

α -Thioureidoalkylation of Functionally Substituted Ureas: II.* Synthesis of Thio Analogs of *N*-Hydroxyalkyl-1,5-diphenylglycolurils

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Abstract—Reactions of *N*-(hydroxyalkyl)ureas with 4,5-dihydroxy-4,5-diphenylimidazolidine-2-thiones gave previously unknown 4,6-dialkyl-1-hydroxyalkyl-3a,6a-diphenyl-5-thioxooctahydroimidazo[4,5-*d*]imidazol-2-ones which may be regarded as thio analogs of *N*-(hydroxyalkyl)glycolurils.

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Replacement of oxygen atom in biologically active compounds by sulfur is known to enhance or change the kind of activity. For example, dithiopiracetam is much more effective than piracetam as nootropic and antihypoxic drug [2]. Substituted thiohydantoins (2-thioxoimidazolidin-4-ones) are stronger inhibitors of fatty acid amide hydrolase than the corresponding oxygen derivatives [3].

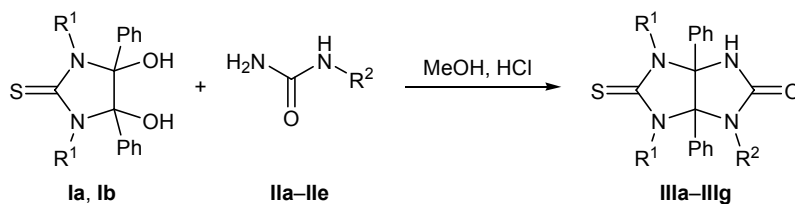
Over several years we were interested in the chemistry of glycolurils (octahydroimidazo[4,5-*d*]imidazole-2,5-diones) [4–9] which constitute a new class of neurotropic compounds [10–12]. Furthermore, it is known that diphenylglycolurils containing alkyl substituents on the nitrogen atoms affect hepatic cytochrome P-450-dependent monooxygenase system [13].

We recently initiated a series of studies aimed at synthesizing glycoluril thio analogs [1, 14]. In the present communication we report on the reaction of *N*-(hydroxyalkyl)ureas **IIa–IIe** (ureido alcohols) with

1,3-dialkyl-4,5-dihydroxy-4,5-diphenylimidazolidine-2-thiones **Ia** and **Ib** as a method of synthesis of 1-hydroxyalkyl-3a,6a-diphenyl-5-thioxooctahydroimidazo[4,5-*d*]imidazol-2-ones **III** (thio analogs of *N*-hydroxyalkyldiphenylglycolurils). The reactions were carried out in boiling methanol in the presence of hydrochloric acid (reaction time 1–3 h; Scheme 1). The dependence of the yield of compounds **III** on the reaction time and amount of HCl was studied using the reaction of imidazolidinethione **Ia** with *N*-(2-hydroxyethyl)urea as model (see table).

It is seen that the yield of thioglycoluril **IIIa** considerably increases as the amount of HCl rises from 0.2 to 0.5 mol per mole of **Ia**; further raising of the amount of HCl to 1 mol almost does not affect the yield of **IIIa**. Increase of the reaction time from 1 to 2–3 h improves the yield of **IIIa** almost twofold. Taking into account these results, compounds **IIIa–IIIg** were synthesized using 1 mol of hydrogen chloride per mole of

Scheme 1.



I, R¹ = Me (**a**), Et (**b**); **II**, R² = HO(CH₂)₂ (**a**), HO(CH₂)₃ (**b**), MeCH(OH)CH₂ (**c**), 4-HOC₆H₄(CH₂)₂ (**d**), HOCH₂CH(Et) (**e**); **III**, R¹ = Me, R² = HO(CH₂)₂ (**a**), HO(CH₂)₃ (**b**), MeCH(OH)CH₂ (**c**), HOCH₂CH(Et) (**d**); R¹ = Et, R² = HO(CH₂)₂ (**e**), 4-HOC₆H₄(CH₂)₂ (**f**), MeCH(OH)CH₂ (**g**).

* For communication I, see [1].

Ia or **Ib**, the reaction time being 2 h; their yields ranged from 59 to 90%.

