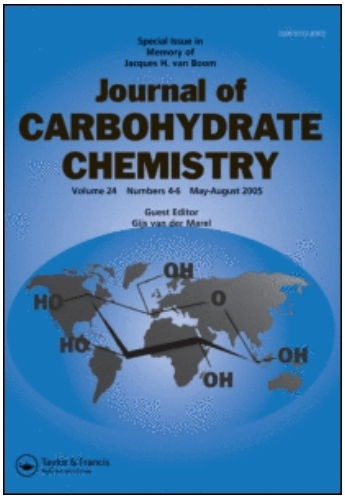


Journal of Carbohydrate Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713617200

SYNTHESIS OF NEW OXAMIDE DERIVATIVES OF D-GLUCOSAMINE AND ALIPHATIC OR AROMATIC AMINES

Andrzej Temeriusz[a]; Boguslawa Piekarska-Bartoszewicz[a]; Magdalena Rowińska[a]
[a] Department of Chemistry, Warsaw University, Warszawa, Poland

Online publication date: 30 April 2001

To cite this Article Temeriusz, Andrzej , Piekarska-Bartoszewicz, Boguslawa and Rowińska, Magdalena(2001) 'SYNTHESIS OF NEW OXAMIDE DERIVATIVES OF D-GLUCOSAMINE AND ALIPHATIC OR AROMATIC AMINES', Journal of Carbohydrate Chemistry, 20: 3, 275 — 283
To link to this Article: DOI: 10.1081/CAR-100104863
URL: http://dx.doi.org/10.1081/CAR-100104863

J. CARBOHYDRATE CHEMISTRY, 20(3&4), 275–283 (2001)

SYNTHESIS OF NEW OXAMIDE DERIVATIVES OF D-GLUCOSAMINE AND ALIPHATIC OR AROMATIC AMINES

Andrzej Temeriusz, Boguslawa Piekarska-Bartoszewicz, and Magdalena Rowińska

Department of Chemistry, Warsaw University, Pasteura 1,
02-093 Warszawa, Poland

ABSTRACT

New oxamides, derivatives of D-glucosamine and aliphatic or aromatic amines were prepared by acylation of methyl 3,4,6-tri-*O*-acetyl-2-acetamido-2-deoxy-α- or -β-D-glucopyranoside (**1c** or **1d**) with oxalyl chloride, followed by reaction with amine. The reaction was assumed to proceed by the intermediate of *N*-carbomethoxy *N*-(methyl 3,4,6-tri-*O*-acetyl-2-deoxy-α or β-D-glucopyranosid-2-yl) oxamic acid chloride which reacted with amines, and afforded *N*-acetyl, *N*-(methyl 3,4,6-tri-*O*-acetyl-2-deoxy-α- or -β-D-glucopyranosid-2-yl), *N′*-alkyl or aryloxamide (**5–7**), and *N*-(methyl 3,4,6-tri-*O*-acetyl-2-deoxy-α- or -β-D-glucopyranosid-2-yl), *N′*-alkyl or aryloxamide (**8–13**).

INTRODUCTION

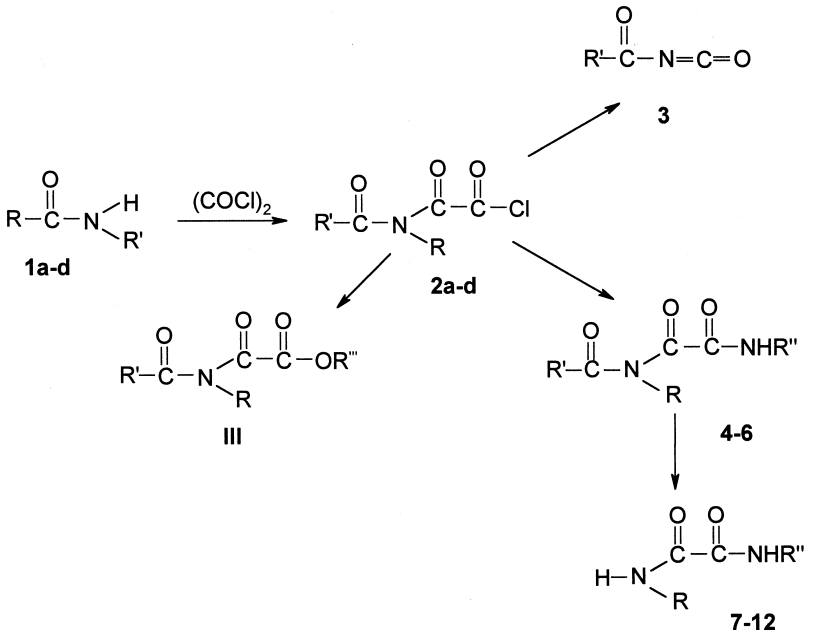
The chemistry of compounds containing the oxalato moiety is an active area of research, the interest in these compounds arising from their applications to catalysis, magnetochemistry, organometalic complexes, and chelating agents.[1] Oxamides are also useful intermediates in the synthesis of α-diketones and important synthons in organic chemistry.[2] Some of them show biological activity as pesticides,[3] plant growth regulators[4], cephalosporin bactericides,[5] and HIV-1 protease inhibitors.[6] Only two methods for preparing *N*,*N′* unsymmetrically substituted oxamides havebeen reported. P.H. Dixneuf *et al*. described the preparation of a series of oxamides, oxamates and oxalates from diisopropenyloxalate.[7] *N′*-Substituted *N*-

