

molecules through hydrogen bonds, O(5)···O(W2)···O(7')( $-\frac{1}{2} + x, -\frac{1}{2} + y, z$ ), O(3')···O(W3)···O(3')( $\frac{3}{2} - x, -\frac{1}{2} + y, -z$ ) and O(4')···O(W4)···O(4')( $\frac{1}{2} + x, \frac{1}{2} + y, z$ ). The water O(W7) molecule located on the twofold axis forms hydrogen bonds to the O(6'') atoms of the rhamnose moiety and to the water O(W6) molecules. As a characteristic feature of the crystal packing, a widely spread intermolecular stacking interaction with a separation of about 3.68 Å is observed between two nearly planar 4-hydroxyphenylbenzopyranone groups which are related by a twofold screw axis as shown in Fig. 2.

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## Structures of (2*R*,4*S*,5*R*)-3,4-Dimethyl-2,5-diphenyl-1,3,2-oxazaphospholidine(*P*-*B*)borane (1) and of (2*R*,5*S*)-5-Methyl-2,4,4-triphenyl-1,3,2-dioxaphospholane(*P*-*B*)borane (2)

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**Abstract.** Compound (1): C<sub>16</sub>H<sub>21</sub>BNOP, m.p. = 383 K, *M<sub>r</sub>* = 285.1, monoclinic, *P*2<sub>1</sub>, *a* = 10.570 (5), *b* = 7.796 (1), *c* = 10.409 (1) Å, β = 107.74 (1)°, *V* = 816.9 (8) Å<sup>3</sup>, *Z* = 2, *D<sub>m</sub>* = 1.158 (floatation in tetrachloromethane–hexane), *D<sub>x</sub>* = 1.159 Mg m<sup>-3</sup>, λ(Mo *K*α) = 0.71070 Å, μ = 0.157 mm<sup>-1</sup>, *F*(000) = 304, *T* = 294 K, *R* = 0.037, *wR* = 0.036 for 1104 significant reflections. Absolute configuration *R* of P atom deduced from known *R* configuration of C(5) and *S* configuration of C(4). Compound (2): C<sub>21</sub>H<sub>22</sub>BO<sub>2</sub>P, m.p. = 431 K, *M<sub>r</sub>* = 348.2, monoclinic,

*P*2<sub>1</sub>, *a* = 17.261 (4), *b* = 10.509 (5), *c* = 10.689 (1) Å, β = 96.73 (1)°, *V* = 1926 (2) Å<sup>3</sup>, *Z* = 4, *D<sub>m</sub>* = 1.199 (floatation in tetrachloromethane–hexane), *D<sub>x</sub>* = 1.201 Mg m<sup>-3</sup>, μ(Mo *K*α) = 0.147 mm<sup>-1</sup>, *F*(000) = 736, *T* = 294 K, *R* = 0.049, *wR* = 0.058 for 3361 significant reflections. Absolute configuration *R* of P atom deduced from known *S* configuration of C(4) atom.

**Introduction.** The *trans* isomers of 2-phenyl-1,3,2-oxazaphospholidine and 1,3,2-dioxaphospholane are

key intermediates in asymmetric synthesis of tertiary phosphine oxides (Jugé & Genet, 1989). The configuration at the phosphorus centre has been established by spectroscopic and chemical methods. The conversion of chiral phosphines into corresponding borane complexes has been proved to be fully stereospecific and proceeds with retention of configuration at the P atom (Imamoto, Kusumoto, Suzuki & Sato, 1985). In our continuing program for the synthesis of chiral organophosphorus compounds from diheterophosphacycloalkane complexes, we have investigated the stereoselectivity and/or the relative configuration of the conversion mentioned above when the P atom is part of a five-membered ring.

