

listed in Table 1,* bond lengths and angles are in Table 2. The thermal ellipsoids drawing (*SHELXTL*; Sheldrick, 1986) of the molecule with the atom-labelling scheme is given in Fig. 1.

Related literature. Similar densely oxygenated cyclopentane rings have been prepared by treatment of an acyclic carbohydrate template with SmI₂ (Enholm & Trivellas, 1989). These highly functionalized rings have also been applied to natural products synthesis (Enholm, Satici & Trivellas, 1989).

* Tables of the crystallographic data, anisotropic thermal parameters, H-atom positional parameters and bond lengths and angles, and the structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54543 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0530]

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Structures of a Pair of Diastereomeric Salts of Ephedrine and Bromo-Substituted Cyclic Phosphoric Acid

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Abstract. (1) (*R*)-(+)-4-*ortho*-Bromophenyl-2-hydroxy-5,5-dimethyl-1,3,2-dioxaphosphorinane 2-oxide (1*R*,2*S*)-(–)- α -[(1-methylamino)ethyl]benzyl alcohol, (–)C₁₀H₁₆NO⁺·(+)-C₁₁H₁₃BrO₄P[–], *M_r* = 486.341, monoclinic, *P*2₁, *a* = 13.3540 (3), *b* = 8.1671 (2), *c* = 10.4791 (3) Å, β = 91.331 (7)°, *V* = 1142.58 (5) Å³, *Z* = 2, *D_x* = 1.414 Mg m^{–3}, Cu *K* α radiation (graphite monochromator), λ = 1.54184 Å, μ (Cu *K* α) = 33.910 cm^{–1}, *F*(000) = 504, *T* = 293 K, final *R* = 0.045, *wR* = 0.054, for 4155 observed unique reflections and 237 variables. (2) (*S*)-(–)-4-*ortho*-Bromophenyl-2-hydroxy-5,5-dimethyl-1,3,2-

dioxaphosphorinane 2-oxide (1*R*,2*S*)-(–)- α -[(1-methylamino)ethyl]benzyl alcohol dihydrate, (–)-C₁₀H₁₆NO⁺·(–)C₁₁H₁₃BrO₄P[–]·2H₂O, *M_r* = 522.372, monoclinic, *C*2, *a* = 20.9469 (9), *b* = 7.7456 (3), *c* = 15.7571 (5) Å, β = 102.013 (5)°, *V* = 2500.5 (2) Å³, *Z* = 4, *D_x* = 1.388 Mg m^{–3}, Cu *K* α radiation (graphite monochromator), λ = 1.54184 Å, μ (Cu *K* α) = 31.931 cm^{–1}, *F*(000) = 1088, *T* = 293 K, final *R* = 0.052, *wR* = 0.063, for 4253 observed unique reflections and 276 variables. For both (1) and (2), the phosphorinane ring is in the usual chair conformation and ephedrine is in the usual extended form. The crystal packing of (1) can be regarded as a chain parallel to the *b* axis of phosphorinane cations

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Table 1. *Crystal and experimental data*

	(1)	(2)
Crystal dimensions (mm)	0.50 × 0.31 × 0.29	0.46 × 0.33 × 0.21
No. of reflections to determine lattice parameters	25	25
θ range (°)	39.9–46.3	31.5–42.7
Max. (sin θ/λ) (Å ⁻¹)	0.6089	0.6090
hkl range	-16 → 16, -9 → 9, -12 → 12	-25 → 25, -8 → 8, -19 → 19
No. of standard reflections	3	3
Drift correction range	0.9991–1.0828	1.0039–1.1509
Empirical absorption correction range	0.970–1.062	0.9158–1.0535*
Total data measured	8479	9311
Unique data (including Friedel pairs)	4295	4687
$R_{int} = \sum(I - \langle I \rangle) / \sum(I)$	0.020	0.030
Data [$I > 3\sigma(I)$] used in the refinement	4155	4253
Parameters refined	237	276
R	0.045	0.052
wR	0.054	0.063
Weighting scheme parameter g in $w = [\sigma^2(F) + gF^2]^{-1}$	0.0003	0.001
S	3.10	4.20
Max. shift/e.s.d. in last cycle	0.031	0.038
Final difference Fourier map peaks min./max. (e Å ⁻³)	-2.12/1.79	-0.75/1.07

* Including *DIFABS* corrections.

and ephedrine anions linked by hydrogen bonds. For (2), the ions are held together by hydrogen bonds involving water molecules to form layers parallel to the *ab* plane.