furyloxamides were prepared by condensation of dimethyl oxalate with furylamine using 1,1′-oxalyldiimidazole, followed by hydrolysis of the resulting amidoester, and condensation with aliphatic or aromatic amines.[4] Recently Katritzky *et al.* reported[8] a synthesis of *N*,*N*′ unsymmetrically substituted oxamides using 1,1′-(1,2-dioxoethane-1,2-diyl)bis-1*H*-benzotriazole. In this paper we wish to describe the synthesis of new oxamides, derivatives of D-glucose and aliphatic or aromatic amines.

RESULTS AND DISCUSSION

According to the literature, the reaction of amides (**1**) with oxalyl chloride gives *N*-acyl, *N*-oxamyl chlorides (**2**). In the case of primary amides (**1a**, R = H) *N*-acyl, *N*-oxamyl chlorides (**2a**, R = H) are unstable and the corresponding acyl isocyanates (**3**) can be obtained.[9] In contrast, the reaction of *N*-alkyl or *N*-aryl amides (**1b**, R = alkyl or aryl) with oxalyl chloride under similar conditions gave stable products (**2b**, R = alkyl or aryl).[10] The reaction of **2b** with an alcohol gave *N*-acyl oxamates (**4**).[10]

The present paper deals with the synthesis of unsymmetrical oxamide derivatives of D-glucosamine and aliphatic or aromatic amines starting from easily available methyl 3,4,6-tri-*O*-acetyl-2-acetamido-2-deoxy-α- or β-D-glucopyranoside (Scheme 1, **1c**[11] or **1d**[12]) and oxalyl chloride. Treatment of **1c** or **1d** with three-fold excess of oxalyl chloride in methylene chloride at −5°C, followed by the addition of amine (aniline, *p*-chloroaniline, diethylamine, *N*-methylaniline and *N*-ethylani-

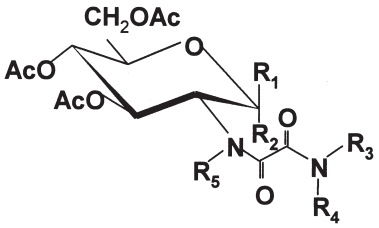


Scheme 1.

MARCEL DEKKER, INC.
270 Madison Avenue, New York, New York 10016

line) at room temperature afforded the corresponding oxamides (**5–13**) which were purified by chromatography on silica gel. The reaction was assumed to proceed by the intermediate *N*-acetyl *N*-(methyl 3,4,6-tri-*O*-acetyl-2-deoxy-α or β-D-glucopyranosid-2-yl) oxamic acid chloride (Scheme 1, **2c, 2d**) which react with amine at −15°C, and afforded *N*-acetyl, *N*-(methyl 3,4,6-tri-*O*-acetyl-2-deoxy-α- or -β-D-glucopyranosid-2-yl), *N′*-alkyl or aryloxamide (**5–7**). The reaction of **1c** or **1d** with oxalyl chloride and amine under drastic conditions (higher temperature) or deacetylation of *N*-acetyl oxamide **5–7** with sodium methoxide and acetylation with acetic anhydride in pyridine at room temperature gave the corresponding oxamides **8–13.**

The structures of **8–13** were confirmed by ^{1}H and ^{13}C NMR. The ^{1}H NMR spectral data are given in the Experimental section. The spectra showed signals corresponding to the glucopyranosyl and amine residues. The signals of protons on the NH groups attached to C-2 of glucopyranosyl residue are split into upfield doublets with $J_{2,NH}$ 9.0–9.5 Hz and appeared upfield (**8–13:** $\delta \approx 7.1$–7.7 ppm,) with respect to those of the NH groups attached to the aromatic residue (**8** and **9:** $\delta \approx 9.2$ ppm).



