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Tris(1*H*-inden-1-yl)phosphane

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In the structure of the title compound, $C_{27}H_{21}P$, at 89 (2) K the P atom is bound to the allylic carbon of the indenyl unit with P—C distances of *ca* 1.88 Å.

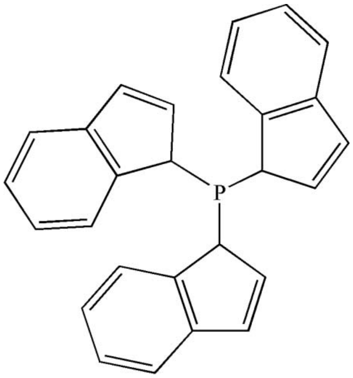
Key indicators

Single-crystal X-ray study
T = 89 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.105
Data-to-parameter ratio = 14.3

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

Comment

The title compound, (I), was synthesized as a precursor during our recent investigations on gallium phosphide synthesis. In (I), the P atom is bonded to the allylic sp^3 carbon position on the indenyl unit (Fig. 1). Selected bond lengths and angles are given in Table 1. The P—C distances are almost 0.1 Å longer in the title compound than those found in $C_{27}H_{21}PS$ [1.787 (5), 1.791 (5), 1.808 (5) Å; Adams *et al.*, 2003] due to the electron-withdrawing effects of the sulfur, and 0.05 Å longer that in triphenylphosphane (*ca* 1.83 Å; Dunne & Orpen, 1991) due to the steric requirements of the indenyl *versus* phenyl groups. The pseudo-C_3 symmetry of this phosphane differs from most aromatic phoshanes because one indenyl group (C19–C27) pivots on the *ipso*-carbon and is bent upwards towards the P atom relative to the other two indenyl groups, resulting in a bent propeller (see Fig. 1).



(I)

The ^{31}P NMR spectrum shows a resonance at +12 p.p.m. for the title compound. This, as well as ^{1}H and ^{13}C NMR spectra, differ from the previously reported spectra for tris-indenylphosphane (−58 p.p.m.; Adams *et al.*, 2003; Heuer *et al.*, 1989). It is clear from the ^{31}P NMR evidence that the title compound, upon heating (5 min at 343 K) or exposure to a Lewis acid (*e.g.* $AlCl_3$), undergoes isomerization and the resulting phosphane shift corresponds to the previously reported value cited above. This indicates that the phosphorus is now bonded to the vinylic carbon on the indenyl unit. This isomerization is irreversible. Suitable cystalline material for analysis has been unobtainable to date.

The compound readily oxidizes when exposed to air, as evidenced by a color change from tan to green.

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Experimental

$C_{27}H_{21}P$ was prepared according to the published procedure (Adams *et al.*, 2003) with some modifications. All manipulations were carried out under an atmosphere of argon. One equivalent of PCl_3 was added to a stirred solution of 3 equivalents of lithium indenyl in Et_2O (40 ml) at 195 K. The reaction mixture was allowed to warm to room temperature and stirred for 15 h. The solution was then filtered and evaporated under reduced pressure. The resulting tan solid was washed with methanol, then pentane. Colorless crystals of X-ray quality were obtained from a saturated dichloromethane solution layered with ethanol at 238 K.

Crystal data

$C_{27}H_{21}P$
$M_r = 376.41$
Monoclinic, $P2_1/n$
$a = 10.540$ (2) Å
$b = 16.371$ (2) Å
$c = 11.9778$ (17) Å
$\beta = 105.191$ (13)°
$V = 1994.5$ (5) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.15$ mm^{-1}
$T = 89$ (2) K
0.31 × 0.30 × 0.08 mm

Data collection

Bruker/Siemens SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{\min} = 0.955$, $T_{\max} = 0.986$
29580 measured reflections
3621 independent reflections
3118 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.039$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.105$
$S = 1.06$
3621 reflections
253 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—P1	1.8842 (18)	C19—P1	1.8797 (18)
C10—P1	1.8760 (18)		
C10—P1—C19	103.57 (8)	C19—P1—C1	99.98 (8)
C10—P1—C1	100.32 (8)		

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.98 Å and with $U_{\rm iso} = 1.2U_{\rm eq}$(C).

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *publCIF* (Westrip, 2006).

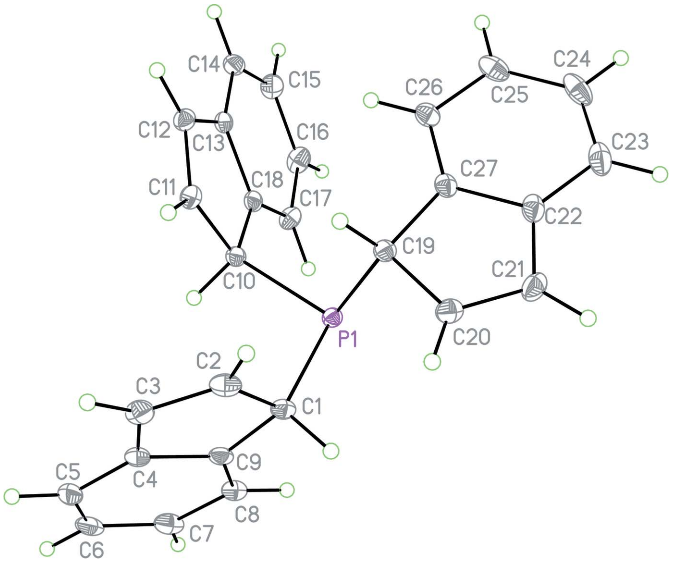

Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids. Hydrogen atoms are shown as spheres of arbitrary radius.

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References

Adams, J. J., Berry, D. E., Curnow, O. J., Fern, G. M., Hamilton, M. L., Kitto, H. J. & Pipal, R. J. (2003). *Aust. J. Chem.* **56**, 1153–1160.
Bruker. (2001). *SADABS.* Version 2.05. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2003). *SMART* (Version 5.630), *SAINT-Plus* (Version 6.45a) and *SHELXTL* (Version 6.12). Bruker AXS Inc., Madison, Wisconsin, USA.
Dunne, B. J. & Orpen, A. G. (1991). *Acta Cryst.* C**47**, 345–347.
Heuer, L., Bode, U. K., Jones, P. G. & Schmuttzler, R. (1989). *Z. Naturforsch. Teil B*, **44**, 1082–1092.
Westrip, S. P. (2006). *publCIF.* In preparation.