1-(2-Hydroxypropyl)- and 1-(1-hydroxybutan-2-yl)-4,6-dialkyl-3a,6a-diphenyl-5-thioxooctahydroimidazo[4,5-*d*]imidazol-2-ones **IIIc**, **IIId**, and **IIIg** whose molecules contain three asymmetric carbon atoms were formed as two diastereoisomers: (2'*R**,3a*S**,6a*R**)- and (2'*R**,3a*R**,6a*S**)-4,6-dialkyl-1-(2-hydroxypropyl)-3a,6a-diphenyl-5-thioxooctahydroimidazo[4,5-*d*]imidazol-2-ones **IIIc** and **IIIg** and (2'*R**,3a*S**,6a*R**)- and (2'*R**,3a*R**,6a*S**)-1-(1-hydroxybutan-2-yl)-4,6-dimethyl-3a,6a-diphenyl-5-thioxooctahydroimidazo[4,5-*d*]imidazol-2-one (**IIId**).

The NMR spectra of compounds **IIIc** and **IIIg** contained double sets of signals from protons and carbon atoms at a ratio of 1:1. By repeated recrystallizations from methanol we isolated one diastereoisomer of **IIIc** and **IIIg**. In the reaction of thione **Ia** with urea **Ile**, one diastereoisomer of **IIId** (**A**) with a small impurity of the other diastereoisomer separated first from the reaction mixture, and then the second diastereoisomer (**B**) containing some impurity of the first one separated. Pure diastereoisomers of **IIId** were isolated in approximately equal amounts (31 and 28%) by recrystallization of the crude products from methanol.

EXPERIMENTAL

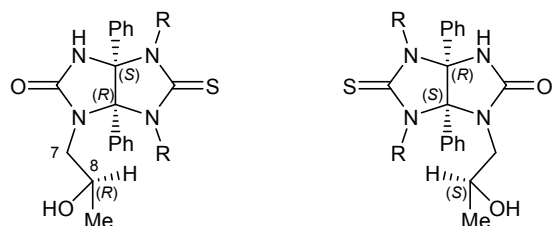
The NMR spectra were recorded on Bruker AM-250 (¹H, 250 MHz) and Bruker AM-300 spectrometers (¹³C, 75.5 MHz) from solutions in DMSO-*d*₆ using

Yields of thioglycoluril **IIIa** in the reaction of 4,5-dihydroxy-1,3-dimethyl-4,5-diphenylimidazolidine-2-thione (**Ia**) with *N*-(2-hydroxyethyl)urea (**IIa**)

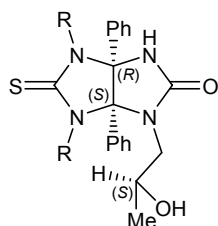
Time, h	HCl, mol	Yield, %
1	0.2	18
1	0.5	32
1	1	34
2	0.5	65
3	0.5	67
2	1	68
3	1	64

tetramethylsilane as internal reference. 1,3-Dialkyl-4,5-dihydroxy-4,5-diphenylimidazolidine-2-thiones **Ia** and **Ib** were synthesized by condensation of *N,N'*-dimethyl- and *N,N'*-diethylureas with 1,2-diphenylethane-1,2-dione according to the procedures reported in [15, 16]. Alcohols **IIa–IIe** were prepared by reaction of 2-aminoethanol, 3-aminopropan-1-ol, 1-aminopropan-2-ol, 2-aminobutan-1-ol, and 4-(2-aminoethyl)-phenol, respectively, with potassium cyanate [17].

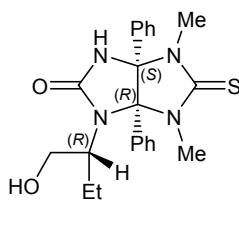
4,6-Dialkyl-2-[hydroxyalkyl, 2-(4-hydroxyphenyl)ethyl]-3a,6a-diphenyl-5-thioxooctahydroimidazo[4,5-*d*]imidazol-2-ones IIIa–IIIg (general procedure). A mixture of 0.005 mol of compound **Ia** or **Ib**, 0.005 mol of urea **IIa–IIe**, 15 ml of methanol, and 0.42 ml (0.005 mol) of concentrated hydrochloric acid was heated for 2 h under reflux. After cooling, the precipitate was filtered off and recrystallized from



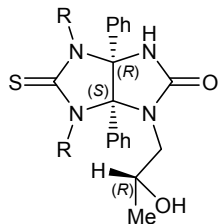
IIIc, IIIg
(3a*S**,6a*R**,8*R**)-Racemate



IIId
(3a*S**,6a*R**,7*R**)-Racemate



IIIc, IIIg
(3a*S**,6a*R**,8*S**)-Racemate



IIId
(3a*S**,6a*R**,7*S**)-Racemate

R = Me (**c**), Et (**g**).

methanol. Individual stereoisomers of **IIIc**, **IIId**, and **IIIg** (as racemates) were isolated by repeated crystallization from methanol.

