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A NEW ROUTE FOR FORMATION OF THE CARBON–PHOSPHORUS BOND, AND SYNTHESIS OF 1,2,4-TRI-*O*-ACETYL-5-DEOXY-3-*O*-METHYL-5-*C*-[(*R*)- AND (*S*)-PHENYLPHOSPHINYL]-α- AND -β-D-XYLOPYRANOSE

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ABSTRACT

Deoxygenation at the carbon atom α to the phosphorus atom of 1,2-*O*-isopropylidene-5-*C*-(methoxyphenylphosphinyl)-3-*O*-methyl-α-D-xylofuranose (**7**), prepared from 1,2-*O*-isopropylidene-3-*O*-methyl-α-D-*xylo*-pentodialdo-1,4-furanose by reaction with methyl phenylphosphinate, was performed by successively treating **7** with 1,1′-(thiocarbonyl)diimidazole (TCDI) and tributyltin hydride. Treatment of the resulting 5-deoxy-1,2-*O*-isopropylidene-5-*C*-(methoxyphenylphosphinyl)-3-*O*-methyl-α-D-xylofuranose with sodium dihydrobis(2-methoxyethoxy)aluminate, followed by mineral acid, and then acetic anhydride in pyridine, gave the four title compounds, which were separated by column chromatography on silica gel and recrystallization, and characterized by ^{1}H-n.m.r.-spectral analysis and their optical rotations.

INTRODUCTION

Sugar analogs having phosphorus in the hemiacetal ring are interesting, not only from the point of view of their chemistry, but also from that of the possible utility of their biological activities. The synthesis of a few compounds (**1–5**) of the pentopyranose type[1–4], prepared from 5-deoxy-5-*C*-phosphinyl-D-pentofuranose precursors afforded by the Michaelis–Arbuzov reaction of 5-deoxy-5-halo derivatives with phosphorus compounds (M–A route), has already been reported. Also, aldoses and glyculoses were treated with dialkyl phosphonate or alkyl phenylphosphinate to give, readily, the corresponding sugar analogs containing the HO-C-P unit[5–10], but this method for the formation of a C–P bond had not yet been applied to the synthesis of sugar analogs having phosphorus in the hemiacetal ring, because deoxygenation of the hydroxyl group on the carbon atom α to the phosphorus atom could not be caused by the ways usually applicable to organic compounds[11].

Successful deoxygenation of a HO-C-P unit at the terminal carbon atom of sugars has now been achieved by successively treating such a compound with 1,1′-

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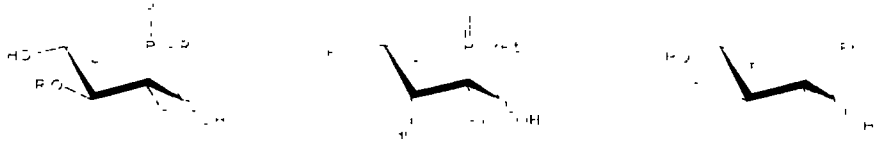
(thiocarbonyl)diimidazole (TCDI) and then tributyltin hydride[11–14], and by this route, the four title compounds were synthesized, each in good yield.

The excellent characteristics of this route for the formation of a C–P bond, compared with the M–A route, are as follows: the phosphorus compounds used as the substrates are readily obtained; the formation of the HO-C-P unit and the deoxygenation thereof are performed for only a short reaction time under mild conditions, and afford good yields; and processing of the reaction mixture is simple. Therefore, this convenient route will evidently be widely used in the future for the formation of a C–P bond in heat-unstable sugar derivatives such as amino sugars.

RESULTS AND DISCUSSION

1,2-*O*-Isopropylidene-3-*O*-methyl-α-D-*xylo*-pentodialdo-1,4-furanose (**6**), obtained by glycol-cleavage oxidation of 1,2-*O*-isopropylidene-3-*O*-methyl-α-D-glucofuranose with sodium metaperiodate[15], was used as the starting material for this synthesis.

