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(3*Z*,3′*Z*)-3,3′-(Ethane-1,2-diylidene)-bis[isobenzofuran-1(3*H*)-one]

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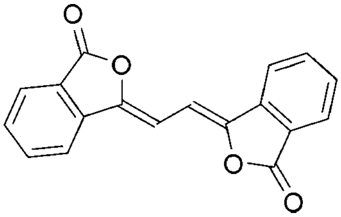
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.066; wR factor = 0.176; data-to-parameter ratio = 16.6.

The title compound, $C_{18}H_{10}O_4$, has been isolated as an impurity in commercially available 6,11-dihydroxy-5,12-naphthacenedione. The title compound exhibits yellow fluorescence in the solid state. The molecule has crystallographic inversion symmetry and is planar, with an r.m.s. deviation of 0.031 (1) Å. The crystal structure is stabilized by C—H⋯O hydrogen bonds and π–π stacking interactions between 3-methyleneisobenzofuran-1(3*H*)-one units [interplanar distance 3.43 (1) Å].

Related literature

For the crystallographic analysis and functionalization of 6,11-dihydroxy-5,12-naphthacenedione, see: Tomura *et al.* (2008); Ono *et al.* (2009). For the synthesis of the title compound, see: Ji *et al.* (2006).



Experimental

Crystal data

$C_{18}H_{10}O_4$
M_r = 290.26
Triclinic, $P\overline{1}$
a = 6.9030 (13) Å
b = 7.0374 (16) Å
c = 7.7792 (13) Å
α = 112.190 (5)°
β = 95.696 (2)°
γ = 107.239 (8)°
V = 324.53 (11) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 173 K
1.00 × 0.50 × 0.40 mm

Data collection

Rigaku/MSC Mercury CCD diffractometer
Absorption correction: none
2656 measured reflections
1661 independent reflections
1323 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.176
S = 0.97
1661 reflections
100 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max}$ = 0.35 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.28 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C3—H3⋯O2$^{\rm i}$	0.95	2.70	3.459 (2)	137
C5—H5⋯O1$^{\rm ii}$	0.95	2.66	3.406 (2)	136
C6—H6⋯O2$^{\rm iii}$	0.95	2.71	3.4700 (19)	138
C9—H9⋯O2$^{\rm iii}$	0.95	2.55	3.3209 (18)	139

Symmetry codes: (i) $-x+2, -y+2, -z+2$; (ii) $x, y, z+1$; (iii) $x, y-1, z$.

Data collection: *CrystalClear* (Rigaku/MSC, 2006); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work was supported by a Grant-in-Aid for Scientific Research (grant No. 20550037) from the Ministry of Education, Culture, Sports, Science and Technology, Japan. The authors thank the Instrument Center of the Institute for Molecular Science for the X-ray structure analysis.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5025).

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supplementary materials

Acta Cryst. (2009). E**65**, o2118 [**doi:10.1107/S1600536809030888**]

(3*Z*,3'*Z*)-3,3'-(Ethane-1,2-diylidene)bis[isobenzofuran-1(3*H*)-one]

K. Ono, O. Tokura and **M. Tomura**

Comment

Through the study of the crystallographic analysis and functionalities of 6,11-dihydroxy-5,12-naphthacenedione (Tomura *et al.*, 2008; Ono *et al.*, 2009), we found the existence of a yellow fluorescent compound as a by-product in the commercially available chemical reagent. After the separation of the by-product, the structure was determined to be the title compound, (I). Although the synthesis of (I) was already reported (Ji *et al.*, 2006), the characterization has not been performed. In this paper, we report the separation, spectral data, and molecular and crystal structures of (I).

The molecular structure of (I) is shown in Fig. 1. The molecule is centrosymmetric and planar with an r.m.s. deviation of 0.031 (1) Å. The distances of the C9—C9 ($-x + 1$, $-y$, $-z + 1$) and C8—C9 bonds are 1.433 (3) and 1.345 (2) Å, respectively, indicating a butadiene skeleton. The bond length of C1—O2 [1.201 (2) Å] is reasonable for carbonyl group. Further, the O1—C1 and O1—C8 bonds result in O—C single bonds with a bond distance of 1.394 (2) Å. These bond distances support the molecular structure of (I).

