# organic compounds

# 2-Iodo-1,3-dimethoxybenzene

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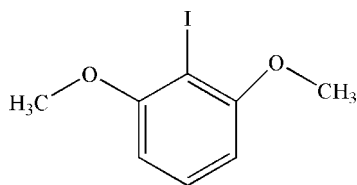
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Crystals of the title compound, $C_8H_9IO_2$, were obtained from a dimethyl sulfoxide solution of 2,6-dimethoxybenzoic acid and iodobenzene diacetate under a nitrogen atmosphere at 353 K. In the crystal structure, molecules are linked by weak C—H$\cdots\pi$ interactions, generating interpenetrating one-dimensional chains of perpendicularly oriented molecules extending along [011] and [01$\bar{1}$]. Chains are also formed through non-bonding C—I$\cdots\pi$ contacts extending in the same directions, projecting a zigzag motif in view down [100]. The I$\cdots Cg$ distance is 3.695 (2) Å and the C—I$\cdots Cg$ angle is 164.17 (14)°. The molecular symmetry $m$ coincides with the mirror plane of the space group $Cmc2_1$, 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–$\pi$ 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

| | |
|---|---|
| $C_8H_9IO_2$ | $V$ = 920.55 (16) Å³ |
| $M_r$ = 264.05 | $Z$ = 4 |
| Orthorhombic, $Cmc2_1$ | Mo $K\alpha$ radiation |
| $a$ = 12.5767 (13) Å | $\mu$ = 3.43 mm⁻¹ |
| $b$ = 8.6788 (8) Å | $T$ = 296 K |
| $c$ = 8.4338 (9) Å | 0.23 × 0.19 × 0.16 mm |

### Data collection

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

### Refinement

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

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

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

Symmetry code: (i) $-x, y + 1, z$. $Cg1$ is the centroid of the C1–C4/C3A/C2A ring.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; 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).

## References

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# supporting information

## 2-Iodo-1,3-dimethoxybenzene

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### S1. 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 PhI(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⋯$\pi$ interactions to generate a one-dimensional supramolecular structure (Fig. 2). The bond length of C1—H1A⋯*Cg*1 is 3.824 (9) Å (Table. 1), *Cg*1 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⋯$\pi$ interactions and their influence on molecular conformation and crystal packing. The authors found 171 intermolecular C—I⋯$\pi$ contacts in the literature, with a mean I⋯C$_{(\pi\text{-system})}$ 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⋯Cg1$^{ii}$ = 3.695 (2) Å) has been found. The C4⋯Cg1$^{ii}$ distance amounts to 5.735 (5) Å, and the angle C4—I1⋯Cg$^{ii}$ is 164.17 (14) °. Symmetry code: (ii = -*x*, *y* + 2, *z* - 1). The C4—H1A⋯$\pi$ and nonbonding C4—I1⋯$\pi$ 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).

