

Novel Synthesis of 2-Phosphinoylphospholane 1-Oxide Derivatives

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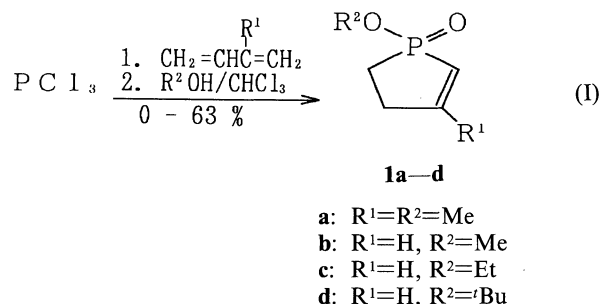
Synopsis. Reactions of 2-phospholene 1-oxides with *N*-bromoacetamide in aqueous organic solvent produced 2-bromo-3-hydroxyphospholane 1-oxides, which reacted with sodium salts of dialkyl phosphonates to afford novel 2-phosphinoylphospholane 1-oxide derivatives. A 4-phosphinoyl-2-phospholene derivative was also prepared.

Phosphorus-in-ring sugar analogs¹⁾ (phosphono sugars),²⁻⁴⁾ in which an oxygen atom in the hemiacetal ring is substituted by a phosphorus atom,^{5,6)} such as 5-deoxy-5-phosphinylaldopyranoses⁷⁾ and 4-deoxy-4-phosphinylaldofuranoses,⁸⁾ are expected to exert some biological activities, as observed in hetero sugars.^{3,6)} In the previous synthesis of sugar analogs having a phosphorus atom in the hemiacetal ring,⁹⁾ the oxygen atom in the hemiacetal ring of the starting sugar materials was replaced by a phosphorus atom via successive reactions of carbon-phosphorus bond formation and a ring-closure reaction with the phosphorus atom.^{1,5)} On the contrary, we have reported a new method which starts from 2-phospholenes: 3-Phosphacyclopentene derivatives were prepared by a Diels-Alder reaction of 1,3-dienes with phosphorus halides, followed by conversion of the carbon-carbon double bond into a *cis* glycol.¹⁰⁾ The introduction of a substituent which included a hetero atom, such as oxygen, nitrogen, sulfur, or phosphorus, into the 2-position of the phospholene, i.e., *O*-, *N*-, *S*-, and *P*-glycoside analogs, respectively, was interesting on the basis of the biological activities of sugar glycosides.¹¹⁻¹⁴⁾ The present note concerns novel chemical conversion of phospholene 1-oxides into new 2- and 4-phosphinoyl derivatives of phospholane and phospholene 1-oxides.

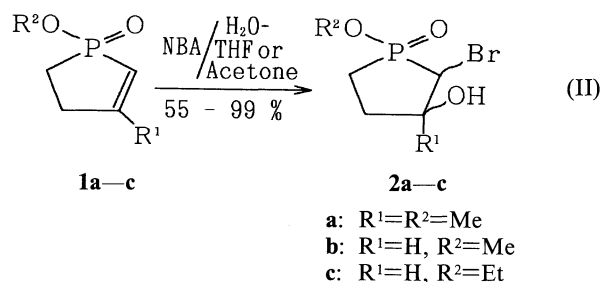
Results and Discussion

2-Phospholenes **1a—c** were prepared by a Diels-Alder reaction of phosphorus trichloride with 1,3-dienes, and the successive alcoholysis of the adducts [Eq. (I)].¹⁵⁾ The alcoholysis of the adduct, i.e., 1-chloro-3-phospholenium chloride, with 2-methyl-2-propanol, however,

was unsuccessful; this was probably because of the unstability of 1-(2-methyl-2-propanoxy)-2-phospholene 1-oxide, as well as the fact that di-*t*-butyl phosphonate was spontaneously hydrolyzed at room temperature, and that triethyl phosphite and di-*t*-butyl peroxide gave triethyl phosphate while triethyl phosphite and diethyl peroxide gave a pentavalent adduct, pentaethoxyphosphorane.^{16,17)} These results are summarized in Table 1.



