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Trimethylindium Based Phosphinoindanes. Synthesis and Molecular Structure of $[Me_2In{-}PPh_2]_3$: An Interesting In_3P_3 Trimer

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Summary: The trimeric phosphinoindane $[Me_2In{-}PPh_2]_3$ *was prepared from the room temperature reaction of trimethylindium with diphenylphosphine in toluene. The highly toluene soluble crystalline phosphinoindane was characterized by* ^1H *NMR,* ^{31}P *NMR, partial elemental analyses, and single crystal X-ray diffraction. The compound crystallizes in the monoclinic space group C2/c (No. 15) with unit cell parameters* $a = 43.130(5)$ Å, $b = 11.461(1)$ Å, $c = 18.887(2)$ Å, $\beta = 107.591(8)°$, $D_{calcd} = 1.48$ g cm^{-3}, *and* $V = 8899(1)$ Å^3, *for* $Z = 8$. *Refinement converged at* $R = 0.025$, $R_w = 0.031$. *The crystal structure reveals that the title compound resides about an* In_3P_3 *six-membered ring in a virtually perfect chair conformation, possessing approximate* C_3 *symmetry. The coordination about both indium and phosphorus may be described as distorted tetrahedral. The mean In–P bond distance is found to be 2.62(1) Å, while the corresponding mean In–C distance is 2.17(1) Å. The title compound represents the first higher-order phosphinoindane oligomer derived from trimethylindium, the simplest indium alkyl.*

Introduction

The organometallic chemistry of indium is perhaps the least developed of the group 13 (III) metals, as aluminum and gallium have traditionally garnered substantial attention. This observation is elegantly underscored by the fact that the first five- and six-coordinate organometallic compounds of indium were only very recently reported[1] while similar high coordination number complexes of aluminum[2,3] and gallium[4] have been known for some time. This disparity notwithstanding, organoindium chemistry is presently in the midst of a vigorous renaissance. The interaction of indium alkyls with phosphines, affording phosphinoindanes, is worthy of particular note. Activity in this regard is driven, arguably in large measure, by a seemingly insatiable quest toward discrete molecular precursors for indium phosphide. The concept of steric loading, the utilization of bulky ligands on both indium and phosphorus, is perhaps the most popular contemporary phosphinoindane preparative method. Typically, this technique has been shown to predominantly yield dimeric phosphinoindanes. Herein, we report the synthesis, characterization, and molecular structure of $[Me_2In{-}PPh_2]_3$ isolated from the room temperature reaction of the simplest indium alkyl, trimethylindium, with diphenylphosphine in toluene according to eq 1. The highly

$$Me_3In + HPPh_2 \xrightarrow[-MeH]{} {}^1/_3[Me_2In{-}PPh_2]_3 \qquad (1)$$

toluene soluble title compound is significant, as it clearly demonstrates that trimeric phosphinoindans may readily be approached from simple, sterically nondemanding indium analogs such as Me_3In. Furthermore, this study suggests that such products may be prepared *via* reasonably facile condensation reactions. The X-ray crystal structure of $[Me_2In{-}PPh_2]_3$ is given in Figure 1a, while Figure 1b provides an isolated view of the In_3P_3 core.

Experimental Section

General Comments. Standard Schlenk techniques were employed in conjunction with an inert atmosphere drybox (Vacuum Atmospheres HE-43 Dri-Lab). Solvents were distilled over sodium benzophenone under an atmosphere of argon prior to use. Trimethylindium was prepared by the method offered by Dennis et al.[5] Diphenylphosphine was purchased from Strem Chemical Co. and used as received. NMR data were recorded on a Bruker AC300 FT-NMR spectrometer. Single crystal X-ray diffraction studies were performed on a Nicolet R3mV diffractometer with graphite-monochromatic Mo Kα radiation ($\lambda = 0.710\,73$ Å). Calculations, structure solution, and refinement were performed using the SHELXTL[6] package of computer programs. Elemental Analyses were performed by E + R Microanalytical Laboratories (Corona, NY).

