Carbohydrate Research, 209 (1991) 311–318
Elsevier Science Publishers B.V., Amsterdam

Note

Synthesis of methyl 3-amino-3,6-dideoxy-α-L-hexopyranosides branched at C-3*

Francisco Santoyo Gonzalez†, Antonio Vargas Berenguel, and Pilar Garcia Mendoza
Departamento de Química Orgánica, Facultad de Ciencias, Universidad de Granada, 18071 Granada (Spain)

(Received March 21st, 1990; accepted for publication, June 22nd, 1990)

3-Amino-3-deoxy sugars are components of aminocyclitol[1], macrolide[2], and anthracycline antibiotics[3]. The therapeutic value of these drugs has stimulated the development of diverse strategies[3b,c] for the synthesis of the component sugars.

One route to branched-chain aminodeoxyhexoses is based on the cyclisation of sugar dialdehydes with C_2 nitro-compounds[4] as exemplified by the synthesis[5] of L-vancosamine. Recently, Gómez Sánchez *et al.*[6] reported that the reaction of methyl nitroacetate, using sodium ethoxide as catalyst, with the dialdehyde **1**, obtained by periodate oxidation of methyl α-L-rhamnopyranoside, gave a crystalline product **2** (13.5%) and a mixture (6.5%) of two isomers of **2**.

We now report the synthesis of branched-chain 3-amino-3,6-dideoxy sugars by cyclisation of **1** with (*a*) methyl nitroacetate, using potassium fluoride[7] as catalyst, and reduction of the major product obtained; and (*b*) cyanoacetamide, using piperidine or sodium ethoxide as catalyst, and Hofmann rearrangement of the product.

The first reaction gave, as the major product, crystalline **2** (31%). The diacetates **3** and **4** were isolated after acetylation of the material in the mother liquors. Compounds **2**–**4** had n.m.r. data identical to those reported[6], and **2** and **5** showed also the same m.p. and optical rotation as those reported[6]. Catalytic (Pt) hydrogenation of **2** in the presence of a stoichiometric amount of hydrochloric acid afforded 98% of the amine **6**, *N,O*-acetylation of which gave **7** (90%).

The second reaction followed by acetylation of the products gave **8** (40%) and **9** (3%). When piperidine alone was used as catalyst, **8** (41%) and traces of **9** were isolated. Following the method[8] for the synthesis of 3-*tert*-butoxycarbonylamino-3-cyano-3-deoxy sugars, treatment of **8** with an excess of lead tetra-acetate and *tert*-butyl alcohol gave **10** (98%), Zemplén deacetylation of which gave the diol **11** (92%). Reduction[8,9] of the nitrile group in **10** with $CoCl_2$–$NaBH_4$ followed by acetylation afforded **12** (62%).

The structures of **2**–**12** were established on the basis of elemental analysis and spectroscopic data (Tables I and II). The configurations at C-2 and C-4 and the

* Presented at EUROCARB V, the Vth European Carbohydrate Symposium, Prague, Czechoslovakia, August 21–25, 1989.

† Author for correspondence.

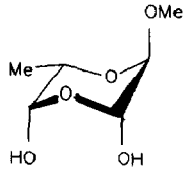
0008-6215/91/$ 03.50

preferred $^1C_4$ (L) conformation were deduced from the ^{1}H-n.m.r. data, namely, the $J_{1,2}$ values of 3.2–4.7 Hz for **2**, **3**, and **5**–**12** (H-1*eq*,2*ax*) and 1.8 Hz for **4** (H-1*eq*,2*eq*), and the $J_{4,5}$ values of 10.0–9.5 Hz for **3**, **8**, **10**, and **12** (H-4*ax*,5*ax*) and < 1.8 Hz for **2**, **4**–**7**, and **9** (H-4*eq*,5*ax*). A long-range coupling ($J_{2,4} \sim 1.6$ Hz) for **4** supported the configuration assigned.

