

2-Iodo-1,3-dimethoxybenzene

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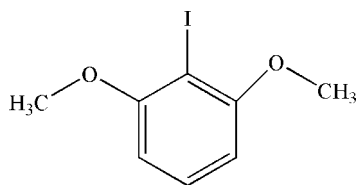
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å;
R factor = 0.019; wR factor = 0.046; data-to-parameter ratio = 15.5.

Crystals of the title compound, $\text{C}_8\text{H}_9\text{IO}_2$, were obtained from a dimethyl sulfoxide solution of 2,6-dimethoxybenzoic acid and iodobenzene diacetate under a nitrogen atmosphere at 353 K. In the crystal structure, molecules are linked by weak $\text{C}-\text{H}\cdots\pi$ interactions, generating interpenetrating one-dimensional chains of perpendicularly oriented molecules extending along $[011]$ and $[0\bar{1}1]$. Chains are also formed through non-bonding $\text{C}-\text{I}\cdots\pi$ contacts extending in the same directions, projecting a zigzag motif in view down $[100]$. The $\text{I}\cdots\text{C}_g$ distance is 3.695 (2) Å and the $\text{C}-\text{I}\cdots\text{C}_g$ angle is 164.17 (14)°. The molecular symmetry m coincides with the mirror plane of the space group $\text{Cmc}2_1$, resulting in a half-molecule in the asymmetric unit ($Z' = \frac{1}{2}$).

Related literature

For the development of a decarboxylative palladation reaction and its use in a Heck-type olefination of arene carboxylates, see: Myers *et al.* (2002). For a novel system for decarboxylative bromination, see: Telvekar & Chettiar (2007). For related structures, see: Kirsop *et al.* (2004); Ali *et al.* (2008). For a database study of C-halogen- π interactions and their influence on molecular conformation and crystal packing, see: Prasanna & Guru Row (2000). For structure validation in chemical crystallography, see: Spek (2009).



Experimental

Crystal data

$\text{C}_8\text{H}_9\text{IO}_2$
 $M_r = 264.05$
Orthorhombic, $\text{Cmc}2_1$
 $a = 12.5767$ (13) Å
 $b = 8.6788$ (8) Å
 $c = 8.4338$ (9) Å
 $V = 920.55$ (16) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 3.43$ mm⁻¹
 $T = 296$ K
 $0.23 \times 0.19 \times 0.16$ mm

Data collection

Bruker P4 diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 1997)
 $T_{\min} = 0.500$, $T_{\max} = 0.616$
(expected range = 0.469–0.578)
2731 measured reflections
850 independent reflections
840 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.019$
 $wR(F^2) = 0.046$
 $S = 1.12$
850 reflections
55 parameters
1 restraint
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.02$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.84$ e Å⁻³
Absolute structure: Flack (1983),
362 Friedel pairs
Flack parameter: -0.05 (4)

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}1-\text{H}1\text{A}\cdots\text{C}_g1^i$	0.93	2.94	3.824 (9)	159

Symmetry code: (i) $-x, y + 1, z$. C_g1 is the centroid of the $\text{C}1-\text{C}4/\text{C}3\text{A}/\text{C}2\text{A}$ ring.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2185).

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supplementary materials

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Comment

Decarboxylation arene carboxylic acids accompanied by simultaneous replacement with different function groups is a useful reaction in organic chemistry (Myers *et al.*, 2002;). Especially iodobenzene derivatives have been found widespread application in organic synthesis because of their selectivity and simplicity of use (Telvekar & Chettiar, 2007). Recently, we found iodobenzene derivatives could be formed by arene carboxylic acid with reaction of $\text{PhI}(\text{OAc})_2$. As part of our studies, we report herein the synthesis and crystal structure of the title compound (Fig. 1). The asymmetric unit of the cell contains a half-molecule ($Z' = 1/2$), which is completed by the space group symmetry m . Atoms I1, C4, C1, H1A occupy the special positions in the mirror plane m . The bond length of C4—I1 is 2.090 (5) Å. The two I—C—C angles, related by mirror symmetry, are 119.5 (2)°.

