C(4)	0.7467 (5)	0.8867 (4)	0.3994 (9)	4.8 (1)
C(5)	0.6969 (6)	0.8521 (4)	0.5851 (9)	4.6 (1)
C(6)	0.5519 (5)	0.8448 (4)	0.6094 (8)	3.8 (1)

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

P—F	1.549 (3)	C(3)—C(4)	1.390 (7)
P—O(2)	1.482 (3)	C(4)—C(5)	1.372 (8)
P—O(1)	1.554 (4)	C(5)—C(6)	1.385 (7)
P—O(3)	1.488 (4)	C(6)—C(1)	1.378 (7)
C(1)—N	1.481 (6)	N—H(7)	0.86 (5)
C(1)—C(2)	1.369 (7)	N—H(8)	0.91 (5)
C(2)—C(3)	1.383 (7)	N—H(9)	1.12 (6)
F—P—O(2)	106.6 (2)	N—C(1)—C(6)	119.0 (8)
F—P—O(1)	103.3 (4)	C(6)—C(1)—C(2)	121.9 (9)
F—P—O(3)	106.9 (4)	C(1)—C(2)—C(3)	118.7 (7)
O(2)—P—O(1)	109.1 (4)	C(2)—C(3)—C(4)	120.1 (9)
O(1)—P—O(3)	112.5 (4)	C(3)—C(4)—C(5)	120.4 (10)
O(2)—P—O(3)	117.2 (5)	C(4)—C(5)—C(6)	119.8 (10)
N—C(1)—C(2)	119.0 (8)	C(5)—C(6)—C(1)	119.2 (9)

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*
N—H(7)···O(3^{i})	0.86 (5)	1.98 (5)	2.822 (5)
N—H(8)···O(2^{ii})	0.91 (5)	1.97 (5)	2.824 (5)
N—H(9)···O(2^{iii})	1.12 (6)	1.62 (6)	2.733 (5)
O(1)—H(1)···O(3^{iv})	0.92 (6)	1.64 (6)	2.554 (6)

Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (iii) $1 - x, 1 - y, 1 - z$; (iv) $2 - x, 1 - y, 1 - z$.

Data were corrected for Lorentz–polarization effects. The structure was solved with the aid of *MULTAN*11/82 (Main *et al.*, 1982) and subsequent difference Fourier syntheses. All calculations were performed with the *MolEN* (Fair, 1990) programs on a Digital MicroVAX 3100 computer.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: DU1082). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Chloroxylenol, C_8H_9ClO

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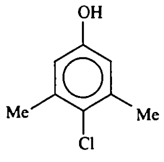
Abstract

The crystal structure of 4-chloro-3,5-dimethylphenol has been determined. There are two independent molecules in the asymmetric unit and the molecules are held together by hydrogen bonding. Each O atom acts as both an acceptor and a donor in the intermolecular hydrogen-bonding scheme.

Comment

The crystal structures of numerous simple aromatic compounds were studied many years ago (Wyckoff, 1969) but today's instrumentation now results in molecular geometries with e.s.d.'s that can often be lower by a factor of ten.

4-Chlorophenol is used as an intermediate in the synthesis of some biocides, fungicides and drugs (Kroschwitz & Howe-Grant, 1992), whereas 4-chloro-3,5-dimethylphenol is a chlorinated phenolic antiseptic which is a bactericide against most Gram-positive bacteria (Reynolds, 1993). The crystal structures of two polymorphic forms of 4-chlorophenol have been reported (Perrin & Michel, 1973) and the molecule is known to form complexes (Shipley & Wallwork, 1967) and clathrates (Ung, Bishop, Craig, Dance & Scudder, 1993) with other molecules. Accurate studies of intermolecular forces exhibited by such molecules are of increasing importance because of the possibilities offered by crystal engineering (Aakeröy & Seddon, 1993). The previously unknown crystal structure of 4-chloro-3,5-dimethylphenol, (1), is now presented.



(1)

The atomic arrangement in molecule *A* of (1) is shown in Fig. 1 and the crystal packing of (1) is shown in Fig. 2. There are no unusual intramolecular bond lengths and angles and a comparison of the two molecules of the asymmetric unit shows no great differences between geometries. As expected, the shortest aromatic bond lengths involve C1 which is attached to an electron withdrawing group and the hydroxy H atom lies in the region of the larger of the two C—C—O bond angles (Fig. 1). The largest geometrical difference in torsion angles is for Cl—C4—C5—C7 which is 2.3 (3) in molecule *A* and −0.2 (3)° in molecule *B*. The molecules are essentially planar and mean-plane calculations through the ring C atoms, indicating small differences between molecules for out-of-plane atoms, are shown in Table 3.

