Synthesis, characterization, and molecular structure of a compound with a ring of Group 13, 15, and 16 atoms, [cyclic] (Me3CCH2)2InP(t-Bu)2In(CH2CMe3)2SePh

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Synthesis, Characterization, and Molecular Structure of a Compound with a Ring of Group 13, 15, and 16 Atoms, $(Me_3CCH_2)_2\overline{InP(t\text{-}Bu)_2In(CH_2CMe_3)_2Se}Ph$

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Summary: The compound $(Me_3CCH_2)_2\overline{InP(t\text{-}Bu)_2In(CH_2CMe_3)_2Se}Ph$ *has been prepared from equimolar quantities of* $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$ *and* $[(Me_3CCH_2)_2InSePh]_2$ *in pentane solution, recrystallized, and fully characterized. This mixed-bridge organoindium species crystallized in the centrosymmetric space group* $P\bar{1}$ *(No. 2) with* $a = 10.069(2)$ Å, $b = 11.627(4)$ Å, $c = 18.452(4)$ Å, $\alpha = 100.74(2)°$, $\beta = 99.33(2)°$, $\gamma = 103.38(2)°$, $V = 2016.9(8)$ Å^3, *and* $Z = 2$. *The structure was solved and refined to* $R = 2.51\%$ *and* $R_w = 2.52\%$ *for those 3747 unique data with* $|F_o| > 6\sigma(|F_o|)$ ($R = 4.51\%$ *for all 5304 data). The two indium atoms are bridged by both a di-tert-butylphosphido ligand and a phenylselenido ligand. The four-membered* In_2SeP *core has a puckered arrangement such that the fold angle about the In···In axis is 20.9°.*

Compounds of the type $[R_2MER'_2]_n$ (M = Al, Ga, In; E = N, P, As) typically have four- and six-membered cyclic structures.[1] If these rings are envisioned to involve a series of donor–acceptor bonds, it should be possible to replace a group 15 moiety (ER'_2) with a Lewis base moiety from another or replace a group 13 moiety (MR_2) with a different Lewis acid. Thus, a series of gallium and indium compounds which have two different bridging atoms, a phosphorus or arsenic and a chlorine or bromine, have been prepared and fully characterized. These compounds have the general formula $R_2ME(SiMe_3)_2MR_2X$ (M = Ga, E = As, R = Ph, X = Cl,[2] Br;[2] M = Ga, E = As, R = CH_2CMe_3,[3] CH_2SiMe_3,[4] CMe_3,[5] X = Cl; M = Ga, E = P, R = Ph, X = Cl;[6] M = In, E = P, R = CH_2CMe_3,[7] CH_2SiMe_3,[8] X = Cl; M = In, E = As, R = CH_2SiMe_3, X = Cl[9]). This interesting class of mixed-bridge compounds has now been extended to include a ring with group 13, 15 and 16 atoms, $(Me_3CCH_2)_2\overline{InP(t\text{-}Bu)_2In(CH_2CMe_3)_2Se}Ph$.

Table I. Selected Bond Lengths and Bond Angles for $(Me_3CCH_2)_2\overline{InP(t\text{-}Bu)_2In(CH_2CMe_3)_2Se}Ph$

