Conwell, E. (1988). In *Semiconductors and Semimetals*, Vol. 27. New York: Academic Press.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
Mizuguchi, J. (1981). *Krist. Tech.* **16**, 695–700.
Wheland, R. C. & Martin, E. I. (1975). *J. Org. Chem.* **40**, 3101–3109.
Zambounis, J. S., Mizuguchi, J., Rihs, G., Chauvet, O. & Zuppiroli, L. (1994). *J. Appl. Phys.* **76**(3). In the press.

Acta Cryst. (1994). **C50**, 2007–2009

4-*tert*-Butyl-2,6-bis[(4-methyl-1-piperazinyl)methyl]phenol

S. Shanmuga Sundara Raj, D. Velmurugan* and E. Subramanian

Department of Crystallography and Biophysics,† *University of Madras, Guindy Campus, Madras 600 025, India*

M. Kandaswamy

Department of Inorganic Chemistry, University of Madras, Guindy Campus, Madras 600 025, India

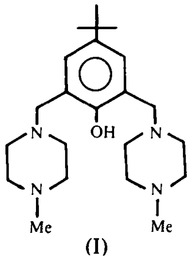
(*Received* 20 *May* 1993; *accepted* 10 *January* 1994)

Abstract

In the title compound, $C_{22}H_{38}N_4O$, both the piperazine rings are in perfect chair conformations and the substituents attached to the N atoms of the piperazine rings are in equatorial positions. The tertiary butyl group is oriented in such a way that the plane of the phenyl group nearly bisects the angle between two of the methyl C atoms of the butyl group, while the third methyl C atom lies in the plane of the phenyl ring.

Comment

The X-ray structure analysis of the title compound, (I), a binucleating ligand, has been undertaken to study the conformational features of the piperazine rings. The compound was synthesized by a modification of a procedure reported in the literature (Hodgkin, 1984). The molecular geometry is in agreement with that reported for similar molecules (Allen *et al.*, 1987; Domenicano, Vaciago & Coulson, 1975; Schwarzenbach, 1968).



(I)

Each piperazine ring adopts a perfect chair conformation (Bassi & Scordamagila, 1977; Sbit, Dupont, Diderberg, Liegeois & Delarge, 1992) similar to that found in morpholine (Shanmuga Sundara Raj, Ponnuswamy, Shanmugam & Kandaswamy, 1993) and piperidine (Shanmuga Sundara Raj, Velmurugan, Ponnuswamy, Shanmugam & Kandaswamy, 1994). The substituents at the N atoms of the piperazine rings are in equatorial positions (Allinger, Carpenter & Karkowski, 1965). The molecular packing is based on van der Waals interactions.

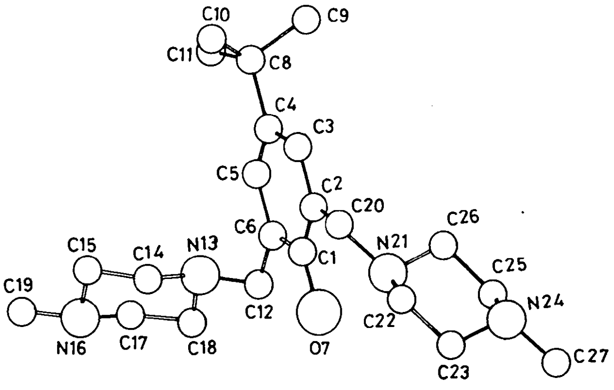


Fig. 1. Perspective view of the molecule with atomic numbering scheme.

Experimental

Crystal data

$C_{22}H_{38}N_4O$
$M_r = 374.6$
Triclinic
$P\bar{1}$
$a = 9.720\ (1)$ Å
$b = 9.945\ (1)$ Å
$c = 12.172\ (3)$ Å
$\alpha = 84.53\ (1)°$
$\beta = 84.97\ (2)°$
$\gamma = 83.49\ (1)°$
$V = 1160.2\ (3)$ Å^3
$Z = 2$
$D_x = 1.07$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 20 reflections
$\theta = 15–25°$
$\mu = 0.45$ mm^{-1}
$T = 298$ K
Transparent needle
0.3 × 0.25 × 0.2 mm
White

---

† DCB Contribution No. 836.

