4,5-Dihydroxyimidazolidin-2-ones in α-ureidoalkylation of *N*-carboxyalkyl-, *N*-hydroxyalkyl-, and *N*-(aminoalkyl)ureas

4.* α-Ureidoalkylation of *N*-(2-acetylaminoethyl)ureas

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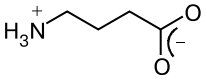
α-Ureidoalkylation of *N*-(2-acetylaminoethyl)ureas with various 4,5-dihydroxyimidazolidin-2-ones was systematically studied. Novel *N*-(2-acetylaminoethyl)glycolurils were obtained. Their yields were found to decrease both when moving from 1,3-H_2- to 1,3-Alk_2-4,5-dihydroxyimidazolidin-2-ones and when increasing the size of the substituent at the second N atom in the starting acetylaminoethylurea. The higher yields were achieved with 4,5-diphenyl-4,5-dihydroxyimidazolidin-2-one as the starting compound. 2-(2-Acetylaminoethyl)-4-methylglycoluril exhibits nootropic activity.

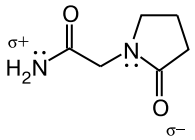
Key words: α-ureidoalkylation, *N*-carbamoylation, *N*-(2-acetylaminoethyl)glycolurils, 4,5-dihydroxyimidazolidin-2-ones, *N*-(2-acetylaminoethyl)ureas.

Glycolurils (2,4,6,8-tetraazabicyclo[3.3.0]octane-3,7-diones) constitute a new class of neurotropic compounds.[2—5] The diurnal tranquilizepr mebicar (2,4,6,8-tetramethyl-2,4,6,8-tetraazabicyclo[3.3.0]octane-3,7-dione) has been introduced into medical practice in Russia.[2]

The mechanism of action of mebicar remains unclear; its effect on the GABAergic system is assumed.[6] GABAergic effects also underlie the action of nootropics.[7] The electronic features of piracetam (nootropil) and GABA (gamma-aminobutyric acid) are somewhat similar with consideration to the zwitterionic nature of the latter.[8]



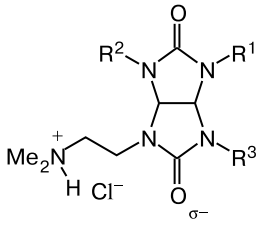
GABA



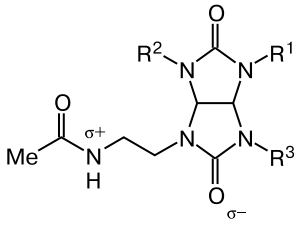
Piracetam

A glycoluril molecule (*e.g.*, **1** and **2**) can be modified in a similar way by creating a partial or integer positive charge on the amino group of a substituent as in piracetam and GABA.

The synthesis of *N*-(2-dimethylaminoethyl)glycoluril hydrochlorides **1** from 4,5-dihydroxyimidazolidin-2-ones and *N*-(2-dimethylaminoethyl)urea hydrochloride has been described previously.[1] The present study is devoted to α-ureidoalkylation of *N*-(2-acetylaminoethyl)ureas **3a—c** with 4,5-dihydroxyimidazolidin-2-ones (DHI) **4a—d** as a route to *N*-(2-acetylaminoethyl)glycolurils **2a—h**.



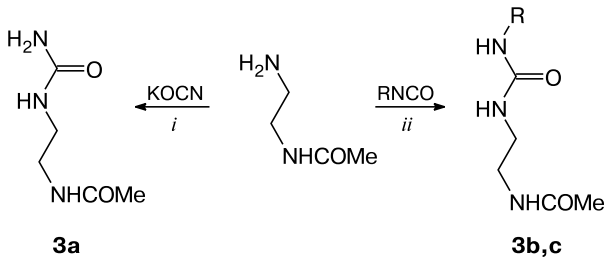
1



2

Ureas **3a—c** were prepared by *N*-carbamoylation of *N*-acetylethylenediamine with potassium cyanate or methyl(phenyl) isocyanate (Scheme 1) as described for the carbamoylation of *N*-substituted ethylenediamines.[9,10] However, in contrast to the published procedures,[9,10] all reactions were carried out in aqueous solutions.