Experimental. Transparent block shaped crystals were prepared from equimolar amounts of (+)-phosphorinane for (1), (-)-phosphorinane for (2), and (-)-ephedrine dissolved in acetone/water (1/1), by slow evaporation. Data sets were collected on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromatized Cu $K\alpha$ radiation using the ω - 2θ scan technique ($0 < \theta < 70^\circ$) with a scan angle of 1.5° and a variable scan rate with a maximum scan time of 30 s per reflection. Profile analysis was performed on all reflections (Lehmann & Larsen, 1974; Grant & Gabe, 1978). Normal Lorentz–polarization corrections and empirical absorption corrections using ψ scans (North, Phillips & Mathews, 1968) were applied. Data pertinent to the intensity data collection and refinement are given in Table 1.* The structure of (1) was solved using heavy-atom Patterson methods and *DIRDIF* (Beurskens, Bosman, Doesburg, Gould, van den Hark, Prick, Noordik, Beurskens, Parthasarathi, Bruins Slot & Haltiwanger, 1984). The structure of (2) was solved using vector-search methods and correlation functions (Beurskens *et al.*, 1984; Beurskens, Gould, Bruins Slot & Bosman, 1987) to orient and position the non-H skeleton of 2-hydroxy-5,5-dimethyl-4-phenyl-

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and full lists of bond lengths, angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54549 (60 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0223]

Table 2. *Atomic coordinates and equivalent isotropic thermal parameters of non-H atoms*

U_{eq} is defined as one third of the trace of the orthogonalized U_j tensor. The first lines give values for (1), the second lines for (2).

