Carbohydrate Research, 72 (1979) 289–296

Note

Formation of phosphonioepimino salts of sugars: a novel neighboring-group participation

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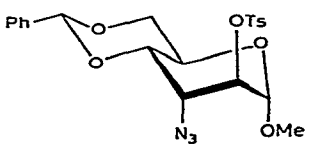
(Received July 10th, 1978; accepted for publication, July 20th, 1978)

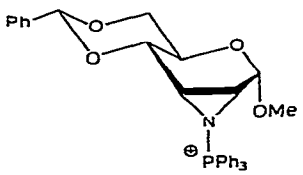
We have described[1,2] the synthesis of sugar phosphinimines which are precursors of carbodi-imide, heterocyclic, and urea derivatives of carbohydrates. In studying the formation of phosphinimino-pyranosides, we now report a novel neighboring-group participation for axially substituted derivatives.

Treatment of methyl 3-azido-4,6-*O*-benzylidene-3-deoxy-2-*O*-toluene-*p*-sulphonyl-α-D-altroside[3] (**1**) with triphenylphosphine afforded methyl 4,6-*O*-benzylidene-2,3-dideoxy-2,3-(*N*-triphenylphosphonioepimino)-α-D-alloside toluene-*p*-sulphonate (**2**) instead of the expected phosphinimine. The ^{1}H-n.m.r. spectrum of **2** showed a singlet for the methyl group of the toluene-*p*-sulphonate anion at δ 2.30, a doublet for H-1 at 5.13 ($J_{1,2}$ 4.0 Hz), and octets for H-2 and H-3 at 3.56 and 3.22, respectively. The doublets of doublets for H-2 and H-3 are further split due to coupling with phosphorus ($^3J_{P,C-H}$ 14.0 Hz). The relatively small chemical shifts for H-2 and H-3 are consistent with the aziridine ring structure. The values $J_{2,3}$ = 6.3 and $J_{3,4}$ = 2.0 Hz indicate a skew-boat conformation of the pyranoid ring. The signal at δ 2.78 indicates that the salt contains one molecule of water. It was shown that without water the product cannot be isolated in the crystalline state.

The X-ray crystal structure[6] of **2** revealed that C-1–C-4 of the pyranoid ring

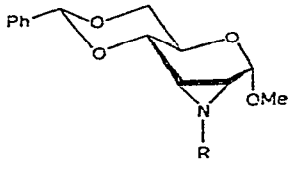


1



2

$TsO^{\ominus} \cdot H_2O$

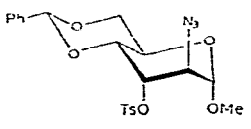


3 R = H
4 R = Ac

are nearly in the same plane (the proper torsion angle is 1.1°) and that the plane of the aziridine ring is almost perpendicular to this plane. The triphenylphosphonio group is *exo* with respect to the pyranoid system. The water molecule is hydrogen-bonded to the pyranose oxygen and to one of the oxygen atoms of the toluene-*p*-sulphonate anion.

Treatment of **2** with aqueous potassium hydroxide afforded the known methyl 4,6-*O*-benzylidene-2,3-dideoxy-2,3-epimino-α-D-alloside[3-5] (**3**), which was acetylated to give the *N*-acetyl derivative[3-5] **4**.

The reaction of methyl 2-azido-4,6-*O*-benzylidene-2-deoxy-3-*O*-toluene-*p*-sulphonyl-α-D-altroside[3] (**5**), which has a structure very similar to that of **1**, with triphenylphosphine afforded not only the expected phosphonioepimino salt **6** (56%) but also another salt (**9**;9%), triphenylphosphine oxide (8%), and the known methyl 4,6-*O*-benzylidene-2,3-dideoxy-2,3-epimino-α-D-mannoside[3,4] (**7**) which was isolated as its *N*-acetyl derivative[3,4] **8** (9%).



