Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Hg1	0.0000	0.0000	0.0000	0.044 (1)
Mn1	0.8145 (1)	0.1510 (1)	0.8800 (1)	0.040 (1)
C1	0.6810 (8)	0.2585 (10)	0.7969 (3)	0.058 (3)
O1	0.5967 (8)	0.3256 (9)	0.7458 (3)	0.086 (3)
C2	0.7599 (9)	0.3781 (10)	0.9364 (3)	0.056 (3)
O2	0.7271 (9)	0.5218 (7)	0.9720 (3)	0.085 (4)
C3	1.0673 (8)	0.2717 (9)	0.8676 (3)	0.054 (3)
O3	1.2240 (6)	0.3474 (8)	0.8584 (3)	0.076 (3)
C4	0.8944 (9)	−0.1007 (11)	0.8390 (3)	0.058 (4)
O4	0.9455 (8)	−0.2528 (8)	0.8135 (3)	0.083 (3)
C5	0.5827 (8)	0.0083 (9)	0.9062 (3)	0.049 (3)
O5	0.4400 (7)	−0.0809 (8)	0.9212 (3)	0.066 (3)

Table 2. *Geometric parameters* (Å, °)

Hg1—Mn1	2.614 (1)	Mn1—C3	1.837 (5)
Mn1—C1	1.829 (6)	Mn1—C4	1.853 (6)
Mn1—C2	1.830 (6)	Mn1—C5	1.855 (5)
Mn1—Hg1—Mn1*a*	180.0	Hg1—Mn1—C3	85.6 (2)
Hg1—Mn1—C1	179.0 (2)	Hg1—Mn1—C4	85.6 (2)
Hg1—Mn1—C2	83.4 (2)	Hg1—Mn1—C5	84.8 (2)

V = 755.4(2) Å^3
Z = 2
D_x = 2.596 Mg m^{-3}
Prism
0.21 × 0.18 × 0.15 mm
Yellow

Data collection

Siemens *R*3*m*/V diffractometer
ω–2θ scans
Absorption correction: empirical *via* ψ scans
T_{min} = 0.14, T_{max} = 0.41
3934 measured reflections
1749 independent reflections
1491 observed reflections [$F>4\sigma(F)$]
R_{int} = 0.019
θ_{max} = 27.5°
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 25$
3 standard reflections monitored every 400 reflections
intensity variation: 0%

Refinement

Refinement on F
Final R = 0.027
wR = 0.029
S = 1.543
1491 reflections
107 parameters
Calculated weights $w=1/[\Sigma^2(F)+0.0001F^2]$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.85 e Å^{-3}
$\Delta\rho_{min}$ = −0.88 e Å^{-3}
Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1990)
Data collection, all calculations and graphics: *SHELXTL-Plus*.

The structure was refined by full-matrix least squares with *SHELXTL-Plus*. All atoms were assigned anisotropic displacement parameters in the refinement.

Lists of structure factors, anisotropic thermal parameters and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55174 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1007]

0108-2701/92/091710-03$06.00

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trans-1,3-Bis(diphenylphosphinoyl)cyclohexane

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Abstract

The relative configuration of the two diphenylphosphinoyl groups was determined as *trans* by X-ray structure analysis. The cyclohexane ring has a chair conformation with one diphenylphosphinoyl group in an equatorial position and the other in an axial position.

Comment

As part of synthetic studies of the Lewis acid-promoted double addition reactions of secondary phosphines with α,β-unsaturated aldehydes and ketones, the title compound (1) was obtained from diphenylphosphine and 2-cyclohexenone in the presence of catalytic amounts of $NbCl_5$ and stoichiometric $BF_3.OEt_2$ (Hashimoto, Maeta, Matsumoto, Morooka, Ohba & Suzuki, 1992). Its melting point is 555-556 K; NMR and IR data are given in Hashimoto *et al.* (1992). The O1—P1—C1—H(C1) and O2—P2—C3—H(C3) torsion angles are nearly 180°. There is an imbalance in the P—C—C bond angles for the Ph groups of 4.7(2)-7.3(2)°, which may be the result of non-bonded H···H repulsions. The H atoms of C1, C8 and C18 and those of C3, C24 and C26 are arranged in