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}/U_{iso}
Br(1)	0.19900 (4)	0	0.06742 (4)	0.0748 (2)
	0.36794 (4)	-0.4046	0.41017 (5)	0.1039 (3)
P(2)	0.06110 (6)	0.45756 (14)	-0.26654 (9)	0.0497 (3)
	0.36075 (5)	0.06879 (17)	0.14384 (5)	0.0560 (3)
O(1)	0.0679 (2)	0.6005 (4)	-0.1629 (3)	0.0614 (9)
	0.43836 (13)	0.0776 (5)	0.15598 (17)	0.0672 (9)
O(3)	0.16686 (17)	0.3660 (4)	-0.2448 (2)	0.0507 (8)
	0.35072 (12)	0.0649 (4)	0.24158 (15)	0.0550 (8)
O(21)	0.0630 (2)	0.5353 (4)	-0.3953 (3)	0.0588 (9)
	0.33339 (18)	0.2329 (5)	0.1040 (2)	0.0719 (11)
O(22)	-0.0222 (2)	0.3434 (4)	-0.2378 (3)	0.0603 (9)
	0.33633 (18)	-0.0963 (5)	0.1001 (2)	0.0684 (11)
C(4)	0.1900 (2)	0.3144 (5)	-0.1148 (3)	0.0458 (10)
	0.38818 (18)	-0.0582 (6)	0.3005 (2)	0.0544 (11)
C(5)	0.1992 (3)	0.4686 (6)	-0.0291 (3)	0.0555 (13)
	0.4618 (2)	-0.0271 (8)	0.3067 (3)	0.0665 (14)
C(6)	0.0956 (3)	0.5519 (6)	-0.0336 (4)	0.0621 (14)
	0.4750 (2)	-0.0450 (8)	0.2148 (3)	0.0755 (18)
C(41)	0.28604 (15)	0.2133 (3)	-0.1197 (3)	0.0498 (11)
	0.36317 (15)	-0.0367 (4)	0.38583 (15)	0.0560 (11)
C(42)	0.3014 (2)	0.0818 (3)	-0.0366 (2)	0.0592 (14)
	0.35418 (15)	-0.1764 (4)	0.43802 (15)	0.0674 (14)
C(43)	0.3914 (2)	-0.0050 (3)	-0.0377 (2)	0.0737 (14)
	0.33152 (15)	-0.1488 (4)	0.51413 (15)	0.0837 (19)
C(44)	0.4659 (2)	0.0397 (3)	-0.1220 (2)	0.085 (2)
	0.31786 (15)	0.0184 (4)	0.53805 (15)	0.084 (2)
C(45)	0.4505 (2)	0.1712 (3)	-0.2052 (2)	0.0812 (18)
	0.32685 (15)	0.1580 (4)	0.48586 (15)	0.0802 (19)
C(46)	0.3606 (2)	0.2580 (3)	-0.2040 (2)	0.0618 (13)
	0.34951 (15)	0.1305 (4)	0.40975 (15)	0.0669 (16)
C(51)	0.2209 (4)	0.4221 (7)	0.1126 (4)	0.0728 (16)
	0.4842 (3)	0.1484 (10)	0.3435 (4)	0.092 (2)
C(52)	0.2808 (4)	0.5864 (7)	0.0720 (5)	0.0725 (17)
	0.5011 (3)	-0.1727 (12)	0.3607 (4)	0.099 (3)
O(10)	0.1965 (2)	-0.1585 (5)	0.3756 (3)	0.0662 (10)
	0.26677 (16)	-0.3751 (5)	0.1384 (3)	0.0746 (11)
N(2)	0.0648 (2)	-0.1030 (4)	0.5780 (3)	0.0528 (10)
	0.20307 (18)	-0.6783 (5)	0.0727 (2)	0.0585 (10)
C(1)	0.1904 (2)	-0.0005 (6)	0.4298 (3)	0.0530 (10)
	0.1980 (2)	-0.3790 (6)	0.1230 (3)	0.0611 (14)
C(2)	0.1648 (3)	-0.0180 (6)	0.5728 (3)	0.0532 (10)
	0.1787 (2)	-0.5659 (6)	0.1367 (3)	0.0648 (16)
C(3)	0.2424 (3)	-0.1173 (7)	0.6486 (4)	0.0700 (14)
	0.2057 (4)	-0.6316 (8)	0.2281 (3)	0.093 (3)
C(11)	0.28399 (19)	0.1003 (4)	0.4152 (3)	0.0659 (16)
	0.17005 (14)	-0.2586 (4)	0.18191 (18)	0.0615 (12)
C(12)	0.3712 (2)	0.0311 (4)	0.3681 (3)	0.096 (2)
	0.20941 (14)	-0.1946 (4)	0.25783 (18)	0.0680 (16)
C(13)	0.4568 (2)	0.1269 (4)	0.3550 (3)	0.148 (5)
	0.18291 (14)	-0.0857 (4)	0.31199 (18)	0.0804 (19)
C(14)	0.4552 (2)	0.2920 (4)	0.3891 (3)	0.132 (4)
	0.11703 (14)	-0.0407 (4)	0.29023 (18)	0.095 (2)
C(15)	0.3680 (2)	0.3612 (4)	0.4362 (3)	0.117 (3)
	0.07767 (14)	-0.1046 (4)	0.21431 (18)	0.096 (3)
C(16)	0.2824 (2)	0.2654 (4)	0.4492 (3)	0.0794 (19)
	0.10417 (14)	-0.2136 (4)	0.16014 (18)	0.0843 (19)
C(20)	0.0099 (3)	-0.0642 (6)	0.6979 (4)	0.0673 (14)
	0.1781 (3)	-0.8568 (7)	0.0676 (4)	0.0829 (19)
O(101)	-	-	-	-
	0.4122 (4)	0.5391 (13)	0.1370 (6)	0.173 (3)
O(102)	-	-	-	-
	0	-0.474 (3)	0	0.226 (7)
O(103)	-	-	-	-
	$\frac{1}{2}$	-0.334 (3)	0	0.258 (8)

1,3,2-dioxaphosphorinane 2-oxide as found earlier (Smits, Beurskens, Parthasarathi, Rijk, Kok & Wynberg, 1987). Full-matrix least-squares refinement based on F was carried out using *SHELX* (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms, except for O(101), O(102) and O(103) in (2), and isotropic H atoms in calculated positions. H atoms connected to O(10) in (1) and connected to O(101), O(102) and O(103) in (2) could neither be

Table 3. Selected bond lengths (Å), bond angles (°) and torsion angles (°) for non-H atoms

