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5,7-Dimethoxyisobenzofuran-1(3H)-one

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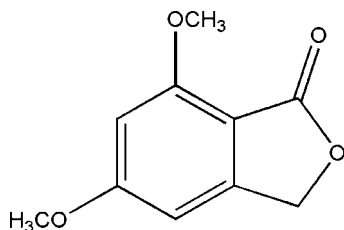
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.052; wR factor = 0.131; data-to-parameter ratio = 12.5.

The asymmetric unit of the title compound, $\text{C}_{10}\text{H}_{10}\text{O}_4$, which has been isolated from rhizoma *Polygonum Cuspidatum*, a Chinese folk medicine, contains two crystallographically independent molecules. The molecules are essentially planar, with a maximum deviation of 0.061 (2) Å from the best planes. The crystal packing is stabilized by weak intermolecular C—H...O hydrogen-bonding interactions, with a stacking direction of the molecules parallel to [101].

Related literature

For the synthesis of 5,7-dimethoxyphthalide, see: Talapatra & Monoj (1980); Dang *et al.* (1999); Orito *et al.* (1995). For the title compound as an intermediate, see: Zuo *et al.* (2008); Lee *et al.* (2001). For the title compound as a byproduct, see: Fürstner *et al.* (2000).



Experimental

Crystal data

$\text{C}_{10}\text{H}_{10}\text{O}_4$
 $M_r = 194.18$
 Monoclinic, $P2_1/c$

$a = 8.532$ (3) Å
 $b = 25.877$ (10) Å
 $c = 8.374$ (3) Å

$\beta = 104.322$ (6)°
 $V = 1791.5$ (11) Å³
 $Z = 8$
 Mo $K\alpha$ radiation

$\mu = 0.11$ mm⁻¹
 $T = 293$ K
 $0.12 \times 0.12 \times 0.10$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.987$, $T_{\max} = 0.989$

7489 measured reflections
 3216 independent reflections
 1766 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.062$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.131$
 $S = 0.93$
 3216 reflections

258 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.18$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C6A}-\text{H6A}\cdots\text{O1B}^{\text{i}}$	0.93	2.51	3.397 (3)	161
$\text{C8A}-\text{H8A1}\cdots\text{O2B}^{\text{ii}}$	0.97	2.53	3.337 (3)	140
$\text{C6B}-\text{H6B}\cdots\text{O1A}^{\text{iii}}$	0.93	2.44	3.325 (3)	159

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

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); 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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2246).

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supplementary materials

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5,7-Dimethoxyisobenzofuran-1(3*H*)-one

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Comment

The compound 5,7-dimethoxyphthalide has been previously reported. It could be obtained by different synthetic strategies, e.g. from 5,7-dihydroxyphthalide (Talapatra & Monoj, 1980), 6-iodo-3-methoxybenzyl alcohols (Dang *et al.*, 1999) or 3,5-dimethoxybenzyl alcohol (Orito *et al.*, 1995). It could act as an intermediate product in the process of synthesizing some significant compounds, such as 5,7-dimethoxy-4-methylphthalide and 5,7-dihydroxy-4-methylphthalide (Zuo *et al.*, 2008), or mycophenolic acid and its analogs (Lee *et al.*, 2001). It was also reported as a byproduct in the synthesis of zearalenone and lasiodiplodin (Fürstner *et al.*, 2000). However, no structural details were provided. In this study, 5,7-dimethoxyphthalide was isolated from the rhizoma *Polygonum cuspidatum* as colorless prismatic crystals.

The molecule (Fig. 1) is essentially planar with a maximum deviation of 0.061 (2) Å from the best planes. The crystal packing is stabilized by weak intermolecular C—H···O hydrogen-bonding interactions with a stacking direction of the molecules parallel to [101] (Fig. 2).

Experimental

The slices of the dried roots of *P. cuspidatum* (10 kg) were extracted with 60% aqueous acetone 3 times (24 h each) at room temperature. The solvent was evaporated in vacuo and some hydrophobic substances precipitated which were filtered off. The filtrate was concentrated to a suitable volume, then chromatographed on a Sephadex LH-20 column eluted with H₂O, aqueous MeOH (10%-70%) and 50% acetone successively to give five fractions. The fraction eluted by 10% MeOH was subjected to MCI gel chromatography eluted with gradient aqueous MeOH solvent. The 30% aqueous MeOH eluate from the MCI column afforded the compound 5,7-dimethoxyphthalide as an amorphous powder. The powder was recrystallized in acetone and produced colourless prismatic crystals.