Treatment of 2-phospholenes **1a—c** with *N*-bromoacetamide (NBA) introduced bromo and hydroxyl groups at the 2- and the 3-positions, respectively, of the phospholene to afford bromohydrins **2a—c** (Eq. (II) and Table 2).¹⁸⁾



The reaction of bromohydrins **2a—c** with trimethyl phosphite did not proceed to afford 2-phosphinoylphos-

Table 1. Preparation of 2-Phospholene 1-Oxides **1**

Product ^{a)}	R ¹	R ²	Reaction conditions		Yield/% ^{b)}	Bp/°C (mmHg)
			Temp/°C	Time/d		
1a	Me	Me	r.t.	8	38	90 (0.25)
1b	H	Me	35	20	54	79 (0.40)
1c	H	Et	35	30	63	78 (0.20)
1d	H	<i>t</i> Bu	35	25	ca. 0	—

a) All products gave satisfactory IR, ¹H and ¹³C NMR spectral data. b) Yield of isolated product based on phosphorus trichloride.

Table 2. Preparation of Bromohydrins **2**

Bromohydrins ^{a)}	R ¹	R ²	Reaction conditions		Yield/% ^{b)}	Mp/°C
			Solvent	Time/h or d		
2a	Me	Me	Acetone-H ₂ O	18 h	84	137—139
2b	H	Me	THF-H ₂ O	3 d	55	oil
2c	H	Et	THF-H ₂ O	4 d	99	oil

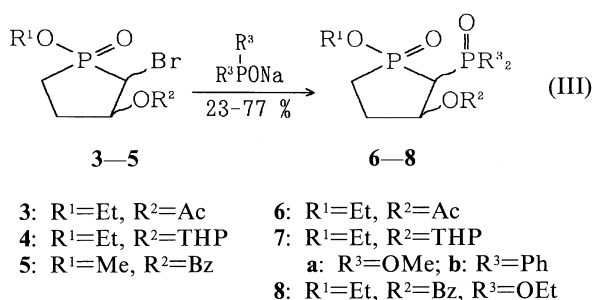
a) All products gave satisfactory IR, ¹H and ¹³C NMR spectral data. b) Yield of isolated product based on 2-phospholene 1-oxide.

Table 3. Becker Reaction of 2-Bromophospholane 1-Oxides **3—5** to Afford 2-Phosphinoyl Derivatives **6—8**

Substrate	Base/Solvent	R ¹	R ²	R ³	Product ^{a)}	Yield/% ^{b)}
3	NaH/THF	Et	Ac	OMe	6a	23
3	NaH/Toluene	Et	Ac	Ph	6b	77
4	NaH/THF	Et	THP	OMe	7a	49
4	NaH/Toluene	Et	THP	Ph	7b	61
5	NaH/Toluene	Me	Bz	OEt	8	29

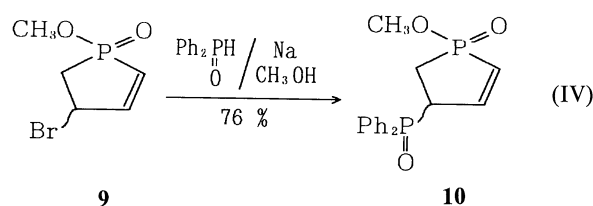
a) All products gave satisfactory IR, ¹H and ¹³C NMR spectral data. b) Yield of isolated product based on **3—5**.

pholane 1-oxide derivatives; however, the ¹H NMR spectra of the reaction mixture showed a disappearance of the OH signal due to an unanalyzable reaction. The hydroxyl group of phospholanes **2a—c** was thus protected as the acetyl, tetrahydropyranyl, and benzoyl derivatives **3—5** by the usual method.¹⁹⁾ The protected 2-bromophospholanes **3—5** were treated with sodium salts of dialkyl phosphonates or diphenylphosphine oxide to afford 2-phosphinoylphospholane 1-oxide derivatives **6—8** [Eq. (III)]. The result is shown in Table 3.



