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Alkylation of Pyrimidine Derivatives with Chloroacetic Acid Esters

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Abstract — Alkylation of 6-methyluracil, 5-hydroxy-6-methyluracil, and 6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-ylammonium sulfate with isopropyl and ethyl chloroacetates afforded previously unknown alkyl 2-(6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-1-yl)acetates, alkyl 2-(1-alkoxycarbonylmethyl-6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-3-yl)acetates, 1,3-bis(alkoxycarbonylmethyl)-6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-ylammonium sulfates, and alkyl 2-[1,3-bis(alkoxycarbonylmethyl)-6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yloxy]acetates.

Vainilavichyus and Syadyaryavichyute [1] showed that the regioselectivity in the alkylation of 2-alkylsulfanyl-4-hydroxypyrimidines with methyl bromoacetate depends on the solvent nature. In nonpolar and weakly polar aprotic solvents, such as dioxane, tetrahydrofuran, and carbon tetrachloride, only the corresponding N^3-substituted products are formed. An exception is diethyl ether, where a mixture of O- and N^3-alkylated isomers is formed. A considerable increase in the fraction of *O*-alkyl isomers is observed in dipolar aprotic solvents. Mixtures of O- and N^3-substituted derivatives are obtained in acetone and acetonitrile, while in DMF and hexamethylphosphoramide (HMPA) the alkylation occurs exclusively at the oxygen atom. Thus, the regioselectivity in the alkylation of 2-alkylsulfanyl-4-hydroxypyrimidine sodium salts with methyl bromoacetate can be controlled by variation of the solvent. It was also shown that the reaction direction depends on the temperature. Reactions of 4-hydroxypyrimidines with methyl bromoacetate at 10–60°C involve only the oxygen atom. At elevated temperatures, mixtures of N- and O-substituted isomers are formed. According to the data of [2], the alkylation of fluorouracil with ethyl 2-chloropropanoate in DMF occurs at the N^1 atom. Reznik and co-workers [3, 4] found that disodium *N*-benzyl- and *N*-methylisocyanurates are alkylated with β,β'-dichlorodiethyl ether and β,β'-dibromodiethyl ether in DMF at the nitrogen atom; the alkylation of pyrimidine derivatives with dibromoalkanes follows an analogous pattern.

We previously showed [5–9] that pyrimidine derivatives exhibit immunotropic, antiphlogistic, membrane-stabilizing, and antiradical activity. For example, hydroxymethyluracil was licensed for wide application in medicine and large-scale manufacture. With the goal of searching for new pyrimidine derivatives as potential immunotropic and antiphlogistic agents, we studied the alkylation of 6-methyluracil (**I**), 5-hydroxy-6-methyluracil (**II**), and 6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-ylammonium sulfate (**III**) with isopropyl chloroacetate (**IV**) and ethyl chloroacetate (**V**). The reactions were carried out in DMF in the presence of tetrabutylammonium bromide as phase-transfer catalyst on heating on a boiling water bath. The alkylation of 6-methyluracil (**I**) of isopropyl chloroacetate (**IV**) in the presence of potassium carbonate and tetrabutylammonium bromide (reactant molar ratio 1 : 2 : 3 : 0.03) afforded 13% of isopropyl 2-(6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-1-yl)acetate (**VI**) and 82% of isopropyl 2-(1-isopropoxycarbonylmethyl-6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-3-yl)acetate (**VII**). Analogous reaction with 5-hydroxy-6-methyluracil (**II**) (ratio **II** : **IV** : K_2CO_3 : Bu_4NBr = 1 : 4 : 4 : 0.03) gave 87% of isopropyl 2-[1,3-bis(isopropoxycarbonylmethyl)-6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yloxy]acetate (**VIII**). Thus the alkylation of **I** with isopropyl chloroacetate occurs at positions *1* and *3*, while compound **II** undergoes alkylation at N^1, N^3, and 5-O.