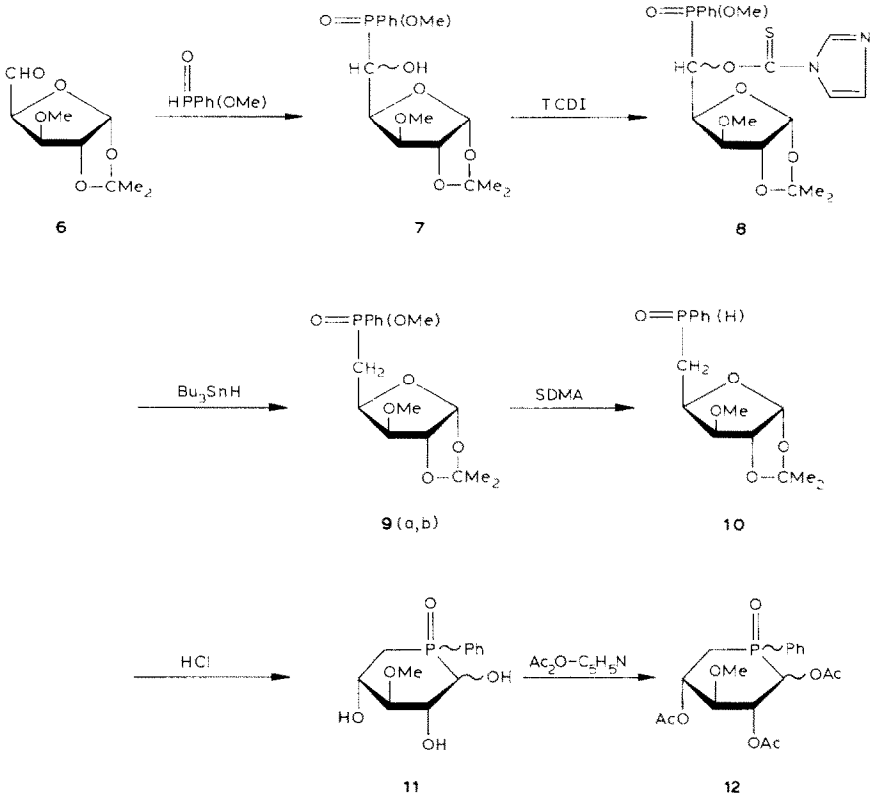
The dialdose derivative **6** was treated with methyl phenylphosphinate and trimethylamine in a refrigerator, to give 1,2-*O*-isopropylidene-5-*C*-(methoxyphenylphosphinyl)-3-*O*-methyl-α-D-xylofuranose (**7**) in quantitative yield [*m/z* 358 (M^+)]. Treatment of **7** with TCDI in 1,2-dichloroethane at 90° (bath) afforded syrupy 5-*O*-(imidazol-1-yl-thiocarbonyl)-1,2-*O*-isopropylidene-5-*C*-(methoxyphenylphosphinyl)-3-*O*-methyl-α-D-xylofuranose (**8**) in 89% yield. **8** showed the characteristic signals (3 H) of the imidazolyl group at δ 6.3–8.3 in the ^{1}H-n.m.r. spectrum, and *m/z* 468 (M^+). Reductive deoxygenation of **8** with tributyltin hydride in refluxing toluene afforded syrupy 5-deoxy-1,2-*O*-isopropylidene-5-*C*-(methoxyphenylphosphinyl)-3-*O*-methyl-α-D-xylofuranose (**9**) in 99% yield; this showed H-5,5′ signals at δ 2.05–2.6 in the ^{1}H n.m.r. spectrum, and *m/z* 342 (M^+). The crude **9** was separated by column chromatography on silica gel, using ethyl acetate–hexane as the eluant, into two fractions: **9a**, colorless needles [R_F 0.54 (EtOAc), 49%], m.p. 111.5–112°, and **9b**, colorless prisms [R_F 0.46 (EtOAc), 49%], m.p. 91.5–92.5°. The precise structure of **9a** or **9b** [probably, (*R*) and (*S*) isomers at the phosphorus atom] could not be determined by ^{1}H-n.m.r.-spectral analysis. On reduction of **9a** and **9b**, the same product was obtained. Reduction of **9** with SDMA in oxolane (tetrahydrofuran; THF) in the usual way[11–14] afforded



1 R = Me, R' = Ph, R'' = Bu
2 R = Et, R' = Ph, R'' = Bu
3 R = Me, R' = OMe, R'' = Bu

4

5



6 7 8

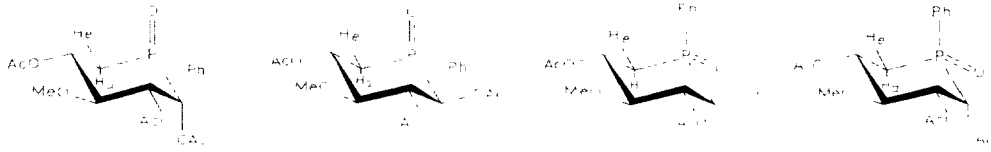
9(a,b) 10

11 12

5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-5-*C*-(phenylphosphinyl)-α-D-xylofuranose (**10**) in 92% yield; this showed an i.r. absorption band at 2340 cm^{-1} (P–H), and in the ^{1}H-n.m.r. spectrum, half a P–H signal, at δ 11.60 (disappearing on deuteration).