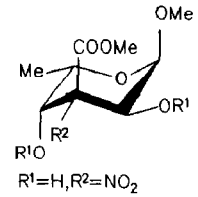
The configuration at C-3 in **6** and **7** is the same as that reported[6] for **2**, that in **3** and **4** remains uncertain, and that in **8** was deduced from the $J_{CN,H\text{-}2} + J_{CN,H\text{-}4}$ value of 18.0 Hz in agreement[10] with an axial orientation for the cyano group and H-2*ax*,4*ax*.

Hofmann[11,12] and Curtius[13] reactions proceed with retention of configuration, so that **10**–**12** should have the configuration at C-3 as depicted, namely RNH equatorial and CN or CH_2NHAc axial.

The above reactions are convenient and short routes to branched 3-amino-3-deoxy sugar derivatives.



1

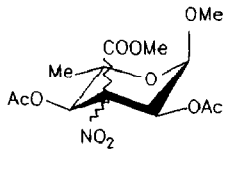


2 R^1=H,R^2=NO_2

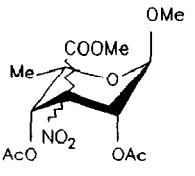
5 R^1=Ac,R^2=NO_2

6 R^1=H,R^2=NH_3Cl

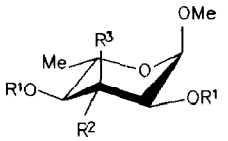
7 R^1=Ac,R^2=NHAc



3



4

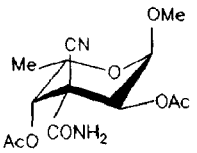


8 R^1=Ac,R^2=$CONH_2$,R^3=CN

10 R^1=Ac,R^2=NHBoc,R^3=CN

11 R^1=H,R^2=NHBoc,R^3=CN

12 R^1=Ac,R^2=NHBoc,R^3=CH_2NHAc



9

EXPERIMENTAL

Melting points were recorded with an Electrothermal apparatus and are uncorrected. Spectral measurements were recorded on Perkin–Elmer 983G (i.r.), Perkin–Elmer 141 ($[\alpha]_D$), and Bruker AM 300, WP 200, and WP 80 instruments (n.m.r.). C.i. (ether)-mass spectra were obtained with a Hewlett–Packard 5988A instrument. Optical rotations were measured at room temperature. Column chromatography was performed on Silica Gel Merck (70–230 mesh, ASTM).

Reaction of **1** *with methyl nitroacetate.* — To a solution of **1** (ref. 7a) (1.0 g, 6.1 mmol) in acetonitrile (30 mL) were added methyl nitroacetate (1 equiv.), potassium

