

6-Methoxyisobenzofuran-1(3H)-one

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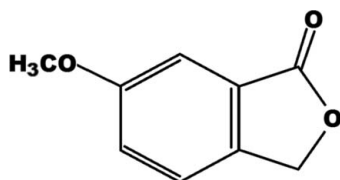
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C—C}) = 0.002$ Å; R factor = 0.050; wR factor = 0.166; data-to-parameter ratio = 16.0.

In the title compound, $\text{C}_9\text{H}_8\text{O}_3$, the molecular skeleton is almost planar [r.m.s. deviation = $0.016(2)$ Å]. Weak intermolecular $\text{C—H}\cdots\text{O}$ and $\text{C—H}\cdots\pi$ interactions consolidate the crystal packing, with the molecules stacking in the $[101]$ direction.

Related literature

For the biological activity of isobenzofuran-1(3H)-one, see: Brady *et al.* (2000); Huang *et al.* (2012); Cardozo *et al.* (2005); Yoganathan *et al.* (2003); Demuner *et al.* (2006). For related structures, see: Sun *et al.* (2009); Mendenhall *et al.* (2003).



Experimental

Crystal data

$\text{C}_9\text{H}_8\text{O}_3$
 $M_r = 164.15$
Monoclinic, $P2_1/c$
 $a = 9.2922(19)$ Å
 $b = 8.4982(12)$ Å
 $c = 9.786(2)$ Å
 $\beta = 90.471(15)^\circ$

$V = 772.8(2)$ Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 293$ K
 $0.28 \times 0.17 \times 0.12$ mm

Data collection

Nonius KappaCCD diffractometer
3304 measured reflections
1741 independent reflections

1285 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.166$
 $S = 1.12$
1741 reflections

109 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.20$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of C2—C7 ring.

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
$\text{C9—H9B}\cdots\text{O2}^i$	0.96	2.55	3.490 (2)	165
$\text{C8—H8B}\cdots\text{Cg1}^{ii}$	0.97	2.84	3.637 (2)	140
$\text{C9—H9C}\cdots\text{Cg1}^{iii}$	0.96	2.91	3.744 (2)	146

Symmetry codes: (i) $-x + 1, -y, -z$; (ii) $x, -y + \frac{3}{2}, z - \frac{1}{2}$; (iii) $x, -y + \frac{3}{2}, z + \frac{1}{2}$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5338).

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supporting information

Acta Cryst. (2012). E68, o2995 [https://doi.org/10.1107/S1600536812039074]

6-Methoxyisobenzofuran-1(3H)-one

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S1. Comment

Isobenzofuran-1(3H)-one fragment, γ -lactone fused to aromatic ring, occurs in several compounds that exhibit biological activities ranging from antibacterial (Brady *et al.*, 2000), antioxidant (Zhu *et al.*, 2012), anticonvulsant (Cardozo *et al.*, 2005), and anti-HIV (Yoganathan *et al.*, 2003) to inhibition of the photosynthetic electron transport (Demuner *et al.*, 2006). In a research program devoted to finding the phytotoxic compounds containing the isobenzofuran-1(3H)-one core, the title compound was synthesized.

The title molecule (Fig. 1) is essentially planar with a mean deviation of 0.016 (2) Å from the best plane formed by 12 non-H atoms. All bond lengths and angles are normal and correspond to those observed in the related compounds (Sun *et al.*, 2009; Mendenhall *et al.*, 2003). The crystal packing is stabilized by weak intermolecular C—H \cdots O and C—H \cdots π interactions (Table 1, Fig. 2), with a stacking direction of the molecules in [101].