In the crystal structure, the molecules are linked to each other through intermolecular C—H···O interactions (Table 1). The molecular arrangements shown in Figs. 2 and 3 run stepwise along the *b* and *c* axes, respectively. Fig. 4 also exhibits a stepwise arrangement along the (021) direction with C—H···O contacts. Furthermore, the 3-methyleneisobenzofuran-1(3*H*)-one rings overlap each other to form a π-stacking structure with an interplanar distance of 3.43 Å, as shown in Fig. 5.

Experimental

The commercially available 6,11-dihydroxy-5,12-naphthacenedione (100 mg, Aldrich) was recrystallized from ethyl acetate (280 ml) to afford pure red crystals of 6,11-dihydroxy-5,12-naphthacenedione (70 mg). The filtrate containing the title compound, (I) was concentrated, and the residue (25 mg) was dissolved in toluene (100 ml). The solution was washed with 1 *M* NaOH solutions (100 ml) ten times over until the NaOH solution was colorless. The organic solution was dried over Na_2SO_4 and concentrated to provide a yellow solid of (I) (*ca* 9% yield). Yellow prisms of the title compound, suitable for X-ray analysis were grown from a dichloromethane solution.

Refinement

All H atoms were placed in geometrically calculated positions, with C—H = 0.95 (aromatic) Å and $U_{iso}(H) = 1.2U_{eq}(C)$ (aromatic), and refined using a riding model.

Figures

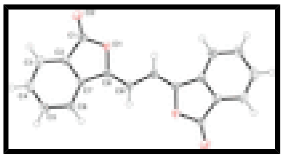
Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms and H atoms are shown as small spheres of arbitrary radii.

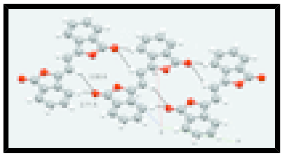
Fig. 2. Aggregation along the *b* axis with C—H···O contacts.

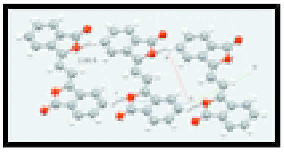
Fig. 3. Aggregation along the *c* axis with C—H···O contacts.

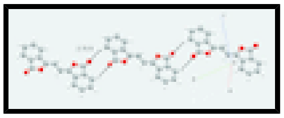
Fig. 4. Aggregation along the (021) direction with C—H···O contacts.

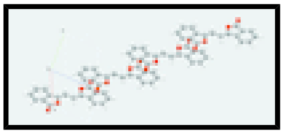
Fig. 5. π-Stacking structure with an interplanar distance of 3.43 Å.

(3*Z*,3'*Z*)-3,3'-(Ethane-1,2-diylidene)bis[isobenzofuran-1(3*H*)-one]

Crystal data

$C_{18}H_{10}O_4$

$M_r = 290.26$

Triclinic, $P\overline{1}$

Hall symbol: -P 1

$a = 6.9030\ (13)$ Å

$b = 7.0374\ (16)$ Å

$c = 7.7792\ (13)$ Å

$\alpha = 112.190\ (5)$°

$\beta = 95.696\ (2)$°

$\gamma = 107.239\ (8)$°

$V = 324.53\ (11)$ Å^3

$Z = 1$

$F_{000} = 150$

$D_x = 1.485$ Mg m^{-3}

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

Cell parameters from 1131 reflections

$\theta = 4.8$–30.8°

$\mu = 0.11$ mm^{-1}

$T = 173$ K

Prism, yellow

1.00 × 0.50 × 0.40 mm

Data collection

Rigaku/MSC Mercury CCD diffractometer

Radiation source: Rotating Anode

Monochromator: Graphite Monochromator

Detector resolution: 14.62 pixels mm^{-1}

1661 independent reflections

1323 reflections with $I > 2\sigma(I)$

$R_{int} = 0.044$

$\theta_{max} = 30.8$°

$T = 173$ K
φ and ω scans
Absorption correction: none
2656 measured reflections

$\theta_{\rm min} = 3.7°$
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 5$
$l = -7 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.176$
$S = 0.97$
1661 reflections
100 parameters
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
$w = 1/[\sigma^2(F_o^2) + (0.1211P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.35$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.28$ e Å^{-3}
Extinction correction: none

Special details

Experimental. IR (KBr, cm^{-1}): 1784, 1645, 1472, 1343, 1281, 1073, 974, 766, 689; ^{1}H NMR (CDCl$_3$, δ p.p.m.): 6.86 (s, 2H), 7.60 (t, J = 7.6 Hz, 2H), 7.77 (t, J = 7.6 Hz, 2H), 7.81 (d, J = 7.6 Hz, 2H), 7.96 (d, J = 7.6 Hz, 2H); MS (EI): m/*z* 290 (M^+), 104; UV-vis (CH$_2$Cl$_2$, nm): 406, 383.

