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Key indicators

Single-crystal X-ray study
 $T = 292$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
Disorder in main residue
 R factor = 0.066
 wR factor = 0.197
Data-to-parameter ratio = 13.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Tetraethyl 2,2'-(*p*-phenylenedimethylene)dimalonateThe molecule of the title compound, $\text{C}_{22}\text{H}_{30}\text{O}_8$, contains a
crystallographic inversion centre located at the middle of the
benzene ring.

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Comment

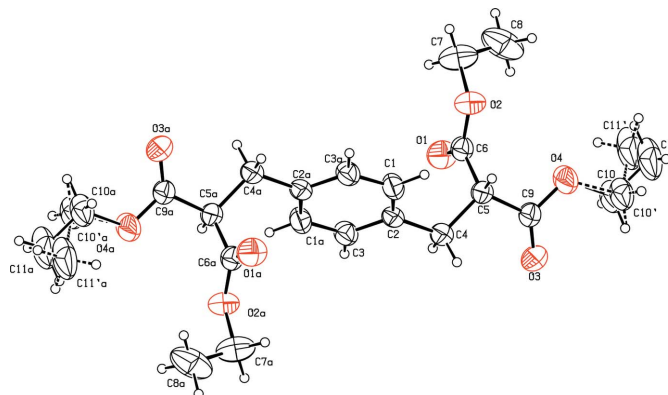
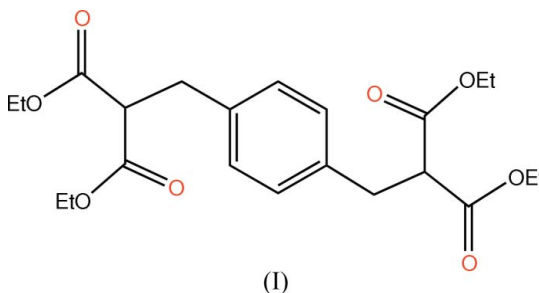
 β -Keto esters are multicoupling reagents with electrophilic
and nucleophilic sites that have proven to be valuable tools in
the synthesis of a wide variety of molecular systems (Benetti *et al.*, 1995). In the present paper, we report the crystal structure
of the title compound, (I). The molecule of (I) lies on a
crystallographic inversion centre located at the middle of the
benzene ring. Selected bond distances and angles are given in
Table 1. One pair of symmetrically related ethyl groups was
found to be disordered over two orientations (Fig. 1). The
molecular structure is stabilized by $\text{C}-\text{H} \cdots \pi$ intermolecular
interactions (Fig. 2); the $\text{C}11-\text{H}11\text{C} \cdots \pi$ distance is
 3.29 Å

Figure 1

View of the molecule of (I), showing the atom-labelling scheme.
Displacement ellipsoids for non-H atoms are drawn at the 50%
probability level. H atoms are represented by circles of arbitrary size.
Both disorder components are shown. [Symmetry code: (a) $1 - x, -y,$
 $1 - z$.]

Experimental

A mixture of 1,4-bis(bromomethyl)benzene (1.32 g, 5 mmol), ethyl malonate (1.53 ml, 10 mmol), potassium carbonate (1.38 g, 10 mmol) and acetone (25 ml) was stirred and refluxed for 8 h (Morenomanas *et al.*, 1993). The solvent was evaporated on a rotary evaporator and the resulting oil was chromatographed on a silica-gel column, yielding the title compound (1.37 g, 65%). Crystals appropriate for data collection were obtained by slow evaporation of an acetone solution at 283 K (m.p. 326–329 K). $^1\text{H NMR}$ (CDCl_3 , 400 MHz, p.p.m.): δ 7.26 (s, 4H), 4.15 (q, 2H, $J = 7.2$ Hz), 3.60 (t, 1H, $J = 7.6$ Hz), 3.17 (d, 2H, $J = 7.6$ Hz), 1.23 (t, 3H, $J = 7.2$ Hz). MS: 423 ($M+1$, 100), 348 (80.99), 274 (90.56), 262 (62.24), 189 (67.31).