As can be seen from Fig. 2, the molecules pack in both a parallel and perpendicular arrangement; the two aromatic rings of the asymmetric unit are inclined to each other by 89.2 (1)°. The hydroxy groups are involved in intermolecular hydrogen bonding where the O atom acts as both an acceptor and a donor. The hydrogen bonds are shown as dotted lines in Fig. 3; it can be seen that each molecule is linked to two other molecules by these interactions. Geometrical details of the hydrogen bonding are given in Table 4.

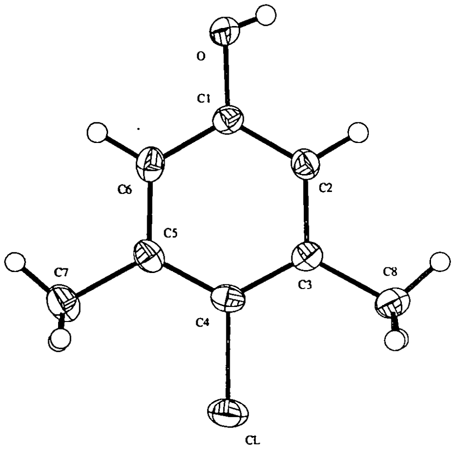


Fig. 1. The atomic arrangement in molecule *A* of (1). Displacement ellipsoids are shown at the 50% probability level.

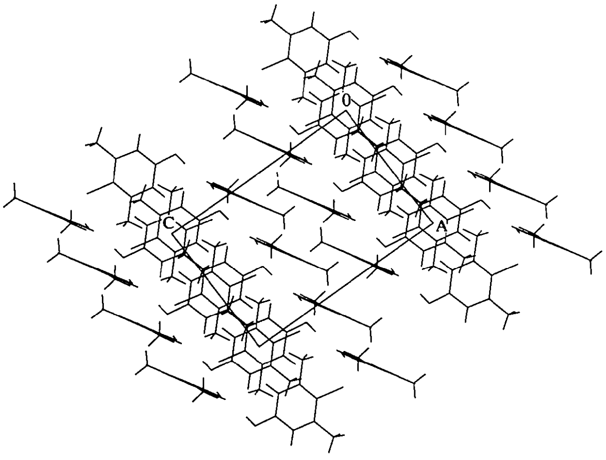


Fig. 2. A view on the 010 plane.

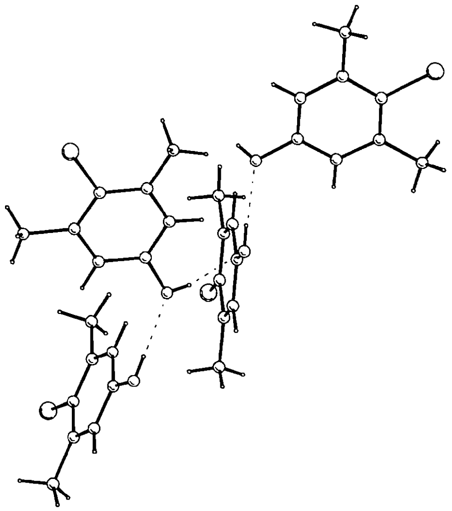


Fig. 3. The hydrogen-bonding scheme.

Interactions between Cl atoms can be very favourable (Sarma & Desiraju, 1986) but here the closest Cl···Cl separation is 4.013 (2) Å (across a centre of symmetry). These values compare with Cl···Cl interactions of 3.574 and 3.727 Å in 2-amino-4-chlorophenol (Ashfaquzzaman & Pant, 1979) and 4.191 (2) Å in 4-chloro-2-isopropyl-5-methylphenol (Rantsordas & Perrin, 1989). There are no significant interactions and the smallest Cl···O non-bonded distances are 3.709 (2) for molecule *A* and 3.700 (2) Å for molecule *B* (both across a centre of symmetry).

Experimental

The title compound was obtained from Aldrich.

Crystal data

C_8H_9ClO Mo $K\alpha$ radiation
M_r = 156.6 λ = 0.71069 Å

Monoclinic
$P2_1/a$
$a = 8.537\,(4)$ Å
$b = 13.936\,(2)$ Å
$c = 12.839\,(3)$ Å
$\beta = 92.92\,(3)°$
$V = 1525.5\,(8)$ Å^3
$Z = 8$
$D_x = 1.364$ Mg m^{-3}

Cell parameters from 250 reflections
$\theta = 2.16–25.03°$
$\mu = 0.424$ mm^{-1}
$T = 140\,(2)$ K
Tablet
0.2 × 0.2 × 0.1 mm
Colourless

