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Synthesis of Acyloxyalkyl Esters of Thiocarbonic and Dithiocarbamic Acids

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Abstract—Reactions of acyloxyalkyl chloride with alkaline salts of alkylxanthic, butyltrithiocarbonic, and diethyldithiocarbamic acids afforded a series of acyloxyalkyl esters of various nature and positions of the acyl groups in the molecule.

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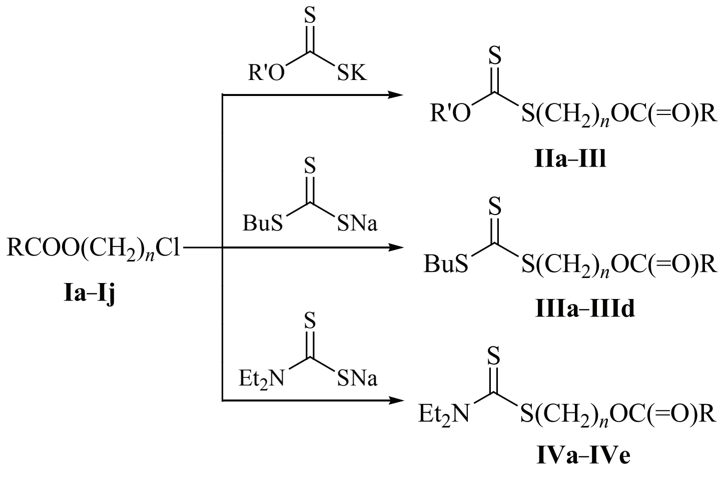
Derivatives of thiocarbonic acids are extensively used in various fields of the national economy, for example, as additives to lubricating oils, flotators, pesticides, vulcanization accelerators, stablizers and additives to the polymeric compositions [1].

In extension of our research in the field of the synthesis and investigation of thiocarbonic acids derivatives [2] we planned to synthesize a series of derivatives of these acids containing in the molecule a carboxy group to improve their lubricity.

We report here the results of the studies on the synthesis of a series of acyloxyalkyl esters of alkylxanthic, butyltrithiocarbonic, and diethyldithiocarbamic acids with a different nature and the place of the acyl group in the molecule.

Acyloxyalkyl esters of thiocarbonic and diethyldithiocarbamic acids were obtained from the acyloxyalkyl chlorides and the alkaline salts of appropriate acids.

The initial acyloxymethyl chlorides **Ia–Id** were obtained in a 80–85% yield from carboxylic acids chlorides and paraformaldehyde in the presence of zinc chloride at 45–50°C by procedure [3].



I, $n = 1$: R = Me (**a**), Et (**b**), C_7H_{15} (**c**), Ph (**d**); $n = 2$: R= Me (**e**), Et (f), Bu (**g**), Vin (**h**), $HSCH_2$ (**i**), Ph (**j**); **II**, $n = 1$: R = Me, R′ = Et (**a**), *i*-Pr (**b**), Bu (**c**); R′ = *i*-Pr, R = Et (**d**), C_7H_{15} (**e**), Ph (f); $n = 2$: R′ = *i*-Pr, R= Me (**g**), Et (**h**); R = Bu, R′ = Et (i), *i*-Pr (**j**); R′ = *i*-Pr, R = Vin (**k**), $HSCH_2$ (**l**); **III**, $n = 1$: R= Me (**a**), Ph (**b**); $n = 2$: R = Me (**c**), Ph (**d**); **IV**, $n = 1$: R = Me (**a**), Ph (**b**); $n = 2$: R = Me (**c**), Bu (**d**), Vin (**e**).

$$RCOCl + HCHO \xrightarrow{ZnCl_2} RCOOCH_2Cl$$

Ia–Id

R = Me (**a**), Et (**b**), C_7H_{15} (**c**), Ph (**d**).