Crystal data

$\text{C}_{22}\text{H}_{30}\text{O}_8$	$D_x = 1.213 \text{ Mg m}^{-3}$
$M_r = 422.46$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1926 reflections
$a = 9.461$ (2) Å	$\theta = 2.4\text{--}21.4^\circ$
$b = 8.3944$ (17) Å	$\mu = 0.09 \text{ mm}^{-1}$
$c = 14.601$ (3) Å	$T = 292$ (2) K
$\beta = 94.069$ (4)°	Block, colourless
$V = 1156.8$ (4) Å ³	$0.20 \times 0.20 \times 0.20 \text{ mm}$
$Z = 2$	

Data collection

Bruker SMART CCD area-detector diffractometer	1518 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.025$
Absorption correction: none	$\theta_{\text{max}} = 25.0^\circ$
5515 measured reflections	$h = -10 \rightarrow 11$
1991 independent reflections	$k = -9 \rightarrow 9$
	$l = -13 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1046P)^2 + 0.3206P]$
$R[F^2 > 2\sigma(F^2)] = 0.066$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.197$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.42 \text{ e } \text{Å}^{-3}$
1991 reflections	$\Delta\rho_{\text{min}} = -0.22 \text{ e } \text{Å}^{-3}$
146 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

C2–C4	1.509 (3)	C6–C5	1.514 (4)
C4–C5	1.526 (3)	C9–O3	1.189 (3)
C6–O1	1.190 (3)	C9–O4	1.304 (3)
C6–O2	1.325 (3)	C9–C5	1.519 (3)
C3–C2–C4	120.1 (2)	O3–C9–O4	123.9 (2)
C1–C2–C4	122.7 (2)	O3–C9–C5	125.1 (2)
C2–C4–C5	115.0 (2)	O4–C9–C5	111.1 (2)
O1–C6–O2	124.3 (3)	C6–C5–C9	108.56 (19)
O1–C6–C5	124.0 (2)	C6–C5–C4	111.5 (2)
O2–C6–C5	111.7 (2)	C9–C5–C4	110.0 (2)

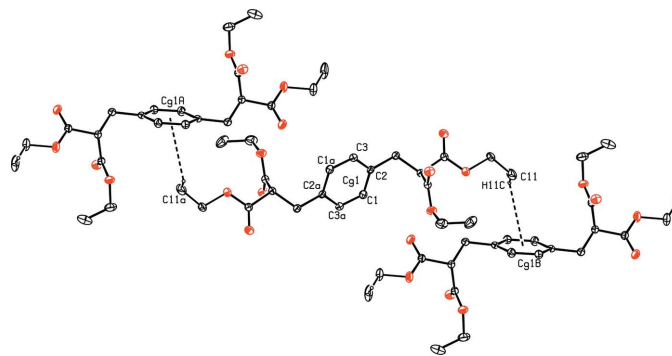


Figure 2

C–H... π intermolecular interactions (dashed lines) in the crystal structure. H atoms have been omitted.

One pair of symmetrically related ethyl groups was found to be disordered over two orientations. The occupancies of the disordered positions C10/C10' and C11/C11' were refined to 0.736 (18)/0.264 (18). All H atoms were refined as riding; C–H (methyl H) = 0.96 Å, C–H (methylene H) = 0.97 Å, C–H (methine H) = 0.98 Å and C–H (aromatic H) = 0.93 Å. The constraint $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$ was applied.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1999); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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References

- Benetti, S., Romagnoli, R., DeRisi, C., Spalluto, G. & Zanirato, V. (1995). *Chem. Rev.* **95**, 1065–1114.
- Bruker (1997). *SMART*. Version 5.054. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (1999). *SAINTE*. Version 6.01. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2001). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
- Morenomanas, M., Pleixats, R. & Riglans, A. (1993). *Synth. Commun.* **23**, 601–612.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.