The molecules in the crystal structure are linked by weak C—H \cdots π interactions to generate a one-dimensional supramolecular structure (Fig. 2). The bond length of C1—H1A \cdots Cg1 is 3.824 (9) Å (Table. 1), Cg1 is the centroid of the C1 C2 C3 C4 C3A C2A ring. In a CSD database study, Prasanna & Guru Row (2000) reported about C-halogen \cdots π interactions and their influence on molecular conformation and crystal packing. The authors found 171 intermolecular C—I \cdots π contacts in the literature, with a mean I \cdots C(π -system) atomic distance of 3.698 (13) Å. In the course of the structure validation (Spek, 2009) of the title compound, a similar geometric parameter (I1 \cdots Cg1ⁱⁱ = 3.695 (2) Å) has been found. The C4 \cdots Cg1ⁱⁱ distance amounts to 5.735 (5) Å, and the angle C4—I1 \cdots Cg1ⁱⁱ is 164.17 (14) Å. Symmetry code: (ii = -x, y + 2, z - 1). The C4—H1A \cdots π and nonbonding C4—I1 \cdots π contacts generate interpenetrating one-dimensional chains of perpendicularly oriented molecules extending along the [0 1 1] and [0 $\bar{1}$ 1] directions, projecting a zigzag motif in view down [1 0 0] (Fig.3).

Experimental

The title compound was obtained from a mixture of 2,6-Dimethoxybenzoic acid (36 mg) with Iodobenzene diacetate (77 mg) in DMSO (2 ml) under a nitrogen atmosphere at 353 K for 24 h. The crude product was isolated and purified by silica gel column chromatography. Colorless prism-shaped crystals of (I) suitable for X-ray diffraction were grown by slow evaporation of a dichloromethane solution at room temperature.

Refinement

All hydrogen atoms were positioned geometrically and treated as riding, with C—H = 0.93 Å (CH) and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, and with C—H = 0.96 Å (CH₃) and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$.

Figures

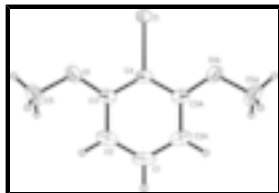


Fig. 1. The molecular structure of the title compound. Symmetry code: $(2 - x, y, z)$. Displacement ellipsoids for non-hydrogen atoms are drawn at the 30% probability level.

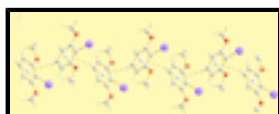


Fig. 2. A view of the one-dimensional weak C—H... π contacts in the title compound.

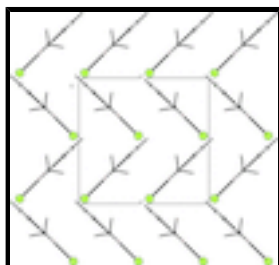


Fig. 3. A view down the a axis showing a section of the zigzag motif of the title compound.

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Crystal data

$C_8H_9IO_2$

$M_r = 264.05$

Orthorhombic, $Cmc2_1$

Hall symbol: C 2c -2

$a = 12.5767$ (13) Å

$b = 8.6788$ (8) Å

$c = 8.4338$ (9) Å

$V = 920.55$ (16) Å³

$Z = 4$

$F_{000} = 504$

$D_x = 1.905$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1365 reflections

$\theta = 3.7$ – 27.5°

$\mu = 3.43$ mm⁻¹

$T = 296$ K

Prism, white

$0.23 \times 0.19 \times 0.16$ mm

Data collection

Bruker P4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 296$ K

ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 1997)