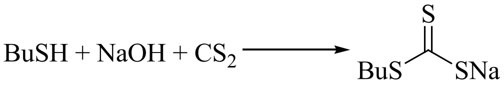
The initial acyloxyethyl chlorides **Ie–Ij** were synthesized by the esterification of carboxylic acids with ethylenechlorohydrin in the presence of KU-2 catalyst. The best yields were obtained at the ratio carboxylic acid–ethylenechlorohydrin 1.2:1 and the amount of KU-2 catalyst of 10% calculated on the acid.

$$RCOOH + ClCH_2CH_2OH \xrightarrow{KU\text{-}2} RCOOCH_2CH_2Cl$$

Ie–Ij

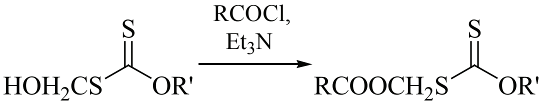
R = Me (**e**), Et (**f**), Bu (**g**), Vin (**h**), $HSCH_2$ (**i**), Ph (**j**).

The xanthates and diethyldithiocarbamate employed as the initial compounds are freely available reagents manufactured by the chemical industry. The sodium butyltrithiocarbonate necessary for the synthesis of the trithiocarbonic acids esters was obtained from sodium butylmercaptide and carbon disulfide in DMF by the known method [4] and was used further without the isolation from the solution.



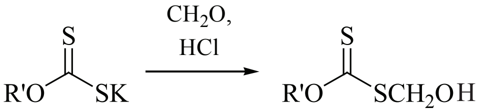
Acyloxymethyl chlorides fairly easily react with the alkaline salts of thioacids, and the nature of the substituent at the thiocarbonyl group virtually does not affect the duration of the reaction and the yield of the target compound. Because of the hydrolysis of the acyloxymethyl chlorides in water and the reactions with the other hydroxyl-containing compounds the reaction with the alkaline salts of thioacids was carried out in aprotic solvent, benzene. At the vigorous stirring of equimolar amounts of the initial substances at 40–45°C within 2–3 h the yield of target compounds reached over 85%.

Acyloxymethyl esters of xanthic acids unlike the other target compounds were also synthesized by an alternative procedure: by the reaction of carboxylic acids chlorides with hydroxymethyl esters of the xanthic acids in the presence of triethylamine.



IIa, IIb

For this reaction in their turn the starting *S*-hydroxymethyl derivatives of xanthic acids were obtained by the treatment with hydrochloric acid of the water solutions of the equimolar amounts of potassium alkylxanthate and formaldehyde by the procedure [5].



The identical refraction indices and IR spectra of acyloxymethyl xanthates with the same substituents prove the identity of compounds obtained by both methods.

Acyloxyethyl esters of thiocarbonic and diethyldithiocarbamic acids were synthesized in more severe conditions. Due to the lower reactivity of the chlorine atom in the acyloxyethyl chlorides their reactions with the thioacid salts was performed in polar solvents (2-propanol, DMF) at higher temperature, 70–75°C, over 4–5 h. The yield of the target products attained 80–90%.

The synthesized derivatives of thiocarbonic and diethyldithiocarbamic acids are light-yellow fluids, well soluble in organic solvents and mineral oils.

The composition and structure of esters synthesized were confirmed by the elemental analysis, IR and ^1H NMR spectra.

The IR spectra of all acyloxyalkyl esters of thiocarbonic acids contain strong absorption bands in the region 1750–1740 cm^{-1} characteristic of the C=O group.

The other specific groups of the acyl fragment give rise to absorption bands in their appropriate frequency regions [6]: The double bond absorption of the acryloyl group is observed in the region 1650–1620 cm^{-1}, the vibrations of the aromatic ring of the benzoyl group appear as a strong band at 1600–1590 cm^{-1}, to the S–H bond of the sulfhydryl group belongs a weak band in the region 2600–2550 cm^{-1}.