Scheme 1



3a **3b,c**

R = Me (**b**), Ph (**c**)

i. H_2O, HCl (pH 1), reflux, 15 min; *ii*. H_2O, 30 min

* For Part 3, see Ref. 1.

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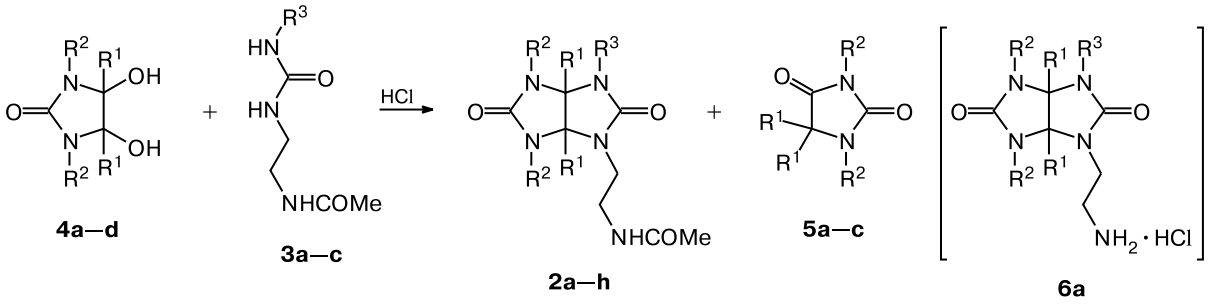
Scheme 2



4a—d **3a—c** **2a—h** **5a—c** **6a**

Compound	R^1	R^2	R^3	Compound	R^1	R^2	R^3	Compound	R^1	R^2	Compound	R^1	R^2
2a	H	H	H	**2f**	H	Me	Ph	**4a**	H	H	**5a**	H	H
2b	H	H	Me	**2g**	H	Et	H	**4b**	H	Me	**5b**	H	Me
2c	H	H	Ph	**2h**	Ph	Me	H	**4c**	H	Et	**5c**	H	Et
2d	H	Me	H	**6a**	H	H	Me	**4d**	Ph	Me			
2e	H	Me	Me										

The target glycolurils **2a**—**h** were obtained by cyclocondensation of ureas **3a**—**c** with DHI **4a**—**d** (Scheme 2) in hot water, methanol, aqueous propan-2-ol, or aqueous methanol, depending on the solubilities of the reagents and the products at pH 1. The reaction time was varied from 1 to 2.5 h.

The dependence of the yields of glycolurils **2** on the reaction time τ was studied with DHI **4a** and urea **3b** as the starting reagents. We found that the yield of compound **2b** is 44% (1 h), 43% (1.5 h), and 27% (2 h). With an increase in the time of the reaction of DHI **4a** with urea **3c**, the yield of glycoluril **2c** decreases from 42% (1.5 h) to 26% (2 h) and 19% (2.5 h). According to the ^{1}H NMR spectra of the dry-evaporated reaction mixtures, the reaction gives not only the target products **2a**—**g** but also known[11,12] hydantoins **5a**—**c** *via* dehydration of DHI **4a**—**c**. At $\tau > 1.5$ h, the reaction mixtures contain an increased fraction of hydantoins and some amount of N-deacylated aminoethylglycolurils; one of them (**6a**) was isolated and characterized. That is why the reaction time should not exceed 1—1.5 h and DHI **4a**—**c** should be used in small excess.

The yields of the target glycolurils also decrease at a reaction temperature above 80 °C (because of the formation of the corresponding hydantoin) and with an increase in the amount of hydrochloric acid, which favors the formation of deacylated aminoethylglycolurils.

While comparing the yield of glycoluril **2a** (49—52%, Table 1) with the yields of compounds **2d** (39—41%) and **2g** (18—20%) or **2b** (43—45%) and **2c** (40—42%), one can conclude that the yields of the glycolurils decrease in the presence of N-alkyl substituents in DHI **4a**—**c** (*cf.* **2a,d,g**) and, to a lesser degree, in the presence of a bulky substituent at the second N atom of the starting *N*-(2-acetylaminoethyl)ureas **3b,c** (*cf.* **2a,b,c**). A reaction of 4,5-diphenyl-DHI **4d** with urea **3a** gave the corresponding glycoluril **2h** in 65—67% yield. Apparently, the phenyl substituents stabilize the carbocations *in situ* generated from the starting DHI upon its dehydration,[13] which makes the reaction with urea more probable than the rearrangement[14] into the corresponding hydantoin.

