Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Diethyl 4,7-dimethoxyindan-2,2-dicarboxylate

Peter Hammershøj* and Jørn B. Christensen

Department of Chemistry, University of Copenhagen, Universitetsparken 5, DK-2100 Copenhagen, Denmark

Correspondence e-mail: jbc@kiku.dk

Key indicators

Single-crystal X-ray study
T = 122 K
Mean σ(C–C) = 0.001 Å
R factor = 0.061
wR factor = 0.149
Data-to-parameter ratio = 48.6

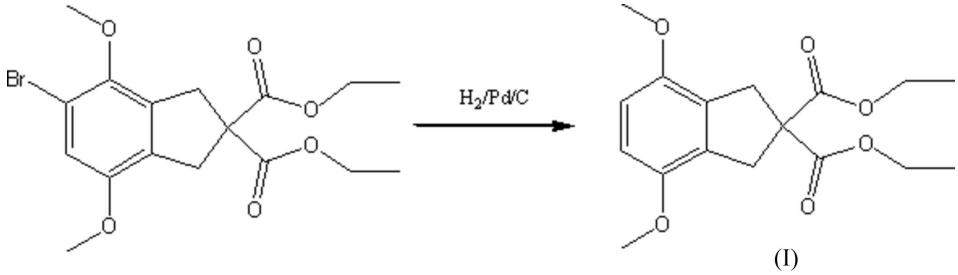
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{17}H_{22}O_6$, crystallizes with two independent molecules in the asymmetric unit. The bond lengths and angles are generally within the normal ranges. The crystal packing is stabilized by weak intermolecular C—H···O hydrogen bonds and weak C—H···π(arene) interactions.

Received 11 August 2005
Accepted 21 September 2005
Online 28 September 2005

Comment

The title compound, (I), was prepared for use as a building block in the syntheses of single-walled carbon nanotube (SWNT) interacting compounds. It crystallizes in the monoclinic space group $P2_1/c$ with two independent molecules in the asymmetric unit (Fig. 1). The corresponding bond lengths and angles of the two molecules agree with each other, but the molecules differ in the orientation of an ester group (Table 1). In both molecules, the methoxy groups are almost coplanar with the benzene ring and the five-membered rings of the indane ring system adopt envelope conformations. The crystal packing of (I) is stabilized mainly by weak intermolecular C—H···O hydrogen bonds and weak C—H ··· π(arene) interactions (Table 2 and Fig. 2). In Table 2, *Cg*1 and *Cg*2 denote the centroids of rings C1*A*—C6*A* and C1*B*—C6*B*, respectively.



(I)

Experimental

Diethyl 5-bromo-4,7-dimethoxyindan-2,2-dicarboxylate (10 g, 24.9 mmol), prepared as described by Hammershøj & Christensen (2005), was dissolved in methanol (300 ml) and Pd/C 10% (cat. amount) was added. The mixture was hydrogenated (5.8 p.s.i.) at room temperature for 20 h. The reaction mixture was filtered and evaporated to dryness *in vacuo*, yielding an off-white solid. Purification by crystallization from ethanol yielded compound (I) as a white powder (yield 63%, m.p. 362–364 K). Single crystals were obtained by slow evaporation from ethanol ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.7 (2H, *s*), 4.2 (4H, *q*, J = 7.3 Hz), 3.41 (4H, *s*), 1.25 (6H, *t*, J = 6.23 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.5, 149.7, 129.5, 109.2, 61.5, 59.7, 55.5, 37.9, 13.9; *m*/*e*: 322 (62), 248 (83.5), 175 (100) and 161

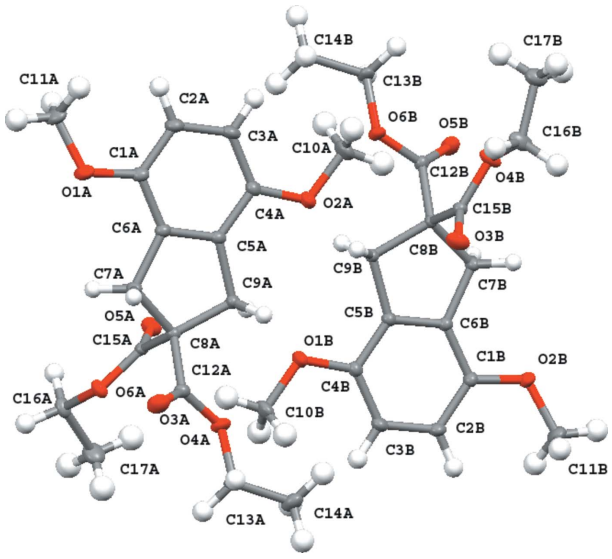


Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

(40%); analysis calculated for $C_{17}H_{22}O_6$: C 63.3, H 6.8%; found: C 63.1, H 6.8%.