Refinement

The H atoms were refined at calculated positions riding on the parent carbon atoms (C—H = 0.95–0.99 Å) with isotropic displacement parameters $U_{iso}(H) = 1.2U(C_{eq})$ or $1.5U(-CH_3)$. All CH₃ hydrogen atoms were allowed to rotate but not to tip.

Figures

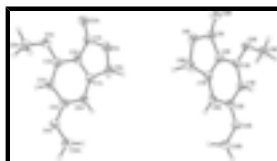


Fig. 1. The molecular structure of 5,7-dimethoxyphthalide, showing the atom-labelling scheme. H atoms are shown as small spheres of arbitrary radius. Displacement ellipsoids are drawn at the 50% probability level.

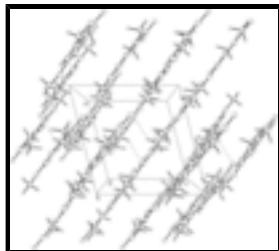


Fig. 2. Molecular packing in the crystal, viewed along the *b* axis. Dashed lines indicate intermolecular C—H...O hydrogen bonds.

5,7-Dimethoxyisobenzofuran-1(3*H*)-one

Crystal data

$C_{10}H_{10}O_4$

$M_r = 194.18$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.532$ (3) Å

$b = 25.877$ (10) Å

$c = 8.374$ (3) Å

$\beta = 104.322$ (6)°

$V = 1791.5$ (11) Å³

$Z = 8$

$F_{000} = 816$

$D_x = 1.440$ Mg m⁻³

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

Cell parameters from 715 reflections

$\theta = 2.6$ – 21.3 °

$\mu = 0.11$ mm⁻¹

$T = 293$ K

Prism, colourless

$0.12 \times 0.12 \times 0.10$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ K

ϕ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.987$, $T_{\max} = 0.989$

7489 measured reflections

3216 independent reflections

1766 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.062$

$\theta_{\max} = 25.2$ °

$\theta_{\min} = 1.6$ °

$h = -7 \rightarrow 10$

$k = -30 \rightarrow 31$

$l = -10 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.052$

$wR(F^2) = 0.131$

$S = 0.93$

3216 reflections

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.053P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.18$ e Å⁻³

$\Delta\rho_{\min} = -0.19$ e Å⁻³

258 parameters

Extinction correction: SHELXL97 (Sheldrick, 2008),

$$F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$$

Primary atom site location: structure-invariant direct methods

Extinction coefficient: 0.0026 (5)

Secondary atom site location: difference Fourier map

Special details

Experimental. The powder of 5,7-dimethoxyphthalide was solved in acetone and produced colorless crystal.

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1A	0.3975 (2)	0.42391 (7)	0.1615 (2)	0.0627 (6)
O2A	0.2605 (3)	0.35976 (8)	0.0110 (2)	0.0674 (6)
O3A	0.4002 (2)	0.25798 (7)	0.1709 (2)	0.0571 (6)
O4A	0.8571 (2)	0.28610 (7)	0.6142 (2)	0.0628 (6)
C1A	0.3670 (4)	0.37248 (11)	0.1270 (3)	0.0518 (8)
C2A	0.4830 (3)	0.34270 (10)	0.2496 (3)	0.0417 (6)
C3A	0.5069 (3)	0.28902 (10)	0.2724 (3)	0.0470 (7)
C4A	0.6341 (3)	0.27237 (10)	0.3971 (3)	0.0471 (7)
H4A	0.6529	0.2372	0.4141	0.057*
C5A	0.7359 (3)	0.30831 (11)	0.4992 (3)	0.0487 (7)
C6A	0.7112 (3)	0.36054 (10)	0.4795 (3)	0.0486 (7)
H6A	0.7780	0.3842	0.5478	0.058*
C7A	0.5835 (3)	0.37640 (10)	0.3545 (3)	0.0452 (7)
C8A	0.5296 (3)	0.43027 (10)	0.3046 (3)	0.0542 (8)
H8A1	0.4942	0.4478	0.3920	0.065*
H8A2	0.6164	0.4500	0.2782	0.065*
C9A	0.4125 (4)	0.20351 (11)	0.2069 (4)	0.0644 (9)
H9A1	0.5177	0.1914	0.2025	0.097*
H9A2	0.3312	0.1853	0.1270	0.097*
H9A3	0.3968	0.1976	0.3150	0.097*
C10A	0.9664 (4)	0.31963 (13)	0.7237 (4)	0.0764 (10)
H10A	1.0202	0.3415	0.6614	0.115*
H10B	1.0451	0.2994	0.7999	0.115*
H10C	0.9072	0.3406	0.7833	0.115*
O1B	-0.0885 (2)	0.46776 (7)	0.6553 (2)	0.0582 (5)
O2B	-0.2380 (2)	0.53224 (8)	0.5190 (2)	0.0674 (6)
O3B	-0.0891 (2)	0.63386 (7)	0.6714 (2)	0.0551 (5)