Although diphenylphosphinoyl derivatives **6b** and **7b** were obtained in good yield, dimethoxyphosphinoyl and diethoxyphosphinoyl derivatives **6a**, **7a**, and **8** were obtained in low yield. The ¹H NMR spectra of 2-(dialkoxyphosphinoyl)phospholane derivatives **6a** and **7a** showed the methoxyl protons as one pair of a doublet, suggesting the presence of diastereomers.

4-Bromo-1-methoxy-2-phospholene (**9**) afforded the expected 4-diphenylphosphinoyl-1-methoxy-2-phospholene 1-oxide (**10**) in 76% yield by a treatment of diphenylphosphine oxide under similar reaction conditions to those for the preparation of 2-phosphinoyl derivatives **6b** and **7b** [Eq. (IV)].



Experimental

All bp's and mp's were uncorrected. The ¹H NMR spectra were measured on Hitachi R-24B (60 MHz) and R-40 (90 MHz) spectrometers with TMS used as an internal standard. ¹³C NMR spectra were measured on a JEOL EX-90 (22.4 MHz) spectrometer with proton decoupling, and IR spectra on a Japan Spectroscopic Co., Ltd. A-3 IR spectrophotometer.

1-Ethoxy-2-phospholene 1-Oxide (1c). Phosphorus trichloride (45 cm³, 500 mmol) reacted with excess 1,3-butadiene (60 cm³, 720 mmol) for 30 d at 35°C. The resulting material was dissolved in CHCl₃ (250 cm³), and then slowly added into excess ethanol (200 cm³) at -20°C. After neutralization of the mixture with solid NaHCO₃, the insoluble material was filtered off, the filtrate was evaporated, and the residue was distilled in vacuo to afford **1c** (47.3 g, 63% yield); bp 78°C/0.20 mmHg;²⁰⁾ ¹H NMR (CDCl₃) δ=1.34 (t, 3H, J_{HCH}=7.0 Hz, P-O-CH₂-CH₃), 1.7—2.1 (m, 2H, P-CH₂-CH₂-), 2.5—2.9 (m, 2H, P-CH₂-CH₂-), 4.10 (dq, 2H, J_{HCH}=7.4 Hz, J_{HCP}=7.4 Hz, P-O-CH₂-), 6.20 (ddt, 1H, J_{HC=CH}=8.4 Hz, J_{HC-CP}=23.7 Hz, J_{HCH}=8.6 Hz, P-CH=CH-), and 7.02 (ddt, 1H, J_{HC=CH}=8.4 Hz, J_{HCP}=48.7 Hz, J_{HC=CCH}=2.6 Hz, P-CH=CH-); ¹³C NMR (CDCl₃) δ=14.7 (d, J_{CCOP}=6.0 Hz, P-O-CH₂-CH₃), 18.5 (d, J_{CP}=96 Hz, P-CH₂-), 25.3 (d, J_{CCP}=15.4 Hz, P-CH₂-CH₂-), 58.9 (d, J_{COP}=6.7 Hz, P-O-CH₂-), 121.9 (d, J_{CP}=120.2 Hz, P-CH=CH-), and 150.0 (d, J_{C-CP}=32.2 Hz, P-CH=CH-).

Similarly, **1a** and **1b** were prepared. **1a:** ¹H NMR (CDCl₃) δ=1.7—2.2 (m, 2H, -CH₂-C=), 2.00 (s, 3H, C-CH₃), 2.3—2.8 (m, 2H, P-CH₂-), 3.69 (d, 3H, J_{HCP}=11.1 Hz, P-O-CH₃), and 5.84 (dm, 1H, J_{HCP}=23.5 Hz, P-CH=C-); ¹³C NMR (CDCl₃) δ=19.6 (d, J_{CC-CP}=36.9 Hz, P-CH=C-CH₃), 21.3 (d, J_{CP}=75.2 Hz, P-CH₂-), 30.1 (d, J_{CCP}=12.8 Hz, P-CH₂-CH₂-), 50.1 (d, J_{COP}=6.0 Hz, P-O-CH₃), 116.1 (d, J=127.6 Hz, P-CH=C-), and 162.8 (d, J_{CCP}=33.6 Hz, P-CH=C-). **1b:** ¹H NMR