The characteristic functional group of all synthesized derivatives of thiocarbonic acids is the C=S group whose stretching vibrations appear in the xanthate spectra as strong bands at 1060–1030 cm^{-1}, in those of trithiocarbonates at 1058–1053 cm^{-1}, of dithiocarbamates, at 990 cm^{-1} in agreement with the published data [6, 7]. The other characteristic absorptions of the xanthate fragments are strong bands in the regions 1250–1235 and

1150–1090 cm^{-1} belonging to ester bonds.

The bands characteristic of amide bonds of dithiocarbamates, often called NCS-bands [6–8] were observed at 1570–1395 and 1420–1260 cm^{-1}.

The absorption bands of the ordinary C–S bonds are known to be weak [6] and due to the uncertainty of their position are as a rule seldom used for the identification. Yet in the spectra of trithiocarbonates beside the absorption band of the C=S group another strong band is observed in the range of 825–790 cm^{-1}. The appearance of this band cannot be ascribed to the vibrations of the bonds C–C, C–H or C–O whose spectral range is well known. It can be positively stated that this band originates just from the structure of the fragment –SC(=S)S– since in the xanthate sperctra where this structure is lacking the band does not appear.

^{1}H NMR spectra of acyloxyalkyl esters of thiocarbonic acids were studied by the examples of acetyloxyalkyl esters of isopropylxanthic **IIb, IIg**, butyltrithiocarbonic **IIIa, IIIc**, and *N*,*N*-diethyldithio-carbamic **IVa, IVc** acids. The acetyl protons in the spectra of all six compounds appear as a singlet at 2.0 ppm.

The methylene protons of the OCH_2S fragment of the acetyloxymethyl esters of thiocarbonic acids **IIb, IIIa, IVa** also give rise to singlets, but in a weaker field, around 5.65 ppm. The protons of the methylene group in the acetoxyethyl esters of the thiocarbonic acids **IIg, IIIc, IVc** are observed at 4.13–4.19 (OCH_2) and 3.25–3.55 ppm (SCH_2). The proton signals of *O*, *S* , and *N*-substituents in the ^{1}H NMR spectra of the derivatives of xanthic, butyltrithiocarbonic, and diethyldithiocarbamic acids appear in the following regions (δ, ppm): OPr-*i*: 1.42 d (6H, $2CH_3$), 5.45 m (1H, CH); SBu: 0.99 t (3H, CH_3), 1.50 m (4H, CH_2CH_2), 3.22 t (2H, SCH_2); NEt_2: 1.30 t (6H, $2CH_3$), 3.80 m (4H, $2CH_2$).

EXPERIMENTAL

IR absorption spectra were recorded on a spectrophotometer Specord 75IR from thin film. ^{1}H NMR spectra were registered on a spectrometer Varian T-60 (60 MHz) in CCl_4, internal reference TMS. The purification and isolation of compounds synthesized was commonly performed by vacuum distillation, and the compounds decomposing at the distillation were purified by absorption column chromatography on a column 1.5 m high consisting of three sections of diameters 4, 3, and 1.5 cm. Silica gel ACK was used as sorbent, eluent benzene–2-propanol, 4 : 1. Eluates were evaporated in a vacuum. Reaction product was controlled by the refraction index.

Acyloxymethyl chlorides Ia–Id were obtained by adding gradually at room temperature 0.2 mol of paraformaldehyde and 0.4 g of zinc chloride to 0.2 mol of carboxylic acid chloride. The reaction mixture was stirred at 45–50°C till paraformaldehyde completely dissolved, the precipitate of zinc chloride was filtered off, and the filtrate was distilled in a vacuum.

Acetoxymethyl chloride (Ia). Yield 18.5 g (85%), bp 102–103°C (760 mm Hg), d_4^{20} 1.2010, n_D^{20} 1.4210. MR_D 22.92, calculated 22.57. Found, %: Cl 32.56. $C_3H_5ClO_2$. Calculated, %: Cl 32.71.