Synthesis of $[Me_2In{-}PPh_2]_3$. Inside the drybox a reaction vessel was charged with toluene (15 mL) and Me_3In (0.500 g, 3.126 mmol). Diphenylphosphine (0.540 mL, 3.103 mmol) was slowly added to the trimethylindium solution. Reaction was immediate although not vigorous. The system was removed from the drybox and placed in the freezer. After several hours a multitude of large, exceedingly well-formed rectangular crystals were afforded (0.937 g) in high yield (92%—based upon diphenylphosphine) (mp = 243 °C). Removal of solvent in vacuo facilitated the isolation of the compound. ^1H NMR (C_6D_6): δ 0.182 (d of t, 18H, In(CH_3)), 6.93–7.39 (mult, 30H, arom H). $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ −54.23 (s). Anal. Calcd (found) for $C_{42}H_{48}P_3In_3$: C, 50.94 (51.17); H, 4.89 (4.99).

Crystal Structure Determination of $[Me_2In{-}PPh_2]_3$. X-ray intensity data for a single crystalline sample of $[Me_2In{-}PPh_2]_3$, mounted in a thin-walled glass capillary under an atmosphere of argon, were collected on a Nicolet R3m/V diffractometer using an $\omega{-}2\theta$ scan technique with Mo Kα radiation ($\lambda = 0.710\,73$ Å)

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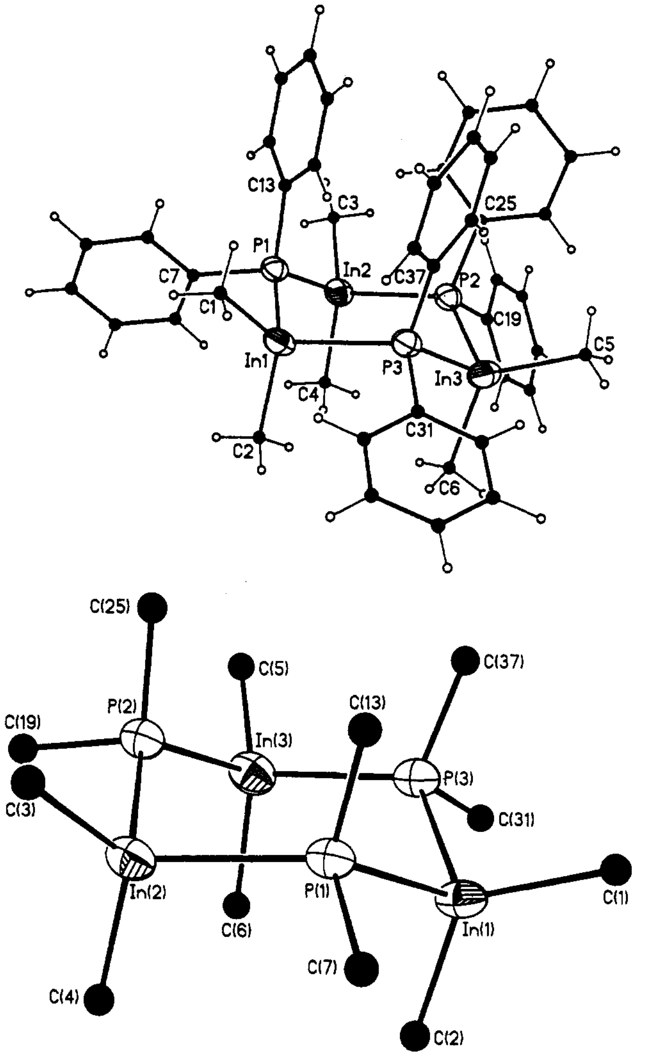

Figure 1. (a, top) Molecular structure of $[Me_2In-PPh_2]_3$. (b, bottom) In_3P_3 core ring of $[Me_2In-PPh_2]_3$.

