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1,3-Diphenyl-1-*o*-tolyl-3,4-dimethylindan

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The title compound, $C_{30}H_{28}$, was prepared by tris-(2,4-dibromophenyl)aminium hexachloroantimonate-induced cyclodimerization of 1-*o*-tolyl-1-phenylethylene in 1,1,1,3,3,3-hexafluoropropan-2-ol. The X-ray analysis of the only product of the reaction confirms that it is the *cis* isomer.

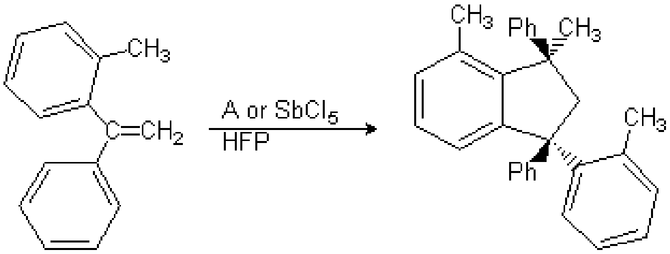
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.057
Data-to-parameter ratio = 8.8

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

Comment

The hole catalyst tris(2,4-dibromophenyl)aminium hexachloroantimonate [$(2,4\text{-}Br_2C_6H_3)_3NSbCl_6$, *A*; E^{red} = 1.66 *versus* SCE; Baker *et al.*, 1965] has been successfully employed for accomplishing novel, selective and highly efficient chemical transformations on a great variety of electron-rich substrates (Bauld, 1989; Nelsen, 1987; Lopez *et al.*, 1994; Caló *et al.*, 1995). Among them, we recently reported the facile aminium salt (*A*) induced cyclodimerization of several 1-aryl-1-phenylethylenes, leading, in dichloromethane solutions, to mixtures of two pairs of diastereoisomeric indan derivatives (Ciminale *et al.*, 1996). By contrast, a similar reaction, carried out in 1,1,1,3,3,3-hexafluoropropan-2-ol (HFP) solution of 1-*o*-tolyl-1-phenylethylene, afforded only the *cis* isomer of the title compound, (I) (Lopez *et al.*, unpublished results).



(I)

The crystal structure of (I) consists of discrete molecules of $C_{30}H_{28}$. The bond distances and angles are in agreement with accepted values. The atoms of the indan group, with the exception of C2, are almost coplanar, with an average deviation of 0.012 (3) Å. The conformation of the the five-membered ring of the indan group can be described as an envelope (Evans & Boeyens, 1989). The out-of-plane displacements from the least-squares plane are 0.121 (3), −0.148 (3), 0,120(3), −0.049 (3) and −0.044 (3) Å for C1, C2, C3, C8 and C9, respectively. C2, at the apex, deviates from the best plane of the other four atoms by 0.371 (2) Å; this compares with an average distance for the other atoms of 0.002 (3) Å. This plane makes an angle of 23.8 (1)° with the C1/C2/C3 plane.

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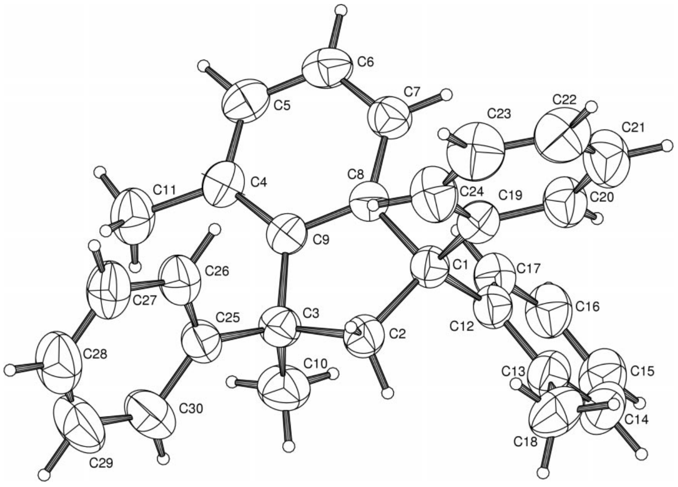

Figure 1
Drawing of the title compound with the atom-numbering scheme. Displacement ellipsoids are at the 50% probability level.

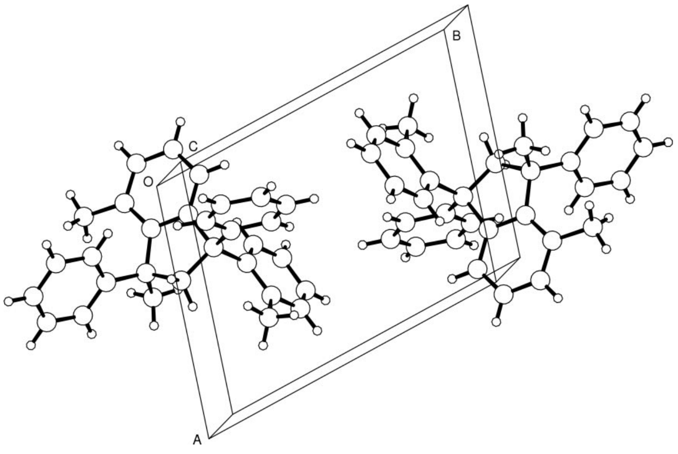

Figure 2
The molecular packing in the unit cell.