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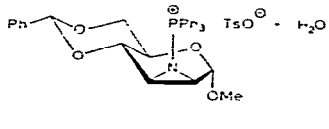
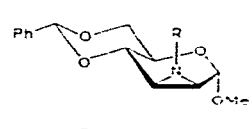
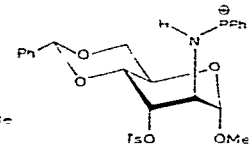


6



7 R = H
8 R = Ac



9

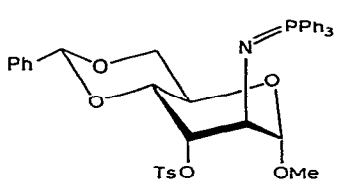
The structure of **6** was similar to that of **2**. The ^{1}H-n.m.r. spectrum contains a singlet at δ 2.30 for the methyl group of the tosylate anion, a singlet for H-1 at 5.16 ($J_{1,2}$ ~0 Hz), and multiplets for H-2 and H-3 at 2.86 ($J_{2,3}$ 6.0 Hz) and 2.70 ($J_{3,4}$ ~0.5 Hz), respectively. The orientation of the aziridine ring in **6** is opposite to that in **2**, *i.e.*, "upward"-related to the plane of the pyranoid ring, as indicated by $J_{2,3}$ and $J_{3,4}$. The presence of one molecule of water in **6** was shown by the signal at δ 2.75.

Thus, **6** is the monohydrate of methyl 4,6-*O*-benzylidene-2,3-dideoxy-2,3-(*N*-triphenylphosphonioepimino)-α-D-mannoside toluene-*p*-sulphonate. As expected, treatment of **6** with potassium hydroxide gave **7**.

The product **9** was methyl 4,6-*O*-benzylidene-2-deoxy-3-*O*-toluene-*p*-sulphonyl-2-(triphenylphosphonioamino)-α-D-altroside toluene-*p*-sulphonate. The i.r. spectrum contained bands at 3100–2700 (NH^+), 1365 (sulphonic ester), and 1180 cm^{-1} (sulphonate anion). The ^{1}H-n.m.r. spectrum contained two singlets for tosyl-methyl at δ 2.26 and 2.32, and a triplet for N-H at 8.41 ($J_{NH,H\text{-}2}$ 11.0 Hz). This J value indicates that N-H and H-2 are antiperiplanar in the dominant rotamer, because of steric hindrance caused by the bulky triphenylphosphonio group. Other signals were δ 4.39 (s, $J_{1,2}$ ~0 Hz, H-1), 3.77 (m, $J_{2,3}$ 3 Hz, H-2), and 4.75 (dd, $J_{3,4}$ 3 Hz, H-3). Both the triplet splitting of NH and the multiplet of H-2 are due to the coupling with phosphorus ($^2J_{P,NH}$ 10.5, $^2J_{P,CH}$ 14.0 Hz). The small value of $J_{1,2}$ points to a distortion of the pyranoid ring caused by the bulky axial substituent on C-2, as in other cases[7,8].

The isolation of **9** indicates that the first step of the reaction is the formation

of the phosphinimine **10**, which cannot be isolated because intramolecular displacement of sulphonate gives the phosphonioepimino derivative **6**. During the reaction, **9** is formed from **10** and the toluene-*p*-sulphonate anion of **6**, with the participation of traces of water in the medium. The cation of **6** then reacts with water, giving an unstable hydroxyphosphorane intermediate (**11**) that decomposes spontaneously to yield **7** and triphenylphosphine oxide.



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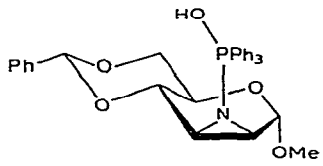
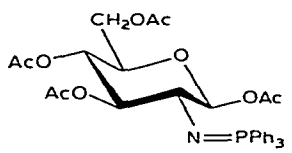


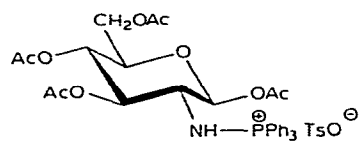
11

When the reactions were carried out in dry dichloroethane, only the phosphonioepimino salt **6** (96%) was obtained; the water required for the isolation of the crystalline hydrate was added after the reaction was complete. When wet dichloromethane was used, only **9** (44%), **7** or its *N*-acetyl derivative **8** (30%), and triphenylphosphine oxide (36%) could be isolated, as **6** was totally decomposed.

That decomposition is due to the simultaneous presence of water and the phosphinimino group, which is a strong proton-acceptor, was proved by treatment of the monohydrate **6** with 1,3,4,6-tetra-*O*-acetyl-2-deoxy-2-(triphenylphosphoranylideneamino)-β-D-glucopyranose[2] (**12**), which afforded **7** [or, after acetylation, **8** (67%)], the toluene-*p*-sulphonate (**13**, 65%) of **12**, and triphenylphosphine oxide (46%).



