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5-Formyl-2-methoxyphenyl 2-iodobenzoate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.007 Å
R factor = 0.036
wR factor = 0.089
Data-to-parameter ratio = 14.4

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

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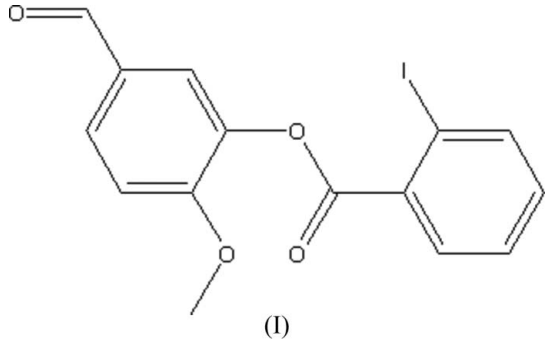
The title compound, $C_{15}H_{11}IO_4$, was synthesized by the *N*,*N*′-dicyclohexylcarbodiimide- and 4-dimethylaminopyridine-catalyzed reaction of 3-hydroxy-4-methoxybenzaldehyde and 2-iodobenzoic acid. In the molecule, the dihedral angle between the two benzene rings is 70.6 (1)°.

Comment

N,*N*′-Dicyclohexylcarbodiimide (DCC) and 4-dimethylaminopyridine (DMAP) are effective catalysts for esterification reactions (Litvin & Kirichenko, 1967; Steglich & Hofle, 1969). We have recently focused on the preparation of benzoic acid phenyl ester derivatives using these catalysts. The title compound, (I), was synthesized by the reaction of 3-hydroxy-4-methoxybenzaldehyde and 2-iodobenzoic acid in the presence of DCC and DMAP.



(I)

The molecular structure of (I) is shown in Fig. 1. The dihedral angle between the atoms of the central ester group (atoms C7/O1/O2) and the C1–C6 benzene ring is 88.2 (1)°,

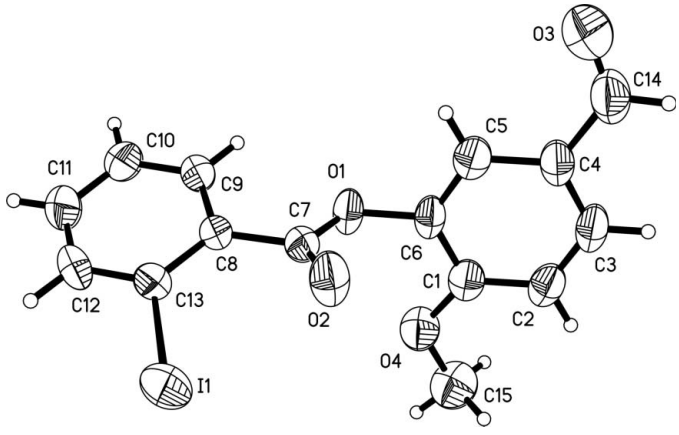


Figure 1
A view of (I), showing displacement ellipsoids drawn at the 30% probability level. H atoms are drawn as spheres of arbitrary radii.

while the dihedral angle between the central ester group and the C8–C13 benzene ring is 21.4 (2)°. The dihedral angle between the two benzene rings is 70.6 (1)°.

In the crystal structure, molecules are linked into centrosymmetric dimers *via* weak C—H···O hydrogen bonds. These dimers are further linked by weak intermolecular C—H···O hydrogen bonds into one-dimensional chains along [110] (Table 1 and Fig. 2).

Experimental

To a solution of 3-hydroxy-4-methoxybenzaldehyde (2 g, 13 mmol) in dry dichloromethane (25 ml, 13 mmol), 2-iodobenzoic acid (3.26 g, 13 mmol) was added in an N_2 atmosphere. *N*,*N*′-Dicyclohexylcarbodiimide (3 g, 14.5 mmol) and 4-dimethylaminopyridine (0.32 g, 2.6 mmol) were then added. The resulting mixture was stirred at room temperature for 10 h. The mixture was then filtered. The insoluble material was washed with dry dichloromethane and the combined filtrates were evaporated. Flash column chromatography (ethyl acetate/petroleum ether, 1:5 *v*/*v*) of the residue gave a white solid, (I) (4.1 g). Colourless crystals were obtained by recrystallization of the title compound from ethyl acetate.

Crystal data

$C_{15}H_{11}IO_4$
M_r = 382.14
Triclinic, $P\overline{1}$
a = 7.4730 (15) Å
b = 8.1841 (17) Å
c = 11.956 (3) Å
α = 74.517 (3)°
β = 86.831 (4)°
γ = 84.019 (3)°
V = 700.6 (3) Å^3
Z = 2
D_x = 1.812 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2008 reflections
θ = 2.4–22.3°
μ = 2.30 mm^{-1}
T = 294 (2) K
Block, colourless
0.40 × 0.30 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.382, T_{max} = 0.604
3709 measured reflections
2630 independent reflections
2089 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 25.8°
$h = -6 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.089
S = 1.04
2630 reflections
183 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0338P)^2 + 1.0404P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 1.05 e Å^{-3}
$\Delta\rho_{min}$ = −1.00 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0335 (19)

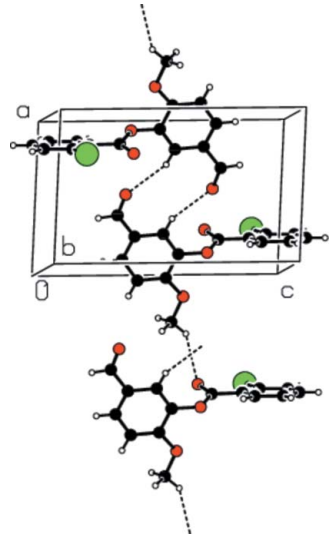


Figure 2
Part of a hydrogen-bonded chain of (I). Hydrogen bonds are drawn as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O3^{i}	0.93	2.52	3.391 (7)	156
C15—H15B···O2^{ii}	0.96	2.52	3.322 (6)	141

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

All H atoms were included in calculated positions and refined using a riding-model approximation, with C—H = 0.93–0.96 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The highest peak in the final difference Fourier map is 0.87 Å from atom I1. The deepest hole in the difference map is 0.83 Å from atom I1.

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

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