Propionyloxymethyl chloride (Ib). Yield 20.8 g (85%), bp 106–107°C (760 mm Hg), d_4^{20} 1.1481 (1.1824 [3]), n_D^{20} 1.4221 (1.4295 [3]). MR_D 27.12, calculated 27.19. Found, %: Cl 29.01. $C_4H_7ClO_2$. Calculated, %: Cl 28.98.

Octanoyloxymethyl chloride (Ic). Yield 31.2 g (81%), bp 114–115°C (760 mm Hg), d_4^{20} 1.0109, n_D^{20} 1.4330. MR_D 49.55, calculated 50.28. Found, %: Cl 18.32. $C_9H_{17}ClO_2$. Calculated, %: Cl 18.44.

Benzoyloxymethyl chloride (Id). Yield 27.3 g (80%), bp 92–93°C (760 mm Hg), d_4^{20} 1.2342, n_D^{20} 1.5276. MR_D 42.54, calculated 42.06. Found, %: Cl 20.20. $C_8H_7ClO_2$. Calculated, %: Cl 20.82.

Acyloxyethyl chlorides Ie–Ij were obtained by esterification of 0.24 mol of carboxylic acid with 0.2 mol of ethylenechlorohydrin in boiling benzene in the presence of cation exchanger KU-2 taken in amount of 10% with respect to the acid. The reaction was stopped after the theoretical quantity of water was azeotropically distilled. Then the reaction mixture was washed in succession with a solution of $NaHCO_3$ and with water till neutral washings, filtered, benzene was distilled off, and the residue was subjected to vacuum distillation.

2-(Acetoxy)ethyl chloride (Ie). Yield 19.8 g (81%), bp 43°C (10 mm Hg), d_4^{20} 1.1553 (1.1700 [9]), n_D^{20} 1.4240 (1.4247 [9]). MR_D 27.07, calculated 27.19. Found, %: Cl 22.85. $C_4H_7ClO_2$. Calculated, %: Cl 23.58.

2-(Propionyloxy)ethyl chloride (If). Yield 21 g (77%), bp 59°C (10 mm Hg), d_4^{20} 1.0824, n_D^{20} 1.4205. MR_D 31.93, calculated 31.81. Found, %: Cl 26.10. $C_5H_9ClO_2$. Calculated, %: Cl 26.00.

2-(Valeroyloxy)ethyl chloride (Ig). Yield 25 g (76%), bp 72°C (10 mm Hg), d_4^{20} 1.0284, n_D^{20} 1.4180. MR_D 40.34, calculated 41.05. Found, %: Cl 21.62. $C_7H_{13}ClO_2$. Calculated, %: Cl 21.58.

2-(Acryloyloxy)ethyl chloride (Ih). Yield 18.2 g (68%), bp 60°C (10 mm Hg), d_4^{20} 1.1491, n_D^{20} 1.4490. MR_D 31.41, calculated 31.34. Found, %: Cl 26.27. $C_5H_7ClO_2$. Calculated, %: Cl 26.39.

2-(Mercaptoacetoxy)ethyl chloride (Ii). Yield 23.2 g (75%), bp 70°C (10 mm Hg), d_4^{20} 1.3099, n_D^{20} 1.5000. MR_D 34.72, calculated 35.00. Found, %: Cl 22.91; S 20.68. $C_4H_7ClSO_2$. Calculated, %: Cl 23.05; S 20.71.

2-(Benzoyloxy)ethyl chloride (Ij). Yield 27.7 g (75%), mp 107°C (from isooctane). Found, %: Cl 18.91. $C_9H_9ClO_2$. Calculated, %: Cl 19.24.

Acyloxymethyl esters of alkylxanthic acids IIa–IIf. To a slurry of 0.2 mol potassium alkylxanthate in benzene at room temperature was added 0.2 mol of acyloxymethyl chloride, and the mixture was stirred for 2–3 h at 40–45°C. The reaction mixture was washed with water, filtered, benzene was distilled off, and the residue was subjected to vacuum distillation.

