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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

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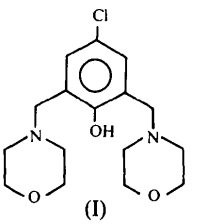
Abstract

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)°.

† DCB Contribution No. 834.



(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.

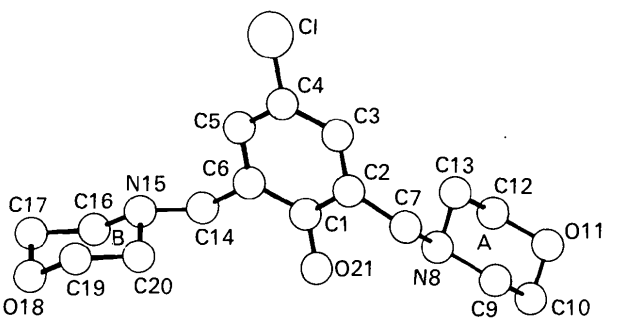


Fig. 1. Perspective view of the molecule with atomic numbering scheme.

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

Refinement

Refinement on F	$w = 1/[\sigma^2(F) + 0.024F^2]$
$R = 0.052$	$(\Delta/\sigma)_{max} = 0.001$
$wR = 0.076$	$\Delta\rho_{max} = 0.231$ e Å^{-3}
$S = 0.73$	$\Delta\rho_{min} = -0.347$ e Å^{-3}
2803 reflections	Extinction correction: none
291 parameters	Atomic scattering factors from *SHELX*76 (Sheldrick, 1976)
All H-atom parameters refined	

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

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

	x	y	z	B_{eq}
C1	0.6172 (2)	−0.0809 (1)	0.7117 (1)	3.16 (5)
C2	0.6024 (2)	0.0047 (1)	0.7842 (1)	3.20 (5)
C3	0.7052 (2)	0.0648 (2)	0.8268 (1)	3.73 (4)
C4	0.8213 (2)	0.0408 (2)	0.7951 (1)	4.27 (4)
C5	0.8358 (2)	−0.0405 (2)	0.7222 (1)	3.99 (5)
C6	0.7349 (2)	−0.1032 (1)	0.6793 (1)	3.43 (5)
C7	0.4747 (2)	0.0321 (1)	0.8139 (1)	3.36 (5)
N8	0.4043 (1)	−0.0817 (1)	0.8314 (1)	2.90 (5)
C9	0.2819 (2)	−0.0485 (2)	0.8617 (1)	3.56 (5)
C10	0.2108 (2)	−0.1633 (2)	0.8847 (2)	4.64 (5)
O11	0.2763 (1)	−0.2340 (1)	0.9566 (1)	4.64 (4)
C12	0.3934 (2)	−0.2694 (2)	0.9267 (1)	4.28 (5)
C13	0.4703 (2)	−0.1580 (1)	0.9049 (1)	3.31 (5)
C14	0.7508 (2)	−0.1927 (2)	0.5997 (1)	4.06 (5)
N15	0.8201 (1)	−0.1370 (1)	0.5262 (1)	3.21 (5)
C16	0.8469 (2)	−0.2296 (2)	0.4558 (1)	4.02 (5)
C17	0.9164 (2)	−0.1704 (2)	0.3800 (1)	4.72 (5)
O18	0.8489 (2)	−0.0706 (2)	0.3356 (1)	5.78 (5)
C19	0.8226 (3)	0.0197 (2)	0.4039 (1)	5.39 (7)
C20	0.7503 (2)	−0.0359 (2)	0.4793 (1)	4.32 (5)
O21	0.5167 (1)	−0.1441 (1)	0.6714 (1)	3.98 (4)
Cl	0.9492 (1)	0.1178 (1)	0.8503 (1)	7.25 (3)

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

C1—C2	1.403 (2)	N8—C13	1.468 (2)
C1—C6	1.408 (3)	C9—C10	1.506 (3)
C1—O21	1.367 (2)	C10—O11	1.418 (3)
C2—C3	1.382 (3)	O11—C12	1.419 (2)
C2—C7	1.504 (3)	C12—C13	1.506 (3)
C3—C4	1.391 (3)	C14—N15	1.464 (2)
C4—C5	1.377 (3)	N15—C16	1.460 (2)
C4—Cl	1.742 (2)	N15—C20	1.455 (2)
C5—C6	1.381 (3)	C16—C17	1.506 (3)
C6—C14	1.508 (2)	C17—O18	1.418 (3)
C7—N8	1.474 (2)	O18—C19	1.420 (3)
N8—C9	1.468 (2)	C19—C20	1.502 (3)
C6—C1—O21	119.0 (1)	C7—N8—C9	109.7 (1)
C2—C1—O21	120.1 (2)	C9—N8—C13	108.9 (1)
C2—C1—C6	120.9 (2)	N8—C9—C10	110.6 (2)
C1—C2—C7	119.9 (2)	C9—C10—O11	111.4 (2)
C1—C2—C3	119.7 (2)	C10—O11—C12	109.4 (2)
C3—C2—C7	120.5 (1)	O11—C12—C13	111.6 (2)
C2—C3—C4	118.9 (2)	N8—C13—C12	110.4 (1)
C3—C4—Cl	117.9 (2)	C6—C14—N15	111.8 (1)
C3—C4—C5	121.6 (2)	C14—N15—C20	111.0 (2)
C5—C4—Cl	120.5 (2)	C14—N15—C16	110.6 (1)
C4—C5—C6	120.7 (2)	C16—N15—C20	108.7 (1)
C1—C6—C5	118.2 (2)	N15—C16—C17	109.9 (2)
C5—C6—C14	120.7 (2)	C16—C17—O18	111.9 (2)
C1—C6—C14	121.1 (2)	C17—O18—C19	109.8 (2)
C2—C7—N8	112.4 (1)	O18—C19—C20	111.2 (2)
C7—N8—C13	111.0 (1)	N15—C20—C19	110.3 (2)
C9—N8—C13—C12	55.3 (2)	C16—N15—C20—C19	−57.8 (2)
C13—N8—C9—C10	−55.5 (2)	C20—N15—C16—C17	57.0 (2)
N8—C9—C10—O11	58.5 (2)	N15—C16—C17—O18	−58.0 (2)
C9—C10—O11—C12	−59.3 (2)	C16—C17—O18—C19	57.8 (2)
C10—O11—C12—C13	59.3 (2)	C17—O18—C19—C20	−58.1 (3)
O11—C12—C13—N8	−58.3 (2)	O18—C19—C20—N15	59.2 (2)

Data collection: Enraf–Nonius CAD-4 software. Cell refinement: *SDP* (Frenz, 1978). Program used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *PARST* (Nardelli, 1983). All calculations were carried out on VAX730 and MicroVAX II computers.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1066). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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