2-(2-Hydroxyethyl)-4,6-dimethyl-3a,6a-diphenyl-5-thioxooctahydroimidazo[4,5-*d*]imidazol-2-one (IIIa). Yield 1.3 g (68%), mp 245–247°C. ¹H NMR spectrum, δ , ppm: 2.75–2.84 m (1H, NCH₂), 2.92 s and 3.11 s (3H each, NMe), 3.42–3.64 m (3H, NCH₂, OCH₂), 4.78 t (1H, OH, *J* = 5.1 Hz), 6.78 br.s (4H, H_{arom}), 7.08–7.13 m (6H, H_{arom}), 8.81 s (1H, NH). ¹³C NMR spectrum, δ , ppm: 30.7 and 32.6 (NMe), 44.8 (NCH₂), 59.1 (OCH₂), 86.7 and 90.9 (C^{3a}, C^{6a}); 127.1, 127.8, 128.1, 128.3, 128.8, 132.3, 133.7 (C_{arom}); 158.9 (C²), 183.0 (C⁵). Found, %: C 62.77; H 5.89; N 14.57; S 8.28. C₂₀H₂₂N₄O₂S. Calculated, %: C 62.81; H 5.80; N 14.65; S 8.38.

2-(3-Hydroxypropyl)-4,6-dimethyl-3a,6a-diphenyl-5-thioxooctahydroimidazo[4,5-*d*]imidazol-2-one (IIIb). Yield 1.23 g (62%), mp 272–274°C. ¹H NMR spectrum, δ , ppm: 1.48–1.60 m (1H, CH₂), 1.80–1.92 m (1H, CH₂), 2.77–2.87 m (1H, NCH₂), 2.93 s and 3.08 s (3H each, NMe), 3.34–3.50 m (3H, NCH₂, OCH₂), 4.43 t (1H, OH, *J* = 5.3 Hz), 6.78 br.s (4H, H_{arom}), 7.10–7.14 m (6H, H_{arom}), 8.71 s (1H, NH). ¹³C NMR spectrum, δ , ppm: 30.6 (CH₂), 32.5 and 32.6 (NMe), 40.3 (NCH₂), 58.3 (OCH₂), 86.7 and 90.6 (C^{3a}, C^{6a}); 127.0, 128.1, 128.3, 128.7, 128.8, 129.2, 132.4, 133.6 (C_{arom}), 158.7 (C²), 182.9 (C⁵). Found, %: C 63.67; H 6.02; N 14.20; S 7.95. C₂₁H₂₄N₄O₂S. Calculated, %: C 63.61; H 6.10; N 14.13; S 8.09.

(2'*R,3a*S**,6a*R**)- and (2'*R**,3a*R**,6a*S**)-1-(2-Hydroxypropyl)-4,6-dimethyl-3a,6a-diphenyl-5-thioxooctahydroimidazo[4,5-*d*]imidazol-2-one (IIIc) (mixture of diastereoisomers).** Yield 1.61 g (81%), mp 236–238°C. ¹³C NMR spectrum, δ , ppm: 21.0, 21.8 (Me); 30.5, 30.7, 32.8, 33.2 (NMe); 50.4, 50.6 (NCH₂); 65.1, 65.4 (OCH); 86.7, 87.0, 91.1 (C^{3a}, C^{6a}); 126.9, 127.0, 128.0, 128.1, 128.3, 128.7, 128.8, 132.3, 133.6, 133.8 (C_{arom}); 159.0, 159.7 (C²); 182.9, 183.0 (C⁵). Found, %: C 63.56; H 6.10; N 14.18; S 7.91. C₂₁H₂₄N₄O₂S. Calculated, %: C 63.61; H 6.10; N 14.13; S 8.09.

Diastereoisomer **A**. ¹H NMR spectrum, δ , ppm: 1.01 d (3H, Me, *J* = 6.2 Hz), 2.66 d.d (1H, NCH₂, *J* = 14.2, 6.6 Hz), 2.93 s and 3.12 s (3H each, NMe), 3.38–3.47 m (1H, NCH₂), 3.77–3.87 m (1H, OCH), 4.76 d (1H, OH, *J* = 3.7 Hz), 6.77 br.s (4H, H_{arom}), 7.12 br.s (6H, H_{arom}), 8.76 s (1H, NH).