The structure of the products was proved by elemental analysis and IR, UV, and ^1H and ^{13}C NMR spectroscopy. Also, their spectral data were compared with those for the O- and N^3-isomers described in [1]. The UV spectra of the products contained absorption

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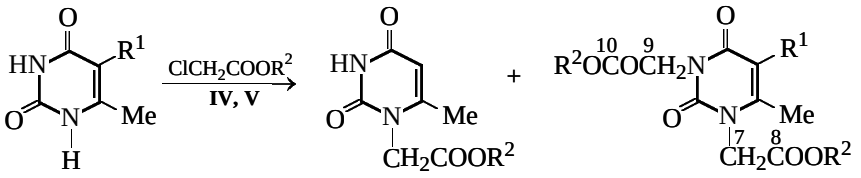

I–III **VI, X** **VII–IX, XI–XIII**

I, VII, XI, R^1 = H; **II**, R^1 = OH; **III, IX, XIII**, R^1 = OSO_3NH_4; **VIII**, R^1 = $OC_{11}H_2C_{12}OOPr$-*i*; **XII**, R^1 = OCH_2COOEt; **IV, VI, VIII, IX**, R^2 = *i*-Pr; **V, X, XI–XIII**, R^2 = Et.

bands in the region 266–272 nm. The absence of a red shift at pH 7 and 14 indicates that the monoalkylation of 6-methyluracil occurs at the N^1 atom (cf. [2]). In the IR spectra of all the products we observed absorption bands at 1640, 1660–1680, and 1720 cm^{-1}, belonging to vibrations of the uracil ring (the lactam carbonyl band 1620–1720 cm^{-1} was clearly defined), strong bands at 1228–1284 cm^{-1}, which are typical of acetates, and ester carbonyl absorption at 1736–1760 cm^{-1}.

The ^1H NMR spectra of compounds **VI–XIII** contained signals from the methyl group on C^6 (δ 2.1–2.4 ppm) and singlets from the N^1CH_2 (δ 3.9–4.5 ppm) and N^3CH_2 protons (δ 4.2–4.6 ppm). In the ^{13}C NMR spectra of compounds **VIII** and **XII** having an OCH_2COOR group in position *5*, the C^5 signal is displaced downfield by 20–25 ppm, while the C^6 signal is displaced upfield by 10–15 ppm, relative to the corresponding signals of the compounds having no substituent on C^5. The chemical shifts of the carbonyl carbon atoms are highly characteristic and are rarely overlapped by signals of the other functional groups; they fall into the δ_C range from 150 to 210 ppm. Carbon atoms of the pyrimidine ring give signals at δ_C 158–162 (C^4=O), 150–158 (C^2=O), 151.3–153.9 (C^6), 100.4–101.75 (C^5), and 13.6–20.0 ppm (6-CH_3).

EXPERIMENTAL

The ^1H and ^{13}C NMR spectra were recorded on a Bruker AM spectrometer at 300 (^1H) and 75.7 MHz (^{13}C) from 1% (^1H) and 10–20% (^{13}C) solutions in $CDCl_3$. The chemical shifts were measured relative to HMDS. The IR spectra were obtained on a UR-20 spectrophotometer with NaCl and LiF prisms; samples were prepared as films or mulls in mineral oil. The melting points were determined on a Boetius device. The elemental compositions were determined on an M-185B CHN Analyzer. The progress of reactions and the purity of products were monitored by TLC on Silufol UV-254 plates using ethanol–aqueous ammonia (4 : 1) as eluent. Spots were detected under UV light (λ 254 nm) or by treatment with iodine vapor. The UV spectra were recorded on a Specord M-400 spectrophotometer in the λ range from 200 to 350 nm from 0.00001% solutions using 10-mm cells.