### S2. 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.

### S3. Refinement

All hydrogen atoms were positioned geometrically and treated as riding, with C—H = 0.93 Å (CH) and $U_{iso}$(H) = 1.2$U_{eq}$(C), and with C—H = 0.96 Å (CH3) and $U_{iso}$(H) = 1.5$U_{eq}$(C).
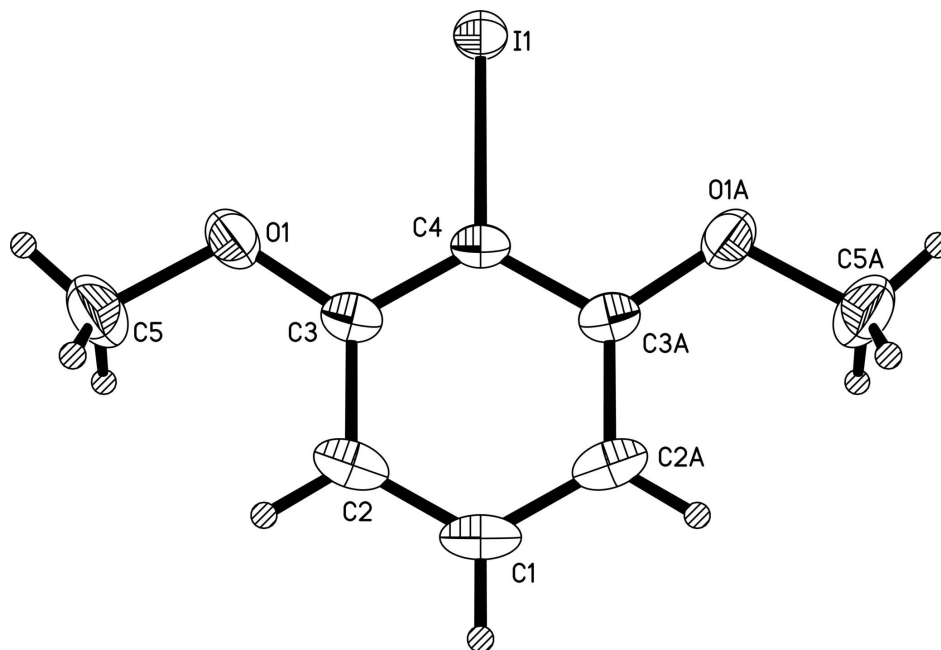
**Figure 1**

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.
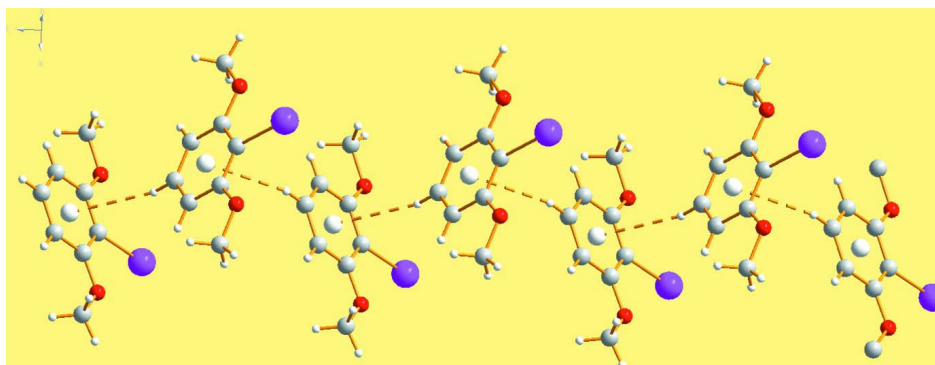