at 21 °C. Cell parameters and an orientation matrix for data collection, obtained from a least-squares analysis of the setting angles of 50 carefully centered reflections in the range $27.43 < 2\theta < 36.61°$ corresponded to a monoclinic cell. The space group was determined to be *C*2/*c* (No. 15). Non-hydrogen atoms were refined using anisotropic thermal parameters. Hydrogen atoms were located by standard difference Fourier techniques and were included in the structure factor calculation at idealized positions ($d_{C-H} = 0.96$ Å). An isotropic group thermal parameter ($U_{iso} = 0.174(8)$ Å^2) was refined for all hydrogen atoms. The structure was solved by direct methods and refined, on the basis of 3744 observed reflections with intensities ($I > 3\sigma(I)$) in the range $3.50 < 2\theta < 45.0°$. Refinement converged at $R = 0.025$, $R_w = 0.031$. Table 1 provides summaries of crystallographic data, data collection, and structural refinement. Table 2 provides the atomic coordinates, while Table 3 provides selected bond distances and angles of the title compound.

Table 1. Crystallographic Data for $[Me_2In-PPh_2]_3$

Crystal Data	
empirical formula	$C_{42}H_{48}P_3In_3$
fw	990.17
color; habit	colorless; parallelepiped
size (mm)	0.20 × 0.22 × 0.31
space group	monoclinic, *C*2/*c* (No. 15)
unit cell dimens	
a (Å)	43.130(5)
b (Å)	11.461(1)
c (Å)	18.887(2)
β (deg)	107.591(8)
V (Å^3)	8899(1)
formula units/cell	8
D_{calc} (g/cm^3)	1.48
F(000)	3936
Data Collection	
unit cell reflns	50 ($27.43 \le 2\theta < 36.61°$)
2θ range (deg)	3.5 to 45.0
scan type	ω–2θ
scan speed (deg min^{-1})	variable, 2.09–14.65
no. of reflns collcd	6299
no. of unique reflns (R_{int})	5849 (0.024)
indices	(47,13,±21)
cryst decomp	linear (10%)
abs coeff (mm^{-1})	1.68
min/max transm	0.69/1.00
Refinement	
final residuals	$R = 0.0249$; $R_w = 0.0305$
goodness-of-fit	$S = 0.85$
largest Δ/σ	0.002
no. of reflns obs ($I > 3\sigma(I)$)	3744
params refined	435
data-to-param ratio	8.61:1
max/min diff peaks (e Å^{-3})	0.45/−0.28

Results and Discussion

Although the organoindium chemistry of phosphorus has not been as extensively developed as other group 13–15 (III–V) compounds such as aminoalanes,[7] the current emphasis on phosphinoindanes is impressive. The following dimeric phosphinoindanes, each residing about In_2P_2 four-membered rings, have been prepared from transformations typical to organoindium chemistry: $[Me_2In-P(SiMe_3)_2]_2$,[8] $[Et_2In-P(Et_2)]_2$,[9] $[i\text{-}Pr(i\text{-}Pr_2P)In-P(i\text{-}Pr_2)]_2$,[10] $[(Me_3SiCH_2)_2In-P(H)t\text{-}Bu]_2$,[11] $[(Me_3SiCH_2)_2In-PPh_2]_2$,[12] $[(Me_3SiCH_2)_2In-P(H)Ada]_2$ (Ada = adamantyl),[13] $[(Me_3SiCH_2)_2In-P(SiMe_3)_2]_2$,[14] $[Me(Me_3SiCH_2)In-P(SiMe_3)_2]_2$,[15] and $[(Me_3SiCH_2)_2In-P(t\text{-}Bu)_2]_2$.[16] Significantly, the trimethylindium based $[Me_2In-P(SiMe_3)_2]_2$ dimer has been shown to afford indium phosphide at 400 °C.[8] The literature reveals only two higher-order phosphinoindane oligomers relevant to this discussion: $[(Me_3CCH_2)_2In-PPh_2]_3$[17] and $[(i\text{-}Pr)In-P(SiPh_3)]_4$.[18] Thus, save for the In_3P_3 trimer and the In_4P_4 cubane, phosphinoindanes which possess molecular cores consisting exclusively of indium and phosphorus atoms are dimers. Furthermore, it is significant that these two higher-order