Isopropyl 2-(6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-1-yl)acetate (VI) and isopropyl 2-(1-isopropoxycarbonylmethyl-6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-3-yl)acetate (VII). Dimethylformamide, 300 ml, and isopropyl chloroacetate, 100 ml, were added to 33 g of 6-methyluracil, 70 g of K_2CO_3, and 5 g of tetrabutylammonium bromide. The mixture was heated for 4 h on a boiling water bath, DMF was distilled off, the precipitate was extracted into chloroform (2 × 100 ml), and the solvent was distilled from the extract under reduced pressure to obtain 112 g of a mixture of compounds **VI** and **VII** which crystallized on storage. By fractional crystallization from alcohol we isolated 7.6 g (13%) of compound **VI**, mp 190–192°C. The product is readily soluble in alcohol, acetone, and chloroform and poorly soluble in water and hexane. R_f 0.63. UV spectrum, nm: pH 7: λ_{min} 231.75, λ_{max} 263.74; pH 14: λ_{min} 241.7, λ_{max} 266.00. IR spectrum, ν, cm^{-1}: 996, 1024 [ω(C=C)]; 1112 (COC); 1024–1232 (=N); 1228–1256 (OCO, acetate); 1376, 1384 [δ_s(*gem*-CH_3)]; 1624, 1708 (C=O, =NC=O); 1736 (C=O, ester); 3048 (C=C); 3193 (NH); 1232 (RCO_2R). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.28 d [6H, $(CH_3)_2CH$, *J* = 6 Hz], 2.17 s (3H, 6-CH_3), 4.55 s (2H, N^1CH_2), 5.09 sext [1H, $CH(CH_3)_2$, *J* = 6 Hz], 5.69 s (1H, 5-H), 9.61 s (1H, NH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 19.60 (6-CH_3), 21.73 [$(CH_3)_2CH$], 45.102 (C^7, N^1CH_2), 70.255 (OCH), 102.52 (C^5), 151.90 (C^6), 153.17 (C^2=O), 162.77 (C^4=O), 167.19 (O=CO, C^8). Found, %: C 45.80; H 6.50; N 10.90. $C_{10}H_{14}N_2O_4 \cdot 2H_2O$. Calculated, %: C 45.80; H 6.92; N 10.68.

Isopropyl 2-(1-isopropoxycarbonylmethyl-6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-3-yl)-acetate (VII) was isolated in 82% yield by crystallization of the mixture of compounds **VI** and **VII**, obtained as described above; mp 98–100°C. The product is readily soluble in alcohol, acetone, and chloroform and poorly soluble in water and hexane. R_f 0.63. IR

spectrum, ν, cm^{-1}: 952, 1040 [ω(C=C)]; 1122 (COC); 1040–1232 (=N); 1228–1256 (OCO, acetate); 1376, 1384 [δ_s(*gem*-CH_3)]: 1672, 1690, 1702, 1720, 1728 (C=O, =NC=O, CO_2); 2856, 2928, 2949, 2952 (CH_2, CH_3); 3048 (=CCH); 1232 (CO_2R). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.25 d.d [12H, 2(CH_3)$_2$CH, J = 6 Hz], 2.17 s (3H, 6-CH_3), 4.54 s (2H, N^1CH_2), 4.59 s (2H, N^3CH_2), 5.05 octet [2H, $CH(CH_3)_2$, J = 6 Hz], 5.67 s (1H, 5-H). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 19.88 (6-CH_3), 21.71 (CH_3), 42.34 (N^1CH_2), 45.99 (N^3CH_2), 69.30 (OCH), 70.16 (OCH), 101.80 (C^5), 151.97 (C^6), 153.17 (C^2=O), 161.36 (C^4=O), 167.19 (O=C^8,10O). Found, %: C 50.30; H 6.79; N 7.80. $C_{15}H_{22}N_2O_6 \cdot 2H_2O$. Calculated, %: C 50.30; H 6.20; N 7.90.