Compounds **2a**—**h** are white hygroscopic powders. When dried over $CaCl_2$, all of them allow satisfactory elemental analysis. Glycolurils **2b** and **2f** contain water of crystallization. Structures **2a**—**h** were proved by ^{1}H and ^{13}C NMR spectroscopy (Table 2). The ^{1}H NMR spectra of the compounds obtained show singlets for the acetyl protons at δ 1.7—1.8, signals for the ethylene fragment at δ 2.7—3.5, two doublets (AB system) of the bridgehead CH—CH protons at δ 5.0—6.0, and signals for the NH protons in the acetamide fragments at δ 7.8—8.5. The ^{13}C NMR spectra exhibit signals for the methyl groups at δ 22.3—23.0 (COMe), the CH_2 groups at δ 35.5—37.9 and 40.0—42.7, the bridgehead CH atoms at δ 62.3—70.1 and 66.2—71.9 (or the bridgehead CPh atoms at δ 82.5 and 87.6), and the CO groups at δ 155.2—159.4, 158.9—161.6, and 169.3—170.5. The ^{1}H NMR spectrum of glycoluril **6a** does not contain the signals for the NH and COMe protons of the acetamide fragment.

The nootropic effect of 2-(2-acetylaminoethyl)-4-methylglycoluril **2b** was examined with mice at the Institute of Technical Chemistry (Ural Division of the Russian Academy of Sciences). Compound **2b** proved to be superior to piracetam in nootropic activity*.

Thus, when studying α-ureidoalkylation of 4,5-dihydroxyimidazolidin-2-ones with *N*-(2-acetylaminoethyl)-

* Comparative quantitative data on the nootropic activities of 2-(2-acetylaminoethyl)-4-methylglycoluril and piracetam will be published elsewhere.

Table 1. Yields, melting points, and elemental analysis data for compounds **2a**—**h**, **3a**—**c**, and **6a**

Compound	Yield (%)	T_m/°C (solvent)	Found / Calculated (%)			Molecular formula
			C	H	N	
2a	49—52	237—239 (H_2O—MeOH, 1 : 1)	42.25 42.29	5.74 5.77	30.87 30.82	$C_8H_{13}N_5O_3$
2b · H_2O	43—45	126—128 (dioxane)	41.71 41.69	6.68 6.61	26.93 27.01	$C_9H_{15}N_5O_3 \cdot H_2O$
2c	40—42	215—217 (H_2O—Pr^iOH, 1 : 2)	55.49 55.44	5.67 5.65	23.06 23.09	$C_{14}H_{17}N_5O_3$
2d	39—41	142—144 (dioxane)	47.08 47.05	6.69 6.71	27.41 27.44	$C_{10}H_{17}N_5O_3$
2e	34—36 (Ref.[20]: 34)	127—130 (acetone) (Ref.[20]: 127—130)	48.97 49.06	7.25 7.16	26.08 26.01	$C_{11}H_{19}N_5O_3$
2f · $0.5H_2O$	14—16	189—191 (dioxane)	56.41 56.46	6.43 6.51	20.63 20.57	$C_{16}H_{21}N_5O_3 \cdot 0.5H_2O$
2g	18—20	151—153 (ethyl acetate)	50.83 50.87	7.49 7.46	24.70 24.72	$C_{12}H_{21}N_5O_3$
2h	65—67	265—267 (acetone)	64.91 64.85	6.22 6.18	17.24 17.19	$C_{22}H_{25}N_5O_3$
3a	50—52	133—135 (methanol)	41.40 41.37	7.67 7.64	28.91 28.95	$C_5H_{11}N_3O_2$
3b	74—76 (Ref.[20]: 76)	155—157 (acetone) (Ref.[20]: 155—157)	45.28 45.27	8.27 8.23	26.36 26.40	$C_6H_{13}N_3O_2$
3c	81—83	189—191 (ethanol) (Ref.[10]: 191)	60.52 60.54	5.51 5.54	19.29 19.25	$C_{11}H_{12}N_3O_2$
6a	8	>300 (methanol)	35.62 35.67	6.04 5.99	29.66 29.72	$C_7H_{14}ClN_5O_2$

ureas, we found out how the yields of earlier unknown *N*-(2-acetylaminoethyl)glycolurils depend on the reaction time, the presence of substituents at the N and C atoms of 4,5-dihydroxyimidazolidin-2-ones, and the size of the substituent at the second N atom of *N*-(2-acetylaminoethyl)ureas.