**Experimental.** Compound (1): Preparation by reaction of BH<sub>3</sub>.THF with the oxazaphospholidine previously obtained from bis(diethylamino)phenylphosphine and (-)-ephedrine. Yield: 70%,  $[\alpha]_D^{20} = +4.3^\circ$  ( $c = 4$ ; CHCl<sub>3</sub>). Colourless crystal: 0.4 × 0.3 × 0.2 mm; precession, P<sub>2</sub>; Philips PW1100 diffractometer, graphite monochromator; Mo K $\alpha$  radiation. Unit cell: least squares on 25 reflections,  $14 < \theta < 15^\circ$ ; intensity measurements by  $\theta$ - $2\theta$  scan;  $(\sin\theta)/\lambda$  range: 0.05–0.71 Å<sup>-1</sup>; scan speed: 0.09° s<sup>-1</sup>; scan range:  $1^\circ + 0.345^\circ \tan\theta$ ; background measured for half of scan time, before and after every scan; three standard reflections measured every 2 h; two octants measured  $hkl$ ,  $\bar{h}kl$ ,  $h\bar{k}l$ ,  $h$  0–11,  $k$  0–9,  $l$  0–12; 1471 independent reflections collected, 1104 observed [ $I > 3\sigma(I)$ ]. Computing program: CRYSTALS (Watkin, Carruthers & Betteridge, 1985); method used to solve

Table 1. Atomic and equivalent isotropic thermal parameters for compound (1) with e.s.d.'s in parentheses

$$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$$

	x	y	z	U <sub>eq</sub> (Å <sup>2</sup> )
P(2)	0.1126 (1)	0.0039 (9)	0.8186 (1)	0.0475
B(1)	0.1335 (8)	-0.197 (1)	0.7285 (8)	0.0738
O(1)	-0.0097 (2)	0.008 (1)	0.8790 (3)	0.0579
N(3)	0.0649 (3)	0.178 (1)	0.7235 (3)	0.0479
C(4)	-0.0169 (4)	0.287 (1)	0.7813 (5)	0.0548
C(5)	-0.0946 (4)	0.158 (1)	0.8389 (5)	0.0560
C(21)	0.2574 (4)	0.053 (1)	0.9616 (4)	0.0474
C(22)	0.3805 (4)	-0.005 (1)	0.9588 (5)	0.0615
C(23)	0.4931 (5)	0.031 (1)	1.0642 (5)	0.0718
C(24)	0.4839 (6)	0.121 (1)	1.1735 (5)	0.0699
C(25)	0.3619 (6)	0.180 (1)	1.1776 (5)	0.0751
C(26)	0.2479 (5)	0.146 (1)	1.0717 (5)	0.0650
C(31)	0.1551 (6)	0.261 (1)	0.6624 (7)	0.0709
C(41)	-0.1031 (6)	0.411 (1)	0.6789 (7)	0.0770
C(51)	-0.2290 (4)	0.107 (1)	0.7470 (4)	0.0543
C(52)	-0.3418 (5)	0.157 (1)	0.7773 (5)	0.0627
C(53)	-0.4660 (5)	0.115 (1)	0.6905 (7)	0.0746
C(54)	-0.4775 (5)	0.030 (1)	0.5719 (6)	0.0747
C(55)	-0.3669 (5)	-0.020 (1)	0.5409 (5)	0.0750
C(56)	-0.2422 (4)	0.020 (1)	0.6272 (4)	0.0660

Table 2. Main interatomic distances (Å) and bond angles (°) for compound (1) with e.s.d.'s in parentheses

P(2)—B(1)	1.869 (7)	O(1)—C(5)	1.457 (6)
P(2)—O(1)	1.601 (3)	N(3)—C(4)	1.465 (6)
P(2)—N(3)	1.662 (4)	C(4)—C(5)	1.529 (6)
P(2)—C(21)	1.821 (4)		
O(1)—P(2)—B(1)	116.5 (3)	C(5)—O(1)—P(2)	113.7 (3)
N(3)—P(2)—B(1)	116.3 (3)	C(4)—N(3)—P(2)	109.8 (3)
N(3)—P(2)—O(1)	94.8 (2)	C(5)—C(4)—N(3)	103.6 (4)
C(21)—P(2)—B(1)	112.7 (3)	C(4)—C(5)—O(1)	106.4 (3)
C(21)—P(2)—O(1)	104.6 (2)	C(51)—C(5)—O(1)	110.4 (4)
C(21)—P(2)—N(3)	110.1 (2)		