	(1)	(2)
Br(1)—C(42)	1.890 (3)	1.858 (3)
P(2)—O(1)	1.596 (3)	1.598 (3)
P(2)—O(3)	1.610 (3)	1.597 (2)
P(2)—O(21)	1.492 (3)	1.479 (4)
P(2)—O(22)	1.488 (3)	1.492 (4)
O(1)—C(6)	1.452 (5)	1.433 (6)
O(3)—C(4)	1.452 (4)	1.443 (5)
C(4)—C(5)	1.550 (6)	1.544 (6)
C(4)—C(41)	1.527 (4)	1.550 (4)
C(5)—C(6)	1.541 (6)	1.536 (6)
C(5)—C(51)	1.553 (5)	1.514 (10)
C(5)—C(52)	1.529 (7)	1.543 (10)
O(10)—C(1)	1.413 (6)	1.410 (6)
N(2)—C(2)	1.507 (5)	1.500 (6)
N(2)—C(20)	1.503 (5)	1.474 (7)
C(1)—C(2)	1.551 (4)	1.530 (7)
C(1)—C(11)	1.507 (4)	1.517 (5)
C(2)—C(3)	1.525 (6)	1.521 (7)
O(1)—P(2)—O(3)	101.99 (15)	102.71 (14)
O(1)—P(2)—O(21)	107.60 (18)	108.0 (2)
O(1)—P(2)—O(22)	110.59 (17)	109.5 (2)
O(3)—P(2)—O(21)	107.00 (14)	107.95 (18)
O(3)—P(2)—O(22)	109.75 (17)	109.15 (18)
O(21)—P(2)—O(22)	118.58 (17)	118.4 (2)
F(2)—O(1)—C(6)	116.4 (3)	116.3 (3)
P(2)—O(3)—C(4)	115.75 (19)	118.0 (2)
O(3)—C(4)—C(5)	108.7 (3)	109.9 (3)
O(3)—C(4)—C(41)	106.6 (2)	104.5 (3)
C(5)—C(4)—C(41)	113.8 (3)	116.2 (3)
C(4)—C(5)—C(6)	106.4 (3)	107.4 (3)
C(4)—C(5)—C(51)	111.5 (4)	113.0 (4)
C(4)—C(5)—C(52)	112.9 (3)	109.4 (5)
C(6)—C(5)—C(51)	106.5 (3)	109.9 (5)
C(6)—C(5)—C(52)	111.0 (4)	105.9 (4)
C(51)—C(5)—C(52)	108.5 (4)	111.0 (5)
O(1)—C(6)—C(5)	111.0 (3)	111.9 (4)
C(4)—C(41)—C(42)	120.4 (2)	122.6 (3)
C(4)—C(41)—C(46)	119.5 (2)	117.3 (3)
Br(1)—C(42)—C(41)	122.29 (19)	123.5 (2)
Br(1)—C(42)—C(43)	117.38 (17)	116.5 (2)
C(2)—N(2)—C(20)	112.5 (3)	114.5 (4)
O(10)—C(1)—C(2)	108.7 (4)	106.6 (4)
O(10)—C(1)—C(11)	113.7 (3)	113.1 (4)
C(2)—C(1)—C(11)	110.5 (3)	110.4 (3)
N(2)—C(2)—C(1)	107.0 (3)	108.5 (4)
N(2)—C(2)—C(3)	109.2 (4)	109.3 (4)
C(1)—C(2)—C(3)	112.9 (3)	113.1 (4)
C(1)—C(11)—C(12)	121.0 (3)	120.6 (3)
C(1)—C(11)—C(16)	119.0 (3)	119.4 (3)
C(6)—O(1)—P(2)—O(3)	-50.7 (3)	49.0 (4)
C(6)—O(1)—P(2)—O(21)	-163.1 (3)	162.9 (3)
C(6)—O(1)—P(2)—O(22)	66.0 (3)	-66.9 (3)
C(4)—O(3)—P(2)—O(1)	53.5 (3)	-49.9 (3)
C(4)—O(3)—P(2)—O(21)	166.4 (3)	-163.8 (3)
C(4)—O(3)—P(2)—O(22)	-63.8 (3)	66.3 (3)
C(5)—C(4)—O(3)—P(2)	-63.8 (3)	58.7 (4)
C(41)—C(4)—O(3)—P(2)	173.2 (2)	-176.0 (2)
O(3)—C(4)—C(5)—C(6)	62.6 (3)	-58.5 (5)
O(3)—C(4)—C(5)—C(51)	178.3 (3)	62.8 (5)
O(3)—C(4)—C(5)—C(52)	-59.4 (4)	-173.1 (4)
C(41)—C(4)—C(5)—C(6)	-178.8 (3)	-176.9 (4)
C(41)—C(4)—C(5)—C(51)	-63.1 (4)	-55.5 (6)
C(41)—C(4)—C(5)—C(52)	59.2 (4)	68.6 (6)
O(3)—C(4)—C(41)—C(42)	-146.5 (3)	141.1 (3)
O(3)—C(4)—C(41)—C(46)	36.4 (4)	-38.9 (4)
C(5)—C(4)—C(41)—C(42)	93.7 (3)	-97.7 (5)
C(5)—C(4)—C(41)—C(46)	-83.3 (3)	82.3 (5)
C(4)—C(5)—C(6)—O(1)	-61.0 (4)	60.0 (6)
C(51)—C(5)—C(6)—O(1)	-180.0 (12)	-63.3 (5)
C(52)—C(5)—C(6)—O(1)	62.2 (5)	176.8 (4)
C(5)—C(6)—O(1)—P(2)	59.5 (4)	-59.1 (5)
C(4)—C(41)—C(42)—Br(1)	9.7 (4)	-2.3 (4)
C(4)—C(41)—C(42)—C(43)	-177.0 (2)	-180.0 (11)
C(46)—C(41)—C(42)—Br(1)	-173.27 (19)	177.7 (2)
C(4)—C(41)—C(46)—C(45)	177.1 (2)	180.0 (18)
Br(1)—C(42)—C(43)—C(44)	173.58 (18)	-177.8 (2)
O(10)—C(1)—C(2)—N(2)	-60.9 (4)	-61.0 (5)
O(10)—C(1)—C(2)—C(3)	59.2 (4)	60.5 (6)
C(11)—C(1)—C(2)—N(2)	173.7 (3)	175.8 (3)
C(11)—C(1)—C(2)—C(3)	-66.2 (5)	-62.7 (5)
O(10)—C(1)—C(11)—C(12)	-8.4 (4)	-17.6 (5)
O(10)—C(1)—C(11)—C(16)	171.4 (3)	163.1 (3)