2 R_1= H, R_2= OMe, R_3 = H, R_4 = Ph, R_5 = H

3 R_1= H, R_2= OMe, R_3 = H, R_4 = *p*ClPh, R_5 = H

4 R_1= H, R_2= OMe, R_3 = Et, R_4 = Et, R_5 = H

5 R_1= H, R_2= OMe, R_3 = Me, R_4 = Ph, R_5 = H

6 R_1= OMe, R_2= H, R_3 = Me, R_4 = Ph, R_5 = H

7 R_1= OMe, R_2= H, R_3= Et, R_4 = Ph, R_5 = H

8 R_1= H, R_2= OMe, R_3= Me, R_4 = Ph, R_5 = OAc

9 R_1= OMe, R_2= H, R_3= Me, R_4 = Ph, R_5 = OAc

10 R_1= OMe, R_2= H, R_3= Et, R_4 = Ph, R_5 = OAc

Scheme 2.

The 2D COSY and HETCOR spectra were also recorded (^{1}H-^{13}C HETCOR spectrum for compound **10** is presented in Figure 1 and for compound **7** in Figure 2). The interpretation of the two-dimension spectrum revealed that in the ^{13}C NMR spectra the order of chemical shifts of sugar carbon signals in the range 75–65 ppm should be: C-3 > C-4 > C-5 (α-anomers) [13] and C-5 > C-3 > C-4 (β-anomers).

EXPERIMENTAL

Melting points were uncorrected. Optical rotations were measured on a Perkin-Elmer Model 241. NMR spectra were recorded in solutions with $CDCl_3$ (internal reference Me_4Si) with a Varian Unity Plus-500 spectrometer. TLC was performed on Silica Gel 60 F_{254} Merck), using dichloromethane-methanol (4:1) as eluent and detection by UV light or by charring with sulfuric acid. Column chromatography was conducted on Silica Gel 60 (Merck 230–400 mesh) with dichloromethane-methanol (4:1) as eluent.

Synthesis of *N*-acetyloxamido derivatives. To a solution of methyl 3,4,6-tri-*O*-acetyl-2-acetamido-2-deoxy-α- or -β-D-glucopyranoside (**1c**[11] or **1d**[12]) (1.4 mmol) in ichloromethane (10 mL) was added a solution of oxalyl chloride (4 mmol) in dichloromethane (10 mL). The reaction mixture was stirred at −5°C

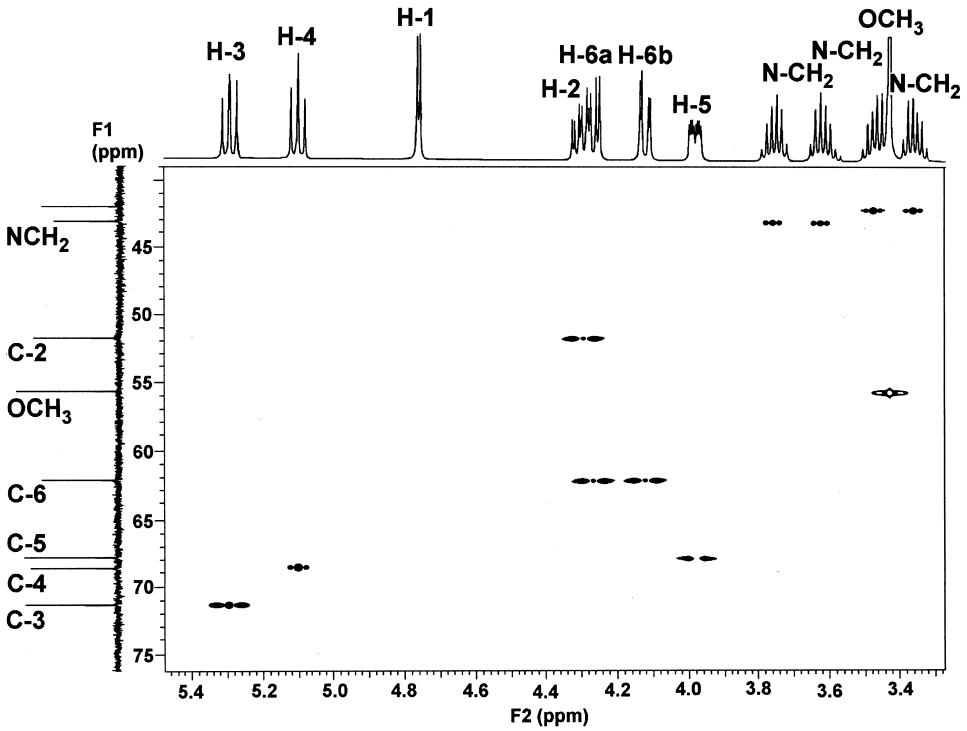


Figure 1. ^{1}H-^{13}C HETCOR spectrum of **10**.