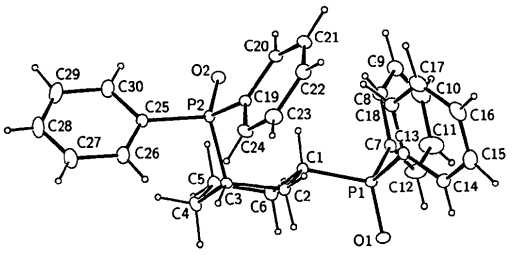
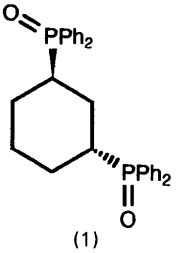


Fig. 1. The molecular structure with thermal ellipsoids at 10% probability level. H atoms are represented by circles of radius 0.1 Å.

approximately equilateral triangles with H···H distances ranging from 2.35(5) to 2.60(3) Å.



(1)

Experimental

Crystal data

$C_{30}H_{30}O_2P_2$
M_r = 484.5
Triclinic
$P\bar{1}$
a = 11.476 (1) Å
b = 13.177 (1) Å
c = 9.267 (1) Å
α = 106.19 (1)°
β = 96.38 (1)°
γ = 96.00 (1)°
V = 1323.9 (2) Å^3
Z = 2

D_x = 1.215 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 38 reflections
θ = 12-15°
μ = 0.183 mm^{-1}
T = 299 K
Prism
0.45 × 0.40 × 0.26 mm
Colourless

Data collection

Rigaku AFC-5 four-circle diffractometer
θ-2θ scans
Absorption correction: none
6361 measured reflections
6059 independent reflections
3866 observed reflections [$|F_o|>3\sigma(|F_o|)$]

R_{int} = 0.012
θ_{max} = 27.5°
h = 0 → 14
k = −17 → 16
l = −12 → 11
5 standard reflections monitored every 100 reflections
intensity variation: 1%

Refinement

Refinement on F
Final R = 0.046
wR = 0.037
S = 2.787
3866 reflections
427 parameters
All H-atom parameters refined
Calculated weights, $w=1/\sigma$