Table 3 (cont.)

	(1)	(2)
C(2)—C(1)—C(11)—C(12)	114.2 (4)	101.7 (4)
C(2)—C(1)—C(11)—C(16)	-66.1 (4)	-77.5 (4)
C(1)—C(2)—N(2)—C(20)	-154.7 (3)	-169.6 (4)
C(3)—C(2)—N(2)—C(20)	82.7 (4)	66.6 (5)
C(1)—C(11)—C(12)—C(13)	179.8 (3)	-179.2 (3)
C(1)—C(11)—C(16)—C(15)	-179.8 (3)	179.2 (3)

located nor calculated at reasonable positions. Although the existence of water O atoms in (2) cannot be confirmed by an independent analysis, peaks in several difference Fourier syntheses, absence of bad Van der Waals contacts and the environment in which the crystals were grown, indicate that water molecules are included. Absolute configuration was determined using *BIJVOET* (Beurskens, Noordik & Beurskens, 1980), $B = 1.000$ (1) for (1) and $B = -0.9798$ (2) for (2), calculated using 200 Friedel pairs. Therefore, structure (2) was inverted. No further empirical absorption corrections were applied to (1). For (2), an additional empirical absorption correction was applied using *DIFABS* (Walker & Stuart, 1983). Positional parameters and temperature factors are given in Table 2. Selected bond lengths, bond angles and torsion angles are given in Table 3. Scattering factors as in *SHELX* (Sheldrick, 1976). Geometry calculations and drawings of the molecules (Fig. 1) and molecular packing (Fig. 2) were obtained using the *EUCLID* package (Spek, 1982). Calculations were performed on NAS9160, VAX3800 and MicroVAX II computers.