structure: MULTAN11/84 (Main, Germain & Woolfson, 1984); calculations performed on a MicroVAX II; scattering factors from *International Tables for X-ray Crystallography* (1974); H atoms located on difference maps and refined with an overall isotropic thermal parameter; absorption correction: DIFABS (Walker & Stuart, 1983); secondary-extinction correction applied; weighting scheme:  $w = \{1 - [\Delta F/6\sigma(F_o)]^2\}^2$  using parameters 0.933, -0.013, 0.390; average shift/e.s.d. = 0.3 in the last cycle;  $S = 1.06$ ;  $N(\text{reflections})/N(\text{parameters}) = 4.5$  (refined by blocks);  $R(F) = 0.036$ ,  $wR(F) = 0.038$ ; max. height in final difference Fourier synthesis:  $0.2 \text{ e \AA}^{-3}$ . Fig. 1 shows the asymmetric unit and Tables 1 and 2 give atomic coordinates and selected distances and bond angles.

Compound (2): Preparation by reaction of BH<sub>3</sub>.THF with the dioxaphospholane previously obtained from dichlorophenylphosphine and (+)-S-1,1-diphenyl-1,2-propanediol. Yield: 50%,  $[\alpha]_D^{20} = -240^\circ$  ( $c = 2$ , CHCl<sub>3</sub>). Data collection and refinement as for compound (1), but colourless crystal: 0.9 × 0.9 × 0.5 mm. Unit cell: least squares on 25 reflections,  $20 < \theta < 21^\circ$ ; scan range  $1.20^\circ + 0.345^\circ \tan\theta$ ; 3526 independent reflections collected, 3361 observed;  $h$  0–20,  $k$  0–12,  $l$  0–12; same

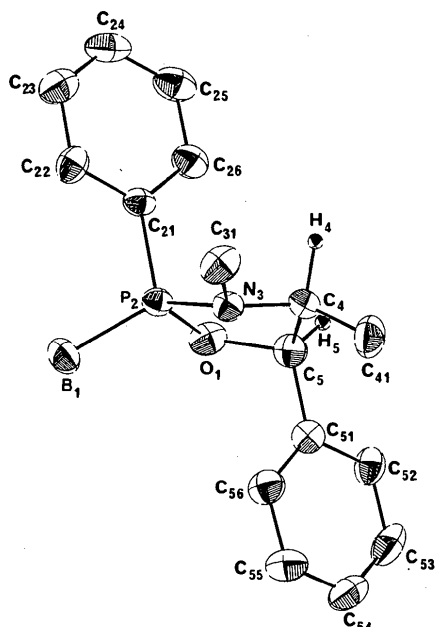


Fig. 1. ORTEP (Johnson, 1976) view of compound (1).

weighting scheme using parameters 4.096, 2.043, 2.495; H atoms given in calculated positions with coordinates not refined but recalculated after each cycle, with a refinable isotropic thermal parameter 20% higher than the thermal parameter of parent atoms; average shift/e.s.d. = 0.10 in the last cycle;  $S = 1.15$ ;  $N(\text{reflections})/N(\text{parameters}) = 7.4$  (refined by blocks);  $R(F) = 0.049$ ,  $wR(F) = 0.058$ ; max. height in final difference Fourier synthesis:  $0.5 \text{ e } \text{Å}^{-3}$ . Fig. 2 shows the molecular drawing and Tables 3 and 4 give atomic coordinates and selected distances and bond angles.\*

**Discussion.** Compound (1): The asymmetric unit contains one  $\text{C}_{16}\text{H}_{21}\text{BNOP}$  molecule (Fig. 1). The main purpose was to determine the absolute configuration of the P atom. The imaginary part of the phosphorus anomalous scattering is too small for detecting significant differences between intensities of Friedel reflections, which prevents a direct determination of the observed enantiomer. Therefore, the only way to proceed is to look at C(4) and C(5) asymmetric atoms for choosing the right enantiomer. The starting compound for the synthesis is (-)-ephedrine and the known absolute configurations of C(4) and C(5) are *S* and *R*, respectively. Under these conditions, one can see (Fig. 1) that the absolute configuration