TABLE I

^{1}H-N.m.r. data (δ in p.p.m., J in Hz) for **2**–**12**

Compound	*Chemical shift*						
	H-1	*H-2*	*H-4*	*H-5*	*Me-5*	*MeO*	*Others*
2[a,b]	4.61d	4.52dd	4.10d	4.24q	1.12d	3.20s	5.97 (d, 1 H, *J* 7.7 Hz, HO-4)[c], 5.12 (d, 1 H, *J* 8.5 Hz, HO-2)[c], and 3.68 (s, 3 H, COOMe).
3[d,e]	4.86d	5.70d	5.15d	4.50m	1.20d	3.29s	3.82 (s, 3 H, COOMe), and 2.10 (s, 6 H, 2 Ac).
4[a,e]	4.71d	5.84[f]	6.03bs	4.24dq	1.18d	3.27s	3.76 (s, 3 H, COOMe), and 2.10–2.00 (2 s, 6 H, 2 Ac).
5[e,g]	4.82d	5.80d	5.85bs	4.50q	1.08d	3.25s	3.80 (s, 3 H, COOMe), and 2.05, 1.98 (2 s, 6 H, 2 Ac).
6[a,b]	4.55d	3.88dd	3.83d	4.32q	1.11d	3.22s	8.45 (bs, 3 H, NH_3)[c], 6.10 (d, 1 H, *J* 6.5 Hz, HO-4)[c], 5.27 (d, 1 H, *J* 8.1 Hz, HO-2)[c], and 3.68 (s, 3 H, COOMe).
7[e,g]	4.70d	5.25d	5.82d	4.75q	1.16d	3.40s	6.65 (bs, 1 H, NH)[c], 3.75 (s, 3 H, COOMe), and 2.25, 2.23, 1.85 (3 s, 9 H, 3 Ac).
8[a,b]	4.87d	5.18d	4.99d	3.94dq	1.11d	3.32s	7.84, 7.82 (2 s, 2 H, NH_2)[c], and 2.10, 2.07 (2 s, 6 H, 2 Ac).
9[a,d]	5.00d	5.49d	5.36s	4.43q	1.15d	3.42s	6.45, 5.90 (2 bs, 2 H, NH_2), and 2.11, 2.09 (2 s, 6 H, 2 Ac).
10[a,e]	4.83d	5.62d	5.38d	4.09dq	1.20d	3.40s	5.01 (s, 1 H, NH)[c], 2.16, 2.14 (2 s, 6 H, 2 Ac), and 1.40 (s, 9 H,Me_3C).
11[a,b]	4.55d	4.00[f]	3.58dd	3.66m	1.15d	3.27s	7.44 (s, 1 H, NH)[c], 5.98 (d, 1 H, *J* 4.7 Hz, HO-4)[c], 5.61 (d, 1 H, *J* 6.1 Hz, HO-2)[c], and 1.40 (s, 9 H, Me_3C).
12[a,e]	4.77d	5.95bs	5.63bd	3.83m	1.10d	3.32s	6.20 (dd, 1 H, *J* 7.9 and 5.0 Hz, CH_2NH), 5.47 (bs, 1 H, NH-3), 3.94 (m, 1 H, CH_2NH), 3.70 (dd, 1 H, *J* 14.8 and 5.0 Hz, CH_2NH), 2.04, 1.99, 1.98 (3 s, 9 H, 3 Ac), and 1.32 (bs, 9 H, Me_3C).

Table continued overleaf

TABLE I *(Continued)*

Compound	*Chemical shift*		
	$J_{1,2}$	$J_{4,5}$	$J_{5,6}$
2	3.4	0	6.5
3	3.7	9.8	6.5
4[h]	1.8	1.6	6.5
5	3.5	0	6.8
6	3.2	0	6.5
7	4.7	1.8	6.5
8	3.8	9.9	6.3
9	3.7	0	6.5
10	4.0	10.0	6.3
11	4.0	9.7	6.0
12	3.7	9.5	6.1

[a] 300 MHz. [b] For solution in $(CD_3)_2SO$. [c] Exchangeable with D_2O. [d] 200 MHz. [e] For solution in $CDCl_3$ (internal Me_4Si). [f] Pseudo-t. [g] 80 MHz. [h] $J_{2,4}$ 1.6 Hz.