Related literature. These crystal structure determinations were undertaken as part of an ongoing research project to gain insight into the processes involved in the resolution of enantiomers *via* diastereomeric salt formation. The synthesis of the chiral acidic resolving phosphorinane agent is described by van der Haest, Wynberg, Leusen & Bruggink (1990). Extending the nomenclature of previous crystal structure determinations of related compounds {(*R*)-(+)-2-hydroxy-5,5-dimethyl-2-oxo-4-phenyl-1,3,2-dioxaphosphorinane (1*R*,2*S*)-(-)- α -[(1-methylamino)ethyl] benzyl alcohol [INAM] (Kok, Wynberg, Smits, Beurskens & Parthasarathi, 1987), (*R*)-(+)-2-hydroxy-5,5-dimethyl-2-oxo-4-phenyl-1,3,2-dioxaphosphorinane (1*S*,2*R*)-(-)- α -[(1-methylamino)ethyl]benzyl alcohol [INAP] (Smits, Beurskens, Kok & Wynberg, 1987), (*R*)-(+)-4-*ortho*-chlorophenyl-2-hydroxy-5,5-dimethyl-2-oxo-1,3,2-dioxaphosphorinane (1*R*,2*S*)-(-)- α -[(1-methylamino)ethyl]benzyl alcohol [CLINAM] (Smits, Beurskens, Parthasarathi, Rijk, Kok & Wynberg, 1987), and (*R*)-(+)-4-*ortho*-chlorophenyl-2-hydroxy-5,5-dimethyl-2-oxo-1,3,2-dioxaphosphorinane (1*S*,2*R*)-(-)- α -[(1-methylamino)ethyl]benzyl alcohol [CLINAP] (Kok, Wijnberg, Parthasarathi, Smits &

Beurskens, 1987)} salt (1) is called BRINAM, and salt (2) is called BRINAP. The resolving power of the phosphorinane agents in these compounds is described by van der Haest *et al.* (1990). A detailed description of the crystal packing, to identify interactions which determine the differences in resolution efficiency of the resolving agent for a pair of diastereomeric salts, has been published (Leusen, Bruins Slot, Noordik, van der Haest, Wynberg & Bruggink, 1991). A second paper on this subject will be published shortly by the same authors.

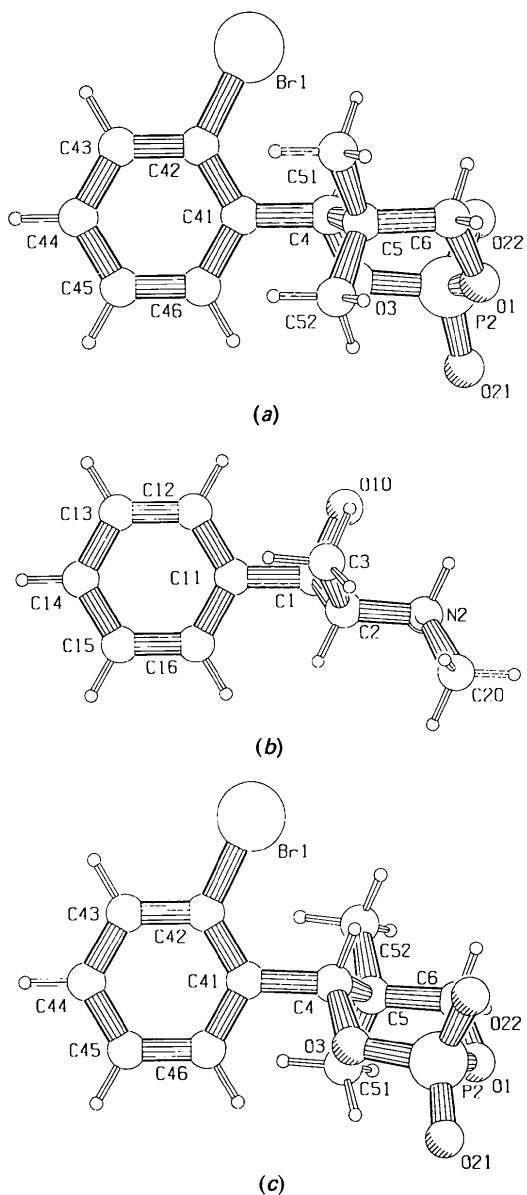


Fig. 1. Perspective drawing of (a) the (+)-phosphorinane moiety of (1), (b) ephedrine, and (c) the (-)-phosphorinane moiety of (2), showing the atomic numbering of non-H atoms.

