Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,4-Dichlorobenzohydroxamic acid

Xian-Mei Shang,[a] Xiang-Gao Meng,[b] Ji-Zhou Wu[a] and Qing-Shan Li[c]*

[a]School of Pharmaceutical Science, Tongji Medical University, HUST, Wuhan 430030, People's Republic of China, [b]Department of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China, and [c]School of Pharmaceutical Science, Shanxi Medical University, Taiyuan 030001, People's Republic of China

Correspondence e-mail: qingshanl@yahoo.com

Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.100
Data-to-parameter ratio = 16.9

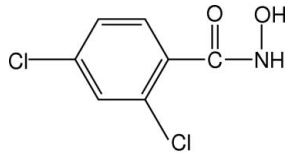
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 27 April 2005
Accepted 24 May 2005
Online 31 May 2005

The title compound, $C_7H_5Cl_2NO_2$, was prepared by reaction of methyl 2,4-dichlorobenzoate with excess NH_2OH in basic solution. In the crystal structure, the molecules are linked into a three-dimensional extended network by O—H···O, N—H···O and C—H···O hydrogen-bond interactions.

Comment

Hydroxamic acid derivatives possess a great variety of biological and pharmaceutical activities (Dobosz *et al.*, 1999). We have synthesized a series of substituted hydroxamic acids, and the crystal structure of one of these, *viz.* the title compound, (I), is reported here.



(I)

The atomic numbering scheme adopted is shown in Fig. 1. The angle between the mean planes through O1/C7/N1/O2 and C1–C6 is 130.7 (2)°. The title molecules are linked *via* O—H···O, N—H···O and C—H···O intermolecular hydrogen bonds (Table 1). These two- or three-center interactions form a three-dimensional extended network (Fig. 2).

Experimental

2,4-Dichlorobenzoic acid (5 g, 0.026 mmol, Acros Organics Company) was added to anhydrous methanol (10 ml), followed by the addition of concentrated hydrochloric acid (2 ml). The mixture

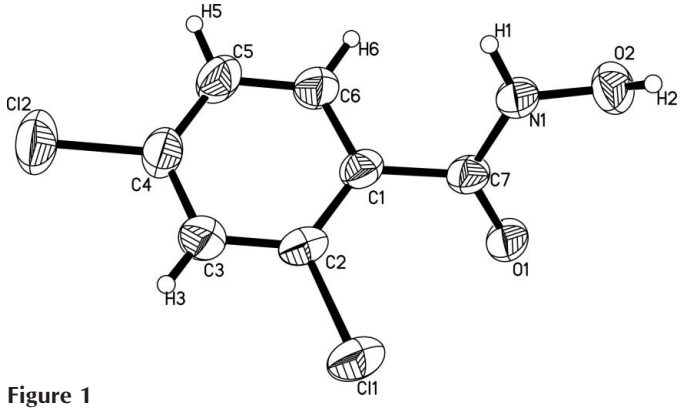


Figure 1
The structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

was refluxed for 7 h and the solution evaporated under vacuum. The oily compound obtained was neutralized by adding saturated Na_2CO_3. Pure methyl 2,4-dichlorobenzoate (yield 4.1 g, 76.5%) was obtained by column chromatography on silica (petroleum ether/ethyl acetate, 100:5). The title compound was obtained from methyl 2,4-dichlorobenzoate by the well known reaction with excess NH_2OH in basic solution (Summers *et al.*, 1990). Single crystals suitable for X-ray analysis were obtained by recrystallization from ethanol.

Crystal data

$C_7H_5Cl_2NO_2$
M_r = 206.02
Orthorhombic, $P2_12_12_1$
a = 4.9893 (8) Å
b = 6.3013 (10) Å
c = 26.790 (4) Å
V = 842.2 (2) Å^3
Z = 4
D_x = 1.625 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1332 reflections
θ = 3.0–21.4°
μ = 0.72 mm^{-1}
T = 292 (2) K
Plate, colorless
0.40 × 0.20 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.761, T_{max} = 0.944
5081 measured reflections
1928 independent reflections
1607 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 27.5°
$h = -6 \rightarrow 6$
$k = -8 \rightarrow 6$
$l = -33 \rightarrow 34$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.100
S = 1.03
1928 reflections
114 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0483P)^2 + 0.0861P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Absolute structure: Flack (1983), 741 Friedel pairs
Flack parameter: 0.01 (10)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O2^{i}	0.82	2.48	3.095 (3)	132
O2—H2···O1^{i}	0.82	2.00	2.735 (3)	149
N1—H1···O1^{ii}	0.86 (3)	1.98 (3)	2.787 (3)	157 (2)
C6—H6···O2^{iii}	0.93	2.58	3.355 (3)	142

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{5}{2}, -z$; (ii) $x-1, y, z$; (iii) $x-\frac{1}{2}, -y+\frac{3}{2}, -z$.

The H atoms bonded to the benzene ring and atom O2 were placed in calculated positions and treated as riding atoms, with C—H = 0.93 Å, O—H = 0.82 Å, and U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(O). The H atom associated with atom N1 was located in a difference map and refined with an N—H distance restraint of 0.86> (3) Å.

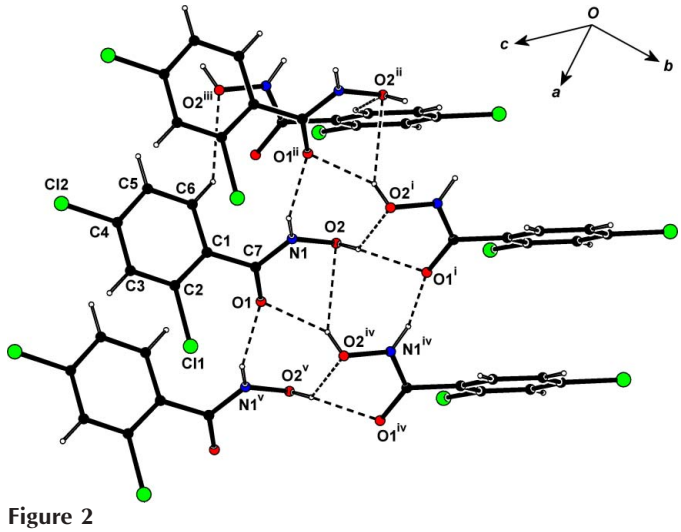


Figure 2
Packing diagram for the title compound, showing the hydrogen-bond interactions as dashed lines. [Symmetry codes: (i) $x-\frac{1}{2}, \frac{5}{2}-y, -z$; (ii) $x-1, y, z$; (iii) $x-\frac{1}{2}, \frac{3}{2}-y, -z$; (iv) $\frac{1}{2}+x, \frac{5}{2}-y, -z$; (v) $1+x, y, z$.]

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge financial support from the Program for New Century Excellent Talents in Universities of China and from the Education Commission of Shanxi Province of China.

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