MARCEL DEKKER, INC.
270 Madison Avenue, New York, New York 10016

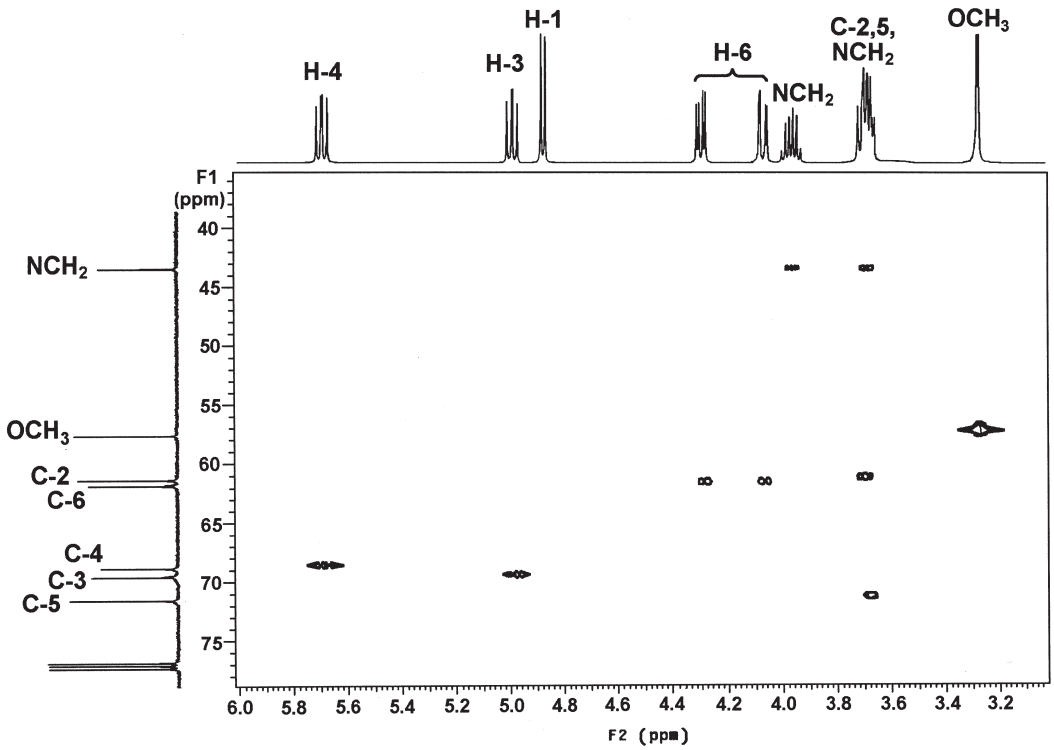


Figure 2. ^{1}H-^{13}C HETCOR spectrum of **7.**

for 10 min and at room temperature during 30 min. TLC then indicated the absence of **1**. Next the reaction mixture was cooled again (−15°C) and amine (5 mmol) was added. The mixture was stirred at −5°C for 2 h. The resulting mixture was successively washed with hydrochloric acid (1M), water, and the saturated solution of sodium hydrogen carbonate, and then dried over magnesium sulfate. The solvent was evaporated in *vacuo* and gave the desired product as a viscous oil which was purified by chromatography with 4:1 dichloromethane − methanol (4:1) as eluent. The product was recrystallized from ethanol.

***N*-acetyl, *N*-(methyl 3,4,6-tri-*O*-acetyl-2-deoxy-α-D-glucopyranosid-2-yl), *N′*-methyl, *N′*-phenyloxamide (5),** ^{1}H NMR δ 7.40–7.28 (m, 5H, Ph), 5.85 (dd, 1H, $J_{4,5}$ = 10.5 Hz, H-4), 4.99 (dd, 1H, $J_{3,4}$ = 9.0 Hz, H-3), 4.68 (m, 1H, H-2), 4.54 (d, 1H, $J_{1,2}$ = 3.0 Hz, H-1), 4.27 (dd, 1H, $J_{5,6a}$ = 4.5 Hz, $J_{6a,6b}$ = 12.0 Hz, H-6a), 4.08 (dd, 1H, $J_{5,6b}$ = 2.0 Hz, H-6b), 4.02 (ddd, 1H, H-5), 3.34 (s, 3H, OCH_3), 3.33 (s, 3H, NCH_3), 2.50 (s, 3H, NAc), 2.07, 2.00, 1.95 (3s, 9H, 3OAc).

