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6-Methoxyisobenzofuran-1(3*H*)-one

Jorge L. Pereira,[a] Róbson R. Teixeira,[a]* Silvana Guilardi[b] and Drielly A. Paixão[b]

[a]Departamento de Química–UFV, Viçosa, MG, Brazil, and [b]Instituto de Química–UFU, Uberlândia, MG, Brazil
Correspondence e-mail: robsonr.teixeira@ufv.br_or_silvana@ufu.br

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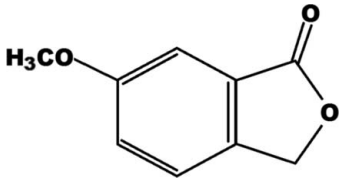
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.050; wR factor = 0.166; data-to-parameter ratio = 16.0.

In the title compound, $C_9H_8O_3$, the molecular skeleton is almost planar [r.m.s. deviation = 0.016 (2) Å]. Weak intermolecular C—H···O and C—H···π 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

$C_9H_8O_3$
M_r = 164.15
Monoclinic, $P2_1/c$
a = 9.2922 (19) Å
b = 8.4982 (12) Å
c = 9.786 (2) Å
β = 90.471 (15)°
V = 772.8 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.28 × 0.17 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
3304 measured reflections
1741 independent reflections
1285 reflections with $I > 2\sigma(I)$
R_{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
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

*Cg*1 is the centroid of C2–C7 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9B···O2^{i}	0.96	2.55	3.490 (2)	165
C8—H8B···$Cg1^{ii}$	0.97	2.84	3.637 (2)	140
C9—H9C···$Cg1^{iii}$	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).

The authors thank Professor Dr Javier Ellena of the IFSC, USP, Brazil, for the X-ray data collection. This work was supported financially by CAPES, CNPq, FUNARBE and FAPEMIG. This work is also a collaborative research project with members of the Rede Mineira de Química (RQ—MG), also supported by FAPEMIG.

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

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supplementary materials

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6-Methoxyisobenzofuran-1(3*H*)-one

Jorge L. Pereira, Róbson R. Teixeira, Silvana Guilardi and Drielly A. Paixão

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···O and C—H···π interactions (Table 1, Fig. 2), with a stacking direction of the molecules in [101].

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. ^{1}H 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); ^{13}C 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 $C_9H_8O_3$, 165.0552; found: 165.0608.

Computing details

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* (Otwinowski & Minor, 1997); 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).

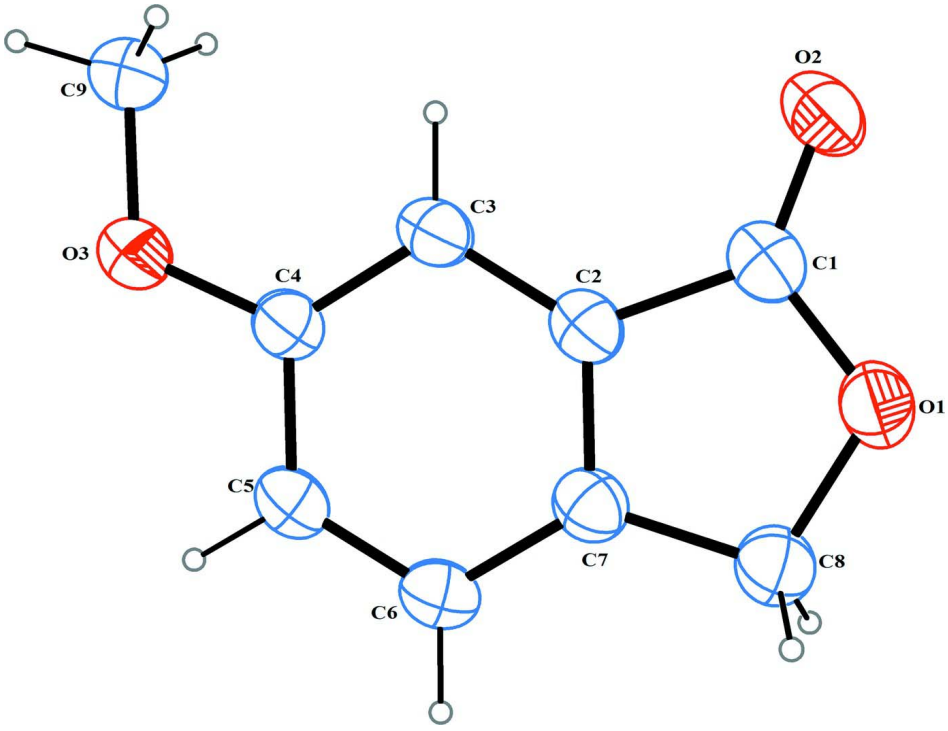

Figure 1

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

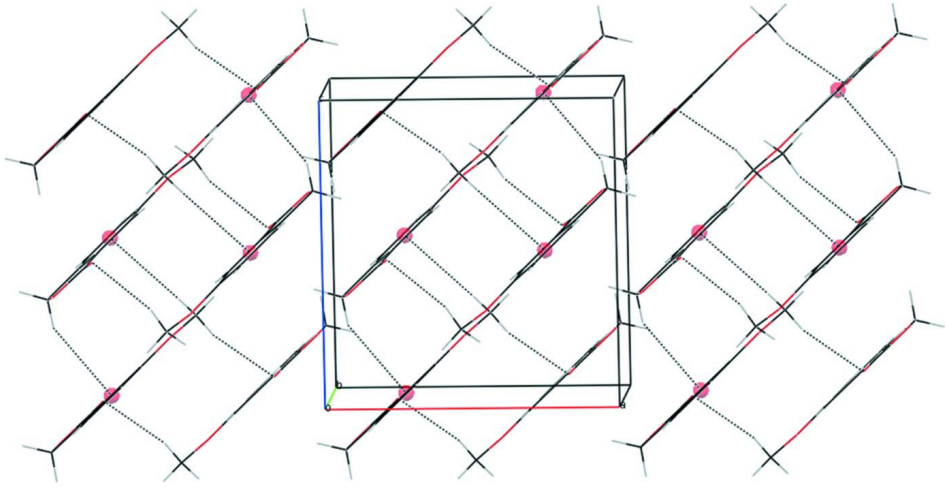

Figure 2

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.

6-Methoxyisobenzofuran-1(3*H*)-one

Crystal data

$C_9H_8O_3$
M_r = 164.15

Monoclinic, $P2_1/c$
a = 9.2922 (19) Å

b = 8.4982 (12) Å
c = 9.786 (2) Å
β = 90.471 (15)°
V = 772.8 (2) Å^3
Z = 4
F(000) = 344
D_x = 1.411 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3082 reflections
θ = 2.1–27.5°
μ = 0.11 mm^{-1}
T = 293 K
Prism, colourless
0.28 × 0.17 × 0.12 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_{int} = 0.036
θ_{max} = 27.5°, θ_{min} = 2.2°
h = −12→12
k = −10→11
l = −12→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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-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 (Å^2)

	x	y	z	U_{iso}*/U_{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, º)

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 (Å, º)

Cg1 is the centroid of C2–C7 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9*B*···O2^{i}	0.96	2.55	3.490 (2)	165
C8—H8*B*···*Cg*1^{ii}	0.97	2.84	3.637 (2)	140
C9—H9*C*···*Cg*1^{iii}	0.96	2.91	3.744 (2)	146

Symmetry codes: (i) −*x*+1, −*y*, −*z*; (ii) *x*, −*y*+3/2, *z*−1/2; (iii) *x*, −*y*+3/2, *z*+1/2.