	Bond Lengths (Å)		
In(1)–Se(1)	2.769(1)	In(1)–P(1)	2.653(2)
In(1)–C(111)	2.194(4)	In(1)–C(121)	2.194(5)
In(2)–Se(1)	2.748(1)	In(2)–P(1)	2.651(2)
In(2)–C(211)	2.181(6)	In(2)–C(221)	2.193(4)
Se(1)–C(1)	1.922(5)	P(1)–C(311)	1.895(5)
P(1)–C(321)	1.891(5)		
	Bond Angles (deg)		
Se(1)–In(1)–P(1)	84.1(1)	Se(1)–In(1)–C(111)	105.9(1)
P(1)–In(1)–C(111)	117.1(1)	Se(1)–In(1)–C(121)	109.4(1)
P(1)–In(1)–C(121)	114.0(1)	C(111)–In(1)–C(121)	119.5(2)
Se(1)–In(2)–P(1)	84.5(1)	Se(1)–In(2)–C(211)	106.7(2)
P(1)–In(2)–C(211)	108.9(2)	Se(1)–In(2)–C(221)	111.0(1)
P(1)–In(2)–C(221)	116.6(1)	C(211)–In(2)–C(221)	122.3(2)
In(1)–Se(1)–In(2)	91.6(1)	In(1)–Se(1)–C(1)	112.6(1)
In(2)–Se(1)–C(1)	101.3(2)	In(1)–P(1)–In(2)	96.4(1)
In(1)–P(1)–C(311)	114.8(1)	In(2)–P(1)–C(311)	112.4(2)
In(1)–P(1)–C(321)	110.1(2)	In(2)–P(1)–C(321)	110.0(2)
C(311)–P(1)–C(321)	112.2(2)	Se(1)–C(1)–C(2)	116.5(4)
Se(1)–C(1)–C(6)	123.7(4)	C(2)–C(1)–C(6)	119.8(5)
C(1)–C(2)–C(3)	119.9(5)	C(2)–C(3)–C(4)	120.5(7)
C(3)–C(4)–C(5)	121.1(8)	C(4)–C(5)–C(6)	119.1(6)
C(1)–C(6)–C(5)	119.7(5)	In(1)–C(111)–C(112)	120.0(3)
In(2)–C(211)–C(212)	124.9(3)	In(1)–C(121)–C(122)	120.7(4)
In(2)–C(221)–C(222)	123.5(3)		

The compound $(Me_3CCH_2)_2\overline{InP(t\text{-}Bu)_2In(CH_2CMe_3)_2Se}Ph$ was prepared from equimolar quantities of the two dimeric reactants $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$[10] and $[(Me_3CCH_2)_2InSePh]_2$[11] in pentane solution at room temperature:

$$[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2 + [(Me_3CCH_2)_2InSePh]_2 \rightarrow 2(Me_3CCH_2)_2\overline{InP(t\text{-}Bu)_2In(CH_2CMe_3)_2Se}Ph$$

The ease of formation of the new ring structure demonstrates the lability of the original four-membered indium–phosphorus and/or indium–selenium rings. The mixed-bridge product was recrystallized from pentane solution in approximately 75% yield and fully characterized in solution and in the solid state, including a single-crystal X-ray structural study.

The unit cell consists of two discrete molecules of $(Me_3CCH_2)_2\overline{InP(t\text{-}Bu)_2In(CH_2CMe_3)_2Se}Ph$ which are separated by normal van der Waals distances. There are no anomalously short intermolecular contacts. A list of interatomic distances and angles is included in Table I. A diagram of the molecule, showing the atomic labeling scheme, is shown in Figure 1.

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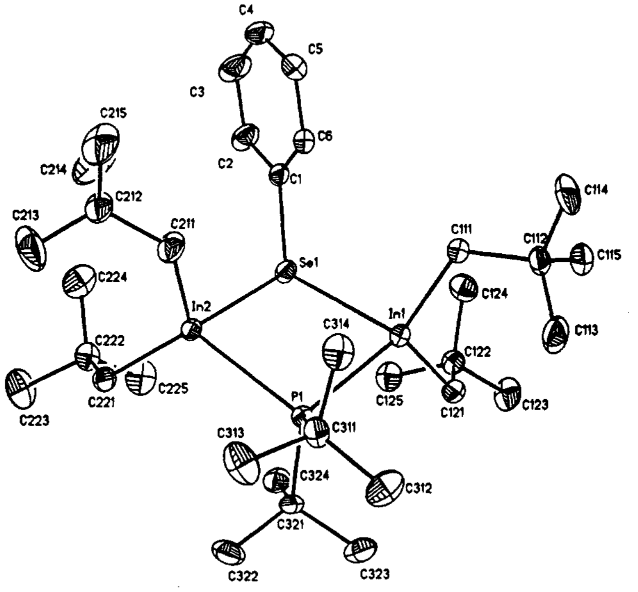

Figure 1. Labeling of atoms in the $(Me_3CCH_2)_2InP(t\text{-}Bu)_2In(CH_2CMe_3)_2SePh$ molecule (ORTEP II diagram, with all hydrogen atoms omitted).