(CDCl₃) δ =1.7–2.1 (m, 2H, $-\text{CH}_2-\text{CH}=\text{}$), 2.5–3.0 (m, 2H, $\text{P}-\text{CH}_2-$), 3.73 (d, 3H, $J_{\text{HCO}}=11.1$ Hz, $\text{P}-\text{O}-\text{CH}_3$), 6.21 (ddm, 1H, $J_{\text{HC}=\text{CH}}=8.6$ Hz, $J_{\text{HC}=\text{CP}}=23.7$ Hz, $\text{P}-\text{CH}=\text{CH}-$), and 7.05 (ddm, 1H, $J_{\text{HC}=\text{CH}}=8.6$ Hz, $J_{\text{HCP}}=49.0$ Hz, $\text{P}-\text{CH}=\text{CH}-$); ¹³C NMR (CDCl₃) δ =18.3 (d, $J_{\text{CP}}=95.4$ Hz, $\text{P}-\text{CH}_2-$), 25.8 (d, $J_{\text{CCP}}=15.4$ Hz, $\text{P}-\text{CH}_2-\text{CH}_2-$), 50.4 (d, $J_{\text{COP}}=6.0$ Hz, $\text{P}-\text{O}-\text{CH}_3$), 121.9 (d, $J_{\text{CP}}=120.9$ Hz, $\text{P}-\text{CH}=\text{}$), and 151.4 (d, $J_{\text{CCP}}=31.6$ Hz, $\text{P}-\text{CH}=\text{CH}-$).

2-Bromo-3-hydroxy-1-methoxyphospholane 1-Oxide (2b). NBA (12.6 g, 90 mmol) was added to a solution of **1b** (9.27 g, 70 mmol) in THF (20 cm³) and water (80 cm³). The mixture was stirred for 3 d at room temperature. The solvent was evaporated, and the residue was extracted with CHCl₃ (40 cm³×5); the CHCl₃ layer was dried (Na₂SO₄) and evaporated to afford product **2b** (9.05 g, 55% yield); oil; ¹H NMR (CDCl₃) δ =1.4–2.6 (m, 4H, $\text{P}-\text{CH}_2-\text{CH}_2-$), 3.3–3.7 (m, 1H, $\text{P}-\text{CHBr}$), 3.71, 3.80 (d×2, 3H, $J_{\text{HCO}}=11.2$ Hz, $\text{P}-\text{O}-\text{CH}_3$), 3.9–4.4 (m, 1H, CHOH), and 5.30 (bs, 1H, OH); IR (KBr) 3500 (OH) and 1240 cm⁻¹ (P=O).

Similarly, **2a** and **2c** were prepared. **2a**: ¹H NMR (CDCl₃) δ =1.46 (s, 3H, $=\text{C}-\text{CH}_3$), 1.7–2.3 (m, 4H, $\text{P}-\text{CH}_2-\text{CH}_2-$), 3.74, 3.79 (d×2, 3H, $J_{\text{HCP}}=11.2$ Hz, $\text{P}-\text{O}-\text{CH}_3$), 3.9–4.0 (m, 1H, $\text{P}-\text{CHBr}$), and 4.74 (bs, 1H, OH). **2c**: ¹H NMR (CDCl₃) δ =1.34 (t, 3H, $J_{\text{HCH}}=7.4$ Hz, $\text{P}-\text{O}-\text{CH}_2-\text{CH}_3$), 1.4–2.6 (m, 4H, $\text{P}-\text{CH}_2-\text{CH}_2-$), 3.5–4.4 (m, 2H, $\text{P}-\text{CHBr}-\text{CHOH}$), 4.17 (dq, 2H, $J_{\text{HCH}}=7.4$ Hz, $J_{\text{HCO}}=7.5$ Hz, $\text{P}-\text{O}-\text{CH}_2-$), and 5.29 (s, 1H, OH).

