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

C(1)—C(2)	1·383 (3)	C(1)—C(6)	1·383 (3)
C(1)—C(7)	1·507 (3)	C(2)—C(3)	1·383 (3)
C(3)—C(4)	1·369 (3)	C(4)—C(5)	1·373 (3)
C(5)—C(6)	1·383 (3)	C(7)—C(8)	1·540 (3)
C(7)—N	1·460 (2)	C(8)—C(9)	1·503 (3)
C(9)—O	1·200 (3)	C(9)—C(10)	1·503 (2)
C(10)—C(11)	1·527 (3)	C(11)—N	1·466 (2)
C(11)—C(12)	1·517 (2)	C(12)—C(17)	1·378 (3)
C(12)—C(13)	1·379 (3)	C(14)—C(15)	1·374 (4)
C(13)—C(14)	1·374 (3)	C(16)—C(17)	1·384 (3)
C(15)—C(16)	1·378 (3)		
C(2)—C(1)—C(6)	118·3 (2)	C(2)—C(1)—C(7)	121·0 (2)
C(6)—C(1)—C(7)	120·7 (2)	C(1)—C(2)—C(3)	121·0 (2)
C(2)—C(3)—C(4)	119·9 (2)	C(3)—C(4)—C(5)	119·8 (2)
C(4)—C(5)—C(6)	120·4 (2)	C(1)—C(6)—C(5)	120·5 (2)
C(1)—C(7)—C(8)	112·5 (1)	C(1)—C(7)—N	110·7 (1)
C(8)—C(7)—N	108·1 (2)	C(7)—C(8)—C(9)	110·3 (2)
C(8)—C(9)—O	122·6 (2)	C(8)—C(9)—C(10)	114·6 (2)
O—C(9)—C(10)	122·8 (2)	C(9)—C(10)—C(11)	111·3 (2)
C(10)—C(11)—N	108·0 (1)	C(10)—C(11)—C(12)	111·0 (2)
N—C(11)—C(12)	111·0 (2)	C(7)—N—C(11)	112·3 (1)
C(11)—C(12)—C(13)	120·4 (2)	C(11)—C(12)—C(17)	121·1 (2)
C(13)—C(12)—C(17)	118·5 (2)	C(12)—C(13)—C(14)	120·8 (2)
C(13)—C(14)—C(15)	120·6 (2)	C(14)—C(15)—C(16)	119·4 (2)
C(15)—C(16)—C(17)	119·8 (2)	C(12)—C(17)—C(16)	121·0 (2)

1985). Atomic coordinates for non-H atoms are given in Table 1 and bond distances and bond angles in Table 2.*

Related literature. 2,6-Diphenyl-4-piperidone (1) was synthesized by Baliah *et al.* (1954) as the major product of the condensation of benzaldehyde, acetone and ammonia, and its structure established by elemental analysis. We have determined the X-ray structure of (1) in order to help understand the conformational selectivity in nucleophilic additions

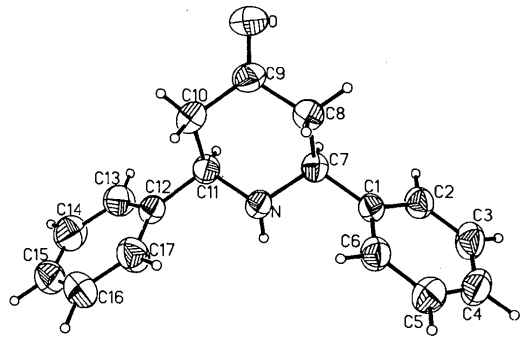

Fig. 1. View of the molecule (40% thermal ellipsoids) showing the atom-numbering scheme and *cis* orientation of the phenyl groups.

to cyclohexanones (Trost, Florez & Jebaratnam, 1987; Singh, Levine & Kasdorf, 1990). The two phenyl groups are oriented *cis* to each other with respect to the piperidone ring (Fig. 1). The dihedral angle between the phenyl rings is 54·3°.

We are grateful to the National Science Foundation (grant No. CHE 830 7022) for providing funds towards upgrading our X-ray diffractometer.