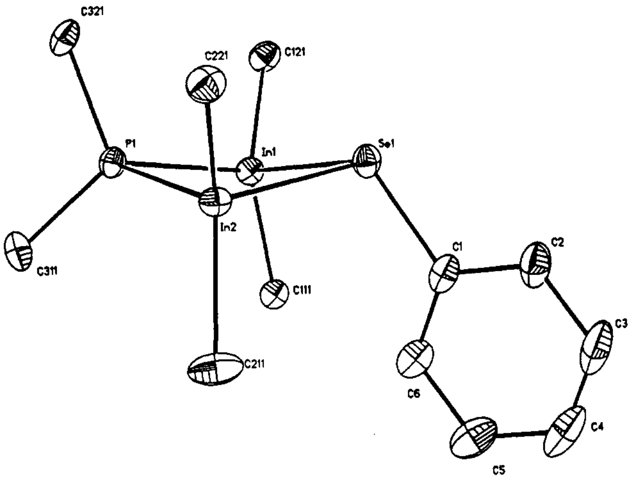

Figure 2. Diagram of the $(C)_2InP(C)_2In(C)_2Se(C)_6$ fragment of the molecule, showing the puckered core.

The four-membered In_2SeP core (Figure 2) has a puckered arrangement with the following In–Se and In–P distances: In(1)– Se(1) = 2.769(1) Å and In(2)– Se(1) = 2.748(1) Å (average 2.759 Å) and In(1)– P(1) = 2.653(2) Å and In(2)– P(1) = 2.651(2) Å (average 2.652 Å). These In–P distances are shorter then those associated with the symmetrically bridged compound $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$, in which the average In–P distance is 2.701 Å.[10] The In–Se distances are slightly elongated relative to those in the symmetrically bridged compound $[(Me_3CCH_2)_2InSePh]_2$, which has an average In–Se distance of 2.744 Å.[11] The fold angle about the In(1)···In(2) axis, as defined by the angle of intersection of the In(1)–Se(1)–In(2) and In(1)–P(1)–In(2) planes, is 20.9°. The fold angle about the Se(1)···P(1) axis, which is defined by the angle of intersection of the planes Se(1)–In(1)–P(1) and Se(1)–In(2)–P(1), is 19.2°. These fold angles are much smaller then the corresponding fold angles in $[(Me_3CCH_2)_2InSePh]_2$, in which that about the In···In axis is 30.8° and that about the Se···Se axis is 31.7°.[11] In comparison, $(Me_3CCH_2)_2InP(SiMe_3)_2In(CH_2CMe_3)_2Cl$ is planar,[7] whereas $(Me_3SiCH_2)_2InP(SiMe_3)_2In(CH_2SiMe_3)_2Cl$ has a puckered ring with a fold angle about the In···In axis of 33.3°. The nonbonding distances across the core in the present molecule are In(1)···In(2) = 3.954 Å and Se(1)···P(1) = 3.631 Å. These distances are intermediate between those for symmetrically bridged di-*tert*-butylphosphido and phenylselenide compounds. Thus, In···In distances vary in the order $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2 > (Me_3CCH_2)_2InP(t\text{-}Bu)_2In(CH_2CMe_3)_2SePh > [(Me_3CCH_2)_2InSePh]_2$. Both the present mixed-bridge compound and the symmetrically bridged phenylselenide compound crystallize with a puckered core. This conformation is adopted because of the low symmetry of the bridging ligands, which allows the ring to fold and reduce the total amount of steric interference between groups attached to the central ring.[11] The symmetrically bridged di-*tert*-butylphosphido derivative has symmetrical ligands bridging the two indium atoms and has a planar In_2P_2 core.

