Table 2. *Bond lengths* (Å) *and bond angles* (°)

C(1)—N(2)	1.453 (4)	C(1)—C(5)	1.543 (4)
C(1)—N(8)	1.455 (4)	N(2)—C(3)	1.440 (4)
N(2)—N(2')	1.380 (4)	C(3)—N(4)	1.470 (4)
N(4)—C(5)	1.470 (4)	N(4)—N(4')	1.360 (4)
C(5)—N(6)	1.438 (3)	N(6)—C(7)	1.456 (4)
N(6)—C(6')	1.373 (4)	C(7)—N(8)	1.459 (4)
N(8)—C(8')	1.364 (4)	N(2')—O(2a)	1.214 (5)
N(2')—O(2b)	1.226 (4)	N(4')—O(4a)	1.222 (4)
N(4')—O(4b)	1.212 (4)	C(6')—O(6)	1.225 (4)
C(6')—C(9)	1.492 (4)	C(8')—O(8)	1.211 (4)
C(8')—C(10)	1.502 (5)		
N(2)—C(1)—C(5)	105.2 (2)	N(2)—C(1)—N(8)	112.5 (2)
C(5)—C(1)—N(8)	102.9 (2)	C(1)—N(2)—C(3)	113.2 (2)
C(1)—N(2)—N(2')	119.9 (2)	C(3)—N(2)—N(2')	116.5 (3)
N(2)—C(3)—N(4)	102.0 (2)	C(3)—N(4)—C(5)	114.4 (2)
C(3)—N(4)—N(4')	116.1 (2)	C(5)—N(4)—N(4')	118.5 (2)
C(1)—C(5)—N(4)	101.4 (2)	C(1)—C(5)—N(6)	104.3 (2)
N(4)—C(5)—N(6)	111.9 (2)	C(5)—N(6)—C(7)	110.0 (2)
C(5)—N(6)—C(6')	129.7 (2)	C(7)—N(6)—C(6')	119.8 (2)
N(6)—C(7)—N(8)	101.8 (2)	C(1)—N(8)—C(7)	112.8 (2)
C(1)—N(8)—C(8')	120.7 (2)	C(7)—N(8)—C(8')	125.8 (2)
N(2)—N(2')—O(2a)	117.6 (3)	N(2)—N(2')—O(2b)	115.9 (3)
O(2a)—N(2')—O(2b)	126.3 (3)	N(4)—N(4')—O(4a)	117.0 (3)
N(4)—N(4')—O(4b)	117.0 (3)	O(4a)—N(4')—O(4b)	125.9 (3)
N(6)—C(6')—O(6)	118.2 (3)	N(6)—C(6')—C(9)	119.0 (3)
O(6)—C(6')—C(9)	122.8 (3)	N(8)—C(8')—O(8)	120.5 (3)
N(8)—C(8')—C(10)	116.2 (3)	O(8)—C(8')—C(10)	123.3 (3)

Fig. 1. A thermal ellipsoid plot of the title compound with ellipsoids drawn at the 20% probability level.

discussed briefly by Archibald, Gilardi, Baum & George (1990).

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Structure of a Chiral Bicyclic β-Hydroxyphosphonamide – an Asymmetric Olefination Intermediate, C25H41N2O2P

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Abstract. (3aR,7aR)-2,3,3a,4,5,6,7,7a-Octahydro-2-[(R)-α-(1-hydroxy-4-*tert*-butylcyclohexyl)phenyl-methyl]-1,3-dimethyl-1H-1,3,2-benzodiazaphosphole 2-oxide, $C_{25}H_{41}N_2O_2P$, $M_r = 432.59$, orthorhombic, $P2_12_12_1$, $a = 10.636$ (2), $b = 12.759$ (1), $c = 18.185$ (2) Å, $V = 2467.8$ (5) Å^3, $Z = 4$, $D_x = 1.164$ Mg m^{-3}, λ(Cu $K\bar{\alpha}$) = 1.54178 Å, $\mu = 1.138$ mm^{-1}, $F(000) = 944$, $T = 293$ K, $R = 0.047$, $wR = 0.039$ for 1884 observed reflections. The title compound is constituted of a (1R,2R)-N,N'-dimethyl-1,2-diaminocyclohexane attached to a tetrahedrally coordinated phosphoryl group. A phenylmethyl group with an R configuration bridges the P atom and the 4-*tert*-butylcyclohexanol ring. The tertiary alcohol is in the axial position and the

4-*tert*-butyl is in the equatorial position. The molecules are held in the crystal through intermolecular hydrogen bonds of the O—H···O type and by van der Waals interactions.

Experimental. The reaction of the anion formed from the chiral non-racemic bicyclic phosphonamide (I) (Hanessian & Beaudoin, 1992) with 4-*tert*-butyl-cyclohexanone gave a crystalline adduct (II) in which the orientations of the substituents at the bond forming sites were elucidated by single-crystal X-ray-diffraction studies.