Experimental

NMR spectra were recorded on Bruker AM-250 (250.13 MHz (^{1}H)) and Bruker AM-300 spectrometers (300.13 (^{1}H) and 75.5 MHz (^{13}C)) in DMSO-d_6. ^{1}H and ^{13}C chemical shifts are given on the δ scale with respect to Me_4Si as the internal standard. Melting points were determined on a GALLENKAMP instrument (Sanyo). Acetylethylenediamine and DHI were prepared according to known procedures from ethylenediamine and ethyl acetate[16] and from glyoxal (diphenylglyoxal) and ureas,[17—20] respectively.

***N*-(2-Acetylaminoethyl)urea (3a).** Concentrated HCl was gradually added to an ice-cooled solution of freshly prepared *N*-acetylethylenediamine (10.2 g, 0.1 mol) in water (5 mL) to pH 1. Then KOCN (8.1 g, 0.1 mol) was added in small portions with stirring. After the addition was completed, the reaction mixture was refluxed for 15 min and stirred for an additional 30 min without heating. On cooling, the mixture was acidified with conc. HCl to pH 1, the solvent was removed on a rotary evaporator, and the product was extracted from the residue with methanol. The precipitate of KCl was filtered off and the filtrate was concentrated on a rotary evaporator to 1/4 of its initial volume and cooled. The precipitate of urea **3a** that formed overnight was filtered off and recrystallized from methanol.

Synthesis of *N*-(2-acetylaminoethyl)-*N′*-methylurea (3b) and *N*-(2-acetylaminoethyl)-*N′*-phenylurea (3c) (general procedure). Methyl or phenyl isocyanate (0.1 mol) was added dropwise in 0.2-mL portions to an ice-cooled solution of *N*-acetylethylenediamine (10.2 g, 0.1 mol) in water (5 mL). After the addition was completed, the reaction mixture was stirred at room temperature for 30 min. The precipitate of urea **3b** that formed was filtered off, washed with a small portion of acetone, and recrystallized from boiling acetone. The precipitate of urea **3c** was filtered off, washed repeatedly with water, and recrystallized from ethanol.

Synthesis of glycolurils 2a—h (general procedure). An appropriate DHI **4a**—**d** (10.1 mmol) and an appropriate urea (10 mmol) were dissolved in water (10 mL) (for **2a,d,e,g**), water—propan-2-ol (2 : 3, 10 mL) (**2b,f**), water—methanol (3 : 2, 15 mL) (**2c**), or methanol (20 mL) (**2h**) with stirring and heating to 50 °C. The resulting solution was acidified with conc. HCl to pH 1 and stirred at 80 °C (or in boiling methanol) for 1 (for **2a,b,g**) or 1.5 h

Table 2. ^{1}H and ^{13}C NMR spectra of compounds **2a—d,f—h***, **3a—c**, and **6a**