Acetoxymethyl ester of ethylxanthate (IIa). Yield 35 g (90%), bp 77–78°C (0.4 mm Hg), d_4^{20} 1.2089, n_D^{20} 1.5252. MR_D 49.26, calculated 49.78. Found, %: C 36.84; H 5.69; S 32.45. $C_6H_{10}O_3S_2$. Calculated, %: C 37.11; H 5.15; S 32.99.

Ester **IIa** was obtained also by an alternative method: To 30.4 g (0.2 mol) of hydroxymethyl ester of ethylxanthic acid in 100 ml of benzene at 5°C was added 20.2 g (0.2 mol) of triethylamine, then 15.7 g (0.2 mol) of acetyl chloride. The reaction mixture was stirred for 30 min at 20°C and 2 h at 35°C. The precipitate was filtered off, the filtrate was washed with water, filtered, benzene was distilled off, and the residue was subjected to vacuum distillation. Yield 34.9 g (90%).

The physical constants and the data of the elemental analysis showed the identity of compounds obtained by two methods.

Acetoxymethyl ester of isopropylxanthic acid (IIb). Yield 37 g (89%), bp 96–98°C (0.5 mm Hg), d_4^{20} 1.1624, n_D^{20} 1.5250. MR_D 54.91, calculated 54.97. Found, %: C 40.21; H 5.65; S 29.98. $C_7H_{12}O_3S_2$. Calculated, %: C 40.28; H 5.77; S 30.77.

Acetoxymethyl ester of butylxanthic acid (IIc). Yield 39 g (88%), bp 102–103°C (0.5 mm Hg), d_4^{20} 1.1384, n_D^{20} 1.5152. MR_D 58.89, calculated 58.60. Found, %: C 42.98; H 6.28; S 28.69. $C_8H_{14}O_3S_2$. Calculated, %: C43.24; H 6.36; S 28.82.

Propionyloxymethyl ester of isopropylxanthic acid (IId). Yield 38.2 g (86%), bp 98–101°C (0.5 mm Hg), d_4^{20} 1.1293, n_D^{20} 1.5150. MR_D 59.37, calculated 59.60. Found, %: C 43.02; H 6.23; S 28.34. $C_8H_{14}O_3S_2$. Calculated, %: C 43.24; H 6.36; S 28.82.

Octanoyloxymethyl ester of isopropylxanthic acid (IIe). Yield 50.2 g (86%). The compound was purified by chromatography. $d_4{}^{20}$ 1.0506, n_D^{20} 1.5025. MR_D 82.20, calculated 82.68. Found, %: C 53.14; H 8.17; S 21.70. $C_{13}H_{24}O_3S_2$. Calculated, %: C 53.42; H 8.22; S 21.91.

Benzoyloxymethyl ester of isopropylxanthic acid (IIf). Yield 47.00 g (87%). The compound was purified by chromatography. $d_4{}^{20}$ 1.1911, n_D^{20} 1.5778. MR_D 75.28, calculated 74.46. Found, %: C 53.10; H 5.02; S 22.93. $C_{12}H_{14}O_3S_2$. Calculated,%: C 53.33; H 5.17; S 23.70.

Acyloxyethyl esters of alkylxanthic acids IIg–IIl. To a mixture of 0.2 mol potassium alkylxanthate in 50 ml of isopropyl alcohol was added 0.2 mol of acyloxyethyl chloride, the reaction mixture was stirred for 4 h at 70–75°C, washed with water, filtered, and the filtrate was subjected to vacuum distillation.

2-(Acetoxy)ethyl ester of isopropylxanthic acid (IIg). Yield 39 g (88%), bp 100–100.5°C (0.4 mm Hg), d_4^{20} 1.1450, n_D^{20} 1.5228. MR_D 59.29, calculated 59.58. Found, %: C 42.85; H 6.76; S 28.78. $C_8H_{14}O_3S_2$. Calculated, %: C 43.22; H 6.35; S 28.85.

