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Synthesis of 1,3-Bis[3-X-2-(X-acetoxy)propyl]-6-methyl-1,2,3,4-tetrahydropyrimidine-2,4-diones

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Abstract—A procedure was developed for synthesizing nitrogen-and-sulfur-containing pyrimidine derivatives by reactions of 1,3-bis(3-chloro-2-hydroxypropyl)-6-methyluracil with 1-butanethiol and chloroacetyl chloride, followed by treatment of the resulting 1,3-bis[3-chloro-2-(chloroacetoxy)propyl]-6-methyl-1,2,3,4-tetrahydropyrimidine-2,4-dione with 1-butanethiol, morpholine, and piperidine. Oxidation of the sulfur-containing products with $NaIO_4$ gave the corresponding sulfoxides.

Functionally substituted acyclic and cyclic sulfides and their oxidation products constitute a group of substances which attract a great interest from the viewpoint of their immunostimulating and antiphlogistic activity [1, 2]; such compounds can also be used as metal extragents and complexing agents [3]. A new class of low-toxic nonsteroidal compounds exhibiting high antiphlogistic activity was obtained on their base [4–6]. A procedure for synthesizing 1,3-bis(3-chloro-2-hydroxypropyl)-6-methyluracil and its derivatives was reported in [7].

With the goal of obtaining new pyrimidine derivatives as potential immunotropic and antiphlogistic drugs, as well as extragents and complexing agents, we have synthesized a series of 1,3-bis[3-X-2-(X-acetoxypropyl)-6-methyl-1,2,3,4-tetrahydropyrimidine-2,4-diones from 1,3-bis(3-chloro-2-hydroxypropyl)-6-methyluracil (**I**) and butanethiol, chloroacetyl chloride, morpholine, and piperidine.

Treatment of compound **I** with butanethiol gave 1,3-bis(3-butylthio-2-hydroxypropyl)-1,2,3,4-tetrahydropyrimidine-2,4-dione (**II**) in good yield. The reaction of compound **I** with chloroacetyl chloride was carried out by the procedure reported in [8]; we thus obtained 1,3-bis[3-chloro-2-(chloroacetoxy)propyl]-1,2,3,4-tetrahydropyrimidine-2,4-dione (**III**) in 77% yiled. Compound **III** was brought into reaction butanethiol to isolate 1,3-bis[3-butylthio-2-(butylthioacetoxy)propyl]-6-methyl-1,2,3,4-tetrahydropyrimidine-2,4-dione (**IV**), while treatment of **III** with morpholine or piperidine afforded 1,3-bis[3-morpholino(piperidino)-2-(morpholino(piperidino)acetoxy)propyl]-6-methyl-1,2,3,4-tetrahydropyrimidine-2,4-diones **V** and **VI**.

Compounds **II** and **IV** were oxidized with sodium periodate in aqueous alcohol at 18–20°C (reaction time 4 h); the products were isolated by extraction into chloroform. The resulting 1,3-bis(3-butylsulfinyl-2-hydroxypropyl)- and 1,3-bis[3-butylsulfinyl-2-(butylsulfinylacetoxy)propyl]-6-methyl-1,2,3,4-tetrahydropyrimidine-2,4-diones **VII** and **VIII** are readily soluble in chloroform, water, and ether, and insoluble in hexane. All the compounds were synthesized for the first time. Their structure was proved by elemental analysis, IR and ^1H and ^{13}C NMR spectroscopy.

The IR spectra of all pyrimidine derivatives contain absorption bands at 1640, 1660–1670, and 1700–1720 cm^{-1}, belonging to vibrations of the uracil ring; strong bands in the region 1228–1256 cm^{-1} are observed only in the spectra of acetates **III**–**VI** and **VIII**; absorption bands in the regions 500–660 and 1248–1252, 1280–1290 cm^{-1} were assigned, respectively, to stretching vibrations of the C–Cl bond and bending vibrations of the CH_2Cl group in compound **III**. The absorption in the region 640–690 cm^{-1} indicates the presence of C–S bonds in compounds **II**, **IV**, **VII**, **VIII**; bands at 1060–1240 cm^{-1} are typical of C–N bonds in tertiary amines (compounds **V** and **VI**). Stretching vibrations of the hydroxy groups in compounds **II** and **VII** appear at 3300–3400 cm^{-1}, and compound **V** shows in the IR spectrum a band at 1120 cm^{-1} due to the C–O–C fragment. In the IR

