# organic compounds

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

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The asymmetric unit of the title compound, $C_{10}H_{10}O_4$, 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).
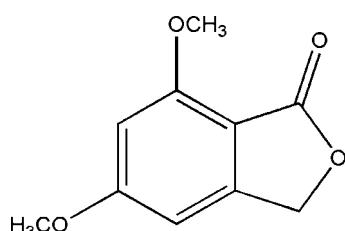


## Experimental

### Crystal data

| | |
|---|---|
| $C_{10}H_{10}O_4$ | $a = 8.532$ (3) Å |
| $M_r = 194.18$ | $b = 25.877$ (10) Å |
| Monoclinic, $P2_1/c$ | $c = 8.374$ (3) Å |

| | |
|---|---|
| $\beta = 104.322$ (6)° | $\mu = 0.11$ mm$^{-1}$ |
| $V = 1791.5$ (11) Å$^3$ | $T = 293$ K |
| $Z = 8$ | $0.12 \times 0.12 \times 0.10$ mm |
| Mo $K\alpha$ radiation | |

### Data collection

| | |
|---|---|
| Bruker SMART APEX CCD area-detector diffractometer | 7489 measured reflections |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | 3216 independent reflections |
| $T_{min} = 0.987$, $T_{max} = 0.989$ | 1766 reflections with $I > 2\sigma(I)$ |
| | $R_{int} = 0.062$ |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.052$ | 258 parameters |
| $wR(F^2) = 0.131$ | H-atom parameters constrained |
| $S = 0.93$ | $\Delta\rho_{max} = 0.18$ e Å$^{-3}$ |
| 3216 reflections | $\Delta\rho_{min} = -0.19$ e Å$^{-3}$ |

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

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

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

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).

## References

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# supporting information

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

**Ming-Xue Sun, Xu Li, Wen-Yong Liu and Kai Xiao**

### S1. 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-methyl-phthaIide (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 ).

### S2. Experimental
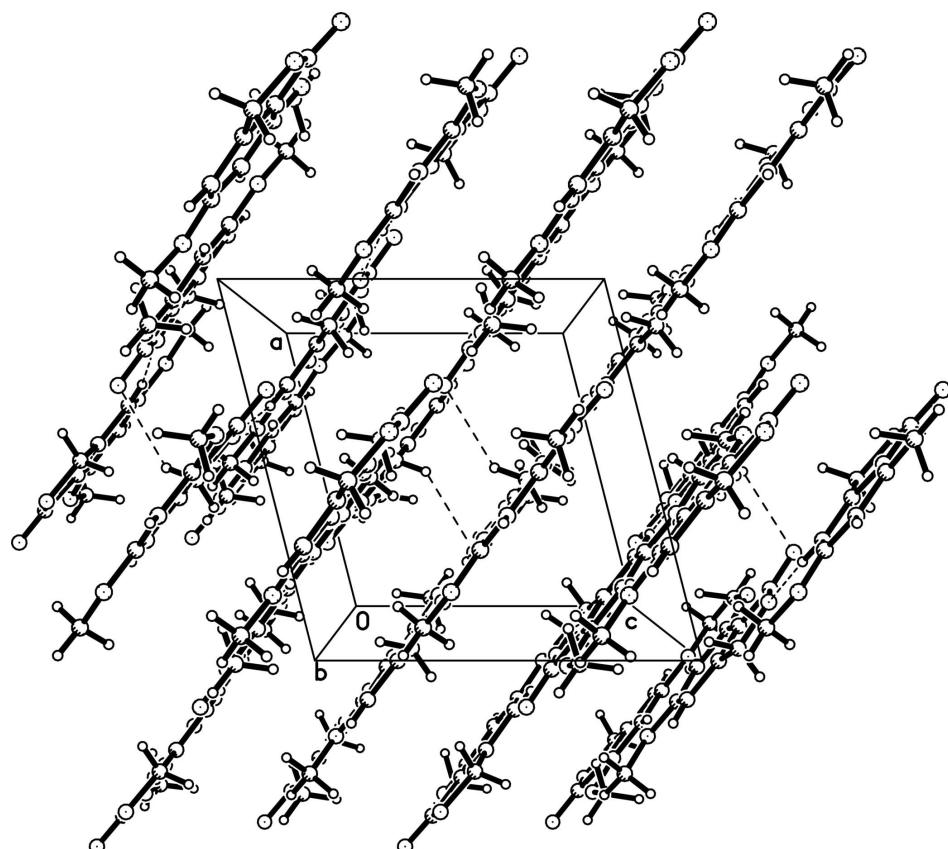
The slices of the dried roots of *P. cuspidatum* (10 kg) were extracted with 60% aqueous acetone 3 times (24 h each) at room temperature. The solvent was evaporated in vacuo and some hydrophobic substances precipitated which were filtered off. The filtrate was concentrated to a suitable volume, then chromatographed on a Sephadex LH-20 column eluted with $H_2O$, aqueous MeOH (10%-70%) and 50% acetone successively to give five fractions. The fraction eluated 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.