Isopropyl 2-[1,3-bis(isopropoxycarbonylmethyl)-6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yloxy]acetate (VIII). Dimethylformamide, 300 ml, was added to 14.2 g of hydroxymethyluracil **II**, 70 g of K_2CO_3, and 3 g of tetrabutylammonium bromide, and 63 ml of isopropyl chloroacetate was then added in 10-ml portions. The mixture was heated for 3 h on a boiling water bath, DMF was distilled off, the residue was dissolved in 200 ml of water, the solution was treated with chloroform (2 × 100 ml), the alkaline extract was neutralized with concentrated hydrochloric acid (1 ml) and washed with water (2 × 50 ml), and the solvent was distilled off. The residue was a light yellow thick liquid which crystallized on storage. Yield 38.5 g (87%), R_f 0.71. IR spectrum, ν, cm^{-1}: 768, 784, 826, 948–1048 [ω(C=C)]; 1104, 1132, 1144, 1152, 1176–1280 (OCO); 1380 [$\delta_s(CH_3)$]; 1048–1296 (=N); 1640, 1648–1744 (C=O, =NC=O, CO_2); 3048 (=CCH); 1232 (CO_2R). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.23 d.d.d [18H, 3(CH_3)$_2$CH, J = 6 Hz], 2.30 s (3H, 6-CH_3), 2.87 m (2H, OCH_2), 4.56 s (2H, N^1CH_2), 4.64 s (2H, N^3CH_2), 5.07 octet [3H, $CH(CH_3)_2$, J = 6 Hz]. ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 13.70 (6-CH_3), 21.74 (CH_3), 42.60 (C^7, N^1CH_2), 48.38 (N^3CH_2), 68.32 (C^{11}, OCH_2), 68.72 (OCH), 69.41 (OCH), 70.12 (OCH), 130.62 (C^5), 142.00 (C^6), 150.69 (C^2=O), 158.31 (C^4=O), 165.00 (O=C^8O), 165.98 (O=C^{10}O), 169.98 (O=C^{12}O). Found, %: C 52.30; H 6.60; N 6.30. $C_{20}H_{30}N_2O_9 \cdot H_2O$. Calculated, %: C 52.17; H 7.00; N 6.08.

1,3-Bis(isopropoxycarbonylmethyl)-6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-ylammonium sulfate (IX). Tetrabutylammonium bromide, 2 g, and potassium carbonate, 14 g, were added to 12 g of 6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-ylammonium sulfate in 150 ml of DMF, and 21 g of isopropyl chloroacetate was then added in succession. The mixture was heated for 6 h on a boiling water bath, the solvent was distilled off, and the residue was diluted with 100 ml of water and extracted with chloroform (3 × 100 ml). The solvent was distilled off from the extract to obtain 7.5 g of compound **IX**. The aqueous phase was concentrated, and the crystals were filtered off to obtain an additional portion of the product, 10 g. Overall yield 17.5 g (80%). IR spectrum, ν, cm^{-1}: 1104, 1120 (COC); 1144–1268 (=N); 1226 (COOR); 1376 [δ_s(*gem*-CH_3)]; 1464 (CH_2, CH_3); 1656, 1688, 1744 (C=O, =NC=O, CO_2); 2936, 2952, 2984 (CH_2, CH_3); no absorption at 3192 cm^{-1} was observed, indicating that the product is substituted at both N^1 and N^3. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.28 d.d [12H, $(CH_3)_2$CH, J = 6 Hz], 2.2 s (3H, 6-CH_3), 4.5 s (2H, N^1CH_2), 4.6 s (2H, N^3CH_2), 5.10 octet [2H, $CH(CH_3)_2$]. ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 19.00 (6-CH_3), 21.25 (CH_3), 42.60 (N^1CH_2), 47.30 (N^3CH_2), 68.80 (OCH), 70.16 (OCH), 102.00 (C^5), 152.07 (C^6), 153.70 (C^2=O), 161.36 (C^4=O), 167.19 (O=$C^{8,10}$O). Found, %: C 40.70; H 5.60; N 9.70; S 7.00. $C_{15}H_{25}N_3O_{10}S$. Calculated, %: C 41.00; H 5.70; N 9.56; S 7.30.

