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Key indicators

Single-crystal X-ray study

T = 298 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.039

wR factor = 0.133

Data-to-parameter ratio = 17.4

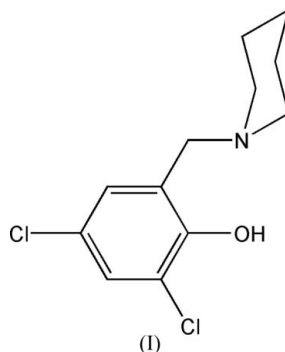
For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

2,4-Dichloro-6-(piperidin-1-ylmethyl)phenol

In the title compound, $\text{C}_{12}\text{H}_{15}\text{Cl}_2\text{NO}$, the piperidine ring adopts a chair conformation. An intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond is observed. The packing of the molecules in the crystal structure is stabilized by $\pi-\pi$ interactions and $\text{Cl}\cdots\text{Cl}$ contacts.

Comment

The pharmacological properties of piperidine derivatives have led to many studies of the design and synthesis of these compounds (Hu *et al.*, 2002; Walker *et al.*, 2005). In addition, a number of these derivatives can act as complexing reagents with metal ions. We have previously studied the application of aminophenol derivatives as ion size recognition reagents (Hirayama *et al.*, 2001), and in this work, describe the crystal structure of 2,4-dichloro-6-(piperidin-1-ylmethyl)phenol, (I), which would be expected to act as an effective chelating reagent.



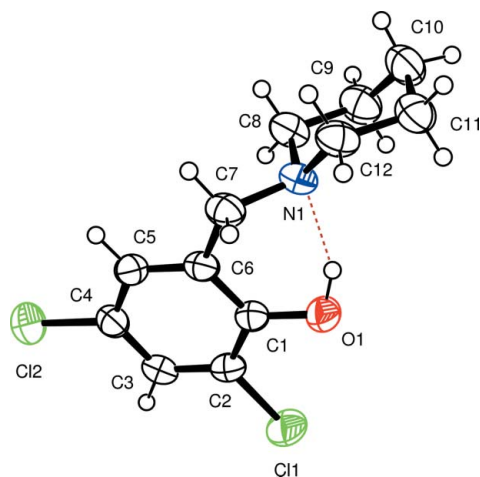
Compound (I) crystallizes in the monoclinic space group $P2_1/c$, with one molecule in the asymmetric unit. The bond lengths and angles observed in the piperidylmethyl group are all in the normal ranges and comparable with those of other related compounds (Deng *et al.*, 2001; Yuan *et al.*, 2004). The piperidine ring adopts the usual chair conformation. The torsion angles $\text{C1}-\text{C6}-\text{C7}-\text{N1}$ and $\text{C5}-\text{C6}-\text{C7}-\text{N1}$ are $44.20(18)$ and $-139.28(14)^\circ$, respectively. There is an intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond (Table 2).

In the crystal structure, the shortest intermolecular $\text{C}\cdots\text{C}$ contact distance is $3.533(2) \text{ \AA}$ for $\text{C4}\cdots\text{C6}^{\text{i}}$ [symmetry code: (i) $-x, -y, -z$]. In addition, weak intermolecular $\text{Cl}\cdots\text{Cl}$ contacts are observed. The contact distances $\text{Cl1}\cdots\text{Cl1}^{\text{ii}}$ and $\text{Cl1}\cdots\text{Cl2}^{\text{iii}}$ are $3.4596(6)$ and $3.5734(6) \text{ \AA}$, respectively [symmetry code: (ii) $-x, -y, 1-z$; (iii) $x, -\frac{1}{2}-y, \frac{1}{2}+z$]. The packing of the molecules in the crystal structure is stabilized by $\pi-\pi$ interactions and $\text{Cl}\cdots\text{Cl}$ contacts between dichlorobenzene groups.

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**Figure 1**

A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size. The dashed line indicates the O—H...N hydrogen bond.

