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2,2'-Piperazin-1,4-diyldimethylenebis[6-(benzyliminomethyl)-4-methylphenol]

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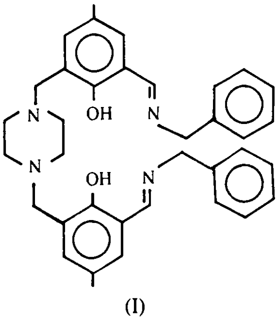
Abstract

The title compound, $C_{36}H_{40}N_4O_2$, was synthesized and the crystal structure solved using X-ray diffraction techniques. The structure has half a molecule in the asymmetric unit and $Z = 2$. The molecule possesses a centre of inversion. The phenyl and benzyl rings lie nearly perpendicular to each other, and the structure is stabilized by van der Waals type interactions.

Comment

A perspective view of the title molecule, (I), showing the atom-numbering scheme is presented in Fig. 1 (the atoms which are not numbered are symmetry related to the numbered atoms). In the phenyl ring (C1–C6) the average bond length of 1.390 Å agrees with average values reported in the literature (Domenicano, Vaciago & Coulsun, 1975). The N atom, N19, of the piperazine ring is sp^3 hybridized (Perales, Cano & Garcia-Blanco, 1977).



(I)

The torsion angles C5—C6—C18—N19 [−46.4 (3)°] and C1—C6—C18—N19 [136.0 (3)°] define the position of the piperazine ring in the molecule. The substituent at N19 is in an equatorial position (Allinger, Carpenter & Karkowski, 1965). There is half a molecule in the asymmetric unit and two molecules in the unit cell. One half of the molecule is related to the other by a centre of inversion. Atoms C20 and C21 of the molecule are connected to C21 and C20 of the second half by 1.512 Å and *vice versa*.

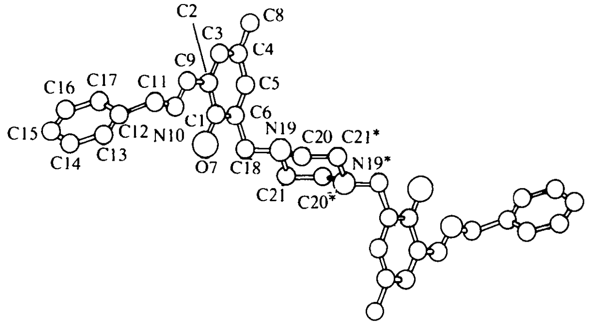


Fig. 1. Perspective view of the molecule with atom-numbering scheme.

All the atoms in the phenyl ring lie in a plane with a maximum deviation of ±0.006 (3) Å, for C1. The phenyl ring forms a dihedral angle of 85.6 (1)° with the benzyl ring to avoid making short contacts. The piperazine moiety makes dihedral angles of 89.9 (1) and 67.5 (1)° with the phenyl and benzyl rings, respectively.

The C—O [1.355 (4) Å] and C=N [1.268 (4) Å] distances are comparable with reported values (Allen *et al.*, 1987). An intramolecular short contact is observed between O7 and N10 [2.597 (3) Å]. A stereoview of the packing of the molecules down the *a* axis is shown in Fig. 2. Packing of the molecules is stabilized by van der Waals interactions.

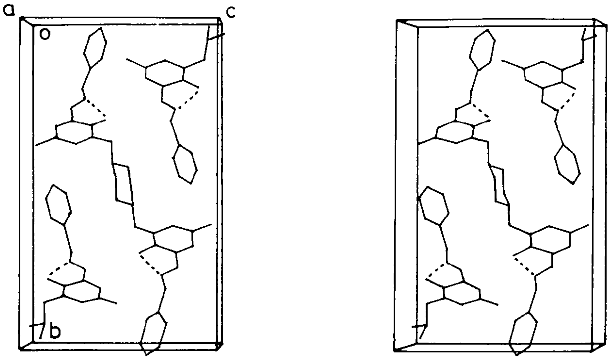


Fig. 2. Stereoview of the packing of molecules down the *a* axis.

Experimental

Paraformaldehyde (2 mol) and piperazine (1 mol) were stirred in 15 ml of acetic acid for an hour. 4-Methyl-2-formylphenol (2 mol) dissolved in 20 ml of acetic acid was added and stirred for 6 h. This 2:2:1 mixture was subjected to Schiff base condensation with two moles of benzylamine to yield the title compound (Hodgkin, 1984). Then the whole mixture was neutralized using saturated Na_2CO_3 and the compound was extracted using chloroform. The crude sample was purified by silica-gel column using *n*-hexane–chloroform (30:70 *v*/*v*) solvent mixture as the eluent. The compound was crystallized by slow evaporation from ether/$CHCL_3$ mixture.

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† DCB contribution No. 867.