S2. Experimental

Starting materials were commercially available and were used without further purification. The synthesis of the title compound was carried out as follows. A tube of 40 ml equipped with a magnetic stir bar was charged with Palladium (II) acetate (156.8 mg, 0.70 mmol), potassium dihydrogen phosphate (3654 mg, 21.0 mmol), 3-methoxy benzoic acid (1064 mg, 7.00 mmol) and dibromomethane (28 ml). The tube was sealed with a teflon cap and the reaction mixture was stirred at 140 °C for 36 h. After this time, the mixture was filtered through a pad of celite. The filtrate was concentrated under reduced pressure and the residue was purified by silica gel column chromatography (hexane-ethyl acetate 2:1 v/v) to afford the title compound in 59% yield (681 mg, 4.15 mmol) as a white solid. The crystals suitable for X-ray crystallographic analysis were obtained by slow evaporation from acetone solution at room temperature. *M.p.* 116.9–118.4 °C. IR (selected bands, cm⁻¹): 3003, 2925, 2837, 1733, 1600, 1585, 1488, 1454, 1431, 1267, 1206, 1028, 973, 869, 749, 690, 545. ¹H NMR (300 MHz, MeOH-*d*₄) δ 3.87 (s, 3H, H-9), 5.26 (s, 2H, H-8), 7.22–7.38 (m, 3H, H-3, H-5 and H-6); ¹³C NMR (75 MHz, MeOH-*d*₄) δ 56.0 (C-9), 69.7 (C-8), 107.7 (C-3), 123.1; 123.3 (C-5 and C-6), 127.3 (C-2), 139.1 (C-7), 160.8 (C-4), 171.4 (C-1). HREIMS *m/z* (*M*+H⁺): Calcd for C₉H₈O₃, 165.0552; found: 165.0608.

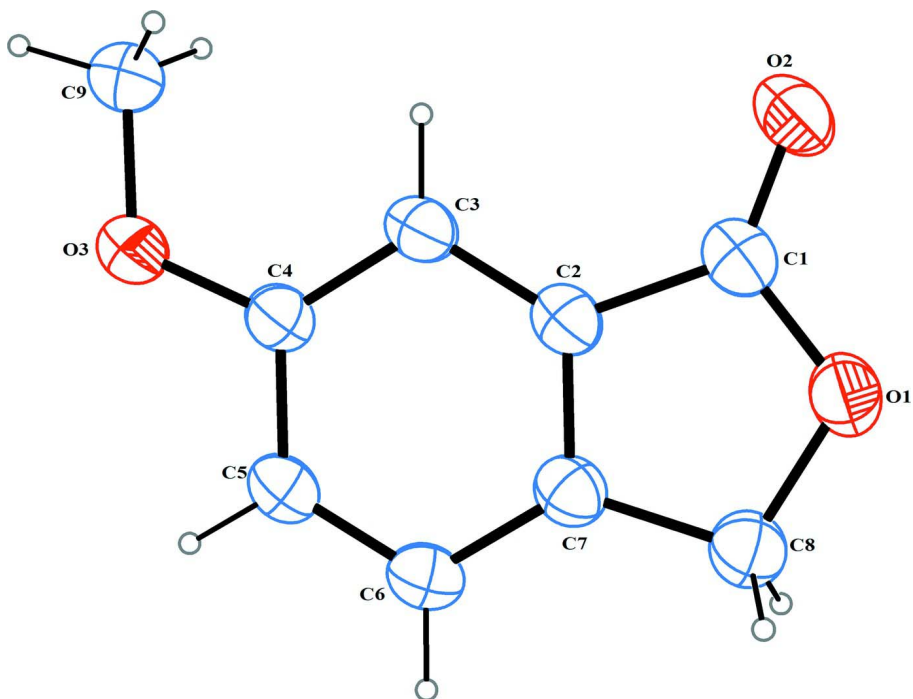


Figure 1

The molecular structure of the title compound, showing the atom labeling and displacement ellipsoids at the 30% probability level.

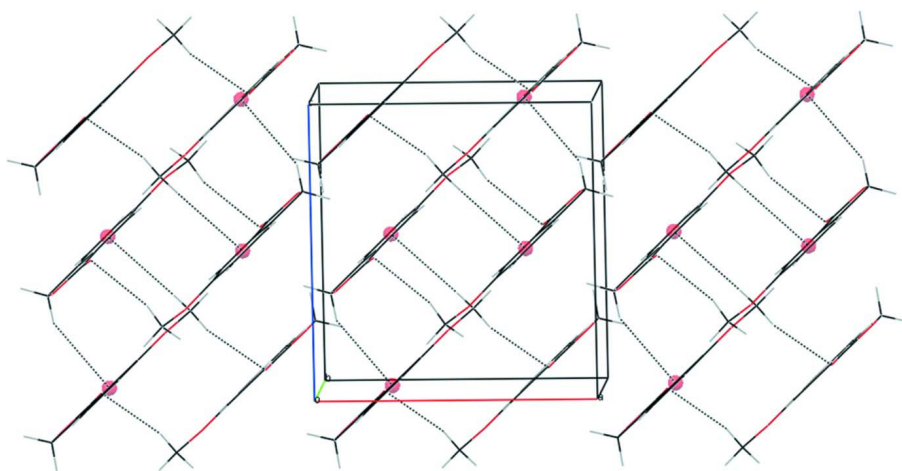


Figure 2

A portion of the crystal packing viewed approximately down the *c* axis. Dotted lines denote intermolecular C—H...O and C—H... π interactions. Centroids of six-membered rings are drawn as red balls.