**Figure 2**

A view of the one-dimensional weak C—H···π contacts in the title compound.
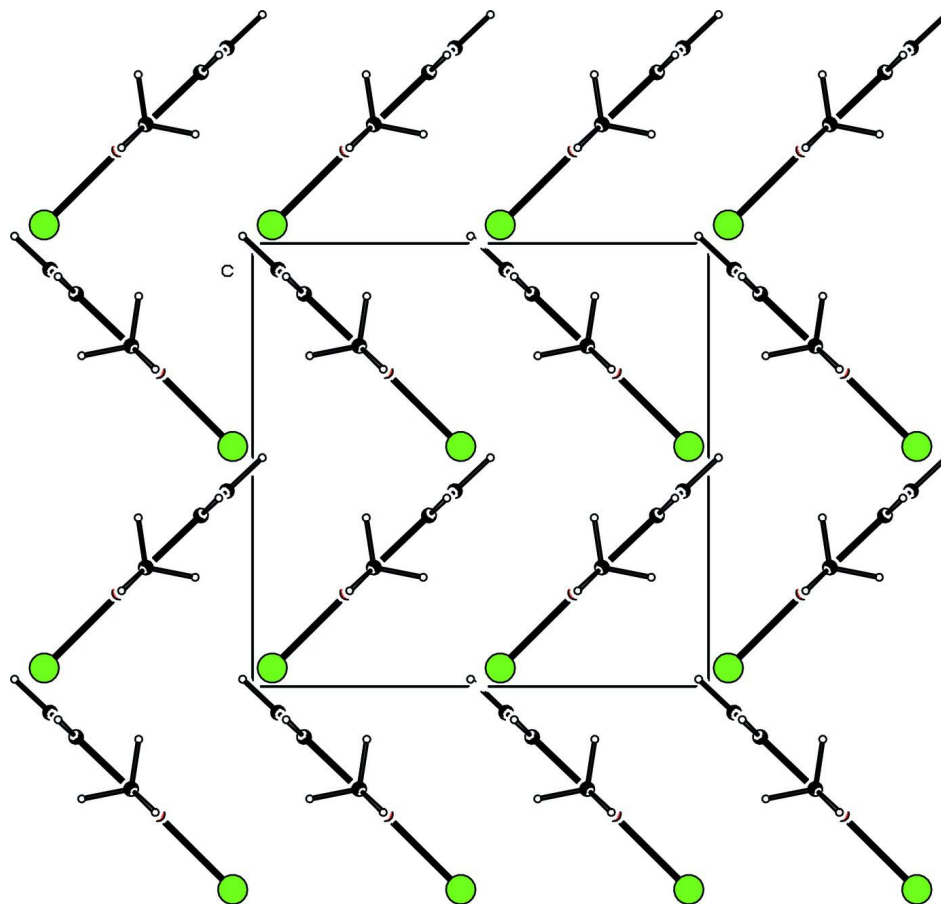
**Figure 3**

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

**2-Iodo-1,3-dimethoxybenzene**

*Crystal data*

| | |
|---|---|
| $C_8H_9IO_2$ | $F(000) = 504$ |
| $M_r = 264.05$ | $D_x = 1.905$ Mg m$^{-3}$ |
| Orthorhombic, $Cmc2_1$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: C 2c -2 | Cell parameters from 1365 reflections |
| $a = 12.5767$ (13) Å | $\theta = 3.7–27.5°$ |
| $b = 8.6788$ (8) Å | $\mu = 3.43$ mm$^{-1}$ |
| $c = 8.4338$ (9) Å | $T = 296$ K |
| $V = 920.55$ (16) Å$^3$ | Prism, white |
| $Z = 4$ | $0.23 \times 0.19 \times 0.16$ mm |

*Data collection*

| | |
|---|---|
| Bruker P4 diffractometer | 2731 measured reflections |
| Radiation source: fine-focus sealed tube | 850 independent reflections |
| Graphite monochromator | 840 reflections with $I > 2\sigma(I)$ |
| $\omega$ scans | $R_{int} = 0.017$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 1997) | $\theta_{max} = 25.5°$, $\theta_{min} = 3.7°$ |
| | $h = -15 \rightarrow 15$ |
| | $k = -10 \rightarrow 7$ |
| $T_{min} = 0.500$, $T_{max} = 0.616$ | $l = -9 \rightarrow 10$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.019$ | H-atom parameters constrained |
| $wR(F^2) = 0.046$ | $w = 1/[\sigma^2(F_o^2) + (0.0245P)^2 + 0.6278P]$ |
| $S = 1.12$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 850 reflections | $(\Delta/\sigma)_{max} = 0.001$ |
| 55 parameters | $\Delta\rho_{max} = 1.02$ e Å$^{-3}$ |
| 1 restraint | $\Delta\rho_{min} = -0.84$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Absolute structure: Flack (1983), 362 Friedel pairs |
| Secondary atom site location: difference Fourier map | Absolute structure parameter: −0.05 (4) |

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

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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 (Å, º)*

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

**sup-4**

| 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 code: (i) −$x$+2, $y$, $z$.

*Hydrogen-bond geometry (Å, º)*

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C1—H1$A$···$Cg$1$^{ii}$ | 0.93 | 2.94 | 3.824 (9) | 159 |

Symmetry code: (ii) −$x$, $y$+1, $z$.