### S3. 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 $U_{iso}(H) = 1.2U(Ceq)$ or $1.5U(–CH_3)$. All $CH_3$ hydrogen atoms were allowed to rotate but not to tip.
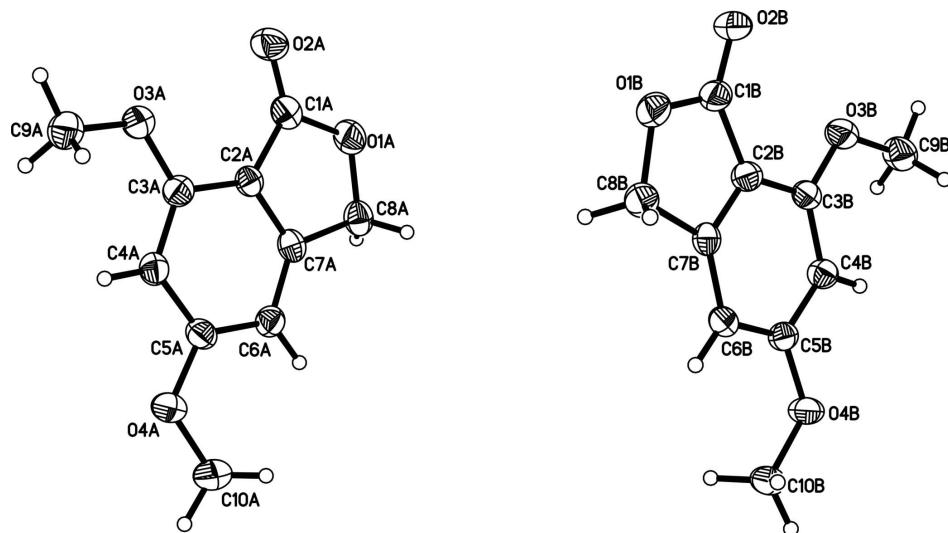
**Figure 1**

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.



**Figure 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*

| | |
|---|---|
| $C_{10}H_{10}O_4$ | $F(000) = 816$ |
| $M_r = 194.18$ | $D_x = 1.440$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 715 reflections |
| $a = 8.532$ (3) Å | $\theta = 2.6–21.3°$ |
| $b = 25.877$ (10) Å | $\mu = 0.11$ mm$^{-1}$ |
| $c = 8.374$ (3) Å | $T = 293$ K |
| $\beta = 104.322$ (6)° | Prism, colourless |
| $V = 1791.5$ (11) Å$^3$ | $0.12 \times 0.12 \times 0.10$ mm |
| $Z = 8$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX CCD area-detector diffractometer | 7489 measured reflections |
| | 3216 independent reflections |
| Radiation source: fine-focus sealed tube | 1766 reflections with $I > 2\sigma(I)$ |
| Graphite monochromator | $R_{int} = 0.062$ |
| $\varphi$ and $\omega$ scans | $\theta_{max} = 25.2°$, $\theta_{min} = 1.6°$ |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) | $h = -7 \rightarrow 10$ |
| | $k = -30 \rightarrow 31$ |
| $T_{min} = 0.987$, $T_{max} = 0.989$ | $l = -10 \rightarrow 8$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.052$ | H-atom parameters constrained |
| $wR(F^2) = 0.131$ | $w = 1/[\sigma^2(F_o^2) + (0.053P)^2]$ |
| $S = 0.93$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 3216 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 258 parameters | $\Delta\rho_{max} = 0.18$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.19$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc$^*$=kFc[1+0.001xFc$^2\lambda^3$/sin(2$\theta$)]$^{-1/4}$ |
| Secondary atom site location: difference Fourier map | Extinction coefficient: 0.0026 (5) |

*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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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) |
| 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 (Å²)*

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

| | | | |
|---|---|---|---|
| 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) |
| 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 (Å, º)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| C6A—H6A···O1Bⁱ | 0.93 | 2.51 | 3.397 (3) | 161 |
| C8A—H8A1···O2Bⁱⁱ | 0.97 | 2.53 | 3.337 (3) | 140 |
| C6B—H6B···O1Aⁱⁱⁱ | 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.