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Crystal data

$C_9H_8O_3$

$M_r = 164.15$

Monoclinic, $P2_1/c$

$a = 9.2922(19) \text{ \AA}$

$b = 8.4982(12) \text{ \AA}$

$c = 9.786(2) \text{ \AA}$

$\beta = 90.471(15)^\circ$

$V = 772.8(2) \text{ \AA}^3$

$Z = 4$
 $F(000) = 344$
 $D_x = 1.411 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 3082 reflections

$\theta = 2.1\text{--}27.5^\circ$
 $\mu = 0.11 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 Prism, colourless
 $0.28 \times 0.17 \times 0.12 \text{ mm}$

Data collection

Nonius KappaCCD
 diffractometer
 Radiation source: Enraf Nonius FR590
 Graphite monochromator
 Detector resolution: 9 pixels mm^{-1}
 CCD rotation images, thick slices scans
 3304 measured reflections

1741 independent reflections
 1285 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$
 $\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 2.2^\circ$
 $h = -12 \rightarrow 12$
 $k = -10 \rightarrow 11$
 $l = -12 \rightarrow 12$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.166$
 $S = 1.12$
 1741 reflections
 109 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0943P)^2 + 0.032P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.93834 (12)	0.04009 (14)	0.16871 (11)	0.0695 (4)
O2	0.79489 (14)	-0.11746 (14)	0.04458 (12)	0.0752 (4)
O3	0.54600 (12)	0.40633 (13)	-0.19372 (11)	0.0699 (4)
C1	0.83141 (17)	0.0134 (2)	0.07489 (15)	0.0612 (4)
C2	0.77893 (16)	0.16711 (17)	0.02593 (13)	0.0552 (4)
C3	0.67205 (15)	0.19885 (18)	-0.07089 (14)	0.0569 (4)
H3	0.6212	0.1185	-0.114	0.068*
C4	0.64497 (16)	0.35569 (18)	-0.09995 (14)	0.0568 (4)
C5	0.72174 (17)	0.47477 (18)	-0.03217 (16)	0.0632 (4)
H5	0.7013	0.5794	-0.0524	0.076*
C6	0.82660 (17)	0.44069 (19)	0.06357 (16)	0.0639 (4)
H6	0.8766	0.5208	0.108	0.077*

C7	0.85594 (16)	0.28364 (18)	0.09213 (14)	0.0572 (4)
C8	0.96195 (18)	0.2080 (2)	0.18656 (16)	0.0663 (5)
H8A	1.0596	0.2365	0.1624	0.08*
H8B	0.9448	0.2392	0.2804	0.08*
C9	0.45913 (19)	0.2893 (2)	-0.25875 (18)	0.0730 (5)
H9A	0.3942	0.3389	-0.3222	0.11*
H9B	0.4051	0.2336	-0.191	0.11*
H9C	0.5197	0.2169	-0.3067	0.11*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0805 (7)	0.0576 (7)	0.0702 (7)	0.0088 (5)	-0.0046 (5)	0.0056 (5)
O2	0.0934 (9)	0.0475 (7)	0.0847 (8)	0.0000 (5)	0.0030 (6)	0.0033 (5)
O3	0.0797 (7)	0.0492 (7)	0.0804 (7)	-0.0024 (5)	-0.0172 (6)	0.0022 (5)
C1	0.0700 (9)	0.0536 (9)	0.0602 (8)	0.0018 (7)	0.0085 (7)	0.0018 (6)
C2	0.0635 (8)	0.0479 (8)	0.0541 (7)	0.0004 (6)	0.0070 (6)	0.0000 (6)
C3	0.0651 (8)	0.0457 (8)	0.0599 (8)	-0.0047 (6)	0.0027 (6)	-0.0034 (6)
C4	0.0635 (8)	0.0480 (8)	0.0588 (8)	-0.0014 (6)	-0.0003 (6)	-0.0001 (6)
C5	0.0757 (9)	0.0428 (8)	0.0708 (9)	0.0005 (6)	-0.0035 (7)	-0.0013 (6)
C6	0.0734 (9)	0.0496 (8)	0.0686 (9)	-0.0049 (7)	-0.0032 (7)	-0.0081 (7)
C7	0.0633 (8)	0.0524 (9)	0.0561 (7)	-0.0001 (6)	0.0046 (6)	-0.0021 (6)
C8	0.0728 (9)	0.0610 (10)	0.0649 (9)	0.0037 (7)	-0.0022 (7)	-0.0042 (7)
C9	0.0790 (10)	0.0593 (10)	0.0805 (10)	-0.0097 (8)	-0.0147 (8)	0.0019 (8)