Experimental

Compound (I) was prepared by the Mannich reaction. 2,4-Dichlorophenol (6.52 g, 40 mmol), piperidine (3.41 g, 40 mmol) and paraformaldehyde (1.20 g, 40 mmol) in methanol (80 ml) were refluxed for 6 h. The mixture was cooled to room temperature, then the solvent was evaporated under vacuum. The resulting oil was extracted with chloroform and evaporated to yield a solid. The product was recrystallized from methanol to give colourless crystals suitable for X-ray analysis. Yield 52.6%; m.p. 335.0–335.4 K. Analysis calculated for $C_{12}H_{15}Cl_2NO$: C 55.40, H 5.81, N 5.38%; found: C 55.49, H 5.87, N 5.37%. 1H NMR ($CDCl_3$, p.p.m., 400 MHz): 1.46–1.69 (*m*, 6H, CH_2), 2.53 (*brs*, 4H, CH_2), 3.65 (*s*, 2H, CH_2), 6.85 (*d*, $J = 2.5$ Hz, 1H, ArH), 7.24 (*d*, $J = 2.5$ Hz, 1H, ArH), 10.2 (*brs*, 1H, OH).

Crystal data

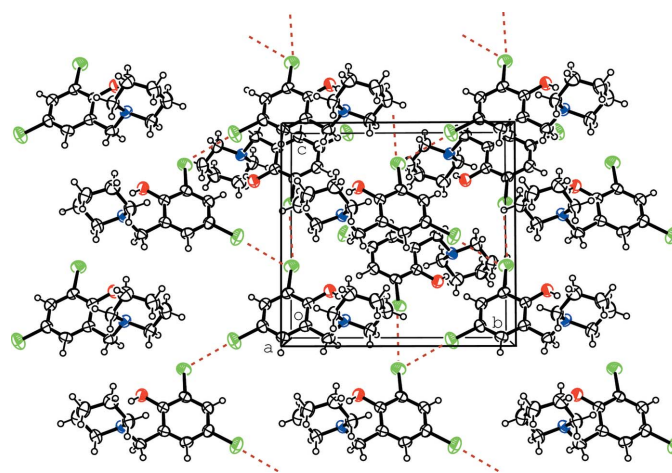
$C_{12}H_{15}Cl_2NO$	$D_x = 1.356 \text{ Mg m}^{-3}$
$M_r = 260.15$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
$a = 9.571(4) \text{ \AA}$	$\theta = 15.5\text{--}17.1^\circ$
$b = 11.794(4) \text{ \AA}$	$\mu = 0.49 \text{ mm}^{-1}$
$c = 11.345(5) \text{ \AA}$	$T = 298.1 \text{ K}$
$\beta = 95.89(3)^\circ$	Prism, colourless
$V = 1273.9(9) \text{ \AA}^3$	$0.50 \times 0.20 \times 0.20 \text{ mm}$
$Z = 4$	

Data collection

Rigaku AFC-7R diffractometer	$\theta_{\max} = 27.5^\circ$
ω - 2θ scans	$h = -12 \rightarrow 12$
Absorption correction: none	$k = -15 \rightarrow 0$
3619 measured reflections	$l = -8 \rightarrow 14$
2937 independent reflections	3 standard reflections
2773 reflections with $F^2 > 2\sigma(F^2)$	every 150 reflections
$R_{\text{int}} = 0.026$	intensity decay: 2.4%

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.039$	$w = 1/[0.0034F_o^2 + 1\sigma(F_o^2)]/(4F_o^2)$
$wR(F^2) = 0.133$	$(\Delta\sigma)_{\max} < 0.001$
$S = 1.01$	$\Delta\rho_{\max} = 0.29 \text{ e \AA}^{-3}$
2776 reflections	$\Delta\rho_{\min} = -0.52 \text{ e \AA}^{-3}$
160 parameters	

**Figure 2**

The packing of the molecules of (I), viewed down the *a* axis, with Cl...Cl contacts shown as dashed lines.

Table 1

Selected geometric parameters (\AA , $^\circ$).

O1—C1	1.3559 (19)	N1—C8	1.466 (2)
N1—C7	1.474 (2)	N1—C12	1.472 (2)
C2...C5 ⁱ	3.609 (2)	Cl1...Cl1 ⁱⁱ	3.4596 (17)
C4...C6 ⁱ	3.533 (2)	Cl1...Cl2 ⁱⁱⁱ	3.5734 (17)
C7—N1—C8	110.88 (12)	N1—C7—C6	111.09 (12)
C7—N1—C12	111.67 (12)	N1—C8—C9	111.12 (14)
C8—N1—C12	110.63 (12)	N1—C12—C11	109.91 (14)

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

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1...N1	0.86	1.87	2.6456 (18)	149

The H atom of the hydroxyl group was found in a difference Fourier map. The other H atoms were placed in idealized positions with $C-H = 0.95 \text{ \AA}$. All the H atoms were refined as riding, with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$.

Data collection: *WinAFC* (Rigaku/MSC, 2004); cell refinement: *WinAFC*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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