***N*-acetyl, *N*-(methyl 3,4,6-tri-*O*-acetyl-2-deoxy-β-D-glucopyranosid-2-yl), *N′*-methyl, *N′*-phenyloxamide (6),** ^{1}H NMR δ 7.35–7.28 (m, 5H, Ph), 5.84 (dd, 1H, $J_{4,5}$ = 9 Hz, H-4), 4.98 (dd, 1H, $J_{3,4}$ = 10.5 Hz, H-3), 4.69 (m, 1H, H-2), 4.54 (d, 1H, $J_{1,2}$ = 3.0 Hz, H-1), 4.27 (dd, 1H, $J_{5,6a}$ = 4.5 Hz, $J_{6a,6b}$ = 12.0 Hz, H-

6a), 4.08 (dd, 1H, $J_{5,6b}$ = 2.5 Hz, H-6b), 4.03 (ddd, 1H, H-5), 3.34 (s, 3H, OCH_3), 3.31 (s, 3H, NCH_3), 2.36 (s, 3H, NAc), 2.06, 2.00, 1.98 (3s, 9H, 3OAc).

***N*-acetyl, *N*-(methyl 3,4,6-tri-*O*-acetyl-2-deoxy-β-D-glucopyranosid-2-yl), *N′*-ethyl, *N′*-phenyloxamide (7),** ^{1}H NMR δ 7.36–7.28 (m, 5H, Ph), 5.69 (dd, 1H, $J_{4,5}$ = 9.5 Hz, H-4), 4.99 (dd, 1H, $J_{3,4}$ = 10.0 Hz, H-3), 4.87 (d, 1H, $J_{1,2}$ = 7.5 Hz, H-1), 4.30 (dd, 1H, $J_{5,6a}$ = 4.0 Hz, $J_{6a,6b}$ = 12.0 Hz, H-6a), 4.06 (dd, 1H, $J_{5,6b}$ = 2.5 Hz, H-6b), 3.96 (m, 1H, NCH_2), 3.71–3.65 (m, 3H, H-2, H-5, NCH_2), 3.27 (s, 3H, OCH_3), 2.36 (s, 3H, NAc), 2.06, 2.00, 1.98 (3s, 9H, 3OAc), 1.18 (t 3H, CH_3).

Synthesis of oxamido derivatives. To a solution of methyl 3,4,6-tri-*O*-acetyl-2-acetamido-2-deoxy-α- or β-D-glucopyranoside (**1c**[11] or **1d**[12]) (1.4 mmol) in dichloromethane (10 mL) was added a solution of oxalyl chloride (4 mmol) in dichloromethane (10 mL). The reaction mixture was stirred at −5°C for 10 min and at room temperature during 30 min. TLC then indicated the absence of **1**. Next the reaction mixture was cooled again (0°C) and amine (10 mmol) was added. The mixture was stirred at room temperature for 2 h. The resulting mixture was successively washed with hydrochloric acid (1M), water, and then a saturated solution of sodium hydrogen carbonate, and then dried over magnesium sulfate. The solvent was evaporated in *vacuo* and gave the desired product as a viscous oil which was purified by chromatography with dichloromethane - methanol (4:1) as eluent. The product was recrystallized from ethanol.

***N*-(methyl 3,4,6-tri-*O*-acetyl-2-deoxy-α-D-glucopyranosid-2-yl), *N′*-phenyloxamide (8),** ^{1}H NMR δ 9.23 (s, 1H, N′HPh), 7.76 (d, 1H, NHGlcp), 7.66–7.19 (m, 5H, Ph), 5.37 (dd, 1H, $J_{3,4}$ = 10.0 Hz, H-3), 5.14 (dd, 1H, $J_{4,5}$ = 9.5 Hz, H-4), 4.79 (d, 1H, $J_{1,2}$ = 3.0 Hz, H-1), 4.33 (ddd, 1H, $J_{2,NH}$ = 9.5 Hz, H-2), 4.30 (dd, 1H, $J_{5,6a}$ = 4.5 Hz, $J_{6a,6b}$ = 12.0 Hz, H-6a), 4.13 (dd, 1H, $J_{5,6b}$ = 2.5 Hz, H-6b), 4.01 (ddd, 1H, H-5), 3.44 (s, 3H, OCH_3), 2.11, 2.03, 1.98 (3s, 9H, 3Ac).

