

## Structure of 3,5-Bis(4-dimethylaminobenzylidene)-1-methyl-4-piperidone Methiodide

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**Abstract.**  $C_{25}H_{32}N_3O^+ \cdot I^-$ ,  $M_r = 517.45$ , triclinic,  $P\bar{1}$ ,  $a = 7.1684$  (8),  $b = 13.144$  (1),  $c = 14.088$  (1) Å,  $\alpha = 66.778$  (7),  $\beta = 88.417$  (8),  $\gamma = 80.164$  (8)°,  $V = 1200.72$  Å<sup>3</sup>,  $Z = 2$ ,  $D_m$ (by flotation) = 1.44,  $D_x = 1.430$  g cm<sup>-3</sup>,  $\lambda$ (Cu  $K\alpha$ ) = 1.5418 Å,  $\mu = 10.88$  cm<sup>-1</sup>,  $F(000) = 528$ ,  $T = 287$  K,  $R = 0.066$  ( $wR = 0.062$ ) for 4448 observed reflections. The central heterocyclic ring exhibits a chair conformation which is obviously flattened at the C(4) end due to the conjugated dienone system. The steric repulsions between H atoms attached

to C(2)/C(6) and those on the phenyl rings cause increases in the bond angles at the C atoms joining the rings and rotations of the phenyl groups about the two C–Ph bonds at the expense of the conjugation energy of the system.

**Experimental.** Title compound prepared according to the literature procedure (McElvain & McMahon, 1949) in a yield of 80% with m.p. 533 K (found: C 57.78, H

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Table 1. Fractional coordinates and equivalent isotropic thermal parameters ( $\times 10^3$ ) with *e.s.d.*'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}$ * (Å <sup>2</sup> )
I	0.28539 (6)	0.27701 (3)	0.03975 (4)	52
O(4)	0.2387 (8)	0.5037 (4)	0.4453 (4)	58
N(1)	0.2750 (7)	0.5533 (4)	0.1408 (3)	32
N(2)	0.936 (1)	0.9783 (5)	0.1640 (6)	61
N(3)	-0.5132 (9)	0.1256 (5)	0.3697 (6)	59
C(1A)	0.4598 (9)	0.4731 (5)	0.1718 (5)	42
C(1B)	0.2441 (9)	0.5919 (5)	0.0261 (5)	41
C(1N2)	1.090 (1)	0.9780 (8)	0.2286 (9)	76
C(2N2)	0.876 (1)	1.0804 (7)	0.0730 (8)	69
C(1N3)	-0.688 (1)	0.1258 (7)	0.4212 (8)	67
C(2N3)	-0.477 (1)	0.0568 (7)	0.3084 (7)	73
C(2)	0.2826 (9)	0.6541 (4)	0.1660 (5)	38
C(3)	0.3296 (8)	0.6158 (4)	0.2793 (4)	37
C(4)	0.2264 (9)	0.5295 (5)	0.3512 (5)	40
C(5)	0.1102 (8)	0.4743 (5)	0.3073 (4)	35
C(6)	0.1152 (9)	0.4960 (6)	0.1937 (5)	45
C(7)	-0.0089 (8)	0.4132 (5)	0.3721 (5)	38
C(8)	0.4659 (9)	0.6479 (5)	0.3199 (5)	39
C(9)	0.5841 (8)	0.7325 (5)	0.2737 (5)	39
C(10)	0.7512 (9)	0.7229 (5)	0.3281 (5)	44
C(11)	0.868 (1)	0.8018 (5)	0.2917 (6)	48
C(12)	0.822 (1)	0.8969 (5)	0.1996 (6)	48
C(13)	0.655 (1)	0.9076 (5)	0.1454 (6)	49
C(14)	0.539 (1)	0.8271 (5)	0.1815 (5)	45
C(15)	-0.1388 (8)	0.3438 (4)	0.3612 (4)	34
C(16)	-0.280 (1)	0.3197 (5)	0.4341 (5)	47
C(17)	-0.405 (1)	0.2502 (5)	0.4363 (6)	49
C(18)	-0.3935 (9)	0.1979 (5)	0.3659 (5)	41
C(19)	-0.253 (1)	0.2213 (5)	0.2935 (5)	45
C(20)	-0.1292 (9)	0.2930 (5)	0.2905 (5)	40

