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2,3,1′,3′-Tetrahydro-[1,2′]biindenylidene

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.068
Data-to-parameter ratio = 17.7

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

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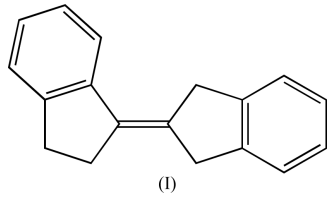
In the crystal structure of the title compound, $C_{18}H_{16}$, the dihedral angle between the least-squares planes through the C atoms of the phenyl rings is 2.82 (4)°. The bond length of the central double bond is 1.3297 (15) Å.

Comment

In the course of our investigations on the four possible biindenylidene isomers, we have already reported a second modification of (*E*)-2,3,2′,3′-tetrahydro-[1,1′]biindenylidene (Jovanovic *et al.*, 2001*a*) and the crystal structures of 1,3,1′,3′-tetrahydro-[2,2′]biindenylidene (Jovanovic *et al.*, 2001*b*) and (*Z*)-2,3,2′,3′-tetrahydro-[1,1′]biindenylidene (Jovanovic *et al.*, 2002*a*). We wished to determine the crystal structure of the fourth isomer, *i.e.* the title compound, (I), and tried to synthesize it, following the description of Bell & Spanswick (1966). The compound obtained was, however, spiro[1,1a,6,6a-tetrahydrocyclopropa[a]indene-1,1′-2′,3′-dihydro-1′H-indene] (Jovanovic *et al.*, 2002*b*). We have now succeeded in isolating (I) from a reaction mixture obtained by treating 1*H*-indene with H_2SO_4 (Jovanovic *et al.*, 2002) and in determining its crystal structure. The formula of the reaction product given in the literature by Bell & Spanswick (1966) is incorrect.



(I)

Experimental

The reaction of indene in the presence of H_2SO_4 was accomplished as described by Dansi & Pasini (1951). The title compound was isolated from the reaction mixture by HPLC on a preparative normal phase column (VP 250/10 Nucleosil 100–7) by isocratic LC elution using *n*-hexane. Recrystallization from propan-2-ol gave colourless crystals (m.p. 359 K).

Crystal data

$C_{18}H_{16}$
M_r = 232.31
Monoclinic, $C2/c$
a = 27.0655 (6) Å
b = 5.6822 (2) Å
c = 19.3829 (6) Å
β = 120.3936 (18)°
V = 2571.26 (13) Å^3
Z = 8
D_x = 1.200 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12358 reflections
θ = 3.1–27.5°
μ = 0.07 mm^{-1}
T = 291 (1) K
Needle, colourless
0.5 × 0.1 × 0.1 mm

Data collection

Nonius KappaCCD diffractometer	1262 reflections with $I > 2\sigma(I)$
310 frames *via* ω-rotation ($\Delta\omega = 1^\circ$) and two times 90 s per frame (three sets at different κ-angles)	$R_{int} = 0.017$ $\theta_{max} = 27.5^\circ$ $h = -34 \rightarrow 34$
Absorption correction: none	$k = -7 \rightarrow 7$
12358 measured reflections	$l = -24 \rightarrow 21$
2909 independent reflections	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0083P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.036$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.069$	$\Delta\rho_{max} = 0.14$ e Å^{-3}
$S = 0.98$	$\Delta\rho_{min} = -0.10$ e Å^{-3}
2909 reflections	Extinction correction: *SHELXL*97
164 parameters	Extinction coefficient: 0.00177 (17)
H-atom parameters constrained	

H atoms were placed in calculated positions, with U_{iso} constrained to be 1.2 times U_{eq} of the carrier atom.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97, *PARST*95 (Nardelli, 1995) and *PLATON* (Spek, 2001).

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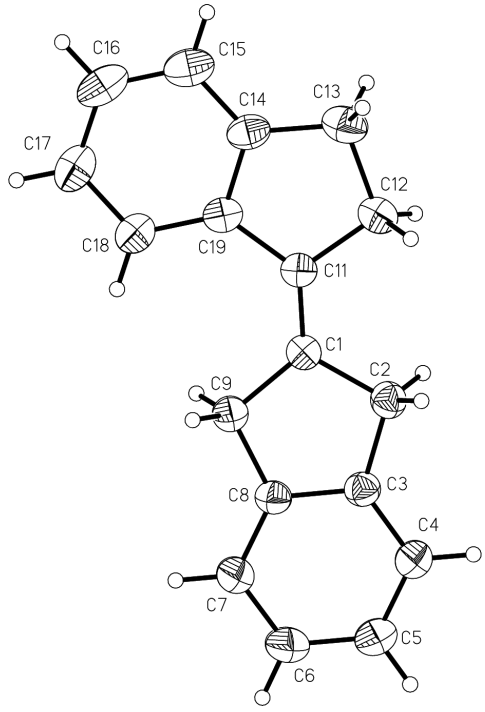

Figure 1
View of the title compound (*XP*; Sheldrick, 1991), showing the labelling of all non-H atoms. Displacement ellipsoids are shown at the 30% probability level. H atoms are drawn as circles of arbitrary radii.

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