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3-(3*H*-Inden-1-ylmethyl)-2,2′-bis(methoxymethoxy)-1,1′-binaphthalenyl

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.058
wR factor = 0.138
Data-to-parameter ratio = 15.2

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

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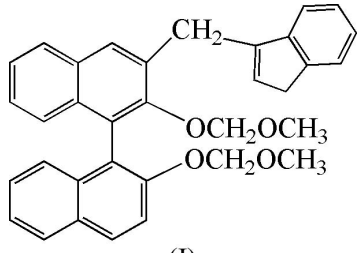
In the title compound, $C_{34}H_{30}O_4$, the dihedral angle between the planes of the indene group and the nearer naphthalene ring system is 84.88 (5)°. No significant hydrogen-bonding interactions are observed in the crystal structure.

Comment

A number of studies have been reported on the introduction of various kinds of substituents into the indenyl ligand in order to study the relationship between the structures of complexes and their catalytic activities (Bruce *et al.*, 1997; Hauptman *et al.*, 1995; Kravchenko *et al.*, 1997; Petoff *et al.*, 1997). 3-Substituted indenes are generally obtained by indenyllithium nucleophilic attack (Grimmer *et al.*, 2000). We report here the crystal structure of the title 1-substituted indene, (I).



(I)

In the molecule of (I) (Fig. 1), the dihedral angle between the two naphthalene ring systems is 70.78 (5)°. The C4—C3—C23—C24 torsion angle of 52.4 (3)° describes the orientation of the 3*H*-inden-1-ylmethyl group with respect to the C1–C10 naphthalene ring system. The dihedral angle between the planes of the indene group and the C1–C10 naphthalene ring system is 84.88 (5)°. The C3—C23—C24 bond angle of 112.9 (2)° is larger than the ideal tetrahedral value of 109.5°. The C3—C23 bond [1.520 (3) Å] is slightly longer than the C23—C24 bond [1.500 (3) Å].

Experimental

To an ice-cooled pink–red solution of indenyllithium (5 mmol) in tetrahydrofuran (THF; 20 ml) was added a solution of 2,2′-bis(methoxymethoxy)-1,1′-binaphthalen-3-ylmethyl methanesulfonate (2.41 g, 5 mmol) in THF, and the mixture was stirred overnight. The resultant red solution was quenched with saturated aqueous NH_4Cl (20 ml) and extracted with ethyl acetate (50 ml). The combined organic layers were washed with 1 *N* aqueous HCl (10 ml), saturated aqueous $NaHCO_3$ (10 ml) and brine (10 ml). The mixture was then dried over $MgSO_4$ and evaporated under reduced pressure, and the residue was purified by column chromatography [SiO_2, petroleum ether–ethyl acetate (10:1 *v*/*v*)] to give a colourless solid (yield 1.6 g,

3.19 mmol, 64%; m.p. 412–413 K). Analysis found: C 81.11, H 5.76%; calculated for $C_{34}H_{30}O_4$: C 81.25, H 6.02%. Block-shaped single crystals of (I) suitable for X-ray diffraction were obtained on evaporation of a solution in THF over several days at room temperature. Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 3054, 2960, 2897, 2825, 1625, 1506, 1466, 1397, 1358, 1241, 1201, 1152, 1086, 1014, 973, 924, 757; ^{1}H NMR (300 MHz, $CDCl_3$, δ, p.p.m.): 7.91, 7.88 (*d*, 1H), 7.81, 7.80 (*d*, 1H), 7.70 (*s*, 1H), 7.53, 7.50 (*d*, 1H), 7.43, 7.40 (*d*, 1H), 7.37, 7.34 (*d*, 1H), 7.32–7.09 (*m*, 8H), 6.20 (*s*, 1H), 5.07, 5.05, 4.99, 4.97 (*q*, 2H), 4.58, 4.56, 4.50, 4.47 (*q*, 2H), 4.15 (*s*, 2H), 3.35 (*s*, 2H), 3.09 (*s*, 3H), 2.81 (*s*, 3H), 2.09 (*s*, 1H).

