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4-Hydroxy-3-iodo-5-methoxybenzaldehyde

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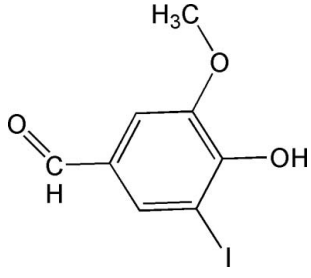
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.022; wR factor = 0.053; data-to-parameter ratio = 13.6.

The title compound, $C_8H_7IO_3$, contains two independent molecules, each lying on a mirror plane. The two molecules differ in the orientation of the –CHO group by a 180° rotation around its bond with the benzene ring. In the mirror plane, each independent molecule is linked to a symmetry-equivalent molecule by intermolecular O—H···O hydrogen bonds, forming a chain along the b axis. Short intermolecular I···O contacts are observed between adjacent chains.

Related literature

For general background, see: Belloni *et al.* (2005); Kahwa *et al.* (1986); Parashar *et al.* (1988); Santos *et al.* (2001); Tynan *et al.* (2005).



Experimental

Crystal data

$C_8H_7IO_3$
M_r = 278.04
Orthorhombic, *Pbam*
a = 16.332 (2) Å
b = 16.344 (2) Å
c = 6.5957 (12) Å
V = 1760.6 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 3.60 mm^{-1}
T = 293 (2) K
0.20 × 0.16 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.533, T_{max} = 0.672
9744 measured reflections
1974 independent reflections
1654 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.053
S = 1.03
1974 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···O2	0.82	2.21	2.661 (4)	115
O1—H1O···O3^{i}	0.82	1.93	2.634 (4)	143
O4—H4O···O5	0.82	2.18	2.639 (4)	116
O4—H4O···O6^{ii}	0.82	2.10	2.746 (4)	135

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2356).

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supplementary materials

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4-Hydroxy-3-iodo-5-methoxybenzaldehyde

Q.-L. Zhou, C.-L. Wang and Z.-L. Jing

Comment

In order to establish control over the preparation of crystalline solid materials so that their architecture and properties are predictable (Belloni *et al.*, 2005; Tynan *et al.*, 2005; Parashar *et al.*, 1988), the synthesis of new and designed crystal structures has become a major strand of modern chemistry. Metal complexes based on Schiff bases have attracted much attention because they can be utilized as model compounds of the active centres

in various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*, 2001). As part of an investigation of the coordination properties of Shiff bases functioning as ligands, we report the synthesis and crystal structure of the title compound, (I).

The asymmetric unit of (I) comprises two independent molecules (Fig. 1), lying on the mirror planes, one at z = 0 and the other at z = 1/2. The two molecules differ in the orientation of the -CHO group by a 180° rotation around its bond with the benzene ring. In both molecules the geometric parameters are normal.

Each independent molecule is linked to a symmetry-equivalent molecule by intermolecular O—H···O hydrogen bonds, forming a chain along the *b* axis, in the mirror plane. The chains formed by each independent molecule are shown in Fig.2 and Fig.3. Short intermolecular I1···O3(x-1/2, 1/2-y, 1-z) [3.099 (3) Å] and I2···O6(x-1/2, 1/2-y, -z) [3.388 (3) Å] contacts are observed between the adjacent chains.]

Experimental

4-Hydroxy-3-iodo-5-methoxy-benzaldehyde (1 g) was added to an anhydrous ethanol (50 ml), with stirring at 350 K. The resulting yellow solution was filtered and the filtrate was allowed to stand in air at room temperature for 10 d, yielding yellow crystals of (I)

Refinement

H atoms were placed in calculated positions [O—H = 0.82 Å and C—H = 0.93 (aromatic) or 0.96 Å (methyl)] and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(O).

Figures

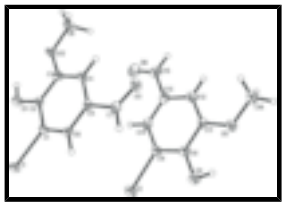

Fig. 1. The asymmetric unit of (I). Displacement ellipsoids are drawn at the 30% probability level.