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O4B	0.3667 (2)	0.60524 (7)	1.1150 (2)	0.0539 (5)
C1B	-0.1230 (3)	0.51938 (12)	0.6263 (3)	0.0522 (8)
C2B	-0.0013 (3)	0.54864 (11)	0.7460 (3)	0.0454 (7)
C3B	0.0172 (3)	0.60196 (10)	0.7725 (3)	0.0415 (7)
C4B	0.1434 (3)	0.61849 (10)	0.8969 (3)	0.0440 (7)
H4B	0.1590	0.6537	0.9162	0.053*
C5B	0.2488 (3)	0.58354 (10)	0.9954 (3)	0.0420 (6)
C6B	0.2297 (3)	0.53046 (10)	0.9714 (3)	0.0418 (6)
H6B	0.2988	0.5069	1.0375	0.050*
C7B	0.1032 (3)	0.51477 (9)	0.8449 (3)	0.0403 (6)
C8B	0.0538 (3)	0.46110 (10)	0.7869 (3)	0.0522 (7)
H8B1	0.1386	0.4442	0.7477	0.063*
H8B2	0.0302	0.4406	0.8750	0.063*
C9B	-0.0723 (4)	0.68778 (10)	0.7035 (4)	0.0620 (8)
H9B1	-0.0863	0.6948	0.8117	0.093*
H9B2	-0.1527	0.7062	0.6232	0.093*
H9B3	0.0335	0.6988	0.6973	0.093*
C10B	0.4778 (3)	0.57130 (11)	1.2222 (3)	0.0589 (8)
H10D	0.4192	0.5485	1.2769	0.088*
H10E	0.5531	0.5913	1.3028	0.088*
H10F	0.5356	0.5514	1.1587	0.088*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0734 (15)	0.0519 (13)	0.0597 (13)	0.0139 (11)	0.0105 (11)	0.0112 (10)
O2A	0.0663 (14)	0.0782 (15)	0.0517 (13)	0.0101 (12)	0.0029 (11)	0.0066 (11)
O3A	0.0585 (13)	0.0487 (13)	0.0580 (12)	0.0039 (10)	0.0028 (10)	-0.0025 (10)
O4A	0.0558 (12)	0.0596 (13)	0.0618 (13)	0.0065 (10)	-0.0066 (11)	-0.0011 (10)
C1A	0.055 (2)	0.059 (2)	0.0442 (18)	0.0101 (16)	0.0194 (16)	0.0078 (15)
C2A	0.0409 (16)	0.0434 (16)	0.0435 (16)	0.0030 (13)	0.0157 (13)	0.0015 (13)
C3A	0.0446 (17)	0.0492 (18)	0.0479 (18)	-0.0023 (14)	0.0129 (14)	-0.0027 (14)
C4A	0.0491 (17)	0.0413 (16)	0.0515 (17)	0.0026 (13)	0.0132 (15)	-0.0009 (13)
C5A	0.0420 (17)	0.0535 (19)	0.0498 (17)	0.0078 (14)	0.0095 (14)	0.0018 (14)
C6A	0.0468 (18)	0.0459 (17)	0.0530 (18)	-0.0022 (13)	0.0120 (15)	-0.0059 (13)
C7A	0.0444 (17)	0.0441 (17)	0.0520 (17)	0.0035 (13)	0.0215 (14)	0.0037 (14)
C8A	0.064 (2)	0.0491 (18)	0.0544 (18)	0.0077 (14)	0.0233 (16)	0.0054 (14)
C9A	0.069 (2)	0.0492 (19)	0.071 (2)	-0.0037 (15)	0.0088 (17)	-0.0008 (15)
C10A	0.068 (2)	0.078 (2)	0.069 (2)	0.0022 (18)	-0.0115 (18)	-0.0118 (18)
O1B	0.0522 (13)	0.0547 (13)	0.0659 (13)	-0.0078 (10)	0.0111 (10)	-0.0179 (10)
O2B	0.0443 (12)	0.0910 (16)	0.0590 (13)	-0.0001 (12)	-0.0020 (10)	-0.0168 (11)
O3B	0.0514 (12)	0.0544 (13)	0.0538 (12)	0.0078 (10)	0.0018 (9)	0.0017 (10)
O4B	0.0504 (12)	0.0478 (11)	0.0521 (12)	0.0024 (9)	-0.0089 (10)	-0.0010 (9)
C1B	0.0381 (18)	0.067 (2)	0.0520 (19)	-0.0063 (15)	0.0115 (15)	-0.0141 (15)
C2B	0.0382 (16)	0.0581 (18)	0.0409 (16)	-0.0021 (14)	0.0117 (13)	-0.0038 (14)
C3B	0.0381 (16)	0.0455 (17)	0.0410 (16)	0.0036 (13)	0.0098 (13)	0.0033 (13)
C4B	0.0428 (16)	0.0402 (16)	0.0474 (16)	-0.0020 (13)	0.0080 (14)	0.0000 (13)
C5B	0.0386 (16)	0.0485 (18)	0.0387 (15)	-0.0018 (13)	0.0090 (13)	-0.0029 (13)