***N*-(methyl 3,4,6-tri-*O*-acetyl-2-deoxy-α-D-glucopyranosid-2-yl), *N′*-*p*-chlorophenyloxamide (9),** ^{1}H NMR δ 9.21 (s, 1H , N′HPh), 7.72 (d, 1H, NHGlcp), 7.62, 7.34 (4d, 4H, Ph), 5.35 (dd, 1H, $J_{3,4}$ = 10.0 Hz, H-3), 5.15 (dd, 1H, $J_{4,5}$ = 9.5 Hz, H-4), 4.78 (d, 1H, $J_{1,2}$ = 3.0 Hz, H-1), 4.33 (ddd, 1H, $J_{2,NH}$ = 9.0 Hz, H-2), 4.30 (dd, 1H, $J_{5,6a}$ = 5.0 Hz, $J_{6a,6b}$ = 12.0 Hz, H-6a), 4.13 (dd, 1H, $J_{5,6b}$ = 2.5 Hz, H-6b), 4.01 (ddd, 1H, H-5), 3.44 (s, 3H, OCH_3), 2.12, 2.04, 1.99 (3s, 9H, 3Ac).

***N*-(methyl 3,4,6-tri-*O*-acetyl-2-deoxy-α-D-glucopyranosid-2-yl), *N′N′*-diethyloxamide (10),** ^{1}H NMR δ 7.31 (d, 1H, $J_{2,NH}$ = 10.8 Hz NHGlcp), 5.30 (dd, 1H, $J_{3,4}$ = 9.5 Hz, H-3), 5.10 (dd, 1H, $J_{4,5}$ = 10.0 Hz, H-4), 4.76 (d, 1H, $J_{1,2}$ = 3.5 Hz, H-1) 4.31 (ddd, 1H, $J_{2,3}$ = 9.5 Hz, H-2), 4.27 (dd, 1H, $J_{5,6a}$ = 4.5 Hz, $J_{6a,6b}$ = 12.0 Hz, H-6a), 4.12 (dd, 1H, $J_{5,6b}$ = 2.5 Hz, H-6b), 3.98 (ddd, 1H, H-5), 3.75, 3.62, 3.46, 3.35 (4m, 4H, $2NCH_2$), 3.42 (s, 3H, OCH_3), 2.10, 2.03, 1.99 (3s, 9H, 3Ac), 1.23, 1.79 (2t, 6H, $3CH_3$).

Table 1. Analytical Data for Compounds **5–13**

Compound	Yield (%)	Mp (°C)	$[\alpha]_D$ (Degrees c 1, $CHCl_3$)	Formula	Analytical Data							
					Calcd				Found			
					C	H	N	Cl	C	H	N	Cl
5	60	118–120	+156.4	$C_{24}H_{30}O_{11}N_2$	55.17	5.79	5.36		55.27	5.83	5.23	
6	4	186–187	−35.7	$C_{24}H_{30}O_{11}N_2$	55.17	5.79	5.36		55.20	5.62	5.46	
7	45	176–179	−42.3	$C_{25}H_{32}O_{11}N_2$	55.96	6.01	5.21		56.01	6.04	5.19	
8	42	164–166	−56.7	$C_{21}H_{26}O_{10}N_2$	54.08	5.62	6.01		54.21	5.75	6.08	
9	53	205–207	+65.1	$C_{21}H_{25}O_{10}N_2Cl$	50.36	5.03	5.59	7.08	50.47	5.11	5.58	7.14
10	71	133–136	+76.3	$C_{19}H_{30}O_{10}N_2$	51.12	6.77	6.27		51.22	6.83	6.29	
11	62	143–145	+42.4	$C_{22}H_{28}O_{10}N_2$	55.00	5.87	5.83		55.02	5.97	5.99	
12	58	206–207	+10.3	$C_{22}H_{28}O_{10}N_2$	55.00	5.87	5.83		55.09	5.89	5.92	
13	54	198–199	+11.7	$C_{23}H_{30}O_{10}N_2$	55.87	6.11	5.67		56.12	6.25	5.66	

_N_-(methyl 3,4,6-tri-_O_-acetyl-2-deoxy-α-D-glucopyranosid-2-yl), _N′_-methyl, _N′_-phenyloxamide (11), ^{1}H NMR δ 7.38–7.31 (m, 3H, Ph), 7.14–7.13 (m, 3H, Ph, NHGlcp), 5.25 (dd, 1H, $J_{3,4}$ = 9.5 Hz, H-3), 5.03 (dd, 1H, $J_{4,5}$ = 10.0 Hz, H-4), 4.58 (d, 1H, $J_{1,2}$ = 3.0 Hz, H-1) 4.20 (dd, 1H, $J_{5,6a}$ = 4.5 Hz, $J_{6a,6b}$ = 12.5 Hz, H-6a), 4.10 (ddd, 1H, $J_{2,NH}$ = 9.0 Hz, H-2), 4.08 (dd, 1H, $J_{5,6b}$ =

Table 2. ^{13}C NMR Data (δ in ppm,) in $CDCl_3$ for Peracetylated Methyl α and β-D-Glucopyranosyloxamides **(5–13)**