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and all bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52885 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

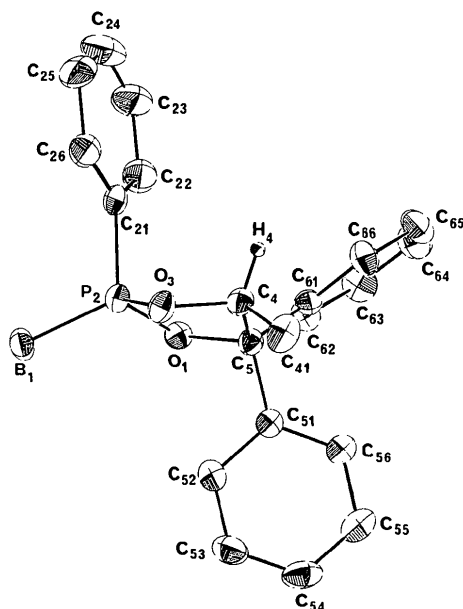


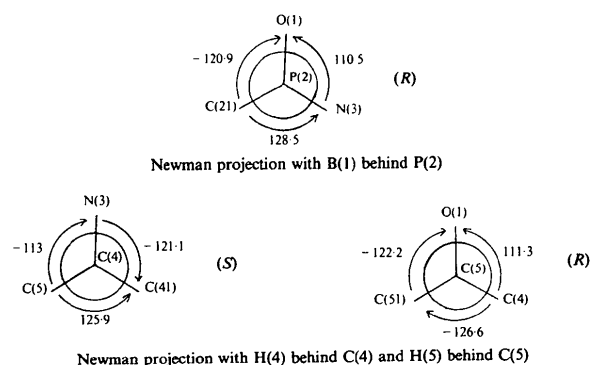
Fig. 2. ORTEP (Johnson, 1976) view of compound (2).

Table 3. Atomic and equivalent isotropic thermal parameters for compound (2) with e.s.d.'s in parentheses