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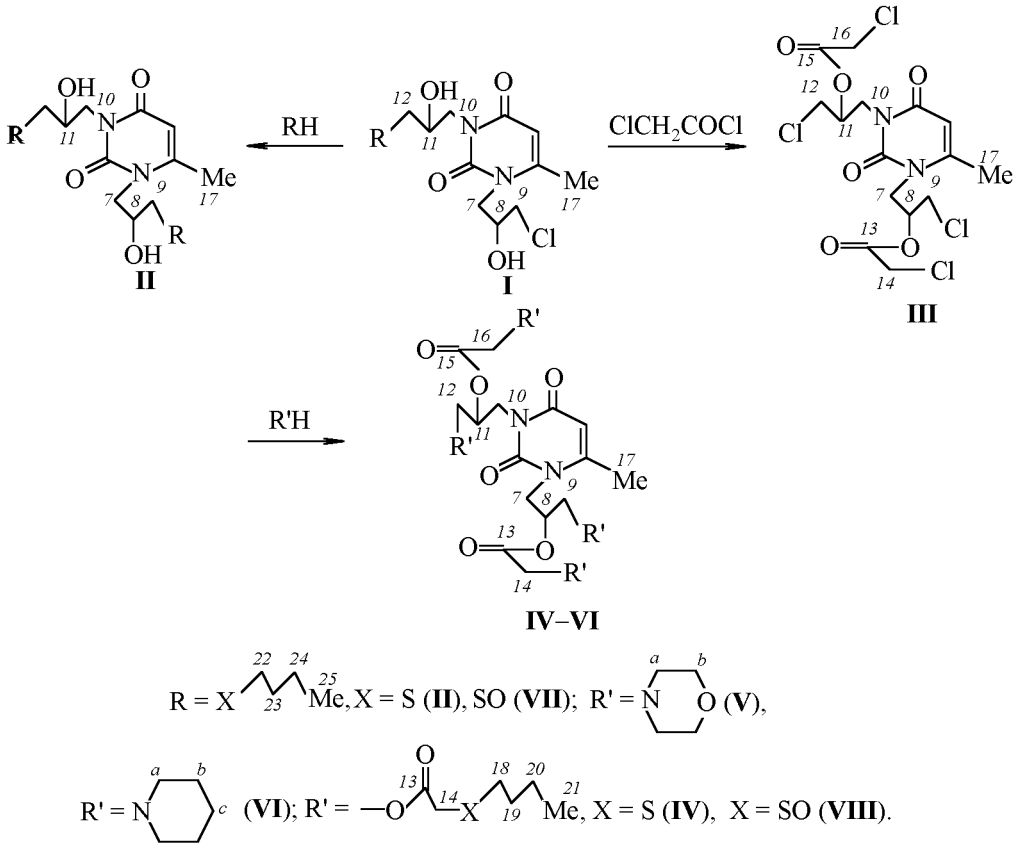
Scheme 1.



II, R = $CH_3CH_2CH_2CH_2S$; **IV**, R' = $CH_3CH_2CH_2CH_2S$; **V**, R' = morpholino; **VI**, R' = piperidino.

spectra of **VII** and **VIII** we observed absorption in the region 1050–1070 cm^{-1}, which is typical of sulfoxides; the lack of absorption bands at 1290, 1340, and 1120–1165 cm^{-1} indicates that no sulfones were formed at the oxidation.