$T_{\min} = 0.500$, $T_{\max} = 0.616$

2731 measured reflections

850 independent reflections

840 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.017$

$\theta_{\text{max}} = 25.5^\circ$

$\theta_{\text{min}} = 3.7^\circ$

$h = -15 \rightarrow 15$

$k = -10 \rightarrow 7$

$l = -9 \rightarrow 10$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.019$	$w = 1/[\sigma^2(F_o^2) + (0.0245P)^2 + 0.6278P]$
$wR(F^2) = 0.046$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.12$	$(\Delta/\sigma)_{\max} = 0.001$
850 reflections	$\Delta\rho_{\max} = 1.02 \text{ e } \text{\AA}^{-3}$
55 parameters	$\Delta\rho_{\min} = -0.84 \text{ e } \text{\AA}^{-3}$
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 362 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: $-0.05 (4)$

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	1.0000	0.95668 (3)	0.54168 (9)	0.04533 (12)
O1	0.8137 (2)	0.7922 (4)	0.7118 (3)	0.0562 (8)
C1	1.0000	0.5564 (7)	0.9408 (11)	0.073 (2)
H1A	1.0000	0.4784	1.0163	0.088*
C2	0.9046 (4)	0.6130 (5)	0.8860 (6)	0.0637 (12)
H2A	0.8409	0.5739	0.9250	0.076*
C3	0.9038 (3)	0.7287 (4)	0.7725 (4)	0.0432 (9)
C4	1.0000	0.7859 (5)	0.7165 (6)	0.0374 (11)
C5	0.7137 (4)	0.7340 (7)	0.7685 (7)	0.0776 (16)
H5A	0.6566	0.7868	0.7160	0.116*
H5B	0.7087	0.7505	0.8808	0.116*
H5C	0.7091	0.6256	0.7464	0.116*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.04655 (17)	0.05029 (18)	0.0391 (2)	0.000	0.000	0.01194 (17)
O1	0.0454 (16)	0.0700 (18)	0.0531 (19)	-0.0124 (14)	0.0033 (14)	0.0111 (15)
C1	0.099 (6)	0.052 (4)	0.068 (5)	0.000	0.000	0.030 (3)
C2	0.085 (3)	0.051 (2)	0.055 (3)	-0.016 (2)	0.009 (2)	0.014 (2)
C3	0.058 (2)	0.0384 (16)	0.033 (2)	-0.0055 (16)	0.0005 (16)	-0.0015 (15)
C4	0.055 (3)	0.030 (2)	0.027 (3)	0.000	0.000	-0.0008 (19)
C5	0.056 (3)	0.086 (4)	0.091 (5)	-0.023 (3)	0.010 (3)	0.007 (3)

Geometric parameters (\AA , $^\circ$)

I1—C4	2.090 (5)	C2—H2A	0.9300
O1—C3	1.359 (5)	C3—C4	1.391 (5)

supplementary materials

O1—C5	1.437 (6)	C4—C3 ⁱ	1.391 (5)
C1—C2	1.376 (6)	C5—H5A	0.9600
C1—C2 ⁱ	1.376 (6)	C5—H5B	0.9600
C1—H1A	0.9300	C5—H5C	0.9600
C2—C3	1.388 (5)		
C3—O1—C5	117.5 (4)	C3 ⁱ —C4—C3	121.0 (5)
C2—C1—C2 ⁱ	121.3 (6)	C3 ⁱ —C4—I1	119.5 (2)
C2—C1—H1A	119.3	C3—C4—I1	119.5 (2)
C2 ⁱ —C1—H1A	119.3	O1—C5—H5A	109.5
C1—C2—C3	119.8 (5)	O1—C5—H5B	109.5
C1—C2—H2A	120.1	H5A—C5—H5B	109.5
C3—C2—H2A	120.1	O1—C5—H5C	109.5
O1—C3—C2	124.0 (4)	H5A—C5—H5C	109.5
O1—C3—C4	116.9 (3)	H5B—C5—H5C	109.5
C2—C3—C4	119.1 (4)		
C2 ⁱ —C1—C2—C3	0.5 (11)	O1—C3—C4—C3 ⁱ	179.6 (3)
C5—O1—C3—C2	-0.7 (6)	C2—C3—C4—C3 ⁱ	0.1 (7)
C5—O1—C3—C4	179.9 (4)	O1—C3—C4—I1	-1.9 (5)
C1—C2—C3—O1	-179.7 (5)	C2—C3—C4—I1	178.6 (3)
C1—C2—C3—C4	-0.3 (7)		