\* Equivalent isotropic  $U$  defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.Table 2. Bond distances (Å) and angles (°) with *e.s.d.*'s in parentheses

O(4)–C(4)	1.234 (8)	C(5)–C(7)	1.349 (7)
N(1)–C(1A)	1.500 (7)	C(7)–C(15)	1.460 (9)
N(1)–C(1B)	1.501 (7)	C(8)–C(9)	1.449 (8)
N(1)–C(2)	1.510 (8)	C(9)–C(10)	1.400 (9)
N(1)–C(6)	1.496 (8)	C(9)–C(14)	1.397 (7)
N(2)–C(1N2)	1.446 (9)	C(10)–C(11)	1.374 (9)
N(2)–C(2N2)	1.452 (9)	C(11)–C(12)	1.399 (8)
N(2)–C(12)	1.384 (9)	C(12)–C(13)	1.399 (9)
N(3)–C(1N3)	1.430 (9)	C(13)–C(14)	1.387 (9)
N(3)–C(2N3)	1.469 (9)	C(15)–C(16)	1.406 (8)
N(3)–C(18)	1.369 (9)	C(15)–C(20)	1.396 (9)
C(2)–C(3)	1.500 (8)	C(16)–C(17)	1.376 (9)
C(3)–C(4)	1.483 (8)	C(17)–C(18)	1.406 (9)
C(3)–C(8)	1.349 (9)	C(18)–C(19)	1.397 (9)
C(4)–C(5)	1.480 (9)	C(19)–C(20)	1.389 (9)
C(5)–C(6)	1.510 (9)		
C(1A)–N(1)–C(1B)	107.6 (4)	C(5)–C(7)–C(15)	133.2 (6)
C(1A)–N(1)–C(2)	110.3 (5)	C(3)–C(8)–C(9)	132.1 (5)
C(1A)–N(1)–C(6)	110.5 (4)	C(8)–C(9)–C(10)	117.9 (4)
C(1B)–N(1)–C(2)	109.0 (4)	C(8)–C(9)–C(14)	124.8 (5)
C(1B)–N(1)–C(6)	108.9 (5)	C(10)–C(9)–C(14)	117.1 (5)
C(2)–N(1)–C(6)	110.4 (5)	C(9)–C(10)–C(11)	121.6 (5)
C(1N2)–N(2)–C(2N2)	117.4 (7)	C(10)–C(11)–C(12)	121.5 (6)
C(1N2)–N(2)–C(12)	120.9 (6)	N(2)–C(12)–C(11)	121.8 (6)
C(2N2)–N(2)–C(12)	119.9 (7)	N(2)–C(12)–C(13)	121.0 (5)
C(1N3)–N(3)–C(2N3)	118.0 (8)	C(11)–C(12)–C(13)	117.2 (6)
C(1N3)–N(3)–C(18)	121.5 (8)	C(12)–C(13)–C(14)	121.3 (5)
C(2N3)–N(3)–C(18)	119.9 (6)	C(9)–C(14)–C(13)	121.3 (6)
N(1)–C(2)–C(3)	109.6 (4)	C(7)–C(15)–C(16)	116.3 (6)
C(2)–C(3)–C(4)	116.6 (5)	C(7)–C(15)–C(20)	126.7 (5)
C(2)–C(3)–C(8)	125.3 (5)	C(16)–C(15)–C(20)	116.9 (6)
C(4)–C(3)–C(8)	117.9 (5)	C(15)–C(16)–C(17)	122.3 (7)
O(4)–C(4)–C(3)	120.7 (6)	C(16)–C(17)–C(18)	120.8 (6)
O(4)–C(4)–C(5)	120.9 (5)	N(3)–C(18)–C(17)	121.2 (6)
C(3)–C(4)–C(5)	118.4 (5)	N(3)–C(18)–C(19)	121.7 (7)
C(4)–C(5)–C(6)	120.4 (5)	C(17)–C(18)–C(19)	117.1 (6)
C(4)–C(5)–C(7)	116.1 (5)	C(18)–C(19)–C(20)	121.9 (7)
C(6)–C(5)–C(7)	123.3 (6)	C(15)–C(20)–C(19)	121.0 (5)
N(1)–C(6)–C(5)	113.4 (5)		