Compounds **II**–**VIII** showed in the ^1H NMR spectra a singlet at δ 2.3–2.4 ppm from the 6-CH_3 group, and a signal at δ 2.9 ppm from methylene protons of the $OCOCH_2Cl$ or $OCOCH_2S$ group. The $OCOCH_2N$ signals in the spectra of **V** and **VI** appear at δ 3.09–3.16 ppm, and the $OCOCH_2SO$ signal of **VIII** is displaced strongly downfield (δ 3.7 ppm) due to the effect of the sulfoxide group. The uracil 5-H signal is observed at about δ 5.6 ppm. Protons of the piperidine and morpholine rings give signals at δ 2.2–2.6 (CH_2NCH_2), 1.25–1.84 ($CH_2CH_2CH_2$), and 3.3–4.1 ppm (CH_2OCH_2). Methyl protons in the alkyl fragments of compounds **II** and **VII** appear as a triplet at δ 0.88 ppm, and the corresponding triplet from the $OCOCH_2SC_4H_9$ group of **IV** and **VIII** is located at δ 1.2 ppm. The ^{13}C carbon signals of carbonyl-containing compounds are highly characteristic, and they are rarely obscured by signals from the other functional groups; the carbonyl carbon signals appear in the range from δ_C 150 to 210 ppm. In the spectra of **IV**–**VI** and **VIII** the OCO signals are located in the region δ_C 166–172 ppm. (C^{13}=O and C^{15}=O) [9]. Carbon atoms of the pyrimidine ring in all the products give signals in the regions δ_C 160–163 (C^4=O), 153–160 (C^2=O), 151.3–153.9 (C^6), 100.4–101.8 (C^5), and 20.7–21.78 ppm (6-CH_3). The ^{13}C chemical shifts of the methylene groups in the $OCOCH_2Cl$ and $OCOCH_2S$ moieties range $OCOCH_2N$ from 37.2 to 40.7 ppm, whereas the corresponding signal from the morpholine and piperidine derivatives is observed in a weaker field, $\Delta\delta_C$ = 16–20 ppm; analogous difference for sulfoxides **VII** and **VIII** is 15 ppm. Thus, the IR and ^1H and ^{13}C NMR spectral parameters of the newly synthesized compounds are consistent with the proposed structures.

EXPERIMENTAL

The IR spectra were recorded on a UR-20 spectrometer from samples prepared as mulls in mineral

oil or thin films. The ^{13}C NMR spectra were obtained on a Bruker AM-300 instrument from 10–20% solutions in $CDCl_3$. The operating frequency was 300 MHz for ^1H and 75.5 MHz for ^{13}C. The chemical shifts were measured relative to TMS. The melting points were determined using a Boetius device. Silufol UV-254 plates were used for thin-layer chromatography; eluent ethanol–aqueous ammonia, 4 : 1; spots were visualized by UV irradiation.

1,3-Bis(3-butylthio-2-hydroxypropyl)-6-methyl-1,2,3,4-tetrahydropyrimidine-2,4-dione (II). Compound I, 62.2 g (0.2 mol), was dissolved on heating in a mixture of 150 ml of DMF and 20 ml of water, and a solution of sodium 1-butanethiolate [prepared by mixing 50 ml of 1-butanethiol with a solution of 24 g (0.6 mol) of NaOH in 100 ml of DMF and 70 ml of water] was added dropwise over a period of 1 h, maintaining the temperature at 50–55°C. The mixture was heated to 80°C, stirred for 2 h at that temperature, and left overnight. Excess 1-butanethiol was distilled off under reduced pressure (bp 45–75°C, bath temperature 85–95°C), the residue was cooled, and the precipitate of sodium chloride was filtered off. The remaining solvent was distilled off from the filtrate at a bath temperature of 130–230°C (15 mm). Yield 80 g (96%). R_f 0.53. Compound **II** is readily soluble in ethanol and ether and poorly soluble in water. IR spectrum, ν, cm^{-1}: 640–690 (C–S), 1220–1270, 1420, 1440 (RCH_2S), 1240 (C=C), 1660, 1710 (C=O, N–C=O), 3440 (2νC=O). ^1H NMR spectrum ($CDCl_3$), δ, ppm (hereinafter, for atom numbering, see Scheme 1): 5.62 s (1H, 5-H), 3.9–4.1 m (2H, OH), 3.83 d.d (2H, 7-H, 10-H, J = 11.2, 6 Hz), 3.75 d.d (2H, 7-H, 10-H, J = 11.2, 6 Hz), 3.5 m (3H, 8-H, 11-H, C**H**OH), 2.72 d (2H, 9-H, 12-H, J = 12 Hz), 2.6 d (2H, 9-H, 12-H, J = 12 Hz), 2.5 t (4H, 22-H, J = 6.5 Hz), 2.3 s (3H, 6-CH_3), 1.55 q (4H, 23-H, J = 7 Hz), 1.35 sext (4H, 24-H, J = 7 Hz), 0.88 t (6H, 2CH_3, J = 6.5 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 163.17 (C^4), 153.80 (C^2), 152.90 (C^6), 101.47 (C^5), 68.55 (C^8), 67.73 (C^{11}), 50.06 (C^7), 46.11 (C^{10}), 37.32 (C^9, C^{12}), 32.21 (2C^{22}), 31.66 (2C^{23}), 21.83 (2C^{24}), 21.78 (6-CH_3), 13.60 (2C^{25}). Found, %: C 54.30; N 8.16; N 6.34; S 15.56. $C_{19}H_{34}N_2O_4S_2$. Calculated, %: C 54.52; H 8.19; N 6.69; S 15.32.

