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Diethyl *trans*-2,3-dibenzoyl-2-butenedioate

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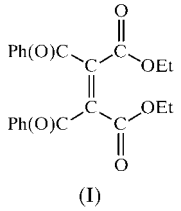
The title compound, $C_{22}H_{20}O_6$, has a central double bond of length 1.3694 (12) Å. Two benzene rings, lying on different sides of the diethyl 2-butenedioate main chain, are almost parallel to each other and nearly perpendicular to the main chain. In the unit cell, two symmetry-equivalent molecules are in general positions and two molecules lie on inversion centres.

Comment

The title compound, (I), is a member of the lignans, which are natural products. Because of its broad range of biological activities (Macrae & Towers, 1984), limited deposition in nature and complicated stereochemistry, much interest has been focused on it (Knorr, 1896; Pelter *et al.*, 1982; Wu *et al.*, 1997). Many consider it to be formed by dimerization of an ArC_3 fragment at the β,β-C atoms through a single bond and the β-C atoms are chiral (Wu *et al.* 1997). In order to deepen the understanding of the structure of the title compound at a molecular level and to provide some information on the synthesis of this class of organic compounds, we determined the crystal structure. We found that the title compound does



(I)

not have chiral C atoms; the two β-C atoms (C12 and C13) are joined by a double bond of length 1.3694 (12) Å and the sums of the bond angles at C12 and C13 are 359.92 (7) and 359.89 (7)°, respectively. Two benzene rings, lying on different sides of the diethyl 2-butenedioate main chain, are almost parallel to each other and nearly perpendicular to the main chain. In the unit cell, one molecule lies in a general position, with its inversion-related partner, and two molecules lie on inversion centres.

Experimental

The title compound was synthesized according to the literature method of Wu *et al.* (1997) and recrystallized from dry ethanol. Crystals suitable for X-ray determination were obtained by slow evaporation from a solution in formyl acetoacetate over a period of three weeks.

Crystal data

$C_{22}H_{20}O_6$
M_r = 380.38
Triclinic, $P\overline{1}$
a = 8.828 (6) Å
b = 15.174 (5) Å
c = 16.420 (7) Å
α = 113.52 (3)°
β = 99.27 (4)°
γ = 83.53 (4)°
V = 1987.6 (17) Å^3
Z = 4
D_x = 1.271 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10.86–12.77°
μ = 0.093 mm^{-1}
T = 293 (2) K
Block, colourless
0.45 × 0.37 × 0.35 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.941, T_{max} = 0.966
6828 measured reflections
6828 independent reflections
6310 reflections with $I > 2\sigma(I)$
θ_{max} = 24.98°
$h = -10 \rightarrow 10$
$k = -18 \rightarrow 16$
$l = 0 \rightarrow 19$
3 standard reflections
every 400 reflections
intensity decay: 0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.017
$wR(F^2)$ = 0.058
S = 0.927
6828 reflections
505 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0320P)^2 + 0.1400P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O11—C11	1.1912 (11)	O21—C21	1.2223 (11)
O12—C11	1.3082 (12)	O22—C21	1.3156 (10)
O12—C15	1.4625 (13)	O22—C23	1.4546 (13)
O13—C127	1.2207 (11)	O23—C217	1.2277 (11)
O14—C14	1.1990 (11)	O31—C31	1.2397 (12)
O15—C14	1.3270 (12)	O32—C31	1.2965 (11)
O15—C17	1.4551 (12)	O32—C33	1.4393 (12)
O16—C117	1.2219 (11)	O33—C317	1.2411 (11)
C11—O12—C15	119.79 (8)	C21—O22—C23	117.89 (7)
C14—O15—C17	116.35 (7)	O21—C21—O22	123.47 (8)
O11—C11—O12	122.68 (8)	O21—C21—C22	124.85 (8)
O11—C11—C12	125.80 (8)	O22—C21—C22	111.68 (7)
O12—C11—C12	111.48 (7)	O22—C23—C24	111.57 (8)
O14—C14—O15	122.78 (8)	O23—C217—C211	120.27 (8)
O14—C14—C13	124.45 (8)	O23—C217—C22	117.57 (7)
O15—C14—C13	112.77 (7)	C31—O32—C33	119.08 (7)
O12—C15—C16	104.34 (10)	O31—C31—O32	122.23 (8)
O15—C17—C18	107.17 (8)	O31—C31—C32	127.29 (8)
O16—C117—C111	120.74 (8)	O32—C31—C32	110.48 (8)
O16—C117—C12	116.22 (8)	O32—C33—C34	106.90 (8)
O13—C127—C121	120.40 (8)	O33—C317—C311	120.51 (8)
O13—C127—C13	116.54 (8)	O33—C317—C32	116.67 (8)

All H atoms were refined as riding (C—H 0.93–0.97 Å).

Data collection: *CAD*-4 *SDP*/*VAX* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *SDP*/*VAX* (Enraf–Nonius, 1989); data reduction: *TEXSAN* (Molecular Structure Corporation, 1989); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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