Compound	^{1}H (DMSO-d_6), δ (*J*/Hz)	^{13}C (DMSO-d_6), δ
2a	1.78 (s, 3 H, COMe); 2.93—3.31 (m, 4 H, 2 CH_2); 5.18, 5.31 (both d, 1 H, CH, *J* = 8.1); 7.25, 7.30, 7.39, 7.84 (all br.s, 1 H, NH)	22.65 (COMe), 36.65 (CH_2), 40.01 (CH_2), 62.33 (CH), 67.74 (CH), 159.36 (CO), 161.15 (CO), 169.44 (CO)
2b	1.77 (s, 3 H, COMe); 2.63 (s, 3 H, NMe); 2.93—3.11 (m, 2 H, 2 CH_2); 3.12—3.32 (m, 2 H, CH_2); 5.10, 5.27 (both d, 1 H, CH, *J* = 8.1); 7.49, 7.56 (both br.s, 1 H, NH); 7.86 (t, 1 H, NH, *J* = 5.1)	22.86 (COMe), 28.05 (NMe), 37.08 (CH_2), 41.00 (CH_2), 66.16 (CH), 67.93 (CH), 158.36 (CO), 161.61 (CO), 170.52 (CO)
2c	1.78 (s, 3 H, COMe); 3.05—3.30 (m, 4 H, 2 CH_2); 5.41, 5.82 (both d, 1 H, CH, *J* = 8.3); 7.05 (t, 1 H, *p*-Ph, *J* = 7.3); 7.31 (t, 2 H, *m*-Ph, *J* = 7.9); 7.55 (d, 2 H, *o*-Ph, *J* = 7.9); 7.71, 7.87, 7.92 (all br.s, 1 H, NH)	22.61 (COMe), 36.21 (CH_2), 40.70 (CH_2), 65.11 (CH), 66.24 (CH), 118.48 (*o*-Ph), 122.73 (*m*-Ph), 128.73 (*p*-Ph), 138.66 (Ph), 155.17 (CO), 161.11 (CO), 169.53 (CO)
2d	1.78 (s, 3 H, COMe); 2.64 (s, 3 H, NMe); 2.82 (s, 3 H, NMe); 2.98—3.39 (m, 4 H, 2 CH_2); 5.06, 5.22 (both d, 1 H, CH, *J* = 8.3); 7.65 (br.s, 1 H, NH); 7.97 (t, 1 H, NH, *J* = 5.1)	22.59 (COMe), 27.81 (NMe), 30.14 (NMe), 36.79 (CH_2), 41.38 (CH_2), 65.65 (CH), 71.31 (CH), 158.25 (CO), 159.51 (CO), 169.52 (CO)
2f	1.78 (s, 3 H, COMe); 2.46 (s, 3 H, NMe); 2.89 (s, 3 H, NMe); 3.18—3.34 (m, 4 H, 2 CH_2); 5.39, 6.00 (both d, 1 H, CH, *J* = 8.5); 7.17 (t, 1 H, *p*-Ph, *J* = 7.3); 7.38 (t, 2 H, Ph-*m*, *J* = 7.7); 7.49 (d, 2 H, Ph-*o*, *J* = 7.7); 8.04 (t, 1 H, NH, *J* = 4.8)	22.97 (COMe), 30.54 (NMe), 30.64 (NMe), 37.00 (CH_2), 42.67 (CH_2), 69.74 (CH), 70.04 (CH), 123.21 (*o*-Ph), 125.25 (*m*-Ph), 129.27 (*p*-Ph), 138.71 (Ph), 156.49 (CO), 158.87 (CO), 170.06 (CO)
2g	1.04 (m, 6 H, 2 Me); 1.79 (s, 3 H, COMe); 2.95—3.25 (m, 6 H, 3 NCH_2); 3.29—3.50 (m, 8 H, 4 NCH_2); 5.18 (dd, 1 H, CH, *J* = 8.3, *J* = 1.9); 5.37 (d, 1 H, CH, *J* = 8.3); 7.67 (s, 1 H, NH); 7.97 (t, 1 H, NH, *J* = 5.4)	12.91 (Me), 13.02 (Me), 22.53 (COMe), 35.53 (CH_2), 36.74 (CH_2), 37.10 (CH_2), 41.28 (CH_2), 63.79 (CH), 68.68 (CH), 157.42 (CO), 159.50 (CO), 169.44 (CO)
2h	1.74 (s, 3 H, COMe); 2.61 (s, 3 H, NMe); 2.86 (s, 3 H, NMe); 2.77—2.91 (m, 1 H, CH_2); 3.21—3.38 (m, 3 H, CH_2); 6.79 (m, 2 H, Ph); 6.89 (m, 2 H, Ph); 7.09 (m, 6 H, Ph); 7.95 (br.s, 1 H, NH); 8.52 (s, 1 H, NH)	22.33 (COMe), 26.13 (NMe), 27.75 (NMe), 37.91 (CH_2), 42.05 (CH_2), 82.48 (CPh), 87.64 (CPh), 127.22, 127.90, 128.04, 128.14, 128.20, 128.39, 132.84, 134.64 (all Ph), 158.42 (CO), 159.52 (CO), 169.32 (CO)
3a	1.78 (s, 3 H, COMe); 2.98—3.05 (m, 4 H, 2 CH_2); 5.49 (s, 2 H, NH_2); 5.99 (br.s, 1 H, NH); 7.89 (br.s, 1 H, NH)	—
3b	1.79 (s, 3 H, COMe); 2.55 (s, 3 H, NMe); 2.97—3.07 (m, 4 H, 2 CH_2); 5.81 (m, 1 H, NH); 5.95 (br.s, 1 H, NH); 7.87 (t, 1 H, NH, *J* = 4.8)	—
3c	1.81 (s, 3 H, COMe); 3.12 (br.s, 4 H, 2 CH_2); 6.17 (br.s, 1 H, NH); 6.88 (t, 1 H, *p*-Ph, *J* = 7.0); 7.21 (t, 2 H, *m*-Ph, *J* = 7.8); 7.39 (d, 2 H, *o*-Ph, *J* = 7.8); 7.94 (br.s, 1 H, NH); 8.54 (s, 1 H, NH)	—
6a	2.62 (s, 3 H, NMe); 2.99—3.14 (m, 2 H, 2 CH_2); 3.21—3.34 (m, 2 H, CH_2); 5.12, 5.27 (both d, 1 H, CH, *J* = 7.9); 7.43, 7.57 (both br.s, 1 H, NH)	—

