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Disordered 4-hydroxyisophthalic acid

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.084
wR factor = 0.214
Data-to-parameter ratio = 12.3

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

Molecules of 4-hydroxyisophthalic acid, $C_8H_6O_5$, are disordered over two equally occupied orientations across a mirror plane. Each molecule contains an O—H···O intramolecular hydrogen bond, and both carboxylic acid groups are involved in $R_2^2(8)$ intermolecular hydrogen bonding. The crystal structure is further supported by π–π-stacking interactions.

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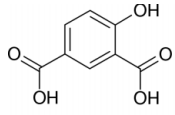
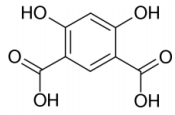
Comment

The title compound, (1), is an antipyretic and an antirheumatic (Negwer, 2000). It may also be found as an impurity in many pharmaceutical preparations containing salicylic acid (Goss, 1998). Our interest in this molecule is related to the possible hydrogen-bonding modes that may be adopted in the crystal structure.



(1)



(2)

The structure of the planar molecule is shown in Fig. 1. The packing of the molecules in the crystal is disordered such that the 4-hydroxy group (and atom H4) in the crystallographic model appear at both the 4 and 4*a* positions, (2), with occupancy parameters of 0.5. This implies that molecules with alternative orientations of the hydroxy group share the same sites in the crystal structure and hence generate an apparent mirror plane in the molecule. A similar disorder is found in space group $P2_1/m$ for (μ_2-propane-1,2-dithiolato-*S*,*S*,*S*′*S*′)-diiron, where a methyl group is equally disordered over two positions across a mirror plane (Zhang *et al.*, 2001).

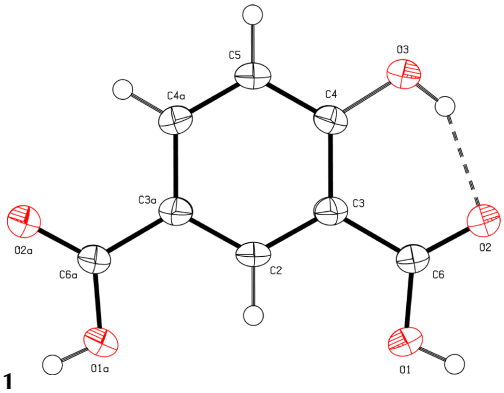


Figure 1
The molecular structure of (1), with disorder omitted. Hydrogen bonding is indicated by a dashed line and displacement ellipsoids are drawn at the 50% probability level. The suffix *a* denotes atoms generated by reflection symmetry.

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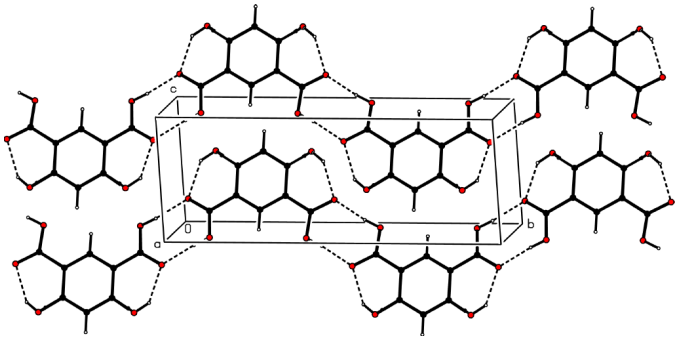

Figure 2
The hydrogen bonding between molecules, with disorder included.

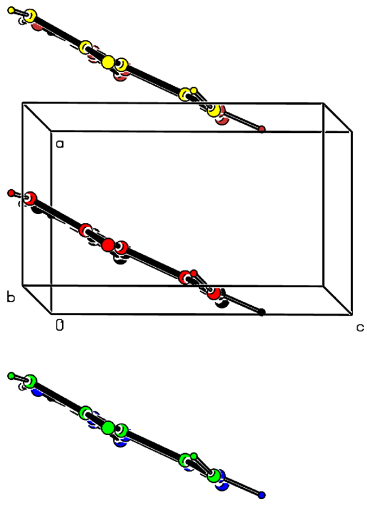

Figure 3
A partial packing diagram, showing molecules involved in π–π-stacking interactions. Disorder is included.

For each molecule, one intramolecular O3—H3···O2 hydrogen bond is present, and each molecule is linked to two other molecules through the formation of $R_2^2(8)$ rings involving the carboxylic acid groups across centres of symmetry (Table 2). This results in a zigzag chain of molecules running in the *b* direction of the unit cell (Fig. 2).

Furthermore, the crystal structure is stabilized by π–π-stacking between the aromatic rings, as shown in Fig. 3. Here the interplanar separation is 3.338 (2) Å, the centroid–centroid separation is 3.683 (2) Å (which corresponds directly to the length of the *a* axis) and the centroid offset is 1.557 (2) Å.

The structure of a hydrated complex between 4-hydroxyisophthalate and cobalt has been reported (Li *et al.*, 2003). The crystal structures of 2-hydroxyisophthalic acid (Solari *et al.*, 1996) and 5-hydroxyisophthalic acid dihydrate (Ermer & Neudorfl, 2001) are also known.

Experimental

The sample was purchased from Avocado Research Chemicals Ltd, England, and recrystallized from acetone.

Crystal data

$C_8H_6O_5$
M_r = 182.13
Monoclinic, $P2_1/m$
a = 3.6831 (3) Å
b = 16.4717 (14) Å
c = 6.0485 (4) Å
β = 90.229 (5)°
V = 366.94 (5) Å^3
Z = 2
D_x = 1.648 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 20 143 reflections
θ = 2.9–27.5°
μ = 0.14 mm^{-1}
T = 120 (2) K
Prism, colourless
0.15 × 0.08 × 0.02 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
T_{min} = 0.987, T_{max} = 0.997
5614 measured reflections
859 independent reflections
598 reflections with $I > 2\sigma(I)$
R_{int} = 0.080
θ_{max} = 27.6°
$h = -4 \rightarrow 4$
$k = -21 \rightarrow 21$
$l = -7 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.084
$wR(F^2)$ = 0.215
S = 1.00
859 reflections
70 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.098P)^2 + 0.6881P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C6	1.296 (4)	C4—O3	1.341 (5)
O2—C6	1.248 (4)	C6—C3	1.474 (4)
O2—C6—O1	123.2 (3)	O1—C6—C3	115.3 (3)
O2—C6—C3	121.5 (3)		
O1—C6—C3—C2	5.0 (5)	O2—C6—C3—C4	4.9 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.84	1.80	2.638 (3)	172
O3—H3···O2	0.84	1.88	2.593 (5)	141

Symmetry code: (i) $1-x, -y, -z$.

The H atoms were placed in calculated positions (O—H = 0.84 Å and C—H = 0.95 Å) and allowed to ride on their parent atoms with isotropic displacement parameters $1.2U_{eq}$ of the parent atom. Final R values are higher than normal and this may be related to the small crystal size and the molecular disorder.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

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