Hydrolysis of **10** with 0.1M hydrochloric acid under nitrogen for 3 h at 110° (bath), and acetylation of the product (**11**) with acetic anhydride–pyridine in the usual way[1–4], afforded a crude mixture (**12**) consisting of crystals in a syrup (91% from **10**). The crude mixture was separated by recrystallization from ethanol and ethanol–hexane, into two fractions, A and B. The syrup was separated by column chromatography on silica gel, using ethyl acetate as the eluant, into three major fractions, which will be referred to as B, C, and D (according to their decreasing R_F values).

Fractions A, B, C, and D respectively gave colorless needles, m.p. 295–296°; colorless needles, m.p. 200–200.5°; a colorless syrup; and a colorless syrup; each exhibited three acetoxyl groups in the ^{1}H-n.m.r. spectrum, and the molecular-ion peak at *m/z* 398, corresponding to $C_{18}H_{23}O_8P$, in the high-resolution mass spectrum of each, and this formula was supported by the elemental analysis of fractions A and B. The structural assignments of these compounds were determined by comparing the ^{1}H-n.m.r. spectra, and the optical rotations, with those of the structurally similar analogs **2, 3,** and **5**.



12a 12b 12c 12d

The ^{1}H-n.m.r. spectra of fractions C and D showed relatively high values of δ for the H-2 and H-4 signals (compared with those of fractions A and B), whereas the remaining signals were essentially similar for the four fractions. The upfield shifts of the H-2 and H-4 signals can be explained in terms of the shielding effect of the phenyl group linked axially to the ring-P atom. The H-1 signal of the β-acetate **12c** consisted of a triplet at δ 5.79, with $J_{1,2}$ 10.7 and $J_{1,P}$ 10.7 Hz, whereas that of the α anomer **12d** showed a double triplet at δ 5.99 with a large $J_{1,P}$ (8.0 Hz) and a small $J_{1,2}$ (2.5 Hz) value, and, probably, $J_{1,5}$ 2.5 Hz, due to the 1,5 W coupling. These splitting patterns of fractions C and D resembled those of 5,6-dideoxy-5-*C*-[(*R*)-phenylphosphinyl]-α- and -β-L-idohexopyranoses[10], and 5-deoxy-5-*C*-[(*S*)-methoxyphosphinyl]-α- and -β-D-xylopyranoses[4]. The optical rotation of fraction C was smaller than that of fraction D. Therefore, fractions C and D were respectively identified as 5-deoxy-5-*C*-[(*S*)-phenylphosphinyl]-β-D-xylopyranose (**12c**) and 5-deoxy-5-*C*-[(*S*)-phenylphosphinyl]-α-D-xylopyranose (**12d**), both in the $^4C_1$(D) conformation.

The shift patterns in the ^{1}H-n.m.r. spectra of fractions A and B were somewhat similar, and showed relatively low δ values for the H-2 and H-4 signals, compared with those for **12c** and **12d**. The optical rotation of fraction A was larger than that of fraction B. Therefore, fractions A and B were respectively considered to be 5-deoxy-5-*C*-[(*R*)-phenylphosphinyl]-α-D-xylopyranose (**12a**) and 5-deoxy-5-*C*-[(*R*)-phenylphosphinyl]-β-D-xylopyranose (**12b**), both in the $^4C_1$(D) conformation.

EXPERIMENTAL

General methods. — Melting points were measured with a micro melting-point apparatus (Yanagimoto Seisakusho, Japan) and are uncorrected. Column chromatography was performed by using Merck Lober silica gel. T.l.c. was conducted on layers of silica G-10 (Nakarai Chemicals, Ltd., Japan). All reactions were monitored by t.l.c., and the products were detected with sulfuric acid–ethanol, or cobalt(II) chloride–ethanol. Optical rotations were determined with an Atago-Polax polarimeter (Atago Ltd., Japan). I.r. spectra were recorded with an A-3 spectrophotometer (Japan Spectroscopic Co., Ltd.). ^{1}H-N.m.r. spectra were recorded, for solutions in $CDCl_3$, with a Hitachi–Perkin–Elmer R-20 (60 MHz) instrument. Chemical shifts are reported as δ values, relative to tetramethylsilane (δ 0.0) as the internal standard. Mass spectra were recorded with a Hitachi RMU7MG GS–MS spectrometer.