1,3-Bis[3-chloro-2-(chloroacetoxy)propyl]-1,2,-3,4-tetrahydropyrimidine-2,4-dione (III). To a solution of 120 g (0.385 mol) of compound **I** in 300 ml of chloroform and 100 ml of pyridine we added dropwise over a period of 2 h a solution of 88 ml (131.6 g, 1.16 mol) of chloroacetyl chloride in 110 ml of chloroform, while cooling with cold water. The mixture was gradually heated to 80°C, stirred for 1 h at that temperature, and left overnight. It was then diluted with 300 ml of water, and the organic phase was separated, washed with water (2 × 70 ml), neutralized to pH 6–7 by adding 2 g of $NaHCO_3$, and dried over $MgSO_4$. The solution was evaporated under reduced pressure using first a water-jet pump and then an oil pump. Yield 92.5 g (77%); dark liquid, readily soluble in organic solvents ($CHCl_3$, C_6H_6, DMF, DMSO) and insoluble in water; R_f 0.74. IR spectrum, ν, cm^{-1}: 560–830 (C–Cl, ωCH_2Cl), 1232, 1256 (O–CO, acetate), 1160–1210 (C–O), 1656, 1664, 1672, 1696, 1704, 1752, 1760 (C=O, N–C=O). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 5.65–5.4 m (1H, 5-H), 5.25 q (2H, 8-H, 11-H), 4.5–4.8 m (4H, 9-H, 12-H, CH_2Cl), 4.08 d.d (2H, 7-H, 10-H, J = 11.3, 5.6 Hz), 3.8 d.d (2H, 7-H, 10-H, CH_2N, J = 11.3, 6.8 Hz), 2.95 s (4H, 14-H, 16-H), 2.34 s (3H, 6-CH_3), ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 164.0 (C^{13}, C^{15}), 161.62 (C^4), 153.72 (C^2), 152.03 (C^6), 101.75 (C^5), 71.37 (C^8), 69.23 (C^{11}), 43.08 (C^9, C^{12}), 40.69 (C^{14}, C^{16}, C^7, C^{10}), 20.7 (6-CH_3). Found, %: C 38.50; H 3.91; Cl 30.64; N 6.00. $C_{15}H_{18}Cl_4N_2O_6$. Calculated, %: C 38.82; H 3.91; Cl 30.55; N 6.04.

