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# 1,1′:4′,1″-Terphenyl-2′,5′-dicarboxylic acid dimethyl sulfoxide-$d_6$ disolvate

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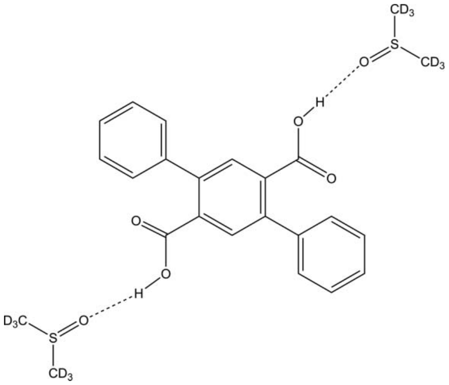
Key indicators: single-crystal X-ray study; $T$ = 150 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.049; $wR$ factor = 0.145; data-to-parameter ratio = 14.2.

The asymmetric unit of the title solvate, $C_{20}H_{14}O_4\cdot 2C_2D_6OS$, contains half of the substituted terephthalic acid molecule and one solvent molecule. The centroid of the central benzene ring in the acid molecule is coincident with a crystallographic inversion center. Neither the carboxyl nor the phenyl substituents are coplanar with the central aromatic ring, showing dihedral angles of 53.18 (11) and 47.83 (11)°, respectively. The dimethyl sulfoxide solvent molecules are hydrogen bonded to the carboxylic acid groups.

## Related literature

For the synthesis of the title compound, see: Deuschel (1951); Ebel & Deuschel (1956). For similar molecules, see: Tanaka *et al.* (2009).



## Experimental

### *Crystal data*

| | |
|---|---|
| $C_{20}H_{14}O_4\cdot 2C_2D_6OS$ | $\gamma$ = 93.127 (2)° |
| $M_r$ = 486.61 | $V$ = 599.26 (14) Å$^3$ |
| Triclinic, $P\overline{1}$ | $Z$ = 1 |
| $a$ = 6.5184 (9) Å | Mo $K\alpha$ radiation |
| $b$ = 8.8273 (12) Å | $\mu$ = 0.26 mm$^{-1}$ |
| $c$ = 10.6017 (14) Å | $T$ = 150 K |
| $\alpha$ = 97.076 (2)° | 0.29 × 0.22 × 0.10 mm |
| $\beta$ = 97.074 (2)° | |

### *Data collection*

| | |
|---|---|
| Siemens SMART CCD area-detector diffractometer | 3730 measured reflections |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2001) | 2101 independent reflections |
| $T_{min}$ = 0.929, $T_{max}$ = 0.975 | 1707 reflections with $I > 2\sigma(I)$ |
| | $R_{int}$ = 0.012 |

### *Refinement*

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.049 | 148 parameters |
| $wR(F^2)$ = 0.145 | H-atom parameters constrained |
| $S$ = 1.04 | $\Delta\rho_{max}$ = 0.53 e Å$^{-3}$ |
| 2101 reflections | $\Delta\rho_{min}$ = −0.17 e Å$^{-3}$ |

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

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| O1—H1O⋯O3$^{i}$ | 0.926 | 1.659 | 2.581 (2) | 174 |

Symmetry code: (i) $x + 1, y, z$.

Data collection: *SMART-NT* (Bruker, 2001); cell refinement: *SAINT-NT* (Bruker, 1999); data reduction: *SAINT-NT*; program(s) used to solve structure: *SHELXTL-NT* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL-NT*; molecular graphics: *SHELXTL-NT*; software used to prepare material for publication: *SHELXTL-NT*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FY2045).

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# supplementary materials

*Acta Cryst.* (2012). E**68**, o1192 [doi:10.1107/S1600536812012056]

## 1,1′:4′,1′′-Terphenyl-2′,5′-dicarboxylic acid dimethyl sulfoxide-$d_6$ disolvate

**Lucian C. Pop, Marcelo Preite, Juan Manuel Manriquez, Andrés Vega and Ivonne Chavez**

### Comment

2,5-Diphenyl-1,4-benzenedicarboxylic acid (2,5-Diphenylterephthalic acid) has been described previously as a precursor towards *trans*-fluorenacenedione, prepared by its dehydration with sulfuric acid (Ebel & Deuschel, 1956).

The compound has a central terephtalic acid core, substituted at positions 2 and 5 with phenyl groups. The molecule has been previoulsy described by Tanaka *et al.* (Tanaka *et al.*, 2009) in the form of an ethanol solvate. Tanaka *et al.* also described a related compound where the phenyl groups were replaced by *p*-fluorophenyl groups, which crystallized as a dimethylformamide solvate.

