Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

3,4,6-Trichloro-5-cyano-2-hydroxybenzamide

Doyle Britton

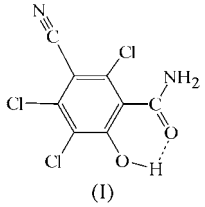
Department of Chemistry, University of Minnesota, Minneapolis, MN 55455-0431, USA
Correspondence e-mail: britton@chem.umn.edu

Received 7 January 2008
Accepted 10 January 2008
Online 9 February 2008

The title compound, $C_8H_3Cl_3N_2O_2$, forms crystals in which hydrogen-bonding and Cl···N interactions appear to be equally important to the structure. The molecules form ribbons held together alternately by cyclic $(CONH)_2$ and cyclic $(ClCCC{\equiv}N)_2$ interactions. The ribbons assemble into layers through Cl···Cl interactions. The layers are held together by NH···N≡C hydrogen bonds, as well as by π–π interactions.

Comment

In 3,5-dichloro-4-cyanobenzoic acid (Britton, 2006), the molecules are involved in cyclic $(COOH)_2$ interactions, graph set $R_2^2(8)$ (Etter, 1990), as well as in cyclic $(ClCCC{\equiv}N)_2$ interactions. Bernstein *et al.* (1995) have suggested that graph-set analysis might be extended to systems other than hydrogen bonding. In this spirit, cyclic Cl···N interactions can be described by the graph set $R_2^2(10)$, with the electron-acceptor Cl replacing H. The title compound, (I), was studied in a search for another example of the same phenomenon.



(I)

Fig. 1 shows the atom labelling and the anisotropic displacement ellipsoids of (I). The bond lengths and angles are normal. The plane of the benzamide group, excluding the H atoms, is rotated 31.2 (10)° out of the plane of the C_6 ring, while the plane of the NH_2 group is rotated another 10 (2)° out of the plane of the benzamide group. The hydroxy group is intramolecularly hydrogen bonded to atom O1, graph set $S(6)$; geometric details are given in Table 1.

One layer of the packing in (I) is shown in Fig. 2. The molecules form ribbons along the $[21\bar{1}]$ direction, held together by the anticipated $(CONH)_2$ cyclic hydrogen bonds and Cl···N interactions. Geometric data for the hydrogen bonds are given in Table 1. Data for the Cl···N interaction are: C4—Cl4···N2^{i} = 171.66 (5)°, Cl4···N2^{i} = 3.0809 (13) Å and Cl4···N2^{i}≡C8^{i} = 129.09 (11)° [symmetry code: (i) $2-x, 2-y, 1-z$]. The ribbons assemble into layers parallel to the (102) plane, held together by Cl···Cl interactions: C3—Cl3···Cl6^{ii} = 138.11 (5)°, Cl3···Cl6^{ii} = 3.3900 (5) Å and Cl3···Cl6^{ii}—C6^{ii} = 136.03 (4)° [symmetry code: (ii) $x, 1+y, z$].

The layers are held together by N—H···N≡C hydrogen bonds. Fig. 3 shows the hydrogen bonds between two molecules in adjacent layers; geometric details are given in Table 1. Pairs of molecules form cyclic dimers, graph set $R_2^2(16)$. In a single layer, alternate molecules form such dimers with adjacent layers on opposite sides of the original sheet.

It would be of interest to determine the structure of the corresponding carboxylic acid. It seems reasonable to suppose that similar layers of molecules might form, but that the stacking of the layers would be different in the absence of interlayer hydrogen bonds.

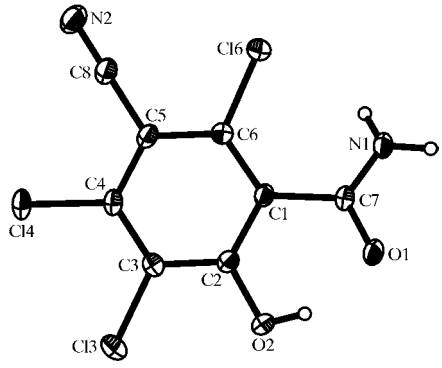


Figure 1
The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

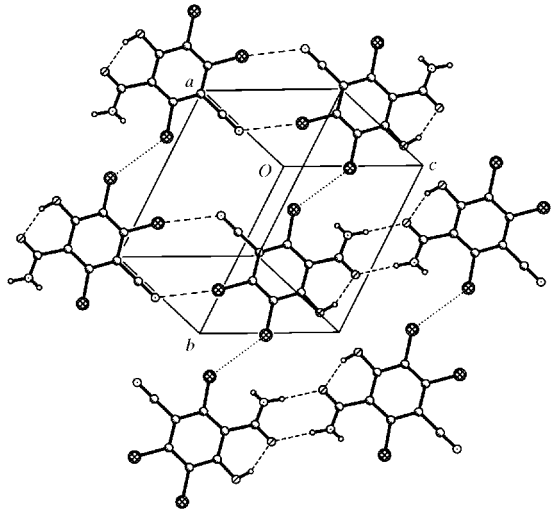


Figure 2
One layer of the crystal packing of (I). Hydrogen bonds and Cl···N≡C interactions are shown as dashed lines. The Cl···Cl interactions are shown as dotted lines.

 DOI: 10.1107/S0108270108001017

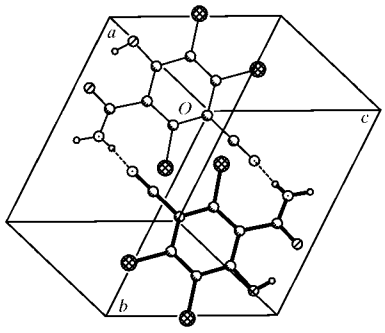


Figure 3
Adjacent molecules of (I) from two different layers. Hydrogen bonds are shown as dashed lines.

Experimental

The title compound was obtained from the Diamond Shamrock Corporation. The crystal used was from the original sample.

Crystal data

$C_8H_3Cl_3N_2O_2$
M_r = 265.47
Triclinic, $P\overline{1}$
a = 6.3020 (6) Å
b = 9.0437 (8) Å
c = 9.3822 (9) Å
α = 109.196 (1)°
β = 94.937 (1)°
γ = 104.076 (1)°
V = 481.72 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.93 mm^{-1}
T = 174 (2) K
0.45 × 0.30 × 0.25 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003; Blessing, 1995)
T_{min} = 0.74, T_{max} = 0.79
5659 measured reflections
2164 independent reflections
2036 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.063
S = 1.07
2164 reflections
149 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Distances and angles (Å, °) in the hydrogen bonds of (I).

X—H···Y—Z	X—H	X—H···Y	H···Y	H···Y—Z	X···Y
O2—H2···O1—C7	0.83 (2)	154 (2)	1.77 (2)	96.4 (6)	2.5542 (14)
N1—H11···O1^{i}—C7^{i}	0.89 (2)	167 (2)	2.07 (2)	123.6 (5)	2.9461 (15)
N1—H12···N2^{ii}—C8^{ii}	0.82 (2)	154 (2)	2.38 (2)	122.1 (5)	3.1417 (17)

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

The solution and refinement of the title compound were straightforward. The H-atom positions and isotropic displacement parameters were refined.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The author would like to thank the late Professor Paul G. Gassman and the Diamond Shamrock Corporation for providing the sample.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN3085). Services for accessing these data are described at the back of the journal.

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Britton, D. (2006). *Acta Cryst.* C**62**, o510–o514.
Bruker (2003). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Sheldrick, G. M. (2003). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.