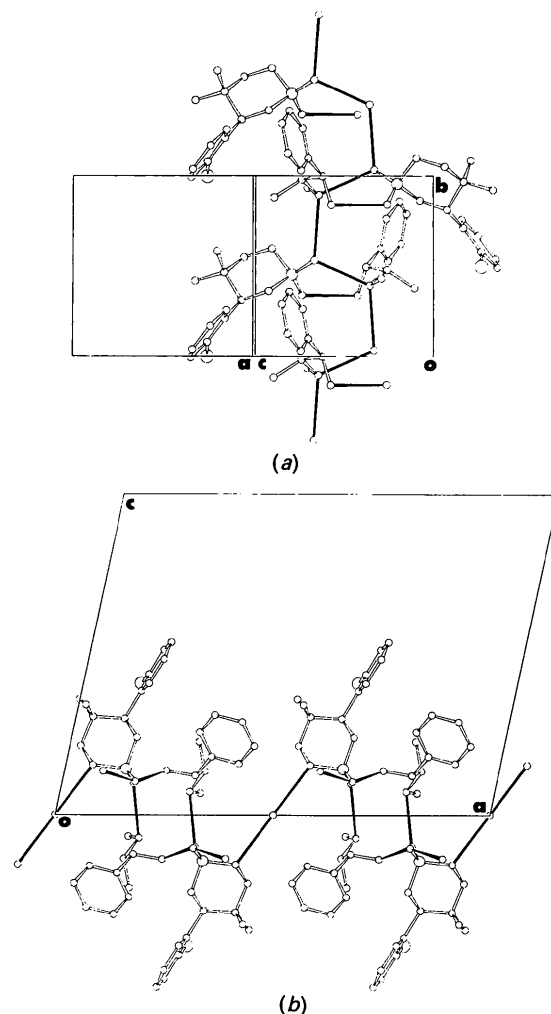


Fig. 2. Molecular packing, showing (a) the hydrogen-bonding chains along the *b* axis of (1), and (b) the two-dimensional hydrogen-bonding pattern parallel to the *ab* plane of (2).

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5-Methoxy-2-(2-methoxyphenyl)-4H-1-benzopyran-4-one (2,5'-Dimethoxyflavone)

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Abstract. C₁₇H₁₄O₄, *M_r* = 282.30, monoclinic, *P*2₁/*c*, *a* = 7.402 (1), *b* = 12.466 (2), *c* = 14.619 (2) Å, β = 97.25 (1)°, *V* = 1338.2 (3) Å³, *Z* = 4, *D_x* = 1.40 g cm⁻³, Cu *K*α, λ = 1.54178 Å, μ = 8.34 cm⁻¹, *F*(000) = 592, *T* = 291 K, *R* = 0.042 for 1684 observed reflections. The dihedral angle between the benzopyran and the phenyl ring mean planes is 10.5 (5)°.

Experimental. Crystals obtained by evaporation from ethanol. *D_m* not measured. Parallelepiped crystal with approximate dimensions 0.24 × 0.16 × 0.07 mm. Lattice parameters refined using 30 reflections in the range 3 ≤ 2θ ≤ 50°. Huber four-circle diffractometer, graphite-monochromatized Cu *K*α radiation, θ–2θ-scan technique. 2406 *hk ± l* measured reflections with sinθ/λ ≤ 0.60 Å⁻¹; 0 ≤ *h* ≤ 8, 0 ≤ *k* ≤ 14, –17 ≤ *l* ≤ 16; 1684 with *I* ≥ 2.5σ(*I*). Standard reflection (045) checked every 50 reflections, no significant deviation. Structure solved by *SHELXS86* (Sheldrick, 1985). H atoms from Fourier difference synthesis. Anisotropic least-squares refinement (*SHELX76*; Sheldrick, 1976) using *F*; H isotropic with common refined temperature factor. *w* = 1/(σ² + 0.00646*F*²), *R* = 0.042, *wR* = 0.052 for 1684

observed reflections. Final maximum shift/e.s.d. = 0.04. *S* = 0.80. Maximum and minimum heights in final difference Fourier synthesis = 0.14 and –0.20 e Å⁻³. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). The atomic parameters are given in Table 1.* Fig. 1 is a stereoscopic view of the molecule, showing the numbering of the atoms (*PLUTO*; Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2.

Related literature. This work forms part of our structural investigations of methoxyflavones (Wallet, Gaydou, Tinant, Declercq, Baldy & Bonifassi, 1990; Wallet, Gaydou, Feneau-Dupont, Tinant, Declercq & Baldy, 1991). Some closely related structures have recently been described (Wallet, Gaydou, Jaud & Baldy, 1990). In 2'-methoxyflavone the torsion angle

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54489 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0073]