The carboxylic acid and the phenyl groups are highly planar. They define dihedral angles of 53.18 (11)° and 47.83 (11)°, respectively, with the central aromatic ring. The corresponding values are 28.2 (2)° and 57.2 (1)°, and 115.3 (1)° and 46.3 (1)°, for both dihedral angles in the ethanol solvate, and in the *p*-fluoro compound, respectively.

The molecule has an inversion center (crystallographic) coincident with the centroid of the central ring, so the point group symmetry of the isolated molecule is $C_i$. The same happens for the ethanol solvate and the *p*-fluoro compounds respectively.

The packing shows a deuterated dimethyl sulfoxide solvent molecule hydrogen bonded with each carboxylic acid group, with $O1\cdots O3^{ii}$ distance of 2.581 (2) Å (ii: $x + 1, y, z$). There are two molecules of solvent for a single diacid molecule, each one defining one of the aforementioned hydrogen bond with each carboxylic acid group. A closely related pattern occurs for the ethanol solvate of the title molecule and the dimethylformamide solvate of the *p*-fluoro derivative (Tanaka *et al.*, 2009): two molecules of solvent, ethanol or dimethylformamide, are bonded by hydrogen bonds to both carboxylic acid groups.

### Experimental

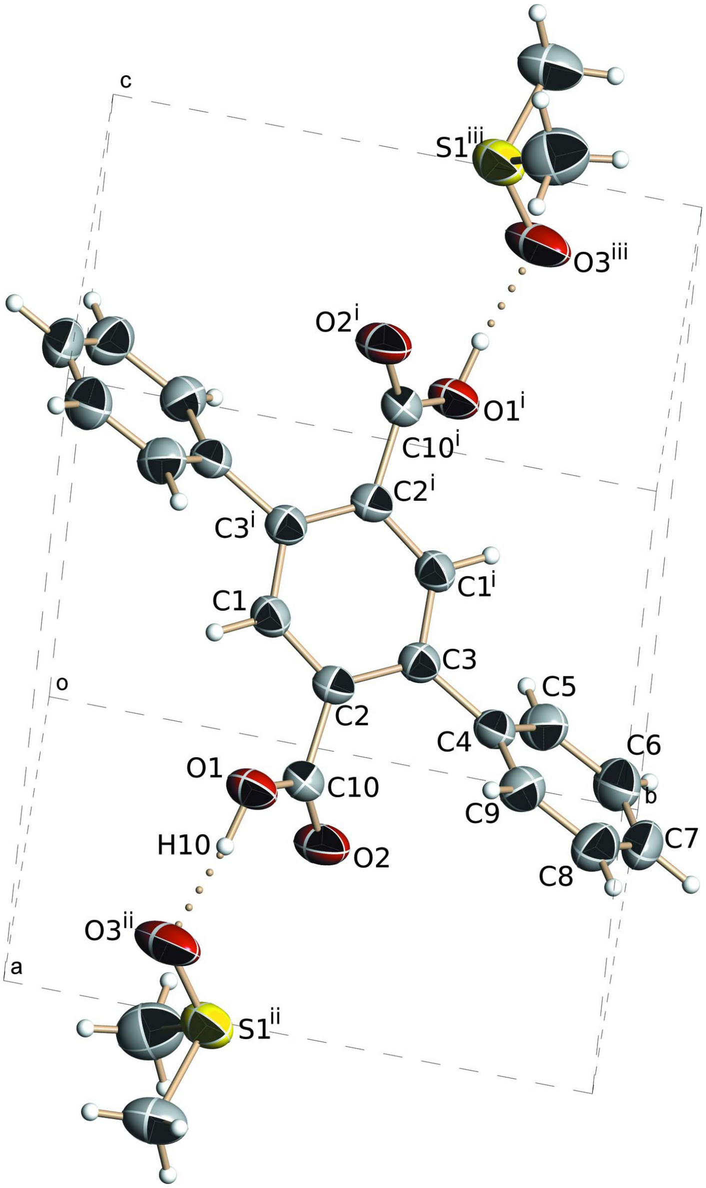
The compound was prepared by a method described in the literature (Deuschel, 1951; Ebel & Deuschel, 1956), slighty modified by using $d_6$-DMSO for crystallization instead of $C_6H_5CN$, giving the DMSO-clathrate. The title compound was prepared in a 93% yield, mp. = 280°C (dec).