12



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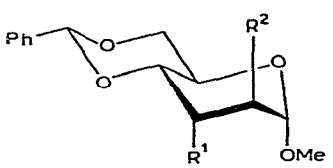

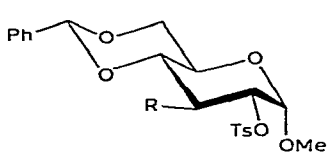

14 R^1 = N_3, R^2 = AcO
15 R^1 = AcO, R^2 = N_3
16 R^1 = N=PPh$_3$, R^2 = AcO
17 R^1 = AcO, R^2 = N=PPh$_3$
18 R^1 = N(Me)$\overset{\oplus}{P}$Ph$_3$I$^{\ominus}$, R^2 = AcO
19 R^1 = AcO, R^2 = N(Me)$\overset{\oplus}{P}$Ph$_3$I$^{\ominus}$

20 R = N_3
21 R = N=PPh$_3$
22 R = N(Me)$\overset{\oplus}{P}$Ph$_3$I$^{\ominus}$

TABLE I

^{1}H-N.M.R. DATA FOR COMPOUNDS **14–19**

Compounds	*Chemical shifts[a] (first-order couplings in Hz, in parentheses)*										
	H-1 ($J_{1,2}$)	*H-2* ($J_{2,3}$)	*H-3* ($J_{3,4}$)	*H-4* ($J_{4,5}$)	*H-5*	*H-6a*	*H-6e*	*H-8*	*AcO*	*OMe*	*Aryl*
14	4.64d (~1)	5.03dd (2.0)		←3.87–4.50m→				5.66s	2.23s	3.51s	7.25–7.55m
15	4.70d (0.6)	4.06dd (2.8)	5.20t (2.8)	←3.65–4.05m→		←4.2–4.5m→		5.61s	2.18s	3.46s	~7.37m
16	4.64d (<1)	4.79dd (2.5)		←3.5–5.1m→				5.66s	2.08s	3.60s	7.2–7.8m
17[b]	4.32d (~0.4)	3.71ddd (2.9)	5.16t (2.9)	4.75dd (8.8)	←3.9–4.5m→			5.73s	2.10s	3.27s	7.2–7.8m
18[c]	4.67d (<1)	4.91dd (1.5)		←3.6–4.6m→				5.85s	2.01s	3.67s	7.3–7.9m
19[d]	5.63d (4.8)	3.40m (3.5)	5.60t (4.5)	4.65dd (8.0)	←4.0–4.4m→			5.87s	1.96s	3.48s	7.3–7.9m

[a] δ scale. Assignments verified by spin decoupling. [b] $J_{P,H-2}$ = 25.3 Hz. [c] NMe δ 3.40d ($^3J_{P,CH_3}$ = 10 Hz). [d] NMe δ 3.44d ($^3J_{P,CH_3}$ = 10 Hz).

The structural requirement for the neighbouring-group participations **1→2** and **5→6** is the presence of *trans*-diaxial tosyloxy and phosphinimino groups. From the corresponding *O*-acetyl derivatives (**14** and **15**), the phosphinimine derivatives **16** and **17** were obtained, and no cyclization to phosphonioepimino salts was observed. The phosphinimine character of these products was indicated by their reaction with methyl iodide to give *N*-methylaminophosphonium salts (**18** and **19**).

In the glucose derivative **20**, where the azide and tosyloxy groups are *trans*-diequatorial, the reaction with triphenylphosphine also affords a phosphinimine **21** that can be converted into the corresponding *N*-methylaminophosphonium iodide **22**.

The structure of these derivatives was supported by their ^{1}H-n.m.r. spectra (see Table I).

Demonstration that the phosphinimino group can participate in neighbouring-group reactions provides a new possibility for the synthesis of epiminopyranose derivatives.

EXPERIMENTAL

General. — Optical rotations were measured with an Opton Polarimeter. I.r. spectra were recorded with Perkin–Elmer 457 and Unicam SP 200 spectrometers. ^{1}H-N.m.r. spectra were recorded with a JEOL PS-100 instrument for solutions in chloroform-*d* with tetramethylsilane as internal standard. The crystal structure was determined by direct methods and refined by least-squares techniques to an *R* of 0.09. X-Ray data were collected on a Philips PW 1100 four-circle automatic diffractometer by using graphite monochromated MoK_α radiation.