1,3-Bis[3-butylthio-2-(butylthioacetoxy)propyl)]-6-methyl-1,2,3,4-tetrahydropyrimidine-2,4-dione (IV). To a solution of sodium 1-butanethiolate, prepared from 24 g (0.6 mol) of sodium hydroxide in 50 ml of water and 60 ml (51.48 g, 0.57 mol) of 1-butanethiol, 200 ml of DMF was added. The mixture was heated to 50°C, 48.1 g (0.1 mol) of compound **III** was added dropwise over a period of 90 min (addition of a large amount of **III** at once leads to spontaneous heating up to 62°C), and the mixture was stirred for 30 min and left overnight. The precipitate of sodium chloride was filtered off, the filtrate was evaporated under reduced pressure (first, using a water-jet pump and then an oil pump), the residue was dissolved in 80 ml of water, the solution was treated with ether (300 ml) and toluene, and the extracts were combined and evaporated. Yield 60 g (88%). Product **IV** is readily soluble in ether, benzene, and toluene. R_f 0.53. IR spectrum, ν, cm^{-1}: 1220–1270, 1420, 1440 (RCH_2S), 1240 (C=C), 1660, 1710 (C=O, N–C=O), 3440 (2νC=O). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 5.62 s (1H, 5-H), 3.94 m (4H, 7-H, 10-H), 3.8–3.5 m (2H, 8-H, 11-H), 2.9 s (4H, 14-H, 16-H), 2.7 d.d (2H, 9-H, 12-H, J = 11.3, 5.8 Hz), 2.62 d.d (2H, 9-H, 12-H, J = 11.3, 6.2 Hz), 2.5 t (8H, 18-H, 22-H, J = 7 Hz), 2.3 s (3H, 17-H), 1.62 sext (8H, 19-H, 20-H, J = 7 Hz), 1.4 sext (8H, 23-H, 24-H, J = 7 Hz), 1.20 t (6H, 21-H, J = 7 Hz), 0.88 t (6H, 25-H, J = 6.5 Hz). ^{13}C NMR spectrum

($CDCl_3$), δ_C, ppm: 172.62 (C^{13}), 169.58 (C^{15}), 162.39 (C^4), 153.37 (C^2), 127.33 (C^6), 100.42 (C^5), 68.22 (C^8), 60.46 (C^{11}), 37.96 (C^{10}), 37.67 (C^7), 37.33 (C^9, C^{12}), 37.24 (C^{14}, C^{16}), 32.90 (C^{19}), 32.52 (C^{22}), 32.50 (C^{23}), 31.12 (C^{18}), 21.20 (C^{20}, C^{24}), 20.97 (6-CH_3, C^{17}), 13.47 (C^{21}, C^{25}). Found, %: C 54.40; H 8.30; N 4.24; S 18.39. $C_{31}H_{54}N_2O_6S_4$. Calculated, %: C 54.83; H 8.02; N 4.13; S 18.89.

6-Methyl-1,3-bis[3-morpholino-2-(morpholinoacetoxy)propyl]-1,2,3,4-tetrahydropyrimidine-2,4-dione (V). Morpholine, 22 ml, was added to 7.7 g (16.6 mmol) of compound **III**; the mixture warmed up to 50°C and was heated to 100–110°C, stirred for 3 h at that temperature, and left overnight. The crystals of $C_4H_8NO \cdot HCl$ were filtered off and washed with chloroform (2×10 ml); 7.8 g of crude morpholine hydrochloride was thus obtained. The solvent was distilled off from the filtrate, and the residue was washed with acetone (10 ml). The product, 12.7 g, was dissolved in acetone, and the solution was left overnight. The precipitate was filtered off, and the filtrate was passed through a column charged with 54 g of Al_2O_3 of activity grade II using acetone as eluent. Two fractions were collected. Removal of the solvent from the first fraction gave 1.6 g (15%) of the product, and 7.8 g (71%) of **V** was isolated from the second fraction. Overall yield 86%; compound **V** is readily soluble in acetone, chloroform, DMF, and ether and poorly soluble in water. IR spectrum, ν, cm^{-1}: 960, 1010 ($\omega C{=}C$), 1640, 1664, 1700 (C=O, NC=O), 1120 (C–O–C), 1156, 1240 (=N–), 1735, 3384 (RCOO). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 5.48 s (1H, 5-H), 4.1–3.33 m (16H, CH_2OCH_2, 7-H, 8-H, 10-H, 11-H), 3.09 s (4H, 14-H, 16-H), 2.42 s (3H, 6-CH_3), 2.6–2.2 m (16H, CH_2NCH_2, 9-H, 12-H). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 167.51 (C^{13}, C^{15}), 162.49 (C^4), 160.6 (C^2), 153.10 (C^6), 101.25 (C^5), 66.65 and 66.53 (CH_2OCH_2), 65.53 (C^8), 63.72 (C^{11}), 59.42 (C^{14}, ^{16}), 53.76 (C^9), 53.50 (C^{12}), 53.7 and 53.61 (CH_2NCH_2), 45.90 (C^7), 41.88 (C^{10}), 13.99 (6-CH_3). Found, %: C 56.0; H 8.0; N 12.9. $C_{31}H_{50}N_6O_{10}$. Calculated, %: C 55.84; H 7.58; N 12.60.