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3825 measured reflections
3619 independent reflections
2751 observed reflections [$I \geq 3\sigma(I)$]

$R_{\rm int} = 0.010$
$\theta_{\rm max} = 65°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 14$
3 standard reflections
frequency: 120 min
intensity variation: <1.5%

Refinement

Refinement on F
$R = 0.061$
$wR = 0.071$
$S = 0.53$
2751 reflections
396 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F) + 0.406F^2]$
$(\Delta/\sigma)_{\rm max} = 0.005$
$\Delta\rho_{\rm max} = 0.27$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.33$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *SHELX*76 (Sheldrick, 1976)

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

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

	x	y	z	$B_{\rm eq}$
C1	0.3755 (2)	−0.0367 (2)	0.7229 (2)	3.91 (5)
C2	0.3057 (2)	−0.1031 (2)	0.6522 (2)	4.01 (5)
C3	0.1621 (2)	−0.0979 (2)	0.6688 (2)	4.08 (4)
C4	0.0848 (2)	−0.0250 (2)	0.7492 (2)	3.94 (5)
C5	0.1579 (2)	0.0417 (2)	0.8165 (2)	4.12 (5)
C6	0.3024 (2)	0.0372 (2)	0.8045 (2)	4.03 (5)
O7	0.5174 (1)	−0.0415 (2)	0.7113 (1)	5.07 (5)
C8	−0.0744 (2)	−0.0210 (2)	0.7610 (2)	4.63 (7)
C9	−0.1170 (3)	−0.1615 (3)	0.7970 (3)	7.58 (11)
C10	−0.1314 (2)	0.0275 (3)	0.6486 (3)	6.56 (9)
C11	−0.1414 (3)	0.0790 (4)	0.8456 (3)	7.47 (8)
C12	0.3783 (2)	0.1120 (2)	0.8795 (2)	5.12 (7)
N13	0.3050 (2)	0.2421 (2)	0.9071 (1)	4.00 (5)
C14	0.2860 (2)	0.3373 (2)	0.8093 (2)	4.65 (5)
C15	0.2117 (3)	0.4709 (2)	0.8417 (2)	5.17 (8)
N16	0.2906 (2)	0.5318 (2)	0.9166 (2)	4.73 (5)
C17	0.3114 (3)	0.4378 (2)	1.0130 (2)	4.85 (7)
C18	0.3838 (2)	0.3030 (2)	0.9825 (2)	4.64 (5)
C19	0.2230 (3)	0.6624 (3)	0.9447 (3)	6.64 (8)
C20	0.3827 (2)	−0.1709 (2)	0.5564 (2)	4.79 (5)
N21	0.5171 (2)	−0.2452 (2)	0.5845 (1)	4.19 (4)
C22	0.6021 (2)	−0.2814 (2)	0.4845 (2)	5.09 (7)
C23	0.7369 (3)	−0.3597 (3)	0.5117 (2)	5.70 (8)
N24	0.7144 (2)	−0.4820 (2)	0.5832 (2)	5.80 (7)
C25	0.6325 (3)	−0.4447 (2)	0.6835 (2)	5.55 (8)
C26	0.4951 (2)	−0.3687 (2)	0.6569 (2)	4.86 (5)
C27	0.8436 (4)	−0.5607 (3)	0.6085 (4)	8.83 (13)