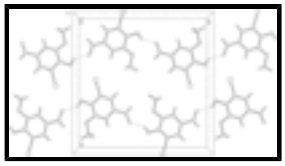

Fig. 2. Hydrogen-bonded (dashed lines) chains formed by one of the independent molecules, viewed down the *c* axis.

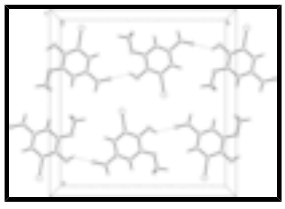

Fig. 3. Hydrogen-bonded (dashed lines) chains formed by other independent molecules, viewed down the *c* axis.

4-Hydroxy-3-iodo-5-methoxybenzaldehyde

Crystal data

$C_8H_7IO_3$	F_{000} = 1056
M_r = 278.04	D_x = 2.098 Mg m^{-3}
Orthorhombic, *Pbam*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2 2ab	Cell parameters from 4592 reflections
a = 16.332 (2) Å	θ = 2.5–26.4º
b = 16.344 (2) Å	μ = 3.60 mm^{-1}
c = 6.5957 (12) Å	*T* = 293 (2) K
V = 1760.6 (4) Å^3	Block, yellow
Z = 8	0.20 × 0.16 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1974 independent reflections
Radiation source: fine-focus sealed tube	1654 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.037
T = 293(2) K	θ_{max} = 26.4º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −16→20
T_{min} = 0.533, T_{max} = 0.672	*k* = −20→19
9744 measured reflections	*l* = −5→8

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.025P)^2 + 0.749P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.022	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.053	$\Delta\rho_{max}$ = 0.47 e Å^{-3}

S = 1.03
1974 reflections
145 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

$\Delta\rho_{min}$ = −0.47 e Å^{-3}
Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	−0.024919 (16)	0.352244 (16)	0.5000	0.03520 (9)
O1	0.13388 (17)	0.45773 (15)	0.5000	0.0385 (7)
H1O	0.1744	0.4873	0.5000	0.058*
O2	0.29270 (16)	0.42133 (16)	0.5000	0.0419 (7)
O3	0.29135 (17)	0.10050 (16)	0.5000	0.0374 (7)
C1	0.0976 (2)	0.3182 (2)	0.5000	0.0271 (8)
C2	0.1575 (2)	0.3788 (2)	0.5000	0.0266 (8)
C3	0.2406 (2)	0.3558 (2)	0.5000	0.0269 (8)
C4	0.2620 (2)	0.2753 (2)	0.5000	0.0292 (8)
H4	0.3170	0.2604	0.5000	0.035*
C5	0.2013 (2)	0.2148 (2)	0.5000	0.0263 (8)
C6	0.1193 (2)	0.2362 (2)	0.5000	0.0287 (8)
H6	0.0791	0.1959	0.5000	0.034*
C7	0.2227 (3)	0.1278 (2)	0.5000	0.0321 (9)
H7	0.1797	0.0904	0.5000	0.038*
C8	0.3785 (2)	0.4039 (3)	0.5000	0.0474 (12)
H8A	0.4087	0.4542	0.5000	0.071*
H8B	0.3921	0.3728	0.3812	0.071*
I2	0.049445 (18)	0.127572 (19)	0.0000	0.04574 (11)
O4	0.15122 (18)	−0.03449 (17)	0.0000	0.0434 (7)
H4O	0.1763	−0.0779	0.0000	0.065*
O5	0.30938 (17)	−0.06768 (16)	0.0000	0.0411 (7)
O6	0.3560 (2)	0.29765 (18)	0.0000	0.0586 (9)
C9	0.1756 (2)	0.1081 (2)	0.0000	0.0311 (9)
C10	0.2044 (2)	0.0276 (2)	0.0000	0.0311 (9)