2-Bromo-1-ethoxy-3-(tetrahydropyranyloxy)phospholane 1-Oxide (4). To an ice-cooled solution of **2c** (1.01 g, 4.1 mmol) and 3,4-dihydro-2H-pyran (1.45 cm³, 16 mmol) in dry dichloromethane (20 cm³) was added a catalytic amount of *p*-toluenesulfonic acid monohydrate. The mixture was stirred for 10 min at 0°C, and then stirred for additional 2 h at room temperature. The mixture was diluted with CHCl₃ (12 cm³), washed successively with saturated brine, saturated NaHCO₃, water, and saturated brine. The organic layer was dried with Na₂SO₄, and evaporated to afford product **4** (1.13 g, 83% yield); oil; ¹H NMR (CDCl₃) δ =1.38 (t, 3H, $J_{\text{HCH}}=7.0$ Hz, $\text{P}-\text{O}-\text{CH}_2-\text{CH}_3$), 1.4–2.9 (m, 10H, $\text{P}-\text{CH}_2-\text{CH}_2-$, $\text{O}-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-$), 3.4–3.7 (m, 1H, $\text{P}-\text{CHBr}$), 3.8–4.0 (m, 1H, CHOTHP), 4.1–4.3 (m, 2H, $\text{O}-\text{CH}_2-\text{CH}_2-$), 4.25 (dq, 2H, $J_{\text{HCH}}=7.4$ Hz, $J_{\text{HCO}}=8.0$ Hz, $\text{P}-\text{O}-\text{CH}_2-$), and 4.7–4.9 (m, 1H, $\text{O}-\text{CH}-\text{O}$).

Similarly, **3** and **5** were prepared.¹⁹⁾ **3** (82% yield): ¹H NMR (CDCl₃) δ =1.36 (t, 3H, $J_{\text{HCH}}=6.9$ Hz, $\text{P}-\text{O}-\text{CH}_2-\text{CH}_3$), 1.8–2.8 (m, 4H, $\text{P}-\text{CH}_2-\text{CH}_2-$), 2.04 (s, 3H, Ac), 3.84 (m, 1H, $\text{P}-\text{CHBr}$), 4.25 (dq, $J_{\text{HCH}}=6.9$ Hz, $J_{\text{HCO}}=7.0$ Hz, $\text{P}-\text{O}-\text{CH}_2-\text{CH}_3$), and 5.15 (m, 1H, CHOAc). **5** (52% yield): ¹H NMR (CDCl₃) δ =1.5–2.9 (m, 4H, $\text{P}-\text{CH}_2-\text{CH}_2-$), 3.75, 3.85 (d×2, 3H, $J_{\text{HCO}}=11.2$ Hz, $\text{P}-\text{O}-\text{CH}_3$), 3.9–4.3 (m, 1H, $\text{P}-\text{CHBr}$), 5.1–5.6 (m, 1H, CHOBz), and 7.1–8.1 (m, 5H, Ph).

2-(Dimethoxyphosphinoyl)-1-ethoxy-3-(tetrahydropyranyloxy)phospholane 1-Oxide (7a). To a toluene (25 cm³) solution of dimethyl phosphonate (121 mg, 1.1 mmol) was slowly added NaH (60% oil, 44.0 mg, 1.1 mmol); the mixture was then stirred for 1 h. To the mixture, 2-bromo-1-ethoxy-3-(tetrahydropyranyloxy)phospholane 1-oxide (**4**, 294.0 mg, 0.90 mmol) in toluene (30 cm³) was slowly added and refluxed for 3 h. An insoluble material was filtered off, and the filtrate was washed with water, and dried (Na₂SO₄). The solvent was evaporated to afford product **7a** (155.4 mg, 49% yield); oil; ¹H NMR (CDCl₃) δ =1.15 (t, 3H, $J_{\text{HCH}}=7.0$ Hz, $\text{P}-\text{O}-\text{CH}_2-\text{CH}_3$), 1.2–2.5 (m, 10H, $\text{P}-\text{CH}_2-\text{CH}_2-$, $\text{O}-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-$), 3.4–4.4 (m, 6H, $\text{O}-\text{CH}_2-\text{CH}_2-$, $\text{P}-\text{O}-\text{CH}_2-\text{CH}_3$, $\text{P}-\text{CH}-\text{CHOTHP}$), 3.55, 3.60 (d×2, 6H, $J_{\text{HCO}}=12.0$ Hz, 2×POCH₃), and 4.5–4.8 (m, 1H, $\text{O}-\text{CH}-\text{O}$).