Methyl 4,6-O-benzylidene-2,3-dideoxy-2,3-(N-triphenylphosphonioepimino)-α-D-

alloside toluene-p-*sulphonate monohydrate* (**2**). — Methyl 3-azido-4,6-*O*-benzylidene-3-deoxy-2-*O*-toluene-*p*-sulphonyl-α-D-altroside (0.46 g, 1 mmol) dissolved in freshly distilled dichloromethane (20 ml) was mixed with triphenylphosphine (0.27 g, 1.03 mmol), and the solution (protected from moisture) was allowed to stand for 2 days at room temperature. The addition of dry ether (120 ml) then gave a white precipitate (0.63 g, 88%), m.p. 210°. Recrystallization from acetonitrile (6 ml) gave **2** (0.45 g, 63%), m.p. 210–211° (dec.), $[\alpha]_D$ +7.6° (*c* 5, dichloromethane).

Anal. Calc. for $C_{39}H_{40}NO_8PS$: N, 1.96; P, 4.34. Found: N, 1.97; P, 4.47.

Methyl 2,3-(N-*acetylepimino*)-*4,6-*O-*benzylidene-2,3-dideoxy-*α-D-*alloside* (**4**).— A solution of **2** (1.04 g, 1.46 mmol) in 1.5M aqueous potassium hydroxide (10 ml) was boiled under reflux for 20 min and then cooled, and the precipitated crystals were filtered off. T.l.c. showed the product to be a mixture of methyl 4,6-*O*-benzylidene-2,3-dideoxy-2,3-epimino-α-D-alloside (**3**) and triphenylphosphine oxide. Conventional treatment of the mixture with acetic anhydride (2 ml) in dry pyridine (3.5 ml) gave **4** (0.28 g, 63%), m.p. 188–189° (from ethanol), $[\alpha]_D$ +148° (*c* 1, chloroform); lit.[4,5] m.p. 187–188°, $[\alpha]_D$ +149° (*c* 0.86, chloroform).

From the ethanolic mother liquor, triphenylphosphine oxide (0.27 g, 67%) was obtained; m.p. and mixture m.p. 153–155° (from benzene–light petroleum).

*Reaction of methyl 2-azido-4,6-*O-*benzylidene-2-deoxy-3-*O-*toluene*-p-*sulphonyl*-α-D-*altroside* (**5**) *with triphenylphosphine.* — (*a*) *In purified dichloromethane.* A mixture of **5** (2.31 g, 5 mmol) and triphenylphosphine (1.37 g, 5.22 mmol) in dried and distilled dichloromethane (30 ml) was stored (with protection from moisture) for 2 days. The addition of dry ether (50 ml) then gave crystals (2.67 g, 75%) which, after recrystallization from dichloromethane–ether, afforded **6** (2.0 g, 56%), m.p. 161–164°, $[\alpha]_D$ +25° (*c* 2, dichloromethane).

Anal. Calc. for $C_{39}H_{40}NO_8PS$: N, 1.96; P, 4.34. Found: N, 1.78; P, 3.99.

Saturation of the filtrate with ether gave a salt-like material (0.45 g, 10%) which was crystallized from ethanol to give pure methyl 4,6-*O*-benzylidene-2-deoxy-3-*O*-toluene-*p*-sulphonyl-2-(triphenylphosphonioamino)-α-D-altroside toluene-*p*-sulphonate (**9**) (0.39 g, 9%) as colorless prisms, m.p. 191–192°, $[\alpha]_D$ +46° (*c* 1, dichloromethane).

Anal. Calc. for $C_{46}H_{46}NO_{10}PS_2$: C, 63.65; H, 5.34; N, 1.61; P, 3.57; S, 7.39. Found: C, 63.10; H, 5.16; N, 1.42; P, 3.66; S, 7.72.

After evaporation of the mother liquor, the residue (containing **7** and triphenylphosphine oxide according to t.l.c.) was treated with acetic anhydride and pyridine to give *N*-acetylated **7** (**8**; 0.13 g, 9%), m.p. 206–207° (from ethanol), identical (t.l.c.) with an authentic sample.

From the ethanolic filtrate, triphenylphosphine oxide (0.11 g, 8%) was isolated: m.p. 152–153° (from cyclohexane).

(*b*) When the reaction was carried out in commercial-grade solvent, no **6** was obtained. From the reaction mixture, **9** (0.38 g, 44%) was precipitated; m.p. 190–191°. Treatment of the mother liquor as in (*a*) afforded **8** (0.09 g, 30%), m.p. 205–206°, and triphenylphosphine oxide (0.10 g, 36%), m.p. 154°.