TABLE II

^{13}C-N.m.r. data for **2**, **3**, and **5–12**

Compound	*C-1*	*C-2*	*C-4*	*C-3*	*C-5*	*C-6*	*MeO*	*Others*
2[a,b]	98.9	65.8	71.7	94.7	64.9	15.9	54.8	164.68(CO), 53.0 ($COOCH_3$)
3[c,d]	96.0	—73.3,70.4—		91.9	64.9	17.3	55.4	169.4, 169.2 (2 CO), 162.2 (*C*OOMe), 53.3 ($COOCH_3$), and 20.5 (2 CH_3CO)
5[a,d]	96.6	67.7	72.5	90.4	64.9	16.1	55.6	169.8, 169.0 (2 CO), 163.5 (*C*OOMe), 54.0 ($COOCH_3$), and 20.8, 20.3 ($2CH_3CO$)
6[a,d]	98.9	65.2	68.9	62.7	65.2	16.5	55.5	167.8 (CO), and 54.0 ($COOCH_3$)
7[d,e]	97.4	69.6	71.3	60.9	64.5	16.3	55.2	173.5, 169.3, 167.9 (4 CO), 52.6 ($COOCH_3$), and 22.7, 21.2, 20.5 (3 CH_3CO)
8[a,b]	94.5	69.3	71.2	52.0	62.9	16.5	54.9	168.8, 168.6 (2 CO), 164.1 ($CONH_2$), 115.7 (CN), and 20.2, 20.1 (2 CH_3CO)
9[a,e]	95.8	66.4	78.8	48.2	62.4	16.0	55.8	170.1, 169.6 (2 CO), 163.7 ($CONH_2$), 117.3 (CN), and 20.7, 20.5 (2 CH_3CO)
10[a,d]	96.2	69.0	71.6	58.5	63.8	16.9	55.5	169.9, 169.6 (2 CO), 152.6 (CONH), 114.9 (CN), 81.7 (CMe_3), 28.1 (CMe_3), and 20.8, 20.7 (2 CH_3CO)
11[a,b]	98.2	69.2	72.7	61.3	65.0	17.3	54.7	154.7 (CO), 116.1 (CN), 79.2 (CMe_3), and 28.0 [$C(CH_3)_3$]
12[a,d]	97.5	69.9	72.4	59.7	64.4	17.7	55.5	175.4, 171.8, 169.0 (3 CO), 154.0 (CON), 79.6 (CMe_3), 41.4 (CH_2N), 28.1 [$C(CH_3)_3$], and 23.2, 20.7 (3 CH_3CO)

[a] 75 MHz. [b] For a solution in $(CD_3)_2SO$. [c] 50 MHz. [d] For a solution in $CDCl_3$ (internal Me_4Si). [e] 20 MHz.

fluoride (0.1 equiv.), and dibenzo-18-crown-6 ether (0.1 equiv.). The reaction mixture was stirred at 45° for 4.5 h and then concentrated. Water (20 mL) was added to the residue, the mixture was extracted with ethyl acetate (3 × 100 mL), and the combined extracts were dried, filtered, and concentrated. Crystallisation of the crude product from ethyl acetate or ether gave methyl 3,6-dideoxy-3-*C*-methoxycarbonyl-3-nitro-α-L-*galacto*-hexopyranoside (**2**; 0.50 g, 31%), m.p. 168–170°, $[\alpha]_D$ −218° (*c* 1, methanol) {lit[6] m.p. 168–170°, $[\alpha]_D$ −222° (chloroform)}; ν_{max}^{KBr} 3484, 3384, 1742, 1557, and 1338 cm^{-1}. For the ^1H- and ^{13}C-n.m.r., see Tables I and II (Found: C, 40.93; H, 5.42; N, 5.13. $C_9H_{15}NO_8$ calc: C, 40.76; H, 5.70; N, 5.28%).

Conventional treatment of the mother liquors with acetic anhydride–acetic acid–acetyl chloride (2:2:5 mL) and column chromatography of the crude product gave, first, a mixture (0.30 g), not studied further, then a mixture (0.52 g, 25%) of methyl 2,4-di-*O*-acetyl-3,6-dideoxy-3-*C*-methoxycarbonyl-3-nitro-α-L-*gluco*(or *allo*)-hexopyranoside (**3**) and methyl 2,4-di-*O*-acetyl-3,6-dideoxy-3-*C*-methoxycarbonyl-3-nitro-α-L-*talo*(or *ido*)hexopyranoside (**4**). Crystallisation from ether–hexane gave **3**, m.p. 160–161°, $[\alpha]_D$ −124° (*c* 1, chloroform); ν_{max}^{KBr} 1759, 1559, and 1348 cm^{-1}. For the ^1H- and ^{13}C-n.m.r., see Tables I and II (Found: C, 44.58; H, 5.24; N, 3.92. $C_{13}H_{19}NO_{10}$ calc.: C, 44.70; H, 5.48; N, 4.01%). The mother liquors contained **3** and **4** in the ratio 1:3.