Similarly, **6a**, **6b**, **7b**, **8**, and **10** (sodium metal/methanol was used as the base/solvent) were prepared. **6a**: ¹H NMR (CDCl₃)

δ =1.33 (t, 3H, $J_{\text{HCH}}=7.4$ Hz, $\text{P}-\text{O}-\text{CH}_2-\text{CH}_3$), 1.3–2.8 (m, 4H, $\text{P}-\text{CH}_2-\text{CH}_2-$), 2.04 (bs, 3H, Ac), 3.69 (d, 6H, $J_{\text{HCO}}=12.0$ Hz, $\text{P}-\text{O}-\text{CH}_3$), and 3.7–4.4 (m, 4H, $\text{P}-\text{O}-\text{CH}_2-\text{CH}_3$, $\text{P}-\text{CH}-\text{CHOAc}$). **6b**: ¹H NMR (CDCl₃) δ =1.40 (t, 3H, $J_{\text{HCH}}=7.0$ Hz, $\text{P}-\text{O}-\text{CH}_2-\text{CH}_3$), 1.7–2.4 (m, 4H, $\text{P}-\text{CH}_2-\text{CH}_2-$), 2.05 (s, 3H, Ac), 3.8–4.4 (m, 1H, $\text{P}-\text{CH}-\text{P}$), 4.11 (dq, 2H, $J_{\text{HCO}}=8.0$ Hz, $J_{\text{HCH}}=7.0$ Hz, $\text{P}-\text{O}-\text{CH}_2-\text{CH}_3$), 5.0–5.5 (m, 1H, CHOAc), and 6.8–7.9 (m, 10H, 2×Ph). **7b**: ¹H NMR (CDCl₃) δ =1.49 (t, 3H, $J_{\text{HCH}}=7.4$ Hz, $\text{P}-\text{O}-\text{CH}_2-\text{CH}_3$), 1.4–2.7 (m, 10H, $\text{P}-\text{CH}_2-\text{CH}_2-$, $\text{O}-\text{CH}-\text{CH}_2-\text{CH}_2-\text{CH}_2-$), 3.4–4.6 (m, 6H, $\text{P}-\text{CH}-\text{CH}-\text{OTHP}$, $\text{O}-\text{CH}_2-$, $\text{P}-\text{O}-\text{CH}_2-\text{CH}_3$), 4.8–5.0 (m, 1H, $\text{O}-\text{CH}-\text{O}$), and 7.0–8.0 (m, 10H, 2×Ph). **8**: ¹H NMR (CDCl₃) δ =1.22 (t, 6H, $J_{\text{HCH}}=7.0$ Hz, 2× $\text{P}-\text{O}-\text{CH}_2-\text{CH}_3$), 1.3–2.4 (m, 4H, $\text{P}-\text{CH}_2-\text{CH}_2-$), 3.5–4.3 (m, 8H, 2× $\text{P}-\text{O}-\text{CH}_2-\text{CH}_3$, $\text{P}-\text{O}-\text{CH}_3$, $\text{P}-\text{CH}-\text{P}$), 5.1–5.5 (m, 1H, $\text{CH}-\text{OBz}$), and 7.1–8.1 (m, 5H, Ph). **10**: ¹H NMR (CDCl₃) δ =2.0–2.8 (m, 2H, $\text{P}-\text{CH}_2-$), 3.2–3.5 (m, 1H, $\text{P}-\text{CH}-$), 3.59 (d, 3H, $J_{\text{HCO}}=10.8$ Hz, $\text{P}-\text{O}-\text{CH}_3$), and 6.0–8.0 (m, 12H, 2×Ph, $\text{P}-\text{CH}=\text{CH}-$).

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