Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.146$
$S = 1.042$
1375 reflections
102 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0845P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.33$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C6	1.219 (2)	N1—C1	1.339 (2)
N1—C5	1.333 (2)	N2—C6	1.356 (2)
O1—C6—N2	122.7 (1)	N2—C6—C5	114.7 (1)
O1—C6—C5	122.6 (2)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1N2···N1	0.89 (2)	2.36 (2)	2.678 (2)	101 (2)
N2—H1N2···O1^{i}	0.89 (2)	2.28 (2)	3.124 (2)	160 (2)
C2—H2···O1^{ii}	0.97 (2)	2.58 (2)	3.220 (2)	124 (2)

Symmetry codes: (i) $x, -y, z - \frac{1}{2}$; (ii) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$.

All the H atoms were refined. C—H distances are in the range 0.97 (2)–0.99 (2) Å.

Data collection: *SMART* (Siemens, 1996*a*). Cell refinement: *SAINT* (Siemens, 1996*b*). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *XP* in *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1420). Services for accessing these data are described at the back of the journal.

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4-Chloro-2-(morpholinomethyl)phenol

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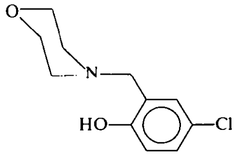
Abstract

The title compound, $C_{11}H_{14}ClNO_2$, crystallizes in the triclinic system with two independent molecules in the asymmetric unit. The molecular packing is governed by C—H···O hydrogen bonds leading to the formation of dimeric pairs.

Comment

Aminomethylphenols have been known for their uses as pigments (Fechner *et al.*, 1982; Fechner, Kranz & Polster, 1983; Fechner, Polster & Kranz, 1983), as insecticides (Nisato & Boveri, 1984) and as intermediates in medicine. Recently, a lot of attention has been paid to aminomethylphenols for their use as excellent mimics for the active site of enzymes. In this paper, we report the structure of a new aminomethylphenol, 4-chloro-2-(morpholinomethyl)phenol, (I), derived from morpholine and *p*-chlorophenol.



(I)

The asymmetric unit of (I) contains two independent molecules (Fig. 1). The two molecules are structurally similar in that the values of the corresponding bond lengths and angles are very similar, and are also comparable with reported values (Shanmuga Sundara Raj *et al.*, 1993; Shanmuga Sundara Raj, Ponnuswamy *et al.*, 1994; Shanmuga Sundara Raj, Velmurugan *et al.*, 1994).

The morpholine rings of the two molecules adopt chair conformations. The best planes of the rings pass

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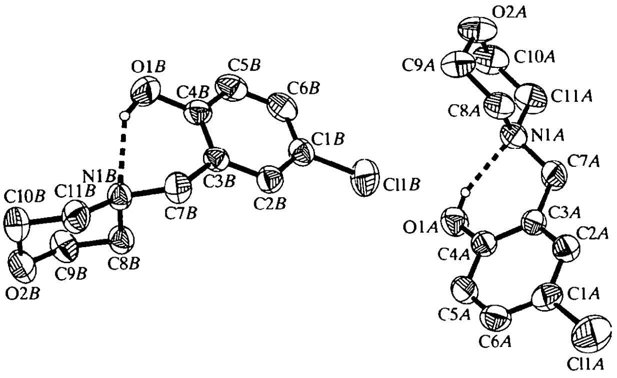

Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

through the C atoms, leaving the O and N atoms on either side with a total puckering amplitude, Q_T, of 0.579 (2) Å for molecule *A* and 0.580 (2) Å for molecule *B* (Cremer & Pople, 1975). The methylene substitution (C7) of the morpholine ring is in an equatorial position.

The phenyl and morpholine rings are oriented at angles of 83.1 (1) and 85.4 (1)° with respect to each other in molecules *A* and *B*, respectively. In addition to van der Waals interactions, intramolecular N—H···O and intermolecular C—H···O hydrogen bonds stabilize the packing of the molecules.

Experimental

To a solution of morpholine (3.5 ml, 40 mmol) and paraformaldehyde (1.8 g, 60 mmol) in dry benzene (80 ml) under a nitrogen atmosphere was added *p*-chlorophenol (40 mmol) at room temperature. The mixture was heated and kept at reflux for 6 h. The solvent was removed *in vacuo* and the crude product was recrystallized from petroleum ether and ethyl acetate to give crystals suitable for X-ray analysis.

