Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-3-Hexene-1,6-diyl dibenzoate

J. Caleb Clark, Tanaji Talele, Mark L. McLaughlin and Frank R. Fronczek*

Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, USA

Correspondence e-mail: ffroncz@lsu.edu

Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.113
Data-to-parameter ratio = 22.8

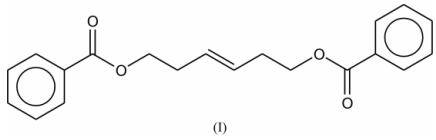
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title molecule, $C_{20}H_{20}O_4$, lies on an inversion center. The central alkene moiety is not coplanar with the remainder of the molecule, forming a dihedral angle of 61.61 (12)° with the O—CH_2—CH_2—C plane and a dihedral angle of 58.70 (11)° with the phenyl plane. The central C=C length is 1.326 (2) Å.

Received 2 June 2003
Accepted 3 June 2003
Online 17 June 2003

Comment

During an attempted cross metathesis reaction, the title compound, (I), was produced in an interesting side reaction. Such cross metathesis reactions, which tend to be unpredictable, occur between two olefins in the presence of an Ru catalyst known as Hoyveda's catalyst (Garber *et al.*, 2000). In the reaction, an olefin first reacts with the Ru catalyst, and then the highly reactive intermediate reacts with a different olefin. In this case, the second step was reaction of another molecule of the first olefin, but-3-enyl benzoate, to form the title dimer. The crystal structure determination was carried out in order to identify the unexpected product.



(I)

The molecule, which lies on a crystallographic inversion center, is illustrated in Fig. 1. The central 3-hexene moiety is nonplanar, with torsion angle C3—C2—C1—$C1^{i}$ of −121.21 (14)°, yielding the dihedral angles given in the abstract [symmetry code: (i) $1-x, 1-y, 1-z$].

The title compound has been previously reported as an intermediate in the synthesis of the corresponding 3,4-diol (Torneiro & Still, 1997).

Experimental

To 15 ml tetrahydrofuran were added but-3-enyl benzoate (0.10 g, 0.56 mmol, 1 eq.) and methyl vinyl ketone (0.85 g, 11.2 mmol, 20 eq.). Hoyveda's catalyst (0.018 g, 0.028 mmol, 0.05 eq) (Garber *et al.*, 2000) was added to the flask in 5 ml dichloromethane. The reaction mixture was stirred until the reaction was complete by TLC. The reaction mixture was purified by column chromatography, to obtain the title compound after removal of solvent.

Crystal data

$C_{20}H_{20}O_4$	$Z = 1$
$M_r = 324.36$	$D_x = 1.270$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 6.829$ (3) Å	Cell parameters from 2385 reflections
$b = 7.508$ (2) Å	$\theta = 2.5$–$30.0°$
$c = 9.460$ (4) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 67.424$ (17)°	$T = 100$ K
$\beta = 81.663$ (15)°	Plate, colorless
$\gamma = 71.322$ (16)°	0.42 × 0.37 × 0.07 mm
$V = 424.1$ (3) Å^3	

Data collection

KappaCCD diffractometer (with Oxford Cryostream)	$R_{int} = 0.021$
ω scans with κ offsets	$\theta_{max} = 30.0°$
9345 measured reflections	$h = -9 \rightarrow 9$
2480 independent reflections	$k = -10 \rightarrow 10$
1979 reflections with $I > 2\sigma(I)$	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 0.0772P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.042$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.113$	$\Delta\rho_{max} = 0.30$ e Å^{-3}
$S = 1.05$	$\Delta\rho_{min} = -0.20$ e Å^{-3}
2480 reflections	
109 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C4	1.3405 (13)	O2—C4	1.2104 (12)
O1—C3	1.4479 (13)	C1—C1^{i}	1.326 (2)
C4—O1—C3	115.89 (7)	C1^{i}—C1—C2	124.60 (12)
C1^{i}—C1—C2—C3	−121.21 (14)	C3—O1—C4—C5	−175.94 (7)
C4—O1—C3—C2	171.48 (8)	O1—C4—C5—C10	0.60 (13)
C1—C2—C3—O1	−167.53 (7)		

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

H atoms were placed in idealized positions, with C—H bond distances 0.95–0.99 Å, and thereafter treated as riding. Displacement parameters for H were assigned as $U_{iso} = 1.2U_{eq}$ of the attached atom.

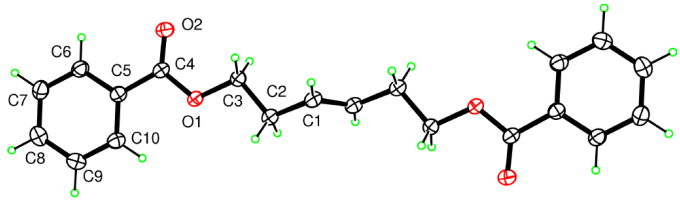


Figure 1
The title compound, showing the atomic numbering scheme, with displacement ellipsoids drawn at the 50% level.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* ; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

The purchase of the diffractometer was made possible by Grant No. LEQSF(1999–2000)-ESH-TR-13, administered by the Louisiana Board of Regents.

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Garber, S. B., Kingsbury, J. S., Gray, B. L. & Hoyveda, A. H. (2000). *J. Am. Chem. Soc.* **122**, 8168–8179.
Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. (1997). *SHELXL*97. University of Göttingen, Germany.
Torneiro, M. & Still, W. C. (1997). *Tetrahedron* **53**, 8739–8750.