C11	0.2889 (2)	0.0137 (2)	0.0000	0.0294 (8)
C12	0.3422 (2)	0.0785 (2)	0.0000	0.0329 (9)
H12	0.3984	0.0690	0.0000	0.039*
C13	0.3128 (3)	0.1583 (2)	0.0000	0.0318 (9)
C14	0.2296 (3)	0.1732 (2)	0.0000	0.0352 (10)
H14	0.2101	0.2266	0.0000	0.042*
C15	0.3720 (3)	0.2257 (3)	0.0000	0.0402 (10)
H15	0.4271	0.2115	0.0000	0.048*
C16	0.3938 (3)	−0.0866 (3)	0.0000	0.0475 (11)
H16A	0.4008	−0.1449	0.0000	0.071*
H16B	0.4190	−0.0638	0.1188	0.071*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.02010 (14)	0.03288 (15)	0.05262 (19)	0.00134 (10)	0.000	0.000
O1	0.0261 (15)	0.0192 (13)	0.070 (2)	−0.0014 (11)	0.000	0.000
O2	0.0207 (14)	0.0257 (14)	0.079 (2)	−0.0031 (11)	0.000	0.000
O3	0.0319 (16)	0.0232 (14)	0.0570 (19)	0.0058 (12)	0.000	0.000
C1	0.0181 (19)	0.0271 (18)	0.036 (2)	−0.0006 (15)	0.000	0.000
C2	0.025 (2)	0.0206 (17)	0.034 (2)	0.0020 (14)	0.000	0.000
C3	0.022 (2)	0.0205 (18)	0.038 (2)	−0.0034 (14)	0.000	0.000
C4	0.023 (2)	0.0285 (19)	0.036 (2)	0.0047 (15)	0.000	0.000
C5	0.028 (2)	0.0206 (17)	0.030 (2)	0.0040 (15)	0.000	0.000
C6	0.028 (2)	0.0242 (19)	0.034 (2)	−0.0050 (15)	0.000	0.000
C7	0.035 (2)	0.0199 (18)	0.042 (2)	0.0007 (16)	0.000	0.000
C8	0.020 (2)	0.045 (3)	0.077 (3)	−0.0009 (19)	0.000	0.000
I2	0.02894 (17)	0.05050 (19)	0.0578 (2)	0.00543 (12)	0.000	0.000
O4	0.0333 (16)	0.0306 (15)	0.066 (2)	−0.0051 (12)	0.000	0.000
O5	0.0323 (16)	0.0268 (14)	0.064 (2)	0.0049 (12)	0.000	0.000
O6	0.067 (2)	0.0269 (16)	0.082 (2)	−0.0079 (15)	0.000	0.000
C9	0.025 (2)	0.034 (2)	0.034 (2)	0.0051 (17)	0.000	0.000
C10	0.031 (2)	0.0284 (19)	0.034 (2)	−0.0059 (16)	0.000	0.000
C11	0.032 (2)	0.0216 (19)	0.035 (2)	0.0016 (16)	0.000	0.000
C12	0.028 (2)	0.036 (2)	0.035 (2)	−0.0005 (17)	0.000	0.000
C13	0.040 (2)	0.0248 (19)	0.030 (2)	−0.0032 (17)	0.000	0.000
C14	0.041 (3)	0.026 (2)	0.038 (2)	0.0044 (18)	0.000	0.000
C15	0.037 (3)	0.038 (2)	0.045 (3)	−0.0093 (19)	0.000	0.000
C16	0.039 (3)	0.038 (2)	0.065 (3)	0.014 (2)	0.000	0.000

Geometric parameters (Å, °)

I1—C1	2.077 (4)	I2—C9	2.085 (4)
O1—C2	1.347 (4)	O4—C10	1.336 (4)
O1—H1O	0.8200	O4—H4O	0.82
O2—C3	1.368 (4)	O5—C11	1.371 (4)
O2—C8	1.430 (5)	O5—C16	1.413 (5)
O3—C7	1.207 (5)	O6—C15	1.205 (5)
C1—C6	1.386 (5)	C9—C14	1.382 (6)