Atom	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**
C-1	98.66	99.48	99.48	97.94	97.85	98.14	98.00	100.9	101.0
C-2	55.64	61.44	61.39	52.57	52.59	51.60	51.61	54.78	54.67
C-3	70.11	69.51	69.51	70.90	70.86	71.10	70.80	71.70	71.79
C-4	69.24	68.86	68.81	68.30	68.14	68.47	68.25	68.73	68.92
C-5	67.62	71.57	71.52	67.71	67.66	67.68	67.57	71.58	71.79
C-6	61.82	61.87	61.87	61.95	61.87	62.03	61.93	62.04	62.14
O-CH_3	55.37	57.65	57.65	55.61	55.61	55.55	55.54	56.78	56.78
C = 0	166.4, 162.9	166.5, 162.3	166.5, 161.7	160.1, 156.5	159.8, 156.5	161.5, 160.9	161.6, 160.4	162.4, 160.5	161.5, 160.5
CH_3CON	25.84	23.62	23.68						
CH_3COO	20.53	20.75, 20.64, 20.43	20.64, 20.43	20.73, 20.66, 20.61	20.74, 20.69, 20.62	20.73, 20.70, 20.63	20.70, 20.75, 20.60	20.64	20.64
CONR	175.4	173.6	173.5						
COOR	170.4, 170.1, 169.5	170.6, 169.8, 169.1	170.6, 169.8, 169.0	170.6, 169.5,	170.7, 169.5,	170.7, 170.6, 169.5	170.7, 170.6, 169.4	170.7, 170.3, 169.4	170.6, 170.2, 169.4
N-CH_2	36.35		43.51			43.13, 42.03			46.19
C-CH_3			12.19			14.76, 12.50			12.35
N-CH_3		36.14					38.77	38.74	36.35
C_{aromat}	142.3, 129.2, 127.8, 126.8	142.2, 129.3, 127.2, 126.6	140.4, 129.2, 127.9, 127.2	136.2, 129.2, 125.6, 119.7	134.8, 130.6, 129.3, 120.9		143.3, 129.3, 127.8, 126.8	143.3, 129.2, 127.6, 125.5	141.7, 129.1, 127.5, 126.6

2.0 Hz, H-6b), 3.92 (ddd, 1H, H-5), 3.49 (s, 3H, OCH_3), 3.33 (s, 3H, NCH_3), 2.06, 2.00, 1.96 (3s, 9H, 3Ac).

***N*-(methyl 3,4,6-tri-*O*-acetyl-2-deoxy-β-D-glucopyranosid-2-yl), *N′*-methyl, *N′*-phenyloxamide (12),** ^{1}H NMR δ 7.38–7.13 (m, 6H, Ph, NHGlcp), 5.31 (dd, 1H, $J_{3,4}$ = 10.5 Hz, H-3), 4.98 (dd, 1H, $J_{4,5}$ = 9 Hz, H-4), 4.63 (d, 1H, $J_{1,2}$ = 8.0 Hz, H-1), 4.22 (dd, 1H, $J_{5,6a}$ = 4.5 Hz, $J_{6a,6b}$ = 12.0 Hz, H-6a), 4.10 (dd, 1H, $J_{5,6b}$ = 2.5 Hz, H-6b), 3.7–3.6 (m 2H, H-2, H-5), 3.43 (s, 3H, OCH_3), 3.39(s, 3H, NCH_3), 2.06, 2.010, 1.99 (3s, 9H, 3OAc).

N-(methyl 3,4,6-tri-O-acetyl-2-deoxy-β-D-glucopyranosid-2-yl), N′-ethyl, N′-phenyloxamide (13), ^{1}H NMR δ 7.38–7.09 (m, 6H, Ph, NHGlcp), 5.29 (dd, 1H, $J_{3,4}$ = 10.5 Hz, H-3), 4.97 (dd, 1H, $J_{4,5}$ = 9 Hz, H-4), 4.61 (d, 1H, $J_{1,2}$ = 8.5 Hz, H-1), 4.23 (dd, 1H, $J_{5,6a}$ = 5.0 Hz, $J_{6a,6b}$ = 12.0 Hz, H-6a), 4.10 (dd, 1H, $J_{5,6b}$ = 2.5 Hz, H-6b), 3.8–3.7 (m 2H, H-2, NCH_2), 3.67–3.62 (m, 2H, H-5, NCH_2), 3.42 (s, 3H, OCH_3), 2.06, 2.00, 1.99 (3s, 9H, 3OAc), 1.16 (t, 3H, CH_3).

ACKNOWLEDGMENTS

This work was supported by Warsaw University, Grant BST 662/14/2000.