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Table 2. Atomic Coordinates (×10⁴) and Equivalent Isotropic Displacement Coefficients (Å² × 10³) for $[Me_2In-PPh_2]_3$

	x	*y*	*z*	U_{eq}[a]
In(1)	1104(1)	1084(1)	8581(1)	57(1)
In(2)	1915(1)	2991(1)	10314(1)	57(1)
In(3)	1091(1)	1170(1)	10957(1)	64(1)
P(1)	1513(1)	2800(1)	8966(1)	48(1)
P(2)	1502(1)	2912(1)	11077(1)	53(1)
P(3)	770(1)	1184(1)	9532(1)	54(1)
C(1)	791(2)	1472(6)	7469(4)	92(3)
C(2)	1396(2)	−468(5)	8867(5)	98(4)
C(3)	2135(2)	4720(6)	10441(4)	84(3)
C(4)	2175(2)	1355(6)	10623(4)	95(3)
C(5)	769(2)	1597(6)	11606(4)	92(3)
C(6)	1352(2)	−455(6)	11056(5)	112(4)
C(7)	1748(1)	2607(4)	8315(3)	50(2)
C(8)	2023(2)	1947(7)	8495(4)	102(4)
C(9)	2189(2)	1757(9)	7987(5)	131(5)
C(10)	2079(2)	2211(7)	7296(4)	90(4)
C(11)	1805(2)	2853(5)	7100(3)	72(3)
C(12)	1642(1)	3053(5)	7602(3)	65(3)
C(13)	1307(1)	4196(4)	8702(3)	47(2)
C(14)	973(1)	4293(5)	8564(3)	59(2)
C(15)	825(2)	5373(5)	8419(3)	71(3)
C(16)	1009(2)	6362(5)	8415(4)	75(3)
C(17)	1335(2)	6263(5)	8541(3)	69(3)
C(18)	1483(1)	5192(5)	8688(3)	57(2)
C(19)	1778(1)	2896(6)	12027(3)	61(2)
C(20)	1857(2)	1869(7)	12426(4)	89(3)
C(21)	2076(2)	1885(10)	13144(5)	116(5)
C(22)	2220(2)	2877(12)	13440(5)	114(5)
C(23)	2150(2)	3904(9)	13060(5)	93(4)
C(24)	1931(2)	3907(6)	12353(4)	76(3)
C(25)	1277(1)	4279(5)	11041(3)	50(2)
C(26)	1102(1)	4468(5)	11543(3)	59(2)
C(27)	915(1)	5447(6)	11486(4)	72(3)
C(28)	898(2)	6248(6)	10932(4)	84(3)
C(29)	1069(2)	6077(5)	10447(4)	76(3)
C(30)	1259(1)	5103(5)	10499(3)	62(2)
C(31)	528(1)	−167(5)	9370(4)	64(3)
C(32)	394(2)	−584(6)	9890(4)	94(4)
C(33)	212(2)	−1589(8)	9770(6)	126(5)
C(34)	165(2)	−2207(7)	9147(8)	136(7)
C(35)	300(2)	−1828(8)	8625(7)	139(6)
C(36)	479(2)	−815(6)	8744(5)	105(4)
C(37)	461(1)	2340(4)	9289(3)	49(2)
C(38)	502(1)	3332(5)	9724(3)	58(2)
C(39)	285(2)	4234(5)	9532(4)	70(3)
C(40)	22(2)	4180(6)	8918(4)	77(3)
C(41)	−18(2)	3196(6)	8487(4)	84(3)
C(42)	197(2)	2293(5)	8664(4)	72(3)

[a] Equivalent isotropic *U* defined as one-third of the trace of the orthogonalized U_{ij} tensor.

phosphinoindan oligomers were only obtained where exceedingly bulky substituents on indium (*i.e.*, Me_3CCH_2–) and phosphorus (*i.e.*, Ph_3Si–) were employed.