*Methyl 2,4-di-*O*-acetyl-3,6-dideoxy-3-*C*-methoxycarbonyl-3-nitro-α-*L*-galacto-hexopyranoside* (**5**). — Conventional treatment of **2** (0.22 g, 0.83 mmol) with acetic anhydride–acetic acid–acetyl chloride (2:2:4 mL), with column chromatography (5:1 ether–hexane) of the crude product, gave **5** (0.28 g, 96%), m.p. 192–194°, $[\alpha]_D$ −166° (*c* 1, chloroform) {lit.[6] m.p. 187–190°, $[\alpha]_D$ −155°} ν_{max}^{KBr} 1758, 1560, 1375, and 1347 cm^{-1}. For the ^1H- and ^{13}C-n.m.r., see Tables I and II (Found: C, 44.53; H, 5.57; N, 4.24. $C_{13}H_{19}NO_{10}$ calc.: C, 44.70; H, 5.48; N, 4.01%).

*Methyl 3-amino-3,6-dideoxy-3-*C*-methoxycarbonyl-α-L-galactopyranoside hydrochloride* (**6**). — A suspension of PtO_2 in water (25 mL) containing M HCl (2.6 mL) was prehydrogenated, then **2** (0.7 g) was added, and the hydrogenation was continued for 60 h. The mixture was filtered, insoluble material was washed with water, and the combined filtrate and washings were concentrated under diminished pressure to give **6** (0.7 g, 98%), m.p. 113–114°, $[\alpha]_D$ −106° (*c* 1, water); ν_{max}^{KBr} 3500–3250, 1741, 1596, 1490, and 1051 cm^{-1}. For the ^1H- and ^{13}C-n.m.r., see Tables I and II. C.i.-mass spectrum: *m/z* 235 (45) (M^+ − HCl), 204 (100) (M^+ − HCl − OCH_3). An elemental analysis could not be obtained because the compound is highly hygroscopic.

Conventional treatment of **6** (0.1 g) with acetic anhydride–pyridine (2:2 mL) at room temperature for 6 h, with column chromatography (ethyl acetate) of the crude product, gave the 2,4-diacetate **7** (0.12 g, 90%), m.p. 150–151° (from ether), $[\alpha]_D$ −111° (*c* 1, chloroform); ν_{max}^{KBr} 3385, 3355, 1741, 1670, 1536, 1229, and 1056 cm^{-1}. For the ^1H- and ^{13}C-n.m.r., see Tables I and II (Found: C, 49.78; H, 6.30; N, 4.00. $C_{15}H_{23}NO_9$ calc.: C, 49.86; H, 6.41; N, 3.88%).

Reaction of **1** *with cyanoacetamide.* — (*a*) *Piperidine as catalyst.* A solution of **1** (ref. 7a) (0.5 g, 3.0 mmol), cyanoacetamide (0.49 g), and piperidine (1%) in 1,4-dioxane (9 mL) and water (3 mL) was stored at room temperature (16 h), then concentrated.

Conventional treatment of the residue with acetic anhydride–acetic acid–acetyl chloride (2:2:4 mL), with column chromatography (ether) of the crude product, gave methyl 2,4-di-*O*-acetyl-3-carbamoyl-3-*C*-cyano-3,6-dideoxy-α-L-*gluco*-hexopyranoside (**8**; 0.39 g, 41%), m.p. 217–218°, $[\alpha]_D$ −131° (*c* 1, chloroform); ν_{max}^{KBr} 3461, 3244, 1765, 1713, 1686, and 1600 cm^{-1}. For the ^1H- and ^{13}C-n.m.r., see Tables I and II (Found: C, 49.50; H, 5.92; N, 9.04. $C_{13}H_{18}N_2O_7$ calc.: C, 49.68; H, 5.77; N, 8.91%).