Data collection

Delft Instruments FAST diffractometer
Absorption correction: none
5996 measured reflections
2251 independent reflections
1887 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0462$
$\theta_{\rm max} = 25.03°$
$h = -9 \rightarrow 7$
$k = -15 \rightarrow 15$
$l = -13 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0407$
$wR(F^2) = 0.0979$
$S = 1.066$
2251 reflections
197 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0621P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$

$\Delta\rho_{\rm max} = 0.514$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.304$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

$U_{\rm iso}$ for H atoms; $U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for others.

	x	y	z	$U_{\rm iso}/U_{\rm eq}$
Cl	0.22774 (7)	1.12372 (4)	0.44203 (5)	0.0349 (2)
O	0.6301 (2)	0.81811 (13)	0.28752 (14)	0.0299 (4)
C1	0.5322 (3)	0.88825 (15)	0.3248 (2)	0.0218 (5)
C2	0.3988 (2)	0.8650 (2)	0.3753 (2)	0.0222 (5)
C3	0.3026 (2)	0.9368 (2)	0.4127 (2)	0.0222 (5)
C4	0.3480 (2)	1.0320 (2)	0.3977 (2)	0.0239 (5)
C5	0.4839 (2)	1.0575 (2)	0.3489 (2)	0.0240 (5)
C6	0.5755 (2)	0.9835 (2)	0.3121 (2)	0.0243 (5)
C7	0.5333 (3)	1.1610 (2)	0.3368 (2)	0.0329 (6)
C8	0.1576 (3)	0.9104 (2)	0.4677 (2)	0.0308 (6)
Cl′	1.07767 (7)	0.89689 (4)	−0.19492 (5)	0.0345 (2)
O′	0.9440 (2)	0.84192 (11)	0.24536 (12)	0.0274 (4)
C1′	0.9697 (2)	0.85655 (15)	0.1414 (2)	0.0216 (5)
C2′	0.8504 (2)	0.85229 (15)	0.0648 (2)	0.0240 (5)
C3′	0.8817 (2)	0.86523 (15)	−0.0401 (2)	0.0235 (5)
C4′	1.0368 (2)	0.88170 (15)	−0.0638 (2)	0.0243 (5)
C5′	1.1596 (2)	0.88608 (15)	0.0126 (2)	0.0256 (6)
C6′	1.1226 (2)	0.87341 (15)	0.1154 (2)	0.0241 (5)
C7′	1.3268 (3)	0.9033 (2)	−0.0145 (2)	0.0346 (6)
C8′	0.7521 (3)	0.8602 (2)	−0.1233 (2)	0.0344 (6)
HO	0.5908 (30)	0.7738 (21)	0.2874 (23)	0.043 (10)
HO′	0.8552 (30)	0.8330 (18)	0.2521 (20)	0.034 (8)

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

	Molecule *A*	Molecule *B*
Cl—C4	1.752 (2)	1.749 (2)
O—C1	1.387 (3)	1.378 (3)
C1—C2	1.377 (3)	1.381 (3)
C1—C6	1.390 (3)	1.384 (3)
C2—C3	1.395 (3)	1.398 (3)
C3—C4	1.399 (3)	1.393 (3)
C3—C8	1.502 (3)	1.500 (3)
C4—C5	1.392 (3)	1.400 (3)
C5—C6	1.391 (3)	1.384 (3)
C5—C7	1.514 (3)	1.505 (3)
C2—C1—O	121.6 (2)	122.4 (2)
O—C1—C6	117.6 (2)	117.2 (2)
C2—C1—C6	120.7 (2)	120.3 (2)
C1—C2—C3	120.6 (2)	120.8 (2)
C2—C3—C4	117.4 (2)	117.5 (2)
C2—C3—C8	120.0 (2)	120.7 (2)
C4—C3—C8	122.5 (2)	121.8 (2)
C5—C4—C3	123.1 (2)	122.7 (2)
C3—C4—Cl	118.4 (2)	118.0 (2)
C5—C4—Cl	118.4 (2)	119.3 (2)
C6—C5—C4	117.4 (2)	117.6 (2)
C6—C5—C7	120.5 (2)	120.4 (2)
C4—C5—C7	122.1 (2)	122.0 (2)
C1—C6—C5	120.7 (2)	121.1 (2)

Table 3. *Deviations* (Å) *from mean planes*

Only the first six ring C atoms were used in the derivation of each mean plane.