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*	$U_{\rm iso}$*/$U_{\rm eq}$
C1	0.7382 (2)	0.6230 (2)	0.8120 (2)	0.0242 (4)
C2	0.7695 (2)	0.5873 (2)	0.9849 (2)	0.0216 (4)
C3	0.8463 (2)	0.7402 (2)	1.1749 (2)	0.0285 (4)
H3	0.8898	0.8939	1.2100	0.034*
C4	0.8568 (2)	0.6597 (3)	1.3105 (2)	0.0320 (4)
H4	0.9049	0.7593	1.4421	0.038*
C5	0.7974 (2)	0.4324 (3)	1.2568 (2)	0.0310 (4)
H5	0.8079	0.3814	1.3531	0.037*
C6	0.7236 (2)	0.2804 (2)	1.0665 (2)	0.0267 (4)
H6	0.6859	0.1271	1.0308	0.032*

C7	0.7070 (2)	0.3616 (2)	0.9299 (2)	0.0204 (3)
C8	0.6284 (2)	0.2534 (2)	0.7233 (2)	0.0207 (3)
C9	0.5404 (2)	0.0392 (2)	0.6005 (2)	0.0223 (4)
H9	0.5313	−0.0668	0.6501	0.027*
O1	0.65059 (16)	0.41594 (15)	0.65779 (15)	0.0248 (3)
O2	0.77382 (19)	0.78743 (17)	0.78771 (18)	0.0364 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0231 (7)	0.0198 (7)	0.0242 (8)	0.0061 (6)	0.0010 (6)	0.0061 (6)
C2	0.0187 (6)	0.0193 (7)	0.0207 (7)	0.0044 (5)	0.0025 (5)	0.0046 (5)
C3	0.0263 (8)	0.0231 (7)	0.0245 (8)	0.0056 (6)	0.0027 (6)	0.0016 (6)
C4	0.0296 (8)	0.0343 (9)	0.0180 (8)	0.0048 (7)	0.0024 (6)	0.0027 (6)
C5	0.0287 (8)	0.0389 (9)	0.0197 (8)	0.0053 (7)	0.0045 (6)	0.0125 (7)
C6	0.0252 (7)	0.0270 (7)	0.0234 (8)	0.0050 (6)	0.0038 (6)	0.0101 (6)
C7	0.0176 (7)	0.0207 (7)	0.0188 (7)	0.0050 (5)	0.0031 (5)	0.0059 (5)
C8	0.0209 (7)	0.0205 (7)	0.0185 (7)	0.0070 (5)	0.0017 (5)	0.0072 (5)
C9	0.0221 (7)	0.0197 (7)	0.0208 (8)	0.0058 (5)	0.0009 (5)	0.0066 (6)
O1	0.0294 (6)	0.0188 (5)	0.0205 (6)	0.0055 (4)	−0.0005 (4)	0.0063 (4)
O2	0.0436 (7)	0.0221 (6)	0.0382 (7)	0.0073 (5)	−0.0006 (5)	0.0136 (5)

Geometric parameters (Å, °)

