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Diethyl 2,3-bis(benzo[1,3]dioxole-5-carbonyl)-but-2-enedioate

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The title compound, $C_{24}H_{20}O_{10}$, is centrosymmetric about the central C═C bond, indicating that the molecule adopts a *trans* configuration and that the aromatic rings are parallel to each other. The crystal structure is stabilized by intermolecular C—H···O interactions.

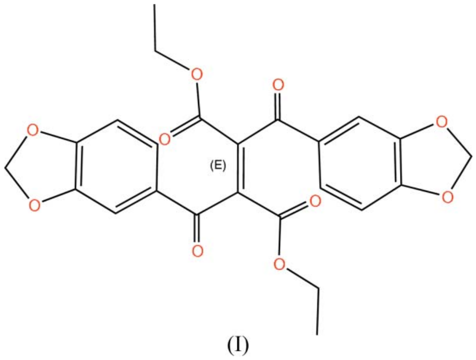
Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.054
wR factor = 0.147
Data-to-parameter ratio = 15.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Acceptor-substituted alkenes are important precursors (Helmut *et al.*, 1999) for the Diels–Alder reaction used to obtain some cyclic compounds (Adembri *et al.*, 1970; Celli *et al.*, 1982). In the present report, the crystal structure of a new alkene, the title compound, (I), is reported. The molecule of (I) is centrosymmetric (Fig. 1 and Table 1). The length of the central C9—C9^i bond [1.334 (3) Å] confirms its double-bond character [symmetry code: (i) $1 - x$, $1 - y$, $2 - z$]. The configuration of (I), from symmetry, is *trans* and the two aromatic rings are parallel to each other. The aromatic ring is effectively coplanar with the five-membered C1/O1/C7/C2/O2 ring, as seen in the dihedral angle between their planes of 0.6 (2)°. The crystal structure is stabilized by C—H···O intermolecular interactions as detailed in Table 2.



(I)

Experimental

Potassium *tert*-butoxide (0.56 g, 5 mmol) was added to a stirred solution of 3-benzo[1,3]dioxol-5-yl-3-oxo-propionic acid ethyl ester (0.24 g, 1 mmol) in anhydrous tetrahydrofuran (THF; 10 ml) under a nitrogen atmosphere. To this mixture, a solution of *N*-bromosuccinimide (0.71 g, 4 mmol) in anhydrous THF (10 ml) was added dropwise and the mixture was stirred overnight at room temperature. THF was removed and water was added, followed by extraction with dichloromethane. The solvent was evaporated to dryness and the compound was purified by column chromatography to yield (I) (0.20 g, 90%) as a green solid. Crystals appropriate for data collection were obtained by the slow evaporation of a methanol–chloroform solution of (I).

Crystal data

$C_{24}H_{20}O_{10}$
$M_r = 468.40$
Monoclinic, $P2_1/c$
$a = 11.1653\ (14)$ Å
$b = 13.3535\ (17)$ Å
$c = 7.738\ (1)$ Å
$\beta = 107.082\ (2)^\circ$
$V = 1102.8\ (2)$ Å^3
$Z = 2$

$D_x = 1.411$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2001 reflections
$\theta = 2.4$–25.6°
$\mu = 0.11$ mm^{-1}
$T = 292\ (2)$ K
Block, green
0.40 × 0.30 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.957$, $T_{\max} = 0.978$
6336 measured reflections

2399 independent reflections
1922 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 27.0^\circ$
$h = -14 \rightarrow 13$
$k = -17 \rightarrow 17$
$l = -9 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.147$
$S = 1.08$
2399 reflections
155 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0737P)^2 + 0.2005P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.24$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C8—C9	1.526 (2)	C9—C9^{i}	1.334 (3)
C9—C10	1.503 (2)		
C3—C4—C8	118.02 (16)	C9^{i}—C9—C8	124.4 (2)
C5—C4—C8	121.60 (16)	C9^{i}—C9—C10	119.6 (2)
C8—C9—C10	115.89 (14)		
O2—C2—C3—C4	179.01 (19)	O3—C8—C9—C10	−81.8 (2)
C5—C4—C8—C9	9.8 (2)	C4—C8—C9—C10	93.80 (18)
C3—C4—C8—C9	−172.13 (14)	C9^{i}—C9—C10—O4	1.2 (3)
O3—C8—C9—C9^{i}	94.5 (3)	C8—C9—C10—O4	177.64 (17)
C4—C8—C9—C9^{i}	−89.9 (3)	C9^{i}—C9—C10—O5	−178.9 (2)

Symmetry code: (i) $-x+1, -y+1, -z+2$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O3^{ii}	0.97	2.46	3.424 (3)	171
C6—H6···O4^{iii}	0.93	2.49	3.364 (2)	157
C11—H11B···O4^{iv}	0.97	2.47	3.430 (3)	170

Symmetry codes: (ii) $-x+2, y+\frac{1}{2}, -z+\frac{5}{2}$; (iii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (iv) $-x+1, -y+1, -z+1$.

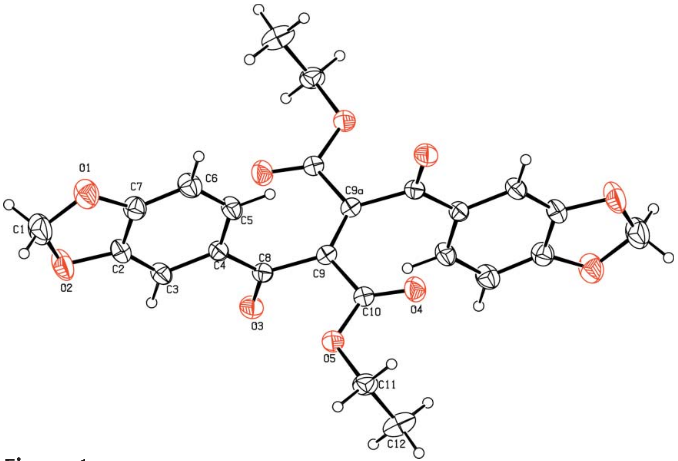


Figure 1
View of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Unlabelled atoms are related to labelled atoms by the symmetry operation $(1 - x, 1 - y, 2 - z)$.

The methyl H atoms were constrained to an ideal geometry, with C—H distances of 0.96 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$, but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93 Å for aromatic H atoms and 0.97 Å for methylene H atoms, and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2001); software used to prepare material for publication: *SHELXTL*.

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