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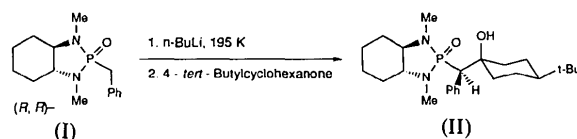
C$_{25}$H$_{41}$N$_2$O$_2$P

Crystals of C$_{25}$H$_{41}$N$_2$O$_2$P were recrystallized from an ethyl acetate–hexane solution. The crystal used for data collection had the following distances between the indicated pairs of faces: 0.08 {001}, 0.16 {01$\bar{1}$} and 0.35 mm for {101}. Accurate cell parameters were derived from least-squares refinement of the setting angles for 25 well centred reflections (41.0 ≤ 2θ ≤ 44.0°) in the counter aperture of an Enraf–Nonius CAD-4 diffractometer. The intensity data were collected with graphite-monochromated Cu K$\bar{\alpha}$ radiation using the ω-scan mode and with scan width (0.80 + 0.14tanθ)°. A set of 2460 unique reflections (2θ$_{max}$ = 140.0°, 0 ≤ h ≤ 11, 0 ≤ k ≤ 15, 0 ≤ l ≤ 22) were measured at 293 K of which 1884 [I ≥ 1.96σ(I)] were retained for structure determination and refinement. The crystal orientation was monitored every 200 measurements while the intensity of four standard reflections was checked every hour. The largest intensity fluctuation was 0.9%. An absorption correction was applied at a later stage in the refinement (Gaussian integration, grid 10 × 10 × 10, transmission coefficient range 0.79–0.92). Finally, the data were corrected for Lp.

The structure was solved by direct methods (SHELXS86; Sheldrick, 1986) and difference Fourier calculations. All non-H atoms were refined anisotropically. All H atoms were found on difference Fourier syntheses and were refined isotropically.

Block-diagonal least-squares refinement was based on F and the function minimized was $\sum w(|F_o| - |F_c|)^2$. In the final stage of the refinement, individual weights based on counting statistics {$w = 1/[\sigma^2(F) + 0.0004(F)^2]$} were applied. Convergence was reached for R = 0.047, wR = 0.039 and a goodness of fit of 1.445 for 436 parameters. The maximum (Δ/σ) was 0.47 while the average (Δ/σ) was 0.10. For all reflections R = 0.067 and wR = 0.042. On the final difference Fourier synthesis, the general background was lower than ±0.08 e Å^{-3}.

The scattering curves for the non-H atoms were taken from Cromer & Mann (1968) and those for the H atoms from Stewart, Davidson & Simpson (1965). Real and imaginary parts of the anomalous dispersion of P atoms were taken from Cromer & Liberman (1970). The set of atomic coordinates presented in Table 1* is in agreement with the absolute configuration based on the known stereochemistry of the (1R,2R)-N,N'-dimethyl-1,2-diaminocyclohexane

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* A packing diagram, lists of anisotropic temperature factors, H-atom coordinates, bond distances and angles for the cyclohexyl and phenyl groups, bond distances and angles involving H atoms, distances to least-squares planes, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54947 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD0065]

Table 1. *Final atomic coordinates with e.s.d.'s in parentheses* (P × 10^5; O, N, C × 10^4) *and equivalent isotropic tempeature factors* (Å^2 × 10^3)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U$_{eq}$
P	56485 (11)	12040 (9)	47265 (6)	42
O(1)	4516 (3)	1871 (2)	4795 (2)	52
O(2)	7098 (2)	3181 (2)	4638 (1)	43
N(1)	5350 (3)	−74 (2)	4646 (2)	44
N(2)	6584 (3)	1056 (3)	5450 (2)	42
C(1)	6286 (4)	−682 (3)	5037 (2)	48
C(2)	5960 (4)	−1798 (3)	5258 (3)	63
C(3)	7107 (5)	−2254 (4)	5661 (3)	81
C(4)	7529 (5)	−1571 (4)	6299 (3)	81
C(5)	7751 (5)	−422 (4)	6086 (2)	66
C(6)	6575 (4)	−33 (3)	5706 (2)	49
C(7)	4704 (5)	−513 (4)	4017 (3)	75
C(8)	6674 (4)	1865 (4)	6015 (2)	59
C(9)	6588 (4)	1611 (3)	3925 (2)	39
C(10)	6882 (4)	2810 (3)	3904 (2)	39
C(11)	8017 (4)	3055 (3)	3415 (2)	42
C(12)	8273 (4)	4240 (3)	3360 (2)	44
C(13)	7144 (4)	4866 (3)	3095 (2)	44
C(14)	5997 (4)	4594 (3)	3564 (2)	53
C(15)	5748 (4)	3419 (3)	3610 (2)	51
C(16)	7407 (4)	6070 (3)	3053 (2)	48
C(17)	8630 (5)	6280 (4)	2618 (3)	80
C(18)	7555 (6)	6539 (4)	3811 (3)	73
C(19)	6316 (5)	6625 (4)	2662 (3)	89
C(20)	7704 (4)	886 (3)	3822 (2)	39
C(21)	8735 (4)	889 (3)	4278 (2)	47
C(22)	9725 (4)	184 (4)	4177 (3)	62
C(23)	9692 (5)	−523 (4)	3611 (3)	74
C(24)	8676 (5)	−533 (4)	3150 (3)	74
C(25)	7687 (5)	164 (3)	3244 (2)	52