Crystal data

$C_{11}H_{14}ClNO_2$
$M_r = 227.68$
Triclinic
$P\bar{1}$
$a = 9.3410\,(1)$ Å
$b = 9.8782\,(1)$ Å
$c = 12.4671\,(2)$ Å
$\alpha = 87.427\,(1)°$
$\beta = 88.548\,(1)°$
$\gamma = 88.604\,(1)°$
$V = 1148.53\,(3)$ Å^3
$Z = 4$
$D_x = 1.317$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 5089 reflections
$\theta = 2.06–38.60°$
$\mu = 0.313$ mm^{-1}
$T = 293\,(2)$ K
Block
0.46 × 0.34 × 0.30 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.870$, $T_{max} = 0.912$
15 271 measured reflections
11 354 independent reflections

4827 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 38.61°$
$h = -16 \rightarrow 14$
$k = -17 \rightarrow 17$
$l = -21 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.205$
$S = 1.007$
11 354 reflections
376 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0860P)^2 + 0.0027P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.380$ e Å^{-3}
$\Delta\rho_{min} = -0.436$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.022 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

	Molecule *A*	Molecule *B*
Cl1—C1	1.744 (2)	1.749 (2)
O1—C4	1.359 (2)	1.361 (2)
N1—C7—C3	112.0 (1)	111.8 (1)
C11—N1—C8—C9	56.5 (2)	57.1 (2)
C10—O2—C9—C8	59.7 (2)	58.8 (2)
N1—C8—C9—O2	−58.5 (2)	−58.6 (2)
C9—O2—C10—C11	−60.1 (3)	−59.3 (2)
C8—N1—C11—C10	−56.7 (2)	−57.5 (2)
O2—C10—C11—N1	59.4 (3)	59.3 (2)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*A*—H1O*A*···N1*A*	0.82	1.97	2.692 (2)	146
O1*B*—H1O*B*···N1*B*	0.82	2.00	2.717 (2)	146
C2*B*—H2*B*···O2*A*^{i}	0.97 (2)	2.42 (2)	3.328 (2)	156 (2)
C5*B*—H5*B*···O2*B*^{ii}	0.98 (3)	2.59 (3)	3.493 (2)	154 (2)

Symmetry codes: (i) $1 + x, y, z$; (ii) $x - 1, y, z$.

All H atoms except the hydroxy H atom (H10) were refined. C—H distances were in the range 0.87 (3)–1.05 (2) Å.

Data collection: *SMART* (Siemens, 1996*a*). Cell refinement: *SAINT* (Siemens, 1996*b*). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1421). Services for accessing these data are described at the back of the journal.

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5-Nitroisophthalic acid hydrate

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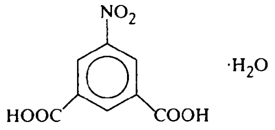
Abstract

In the title compound, $C_8H_5NO_6\cdot H_2O$, the carboxylic acid groups are coplanar with the phenyl ring, whereas the nitro group is twisted from it by 26.1 (3)°. In the crystal, the molecules are linked by O—H···O hydrogen bonds through the water molecules and by weak C—H···O hydrogen bonds involving the nitro groups, to form infinite zigzag sheets.

Comment

Crystal engineering (Desiraju, 1989) offers the opportunity to design and construct solids with a defined architecture (Goswami *et al.*, 1999) from relatively simple starting components. Co-operative non-covalent bonding interactions control the nucleation of supramolecular assemblies which grow into well defined supramolecular arrays (Duchamp & Marsh, 1969) and finally into a macroscopic aggregate, namely, a crystal. Thus, hydrogen-bonding units comprising a sequence of acceptor ($-NO_2$ or $-C{=}O$) and donor ($-NH_2$ or $-OH$) sites have been used to direct the formation of a variety of organic supramolecular entities (Whitesides *et al.*, 1991). The basic motif is an open network comprising rings of three molecules of 5AIPA (5AIPA is 5-aminoisophthalic acid) and three molecules of 5NIPA (5NIPA is 5-nitroaminoisophthalic acid) hydrogen bonded together through pairs of O—H···O and N—H···O hydrogen bonds between two carbonyl groups and between amine nitro groups. In a continuation of this general line of study, the title compound, (I), was studied by X-ray crystallography and the results are presented here.



(I)

The X-ray structure of a water-free pseudo-polymorph of 5NIPA and angular parameters for several substituents of benzene rings, including NO_2 and COOH, were calculated using regression by Colapietro *et al.* (1984). In addition, Domenicano *et al.* (1990) have performed extensive work on substituted benzene derivatives using *ab initio* molecular-orbital calculations,

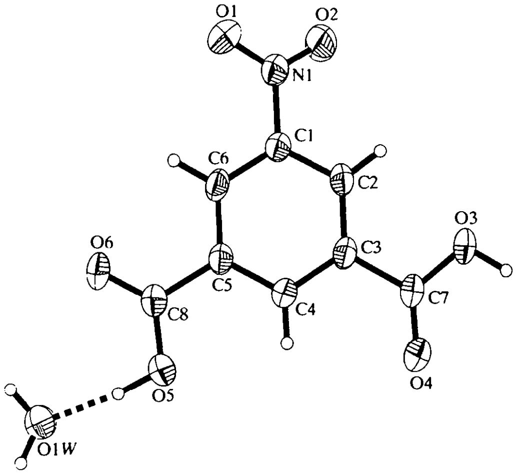


Fig. 1. The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms are drawn as circles of arbitrary radii.

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