6-Methyl-1,3-bis[3-piperidino-2-(piperidinoacetoxy)propyl)-1,2,3,4-tetrahydropyrimidine-2,4-dione (VI) was synthesized as described above for compound **V** from 4.8 g (0.01 mol) of compound **III** and 15 ml of piperidine. After removal of the solvent under reduced pressure (oil pump), the residue was passed through a column charged with Al_2O_3 of activity grade II to isolate 4.1 g (62%) of compound **VI**. The product is readily soluble in chloroform, acetone, and DMF, sparingly soluble in ether, and insoluble in hexane. Starting from 5.6 g (0.013 mol) of **III** and 20 ml of piperidine, after appropriate treatment, dissolution in acetone, and removal of $C_5H_{11}N \cdot HCl$, we obtained 7.5 g (88%) of product **VI**. R_f 0.60. Insofar as compound **VI** is soluble in water, it was not treated with water to remove salts. IR spectrum, ν, cm^{-1}: 928, 1008 ($\omega C{=}C$), 1082–1296 (C–N), 1156, 1240 (=N–), 1644, 1680, 1696 (C=O, NC=O), 3440 (RCOO). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 5.6 s (1H, 5-H), 4.2–3.7 m (6H, 7-H, 8-H, 10-H, 11-H), 3.16 s (4H, 14-H, 16-H), 2.49–2.76 m (4H, 9-H, 12-H), 2.31 s (3H, 6-CH_3), 2.56–2.19 m (16H, CH_2NCH_2), 1.84–1.25 m (24H, $CH_2CH_2CH_2$). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 167.3 (C^{13}, C^{15}), 162.20 (C^4), 159.30 (C^2), 153.80 (C^6), 100.90 (C^5), 62.30 (C^8), 62.0 (C^{11}), 54.44 and 54.10 (CH_2NCH_2), 53.98 (C^{14}, C^{16}), 53.50 (C^9), 53.20 (C^{12}), 46.2 (C^7), 45.07 (C^{10}), 26.50 and 25.90 ($NCH_2\mathbf{C}CH_2$), 24.64 and 23.70 ($NCH_2CH_2\mathbf{C}CH_2$), 20.54 (6-CH_3). Found, %: C 63.70; H 9.00; N 12.40. $C_{35}H_{58}N_6O_6$. Calculated, %: C 63.80; H 8.87; N 12.75.