$$U_{\text{eq}} = (U_{11}U_{22}U_{33})^{1/3}$$

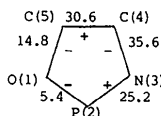
	x	y	z	$U_{\text{eq}} (\text{Å}^2)$
First molecule				
P(2)	0.53968 (5)	0.1961	0.57797 (7)	0.0443
B(1)	0.6224 (3)	0.2805 (5)	0.5068 (4)	0.0568
O(1)	0.5127 (1)	0.2636 (2)	0.7024 (2)	0.0432
O(3)	0.5613 (2)	0.0632 (3)	0.6364 (2)	0.0530
C(4)	0.5101 (2)	0.0380 (3)	0.7330 (3)	0.0465
C(5)	0.4999 (2)	0.1686 (3)	0.7984 (3)	0.0409
C(21)	0.4517 (2)	0.1817 (4)	0.4706 (3)	0.0449
C(22)	0.3865 (2)	0.2589 (5)	0.4819 (4)	0.0686
C(23)	0.3222 (3)	0.2501 (6)	0.3930 (5)	0.0822
C(24)	0.3198 (3)	0.1717 (6)	0.2975 (5)	0.0866
C(25)	0.3824 (4)	0.0958 (6)	0.2820 (4)	0.0806
C(26)	0.4508 (3)	0.0996 (4)	0.3715 (4)	0.0656
C(41)	0.5470 (3)	-0.0685 (4)	0.8152 (4)	0.0595
C(51)	0.5614 (2)	0.1923 (3)	0.9114 (3)	0.0418
C(52)	0.6364 (2)	0.2302 (4)	0.8943 (4)	0.0593
C(53)	0.6930 (2)	0.2431 (5)	0.9967 (4)	0.0708
C(54)	0.6755 (3)	0.2203 (4)	1.1156 (4)	0.0653
C(55)	0.6014 (3)	0.1849 (5)	1.1347 (3)	0.0659
C(56)	0.5449 (2)	0.1702 (4)	1.0324 (3)	0.0579
C(61)	0.4178 (2)	0.1884 (4)	0.8343 (3)	0.0429
C(62)	0.3921 (2)	0.3101 (4)	0.8533 (4)	0.0536
C(63)	0.3192 (2)	0.3330 (4)	0.8924 (4)	0.0655
C(64)	0.2716 (2)	0.2306 (6)	0.9129 (4)	0.0739
C(65)	0.2956 (2)	0.1107 (5)	0.8930 (5)	0.0710
C(66)	0.3692 (2)	0.0871 (4)	0.8543 (4)	0.0580
Second molecule				
P(2)	1.05104 (5)	0.4438 (1)	1.06664 (7)	0.0427
O(1)	1.0189 (1)	0.5127 (2)	1.1842 (2)	0.0422
O(3)	1.0729 (1)	0.3113 (3)	1.1338 (2)	0.0512
B(1)	1.1344 (3)	0.5255 (6)	0.9976 (4)	0.0629
C(4)	1.0205 (2)	0.2922 (3)	1.2299 (3)	0.0465
C(5)	1.0106 (2)	0.4249 (3)	1.2884 (3)	0.0431
C(21)	0.9661 (2)	0.4165 (3)	0.9532 (3)	0.0392
C(22)	0.8953 (2)	0.4752 (4)	0.9667 (3)	0.0592
C(23)	0.8329 (2)	0.4556 (6)	0.8749 (4)	0.0723
C(24)	0.8406 (3)	0.3813 (5)	0.7743 (4)	0.0707
C(25)	0.9110 (3)	0.3219 (5)	0.7605 (5)	0.0701
C(26)	0.9732 (2)	0.3422 (4)	0.8516 (4)	0.0598
C(41)	1.0563 (3)	0.1865 (4)	1.3170 (3)	0.0583
C(51)	1.0748 (2)	0.4571 (3)	1.3953 (3)	0.0450
C(52)	1.1460 (2)	0.5047 (4)	1.3695 (4)	0.0616
C(53)	1.2049 (3)	0.5282 (5)	1.4673 (5)	0.0730
C(54)	1.1930 (3)	0.5045 (5)	1.5889 (5)	0.0741
C(55)	1.1224 (3)	0.4587 (5)	1.6165 (4)	0.0745
C(56)	1.0634 (2)	0.4354 (4)	1.5192 (3)	0.0606
C(61)	0.9297 (2)	0.4468 (4)	1.3289 (3)	0.0429
C(62)	0.9045 (2)	0.5704 (4)	1.3429 (3)	0.0525
C(63)	0.8328 (2)	0.5956 (4)	1.3854 (4)	0.0614
C(64)	0.7866 (2)	0.4963 (5)	1.4162 (4)	0.0638
C(65)	0.8101 (3)	0.3732 (5)	1.4012 (4)	0.0654
C(66)	0.8822 (2)	0.3473 (4)	1.3583 (4)	0.0580

at the P atom is *R*. Torsion angles ( $^\circ$ ) calculated for P(2), C(4) and C(5) environments (see below) confirm this result.



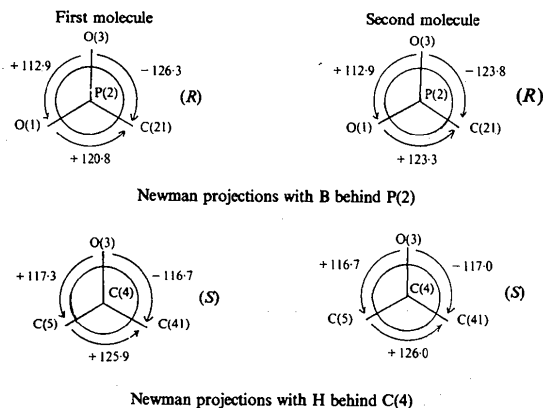
Newman projection with H(4) behind C(4) and H(5) behind C(5)