### Refinement

The hydrogen atoms positions were calculated after each cycle of refinement with *SHELXL* (Bruker,1999) using a riding model, with C—H distances in the range of 0.93 to 0.96 Å. $U_{iso}$(H) values were set equal to $1.5U_{eq}$ of the parent carbon atom for methyl groups, and $1.2U_{eq}$ for the others. The carboxylic acid hydrogen atom was located in the difference Fourier map, and its coordinates were subsequentely kept fixed (by adding 10 to the coordinates in SHELXL), while $U_{iso}$(H) was left free to refine.

## Computing details

Data collection: *SMART-NT* (Bruker, 2001); cell refinement: *SAINT-NT* (Bruker, 1999); data reduction: *SAINT-NT* (Bruker, 1999); program(s) used to solve structure: *SHELXTL-NT* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL-NT* (Sheldrick, 2008); molecular graphics: *SHELXTL-NT* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL-NT* (Sheldrick, 2008).



### Figure 1

Molecular structure diagram for **I**, showing the numbering scheme. Displacement ellipsoids are drawn at the 25% probability level and H atoms are shown as spheres of arbitrary radii. Symmetry codes (i): $-x+1, -y+1, -z+1$; (ii) $x + 1, y, z$; (iii): $-x, -y+1, -z+1$.

## 1,1′:4′,1″-Terphenyl-2′,5′-dicarboxylic acid dimethyl sulfoxide-$d_6$ disolvate

*Crystal data*

$C_{20}H_{14}O_4 \cdot 2C_2D_6OS$
$M_r = 486.61$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.5184$ (9) Å
$b = 8.8273$ (12) Å
$c = 10.6017$ (14) Å
$\alpha = 97.076$ (2)°
$\beta = 97.074$ (2)°
$\gamma = 93.127$ (2)°
$V = 599.26$ (14) Å$^3$

$Z = 1$
$F(000) = 250$
$D_x = 1.348$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1643 reflections
$\theta = 2.3$–24.2°
$\mu = 0.26$ mm$^{-1}$
$T = 150$ K
Block, colorless
0.29 × 0.22 × 0.10 mm

*Data collection*

Siemens SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.929$, $T_{max} = 0.975$

3730 measured reflections
2101 independent reflections
1707 reflections with $I > 2\sigma(I)$
$R_{int} = 0.012$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.3°$
$h = -7 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -12 \rightarrow 12$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.145$
$S = 1.04$
2101 reflections
148 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
$w = 1/[\sigma^2(F_o^2) + (0.0891P)^2 + 0.1414P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.53$ e Å$^{-3}$
$\Delta\rho_{min} = -0.17$ 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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.6023 (3) | 0.3696 (2) | 0.48626 (19) | 0.0397 (5) |
| H1 | 0.6737 | 0.2814 | 0.4770 | 0.048* |
| C2 | 0.6546 (3) | 0.4883 (2) | 0.41945 (19) | 0.0371 (5) |
| C10 | 0.8077 (3) | 0.4593 (2) | 0.3263 (2) | 0.0408 (5) |