2-(Propionyloxy)ethyl ester of isopropylxanthic acid (IIh). Yield 40.6 g (86%), bp 110–111°C (0.4 mm Hg), $d_4{}^{20}$ 1.1287, n_D^{20} 1.5220. MR_D 63.86, calculated 64.21. Found, %: C 45.20; H 7.07; S 27.78. $C_9H_{16}O_3S_2$. Calculated, %: C 45.76; H 6.78; S 27.11.

2-(Valeroyloxy)ethyl ester of ethylxanthic acid (IIi). Yield 42.5 g (85%), bp 116–117°C (0.4 mm Hg), d_4^{20} 1.1075, n_D^{20} 1.5128. MR_D 68.92, calculated 67.82. Found, %: C 47.79; H 7.31; S 25.78. $C_{10}H_{18}O_3S_2$. Calculated, %: C 48.00; H 7.20; S 25.60.

2-(Valeroyloxy)ethyl ester of isopropylxanthic acid (IIj). Yield 46.4 g (84%), bp 129°C (0.4 mm Hg), d_4^{20} 1.0833, n_D^{20} 1.5040. MR_D 72.27, calculated 72.26. Found, %: C 49.25; H 7.78; S 23.98. $C_{11}H_{20}O_3S_2$. Calculated, %: C 49.97; H 7.63; S 24.26.

2-(Acryloyloxy)ethyl ester of isopropylxanthic acid (IIk). Yield 38.4 g (82%), bp 121°C (0.45 mm Hg), d_4^{20} 1.1609, n_D^{20} 1.5337. MR_D 62.70, calculated 63.74.

Found, %: C 46.28; H 5.88; S 27.22. $C_9H_{14}O_3S_2$. Calculated, %: C 46.15; H 5.98; S 27.34.

2-(Mercaptoacetoxy)ethyl ester of isopropylxanthic acid (III). Yield 36.8 g (83%), bp 176°C (0.45 mm Hg), d_4^{20} 1.2570, n_D^{20} 1.5740. MR_D 66.77, calculated 67.40. Found, %: C 37.83; H 6.18; S 37.65. $C_8H_{14}O_3S_3$. Calculated, %: C 37.78; H 5.56; S 37.82.

Acetoxymethyl ester of butyltrithiocarbonic acid (IIIa). To 18 g (0.2 mol) of butylmercaptane in 50 ml of DMF was slowly added 8 g (0.2 mol) of sodium hydroxide, and the mixture was stirred for 2.5 h at 60–70°C. Then at 20–25°C 19 g (0.25 mol) of carbon disulfide was added, 21.7 g (0.2 mol) of acetoxymethyl chloride was added by portions, and the mixture was stirred for 2–3 h at 40–45°C. The reaction mixture was washed with water, filtered, the solvent was distilled off, and the residue was distilled in a vacuum. Yield 40 g (85%), bp 129–130°C (0.5 mm Hg), d_4^{20} 1.1648, n_D^{20} 1.5652. MR_D 66.68, calculated 66.62. Found, %: C 40.32; H 5.93; S 40.38. $C_8H_{14}O_2S_3$. Calculated, %: C 40.33; H 5.92; S 40.33.

Benzoyloxymethyl ester of butyltrithiocarbonic acid (IIIb) was obtained similarly. Yield 51.6 g (86%). The compound was purified by chromatography. d_4^{20} 1.1729, n_D^{20} 1.5940. MR_D 86.83, calculated 86.11. Found, %: C 51.75; H 5.15; S 31.10. $C_{13}H_{16}O_2S_3$. Calculated, %: C 52.00; H 5.33; S 31.96.