Crystal data

$C_{36}H_{40}N_4O_2$
$M_r = 560.7$
Monoclinic
$P2_1/n$
$a = 5.931\ (2)$ Å
$b = 20.811\ (3)$ Å
$c = 12.479\ (1)$ Å
$\beta = 98.68\ (3)°$
$V = 1522.64$ Å^3
$Z = 2$
$D_x = 1.223$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 15–23°$
$\mu = 0.609$ mm^{-1}
$T = 298$ K
Needle
0.50 × 0.42 × 0.23 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan
$T_{\min} = 0.823$, $T_{\max} = 0.976$
3288 measured reflections
2892 independent reflections
1698 observed reflections $[I \geq 3\sigma(I)]$

$R_{\rm int} = 0.019$
$\theta_{\max} = 70°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 25$
$l = -15 \rightarrow 15$
3 standard reflections monitored every 200 reflections intensity decay: <1.2%

Refinement

Refinement on F
$R = 0.05$
$wR = 0.06$
$S = 1.15$
1698 reflections
266 parameters
$w = 1/[\sigma^2(F) + 0.0038F^2]$

$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 0.358$ e Å^{-3}
$\Delta\rho_{\min} = -0.212$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *SHELX76* (Sheldrick, 1976)

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

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
C1	0.5983 (5)	0.1854 (1)	−0.18580 (2)	3.85 (7)
C2	0.5001 (5)	0.2018 (1)	−0.29210 (2)	4.09 (8)
C3	0.6013 (5)	0.1813 (1)	−0.37940 (2)	4.43 (6)
C4	0.7969 (5)	0.1448 (1)	−0.36560 (2)	4.28 (6)
C5	0.8893 (5)	0.1289 (1)	−0.25950 (2)	4.03 (7)
C6	0.7950 (5)	0.1476 (1)	−0.17010 (2)	3.73 (7)
O7	0.5074 (4)	0.2071 (1)	−0.09930 (1)	5.14 (5)
C8	0.9113 (7)	0.1237 (2)	−0.45970 (1)	5.84 (11)
C9	0.2943 (5)	0.2419 (1)	−0.31250 (1)	4.78 (9)
N10	0.2024 (4)	0.2657 (1)	−0.23610 (2)	5.15 (9)
C11	−0.0086 (5)	0.3034 (1)	−0.26520 (3)	6.08 (11)
C12	0.0175 (5)	0.3713 (1)	−0.22590 (2)	4.04 (8)
C13	−0.1383 (6)	0.3994 (1)	−0.16770 (3)	5.77 (11)
C14	−0.1195 (7)	0.4631 (2)	−0.13770 (3)	7.01 (12)
C15	0.0552 (8)	0.4994 (2)	−0.16410 (3)	6.80 (11)
C16	0.2120 (6)	0.4726 (1)	−0.22010 (3)	5.47 (9)
C17	0.1920 (5)	0.4093 (1)	−0.25060 (2)	4.27 (8)
C18	0.9120 (5)	0.1292 (1)	−0.05750 (2)	4.32 (7)
N19	0.9788 (3)	0.0619 (1)	−0.05140 (1)	3.35 (5)
C20	0.7810 (4)	0.0209 (1)	−0.04710 (2)	3.66 (5)
C21	1.1511 (4)	0.0492 (1)	0.04270 (2)	3.54 (4)

Table 2. *Selected geometric parameters (Å, °)*

C1—C2	1.408 (3)	N10—C11	1.475 (4)
C1—C6	1.399 (4)	C11—C12	1.496 (3)
C1—O7	1.355 (4)	C12—C13	1.388 (5)
C2—C3	1.388 (4)	C12—C17	1.378 (4)
C2—C9	1.469 (4)	C13—C14	1.377 (6)
C3—C4	1.376 (4)	C14—C15	1.362 (6)
C4—C5	1.394 (3)	C15—C16	1.364 (6)
C4—C8	1.508 (4)	C16—C17	1.371 (3)
C5—C6	1.376 (4)	C18—N19	1.454 (3)
C6—C18	1.518 (3)	N19—C20	1.458 (3)
C9—N10	1.268 (4)	N19—C21	1.460 (3)
C6—C1—O7	120.1 (2)	C9—N10—C11	117.9 (3)
C2—C1—O7	120.8 (2)	N10—C11—C12	112.3 (2)
C2—C1—C6	119.1 (2)	C11—C12—C17	120.9 (2)
C1—C2—C9	121.1 (2)	C11—C12—C13	121.7 (3)
C1—C2—C3	119.9 (2)	C13—C12—C17	117.4 (3)
C3—C2—C9	119.0 (2)	C12—C13—C14	121.0 (3)
C2—C3—C4	121.9 (2)	C13—C14—C15	120.1 (4)
C3—C4—C8	122.3 (2)	C14—C15—C16	120.0 (4)
C3—C4—C5	117.0 (2)	C15—C16—C17	120.0 (3)
C5—C4—C8	120.8 (3)	C12—C17—C16	121.6 (3)
C4—C5—C6	123.6 (2)	C6—C18—N19	111.9 (2)
C1—C6—C5	118.5 (2)	C18—N19—C21	111.6 (2)
C5—C6—C18	119.7 (2)	C18—N19—C20	110.6 (2)
C1—C6—C18	121.7 (2)	C20—N19—C21	109.5 (2)
C2—C9—N10	122.1 (2)		

Refinement was performed by full-matrix least-squares methods. All H-atom parameters were refined except the phenolic H atom which could not be located from the difference Fourier map.

Data reduction: *SDP* (Frenz, 1978). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *PLUTO* (Motherwell and Clegg, 1978). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1018). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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