1,2-O-Isopropylidene-5-C-(methoxyphenylphosphinyl)-3-O-methyl-α-D-xylo-

furanose (**7**). — A mixture of compound **6** (2.21 g), methyl phenylphosphinate (2.5 mL), and triethylamine (3.0 mL) was kept in a refrigerator for 10 h (as, after ~1 h at room temperature, a partially decomposed mixture is obtained), diluted with chloroform and aqueous $NaHCO_3$ (to decompose the excess of phosphinate), and the chloroform layer washed with water, dried (Na_2SO_4), and evaporated *in vacuo*, to give **7** in quantitative yield as a colorless syrup; $[\alpha]_D^{20}$ −28.6° (*c* 1.70, $CHCl_3$); ν_{max}^{KBr} 3450 cm^{-1} (OH); ^{1}H-n.m.r. data: δ 1.36, 1.53 (2 s, 6 H, CMe_2), 3.06, 3.40 (2 s, 3 H, OMe-3, overlapping with H-5), 3.56, 3.60, 3.73, 3.79 (4 s, 3 H, P-OMe), 3.83 (d, 1 H, $J_{3,4}$ 2.0 Hz, H-3), 4.45 (d, 1 H, $J_{1,2}$ 3.2 Hz, overlapping with H-4, H-2), 5.35 (broad, 1 H, disappearing on deuteration, OH), 5.82 (d, 1 H, $J_{1,2}$ 3.2 Hz, H-1), and 7.05–8.1 (m, 5 H, P-C_6H_5); *m/z* 358 (M^+).

*5-O-(Imidazol-1-yl-thiocarbonyl)-1,2-O-isopropylidene-5-C-(methoxyphenylphosphinyl)-3-O-methyl-α-*D*-xylofuranose* (**8**). — To a solution of **7** (3.81 g) in 1,2-dichloroethane (50 mL) was added TCDI (2.70 g), and the mixture was heated for 30 min at 90° (bath), cooled, diluted with dichloromethane, washed with cold dilute HCl, saturated aqueous $NaHCO_3$, and water, dried (Na_2SO_4), and evaporated *in vacuo*, to give **8** (4.44 g, 89%) as a syrup; $[\alpha]_D^{20}$ ~ −0.2° (*c* 1.10, $CHCl_3$); ^{1}H-n.m.r. data: δ 1.27, 1.36, 1.42 (3 s, 6 H, CMe_2), 3.02, 3.34 (2 s, 3 H, OMe-3, overlapping with H-5), 3.66, 3.84 (2 s, 3 H, overlapping with H-3, P-OMe), 4.46, 4.47 (2 d, 1 H, $J_{1,2}$ 3.4 Hz, overlapping with H-4, H-2), 5.75, 5.79 (2 d, 1 H, $J_{1,2}$ 3.4 Hz, H-1), and 6.2–8.3 (m, 8 H, P-C_6H_5 and imidazolyl-H-3); *m/z* 468 (M^+).

*5-Deoxy-1,2-O-isopropylidene-5-C-(methoxyphenylphosphinyl)-3-O-methyl-α-*D*-xylofuranose* (**9a, 9b**). — To a solution of **8** (3.89 g) in toluene (150 mL) was added tributyltin hydride (3.5 mL), and the mixture was heated for 1.5 h at 120° (bath), cooled, and evaporated *in vacuo*. The residue was dissolved in acetonitrile–hexane, and the layers were separated. The acetonitrile layer was washed with hexane (to extract the tin compound), and evaporated *in vacuo*, to give crude mixture **9** as a syrup (2.82 g, 99%). By chromatography on a column of silica gel with 9:1 EtOAc–hexane which was gradually changed to EtOAc, as the eluant, the crude mixture was separated into **9a**, colorless needles (1.40 g, 49%) and **9b**, colorless prisms (1.39 g, 49%).