Geometric parameters (Å, °)

O1—C1	1.366 (2)	C5—C6	1.377 (2)
O1—C8	1.454 (2)	C5—H5	0.93
O2—C1	1.199 (2)	C6—C7	1.390 (2)
O3—C4	1.3635 (19)	C6—H6	0.93
O3—C9	1.427 (2)	C7—C8	1.491 (2)
C1—C2	1.473 (2)	C8—H8A	0.97
C2—C7	1.380 (2)	C8—H8B	0.97
C2—C3	1.393 (2)	C9—H9A	0.96
C3—C4	1.385 (2)	C9—H9B	0.96
C3—H3	0.93	C9—H9C	0.96
C4—C5	1.402 (2)		
C1—O1—C8	110.59 (12)	C5—C6—H6	120.8
C4—O3—C9	117.14 (13)	C7—C6—H6	120.8
O2—C1—O1	121.50 (16)	C2—C7—C6	119.63 (15)
O2—C1—C2	130.52 (17)	C2—C7—C8	108.60 (14)
O1—C1—C2	107.98 (14)	C6—C7—C8	131.77 (14)
C7—C2—C3	122.97 (15)	O1—C8—C7	104.50 (12)
C7—C2—C1	108.34 (15)	O1—C8—H8A	110.9
C3—C2—C1	128.69 (14)	C7—C8—H8A	110.9
C4—C3—C2	116.94 (14)	O1—C8—H8B	110.9

C4—C3—H3	121.5	C7—C8—H8B	110.9
C2—C3—H3	121.5	H8A—C8—H8B	108.9
O3—C4—C3	124.20 (14)	O3—C9—H9A	109.5
O3—C4—C5	115.37 (14)	O3—C9—H9B	109.5
C3—C4—C5	120.43 (15)	H9A—C9—H9B	109.5
C6—C5—C4	121.64 (15)	O3—C9—H9C	109.5
C6—C5—H5	119.2	H9A—C9—H9C	109.5
C4—C5—H5	119.2	H9B—C9—H9C	109.5
C5—C6—C7	118.38 (15)		
C8—O1—C1—O2	179.83 (13)	O3—C4—C5—C6	-178.97 (12)
C8—O1—C1—C2	-0.01 (16)	C3—C4—C5—C6	0.7 (2)
O2—C1—C2—C7	-179.93 (15)	C4—C5—C6—C7	0.2 (2)
O1—C1—C2—C7	-0.11 (16)	C3—C2—C7—C6	0.6 (2)
O2—C1—C2—C3	-0.6 (3)	C1—C2—C7—C6	179.99 (12)
O1—C1—C2—C3	179.23 (12)	C3—C2—C7—C8	-179.21 (12)
C7—C2—C3—C4	0.2 (2)	C1—C2—C7—C8	0.18 (16)
C1—C2—C3—C4	-179.01 (12)	C5—C6—C7—C2	-0.8 (2)
C9—O3—C4—C3	4.2 (2)	C5—C6—C7—C8	178.95 (14)
C9—O3—C4—C5	-176.13 (13)	C1—O1—C8—C7	0.11 (15)
C2—C3—C4—O3	178.74 (12)	C2—C7—C8—O1	-0.18 (15)
C2—C3—C4—C5	-0.9 (2)	C6—C7—C8—O1	-179.96 (14)

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

Cg1 is the centroid of C2—C7 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C9—H9B \cdots O2 ⁱ	0.96	2.55	3.490 (2)	165
C8—H8B \cdots Cg1 ⁱⁱ	0.97	2.84	3.637 (2)	140
C9—H9C \cdots Cg1 ⁱⁱⁱ	0.96	2.91	3.744 (2)	146

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