Acyloxyethyl esters of butyltrithiocarbonic acid IIIc, IIId were obtained and isolated analogously to the acyloxymethyl esters with the only difference that after the addition of the acetoxyethyl chloride the reaction mixture was stirred for 4–5 h at 75–80°C.

2-(Acetoxy)ethyl ester of butyltrithiocarbonic acid (IIIc). Yield 43.8 g (87%), bp 105–106°C (0.45 mm Hg), d_4^{20} 1.1507, n_D^{20} 1.5600. MR_D 70.91, calculated 71.24. Found, %: C 42.38; H 6.77; S 37.52. $C_9H_{16}O_2S_3$. Calculated, %: C 42.85; H 6.35; S 38.09.

2-(Benzoyloxy)ethyl ester of butyltrithiocarbonic acid (IIId). Yield 54 g (86%). The compound was purified by chromatography. d_4^{20} 1.1759, n_D^{20} 1.5900. MR_D 90.26, calculated 90.72. Found, %: C 53.90; H 5.62; S 29.80. $C_{14}H_{18}O_2S_3$. Calculated, %: C 53.50; H 5.73; S 30.57.

Acetoxymethyl ester of diethyldithiocarbamic acid (IVa). To a mixture of 45 g (0.2 mol) of sodium diethyldithiocarbamate trihydrate in 50 ml of benzene was added 21.7 g (0.2 mol) of acetoxymethyl chloride, and the mixture was stirred for 2–3 h at 40–45°C. The reaction mixture was washed with water, filtered, benzene was distilled off, and the residue was subjected to vacuum distillation. Yield 37.6 g (85%), bp 130–131°C (0.6 mm Hg), d_4^{20} 1.1646, n_D^{20} 1.5567. MR_D 61.12, calculated 60.98. Found, %: C 43.31; H 7.32; N 6.30 S 28.90. $C_8H_{15}NO_2S_2$. Calculated, %: C 43.42; H 6.80; N 6.33; S 28.95.

Benzoyloxymethyl ester of diethyldithiocarbamic acid (IVb) was obtained similarly. Yield 48.7 g (86%), white crystals, mp 39–40°C (from heptane). Found, %: C 55.18; H 6.20; N 4.90; S 22.59. $C_{13}H_{17}NO_2S_2$. Calculated, %: C 55.12; H 6.00; N 4.94; S 22.61.

Acyloxyethyl esters of diethyldithiocarbamic acid IVc–IVe were obtained in the same way as the acyloxymethyl esters, but the reaction was carried out in DMF at 75–80°C over 4–5 h.

Acetoxyethyl ester of diethyldithiocarbamic acid (IVc). Yield 41.3 g (88%), bp 128°C (0.5 mm Hg), d_4^{20} 1.1346, $n_D{}^{20}$ 1.5518. MR_D 66.25, calculated 65.60. Found, %: C 45.79; H 7.39; N 6.12; S 26.9. $C_9H_{17}NO_2S_2$. Calculated, %: C 45.95; H 7.23; N 5.95; S 27.23.

2-(Valeroyloxy)ethyl ester of diethyldithiocarbamic acid (IVd). Yield 47.6 g (86%), bp 154°C (0.4 mm Hg), d_4^{20} 1.0784, $n_D{}^{20}$ 1.5338. MR_D 79.93, calculated 79.45. Found, %: C 51.83; H 8.49; N 5.15; S 22.95. $C_{12}H_{23}NO_2S_2$. Calculated, %: C 51.98; H 8.33; N 5.05; S 23.10.

2-(Acryloyloxy)ethyl ester of diethyldithiocarbamic acid (IVe). Yield 42 g (85%). The compound was purified by chromatography. d_4^{20} 1.1395, n_D^{20} 1.5645. MR_D 70.65, calculated 69.75. Found, %: C 48.67; H 6.79; N 5.55; S 25.81. $C_{10}H_{17}O_2S_2$. Calculated, %: C 48.57; H 6.88; N 5.66; S 25.91.

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