Compound **9a**, R_F 0.54 (EtOAc); m.p. 111.5–112° (after recrystallization from EtOAc–hexane), $[\alpha]_D^{18}$ −56.8° (*c* 1.76, $CHCl_3$); ^{1}H-n.m.r. data: δ 1.39, 1.45 (2 s, 6 H, CMe_2), 2.41 (dd, 2 H, $J_{5,P}$ 14.8, $J_{5,5'}$ 7.2 Hz, H-5,5'), 3.03 (s, 3 H, OMe-3), 3.58 (d, 3 H, J_P 11.5 Hz, overlapping with H-3, P-OMe), 4.46 (d, 1 H, $J_{1,2}$ 3.8 Hz, overlapping with H-4, H-2), 5.72 (d, 1 H, $J_{1,2}$ 3.8 Hz, H-1), and 7.3–8.0 (m, 5 H, P-C_6H_5); *m/z* 342 (M^+).

Anal. Calc. for $C_{16}H_{23}O_6P$: C, 56.14; H, 6.77. Found: C, 56.08; H, 6.79.

Compound **9b**, R_F 0.46 (EtOAc); m.p. 91.5–92.5° (after recrystallization from EtOAc–hexane), $[\alpha]_D^{18}$ −36.8° (*c* 1.90, $CHCl_3$); ^{1}H-n.m.r. data: δ 1.36, 1.44 (2 s, 6 H, CMe_2), 2.05–2.6 (m, 2 H, H-5,5'), 3.45 (s, 3 H, OMe-3), 3.61 (d, 3 H, J_P 11.2 Hz, P-OMe), 3.62 (d, 1 H, $J_{3,4}$ 3.7 Hz, H-3), 4.0–4.55 (m, 1 H, H-4), 4.48 (d, 1 H, $J_{1,2}$ 3.8 Hz, H-2), 5.72 (d, 1 H, $J_{1,2}$ 3.8 Hz, H-1), and 7.3–8.0 (m, 5 H, P-C_6H_5); *m/z* 342 (M^+).

Anal. Calc. for $C_{16}H_{23}O_6P$: C, 56.14; H, 6.77. Found: C, 56.12; H, 6.85.

5-Deoxy-1,2-O-isopropylidene-5-C-(phenylphosphinyl)-3-O-methyl-α-D-xylofuranose (**10**). — To a solution of **9** (1.31 g) in THF (40 mL) was added a 70% solution of SDMA (2.5 g, in benzene) plus THF (20 mL) at 0° under nitrogen. After 30 min, a small amount of water containing conc. HCl (0.2 mL) was added at 0° (to decompose the excess of SDMA), the mixture filtered, and the filtrate evaporated *in vacuo*. A solution of the residue in chloroform was washed with water, dried (Na_2SO_4), and evaporated *in vacuo*, to give **10** (1.09 g, 92%) as a colorless syrup. The i.r. and ^{1}H-n.m.r. spectra of **10** from **9a** and **9b** were respectively identical; $[\alpha]_D^{18}$ −21.4° (*c* 2.08, $CHCl_3$), ν_{max}^{KBr} 2340 cm^{-1} (P-H); ^{1}H-n.m.r. data: δ 1.28, 1.32, 1.47 (3 s, 6 H, CMe_2), 2.1–2.7 (m, 2 H, H-5,5′), 3.47 (d, 3 H, J_P 8.0 Hz, P-OMe), 3.55, 3.57 (2 d, 1 H, $J_{3,4}$ 1.9 Hz, H-3), 3.9–4.9 (m, 1 H, H-4), 4.51, 4.55 (2 d, 1 H, $J_{1,2}$ 4.1 Hz, H-2), 5.77, 5.80 (2 d, 1 H, $J_{1,2}$ 4.1 Hz, H-1), 7.3–8.0 (m, 5 H, P-C_6H_5), and 11.60 (m, $^1/_2$ H, disappeared on deuteration, P-H); *m/z* 312 (M^+).

Hydrolysis of **10** *and 1,2,4-tri-O-acetyl-5-deoxy-3-O-methyl-5-C-[(R) and (S)-phenylphosphinyl]-α- and -β-D-xylopyranose* (**12a–d**). — To a solution of **10** (238 mg) in ethanol (5 mL) was added 0.1M HCl (10 mL). The mixture was heated under nitrogen for 3 h at 110° (bath), the ethanol being allowed to evaporate gradually, cooled, diluted with water, and the acid neutralized with Amberlite IR-45 ion-exchange resin; this was then washed with water (20 mL × 5), and filtered, and the filtrate and washings were combined, and evaporated *in vacuo*, to give syrupy **11**. This was treated with acetic anhydride (4 mL) in dry pyridine (15 mL) in the usual way[13,14], to give crude mixture **12** as a white, amorphous material or a syrup (275 mg, 91% from **10**); on crystallization from ethanol–hexane, this was separated into colorless needles (96 mg) and a syrup (150 mg). The crystals were carefully recrystallized from ethanol and ethanol–hexane, to give **12a** and **12b**. The syrup was separated by chromatography on a column of silica gel by elution with EtOAc, which was gradually changed to 50:1 EtOAc–methanol, to give **12b**, **12c**, and **12d**.