Diastereoisomer **B**. Yield 0.59 g (30%), mp 246–248°C. ¹H NMR spectrum, δ , ppm: 1.01 d (3H, Me, *J* = 6.2 Hz), 2.72 d.d (1H, NCH₂, *J* = 14.2, 5.1 Hz),

2.94 s and 3.09 s (3H each, NMe), 3.38–3.47 m (1H, NCH₂), 3.97–4.08 m (1H, OCH), 4.77 d (1H, OH, *J* = 2.8 Hz), 6.77 br.s (4H, H_{arom}), 7.12 br.s (6H, H_{arom}), 8.87 s (1H, NH).

(2'*R,3a*S**,6a*R**)- and (2'*R**,3a*R**,6a*S**)-1-(1-Hydroxybutan-2-yl)-4,6-dimethyl-3a,6a-diphenyl-5-thioxooctahydroimidazo[4,5-*d*]imidazol-2-one (IIId).** Yield of diastereoisomer mixture 1.21 g (59%).

Diastereoisomer **A**. Yield 0.63 g (31%), mp 282–284°C. ¹H NMR spectrum, δ , ppm: 0.84 t (3H, Me, *J* = 7.4 Hz), 1.89–2.04 m and 2.14–2.28 m (1H each, CH₂), 2.75–2.83 m (1H, CH), 2.89 s and 3.18 s (3H each, NMe), 2.56–3.61 m and 2.69–3.75 m (1H each, OCH₂), 4.76 t (1H, OH, *J* = 5.1 Hz), 6.87–6.96 m (4H, H_{arom}), 7.08–7.18 m (6H, H_{arom}), 8.59 s (1H, NH). ¹³C NMR spectrum, δ , ppm: 12.8 (Me), 25.5 (CH₂), 30.8 and 33.8 (NMe), 57.6 (CH), 63.2 (OCH₂), 87.2 and 91.0 (C^{3a}, C^{6a}); 127.1, 128.0, 128.1, 128.2, 128.4, 128.6, 128.7, 133.1, 134.0 (C_{arom}); 158.7 (C²), 183.0 (C⁵). Found, %: C 64.42; H 6.43; N 13.59; S 7.67. C₂₂H₂₆N₄O₂S. Calculated, %: C 64.36; H 6.38; N 13.65; S 7.81.

Diastereoisomer **B**. Yield 0.58 g (28%), mp 212–214°C. ¹H NMR spectrum, δ , ppm: 0.86 t (3H, Me, *J* = 7.4 Hz), 1.43–1.59 m and 1.89–2.04 m (1H each, CH₂), 2.87–2.92 m (1H, CH), 2.92 s and 3.11 s (3H each, NMe), 3.85–4.00 m (2H, OCH₂), 4.96 t (1H, OH, *J* = 4.5 Hz), 6.87–6.96 m (4H, H_{arom}), 7.08–7.18 m (6H, H_{arom}), 8.68 s (1H, NH). ¹³C NMR spectrum, δ , ppm: 10.8 (Me), 23.2 (CH₂), 30.6 and 34.0 (NMe), 57.3 (CH), 62.2 (OCH₂), 87.8 and 90.7 (C^{3a}, C^{6a}); 127.2, 128.0, 128.1, 128.2, 128.4, 128.7, 128.8, 130.5, 132.9, 133.6 (C_{arom}); 159.1 (C²), 183.0 (C⁵). Found, %: C 64.39; H 6.44; N 13.56; S 7.72. C₂₂H₂₆N₄O₂S. Calculated, %: C 64.36; H 6.38; N 13.65; S 7.81.

4,6-Diethyl-1-(2-hydroxyethyl)-3a,6a-diphenyl-5-thioxooctahydroimidazo[4,5-*d*]imidazol-2-one (IIIe). Yield 1.85 g (90%), mp 212–215°C. ¹H NMR spectrum, δ , ppm: 1.14 t and 1.32 t (3H each, Me, *J* = 6.6 Hz), 2.88–2.96 m (1H, NCH₂), 3.03–3.12 m (2H, NCH₂), 3.37–3.56 m (4H, NCH₂, OCH₂), 3.71–3.79 m (1H, OCH₂), 4.78 t (1H, OH, *J* = 5.1 Hz), 6.74 m (4H, H_{arom}), 7.15 m (6H, H_{arom}), 8.67 s (1H, NH). ¹³C NMR spectrum, δ , ppm: 13.5, 14.4 (Me); 39.3, 40.4, 44.0 (NCH₂); 59.1 (OCH₂); 87.2, 91.7 (C^{3a}, C^{6a}); 127.3, 127.8, 128.0, 128.2, 128.9, 129.0, 132.4, 134.0 (C_{arom}); 159.0 (C²); 182.8 (C⁵). Found, %: C 64.33; H 6.34; N 13.66; S 7.74. C₂₂H₂₆N₄O₂S. Calculated, %: C 64.36; H 6.38; N 13.65; S 7.81.