(b) Sodium ethoxide as catalyst. — The solution obtained by the reaction of sodium (0.14 g) in ethanol (10 mL) was added in portions during 3 min to a stirred solution of **1** (ref. 7a) (1.0 g, 6.1 mmol) and cyanoacetamide (0.51 g) in ethanol (30 mL) at 0°. The mixture was kept at 0° for 20 min, then neutralised with Amberlite IR-120 (H^+) resin, filtered, and concentrated. The crude product was treated conventionally with acetic anhydride–acetic acid–acetyl chloride (4:4:12 mL). Column chromatography (4:1 ether–hexane) of the crude product gave, first, methyl 2,4-di-*O*-acetyl-3-carbamyl-3-*C*-cyano-3,6-dideoxy-α-L-*galacto*-hexopyranoside (**9**; 0.06 g, 3%), m.p. 199–200°, $[\alpha]_D$ −140° (*c* 1, chloroform); ν_{max}^{KBr} 3432, 3207, 1755, 1715, 1684, and 1619 cm^{-1}. For the ^1H- and ^{13}C-n.m.r., see Tables I and II (Found: C, 49.42; H, 5.35; N, 9.07. $C_{13}H_{18}N_2O_7$ calc.: C, 49.68; H, 5.77; N, 8.91%).

Eluted second was **8** (0.76 g, 40%).

*Methyl 2,4-di-*O*-acetyl-3-*tert*-butoxycarbonylamino-3-*C*-cyano-3,6-dideoxy-α-L-glucopyranoside* (**10**). — Lead tetra-acetate (4.50 g) was added to a solution of **8** (0.66 g) in *tert*-butyl alcohol–*N*,*N*-dimethylformamide (10:5 mL). The mixture was stirred and boiled under reflux for 20 min, then cooled, toluene (100 mL) and ether (25 mL) were added, and the solution was filtered, washed with water (2 × 50 mL), dried ($MgSO_4$), and concentrated. Column chromatography (2:1 ether–hexane) of the crude product gave **10** (0.80 g, 98%), m.p. 166–167° (from ether–hexane), $[\alpha]_D$ −87.5° (*c* 1, chloroform); ν_{max}^{KBr} 3319, 1758, and 1720 cm^{-1}. For the ^1H- and ^{13}C-n.m.r., see Tables I and II (Found: C, 52.74; H, 6.70; N, 7.46. $C_{17}H_{26}N_2O_8$ calc.: C, 52.84; H, 6.78; N, 7.25%).

*Methyl 3-*tert*-butoxycarbonylamino-3-C-cyano-3,6-dideoxy-α-L-glucopyranoside* (**11**). — To a solution of **10** in dry methanol (20 mL) at ~ −15° was added freshly prepared methanolic NaOMe (10 mL, 10%). After storage for 20 min at room temperature, the solution was concentrated, and column chromatography (3:1 ether–hexane) of the residue gave **11** (0.15 g, 92%), m.p. 175–176° (from ether–hexane), $[\alpha]_D$ −101° (*c* 1, methanol); ν_{max}^{KBr} 3538, 3476, 3275, 2265, and 1738 cm^{-1}. For the ^1H- and ^{13}C-n.m.r., see Tables I and II (Found: C, 51.40; H, 7.52; N, 9.47. $C_{13}H_{22}N_2O_6$ calc.: C, 51.65; H, 7.33; N, 9.26%).