* Lists of anisotropic thermal parameters, H-atom coordinates, least-squares planes and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52990 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Bis(ketimino)(amino)phosphine

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Abstract. 1,1,5,5-Tetraphenyl-3-(2,4,6-tri-*tert*-butylphenylamino)-2,4-diaza-3-phosphapenta-1,4-diene, $C_{44}H_{50}N_3P$, $M_r = 651{\cdot}84$, triclinic, $P\bar{1}$, $a = 10{\cdot}396\ (3)$, $b = 10{\cdot}771\ (3)$, $c = 18{\cdot}178\ (6)$ Å, $\alpha = 102{\cdot}70\ (2)$, $\beta = 104{\cdot}37\ (2)$, $\gamma = 93{\cdot}60\ (3)°$, $V = 1909$ Å^3, $Z = 2$, $D_x = 1{\cdot}13$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}10$ mm^{-1}, $F(000) = 700$, $T = 298$ K, $R = 0{\cdot}071$ for 2674 observed reflections. The

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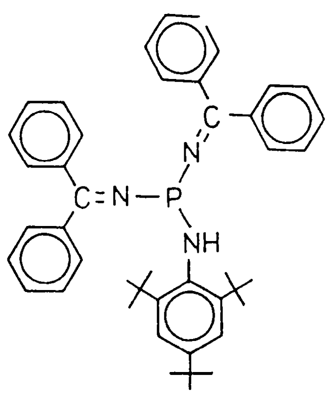
2,4-diaza-3-phosphapenta-1,4-diene can be described as a phosphine. The trigonal-pyramidal P atom is coordinated by two ketimino fragments and one amino group. The N—P—N angles are 101·0 (2), 97·4 (3) and 98·2 (2)°, respectively. The P—N distances are not equivalent: P—N(amino) = 1·701 (6), P—N(imino) = 1·715 (5) and 1·741 (5) Å, respectively.

Experimental. The title compound (1) was prepared as follows. A diethyl ether solution of lithiated diphenylimine was added to an equimolar solution of chloro(2,4,6-tri-*tert*-butylphenylimino)phosphane in diethyl ether at 195 K. After 2 h the reaction mixture was warmed up to room temperature. The solvent was removed and the residue redissolved in hexane.



(1)

Filtration and cooling the filtrate to 278 K gave orange crystals (Gärtner, 1988). The crystal dimensions were 0·2 × 0·4 × 0·5 mm. X-ray data were measured on a Nicolet *R*3*m* four-circle diffractometer with graphite-monochromated Mo *Kα* radiation. The *ω*-scan mode was used (scan rate 4·0–29·3° min^{-1}, depending on intensity). The cell constants were determined by least-squares fit of 24 reflections in the range $17 < 2\theta < 22°$. The intensities of 6546 reflections were measured ($2\theta_{max} = 45°$). Three check reflections showed no significant intensity variation (1·0%). The data were averaged to 5008 unique reflections ($R_{int} = 0·006$, *hkl* range *h* −11→11, *k* −11→11, *l* 0→19), 2674 of which, with $F > 4\sigma(F)$, were used for all calculations (*SHELXTL*, Sheldrick, 1978; *SHELXTL-Plus*, Sheldrick, 1989). Absorption and extinction corrections were not deemed necessary, but two low-angle reflections had to be ignored during refinement. The structure was solved by direct methods. Full-matrix least-squares refinement on F_o converged to $R = 0·071$, $wR = 0·053$ and $S = 1·50$. Anisotropic atomic displacement parameters were used for the non-H atoms. All the H atoms were found in a $\Delta\rho$ map, but were refined using a riding model and idealized geometry [$U(H) = 1·2U_{eq}(C)$]. The H atom bonded to N(1) was refined with N—H = 0·90 Å and $U(H) = 0·06$ Å^2. 436 parameters were refined, weighting scheme $w^{-1} = \sigma^2(F_o) + 0·0001F_o^2$, which led to a featureless analysis of variance in terms of sin*θ* and F_o, a maximum value $\Delta/\sigma = 0·017$, and maximum and minimum heights in final $\Delta\rho$ map = 0·32 and −0·23 e Å^{-3}, respectively. Atomic scattering factors were those stored in *SHELXTL* and *SHELXTL-Plus* which were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic parameters are given in Table 1,* selected bond distances and

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement parameters* (Å^2 × 10^3)

Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
P(1)	5474 (2)	1473 (2)	2258 (1)	41 (1)
N(1)	5588 (5)	979 (5)	3096 (3)	39 (2)
C(1)	4473 (6)	1028 (5)	3428 (3)	35 (3)
C(2)	4577 (6)	1816 (5)	4182 (3)	37 (3)
C(3)	3395 (6)	2088 (5)	4372 (3)	39 (3)
C(4)	2136 (6)	1631 (6)	3859 (4)	37 (3)
C(5)	2094 (6)	754 (5)	3180 (3)	39 (3)
C(6)	3226 (6)	356 (6)	2970 (3)	38 (3)
C(7)	5930 (6)	2331 (6)	4804 (3)	45 (3)
C(8)	5744 (6)	2959 (7)	5611 (3)	75 (4)
C(9)	6748 (6)	1219 (6)	4928 (4)	62 (3)
C(10)	6762 (6)	3358 (6)	4581 (4)	64 (3)
C(11)	854 (6)	2034 (6)	4086 (4)	50 (3)
C(12)	1015 (7)	3441 (7)	4477 (5)	99 (5)
C(13)	560 (7)	1241 (7)	4632 (4)	92 (5)
C(14)	−353 (6)	1742 (7)	3369 (4)	90 (4)
C(15)	2974 (6)	−850 (6)	2271 (3)	45 (3)
C(16)	2321 (7)	−545 (6)	1489 (3)	77 (4)
C(17)	1988 (6)	−1867 (6)	2411 (4)	62 (3)
C(18)	4213 (6)	−1522 (5)	2222 (4)	72 (4)
N(2)	6550 (5)	2903 (4)	2612 (3)	45 (2)
C(19)	6100 (7)	3976 (6)	2581 (3)	45 (3)
C(20)	4651 (7)	4162 (5)	2328 (4)	43 (3)
C(21)	4139 (8)	4656 (6)	1699 (4)	63 (4)
C(22)	2790 (8)	4784 (6)	1460 (4)	68 (4)
C(23)	1929 (7)	4401 (7)	1851 (5)	76 (4)
C(24)	2405 (8)	3904 (7)	2468 (5)	82 (5)
C(25)	3755 (7)	3775 (6)	2722 (4)	62 (4)
C(26)	6719 (7)	6384 (6)	2970 (3)	55 (3)
C(27)	7665 (9)	7457 (7)	3189 (4)	69 (4)
C(28)	8979 (9)	7319 (7)	3245 (4)	74 (4)
C(29)	9383 (8)	6118 (7)	3096 (4)	75 (4)
C(30)	8450 (8)	5044 (7)	2886 (4)	66 (4)
C(31)	7109 (7)	5175 (6)	2818 (3)	48 (3)
N(3)	6551 (5)	512 (4)	1907 (3)	42 (2)
C(32)	7041 (6)	600 (6)	1339 (3)	41 (3)
C(33)	5649 (7)	1850 (7)	503 (4)	64 (4)
C(34)	5469 (9)	2843 (9)	143 (4)	84 (5)
C(35)	6531 (11)	3719 (8)	230 (5)	88 (5)
C(36)	7781 (7)	3592 (7)	656 (5)	83 (5)
C(37)	7964 (7)	2571 (6)	1008 (4)	65 (4)
C(38)	6886 (7)	1694 (6)	935 (3)	43 (3)
C(39)	7966 (7)	−1459 (6)	1406 (4)	60 (3)
C(40)	8551 (7)	−2498 (7)	1106 (5)	70 (4)
C(41)	8947 (8)	−2561 (8)	434 (5)	81 (4)
C(42)	8752 (8)	−1600 (8)	60 (4)	81 (4)
C(43)	8165 (7)	−543 (7)	359 (4)	63 (4)
C(44)	7766 (6)	−460 (6)	1038 (4)	45 (3)

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles, non-bonded distances, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53095 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