C1—C2	1.392 (5)	C9—C10	1.396 (5)
C2—C3	1.408 (5)	C10—C11	1.399 (5)
C3—C4	1.363 (5)	C11—C12	1.371 (5)
C4—C5	1.400 (5)	C12—C13	1.390 (5)
C4—H4	0.93	C12—H12	0.93
C5—C6	1.384 (5)	C13—C14	1.379 (6)
C5—C7	1.464 (5)	C13—C15	1.465 (5)
C6—H6	0.93	C14—H14	0.93
C7—H7	0.93	C15—H15	0.93
C8—H8A	0.96	C16—H16A	0.96
C8—H8B	0.96	C16—H16B	0.96
C2—O1—H1O	109.5	C10—O4—H4O	109.5
C3—O2—C8	117.0 (3)	C11—O5—C16	116.8 (3)
C6—C1—C2	120.5 (3)	C14—C9—C10	120.7 (4)
C6—C1—I1	120.4 (3)	C14—C9—I2	120.9 (3)
C2—C1—I1	119.1 (3)	C10—C9—I2	118.4 (3)
O1—C2—C1	118.7 (3)	O4—C10—C9	119.8 (4)
O1—C2—C3	122.1 (3)	O4—C10—C11	121.1 (3)
C1—C2—C3	119.2 (3)	C9—C10—C11	119.0 (3)
C4—C3—O2	126.7 (3)	C12—C11—O5	126.4 (4)
C4—C3—C2	120.3 (3)	C12—C11—C10	120.1 (3)
O2—C3—C2	113.0 (3)	O5—C11—C10	113.5 (3)
C3—C4—C5	120.1 (3)	C11—C12—C13	120.3 (4)
C3—C4—H4	120.0	C11—C12—H12	119.8
C5—C4—H4	120.0	C13—C12—H12	119.8
C6—C5—C4	120.4 (3)	C14—C13—C12	120.4 (4)
C6—C5—C7	118.5 (3)	C14—C13—C15	121.1 (4)
C4—C5—C7	121.1 (3)	C12—C13—C15	118.5 (4)
C5—C6—C1	119.5 (3)	C13—C14—C9	119.5 (4)
C5—C6—H6	120.2	C13—C14—H14	120.2
C1—C6—H6	120.2	C9—C14—H14	120.2
O3—C7—C5	125.5 (4)	O6—C15—C13	126.2 (4)
O3—C7—H7	117.3	O6—C15—H15	116.9
C5—C7—H7	117.3	C13—C15—H15	116.9
O2—C8—H8A	109.5	O5—C16—H16A	109.5
O2—C8—H8B	109.5	O5—C16—H16B	109.5
H8A—C8—H8B	109.5	H16A—C16—H16B	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O···O2	0.82	2.21	2.661 (4)	115
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Symmetry codes: (i) −*x*+1/2, *y*+1/2, −*z*+1; (ii) −*x*+1/2, *y*−1/2, −*z*.