C1—O2	1.2007 (18)	C5—C6	1.388 (2)
C1—O1	1.3935 (17)	C5—H5	0.9500
C1—C2	1.465 (2)	C6—C7	1.393 (2)
C2—C3	1.389 (2)	C6—H6	0.9500
C2—C7	1.3914 (18)	C7—C8	1.4543 (19)
C3—C4	1.378 (2)	C8—C9	1.3447 (19)
C3—H3	0.9500	C8—O1	1.3944 (17)
C4—C5	1.405 (2)	C9—C9^{i}	1.433 (3)
C4—H4	0.9500	C9—H9	0.9500
O2—C1—O1	120.77 (15)	C4—C5—H5	119.2
O2—C1—C2	132.22 (15)	C5—C6—C7	117.42 (14)
O1—C1—C2	107.01 (12)	C5—C6—H6	121.3
C3—C2—C7	122.43 (14)	C7—C6—H6	121.3
C3—C2—C1	129.48 (14)	C2—C7—C6	120.35 (14)
C7—C2—C1	108.08 (13)	C2—C7—C8	107.24 (13)
C4—C3—C2	117.22 (14)	C6—C7—C8	132.40 (13)
C4—C3—H3	121.4	C9—C8—O1	120.45 (13)
C2—C3—H3	121.4	C9—C8—C7	131.48 (13)
C3—C4—C5	120.95 (14)	O1—C8—C7	108.04 (11)
C3—C4—H4	119.5	C8—C9—C9^{i}	124.02 (16)
C5—C4—H4	119.5	C8—C9—H9	118.0
C6—C5—C4	121.59 (15)	C9^{i}—C9—H9	118.0
C6—C5—H5	119.2	C1—O1—C8	109.58 (12)
O2—C1—C2—C3	2.1 (3)	C5—C6—C7—C2	2.1 (2)

O1—C1—C2—C3	−177.95 (14)	C5—C6—C7—C8	−176.59 (13)
O2—C1—C2—C7	−177.93 (16)	C2—C7—C8—C9	−176.24 (14)
O1—C1—C2—C7	2.00 (15)	C6—C7—C8—C9	2.6 (3)
C7—C2—C3—C4	−0.7 (2)	C2—C7—C8—O1	1.63 (15)
C1—C2—C3—C4	179.22 (13)	C6—C7—C8—O1	−179.57 (13)
C2—C3—C4—C5	1.7 (2)	O1—C8—C9—C9^{i}	−1.6 (3)
C3—C4—C5—C6	−0.8 (2)	C7—C8—C9—C9^{i}	176.07 (16)
C4—C5—C6—C7	−1.1 (2)	O2—C1—O1—C8	178.96 (12)
C3—C2—C7—C6	−1.2 (2)	C2—C1—O1—C8	−0.98 (16)
C1—C2—C7—C6	178.83 (12)	C9—C8—O1—C1	177.79 (11)
C3—C2—C7—C8	177.76 (13)	C7—C8—O1—C1	−0.36 (16)
C1—C2—C7—C8	−2.20 (15)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2^{ii}	0.95	2.70	3.459 (2)	137
C5—H5···O1^{iii}	0.95	2.66	3.406 (2)	136
C6—H6···O2^{iv}	0.95	2.71	3.4700 (19)	138
C9—H9···O2^{iv}	0.95	2.55	3.3209 (18)	139