P(1)—N(1)	1·701 (6)	P(1)—N(2)	1·741 (5)
P(1)—N(3)	1·715 (5)	N(1)—C(1)	1·432 (9)
N(2)—C(19)	1·281 (8)	C(19)—C(20)	1·502 (9)
C(19)—C(31)	1·521 (9)	N(3)—C(32)	1·278 (9)
C(32)—C(38)	1·516 (10)	C(32)—C(44)	1·486 (9)
N(1)—P(1)—N(2)	101·0 (2)	N(1)—P(1)—N(3)	97·4 (3)
N(2)—P(1)—N(3)	98·2 (2)	P(1)—N(1)—C(1)	119·2 (4)
N(1)—C(1)—C(2)	121·3 (5)	N(1)—C(1)—C(6)	118·9 (5)
C(2)—C(1)—C(6)	119·6 (6)	P(1)—N(2)—C(19)	120·5 (4)
N(2)—C(19)—C(20)	125·7 (5)	N(2)—C(19)—C(31)	117·8 (6)
C(20)—C(19)—C(31)	116·5 (6)	P(1)—N(3)—C(32)	126·5 (4)
N(3)—C(32)—C(38)	124·0 (6)	N(3)—C(32)—C(44)	117·8 (6)
C(38)—C(32)—C(44)	118·1 (6)		

angles in Table 2. Fig. 1 shows a plot with the atom numbering.

Related literature. Recently we showed (Niecke, Nieger, Gärtner-Winkhaus & Kramer, 1990) that reaction of lithiated ketimines LiN=CR_2 (R_2 = ^{t}Bu$_2$, fluorenyl) with the chloro(arylimino)phosphane Cl—P=N—Ar (Ar = 2,4,6-^{t}Bu$_3$C$_6$H$_2$) gives the heterobutadienes R_2 = N—P=N—Ar with unusual short P—N single bonds in both *cis* and *trans* arrangements of the NPN skeleton. The reaction with the lithiated ketimine LiN=CPh$_2$ resulted in the structure described above.

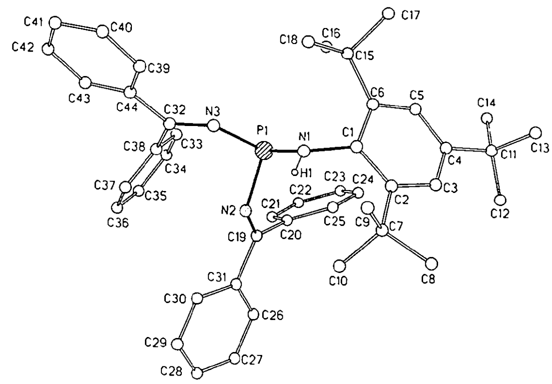


Fig. 1. A perspective view of the bis(ketimino)(amino)phosphine molecule with the atom numbering.

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The Structure of (4R*,7R*)-4,10,11,11-Tetramethyl-5-oxobicyclo[5.3.1]undec-1(10)-en-4-carbaldehyde

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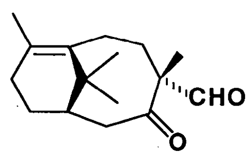
Abstract. $C_{16}H_{24}O_2$, M_r = 248·36, orthorhombic, $P2_12_12_1$, a = 9·0745 (9), b = 12·1499 (15), c = 12·8820 (10) Å, V = 1420·3 (3) Å^3, Z = 4, D_x = 1·16 g cm^{-3}, μ = 0·6946 cm^{-1}, Mo $K\alpha$, λ = 0·7107 Å, F(000) = 544, T = 298 K, R = 0·0624 for 1238 reflections where $F_o \geq 4[\sigma(F_o)]$. The cyclooctane ring is in the usual boat–chair conformation. The bridgehead atom of the alkene group, C7, has one short [C7—C6 1·485 (6) Å] and one long [C7—C11, 1·541 (6) Å] C_{sp^2}—C_{sp^3} bond. The alkene group is presumably distorted due to ring strain. The six C atoms comprising the group are non-planar [max. dev. 0·114 (7) Å for C9]. The two nearly planar fragments (C6, C7, C8, C11 and C7, C8, C9, C18) are twisted by 8·9 (3)°.

Experimental. (1) was synthesized by sequential formylation and methylation of the corresponding [5.3.1]bicyclic ketone which was prepared by an anionic oxy-Cope rearrangement of the appropriate dienol precursor (Martin, White & Wagner, 1982).



(1)

The full details of the synthesis of (1) are presented elsewhere (Martin, White, Wagner, Guinn, Tanaka,

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