$(\Delta/\sigma)_{max}$ = 0.115
$\Delta\rho_{max}$ = 0.241 e Å^{-3}
$\Delta\rho_{min}$ = −0.296 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
P1	0.12729 (6)	0.25541 (5)	0.31258 (8)	0.0376 (3)
P2	0.30505 (6)	0.64368 (5)	0.46583 (8)	0.0386 (3)
O1	−0.0029 (2)	0.2198 (1)	0.2794 (2)	0.0495 (9)
O2	0.3919 (2)	0.5884 (1)	0.3759 (2)	0.050 (1)
C1	0.1697 (2)	0.3870 (2)	0.2926 (3)	0.036 (1)
C2	0.1244 (3)	0.4719 (2)	0.4146 (3)	0.038 (1)
C3	0.1501 (2)	0.5849 (2)	0.4006 (3)	0.039 (1)
C4	0.1077 (3)	0.5877 (3)	0.2377 (4)	0.053 (2)
C5	0.1519 (3)	0.5016 (3)	0.1176 (4)	0.056 (2)
C6	0.1195 (3)	0.3910 (2)	0.1343 (3)	0.050 (2)
C7	0.2069 (2)	0.1659 (2)	0.1878 (3)	0.041 (1)
C8	0.3276 (3)	0.1850 (3)	0.1870 (4)	0.059 (2)
C9	0.3825 (4)	0.1142 (3)	0.0874 (5)	0.077 (2)
C10	0.3192 (4)	0.0245 (3)	−0.0123 (5)	0.086 (3)
C11	0.2013 (4)	0.0041 (4)	−0.0136 (5)	0.110 (3)
C12	0.1447 (3)	0.0755 (3)	0.0858 (4)	0.083 (2)
C13	0.1861 (2)	0.2610 (2)	0.5045 (3)	0.040 (1)
C14	0.1157 (3)	0.2116 (2)	0.5844 (4)	0.057 (2)
C15	0.1587 (5)	0.2129 (3)	0.7314 (4)	0.075 (2)
C16	0.2704 (4)	0.2618 (3)	0.7962 (5)	0.077 (2)
C17	0.3408 (4)	0.3128 (3)	0.7210 (4)	0.073 (2)
C18	0.2986 (3)	0.3114 (3)	0.5740 (4)	0.058 (2)
C19	0.3326 (2)	0.6451 (2)	0.6623 (3)	0.039 (1)
C20	0.4285 (3)	0.5969 (2)	0.7041 (4)	0.049 (2)
C21	0.4550 (3)	0.5954 (3)	0.8532 (4)	0.063 (2)
C22	0.3876 (3)	0.6392 (3)	0.9585 (4)	0.070 (2)
C23	0.2907 (3)	0.6863 (3)	0.9195 (4)	0.070 (2)
C24	0.2639 (3)	0.6880 (3)	0.7706 (4)	0.057 (2)
C25	0.3076 (3)	0.7804 (2)	0.4618 (3)	0.043 (1)
C26	0.2322 (3)	0.8467 (3)	0.5301 (4)	0.070 (2)
C27	0.2341 (4)	0.9490 (3)	0.5156 (6)	0.094 (3)
C28	0.3107 (5)	0.9829 (4)	0.4305 (6)	0.104 (3)
C29	0.3858 (5)	0.9189 (4)	0.3620 (5)	0.100 (3)
C30	0.3861 (4)	0.8174 (3)	0.3788 (4)	0.072 (2)

Table 2. *Geometric parameters* (Å, °)

P1—O1	1.489 (2)	P2—O2	1.489 (2)
P1—C1	1.817 (3)	P2—C3	1.827 (3)
P1—C7	1.807 (3)	P2—C19	1.807 (3)
P1—C13	1.809 (3)	P2—C25	1.810 (3)
O1—P1—C1	113.5 (1)	P1—C1—C2	110.1 (2)
O1—P1—C7	112.0 (1)	P1—C1—C6	110.3 (2)
O1—P1—C13	111.5 (1)	P2—C3—C2	112.7 (2)
C1—P1—C7	106.2 (1)	P2—C3—C4	112.2 (2)
C1—P1—C13	106.6 (1)	P1—C7—C8	123.5 (2)
C7—P1—C13	106.6 (1)	P1—C7—C12	118.6 (2)
O2—P2—C3	115.2 (1)	P1—C13—C14	118.3 (2)
O2—P2—C19	111.6 (1)	P1—C13—C18	123.0 (2)
O2—P2—C25	112.2 (1)	P2—C19—C20	117.0 (2)
C3—P2—C19	105.3 (1)	P2—C19—C24	124.3 (2)
C3—P2—C25	104.0 (1)	P2—C25—C26	123.4 (3)
C19—P2—C25	107.9 (1)	P2—C25—C30	117.6 (2)

Program(s) used to solve structure: *Xtal*3.0 *SIMPEL, GENTAN* (Hall & Stewart, 1990). Program(s) used to refine structure: *Xtal*3.0 *CRYLSQ* (Hall & Stewart, 1990). Molecular graphics: *Xtal*3.0 *ORTEP* (Hall & Stewart, 1990). Software used to prepare material for publication: *Xtal*3.0 *BONDLA*, *CIFIO* (Hall & Stewart, 1990).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55206 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1010]

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Structure of Oxatomide Monohydrate: an Anti-Allergic Drug

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Abstract

The crystal structure of the monohydrate of oxatomide, 1-{3-[4-(diphenylmethyl)-1-piperazinyl]propyl}-1,3-dihydro-2*H*-benzimidazol-2-one, has been determined at 100 K. The oxatomide molecule adopts an extended conformation with a planar benzimidazolone fragment. The water molecule has a cohesive function, connecting three oxatomide molecules by intermolecular hydrogen bonds.

