrometer in $CDCl_3$. Chemical shifts were measured against TMS. Mass spectra were obtained on a VG-7070 E mass spectrometer (ionizing voltage 70 V). Chromatographic analysis of reaction mixtures and evaluation of purity of compounds synthesized was carried out on an LKhM-8MD chromatograph equipped with 3000× 3 mm steel column filled with 5% of polyethyleneglycol succinate on Dinochrom P, carrier gas helium (40 ml min^{-1}), detector katharometer, column temperature 150°C, evaporator temperature 220°C.

Antimicrobial action of compounds **IX–XIII** in the M-11 lubricant was studied on their 0.25–1% solutions in this oil. The evaluation of antimicrobial action was carried out in thermo- and moisture-control cell by means of the hole method. Experiments were carried out at 28–30°C for 2–3 days. Fungi and bacteria were used as test microorganisms.

Antimicrobial action of compounds **IX–XIII** was studied by the serial dilution method on several strains of microorganisms. Sarcopeptone agar with pH 7.2-6.4 for bacteria and the Sabourad media for fungi were used as the culture media. Dilution degree was 1:200, 1:400, 1:800, 1:1600, and 1:3200. Ethanol, phenol, chloramine, rivanol, and nitrofungin were used in these studies as reference substances with the same dilution degree. Seeding was carried out after 10, 20, 30, 40, and 60 min for the bacteria and fungi.

Results of testing the bactericidal and fungicidal properties of aminomethoxyderivatives of 1-(ethylsulfanyl)pentane **IX–XIII** in M-11 oil

Compound	Concentration of additive, %	Diameter of zone of suppression of microorganisms, mm		
		fungi		bacteria
		Aspergillus niger	*Candida tropicalis*	*Pseudomonas aeruginosa*
IX	1.0	40	36	32
	0.50	20	18	16
	0.25	15	14	13
X	1.0	4	32	36
	0.50	20	16	18
	0.25	16	15	14
XI	1.0	28	32	32
	0.50	14	16	16
	0.25	12	11	12
XII	1.0	28	32	32
	0.50	14	16	16
	0.25	14	12	11
XIII	1.0	32	32	36
	0.50	16	16	18
	0.25	11	12	13
8-Oxyquinoline	1.0	28	28	36
	0.50	14	14	16
	0.25	9	8	14
M-11 without the additive	0	+[a]	+[a]	+[a]

[a] (+)-abundant growth around the hole on the Petri cup.

Synthesis of 1-(ethylsulfanyl)pentan-2-ol (III). To a mixture of 31 g of 1-ethanethiol **I**, 20 g of sodium hydroxide, and 30 g of water 83.52 g of 1-bromopentan-2-ol **II** was added under vigorous stirring. After the addition was completed stirring was continued for 3 h. After cooling the reaction mixture was treated with 20 ml of 5% NaOH and 50 ml of benzene, the organic layer was separated, washed with 5% NaOH and with water until neutral pH, and then dried over anhydrous sodium sulfate. After removing the solvent the residue was distilled in a vacuum to give 46 g (62%) of

1-(ethylthio)pentan-2-ol **III**, bp 82–84°C (2 mm Hg), n_D^{20} 1.4746, d_4^{20} 0.9488, MR_D 43.96, calculated 44.02. IR spectrum, ν, cm^{-1}: 3500 (OH), 2930 (CH_3), 2880 (CH_2), 1200 (C–N), 740 (C–S). ^1H NMR spectrum, δ, ppm: 0.9 t (3H, CH_3^8), 1.3 t [3H, SCH_2–CH_3, $J(H^1,H^2)$ 7 Hz], 1.5 m (4H, $2CH_2$), 2.5 t (2H, $2SC^2H_2$), 2.7 s (OH), 2.75 q [2H, SC^4H_2CH, $J(H^4,H^5)$ 7.2 Hz], 3.7 m (OCH). Mass spectrum, *m/z* (I_{rel}, %): $[M + H]^+$ 149 (27), $[M]^+$ 148 (30), $[M - OH]^+$ 131 (100), 117 (8), 89 (6), 53 (30), 52 (72), 45 (15). Found, %: C 56.70, H 10.82, S 21.54. $C_7H_{15}OS$. Calculated, %: C 56.81, H 10.90, S 21.66.