C6B	0.0381 (16)	0.0438 (16)	0.0437 (16)	0.0047 (12)	0.0107 (13)	0.0030 (12)
C7B	0.0417 (16)	0.0397 (16)	0.0431 (15)	0.0003 (13)	0.0175 (13)	-0.0005 (13)
C8B	0.0486 (18)	0.0493 (18)	0.0581 (18)	-0.0027 (14)	0.0118 (14)	-0.0050 (14)
C9B	0.064 (2)	0.050 (2)	0.068 (2)	0.0100 (15)	0.0074 (16)	0.0063 (15)
C10B	0.0473 (19)	0.062 (2)	0.0578 (19)	0.0068 (15)	-0.0056 (15)	-0.0028 (15)

Geometric parameters (Å, °)

O1A—C1A	1.373 (3)	O1B—C1B	1.376 (3)
O1A—C8A	1.437 (3)	O1B—C8B	1.434 (3)
O2A—C1A	1.200 (3)	O2B—C1B	1.202 (3)
O3A—C3A	1.346 (3)	O3B—C3B	1.357 (3)
O3A—C9A	1.440 (3)	O3B—C9B	1.422 (3)
O4A—C5A	1.355 (3)	O4B—C5B	1.354 (3)
O4A—C10A	1.428 (3)	O4B—C10B	1.434 (3)
C1A—C2A	1.458 (4)	C1B—C2B	1.463 (4)
C2A—C7A	1.377 (3)	C2B—C7B	1.372 (3)
C2A—C3A	1.410 (4)	C2B—C3B	1.400 (4)
C3A—C4A	1.376 (3)	C3B—C4B	1.368 (3)
C4A—C5A	1.408 (4)	C4B—C5B	1.393 (3)
C4A—H4A	0.9300	C4B—H4B	0.9300
C5A—C6A	1.371 (4)	C5B—C6B	1.392 (4)
C6A—C7A	1.374 (3)	C6B—C7B	1.373 (3)
C6A—H6A	0.9300	C6B—H6B	0.9300
C7A—C8A	1.495 (3)	C7B—C8B	1.497 (3)
C8A—H8A1	0.9700	C8B—H8B1	0.9700
C8A—H8A2	0.9700	C8B—H8B2	0.9700
C9A—H9A1	0.9599	C9B—H9B1	0.9599
C9A—H9A2	0.9599	C9B—H9B2	0.9599
C9A—H9A3	0.9599	C9B—H9B3	0.9599
C10A—H10A	0.9599	C10B—H10D	0.9599
C10A—H10B	0.9599	C10B—H10E	0.9599
C10A—H10C	0.9599	C10B—H10F	0.9599
C1A—O1A—C8A	110.8 (2)	C1B—O1B—C8B	110.8 (2)
C3A—O3A—C9A	116.7 (2)	C3B—O3B—C9B	117.3 (2)
C5A—O4A—C10A	117.5 (2)	C5B—O4B—C10B	117.7 (2)
O2A—C1A—O1A	120.2 (3)	O2B—C1B—O1B	120.0 (3)
O2A—C1A—C2A	132.1 (3)	O2B—C1B—C2B	132.7 (3)
O1A—C1A—C2A	107.7 (2)	O1B—C1B—C2B	107.3 (2)
C7A—C2A—C3A	119.4 (2)	C7B—C2B—C3B	120.2 (2)
C7A—C2A—C1A	108.8 (2)	C7B—C2B—C1B	109.1 (3)
C3A—C2A—C1A	131.8 (3)	C3B—C2B—C1B	130.6 (3)
O3A—C3A—C4A	125.1 (3)	O3B—C3B—C4B	124.3 (2)
O3A—C3A—C2A	116.7 (2)	O3B—C3B—C2B	118.0 (2)
C4A—C3A—C2A	118.2 (2)	C4B—C3B—C2B	117.7 (2)
C3A—C4A—C5A	120.4 (3)	C3B—C4B—C5B	121.3 (2)
C3A—C4A—H4A	119.8	C3B—C4B—H4B	119.4
C5A—C4A—H4A	119.8	C5B—C4B—H4B	119.4
O4A—C5A—C6A	124.8 (3)	O4B—C5B—C6B	123.7 (2)