The bridging phenylselenide ligand is associated with a Se(1)–C(1) bond length of 1.922(5) Å and bond angles of In(1)–Se(1)–C(1) = 112.6(1)° and In(2)–Se(1)–C(1) = 101.3(2) Å. The Se–C distance is identical to the Se–C distances in $[(Me_3CCH_2)_2InSePh]_2$. The phenyl group is bent out of the In(1)–Se(1)–In(2) plane by 25.9°. The orientation of this ligand is determined by intermolecular packing rather then intramolecular forces.

The bridging di-*tert*-butylphosphido ligand is associated with the phosphorus–carbon distances P(1)–C(311) = 1.895(5) Å, P(1)–C(321) = 1.891(5) Å (average 1.893 Å); the interligand angle C(311)–P(1)–C(321) is 112.2(2)°. These P–C distances are very similar to those associated with $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$. The plane formed by C(311)–P(1)–C(321) intersects the In(1)–P(1)–In(2) plane at an angle of 89.2°. The equivalent angle in $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$ is 88.6°.

There are two neopentyl ligands associated with each indium atom. In–C distances are In(1)–C(111) = 2.194(4) Å, In(1)–C(121) = 2.194(5) Å, In(2)–C(211) = 2.181(6) Å and In(2)–C(221) = 2.193(4) Å (average = 2.191(5) Å). Interligand angles are C(111)–In(1)–C(121) = 119.5(2)° and C(211)–In(2)–C(221) = 122.3(2)° (average 120.9°). These angles are intermediate between those in $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$ (average interligand angle 115.2°) and $[(Me_3CCH_2)_2InSePh]_2$ (average interligand angle 133.6°).

The NMR spectroscopic data confirm the existence of a single, unique compound in benzene and toluene solutions rather than an equilibrium mixture of reactants and products. A comparison of the ^1H and ^{31}P NMR chemical shift data for $(Me_3CCH_2)_2InP(t\text{-}Bu)_2In(CH_2CMe_3)_2SePh$, $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$,[10] and $[(Me_3CCH_2)_2InSePh]_2$[11] is provided in Table II. It is significant that the resonance for the *tert*-butyl group protons bound to phosphorus in $(Me_3CCH_2)_2InP(t\text{-}Bu)_2In(CH_2CMe_3)_2SePh$ is a doublet at 1.36 ppm, whereas in $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$ the resonance for the tert-butyl group protons is a triplet at 1.46 ppm.[10] The trans coupling of the protons of the *tert*-butyl group to phosphorus in $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$ is not possible for the mixed-bridge compound with one phosphorus atom. The chemical shifts of the phosphorus atoms in the ^{31}P NMR spectra of the

Table II. Comparison of ^{1}H and ^{31}P NMR Data (ppm)

	$(Me_3CCH_2)_2InP(t\text{-}Bu)_2In(CH_2CMe_3)_2SePh$	$[(Me_3CCH_2)_2InSePh]$	$[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$
$InCCCH_3$	1.26 (s)	1.12 (s)	1.39 (s)
$PCCH_3$	1.36 (d)		1.46 (t)
$InCH_2$	1.45 (s)	1.47 (s)	1.54 (s)
SeC_6H_5	6.91 (tt)	6.94 (m)	
	6.98 (tm)		
	7.67 (dm)	7.44 (m)	
^{31}P	44.2		55.0

reactant and product differ by 11.0 ppm. The X-ray structural study identified the presence of unique neopentyl and *tert*-butyl groups due to the relative position of the phenyl group on selenium, but these were not observed in solution by NMR spectroscopy. The two neopentyl groups bound to indium are equivalent, as are the two *tert*-butyl groups on phosphorus at room temperature and at −80 °C in toluene. Either the substituents on indium and phosphorus are not differentiated by the phenyl group or, more likely, an exchange process makes the protons of each group equivalent. The most likely process would appear to be rapid inversion of the puckered four-membered ring, even at −80 °C. Alternatively, ring cleavage and rotation would be required.