4,6-Diethyl-1-[2-(4-hydroxyphenyl)ethyl]-3a,6a-diphenyl-5-thioxooctahydroimidazo[4,5-*d*]imidazol-

2-one (III_f). Yield 2.06 g (85%), mp 133–135°C. ¹H NMR spectrum, δ , ppm: 1.18 t and 1.34 t (3H each, Me, J = 6.6 Hz), 2.63–2.72 m and 2.77–2.87 m (1H each, CH₂), 2.95–3.12 m (2H, NCH₂), 3.36–3.63 m (3H, NCH₂), 3.73–3.83 m (1H, NCH₂), 6.64 m (2H, C₆H₄), 6.74 m (4H, H_{arom}), 6.97 d (2H, C₆H₄, J = 8.1 Hz), 7.08–7.16 m (6H, H_{arom}), 8.66 s (1H, NH), 9.20 s (1H, OH). Found, %: C 69.08; H 6.16; N 11.55; S 6.51. C₂₈H₃₀N₄O₂S. Calculated, %: C 69.11; H 6.21; N 11.51; S 6.59.

(2'*R,3*aS**,6*aR**)- and (2'*R**,3*aR**,6*aS**)-4,6-Diethyl-1-(2-hydroxypropyl)-3*a*,6*a*-diphenyl-5-thioxooctahydroimidazo[4,5-*d*]imidazol-2-one (III_g)** (mixture of diastereoisomers). Yield 1.4 g (66%), mp 207–209°C. Found, %: C 65.11; H 6.67; N 13.18; S 7.42. C₂₃H₂₈N₄O₂S. Calculated, %: C 65.07; H 6.65; N 13.20; S 7.55.

Diastereoisomer **A**. ¹H NMR spectrum, δ , ppm: 1.04 d (3H, Me, J = 6.2 Hz), 1.17 t (3H, Me, J = 6.6 Hz), 1.33 t (3H, Me, J = 6.7 Hz), 2.79–2.85 m (1H, CH₂), 2.95–3.11 m (2H, CH₂), 3.31–3.43 s (3H, CH₂), 3.58–3.69 m (1H, CH₂), 3.82–3.93 m (1H, CH₂), 4.05–4.13 m (1H, CH), 4.70 br.s (1H, OH), 6.63–6.88 m (4H, H_{arom}), 7.03–7.24 m (6H, H_{arom}), 8.65 s (1H, NH). ¹³C NMR spectrum, δ _C, ppm: 13.6, 14.7, 21.5 (Me); 39.5, 40.6, 48.7 (NCH₂); 65.8 (CH); 87.0, 92.2 (C^{3*a*}, C^{6*a*}); 128.0, 128.2, 129.0, 132.0, 133.7 (C_{arom}); 158.9 (C²); 182.7 (C⁵).

Diastereoisomer **B**. mp 237–239°C. ¹H NMR spectrum, δ , ppm: 1.03 d (3H, Me, J = 6.2 Hz), 1.16 t (3H, Me, J = 6.6 Hz), 1.33 t (3H, Me, J = 6.7 Hz), 2.79–2.85 m (1H, CH₂), 3.00–3.17 m (2H, CH₂), 3.31–3.43 s (3H, CH₂), 3.51–3.58 m (1H, CH₂), 3.74–3.81 m (1H, CH₂), 4.05–4.13 m (1H, CH), 4.72 br.s (1H, OH), 6.66–6.90 m (4H, H_{arom}), 7.03–7.24 m (6H, H_{arom}), 8.70 s (1H, NH). ¹³C NMR spectrum, δ _C, ppm: 13.6, 14.5, 21.2 (Me); 39.3, 40.9, 50.1 (NCH₂); 65.0 (CH); 87.2, 92.4 (C^{3*a*}, C^{6*a*}); 128.1, 128.2, 129.0, 132.3, 133.9 (C_{arom}); 159.7 (C²); 182.8 (C⁵).

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