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

Fig. 1

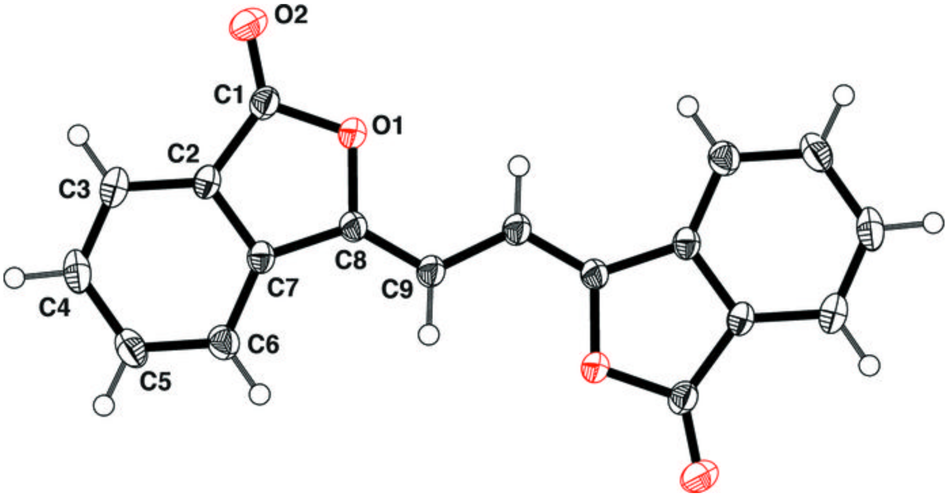

Fig. 2

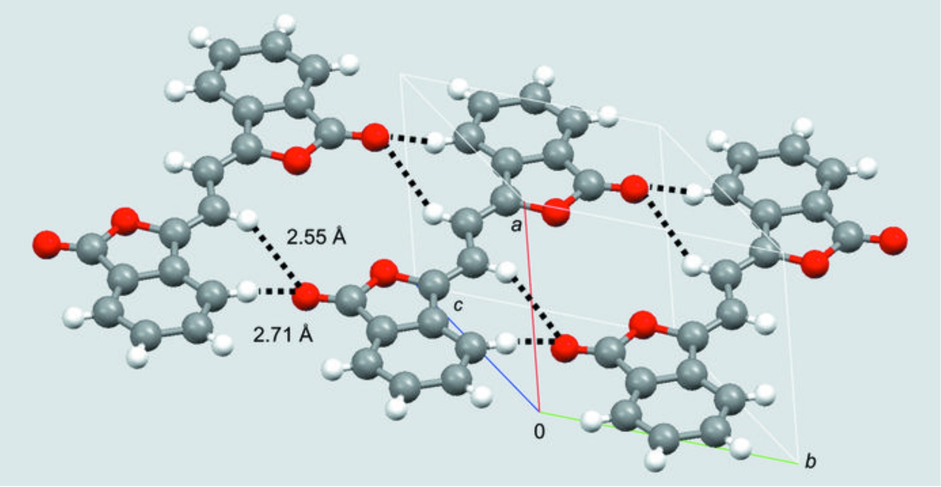

Fig. 3

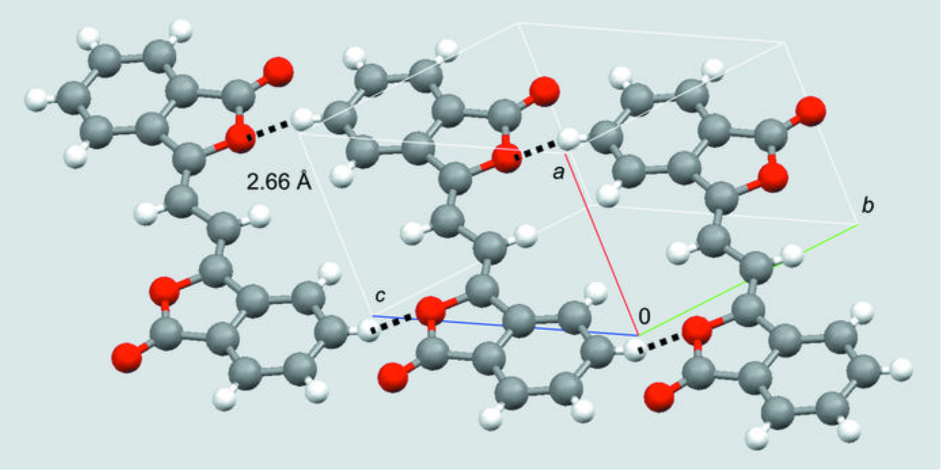

Fig. 4

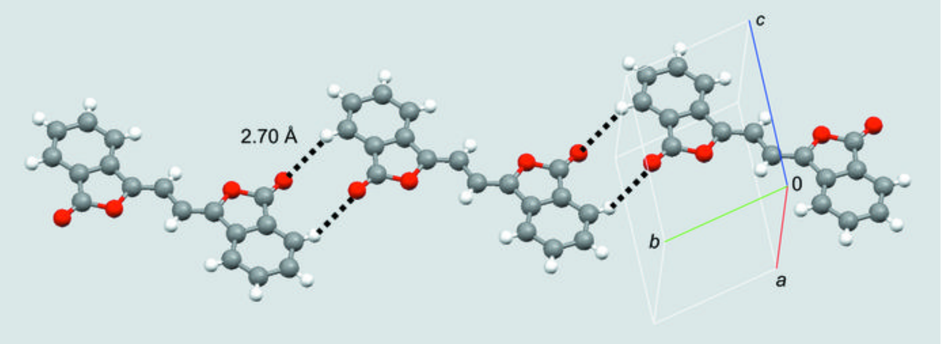

Fig. 5