6.32, N 7.91%;  $C_{25}H_{32}N_3O^+I^-$  requires C 58.03, H 6.23, N 8.12%). Crystallized from mixture of MeOH and cyclohexane. Orange crystal with dimensions 0.15 × 0.20 × 0.30 mm. Cell parameters by least squares using 25 reflections with  $21.76 < \theta < 33.08^\circ$ . Enraf-Nonius CAD-4 diffractometer used for data collection, 4924 unique reflections,  $-8 \leq h \leq 8$ ,  $-16 \leq k \leq 16$ ,  $0 \leq l \leq 17$ ,  $[(\sin\theta)/\lambda]_{\max} = 0.62653 \text{ \AA}^{-1}$ . 4448 reflections with  $I > 2\sigma(I)$  used in refinement. Three intensity and orientation monitor reflections, intensity fluctuation within 1.6%. Absorption applied, max. and min. corrections of 8.445 and 2.876. No extinction correction. Merging  $R$  based on intensities of 0.0290 for 465 replicate reflections. Structure solved by direct methods using *XTAL* (Hall & Stewart, 1988), all non-H atoms found on  $E$  map and refined anisotropically; H atoms calculated and not refined.  $R = 0.066$ ,  $wR = 0.062$  [ $w = 1/\sigma^2(F)$ ],  $S = 4.477$  for 4448 observed reflections, 271 parameters refined.  $F$  magnitudes used in LS refinement. Final  $(\Delta/\sigma)_{\text{av}} = 0.023$ ,  $(\Delta/\sigma)_{\max} = 0.38$ .  $\Delta\rho$  in final difference map were +0.81 and  $-0.77 \text{ e \AA}^{-3}$  (other larger peaks +1.59, +1.34 and +1.04  $\text{e \AA}^{-3}$  within 1.34  $\text{\AA}$  from  $I^-$ ; holes  $-1.87$ ,  $-0.97$  and  $-0.84 \text{ e \AA}^{-3}$  within 1.21  $\text{\AA}$  from  $I^-$ ). Atomic scattering factors from *International Tables for*

*X-ray Crystallography* (1974). All calculations performed on a VAX8650 computer at the University of Saskatchewan.

The atomic parameters are summarized in Table 1.\* Bond distances and angles are listed in Table 2. Fig. 1 is an *ORTEP* drawing (Johnson, 1976) of the title compound.

**Related literature.** Other structures in this series of cyclic conjugated *bis*(benzylidene)ketones, which have various cytotoxicities to P388 leukemia cells (Warrington, Fang, Dimmock & Arora, 1988), have been previously reported (Jia, Quail, Arora & Dimmock, 1988, 1989). The title compound shows similar geometrical features to those of related molecules that mainly result from the steric repulsions (Tokuno, Matsui, Miyoshi, Asao, Ohashi & Kihara, 1986). In this series of molecules, the title compound has the highest bioactivity.

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\* Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51758 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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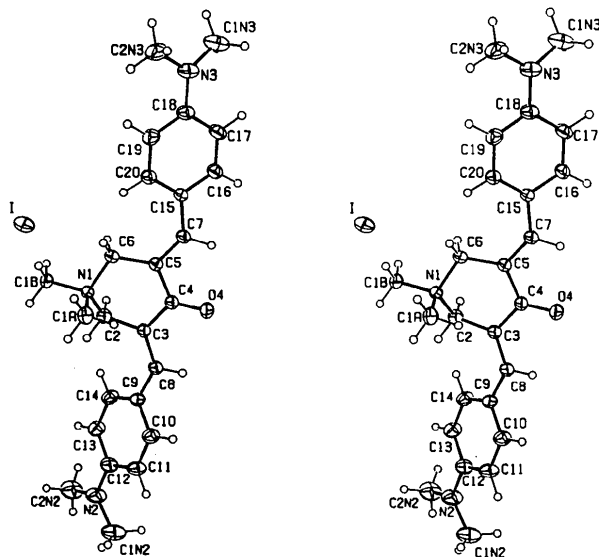


Fig. 1. Stereoscopic *ORTEP* view (Johnson, 1976) with atomic numbering.