The melting point and solubility properties of the bulk solid are consistent with the molecule identified by the X-ray structural study but not consistent with a mixture of the two starting compounds, $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$ and $[(Me_3CCH_2)_2InSePh]_2$. The melting point of the product (144–145 °C) is sharp and higher than the melting point of $[(Me_3CCH_2)_2InSePh]_2$ (103.8–105.5 °C)[11] but lower than the decomposition temperature of $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$ (200–202 °C).[10] The mixed-bridge compound is also significantly more soluble in pentane and benzene than either of the two compounds used to make it. The phosphide $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$ is almost insoluble in pentane and has insufficient solubility in benzene for a cryoscopic molecular weight study (0.01–0.10 *m*).[11]

Experimental Section

Compounds described in this investigation were extremely sensitive to oxygen and moisture and were handled in a standard vacuum line or under a purified argon atmosphere. The starting compounds $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$[10] and $[In(CH_2CMe_3)_2(SePh)]_2$[11] were prepared and purified by literature methods. Solvents were dried by conventional procedures. Elemental analyses were performed by E + R Microanalytical Laboratory, Inc., Corona, NY. Infrared spectra of Nujol mulls between CsI plates were recorded by means of a Perkin-Elmer 683 spectrometer. The ^{1}H NMR spectra were recorded at 400 MHz by means of a Varian VXR-400 S spectrometer. Proton chemical shifts were reported in δ units (ppm) and were referenced to C_6H_6 at δ 7.15 ppm. The $^{13}C\{^1H\}$ NMR spectra were recorded by means of a Varian Gemini 300 at 75 MHz. The ^{13}C spectra were reported relative to C_6D_6 at δ 128.0 ppm. The $^{31}P\{^1H\}$ and ^{31}P NMR spectra were recorded at 161.9 MHz by means of a Varian VXR-400 S spectrometer. The ^{31}P spectra were reported relative to H_3PO_4 at δ 0.0 ppm via an external standard. All samples for NMR spectra were contained in sealed NMR tubes. Melting points were observed in sealed capillaries under purified argon. Molecular weights were measured cryoscopically in benzene solution by using an instrument similar to that described by Shriver and Drezdzon.[12]