supplementary materials

O4A—C5A—C4A	113.5 (2)	O4B—C5B—C4B	114.9 (2)
C6A—C5A—C4A	121.6 (3)	C6B—C5B—C4B	121.3 (2)
C5A—C6A—C7A	117.1 (2)	C7B—C6B—C5B	116.4 (2)
C5A—C6A—H6A	121.5	C7B—C6B—H6B	121.8
C7A—C6A—H6A	121.5	C5B—C6B—H6B	121.8
C6A—C7A—C2A	123.3 (2)	C2B—C7B—C6B	123.1 (2)
C6A—C7A—C8A	128.5 (3)	C2B—C7B—C8B	107.9 (2)
C2A—C7A—C8A	108.2 (2)	C6B—C7B—C8B	129.0 (2)
O1A—C8A—C7A	104.5 (2)	O1B—C8B—C7B	104.8 (2)
O1A—C8A—H8A1	110.9	O1B—C8B—H8B1	110.8
C7A—C8A—H8A1	110.9	C7B—C8B—H8B1	110.8
O1A—C8A—H8A2	110.9	O1B—C8B—H8B2	110.8
C7A—C8A—H8A2	110.9	C7B—C8B—H8B2	110.8
H8A1—C8A—H8A2	108.9	H8B1—C8B—H8B2	108.9
O3A—C9A—H9A1	109.5	O3B—C9B—H9B1	109.5
O3A—C9A—H9A2	109.5	O3B—C9B—H9B2	109.5
H9A1—C9A—H9A2	109.5	H9B1—C9B—H9B2	109.5
O3A—C9A—H9A3	109.5	O3B—C9B—H9B3	109.5
H9A1—C9A—H9A3	109.5	H9B1—C9B—H9B3	109.5
H9A2—C9A—H9A3	109.5	H9B2—C9B—H9B3	109.5
O4A—C10A—H10A	109.5	O4B—C10B—H10D	109.5
O4A—C10A—H10B	109.5	O4B—C10B—H10E	109.5
H10A—C10A—H10B	109.5	H10D—C10B—H10E	109.5
O4A—C10A—H10C	109.5	O4B—C10B—H10F	109.5
H10A—C10A—H10C	109.5	H10D—C10B—H10F	109.5
H10B—C10A—H10C	109.5	H10E—C10B—H10F	109.5
C8A—O1A—C1A—O2A	-179.8 (2)	C8B—O1B—C1B—O2B	179.5 (2)
C8A—O1A—C1A—C2A	-0.5 (3)	C8B—O1B—C1B—C2B	-1.5 (3)
O2A—C1A—C2A—C7A	178.2 (3)	O2B—C1B—C2B—C7B	178.6 (3)
O1A—C1A—C2A—C7A	-1.0 (3)	O1B—C1B—C2B—C7B	-0.3 (3)
O2A—C1A—C2A—C3A	-0.6 (5)	O2B—C1B—C2B—C3B	0.5 (5)
O1A—C1A—C2A—C3A	-179.8 (3)	O1B—C1B—C2B—C3B	-178.5 (2)
C9A—O3A—C3A—C4A	6.2 (4)	C9B—O3B—C3B—C4B	-3.4 (4)
C9A—O3A—C3A—C2A	-172.6 (2)	C9B—O3B—C3B—C2B	177.8 (2)
C7A—C2A—C3A—O3A	177.0 (2)	C7B—C2B—C3B—O3B	180.0 (2)
C1A—C2A—C3A—O3A	-4.3 (4)	C1B—C2B—C3B—O3B	-2.0 (4)
C7A—C2A—C3A—C4A	-1.9 (4)	C7B—C2B—C3B—C4B	1.1 (4)
C1A—C2A—C3A—C4A	176.8 (3)	C1B—C2B—C3B—C4B	179.1 (2)
O3A—C3A—C4A—C5A	-178.2 (2)	O3B—C3B—C4B—C5B	-179.5 (2)
C2A—C3A—C4A—C5A	0.6 (4)	C2B—C3B—C4B—C5B	-0.7 (4)
C10A—O4A—C5A—C6A	0.5 (4)	C10B—O4B—C5B—C6B	0.2 (4)
C10A—O4A—C5A—C4A	-179.9 (2)	C10B—O4B—C5B—C4B	179.1 (2)
C3A—C4A—C5A—O4A	-178.9 (2)	C3B—C4B—C5B—O4B	-179.2 (2)
C3A—C4A—C5A—C6A	0.7 (4)	C3B—C4B—C5B—C6B	-0.2 (4)
O4A—C5A—C6A—C7A	179.0 (2)	O4B—C5B—C6B—C7B	179.6 (2)
C4A—C5A—C6A—C7A	-0.6 (4)	C4B—C5B—C6B—C7B	0.8 (4)
C5A—C6A—C7A—C2A	-0.8 (4)	C3B—C2B—C7B—C6B	-0.6 (4)
C5A—C6A—C7A—C8A	-179.5 (3)	C1B—C2B—C7B—C6B	-179.0 (2)
C3A—C2A—C7A—C6A	2.1 (4)	C3B—C2B—C7B—C8B	-179.8 (2)