5-C-[(R)-Phenylphosphinyl]-α-D-xylopyranose derivative (**12a**). R_f 0.56 (EtOAc); colorless needles (52 mg, 17% from **10**), m.p. 295–296° (recrystallized from ethanol), $[\alpha]_D^{18}$ −31.4° (*c* 2.23, $CHCl_3$); ^{1}H-n.m.r. data: δ 1.86, 2.00, 2.03 (3 s, 9 H, 3 OAc), 2.1–3.0 (m, 2 H, H-5,5′), 3.47 (s, 3 H, OMe-3), 3.56 (t, 1 H, $J_{2,3} = J_{3,4} = 9.0$ Hz, H-3), 5.15–5.95 (m, 3 H, H-1,2,3), and 7.15–8.0 (m, 5 H, P-C_6H_5); *m/z* 398 (M^+).

Anal. Calc. for $C_{18}H_{23}O_8P$: C, 54.27; H, 5.82. Found: C, 54.30; H, 5.79.

5-C-[(R)-Phenylphosphinyl]-β-D-xylopyranose derivative (**12b**). R_f 0.56 (EtOAc); colorless needles (51 mg, 17% from **10**); m.p. 200–200.5° (recrystallized ethanol–hexane), $[\alpha]_D^{18}$ −343.6° (*c* 1.79, $CHCl_3$); ^{1}H-n.m.r. data: δ 1.96, 1.99, 2.08 (3 s, 9 H, 3 OAc), 2.25–3.15 (m, 2 H, H-5,5′), 3.52 (s, 3 H, OMe-3), 3.67 (t, 1 H, $J_{2,3} = J_{3,4} = 9.0$ Hz, H-3), 5.15–5.9 (m, 3 H, H-1,2,4), and 7.15–8.0 (m, 5 H, P-C_6H_5); *m/z* 398 (M^+).

Anal. Calc. for $C_{18}H_{23}O_8P$: C, 54.27; H, 5.82. Found: C, 53.88; H, 5.81.

5-C-[(S)-Phenylphosphinyl]-β-D-xylopyranose derivative (**12c**). R_f 0.36

(EtOAc), colorless syrup (25 mg, 8% from **10**); $[\alpha]_D^{18}$ −223.5° (*c* 3.49, $CHCl_3$); ^{1}H-n.m.r. data: δ 1.99, 2.02, 2.08 (3 s, 9 H, 3 OAc), 2.0–3.2 (m, 2 H, H-5,5'), 3.45 (s, 3 H, OMe-3), 3.60 (t, 1 H, $J_{2,3} = J_{3,4} = 8.8$ Hz, H-3), 4.7–5.3 (m, 2 H, H-2,4), 5.79 (t, 1 H, $J_{1,P} = J_{1,2} = 10.7$ Hz, H-1), and 7.4–8.1 (m, 5 H, P-C_6H_5); *m/z* 398 (M^+).

5-C-[(S)-Phenylphosphinyl]-α-D-xylopyranose derivative (**12d**), R_F 0.21 (EtOAc), colorless syrup (37 mg, 12% from **10**); $[\alpha]_D^{18}$ −16.6° (*c* 5.13, $CHCl_3$); ^{1}H-n.m.r. data: δ 2.02, 2.10, 2.21 (3 s, 9 H, 3 OAc), 2.3–3.1 (m, 2 H, H-5,5'), 3.41 (s, 3 H, OMe-3), 3.62 (t, 1 H, $J_{2,3} = J_{3,4} = 10.0$ Hz, H-3), 4.5–5.1 (m, 2 H, H-2,4), 5.99 (2 t, 1 H, $J_{1,P}$ 8.0, $J_{1,2}$ 2.5 Hz, and probably, $J_{1,5}$ 2.5 Hz, H-1), and 7.3–8.0 (m, 1 H, P-C_6H_5); *m/z* 398 (M^+).

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