Symmetry codes: (i) $-x+2, y, z$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1A \cdots Cg1 ⁱⁱ	0.93	2.94	3.824 (9)	159

Symmetry codes: (ii) $-x, y+1, z$.

Fig. 1

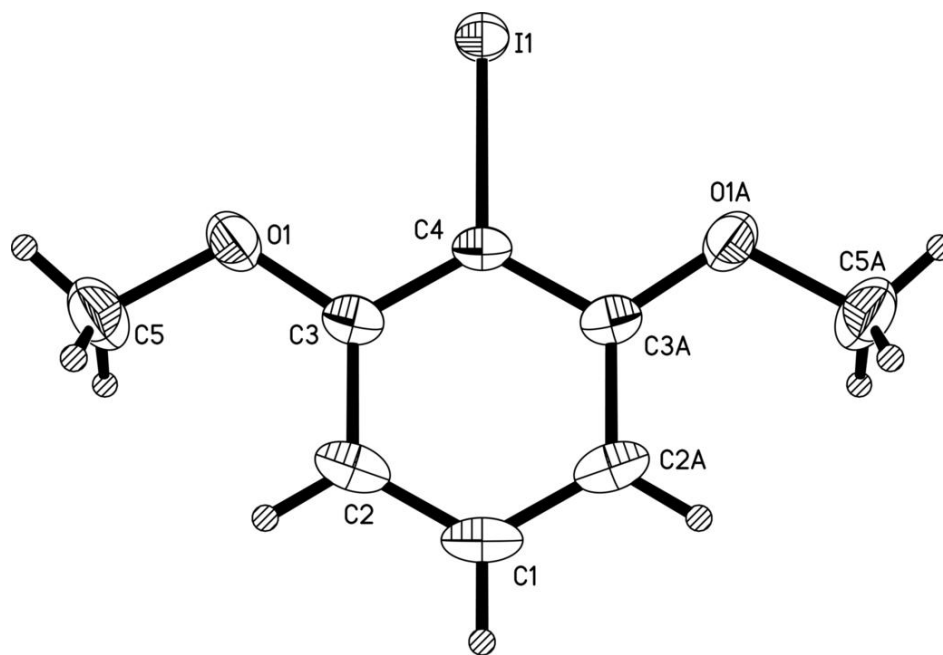


Fig. 2

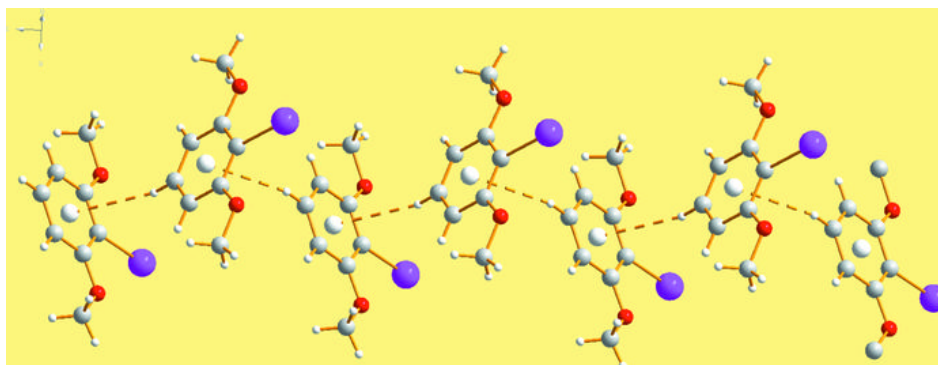


Fig. 3