The In_3P_3 core of the title compound assumes a virtually perfect chair conformation possessing approximate C_3 symmetry. The endocyclic P–In–P bond angles range from 99.8(1) to 104.0(1)°, while the exocyclic C–In–C bond angles are reasonably distorted ranging from 123.6(3) to 125.7(2)°. A similar trend is observed about the phosphorus atoms where the mean C–P–C bond angle of 103.6(4)° compares to an endocyclic In–P–In bond angle range of 118.2(1)–122.2(1)°. The coordination about the In_3P_3 core atoms may be best described as distorted tetrahedral. The $In-P_{mean}$ and $In-C_{mean}$ bond distances are determined to be 2.62(1) and 2.17(1) Å, respectively. In preliminary investigations $[Me_2In-PPh_2]_3$, much like $[Me_2In-P(SiMe_3)_2]_2$,[19] has shown little reactivity toward Lewis bases such as amines, ethers, or other phosphines. Furthermore heating of the trimer did not afford a higher-order oligomer.

Table 3. Selected Bond Distances (Å) and Angles (deg) for $[Me_2In-PPh_2]_3$

	Distances		
In(1)–P(1)	2.593(1)	In(1)–P(3)	2.625(2)
In(1)–C(1)	2.172(6)	In(1)–C(2)	2.151(6)
In(2)–P(1)	2.622(1)	In(2)–P(2)	2.612(2)
In(2)–C(3)	2.178(7)	In(2)–C(4)	2.172(7)
In(3)–P(2)	2.633(2)	In(3)–P(3)	2.628(2)
In(3)–C(5)	2.168(9)	In(3)–C(6)	2.156(7)
P(1)–C(7)	1.828(7)	P(1)–C(13)	1.823(5)
P(2)–C(19)	1.830(5)	P(2)–C(25)	1.832(6)
P(3)–C(31)	1.841(6)	P(3)–C(37)	1.836(5)
	Angles		
P(1)–In(1)–P(3)	104.0(1)	P(1)–In(1)–C(1)	106.7(2)
P(3)–In(1)–C(1)	110.1(2)	P(1)–In(1)–C(2)	105.1(2)
P(3)–In(1)–C(2)	105.4(2)	C(1)–In(1)–C(2)	123.9(3)
P(1)–In(2)–P(2)	99.8(1)	P(1)–In(2)–C(3)	108.7(2)
P(2)–In(2)–C(3)	108.5(2)	P(1)–In(2)–C(4)	109.0(2)
P(2)–In(2)–C(4)	101.8(2)	C(3)–In(2)–C(4)	125.7(2)
P(2)–In(3)–P(3)	102.5(1)	P(2)–In(3)–C(5)	108.0(2)
P(3)–In(3)–C(5)	110.3(2)	P(2)–In(3)–C(6)	109.1(2)
P(3)–In(3)–C(6)	101.2(2)	C(5)–In(3)–C(6)	123.6(3)
In(1)–P(1)–In(2)	122.0(1)	In(1)–P(1)–C(7)	100.8(2)
In(2)–P(1)–C(7)	108.9(2)	In(1)–P(1)–C(13)	110.9(2)
In(2)–P(1)–C(13)	108.4(2)	C(7)–P(1)–C(13)	104.1(3)
In(2)–P(2)–In(3)	122.2(1)	In(2)–P(2)–C(19)	101.0(2)
In(3)–P(2)–C(19)	107.4(2)	In(2)–P(2)–C(25)	113.0(2)
In(3)–P(2)–C(25)	108.2(2)	C(19)–P(2)–C(25)	103.0(3)
In(1)–P(3)–In(3)	118.2(1)	In(1)–P(3)–C(31)	104.5(2)