Ethyl 2-(6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-3-yl)acetate (X) and ethyl 2-(1-ethoxycarbonylmethyl-6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-3-yl)acetate (XI). Ethyl chloroacetate, 25 ml, was added in portions to a mixture of 12.6 g of 6-methyluracil, 27 g of K_2CO_3, and 3 g of tetrabutylammonium bromide in 50 ml of dioxane. The mixture was stirred for 4 h at 40–75°C and was left overnight. It was then diluted with 300 ml of water, and the precipitate was filtered off. The filtrate was evaporated to obtain 22 g (74%) of a thick liquid which was treated with DMF and acetone. The crystals (compound **X**) were filtered off. Yield 6.5 g, mp 220–222°C, R_f 0.69. UV spectrum, nm: pH 7: λ_{min} 238.6, λ_{max} 271.0; pH 14: λ_{min} 240.00, λ_{max} 272.0. IR spectrum, ν, cm^{-1}: 544, 632, 820, 850, 1050 [ω(C=C)]; 1050–1240 (=N); 1160, 1168, 1204, 1268, 1284 (OCO); 1380 [δ_s(*gem*-CH_3)]; 1624, 1640, 1672, 1704, 1728 (C=O, =NC=O, OCO); 2864, 2960 (CH_2, CH_3); 3256, 3384 (H_2O). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.1 t (3H, CH_3, J = 7.1 Hz), 2.40 s (3H, 6-CH_3), 4.0 s (2H, N^1CH_2), 4.30 t (2H, OCH_2, J = 7.1 Hz), 5.07 s (1H, 5-H), 9.4 s (1H, NH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 13.40 (CH_2CH_3), 17.13 (6-CH_3), 43.90 (C^7, N^1CH_2), 61.30 (CH_2CH_3), 99.00 (C^5), 151.50 (C^6), 155.30 (C^2=O), 161.40 (C^4=O), 163.70 (O=C^8O). Found, %: C 44.14; H 4.78; N 9.00. $C_9H_{12}N_2O_4$. Calculated, %: C 43.55; H 6.50; N 11.28.

The solvent was distilled off from the mother liquor to isolate 13.2 g of compound **XI** as a thick liquid. The product is readily soluble in water and poorly soluble in acetone and DMF. R_f 0.69. IR spectrum, ν, cm^{-1}: 544, 632, 820, 850, 1050 [ω(C=C)];

1050–1240 (=N); 1160, 1168, 1204, 1268, 1284 (OCO); 1380 [δ_s(*gem*-CH_3)]; 1624, 1640, 1672, 1704, 1728 (C=O, =NC=O, OCO); 2864, 2960 (CH_2, CH_3); 3256, 3384 (H_2O). UV spectrum, nm: pH 7: λ_{min} 240.6, λ_{max} 270.0. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.13 t (6H, C*H*$_3$CH$_2$, *J* = 7 Hz), 2.10 s (3H, 6-C*H*$_3$), 3.90 s (2H, N^{1}CH$_2$), 4.2 s (2H, N^{3}CH$_2$), 4.4 t (4H, C*H*$_2$CH$_3$, *J* = 7 Hz), 5.68 s (1H, 5-H). ^{13}C NMR spectrum (CDCl3), δ_C, ppm: 13.60 (6-CH_3), 14.13 (CH_2*C*H$_3$), 44.90 (C^7, N^{3}CH$_2$), 49.38 (N^{1}CH$_2$), 61.20 (*C*H$_2$CH$_3$), 61.81 (*C*H$_2$CH$_3$), 99.90 (C^5), 155.50 (C^6), 158.30 (C^2=O), 161.41 (C^4=O), 163.70 (O=C^{8}O), 166.36 (O=C^{10}O). Found, %: C 52.76; H 6.44; N 9.00. $C_{13}H_{18}N_2O_6$. Calculated, %: C 52.34; H 6.08; N 9.39.