| | | | | |
|---|---|---|---|---|
| O1 | 0.9582 (2) | 0.37837 (18) | 0.36916 (14) | 0.0503 (4) |
| O2 | 0.7903 (3) | 0.5003 (2) | 0.22114 (16) | 0.0603 (5) |
| H10 | 1.0348 | 0.3461 | 0.3042 | 0.073 (8)* |
| C3 | 0.5515 (3) | 0.6244 (2) | 0.43352 (19) | 0.0385 (5) |
| C4 | 0.6018 (3) | 0.7627 (2) | 0.3727 (2) | 0.0413 (5) |
| C5 | 0.4423 (4) | 0.8397 (3) | 0.3156 (2) | 0.0532 (6) |
| H5 | 0.3059 | 0.8007 | 0.3118 | 0.064* |
| C6 | 0.4828 (5) | 0.9733 (3) | 0.2644 (3) | 0.0637 (7) |
| H6 | 0.3746 | 1.0236 | 0.2265 | 0.076* |
| C7 | 0.6846 (5) | 1.0312 (3) | 0.2699 (3) | 0.0640 (7) |
| H7 | 0.7132 | 1.1199 | 0.2343 | 0.077* |
| C8 | 0.8446 (4) | 0.9578 (3) | 0.3283 (2) | 0.0607 (7) |
| H8 | 0.9807 | 0.9980 | 0.3329 | 0.073* |
| C9 | 0.8033 (4) | 0.8244 (3) | 0.3801 (2) | 0.0500 (6) |
| H9 | 0.9118 | 0.7760 | 0.4200 | 0.060* |
| S1 | 0.18071 (11) | 0.35077 (8) | 0.07652 (6) | 0.0630 (3) |
| O3 | 0.1731 (3) | 0.2709 (3) | 0.19405 (18) | 0.0834 (7) |
| C11 | 0.4021 (4) | 0.2871 (4) | 0.0103 (3) | 0.0744 (8) |
| D11A | 0.5249 | 0.3310 | 0.0647 | 0.112* |
| D11B | 0.4020 | 0.3183 | −0.0733 | 0.112* |
| D11C | 0.3993 | 0.1776 | 0.0035 | 0.112* |
| C12 | −0.0093 (5) | 0.2526 (4) | −0.0412 (3) | 0.0850 (10) |
| D12A | 0.0088 | 0.1449 | −0.0479 | 0.127* |
| D12B | 0.0036 | 0.2878 | −0.1222 | 0.127* |
| D12C | −0.1442 | 0.2715 | −0.0180 | 0.127* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0418 (11) | 0.0386 (11) | 0.0396 (11) | 0.0091 (8) | 0.0070 (9) | 0.0044 (9) |
| C2 | 0.0362 (10) | 0.0422 (11) | 0.0333 (10) | 0.0052 (8) | 0.0054 (8) | 0.0045 (8) |
| C10 | 0.0433 (11) | 0.0400 (11) | 0.0397 (12) | 0.0049 (9) | 0.0094 (9) | 0.0030 (9) |
| O1 | 0.0478 (9) | 0.0628 (10) | 0.0453 (9) | 0.0194 (7) | 0.0156 (7) | 0.0105 (7) |
| O2 | 0.0705 (11) | 0.0738 (12) | 0.0461 (10) | 0.0263 (9) | 0.0230 (8) | 0.0208 (8) |
| C3 | 0.0386 (11) | 0.0411 (11) | 0.0359 (11) | 0.0049 (8) | 0.0046 (8) | 0.0050 (8) |
| C4 | 0.0470 (12) | 0.0416 (12) | 0.0377 (11) | 0.0087 (9) | 0.0115 (9) | 0.0065 (9) |
| C5 | 0.0520 (13) | 0.0555 (15) | 0.0569 (14) | 0.0134 (11) | 0.0118 (11) | 0.0177 (11) |
| C6 | 0.0789 (18) | 0.0539 (15) | 0.0655 (17) | 0.0245 (13) | 0.0157 (14) | 0.0211 (12) |
| C7 | 0.098 (2) | 0.0383 (13) | 0.0591 (16) | 0.0018 (13) | 0.0223 (14) | 0.0104 (11) |
| C8 | 0.0687 (16) | 0.0521 (15) | 0.0605 (16) | −0.0109 (12) | 0.0133 (13) | 0.0055 (12) |
| C9 | 0.0497 (13) | 0.0495 (13) | 0.0519 (14) | 0.0036 (10) | 0.0091 (10) | 0.0090 (10) |
| S1 | 0.0719 (5) | 0.0675 (5) | 0.0573 (5) | 0.0266 (3) | 0.0223 (3) | 0.0148 (3) |
| O3 | 0.1020 (16) | 0.1079 (16) | 0.0601 (12) | 0.0620 (13) | 0.0431 (11) | 0.0331 (11) |
| C11 | 0.0644 (17) | 0.102 (2) | 0.0625 (17) | 0.0194 (16) | 0.0214 (14) | 0.0155 (16) |
| C12 | 0.0634 (18) | 0.104 (2) | 0.091 (2) | 0.0067 (17) | 0.0121 (16) | 0.0226 (19) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.385 (3) | C6—H6 | 0.9300 |
| C1—C3$^{i}$ | 1.391 (3) | C7—C8 | 1.380 (4) |

