Structure of 2,6-Diphenyl-4-piperidone

By P. Singh, S. G. Levine and K. Kasdorf

Department of Chemistry, North Carolina State University, Raleigh, NC 27695-8204, USA

Abstract. $C_{17}H_{17}NO$, $M_r = 251.33$, triclinic, $P\bar{1}$, $a = 5.669$ (1), $b = 10.706$ (3), $c = 11.952$ (4) Å, $\alpha = 105.78$ (2), $\beta = 92.40$ (2), $\gamma = 96.35$ (2)°, $V = 691.8$ (3) Å^3, $Z = 2$, $D_x = 1.21$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.70$ cm^{-1}, $F(000) = 268$, $T = 298$ K, $R = 0.0451$ for 1952 reflections with $I \geq 2.5\sigma(I)$. The two phenyl groups attached to the piperidone ring are oriented *cis* to each other with respect to the piperidone ring. The molecule possesses an approximate mirror symmetry, the mirror plane passing through the atoms N, C(9) and O. The dihedral angles C(17)—C(12)—C(11)—C(10) [−76.0 (2)°] and C(6)—C(1)—C(7)—C(18) [73.1 (2)°] are consistent with this symmetry. There are no hydrogen bonds in the crystal.

Experimental. 2,6-Diphenyl-4-piperidone (1) was synthesized according to the literature procedure (Baliah, Ekambaram & Govindrajan, 1954) and crystallized by cooling a hot concentrated solution of the com-

(1)
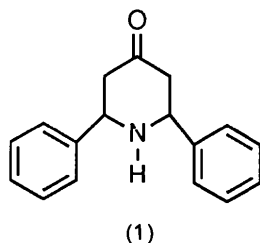
pound in cyclohexane down to room temperature. A crystal measuring approximately 0.72 × 0.35 × 0.05 mm was used for data collection using Mo $K\alpha$ radiation on a Nicolet $R3m/\mu$ diffractometer equipped with a graphite monochromator. The cell dimensions were obtained by a least-squares refinement of the setting angles of 16 reflections. The intensity data were collected by the ω-scan method between 3 and 60° in 2θ at variable scan speeds between 3.9 and 29.3° min^{-1} depending upon intensity; range in *hkl*: $-7 < h < 7$, $-15 < k < 14$, $0 < l < 16$. Stationary backgrounds were measured for half the scan time on each side of a peak. Two standard reflections, measured after every 48 reflections to monitor the instrument and crystal stability, showed a maximum

fluctuation in intensity of ±2.5%. 4053 reflections were measured which yielded 1952 reflections with $I \geq 2.5\sigma(I)$; $R_{int} = 0.011$. The intensities were corrected for background, and Lorentz and polarization effects but not for absorption ($\mu = 0.70$ cm^{-1}).

The structure was solved by direct methods and refined by blocked-cascade least squares (Sparks, 1961). The function minimized was $\Sigma w(|F_o| - |F_c|)^2$, where $w = 1/[\sigma_F^2 - 0.0005F^2]$. The non-H atoms were refined with anisotropic thermal parameters. The H atoms were located from a difference electron-density map and refined with isotropic thermal parameters. An isotropic secondary-extinction correction was applied to the intensities, the refined value of the extinction coefficient being 4.0 (2) × 10^{-6}. The final $R = 0.0451$, $wR = 0.0555$, goodness-of-fit parameter $S = 1.43$. The maximum $\Delta/\sigma = 0.3$ in the final cycle of least-squares refinement, and the maximum and minimum peaks in the final ΔF map were +0.17 and −0.14 e Å^{-3}, respectively. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Computations were performed on a Data General Desktop Microeclipse computer, with the crystallographic program package *SHELXTL* (Sheldrick,

Table 1. *Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters (Å$^2 \times 10^3$)*

	x	y	z	U^*
C(1)	2332 (3)	9023 (2)	8180 (1)	40 (1)
C(2)	842 (4)	9986 (2)	8447 (2)	48 (1)
C(3)	1301 (4)	11128 (2)	8121 (2)	58 (1)
C(4)	3248 (4)	11315 (2)	7518 (2)	58 (1)
C(5)	4755 (4)	10374 (2)	7253 (2)	58 (1)
C(6)	4314 (4)	9237 (2)	7589 (2)	49 (1)
C(7)	1783 (3)	7759 (2)	8500 (2)	42 (1)
C(8)	3483 (4)	7667 (2)	9505 (2)	51 (1)
C(9)	2929 (4)	6367 (2)	9753 (2)	49 (1)
O	2617 (3)	6269 (1)	10714 (1)	67 (1)
C(10)	2705 (2)	5205 (2)	8694 (2)	52 (1)
C(11)	1068 (3)	5394 (2)	7721 (2)	42 (1)
N	1946 (3)	6638 (1)	7499 (1)	43 (1)
C(12)	993 (3)	4264 (2)	6625 (2)	43 (1)
C(13)	−888 (4)	3273 (2)	6357 (2)	55 (1)
C(14)	−907 (5)	2210 (2)	5403 (2)	67 (1)
C(15)	951 (5)	2113 (2)	4695 (2)	64 (1)
C(16)	2842 (5)	3096 (2)	4949 (2)	70 (1)
C(17)	2856 (4)	4163 (2)	5914 (2)	59 (1)

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

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)

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

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

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°.

* 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

By Martin Nieger, Edgar Niecke and Christiane Gärtner-Winkhaus

Institut für Anorganische Chemie der Universität, Gerhard-Domagk-Straße 1, D-5300 Bonn, Federal Republic of Germany

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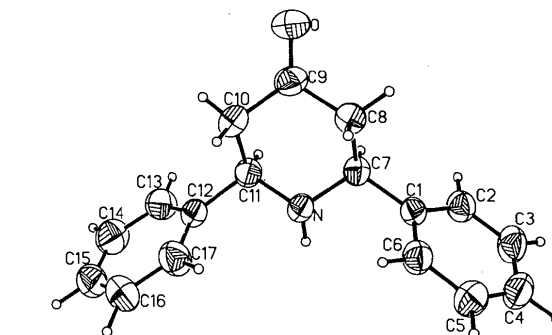10·396$ (3), $b = 10·771$ (3), $c = 18·178$ (6) Å, $\alpha = 102·70$ (2), $\beta = 104·37$ (2), $\gamma = 93·60$ (3)°, $V = 1909$ Å^3, $Z = 2$, $D_x = 1·13$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 0·10$ mm^{-1}, $F(000) = 700$, $T = 298$ K, $R = 0·071$ for 2674 observed reflections.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