Fig. 1

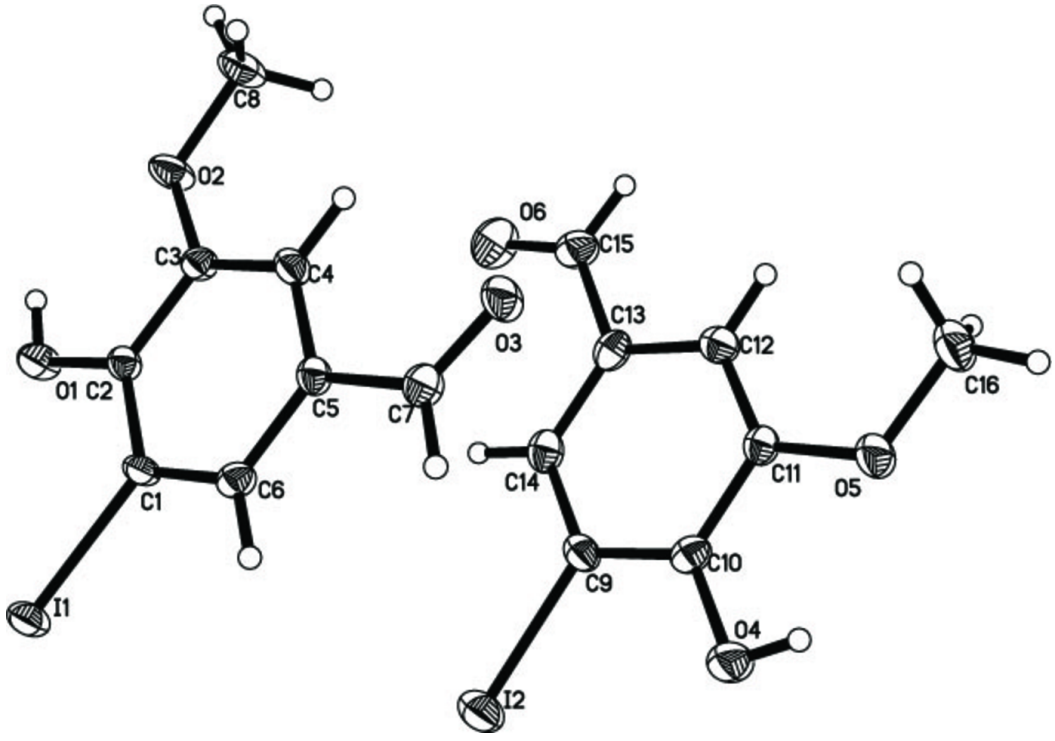
C8
O2
C3
C4
O6
C15
C13
O3
C12
C16
O1
C2
C5
C7
C14
C11
O5
C1
C6
C10
C9
I1
O4
I2

Fig. 2

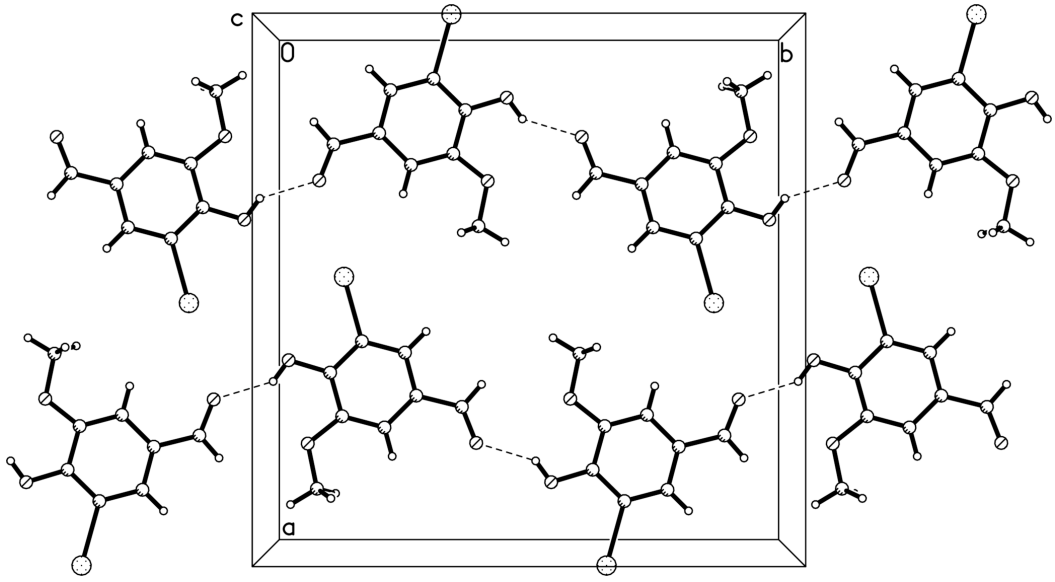

Fig. 3