1,3-Bis(3-butylsulfinyl-2-hydroxypropyl)-6-methyl-1,2,3,4-tetrahydropyrimidine-2,4-dione (VII). To 4.2 g (0.01 mol) of compound **II** we added 100 ml of water and 50 ml of alcohol, and 5 g (0.023 mol) of $NaIO_4$ was added over a period of 15 min, while cooling with cold water. The mixture was stirred for 4 h at 18–20°C and was left overnight. The crystals were filtered off, the filtrate was treated with chloroform (3×70 ml), the organic phase was separated and evaporated, and the residue was washed with acetone. Yield 3.2 g (70%), mp >350°C (from $CHCl_3$), R_f 0.78. IR spectrum, ν, cm^{-1}: 760 (RCH_2S), 1050 (S=O), 1130, 1240, 1340 (C–N), 1400 (δCH_3), 1490 (δCH_2CH_3), 1680, 1710 (C=O, NC=O), 2880 ($C{-}H_{sym}$, RCH_2S), 2940, 2980 ($C{-}H_{asym}$), 3360 (OH). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 4.2–3.6 br.s (2OH), 5.5 s (1H, 5-H), 4.7 m (2H, C**H**OH), 4.2–3.8 m (4H, CH_2N), 3.1–2.8 m (4H, 9-H, 12-H), 2.8–2.5 m (4H, 22-H), 2.27 s (3H, 6-CH_3), 1.68 sext (4H, CH_2CH_2, J = 6.5 Hz), 1.40 sext (4H, C**H**$_2CH_3$, J = 6.5 Hz), 0.88 t (6H, CH_3, J = 6 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 162.5 (C^4), 153.6 (C^2), 151.33 (C^6), 101.07 (C^5), 63.2 (C^8), 61.9 (C^{11}), 58.18 (C^9), 58.1 (C^{22}), 57.8 (C^{12}), 56.4 (C^{22}), 52.0 (C^{10}), 51.9 (C^7), 24.3 (C^{23}), 21.6 (C^{24}), 20.9 (C^{17}, 6-CH_3), 13.47 (C^{25}). Found, %: C 50.80; H 7.70; N 5.80; S 14.57. $C_{19}H_{34}N_2O_6S_2$. Calculated, %: C 50.64; H 7.61; N 6.22; S 14.23.

1,3-Bis[3-butylsulfinyl-2-(butylsulfinylacetoxy)-propyl]-6-methyl-1,2,3,4-tetrahydropyrimidine-2,4-dione (VIII). To 3.4 g (0.005 mol) of compound **IV** we added 50 ml of water, 30 ml of alcohol, and

a solution of 5.35 g (0.025 mol) of $NaIO_4$ in 50 ml of water was added dropwise over a period of 1 h at room temperature. The mixture was stirred for 3 h and left overnight. It was then treated with 60 ml of chloroform, the organic phase was separated, and the aqueous phase was extracted with chloroform (2×20 ml). The chloroform extracts were combined and evaporated to obtain 2.8 g (76%) of compound **VIII** as a solid substance. IR spectrum, ν, cm^{-1}: 640, 760 (RCH_2S), 1070 (S=O), 1120, 1240 (C–N), 1390 (δCH_3, sym.), 1680, 1720 (C=O, NC=O), 2890, 2950 (RCH_2CH_3), 3360 (OH). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 5.6–5.2 m (2H, OCH), 5.2 s (1H, 5-H), 4.3–3.9 m (4H, NCH_2), 3.9–3.5 m (4H, $COCH_2S$), 3.3–2.5 m (12H, SCH_2), 2.29 s (3H, 6-CH_3), 1.69 sext (8H, CH_2CH_2, J = 6 Hz), 1.39 sext (8H, CH_2CH_3, J = 6.5 Hz), 0.89 t.t (12H, CH_3, J = 7 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 166.34 (C^{13}, C^{15}), 162.6 (C^4), 159.9 (C^2), 153.9 (C^6), 101.34 (C^5), 71.9 (C^8), 70.0 (C^{11}), 55.32 (C^{14}, C^{16}), 54.51 (C^9, C^{12}), 52.52 (C^{18}), 52.40 (C^{22}), 47.1 (C^7), 42.4 (C^{10}), 24.48 (C^{19}, C^{23}), 21.82 (C^{20}, C^{24}), 20.86 (C^{17}, 6-CH_3), 13.58 (C^{25}, C^{21}). Found, %: C 45.90; H 6.90; N 3.20; S 17.06. $C_{31}H_{54}N_2O_{10}S_4$. Calclated, %: C 50.11; H 7.33; N 3.77; S 17.26. The product contains 4 molecules of crystallization water. Calculated for $C_{31}H_{54}N_2O_{10}S_4 \cdot 4H_2O$, %: C 45.68; H 7.67; N 3.44; S 15.73.

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