Table 2. *Selected bond distances* (Å), *bond angles* (°) *and torsion angles* (°) *with e.s.d.'s in parentheses*

P—O(1)	1.480 (3)	P—N(1)	1.668 (3)
P—N(2)	1.660 (3)	P—C(9)	1.842 (4)
N(1)—C(1)	1.449 (5)	N(1)—C(7)	1.447 (6)
N(2)—C(6)	1.465 (5)	N(2)—C(8)	1.460 (5)
C(9)—C(20)	1.517 (6)	C(9)—C(10)	1.562 (6)
C(10)—O(2)	1.435 (5)		

O(1)—P—N(1)	114.5 (2)	C(6)—N(2)—C(8)	116.6 (3)
O(1)—P—N(2)	119.1 (2)	N(1)—C(1)—C(2)	118.4 (4)
O(1)—P—C(9)	110.2 (2)	N(2)—C(6)—C(5)	116.8 (4)
N(1)—P—C(9)	108.0 (2)	C(9)—C(20)—C(21)	123.2 (4)
N(2)—P—C(9)	109.5 (2)	P—C(9)—C(10)	113.8 (3)
N(1)—P—N(2)	94.2 (2)	P—C(9)—C(20)	110.5 (3)
P—N(1)—C(1)	110.5 (3)	C(10)—C(9)—C(20)	116.0 (3)
N(1)—C(1)—C(6)	104.1 (3)	O(2)—C(10)—C(9)	109.4 (3)
C(1)—C(6)—N(2)	105.5 (3)	O(2)—C(10)—C(11)	110.3 (3)
C(6)—N(2)—P	110.8 (3)	O(2)—C(10)—C(15)	106.5 (3)
P—N(1)—C(7)	122.6 (3)	C(9)—C(10)—C(11)	111.8 (3)
C(1)—N(1)—C(7)	120.5 (3)	C(9)—C(10)—C(15)	110.4 (3)
P—N(2)—C(8)	121.1 (3)	C(9)—C(20)—C(25)	119.0 (4)