Another point is the oxazaphospholidine ring conformation. From the torsion angles, it can be seen that the conformation is neither symmetrical twist nor symmetrical envelope. The ring is very distorted, with the following distances (Å) of atoms from the



least-squares mean plane: O(1) -0.017, P(2) -0.098, N(3) 0.197, C(4) -0.213 and C(5) 0.131. The best calculated mean plane corresponds to O(1), P(2), N(3) and C(5) atoms, with C(4) out of the plane; distances (Å) from this four-atom mean plane are: O(1) 0.035, P(2) -0.030, N(3) 0.019, C(5) -0.024 and C(4) -0.501. The C(4) out-of-plane position results from the semi-pyramidal configuration of N(3); the sum of the bond angles around N(3) is 348°. The deviation of C(4) from the plane leads the C(4)-C(41) bond to be almost equatorial to this plane (107.2° with the normal to the plane). The conformation of our 1,3,2-oxazaphospholidine ring is very similar to that observed for C<sub>10</sub>H<sub>13</sub>CINOPS (Bartczak, Galdecki & Rutowska, 1983) where substituents on the P atom are Cl and S atoms instead of phenyl and borane groups.

Compound (2): The asymmetric unit consists of two C<sub>21</sub>H<sub>22</sub>O<sub>2</sub>PB molecules; one of them is shown in Fig. 2. The absolute configuration of the P atom has been determined as for (1). In comparison with the known *S* configuration of the C(4) atom, Fig. 2 shows an *S* absolute configuration for P(2). This result is confirmed by calculated torsion angles (°) of P(2) and C(4) environments for both molecules of asymmetric unit.



Concerning the oxazaphospholidine ring conformation, torsion angles show that it is neither a

Table 4. Main interatomic distances (Å) and bond angles (°) for compound (2) with e.s.d.'s in parentheses

	First molecule	Second molecule
P(2)—O(1)	1.623 (2)	1.604 (2)
P(2)—O(3)	1.558 (3)	1.592 (3)
P(2)—C(21)	1.799 (3)	1.813 (3)
P(2)—B(1)	1.912 (4)	1.897 (5)
O(1)—C(5)	1.467 (4)	1.466 (4)
C(5)—C(4)	1.559 (5)	1.546 (5)
C(4)—O(3)	1.460 (4)	1.460 (4)
O(3)—P(2)—O(1)	98.2 (1)	97.3 (1)
C(21)—P(2)—O(1)	104.9 (1)	105.6 (1)
C(21)—P(2)—O(3)	109.0 (2)	107.0 (1)
B(1)—P(2)—O(1)	115.0 (2)	116.6 (2)
B(1)—P(2)—O(3)	114.9 (2)	114.9 (2)
B(1)—P(2)—C(21)	113.5 (2)	113.7 (2)
C(5)—O(1)—P(2)	110.9 (2)	112.6 (2)
C(4)—C(5)—O(1)	104.6 (2)	103.6 (2)
O(3)—C(4)—C(5)	105.5 (3)	105.5 (3)
C(4)—O(3)—P(2)	108.0 (2)	107.7 (2)

symmetrical twist nor a symmetrical envelope for both molecules of the asymmetric unit.



As for (1), least-squares mean planes have been calculated. For both molecules of the asymmetric unit, the five-membered ring is very distorted, with the following distances (Å) to the mean plane:

First molecule: O(1) -0.03; P(2) 0.145; O(3) -0.225; C(4) 0.200; C(5) -0.087.

Second molecule: O(1) -0.00; P(2) 0.119; O(3) -0.217; C(4) 0.219; C(5) -0.120.

The best four-atom mean plane consists of P(2) O(1) C(4) C(5), with O(3) out of the plane. The calculated distances (Å) to this mean plane are:

First molecule: O(1) -0.033; P(2) 0.019; C(4) -0.020; C(5) 0.033; O(3) 0.515.

Second molecule: O(1) -0.066; P(2) 0.039; C(4) -0.039; C(5) 0.066; O(3) 0.508.

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