Ethyl 2-[1,3-bis(ethoxycarbonylmethyl)-6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yloxy]acetate (XII). Ethyl chloroacetate, 40 g, was added in portions to a mixture of 14.2 g of hydroxymethyluracil **II**, 43 g of K_2CO_3, and 4 g of tetrabutylammonium bromide in 60 ml of DMF. The mixture was heated for 3 h on a boiling water bath, cooled, and neutralized with hydrochloric acid to pH 7, DMF was distilled off under reduced pressure (water-jet pump), the residue was dissolved in 100 ml of water, and the precipitate was filtered off and washed with acetone and DMF. The solvent was distilled off to obtain 30 g (74%) of compound **XII** as a light brown thick liquid. The product is readily soluble in water and poorly soluble in acetone and DMF. R_f 0.69. IR spectrum, ν, cm^{-1}: 800, 850, 1050 [ω(C=C)]; 1050–1240 (=N); 1256 (OCO); 1380 [δ_s(*gem*-CH_3)]; 1680, 1720, 1760 (C=O, =NC=O, OCO); 2950, 3000 (CH_2, CH_3); 3300 (H_2O). UV spectrum, nm: pH 7: λ_{min} 243.6, λ_{max} 274.0. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.23 t (9H, 3C*H*$_3$CH$_2$, *J* = 6 Hz), 2.30 s (3H, 6-C*H*$_3$), 2.87 m (8H, 4OCH$_2$), 4.56 s (2H, N^{1}CH$_2$), 4.64 s (2H, N^{3}CH$_2$). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 13.70 (6-CH_3), 14.74 (3*C*H$_3$CH$_2$), 44.60 (C^7, N^{1}CH$_2$), 46.38 (N^{3}CH$_2$), 61.2 (*C*H$_2$CH$_3$), 61.41 (*C*H$_2$CH$_3$), 62.12 (*C*H$_2$CH$_3$), 64.36 (O*C*H$_2$), 125.62 (C^5), 141.00 (C^6), 149.69 (C^2=O), 161.31 (C^4=O), 163.70 (O=C^{8}O), 166.36 (O=C^{10}O), 167.00 (O=C^{12}O). Found, %: C 48.30; H 6.33; N 7.00. $C_{17}H_{24}N_2O_9$. Calculated, %: C 48.80; H 6.26; N 6.69.

1,3-Bis(ethoxycarbonylmethyl)-6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-ylammonium sulfate (XIII). Tetrabutylammonium bromide, 2 g, and potassium carbonate, 14 g, were added to 12 g of 6-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-ylammonium sulfate in 150 ml of DMF, and 18.4 g of ethyl chloroacetate was then added in portions. The mixture was heated for 6 h on a boiling water bath, the solvent was distilled off, 100 ml of water was added to the residue, and the solution was extracted with chloroform (3 × 100 ml). The solvent was distilled off from the extract to obtain 14.7 g (81%) of compound **XIII** as a light yellow thick liquid. IR spectrum, ν, cm^{-1}: 1104, 1120 (COC); 1144–1268 (=N); 1226 (COOR); 1376 [δ_s(*gem*-CH_3)]; 1464 (CH_2, CH_3); 1656, 1688, 1744 (C=O, =NC=O, CO_2); 2936, 2952, 2984 (CH_2, CH_3). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.13 t (6H, 2C*H*$_3$CH$_2$, *J* = 7 Hz), 2.10 s (3H, 6-CH_3), 3.90 s (2H, N^{1}CH$_2$), 4.2 s (2H, N^{3}CH$_2$), 4.4 t (4H, C*H*$_2$CH$_3$, *J* = 7 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 13.60 (6-CH_3), 14.13 (CH_2*C*H$_3$), 44.90 (C^7, N^{1}CH$_2$), 49.38 (N^{3}CH$_2$), 61.20 (*C*H$_2$CH$_3$), 61.81 (*C*H$_2$CH$_3$), 99.90 (C^5), 155.50 (C^6), 158.30 (C^2=O), 161.41 (C^4=O), 163.70 (O=C^{8}O), 166.36 (O=C^{10}O). Found, %: C 37.50; H 5.30; N 10.00; S 7.50. $C_{13}H_{21}N_3O_{10}S$. Calculated, %: C 37.96; H 5.14; N 10.21; S 7.79.

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