| | | | |
|---|---|---|---|
| C1—H1 | 0.9300 | C7—H7 | 0.9300 |
| C2—C3 | 1.410 (3) | C8—C9 | 1.386 (3) |
| C2—C10 | 1.498 (3) | C8—H8 | 0.9300 |
| C10—O2 | 1.209 (3) | C9—H9 | 0.9300 |
| C10—O1 | 1.314 (3) | S1—O3 | 1.5101 (19) |
| O1—H10 | 0.926 | S1—C12 | 1.754 (3) |
| C3—C1$^{i}$ | 1.391 (3) | S1—C11 | 1.769 (3) |
| C3—C4 | 1.489 (3) | C11—D11A | 0.9600 |
| C4—C9 | 1.384 (3) | C11—D11B | 0.9600 |
| C4—C5 | 1.390 (3) | C11—D11C | 0.9600 |
| C5—C6 | 1.384 (3) | C12—D12A | 0.9600 |
| C5—H5 | 0.9300 | C12—D12B | 0.9600 |
| C6—C7 | 1.377 (4) | C12—D12C | 0.9600 |

| | | | |
|---|---|---|---|
| C2—C1—C3$^{i}$ | 123.41 (19) | C6—C7—H7 | 120.0 |
| C2—C1—H1 | 118.3 | C8—C7—H7 | 120.0 |
| C3$^{i}$—C1—H1 | 118.3 | C7—C8—C9 | 120.2 (2) |
| C1—C2—C3 | 119.46 (18) | C7—C8—H8 | 119.9 |
| C1—C2—C10 | 117.34 (18) | C9—C8—H8 | 119.9 |
| C3—C2—C10 | 122.99 (19) | C4—C9—C8 | 120.4 (2) |
| O2—C10—O1 | 123.55 (19) | C4—C9—H9 | 119.8 |
| O2—C10—C2 | 123.75 (19) | C8—C9—H9 | 119.8 |
| O1—C10—C2 | 112.63 (18) | O3—S1—C12 | 105.90 (15) |
| C10—O1—H10 | 110.50 | O3—S1—C11 | 105.11 (13) |
| C1$^{i}$—C3—C2 | 117.12 (19) | C12—S1—C11 | 98.15 (16) |
| C1$^{i}$—C3—C4 | 118.39 (18) | S1—C11—D11A | 109.5 |
| C2—C3—C4 | 124.45 (18) | S1—C11—D11B | 109.5 |
| C9—C4—C5 | 118.6 (2) | D11A—C11—D11B | 109.5 |
| C9—C4—C3 | 121.63 (19) | S1—C11—D11C | 109.5 |
| C5—C4—C3 | 119.6 (2) | D11A—C11—D11C | 109.5 |
| C6—C5—C4 | 121.2 (2) | D11B—C11—D11C | 109.5 |
| C6—C5—H5 | 119.4 | S1—C12—D12A | 109.5 |
| C4—C5—H5 | 119.4 | S1—C12—D12B | 109.5 |
| C7—C6—C5 | 119.5 (2) | D12A—C12—D12B | 109.5 |
| C7—C6—H6 | 120.2 | S1—C12—D12C | 109.5 |
| C5—C6—H6 | 120.2 | D12A—C12—D12C | 109.5 |
| C6—C7—C8 | 120.1 (2) | D12B—C12—D12C | 109.5 |

| | | | |
|---|---|---|---|
| C3$^{i}$—C1—C2—C3 | 1.1 (3) | C2—C3—C4—C9 | −48.8 (3) |
| C3$^{i}$—C1—C2—C10 | −173.80 (19) | C1$^{i}$—C3—C4—C5 | −46.1 (3) |
| C1—C2—C10—O2 | 137.8 (2) | C2—C3—C4—C5 | 136.4 (2) |
| C3—C2—C10—O2 | −37.0 (3) | C9—C4—C5—C6 | 1.4 (4) |
| C1—C2—C10—O1 | −39.3 (3) | C3—C4—C5—C6 | 176.4 (2) |
| C3—C2—C10—O1 | 145.9 (2) | C4—C5—C6—C7 | 0.0 (4) |
| C1—C2—C3—C1$^{i}$ | −1.1 (3) | C5—C6—C7—C8 | −1.2 (4) |
| C10—C2—C3—C1$^{i}$ | 173.57 (18) | C6—C7—C8—C9 | 0.9 (4) |
| C1—C2—C3—C4 | 176.46 (19) | C5—C4—C9—C8 | −1.8 (3) |

| | | | |
|---|---|---|---|
| C10—C2—C3—C4 | −8.9 (3) | C3—C4—C9—C8 | −176.6 (2) |
| C1$^{i}$—C3—C4—C9 | 128.7 (2) | C7—C8—C9—C4 | 0.6 (4) |

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H10···O3$^{ii}$ | 0.926 | 1.659 | 2.581 (2) | 174 |

Symmetry code: (ii) *x*+1, *y*, *z*.