REFERENCES

1. Casellato, U.; Guerriero, P.; Tamburini, S.; Vigato, P.A. Metal Complexes with Disubstituted Oxamic Ligands. Inorg. Chim. Acta **1997**, *260*, 1–9.
2. Mueller-Westerhoff, U.T.; Zhou, M. A Simple Synthesis of Symmetrical α-Diones from Organometalic Reagents and 1,4-Dimethylpiperazine-2,3-dione.Tetrahedron Lett. **1993**, *34*, 571–574. Mueller-Westerhoff, U.T.; Zhou, M. Synthesis of Symmetrically and Unsymmetrically Substituted α-Diones from Organometallic Reagents and 1,4-Dialkylpiperazine-2,3-diones. J. Org. Chem. **1994**, *59*, 4988–1992.
3. Boger, M.; Drabek J.; Neumann, R. Brit. U.K. Pat Appl. GB 2.145,716, 1984.
4. Kitagawa, T.; Tsutsui, C.; Hayashi, K.; Yamato, A. Preparation and Plant Growth Regulatory Activity of *N′*-Substituted *N*-Furfuryloxamides. Chem. Pharm. Bull. **1998**, *46*, 514–517; Kitagawa, T.; Tsutsui, C. Preparation and Plant Growth Regulatory Activity of *N′*-Chloro-Substituted Phenyl- and Benzyl-*N*-furfuryloxamides. *ibid.* **1998**, *46*, 1308–1310, Kitagawa, T.; Tsutsui, C. Preparation of *N′*-[2-(5,6-Dimethylbenzothiazolyl)]-*N*-furfuryloxamide with Plant Growth Regulatory Activity. *ibid.* **2000**, *48*, 1363–1366.
5. Treuner, U.D.; Breuer, H. U.S.Patent 4,113,943, 1978.
6. Jadhav, P.K.; Man, H.W. Synthesis of 7-Membered Cyclic Oxamides: Novel HIV-1 Protease Inhibitors. Tetrahedron Lett. **1996**, *37*, 1153–1156; Medou, M.; Priem, G.; Quélever, G.; Camplo, M.; Kraus, J.K. Synthesis of New 7-Membred α-Phenylthio Cyclic Oxamides: HIV-Inhibitors. Tetrahedron Lett. **1998**, *39*, 4021–4024.
7. Neveux, M.; Bruneau, C.; Lécolier S.; Dixneuf, P.H. Novel Synthesis of Oxamides, Oxamates and Oxalates from Diisopropenyl Oxalate. Tetrahedron **1993**, *49*, 2629–2640.

8. Katritzky, A.R.; Levell, J.R.; Pleynet, D.P.M. General Synthesis of Unsymmetrical Tetrasubstituted Oxamides via 1,1′-(1,2-Dioxoethane-1,2-diyl)bis-1*H*-benzotriazole. Synthesis **1998**, (*2*), 153–156.
9. Berrée, F.; Michelot, G.; Le Corre, M. *N*-Boc Ethyl Oxamate: a New Nitrogen Nucleophile for Use in Mitsunobu Reactions. Tetrahedron Lett. **1998,** *39*, 8275–8276; Bazureau, J.P.; Le Roux, J.; Le Corre, M. A New Rout to Heterocyclic α-Dehydro α-Amino Esters. Tetrahedron Lett. **1988**, *29*, 1921–1922; Speziale A.J.; Smith, L.R. A New and Convenient Synthesis of Acyl Isocyanates. J. Org. Chem. **1962**, *27*, 3742–3743.
10. Stoffel, P.J. The Preparation of Oxazolidinetriones. A Novel Cyclic Anhydride Derived from Carbamates and Oxalyl Chloride. J. Org. Chem. **1964**, *29*, 2796–2799.
11. Flowers, M.H.; Jeanloz, R.W. The Synthesis of the Repeating Unit of Hyaluronic Acid. Biochemistry **1964**, *3*, 123–125.
12. Inouye, Y.; Onodera, K.; Kitaoka, S.; Ochiai, H. An Acyl Migration in Acetohalogenoglucosamines. J. Am. Chem. Soc. **157**, *79*, 4218–4222.
13. Wawer, I.; Piekarska-Bartoszewicz, B.; Temeriusz, A. ^{13}C CP MAS and High-Resolution ^{1}H, ^{13}C, ^{15}N NMR Study of New Ureido Sugars, Derivatives of 2-Amino-2-deoxy-β-D-glucopyranose and L-Amino Acid. Carbohydr. Res. **1995**, *279*, 83–91.

Received July 20, 2000
Accepted March 13, 2001

Request Permission or Order Reprints Instantly!