Synthesis of $(Me_3CCH_2)_2InP(t\text{-}Bu)_2In(CH_2CMe_3)_2SePh$. Stoichiometric amounts of $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$[10] (0.251 g, 0.311 mmol) and $[In(CH_2CMe_3)_2SePh]_2$[11] (0.258 g, 0.312 mmol) were combined in pentane (25 mL) at room temperature and stirred for 2 h. The product was filtered and then recrystallized from pentane to yield X-ray quality-crystals of $(Me_3CCH_2)_2InP(t\text{-}Bu)_2In(CH_2CMe_3)_3SePh$ (0.392 g, 0.481 mmol, 77% based on $[(Me_3CCH_2)_2InP(t\text{-}Bu)_2]_2$). Mp: 144–145 °C. Anal. Calcd for $C_{29}H_{56}In_2PSe$: C, 50.08; H, 8.28. Found: C, 50.08; H, 8.38. ^{1}H NMR (C_6D_6): δ 1.27 (s, 36 H, $InCCCH_3$), 1.35 (d, 18 H, $PCCH_3$, J = 14 Hz), 1.45 (s, 8H, $InCH_2$, $w_{1/2}$ = 6 Hz), 6.91 (tt, 1 H, *p*-H of Ph, ^3J = 7 Hz, ^4J = 2 Hz), 6.98 (tm, 2 H, *m*-H of Ph, ^3J = 7 Hz, ^4J = 2 Hz), 7.67 (dm, 2 H, *o*-H of Ph, ^3J = 7 Hz, ^4J = 1 Hz). ^{1}H NMR ($C_6D_5CD_3$): δ 1.26 (s, $InCCCH_3$), 1.36 (d, J = 13 Hz, $PCCH_3$), 1.45 (s, $InCH_2$), 6.90 (t, *p*-H of Ph, J = 8 Hz), 6.97 (t, *m*-H of Ph, J = 7 Hz), 7.62 (d, *o*-H of Ph, J = 8 Hz). ^{1}H NMR (−80 °C, $C_6D_5CD_3$): δ 1.25 (d, J = 13 Hz, $PCCH_3$), 1.29 (s, $InCCCH_3$), 1.46 (s, $InCH_2$), 6.75 (t, *p*-H of Ph), 6.85 (m, *m*-H of Ph), 7.64 (d, *o*-H of Ph, J = 7 Hz). $^{31}P\{^1H\}$ NMR (C_6D_6): δ 44.2 (s, $PCCH_3$). ^{31}P NMR (C_6D_6): δ 44.2 (m, $PCCH_3$, J = 13 Hz). $^{31}P\{^1H\}$ NMR ($C_6D_5CD_3$): δ 46.3 (s, $PCCH_3$). $^{31}P\{^1H\}$ NMR (−80 °C, $C_6D_5CD_3$): δ 45.4 (s, $PCCH_3$). $^{13}C\{^1H\}$ NMR (C_6D_6): δ 33.1 (s, $InCH_2\mathit{C}Me_3$), 33.4 (d, $PC\mathit{Me}_3$, J_{CP} = 5 Hz), 34.8 (s, $InCH_2C\mathit{Me}_3$), 35.2 (d, $P\mathit{C}Me_3$, J_{CP} = 7 Hz), 40.0 (d, $In\mathit{C}H_2CMe_3$, J_{CP} = 11 Hz), 126.2 (s, *p*-C of Ph), 128.9 (s, *m*-C of Ph), 135.5 (s, *o*-C of Ph). IR (Nujol mull): 1576 (w), 1358 (vs, sh), 1297 (vw), 1231 (m), 1212 (vw), 1170 (w), 1112 (m), 1093 (w), 1066 (w), 1017 (m), 1010 (m), 997 (m), 960 (vw), 926 (vw), 831 (vw), 804 (vw), 731 (vs), 688 (s), 661 (m), 569 (w), 462 (w), 446 (vw), 376 (vw) cm^{-1} Cryoscopic molecular weight, formula weight 815.48 (obsd molality, obsd mol wt, association): 0.0512, 814.6, 1.00; 0.0379, 811.1, 0.99; 0.0239, 852.7, 1.05.

Collection of X-ray Diffraction Data for $(Me_3CCH_2)_2InP(t\text{-}Bu)_2In(CH_2CMe_3)_2SePh$. A crystal of the mixed-bridge indium species was selected and sealed into a 0.3-mm thin-walled capillary under strictly anaerobic conditions. The capillary was then mounted and aligned on a Siemens R3m/V single crystal-diffractometer. Data collection was performed as described previously.[13] Details of the data collection appear in Table III.

The crystal belongs to the triclinic crystal system, the space group being $P\bar{1}$ (No. 2). This choice of space group was made on the basis of intensity statistics and was later confirmed by the successful solution of the crystal structure in the centrosymmetric space group $P\bar{1}$. All data were corrected for Lorentz and polarization effects. An empirical absorption correction was applied. A total of 10 608 reflections (a complete shell with 2θ = 5–45° for Mo Kα radiation) were collected and merged (R_{int} = 1.69%) into a unique set of 5304 reflections.