| P—N(1)—C(1)—C(6)	36.2 (4)	N(1)—P—C(9)—C(10)	177.0 (3)
N(1)—C(1)—C(6)—N(2)	−39.3 (4)	N(2)—P—C(9)—C(10)	−81.7 (3)
C(1)—C(6)—N(2)—P	27.9 (4)	N(1)—P—C(9)—C(20)	−50.5 (3)
C(6)—N(2)—P—N(1)	−6.5 (3)	N(2)—P—C(9)—C(20)	50.8 (3)
N(2)—P—N(1)—C(1)	−18.3 (3)	O(2)—C(10)—C(9)—C(20)	−93.1 (4)
O(1)—P—N(1)—C(1)	65.3 (4)	O(2)—C(10)—C(11)—C(12)	−60.9 (4)
O(1)—P—N(1)—C(1)	−143.0 (3)	O(2)—C(10)—C(15)—C(14)	62.5 (4)
O(1)—P—N(2)—C(8)	−27.5 (4)	C(1)—N(1)—P—C(9)	93.8 (3)
O(1)—P—N(2)—C(6)	114.6 (3)	C(7)—N(1)—P—C(9)	−57.9 (4)
O(1)—P—C(9)—C(10)	51.2 (3)	C(6)—N(2)—P—C(9)	−117.3 (3)
O(1)—P—C(9)—C(20)	−176.3 (3)	C(8)—N(2)—P—C(9)	100.7 (3)
P—N(1)—C(1)—C(2)	159.1 (3)	C(1)—C(6)—N(2)—C(8)	171.9 (3)
P—N(2)—C(6)—C(5)	151.7 (3)	C(2)—C(1)—N(1)—C(7)	−48.5 (5)
P—C(9)—C(10)—O(2)	36.8 (4)	C(5)—C(6)—N(2)—C(8)	−64.4 (5)
P—C(9)—C(10)—C(11)	159.3 (3)	C(6)—C(1)—N(1)—C(7)	−171.4 (4)
P—C(9)—C(10)—C(15)	−80.0 (4)	C(9)—C(10)—C(11)—C(12)	177.1 (3)
P—C(9)—C(20)—C(21)	−72.5 (5)	C(9)—C(10)—C(15)—C(14)	−178.8 (3)
P—C(9)—C(20)—C(25)	106.5 (4)	C(9)—C(20)—C(21)—C(22)	177.9 (4)
N(1)—P—N(2)—C(8)	−148.5 (3)	C(9)—C(20)—C(25)—C(24)	−177.9 (4)
N(2)—P—N(1)—C(7)	−170.0 (3)	C(10)—C(9)—C(20)—C(21)	59.0 (5)
N(1)—C(1)—C(2)—C(3)	−179.9 (4)	C(10)—C(9)—C(20)—C(25)	−122.1 (4)
N(1)—C(1)—C(6)—C(5)	−166.6 (3)	C(11)—C(10)—C(9)—C(20)	29.4 (5)
N(2)—C(6)—C(1)—C(2)	−167.2 (3)	C(15)—C(10)—C(9)—C(20)	150.1 (3)
N(2)—C(6)—C(5)—C(4)	−179.7 (4)		
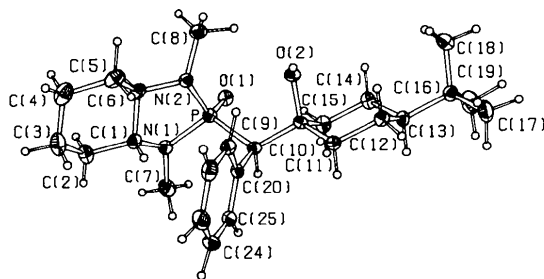
Fig. 1. An *ORTEP* (Johnson, 1965) drawing showing the atomic numbering of $C_{25}H_{41}N_2O_2P$; 40% probability level. The H atoms are represented by spheres of arbitrary size.
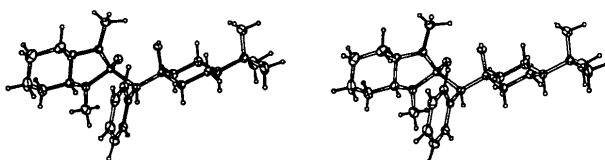

Fig. 2. Stereopair showing the molecular conformation.

moiety. For the other configuration, $R = 0.055$, $wR = 0.047$ and $S = 1.726$. Selected bond distances, bond angles and torsion angles are presented in Table 2.

The programs used here are: modified versions of *NRC*-3, Lp absorption correction; *NRC*-2, data reduction; *NRC*-10, bond distances and angles; and *NRC*-22, mean planes (Ahmed, Hall, Pippy & Huber, 1973); *SHELXS*86 (Sheldrick, 1986); *FORDAP*, Fourier and Patterson maps (A. Zalkin, unpublished); *NUCLS*, least-squares refinement (Doedens & Ibers, 1967); and *ORTEP*, Figs. 1 and 2 (Johnson, 1965).

In the crystal, the molecules are held by hydrogen bonds between O(2)—H(O2) and O(1) where O(2)—

$H(O2) = 0.79$ (3), $H(O2)\cdots O(1^a) = 1.99$ (3), $O(2)\cdots O(1^a) = 2.772$ (4) Å, $O(2)—H(O2)\cdots O(1^a) = 172$ (3)°, and $O(1^a)$ is related to $O(1)$ by $\frac{1}{2} + x$, $\frac{1}{2} - y$, $1 - z$.

Related literature. The anion generated from the phosphonamide (I) showed the same facial selectivity as observed with electrophiles in previous studies by Hanessian, Delorme Beaudoin & Leblanc (1984), Bélanger-Gariépy, Delorme, Hanessian & Brisse (1986), Bélanger-Gariépy, Bennani, Hanessian & Brisse (1989), Bennani, Bélanger-Gariépy & Hanessian (1990), Hanessian, Bennani & Delorme (1990) and Bennani & Hanessian (1991).

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Structure of a Chiral Bicyclic β-Hydroxyphosphonamide – a Product of Kinetic Resolution in an Olefination Reaction, $C_{22}H_{35}N_2O_2P$

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Abstract. $C_{22}H_{35}N_2O_2P$, $M_r = 390.51$, monoclinic, $P2_1$, $a = 9.101$ (3), $b = 8.612$ (6), $c = 13.790$ (5) Å, $\beta = 92.29$ (3)°, $V = 1080.0$ (9) Å^3, $Z = 2$, $D_x = 1.201$ Mg m^{-3}, $\lambda(\text{Mo }K\bar{\alpha}) = 0.71069$ Å, $\mu = 0.14$ mm^{-1}, $T = 293$ K, $R = 0.044$, $wR = 0.031$ for 1355 observed reflections. The title compound,

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