(*c*) *In dry 1,2-dichloroethane.* A solution of **5** (0.46 g, 1 mmol) and triphenylphosphine (0.27 g, 1.03 mmol) in dry 1,2-dichloroethane (4 ml) was protected from moisture and kept at room temperature overnight. Addition of ether (30 ml) precipitated thin needles of **6** which were repeatedly precipitated from dichloromethane–ether to afford the pure product (0.55 g, 77%), m.p. 160–162°, $[\alpha]_D$ +23° (*c* 5, dichloromethane).

*Methyl 2,3-(*N*-acetylepimino)-4,6-O-benzylidene-2,3-dideoxy-α-*D*-mannoside* (**8**). — (*a*) A solution of **6** (0.90 g, 1.26 mmol) in M aqueous potassium hydroxide (18 ml) was boiled under reflux for 1 h and then cooled and filtered, to give a mixture (0.65 g) of **7** and triphenylphosphine oxide. Conventional acetylation of this solid with acetic anhydride and dry pyridine gave **8** (0.22 g, 57%), m.p. 208–210° (from ethanol), $[\alpha]_D$ +48.5° (*c* 1, chloroform); lit.[3] m.p. 205–206°, $[\alpha]_D$ +49.3° (*c* 0.69, chloroform).

From the mother liquor, triphenylphosphine oxide (0.23 g, 66%) was obtained; m.p. 153–155° (from cyclohexane).

(*b*) A solution of **6** (0.56 g, 0.785 mmol) and **12** (0.52 g, 0.86 mmol) in dry dichloromethane (8 ml) was left to stand for 4 h, and then saturated with dry ether (80 ml) to give a white, salt-like product (**13**; 0.40 g, 66%), m.p. 108–111°, $[\alpha]_D$ +15° (*c* 2, chloroform). According to i.r. spectra and t.l.c. (alumina, chloroform), the product was identical with an authentic sample.

After evaporation of the mother liquor, the residue (containing **7** and triphenylphosphine oxide according to t.l.c.) was treated with acetic anhydride and pyridine to give **8** (0.16 g, 67%), m.p. 206–208° (from ethanol), $[\alpha]_D$ +47.9° (*c* 1, chloroform).

From the ethanolic mother liquor, triphenylphosphine oxide (0.10 g, 46%) was isolated; m.p. 155–157° (from cyclohexane).

*1,3,4,6-Tetra-O-acetyl-2-deoxy-2-(triphenylphosphonioamino)-β-*D*-glucopyranose toluene-*p*-sulphonate* (**13**). — A solution of **12** (0.61 g, 1.0 mmol) and toluene-*p*-sulphonic acid monohydrate (0.20 g, 1.05 mmol) in acetic anhydride (2.5 ml) was mixed with dry ether to precipitate a solid product (0.75 g), m.p. 109–112°. The salt was repeatedly precipitated with ether from solution in dichloromethane to give **13** (0.70 g, 90%), as a powder, m.p. 110–113°, $[\alpha]_D$ +15° (*c* 2, chloroform); ν_{max}^{KBr} 3000–2600 (NH^+), 1740 (AcO), and 1190 cm^{-1} (SO_3^-).

Anal. Calc. for $C_{39}H_{42}NO_{12}PS$: N, 1.80; P, 3.98. Found: N, 1.77; P, 3.85.

*Methyl 3-O-acetyl-2-azido-4,6-O-benzylidene-2-deoxy-α-*D*-altroside* (**15**). — Conventional acetylation of methyl 2-azido-4,6-*O*-benzylidene-2-deoxy-α-D-altroside[3] (3 g, 9.76 mmol) with acetic anhydride–pyridine gave syrupy **15** (3.06 g, 90%), $[\alpha]_D$ +37° (*c* 1, chloroform); $\nu_{max}^{CHCl_3}$ 2100 (N_3) and 1730 cm^{-1} (AcO).

Anal. Calc. for $C_{16}H_{19}N_3O_6$: C, 55.01; H, 5.48; N, 12.03. Found: C, 55.32; H, 5.25; N, 12.28.

*Methyl 3-O-acetyl-4,6-O-benzylidene-2-deoxy-2-(triphenylphosphoranylidene-amino)-α-*D*-altroside* (**17**). — To a solution of **15** (2.79 g, 8 mmol) in dry ether (30 ml) was added triphenylphosphine (2.10 g, 8 mmol). The mixture was kept (with pro-

tection from moisture) for 5 h and then evaporated to give **17** as a colorless glass (4.7 g, 99%), $[\alpha]_D$ +19° (*c* 1, dichloromethane).