Plane 1							
C1	−0.008 (1)	C2	0.007 (1)	C3	0.000 (2)	C4	−0.007 (2)
C5	0.007 (1)	C6	0.001 (1)	C7	0.044 (4)	C8	0.017 (3)
Cl	−0.042 (3)	O	0.006 (3)	HO	−0.14 (3)		
Plane 2							
C1′	0.000 (1)	C2′	−0.002 (1)	C3′	0.003 (1)	C4′	−0.001 (1)
C5′	−0.002 (1)	C6′	0.002 (1)	C7′	−0.015 (4)	C8′	−0.005 (4)
Cl′	−0.010 (3)	O′	−0.036 (3)	HO′	−0.04 (3)		

Table 4. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O′—HO′···O	0.78 (3)	2.01 (3)	2.781 (3)	173 (3)
O—HO···O′$^{\rm i}$	0.70 (3)	2.10 (3)	2.776 (2)	163 (3)

Symmetry code: (i) $x - \frac{1}{2}, \frac{3}{2} - y, z$.

Data corrections were applied for Lorentz and polarization effects but not for absorption. There was no reasonable decay as indicated by the equivalence of common reflections measured at the beginning and end of data collection. The structure was solved by the Patterson method using *SHELXS*86 (Sheldrick, 1990) and from subsequent difference Fourier calculations using *SHELXL*93 (Sheldrick, 1993). The non-H atoms were freely refined with anisotropic displacement parameters. The H atoms of the hydroxy groups were also freely refined but with isotropic displacement parameters, and the remaining H atoms were allowed to ride on their attached C atoms with common displacement parameters according to their type (aromatic or methyl, molecule *A* or molecule *B*). Figures were prepared using *SNOOPI* (Davies, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1115). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Methyl 3,5-Dinitro-*trans*-cinnamate

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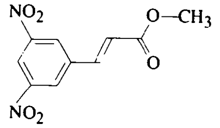
Abstract

The title compound, $C_{10}H_8N_2O_6$, forms C—H···O hydrogen-bonded inversion dimers in the crystal with a calculated (AM1) energy of −8.2 kcal mol^{-1} (1 cal = 4.184 J). These dimers in turn form a planar sheet structure which is stacked along the [100] direction.

Comment

The C—H···O hydrogen bonds were found to be structure determining in the crystal structure of 3,5-dinitrocinnamic acid, (2), wherein the carboxyl groups were related by the uncommon twofold axis rather than the expected inversion centre (Desiraju & Sharma, 1991). Furthermore, one of the nitro groups in this molecule is involved in a C—H···O self-recognition motif (Sharma, Panneerselvam, Pilati & Desiraju, 1993). To further investigate the C—H···O bond-forming ability of the 3,5-dinitrocinnamyl skeleton, we undertook the structure determination of the corresponding methyl ester, (1).



(1)

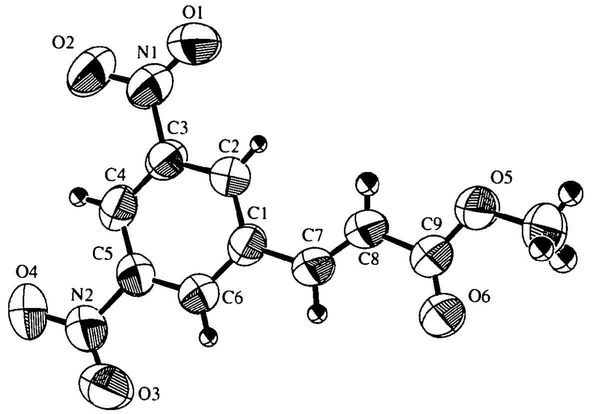


Fig. 1. An *ORTEP*II (Johnson, 1976) diagram of compound (1). Displacement ellipsoids are plotted at the 50% probability level. For clarity, H atoms are drawn as small circles.

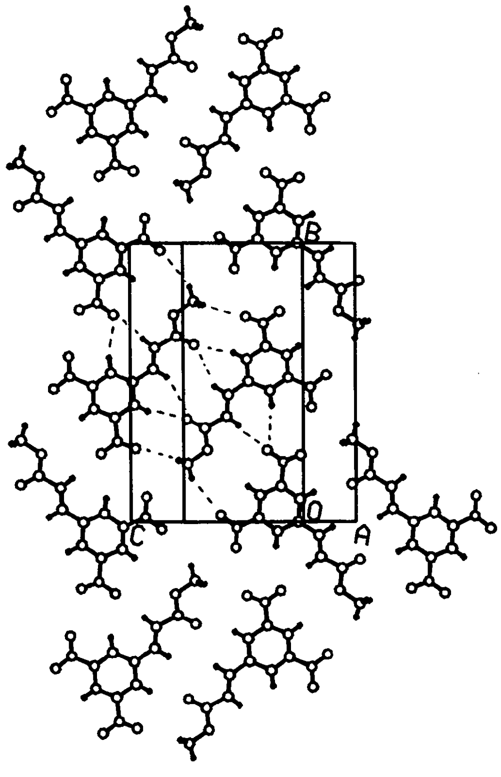


Fig. 2. The crystal structure of (1) along the [100] direction showing the planar structure.

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