Crystal data

$C_{34}H_{30}O_4$	D_x = 1.299 Mg m^{-3}
M_r = 502.58	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 882 reflections
a = 16.507 (6) Å	θ = 2.9–24.6°
b = 8.361 (3) Å	μ = 0.08 mm^{-1}
c = 18.907 (7) Å	T = 293 (2) K
β = 100.076 (6)°	Block, colourless
V = 2569.2 (15) Å^3	0.24 × 0.12 × 0.10 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3230 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.044
Absorption correction: none	θ_{max} = 26.4°
14 431 measured reflections	$h = -15 \rightarrow 20$
5260 independent reflections	$k = -8 \rightarrow 10$
	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2 + 0.7205P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.058	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.138	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
S = 1.09	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
5260 reflections	
345 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.389 (3)	O4—C33	1.379 (3)
O1—C21	1.432 (3)	O4—C34	1.417 (4)
O2—C12	1.375 (3)	C1—C11	1.494 (3)
O2—C33	1.414 (3)	C3—C23	1.520 (3)
O3—C21	1.367 (3)	C23—C24	1.500 (3)
O3—C22	1.420 (3)	C24—C25	1.335 (3)
C2—O1—C21	112.70 (17)	C24—C23—C3	112.9 (2)
C12—O2—C33	119.11 (18)	C25—C24—C28	108.5 (2)
C21—O3—C22	113.6 (2)	C25—C24—C23	128.2 (2)
C33—O4—C34	113.4 (2)		
C2—C1—C11—C12	69.4 (3)	C2—C1—C11—C16	−110.5 (2)
C6—C1—C11—C12	−106.3 (2)	C6—C1—C11—C16	73.8 (3)

All H atoms were placed in calculated positions, with C—H distances in the range 0.93–0.97 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2 or 1.5 (methyl) U_{eq}(C).

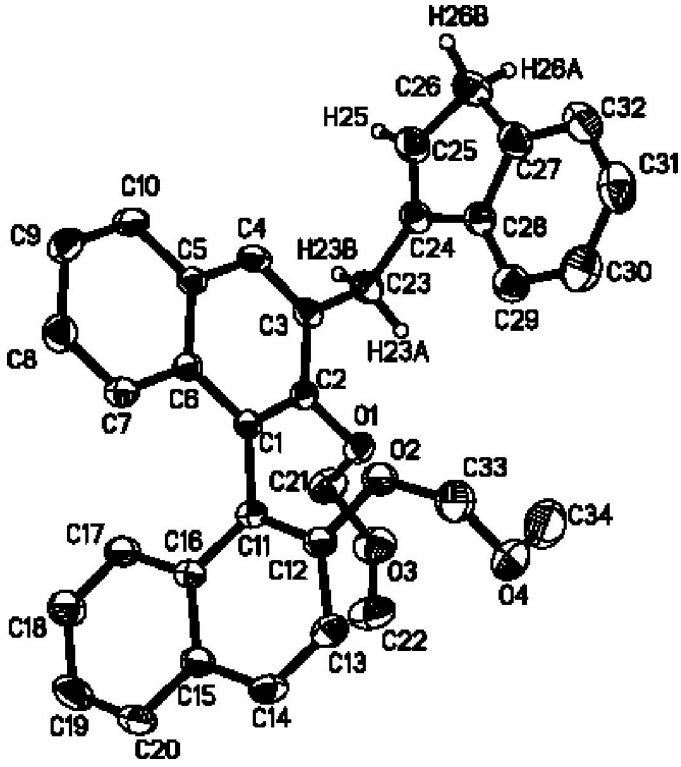


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. Most of the H atoms have been omitted for clarity.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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References

Bruce, M. D., Coates, G. W., Hauptman, E. & Waymouth, R. M. (1997). *J. Am. Chem. Soc.* **119**, 11174–11182.
Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1998). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Grimmer, N. E., Coville, N. J., Koning, C. B., Smith, J. M. & Cook, L. M. (2000). *J. Organomet. Chem.* **616**, 112–127.
Hauptman, E. & Waymouth, R. M. (1995). *J. Am. Chem. Soc.* **117**, 11586–11587.
Kravchenko, R., Masood, A. & Waymouth, R. M. (1997). *Organometallics*, **16**, 3635–3639.
Petoff, J. L. M., Bruce, M. D. & Waymouth, R. M. (1997). *Organometallics*, **16**, 5909–5916.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.