*Methyl 3-C-acetamidomethyl-2,4-di-O-acetyl-3-*tert*-butoxycarbonylamino-3,6-dideoxy-α-L-glucopyranoside* (**12**). — $CoCl_2$ (0.65 g) was added to a solution of **10** in methanol (45 mL). $NaBH_4$ (0.65 g) was then added in small portions during 15 min with stirring. The mixture was kept at room temperature for 60 min, aqueous 30% NH_4Cl solution (25 mL) was added, the solution was concentrated, the residue was extracted with ethyl acetate (3 × 50 mL), and the combined extracts were dried ($MgSO_4$) and concentrated. The crude product was treated conventionally with acetic anhydride–pyridine (3:2 mL). Column chromatography (ethyl acetate) of the product gave **12** (0.14

g, 62%), isolated as a syrup, $[\alpha]_D$ −65° (*c*1, chloroform); ν_{max}^{film} 3325, 1750, 1714, 1654, and 1368 cm^{-1}. For the ^1H- and ^{13}C-n.m.r., see Tables I and II. C.i.-mass spectrum: *m/z* 433 (32) ($M^+ + 1$), 377 (100) ($M^+ + 1 - C_4H_8$).

ACKNOWLEDGMENTS

This work was supported by the CAICYT (PB85-0390) and the Consejería de Educación y Ciencia of the Junta de Andalucía.

REFERENCES

1 S. Umezawa, *Adv. Carbohydr. Chem. Biochem.*, 30 (1974) 111–182; K. L. Rinehart and T. Suami (Eds.), *ACS Symp. Ser.*, 125 (1980).
2 S. Omura (Ed.), *Macrolide Antibiotics: Chemistry, Biology and Practice*, Academic Press, Orlando, Florida, 1984.
3 (*a*) F. Arcamone, in P. G. Sammes (Ed.), *Topics in Antibiotic Chemistry*, Vol. 2, Ellis Horwood, Chichester, 1978, pp. 99–239; (*b*) I. F. Pelivas, C. Monneret, and P. Herczegh, *Synthetic Aspects of Aminodeoxy Sugars of Antibiotics*, Springer-Verlag, Berlin–Heidelberg, 1988; (*c*) F. M. Hauser and S. Ellenberger, *Chem. Rev.*, 86 (1986) 35–67; (*d*) F. Arcamone, *Doxorubicin Anticancer Antibiotics*, Academic Press, New York, 1981; (*e*) D. Horton and W. Priebe, in H. S. El Khadem (Ed.), *Anthracycline Antibiotics*, Academic Press, New York, 1982, pp. 197–224.
4 Ref. 3*b*, pp. 68–69.
5 J. S. Brimacombe and L. W. Doner, *J. Chem. Soc, Perkin Trans. 1*, (1974) 62–65.
6 F. Cabrera Escribano, R. Fernandez Fernandez, A. Gomez Sanchez, I. Hermosin Gutierrez, A. Lopez Castro, and M. D. Estrada, *Carbohydr. Res.*, 199 (1990) 129–137.
7 (*a*) F. Santoyo Gonzalez and A. Vargas Berenguel, *Tetrahedron*, (*b*) F. Santoyo Gonzalez, A. Vargas Berenguel, and J. Molina Molina, *Carbohydr. Res.*, 209 (1991) 155–165.
8 F. Santoyo Gonzalez, A. Vargas Berenguel, F. Hernandez Mateo, and P. Garcia Mendoza, *Carbohydr. Res.*, 209 (1990) 131–143.
9 T. Satoh, S. Suzuki, Y. Suzuki, Y. Miyaji, and Z. Imai, *Tetrahydron Lett.*, (1969) 4555–4558; B. Ganem and J. O. Osby, *Chem. Rev.*, 86 (1986) 763–780.
10 J. L. Marshall, *Methods Stereochem. Anal.*, 2 (1983) 11–33.
11 H. E. Baumgarten and A. Staklis, *J. Am. Chem. Soc.*, 87 (1965) 1141–1142.
12 E. S. Wallis and J. F. Lane, *Org. React.*, 3 (1946) 267–306.
13 P. A. S. Smith, *Org. React.*, 3 (1946) 337–449.