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

C1—C2	1.396 (3)	N13—C18	1.460 (3)
C1—C6	1.389 (3)	C14—C15	1.508 (3)
C1—O7	1.370 (2)	C15—N16	1.456 (4)
C2—C3	1.389 (3)	N16—C17	1.443 (3)
C2—C20	1.502 (3)	N16—C19	1.445 (4)
C3—C4	1.395 (3)	C17—C18	1.505 (3)
C4—C5	1.389 (5)	C20—N21	1.477 (3)
C4—C8	1.538 (3)	N21—C22	1.461 (3)
C5—C6	1.395 (3)	N21—C26	1.467 (3)
C6—C12	1.518 (3)	C22—C23	1.493 (3)
C8—C9	1.516 (4)	C23—N24	1.453 (3)
C8—C10	1.537 (4)	N24—C25	1.453 (3)
C8—C11	1.549 (4)	N24—C27	1.443 (4)
C12—N13	1.458 (3)	C25—C26	1.503 (3)
N13—C14	1.460 (3)		
C6—C1—O7	119.0 (2)	C12—N13—C18	109.4 (2)
C2—C1—O7	120.3 (2)	C12—N13—C14	112.2 (2)
C2—C1—C6	120.7 (2)	C14—N13—C18	108.4 (2)
C1—C2—C20	120.9 (2)	N13—C14—C15	110.5 (2)
C1—C2—C3	118.2 (2)	C14—C15—N16	110.9 (2)
C3—C2—C20	120.8 (2)	C15—N16—C19	111.3 (2)
C2—C3—C4	123.0 (2)	C15—N16—C17	108.7 (2)
C3—C4—C8	120.2 (2)	C17—N16—C19	112.4 (2)
C3—C4—C5	117.0 (2)	N16—C17—C18	111.7 (2)
C5—C4—C8	122.8 (2)	N13—C18—C17	111.0 (2)
C4—C5—C6	122.1 (2)	C2—C20—N21	112.9 (2)
C1—C6—C5	119.0 (2)	C20—N21—C26	110.5 (2)
C5—C6—C12	120.4 (2)	C20—N21—C22	110.8 (2)
C1—C6—C12	120.6 (2)	C22—N21—C26	109.5 (2)
C4—C8—C11	112.0 (2)	N21—C22—C23	111.3 (2)
C4—C8—C10	109.1 (2)	C22—C23—N24	111.0 (2)
C4—C8—C9	110.3 (2)	C23—N24—C27	111.9 (2)
C10—C8—C11	107.6 (2)	C23—N24—C25	109.1 (2)
C9—C8—C11	108.7 (2)	C25—N24—C27	111.2 (3)
C9—C8—C10	108.8 (2)	N24—C25—C26	110.9 (2)
C6—C12—N13	113.8 (2)	N21—C26—C25	110.1 (2)

Cell refinement and data reduction: *SDP* (Frenz, 1978). Structure solution: *SHELXS*86 (Sheldrick, 1985). Structure refinement: *SHELX*76 (Sheldrick, 1976). Molecular diagrams: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *PARST* (Nardelli, 1983). Computers used: VAX 730, MicroVAX II.

SSSR thanks the University of Madras for providing a fellowship.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: HA1065). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.

Allinger, N. L., Carpenter, J. G. D. & Karkowski, F. M. (1965). *J. Am. Chem. Soc.* **87**, 1232–1236.

Bassi, I. W. & Scordamagila, R. (1977). *Macromol. Chem.* **178**, 2063–2070.

Domenicano, A., Vaciago, A. & Coulsun, C. A. (1975). *Acta Cryst.* **B31**, 221–234.

Frenz, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – a Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution. Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft Univ. Press.

Hodgkin, J. H. (1984). *Aust. J. Chem.* **37**, 2371–2378.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures*. Univ. of Cambridge, England.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Sbit, M., Dupont, L., Dideberg, O., Liegeois, J. F. & Delarge, J. (1992). *Acta Cryst.* **C48**, 1851–1853.

Schwarzenbach, D. (1968). *J. Chem. Phys.* **48**, 4134–4140.

Shanmuga Sundara Raj, S., Ponnuswamy, M. N., Shanmugam, G. & Kandaswamy, M. (1993). *J. Crystallogr. Spectrosc. Res.* **23**, 607–610.

Shanmuga Sundara Raj, S., Velmurugan, D., Ponnuswamy, M. N., Shanmugam, G. & Kandaswamy, M. (1994). *J. Chem. Cryst.* **24**, 187–191.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* Univ. of Cambridge, England.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.