Aminomethoxy derivatives of 1-(ethylsulfanyl) pentane (IX–XIII) *(general procedure).* To a mixture of 0.03 mol of 1-(ethylsulfanyl)pentan-2-ol, 0.03 mol of paraformaldehyde and 30 ml of benzene 0.03 mol of secondary amine **IV–VIII** was added dropwise with stirring at 15–20°C. Stirring was continued for extra 3–4 h at 40–45°C. After that benzene was distilled off, and the residue was distilled in a vacuum.

2-(*N,N*-Diethylaminomethoxy)-1-(ethylsulfanyl)-pentane (IX) was prepared from 4.45 g of 1-(ethylsulfanyl)pentan-2-ol **III,** 0.9 g of paraformaldehyde, and 2.19 g of diethylamine **IV**. Yield 4.7 g (67%), bp 108–110°C (1 mm Hg), n_D^{20} 1.4654, d_4^{20} 0.9098, MR_D 70.98, calculated 71.27. IR spectrum, ν, cm^{-1}: 2920 (CH_3), 2880 (CH_2), 730 (C–S). ^1H NMR spectrum, δ, ppm: 0.9 t (3H, C^7H_3), 1.2 m (9H, CH_3CH_2S, CH_3–CH_2–N–CH_2–CH_3), 1.35 m (4H, C^6H_2, C^7H_2), 2.4 m (4H, SC^2H_2, SC^3H_2), 2.6 q (4H, $2NCH_2$), 2.7 m (2H, SC^4H_2CH), 3.2 quintet (1H, OC^5H), 4.2 d.d (2H, OCH_2N). Mass spectrum (electron impact), *m/z* (I_{rel}, %): 233 (10) $[M]^+$, 162 (9) $[M - C_2H_5S]^+$, 131 (15) $[M - C_5H_{12}NO]^+$, 119 (100), 75 (60) $[C_3H_7S]^+$, 61 (20) $[C_2H_5S]^+$, $[M]^+$ calculated 233.92. Found, %: C 61.59, H 11.60, N 5.94, S 13.68. $C_{12}H_{27}NOS$. Calculated, % C 61.75, H 11.66, N 6.00, S 13.74.

2-(*N,N*-Dibutylaminomethoxy)-1-(ethylsulfanyl)-pentane (X) was prepared from 4.45 g of 1-(ethylsulfanyl)pentan-2-ol **III**, 0.9 g of paraformaldehyde, and 3.87 g of dibutylamine **V.** Yield 6.23 g (74%), bp 150–152°C (1 mm Hg), n_D^{20} 1.4630, d_4^{20} 0.8876, MR_D 89.84, calculated 89.86. IR spectrum, ν, cm^{-1}: 2930 (CH_3), 2875 (CH_2), 1200 (C–N), 730 (C–S). ^1H NMR spectrum, δ, ppm: 0.9 t (9H, $3CH_3$), 1.2 t (3H, C^1H_3), 1.4 m (12H, $6CH_2$), 2.40–2.45 m (8H, $2NCH_2$, $2SCH_2$), 3.2 quintet (1H, OCH), 4.2 d.d (2H, OCH_2N). Mass spectrum (electron impact), *m/z* (I_{rel}, %): 290 (10) $[M + H]^+$, 289 (15) $[M]^+$, 228 (20) $[M - C_2H_5S]^+$, 168 (20) $[M - C_8H_{18}N]^+$, 130 (100) $[M - C_{10}H_{21}OS]^+$, 98 (75), 58 (50). $[M]^+$ calculated 289.53. Found, %: C 66.19, H 12.11, N 4.78, S 10.98. $C_{16}H_{35}NOS$. Calculated, %: C 66.38, H 12.18, N 4.84, S 11.07.