The phenyl rings are *cis* with respect to the indan group. The angle between their least-squares planes is 35.3 (1)°. The C3—C25 and C1—C19 bonds make angles of 44.5 (1) and 43.4 (1)°, respectively, with the best plane of the indan group, while bonds C3—C10 and C1—C19 form angles of 68.9 (2) and 66.8 (1)°. Furthermore, the distance between C19 and C25 is 4.717 (4) Å compared with a distance between C10 and C12 of 3.519 (4) Å, thus relieving steric strain between the two phenyl groups.

Experimental

A catalytic amount of tris(2,4-dibromophenyl)aminium hexachloroantimonate (*A*; 0.105 g, 0.10 mmol) was added rapidly to a stirred HFP (10 ml) solution of 1-*o*-tolyl-1-phenylethylene (0.194 g, 1 mmol) at room temperature. The reaction mixture, whose intensely green colour faded slowly, was monitored by TLC (petroleum ether/ethyl acetate, 20:1) and by GC–MS spectrometry, revealing the total disappearance of the starting material within 6 h, and the simultaneous formation of the main reaction product with molecular peak *m*/*z* = 388. The excess of aminium salt was destroyed by addition of 2 ml of ethyl ether, then the reaction mixture was poured into 10 ml of water and extracted with ethyl ether (10 ml × 2). The organic phase was separated, dried over sodium sulfate and the solvent removed *in vacuo*. Column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1) of the residue gave a pale-yellow crystalline product (0.175 g, 90%). Recrystallization from methanol furnished colourless crystals of pure *cis*-1,3-diphenyl-1-*o*-tolyl-3,4-dimethylindan: m.p. 448–449 K with elemental analysis consistent with $C_{30}H_{28}$; calculated: C 92.73, H 7.26%; found: C 92.87, H 7.16%; IR (KBr) 3055, 3020, 2967, 1492, 1444, 770, 758, 744, 731, 697, 637 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 7.36–6.63 (*m*, 17H), 3.53 (*d, J* = 13.8 Hz, 1H), 2.78 (*d, J* = 13.8 Hz, 1H), 1.91, (*s*, 3H), 1.53 (*s*, 3H), 1.42 (*s*, 3H) p.p.m.; ^{13}C NMR ($CDCl_3$): δ_C 150.7, 149.6, 149.4, 148.4, 147.9, 147.7, 136.6, 134.5, 132.2, 129.9, 129.0. 128.5, 128.2, 127.1, 126.4, 126.0, 125.7, 125.6, 125.0, 60.9, 60.8, 51.7, 24.4, 22.3, 18.9 p.p.m.; MS *m*/*z* (relative abundance): 388 (M$^+$, 20), 373 (11), 297 (100), 282 (19), 265 (13) p.p.m..

Crystal data

$C_{30}H_{28}$
M_r = 388.55
Triclinic, $P\overline{1}$
a = 8.577 (2) Å
b = 10.997 (3) Å
c = 12.327 (3) Å
α = 92.56 (2)°
β = 93.38 (2)°
γ = 106.67 (2)°
V = 1109.6 (5) Å^3

Z = 2
D_x = 1.163 Mg m^{-3}
Cu *K*α radiation
Cell parameters from 25 reflections
θ = 29.2–38.9°
μ = 0.49 mm^{-1}
T = 293 K
Prism, colourless
0.35 × 0.20 × 0.15 mm

Data collection

Rigaku AFC-5 four-circle diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (North *et al.*, 1968)
T_{min} = 0.931, T_{max} = 1.000
3828 measured reflections
3495 independent reflections
2397 reflections with $F > 6\sigma(F)$

R_{int} = 0.029
θ_{max} = 62.0°
$h = 0 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$
3 standard reflections every 97 reflections intensity decay: none

Refinement

Refinement on *F*
R = 0.045
wR = 0.057
S = 0.94
2397 reflections
271 parameters

H-atom parameters constrained
$w = 1/(5.1485 + 0.0617F + 0.0005F^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

The poor data/parameter ratio of 8.85 is a result of the weak diffracting power of the crystal. The positions of the H atoms were calculated geometrically at a distance of 0.96 Å from the corresponding C atom, and a riding model was used during their refinement. The orientation of the methyl H atoms was not refined.

Data collection: *MSC*/*AFC Diffractometer Control Software* (Molecular Structure Coorporation, 1988); cell refinement: *MSC*/*AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Coorporation, 1985); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *CAOS* (Camalli & Spagna, 1994); molecular graphics: *CAOS*; software used to prepare material for publication: *CAOS*.

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