* The ^{1}H and ^{13}C NMR spectra of glycoluril **2e** were cited earlier.[15]

(**2c—f,h**). The solvent was removed on a rotary evaporator. Compounds **2d—g** were extracted from the oily residue with boiling ether (4×5 mL) and triturated with ethyl acetate—methanol (1 : 1) (for **2a**), propan-2-ol (**2b,c,f**), ethyl acetate—ether (1 : 1) (**2d,e,g**), or water (**2h**). The precipitate that formed was filtered off and recrystallized from an appropriate solvent (see Table 1). This procedure was used to obtain 2-(2-acetylaminoethyl)-2,4,6,8-tetraazabicyclo[3.3.0]octane-3,7-dione (**2a**), 2-(2-acetylaminoethyl)-4-methyl-2,4,6,8-tetraazabicyclo[3.3.0]octane-3,7-dione (**2b**), 2-(2-acetylaminoethyl)-4-phenyl-2,4,6,8-tetraazabicyclo[3.3.0]octane-3,7-dione (**2c**), 6-(2-acetylaminoethyl)-2,4-dimethyl-2,4,6,8-tetraazabicyclo[3.3.0]octane-3,7-dione (**2d**), 2-(2-acetylaminoethyl)-4,6,8-trimethyl-2,4,6,8-tetraazabicyclo[3.3.0]octane-3,7-dione (**2e**), 2-(2-acetylaminoethyl)-6,8-dimethyl-4-phenyl-2,4,6,8-tetraazabicyclo[3.3.0]octane-3,7-dione (**2f**), 6-(2-acetylaminoethyl)-2,4-diethyl-2,4,6,8-tetraazabicyclo[3.3.0]octane-3,7-dione (**2g**), and 6-(2-acetylaminoethyl)-2,4-dimethyl-1,5-diphenyl-2,4,6,8-tetraazabicyclo-[3.3.0]octane-3,7-dione (**2h**).

2-(2-Aminoethyl)-4-methylglycoluril hydrochloride (6a). Concentrated HCl (0.2 mL) was added to a solution of DHI **4a** (1.18 g, 10 mmol) and urea **3b** (1.59 g, 10 mmol) in water (10 mL). The

reaction mixture was stirred at 80 °C for 2 h. The solvent was removed on a rotary evaporator and the oily residue was triturated with acetone—methanol (1 : 1). The precipitate of compound **6a** that formed was filtered off and recrystallized. The filtrate was concentrated *in vacuo* and the product was extracted from the residue with boiling ether (4×5 mL) and triturated with propan-2-ol. The resulting precipitate of hydrate **2b**·H_2O (0.51 g, 20%) was filtered off and recrystallized.

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