Crystal data

$C_{17}H_{22}O_6$	D_x = 1.306 Mg m^{-3}
M_r = 322.35	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 37980 reflections
a = 16.569 (1) Å	θ = 1.2–40.0°
b = 8.033 (1) Å	μ = 0.10 mm^{-1}
c = 24.643 (3) Å	T = 122 (2) K
β = 90.232 (7)°	Prism, white
V = 3279.9 (6) Å^3	0.4 × 0.31 × 0.25 mm
Z = 8	

Data collection

Nonius KappaCCD area-detector diffractometer	20169 independent reflections
	12472 reflections with $I > 2\sigma(I)$
ω and φ scans	R_{int} = 0.079
Absorption correction: Gaussian integration (Coppens, 1970)	θ_{max} = 40.0°
	$h = -29 \rightarrow 29$
T_{min} = 0.977, T_{max} = 0.988	$k = -14 \rightarrow 14$
141916 measured reflections	$l = -43 \rightarrow 44$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0458P)^2 + 1.6487P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.061	
$wR(F^2)$ = 0.149	
S = 1.08	$(\Delta/\sigma)_{max}$ = 0.001
20169 reflections	$\Delta\rho_{max}$ = 0.65 e Å^{-3}
415 parameters	$\Delta\rho_{min}$ = −0.32 e Å^{-3}
H-atom parameters constrained	

Table 1
Selected torsion angles (°).

C11*A*—O1*A*—C1*A*—C2*A*	13.47 (14)
C10*A*—O2*A*—C4*A*—C3*A*	2.88 (15)
C15*A*—O6*A*—C16*A*—C17*A*	81.15 (11)
C10*B*—O1*B*—C4*B*—C3*B*	−1.98 (15)
C11*B*—O2*B*—C1*B*—C2*B*	−12.14 (14)
C15*B*—O4*B*—C16*B*—C17*B*	−163.66 (9)

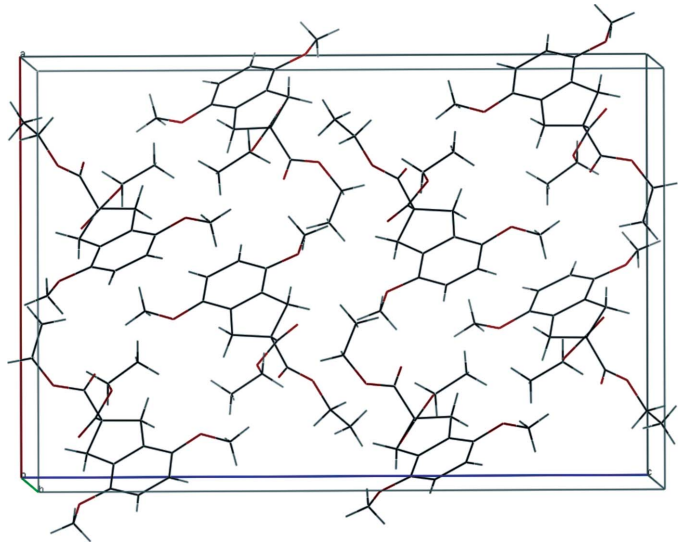


Figure 2
The crystal packing of (I).

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

D—H···A	D—H	H···A	D···A	D—H···A
C7*A*—H7*AB*···O3*A*	0.99	2.48	2.8639 (13)	102
C9*A*—H9*AB*···O5*A*	0.99	2.48	2.8501 (12)	102
C11*A*—H11*AC*···O3*B*^{i}	0.98	2.55	3.3757 (15)	142
C7*B*—H7*BA*···O5*B*	0.99	2.54	2.8778 (13)	100
C10*B*—H10*BA*···O5*A*	0.98	2.55	3.4685 (15)	156
C13*A*—H13*AB*···*Cg*1^{ii}	0.99	2.65	3.615 (1)	164
C13*B*—H13*BA*···*Cg*2^{iii}	0.99	2.67	3.633 (1)	165

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

H atoms were placed in idealized positions and allowed to ride on their parent atoms, with C—H = 0.95–0.99 Å and U_{iso}(H) = 1.2–1.5U_{eq}(C).

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DIRAX* (Duisenberg, 1992); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

The authors thank Flemming Hansen for collecting the diffraction data and the Centre for Crystallographic Studies for the use of their equipment.

References

Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Coppens, P. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 255–270. Copenhagen: Munksgaard.
Duisenberg, A. J. M. (1992). *J. Appl. Cryst.* **25**, 92–96.
Duisenberg, A. J. M., Kroon-Batenburg, L. M. J. & Schreurs, A. M. M. (2003). *J. Appl. Cryst.* **36**, 220–229.
Hammershøj, P. & Christensen, J. B. (2005). *Acta Cryst.* E**61**, o3278–o3280..
Nonius (1999). *COLLECT*. Nonius BV, Delft, The Netherlands.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.