2-(Piperidinomethoxy)-1-(ethylsulfanyl)pentane (XI) was prepared from 4.45 g of 1-(ethylsulfanyl) pentan-2-ol **III**, 0.9 g of paraformaldehyde, and 2.55 g of piperidine **VI**. Yield 5.1 g (69%), bp 127–128°C (1 mm Hg), n_D^{20} 1.4798, d_4^{20} 0.9452, MR_D 73.74, calculated 73.86. IR spectrum, ν, cm^{-1}: 2930 (CH_3), 2870 (CH_2), 1220 (C–N), 730 (C–S). ^1H NMR spectrum, δ, ppm: 0.95 t (3H, C^8H_3), 1.25 t (3H, CH_3–CH_2S), 1.3–1.4 m (10H, $5CH_2$), 2.40 m [4H, $2NCH_2$, $J(H^1,H^2)$ 10.9 Hz], 2.55 m [2H, SC^2H_2, $J(H^1,H^2)$ 7.1 Hz], 2.75 quartet [2H, SC^4H_2–CH, $J(H^4,H^5)$ 7.2 Hz] 3.3 quintet (1H, OCH), 4.2 d.d (2H, OCH_2N). Mass spectrum (electron impact), *m/z* (I_{rel}, %): 245 (10) $[M]^+$, 184 (9) $[M - C_2H_5S]^+$, 161 (100) $[M - C_5H_{10}N]^+$, 146 (15) $[C_7H_{14}OS]^+$, 75 (30). $[M]^+$ calculated 245.43. Found, %: C 63.52, H 11.02, N 5.65, S 13.02. $C_{13}H_{27}NOS$. Calculated, %: C 63.73, H 11.11, N 5.61, S 13.09.

2-(Morpholinomethoxy)-1-(sulfanyl)pentane (XII) was prepared from 4.45 g of 1-(ethylsulfanyl) pentan-2-ol **III**, 0.9 g of paraformaldehyde, and 2.61 g of morpholine **VII**. Yield 5.2 g (70%), bp 140–142°C (3 mm Hg), n_D^{20} 1.4786, d_4^{20} 0.9894, MR_D 70.74, calculated 70.98. IR spectrum, ν, cm^{-1}: 2910 (CH_3), 2840 (CH_2), 1220 (C–N), 730 (C–S). ^1H NMR spectrum, δ, ppm: 0.95 t (3H, C^8H_3), 1.3 t (3H, C^1H_3), 1.4 m (4H, $2CH_2$), 2.4-2.7 m (8H, $2NCH_{2\ morf.}$, $2SCH_2$), 3.2 m [4H, $2CH_2O_{morf}$, $J(H^3H^5)$ 12.5 Hz, $J(H^2H^6)$ 11.5 Hz], 3.5 quintet (OCH), 4.2 d.d (2H, OCH_2N). Mass spectrum (electron impact), *m/z* (I_{rel}, %): 247 (10) $[M]^+$, 186 (9) $[M - C_2H_5S]^+$, 131 (15) $[M - C_5H1_0NO_2]^+$, 82 (100). $[M]^+$ calculated 247.4. Found, %: C 58.06, H 10.11, N 5.61, S 12.88. $C_{12}H_{25}NO_2S$. Calculated, %: C 58.26, H 10.18, N 5.66, S 12.96.

2-(Hexamethyleniminomethoxy)-1(ethylsulfanyl)-pentan (XIII) was prepared from 4.45 g of ethylsulfanyl)pentan-2-ol **III**, 0.9 g of paraformaldehyde, and 2.97 g of hexanethylenimine **VIII**. Yield 5.6 g (72%), bp 132–134°C (2 mm Hg), n_D^{20} 1.4850, d_4^{20} 0.9472, MR_D 78.45, calculated 78.51. IR spectrum, ν, cm^{-1}: 2920 (CH_3), 2840 (CH_2), 1200 (C–N), 730 (C–S). ^1H NMR spectrum, δ, ppm: 0.95 t (3H, C^8H_3), 1.25 t (3H, C^1H^3), 1.4 m (12H, $6CH_2$), 2.4 m (8H, $2NCH_2$, $2SCH_2$), 3.2 quintet (1H, OCH), 4.2 d.d (2H, OCH_2N).

Mass spectrum (electron impact), *m/z* (I_{rel}, %): 259 (8) $[M]^+$, 198 (9) $[M - C_2H_5S]^+$, 146 (14) $[C_7H_{14}OS]^+$, 130 (15) $[M - C_7H_{15}NO]^+$, 113 (100) $[C_7H_{15}N]^+$, 82 (33). $[M]^+$ calculated 259.46. Found, %: C 64.71, H 11.22, N 5.36, S 12.30. $C_{14}H_{29}NOS$. Calculated, %: C 64.92, H 11.29, N 5.36, S 12.30.

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