Comment

Oxatomide (1) is a potent broad-scale anti-allergic drug by virtue of its inhibition of both the release and the action of allergic mediators (Awouters *et al.*, 1977). The structure analysis of oxatomide was carried out as part of an investigation into the biologically active conformation of certain H_1-histamine-receptor agonists and antagonists (Richards, Brogden, Heel, Speight & Avery, 1984). A comparison of the crystal structure conformation with conformations obtained by molecular modelling of the active site could provide more insight into the actions of pharmaceuticals at the molecular level. The two central torsion angles in the propyl residue, N2—C8—C9—C10 and C8—C9—C10—N3, are 178.0(3) and 169.2(3)° respectively, resulting in an extended conformation rather than a folded one. The benzimidazolone moiety is planar and rotated through 78.8(3)° with respect to the almost planar C8—C9—C10—N3 chain. The piperazine ring has a chair conformation, with the substituents on both N atoms in equatorial positions. All hydrogen bonds are intermolecular. The cohesive role of water in the hydrogen-bond framework is embodied by its interaction as a donor to the O1 atom of the carbonyl group of an oxatomide residue at (x, y, z) and to N3 in the piperazine ring of a residue at ($1/2-x$, $1/2+y$, $1/2-z$), and as an acceptor of a hydrogen bond donated by N1—H of the residue at ($1/2-x$, $1/2-y$, $1-z$). The donor-acceptor distances are 2.801(3), 2.846(4) and 2.756(4) Å respectively, and the donor-hydrogen-acceptor angles are 173(5), 176(4) and 169(4)°.

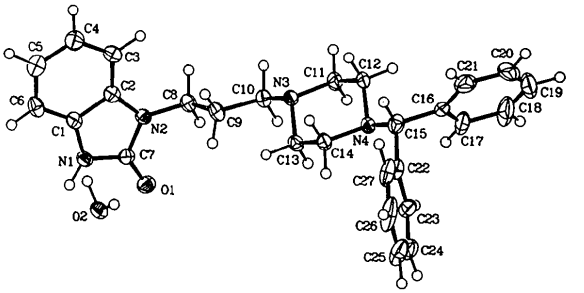
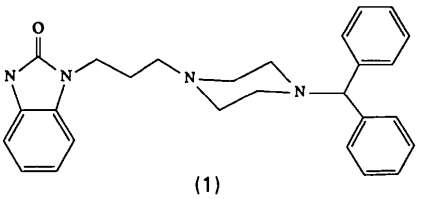


Fig. 1. View (Johnson, 1976) of oxatomide monohydrate showing the labelling of the non-H atoms. Thermal ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of arbitrary radius.



(1)

Experimental

Crystal data

$C_{27}H_{30}N_4O.H_2O$
M_r = 444.58
Monoclinic
$C2/c$
a = 31.1173 (16) Å
b = 8.9219 (6) Å
c = 19.0721 (14) Å
β = 112.853 (5)°
V = 4879.3 (6) Å^3
Z = 8
D_x = 1.210 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10.84–17.66°
μ = 0.73 mm^{-1}
T = 100 K
Platelet
0.55 × 0.20 × 0.15 mm
Colourless
Crystal source: Janssen Pharmaceutica, Beerse, Belgium

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
4793 measured reflections
4353 independent reflections
3005 observed reflections [$I>2.5\sigma(I)$]
R_{int} = 0.0409
θ_{max} = 25.37°
h = −34 → 35
k = −10 → 0
l = −22 → 0
3 standard reflections
frequency: 60 min
intensity variation: 1.2%

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