Although the title compound and associated bond distances and angles are easily compared with the trimethylindium and diphenylphosphine based dimeric phosphinoindanes $[Me_2In-P(SiMe_3)_2]_2$[19] ($In-P_{mean}$ = 2.627(2) Å; $In-C_{mean}$ = 2.160(3) Å) and $[(Me_3SiCH_2)_2In-PPh_2]_2$[20] ($In-P_{mean}$ = 2.654(4) Å; $In-C_{mean}$ = 2.202(8) Å), respectively, perhaps the most intriguing comparison is found with the only other reported trimeric phosphinoindane, $[(Me_3CCH_2)_2In-PPh_2]_3$.[17] The neopentyl based phosphinoindane trimer $[(Me_3CCH_2)_2In-PPh_2]_3$, prepared from a metathesis reaction involving dineopentylindium chloride, $(Me_3CCH_2)_2InCl$, and potassium diphenyl phosphide, $KPPh_2$, was shown to exist as a monomer–dimer equilibrium mixture in benzene while a trimer is observed in the solid state. Furthermore, no other oligomers were observed in solution. The solution behavior of the title compound appears consistent with that observed in the solid state. The In–P distances in $[(Me_3CCH_2)_2In-PPh_2]_3$ were shown to be 2.677(1) and 2.699(2) Å, while the In–C distances were 2.182(6) and 2.210(7) Å. Spanning a range from 2.593(1) to 2.633(2) Å, the In–P bond distances in the title compound are noticeably shorter. A similar trend is also observed for the In–C bond distances. The neopentylindium based phosphinoindane trimer resides about a similar In_3P_3 chair conformation core. The ^{31}P NMR of $[Me_2In-PPh_2]_3$ is noteworthy as only one signal of δ at −54.23 ppm was observed. This compares to a value of −50.30 ppm for $[(Me_3SiCH_2)_2In-PPh_2]_2$. For a comparative basis, the δ chemical shift for the trigonal planar $In(P\text{-}t\text{-}Bu_2)_3$[9] is 70.80 ppm.

The preparation of a trimeric phosphinoindane based upon trimethylindium is noteworthy and suggests that higher-order oligomers need not, exclusively, be prepared using large sterically demanding ligands on indium but may also be reasonably approached from simple indium alkyls. While the mechanism affording the title compound remains unclear, it is interesting that reaction of Me_3In with $P(SiMe_3)_3$ (as noted earlier), clearly a very sterically

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demanding phosphine compared to $HPPh_2$, yields only a dimer. As an interesting aside, diphenylphosphine has also been employed in organoaluminum chemistry. In particular, reaction of $HPPh_2$ with diisobutylaluminum hydride in heptane has been shown in this laboratory to yield the crystalline dimer $[(i\text{-Bu})_2Al\text{–}PPh_2]_2$.[21] Particularly germane to this study is the convenient comparison of the title compound to the $[Me_2In\text{–}PPh_2]_2$ dimer, isolated from the room temperature reaction of Me_2InCl with $(Me_3Si)PPh_2$ in toluene.[22] The dimer was accompanied by Me_3SiCl elimination, while (as mentioned earlier) concomitant methane elimination is associated with the formation of the trimer. It is somewhat intriguing that the corresponding dimer was not isolated from the reaction which afforded the title trimeric phosphinoindane. This observation continues to be investigated in this laboratory. These findings, nonetheless, clearly suggest that sterics alone may not be the lone determinant in the association of phosphinoindanes but a number of factors, possibly including ring strain and reaction solvent, may also play important roles.

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Supplementary Material Available: Textual summary of data collection and refinement and tables of crystal data, bond distances and angles, atomic coordinates, and thermal parameters (8 pages). Ordering information is given on any current masthead page.

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