Acta Cryst. (1994). C**50**, 2009–2010

4-Chloro-2,6-bis(morpholinomethyl)phenol

S. Shanmuga Sundara Raj, D. Velmurugan* and E. Subramanian

Department of Crystallography and Biophysics,†
University of Madras, Guindy Campus,
Madras 600 025, India

M. Kandaswamy

Department of Inorganic Chemistry,
University of Madras, Guindy Campus,
Madras 600 025, India

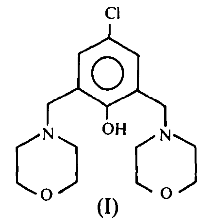
(*Received* 20 *May* 1993; *accepted* 10 *January* 1994)

Abstract

In the title compound, $C_{16}H_{23}ClN_2O_3$, both morpholino rings adopt chair conformations with an angle of 49.4 (1)° between their mean planes. The maximum puckering amplitude of the morpholino rings is calculated as $\varphi_T = 0.57$ Å for both rings. The molecules are held together by van der Waals forces and one C—H···O hydrogen bond of 3.298 (2) Å.

Comment

The ligand was prepared by a modification of a procedure reported in the literature (Hodgkin, 1984), employing the Mannich reaction. Each morpholino ring assumes a perfect chair conformation. The molecular geometry is in agreement with that of similar molecules except that the average C—O bond length in the morpholino rings, 1.419 Å, is slightly greater than the literature value of 1.364 Å (Allen *et al.*, 1987; Swaminathan, Sundaralingam, Chattopathyaya & Reese, 1980; Baydar, Boyd, Stride & Lindley, 1984; Shanmuga Sundara Raj, Ponnuswamy, Shanmugam & Kandaswamy, 1993; Ianelli *et al.*, 1992*a*,*b*). The mean planes passing through the four C atoms of the morpholino rings *A* and *B* make dihedral angles of 67.4 (1) and 70.3 (1)°, respectively, with the plane of the phenyl ring, and are inclined to each other at 49.4 (1)°.



(I)

An intermolecular short contact between the atoms C9 and O18 [$-x+1$, $-y$, $-z+1$; 3.298 (2) Å] is suggestive of a C—H···O hydrogen bond. In addition, intramolecular contact distances involving atoms C14 and O21 [2.851 (2) Å], and N8 and O21 [2.750 (2) Å] are suggestive of hydrogen-bonded interactions. The packing of the molecules is stabilized mainly by van der Waals interactions.

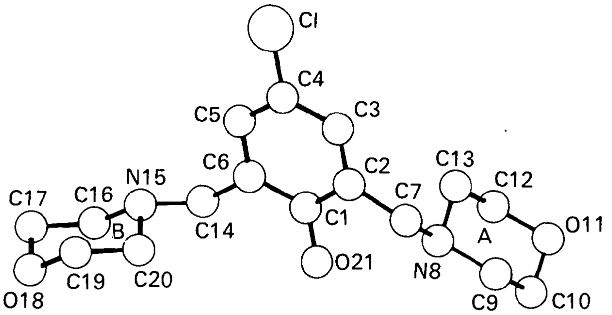


Fig. 1. Perspective view of the molecule with atomic numbering scheme.

Experimental

Crystal data

$C_{16}H_{23}ClN_2O_3$
$M_r = 326.82$
Monoclinic
$P2_1/c$
$a = 10.798$ (2) Å
$b = 10.771$ (3) Å
$c = 14.235$ (4) Å
$\beta = 94.65$ (2)°
$V = 1650.1$ (7) Å^3
$Z = 4$
$D_x = 1.32$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 22 reflections
$\theta = 15$–25°
$\mu = 2.18$ mm^{-1}
$T = 295$ K
Needle
0.25 × 0.22 × 0.15 mm
White

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
$T_{\min} = 0.984$, $T_{\max} = 0.999$
3329 measured reflections
3065 independent reflections
2803 observed reflections [$I \geq 3\sigma(I)$]

$R_{\rm int} = 0.013$
$\theta_{\max} = 70$°
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 17$
3 standard reflections
frequency: 120 min
intensity variation: ≤ 1.35%

---

† DCB Contribution No. 834.