Anal. Calc. for $C_{34}H_{34}NO_6P$: P, 5.31; Ac, 7.38. Found: P, 5.54; Ac, 7.42.

Methyl 3-O-acetyl-4,6-O-benzylidene-2-deoxy-N-methyl-2-(triphenylphosphonioamino)-α-D-altroside iodide (**19**). — A solution of **17** (0.30 g, 0.514 mmol) in methyl iodide (4 ml) was kept for 24 h and then evaporated. Repeated precipitation of the solid residue with ether from solution in dichloromethane afforded **19** as yellow powder (0.36 g, 96%), m.p. 140–145°, $[\alpha]_D$ +20.7° (*c* 1, chloroform).

Anal. Calc. for $C_{35}H_{37}INO_6P$: I, 17.49; P, 4.27. Found: I, 18.08; P, 4.19.

Methyl 2-O-acetyl-4,6-O-benzylidene-3-deoxy-3-(triphenylphosphoranylideneamino)-α-D-altroside (**16**). — A suspension of methyl 2-*O*-acetyl-3-azido-4,6-*O*-benzylidene-3-deoxy-α-D-altroside[3] (**14**; 1.05 g, 3 mmol) in dry ether (10 ml) was treated with triphenylphosphine (0.79 g, 3 mmol) overnight. Evaporation of the resulting solution gave **16** (1.75 g, 99%) as a colorless syrup, $[\alpha]_D$ +41° (*c* 1, dichloromethane). Completion of the reaction was indicated by the absence of the azide band at 2100 cm^{-1} in the i.r. spectrum.

Methyl 2-O-acetyl-4,6-O-benzylidene-3-deoxy-N-methyl-3-(triphenylphosphonioamino)-α-D-altroside iodide (**18**). — A solution of **16** (1.60 g, 2.74 mmol) in methyl iodide (5 ml) was stored for 24 h and then mixed with dry ether (25 ml) to give a solid that was repeatedly precipitated with ether from its solution in dichloromethane to yield **18** as a yellow powder (1.75 g, 88%), m.p. 117–120° (dec.), $[\alpha]_D$ +8° (*c* 3, chloroform).

Anal. Calc. for $C_{35}H_{37}INO_6P$: I, 17.49; P, 4.27. Found: I, 17.76; P, 3.89.

Methyl 3-azido-4,6-O-benzylidene-3-deoxy-2-O-toluene-p-sulphonyl-α-D-glucoside (**20**). — Methyl 3-azido-4,6-*O*-benzylidene-3-deoxy-α-D-glucoside[3] (1.00 g, 3.25 mmol) was added to a solution of toluene-*p*-sulphonyl chloride (2 g) in dry pyridine (5 ml). The mixture was stored for 4 days at room temperature, and then poured into ice–water to precipitate colorless crystals (1.40 g, 93%), m.p. 96–97°, which were recrystallized from ethanol to give **20** (1.12 g, 75%), m.p. 97–98°, $[\alpha]_D$ +35° (*c* 2.3, dichloromethane).

Anal. Calc. for $C_{21}H_{23}N_3O_7S$: C, 54.65; H, 5.02; N, 9.11; S, 6.95. Found: C, 54.77; H, 5.31; N, 9.02; S, 6.98.

Methyl 4,6-O-benzylidene-3-deoxy-N-methyl-2-O-toluene-p-sulphonyl-3-(triphenylphosphonioamino)-α-D-glucopyranoside iodide (**22**). — A solution of **20** (0.46 g, 1.0 mmol) in distilled dichloromethane (20 ml) and triphenylphosphine (0.27 g, 1.03 mmol) was stored overnight with protection from moisture. Evaporation of the solution gave **21** as a glass (0.71 g), $[\alpha]_D$ +32.5° (*c* 2, dichloromethane), which was sufficiently pure for the following step.

A solution of **21** (0.50 g, 0.72 mmol) in methyl iodide (2.5 ml) was kept for 24 h and then mixed with dry ether (25 ml) to precipitate crude **22** (0.50 g, 83%). Precipitation from dichloromethane solution with ether afforded **22** (0.39 g, 65%) as a powder, m.p. 132–136°, $[\alpha]_D$ +50.5° (*c* 2, chloroform).

Anal. Calc. for $C_{40}H_{41}INO_7PS$: I, 15.15; P, 3.70. Found: I, 15.40; P, 3.61.

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