C1A—C2A—C7A—C6A	-176.9 (2)	C1B—C2B—C7B—C8B	1.8 (3)
C3A—C2A—C7A—C8A	-178.9 (2)	C5B—C6B—C7B—C2B	-0.4 (4)
C1A—C2A—C7A—C8A	2.1 (3)	C5B—C6B—C7B—C8B	178.7 (2)
C1A—O1A—C8A—C7A	1.7 (3)	C1B—O1B—C8B—C7B	2.5 (3)
C6A—C7A—C8A—O1A	176.6 (2)	C2B—C7B—C8B—O1B	-2.6 (3)
C2A—C7A—C8A—O1A	-2.3 (3)	C6B—C7B—C8B—O1B	178.2 (2)

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

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C6A—H6A \cdots O1B ⁱ	0.93	2.51	3.397 (3)	161
C8A—H8A1 \cdots O2B ⁱⁱ	0.97	2.53	3.337 (3)	140
C6B—H6B \cdots O1A ⁱⁱⁱ	0.93	2.44	3.325 (3)	159

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

Fig. 1

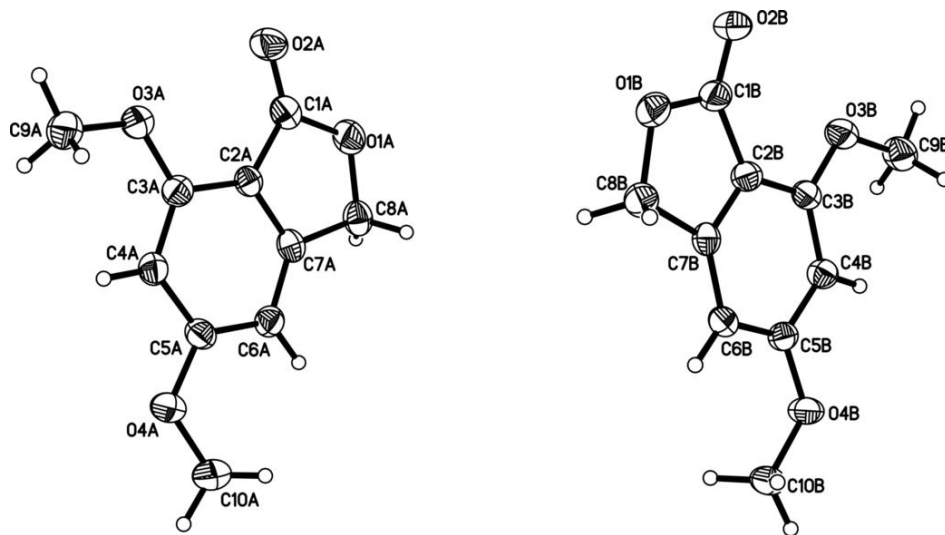


Fig. 2