Solution and Refinement of the Structure. All crystallographic calculations were carried out with the aid of the Siemens SHELXTL PLUS program set.[14] The analytical scattering

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Table III. Experimental Data for the X-ray Diffraction Study of $(Me_3CCH_2)_2InP(t\text{-}Bu)_2In(CH_2CMe_3)_2SePh$

	Crystal Data
empirical formula	$C_{34}H_{67}In_2PSe$
cryst syst	triclinic
space group	$P\bar{1}$
unit cell dimens	$a = 10.069(2)$ Å $b = 11.627(4)$ Å $c = 18.452(4)$ Å $\alpha = 100.74(2)°$ $\beta = 99.33(2)°$ $\gamma = 103.38(2)°$
volume	2016.9(8) Å^3
Z	2
fw	815.4
density (calcd)	1.343 Mg/m^3
abs coeff	1.996 mm^{-1}
F(000)	836
	Data Collection
diffractometer used	Siemens R3m/V
radiation	Mo Kα (λ = 0.710 73 Å)
temp (K)	297
monochromator	highly oriented graphite cryst
2θ range	5.0–45.0°
scan type	2θ–θ
scan speed	constant; 2.00°/min in ω
scan range (ω)	0.50° plus Kα separation
bkgd measurement	stationary cryst and stationary counter at beginning and end of scan, each for 25.0% of total scan time
std rflns	3 measd every 97 rflns
index ranges	$-10 \le h \le 10, -12 \le k \le 12 -19 \le l \le 19$
no. of rflns collected	10 608
no. of indep rflns	5304 (R_{int} = 1.69%)
no. of obsd rflns	3747 ($F > 6.0(F)$)
abs cor	semiempirical
min/max transmissn	0.5447/0.7183
	Solution and Refinement
system used	Siemens SHELXTL PLUS (VMS)
soln	direct methods
refinement method	full-matrix least squares
quantity minimized	$\sum w(F_o - F_c)^2$
extinction cor	$\chi = 0.00014(4)$, where $F^* = F[1 + 0.002\chi F^2/(\sin 2\theta)]^{-1/4}$
H atoms	Riding model, fixed isotropic U
weighting scheme	$w^{-1} = \sigma^2(F) + 0.0002F^2$
no. of params refined	344
final R indices (all data)	R = 4.51%, R_w = 3.39%
R indices (6σ data)	R = 2.51%, R_w = 2.52%
goodness of fit	1.07
largest and mean Δ/σ	0.002, 0.000
data-to-param ratio	15.4:1
largest diff peak	0.94 e Å^{-3}
largest diff hole	–0.68 e Å^{-3}

factors for neutral atoms were corrected for both components ($\Delta f'$ and $i\Delta f''$) of anomalous dispersion.[15]

The structure was solved by the use of direct methods. Positional and anisotropic thermal parameters for all nonhydrogen atoms were refined. Hydrogen atoms were placed at calculated positions with d(C–H) = 0.96 Å[16] and the appropriate staggered- tetrahedral geometry. The isotropic thermal parameter of each hydrogen atom was fixed equal to the U_{eq} value for that carbon atom to which it was bonded.

Table IV. Final Atomic Parameters for $(Me_3CCH_2)_2InP(t\text{-}Bu)_2In(CH_2CMe_3)_2SePh$

	x	y	z	U(eq),[a] Å^2
In(1)	610(1)	2779(1)	2719(1)	41(1)
In(2)	3010(1)	5779(1)	2407(1)	38(1)
Se(1)	878(1)	5248(1)	3154(1)	45(1)
P(1)	3058(1)	3494(1)	2348(1)	39(1)
C(1)	–561(5)	5742(5)	2572(3)	50(2)
C(2)	–868(6)	6786(5)	2913(3)	70(3)
C(3)	–1835(8)	7235(7)	2515(5)	94(4)
C(4)	–2519(7)	6644(7)	1819(5)	92(4)
C(5)	–2235(6)	5596(6)	1460(4)	81(3)
C(6)	–1245(5)	5145(5)	1849(3)	58(2)
C(111)	–1239(5)	2048(4)	1791(3)	47(2)
C(112)	–1960(5)	676(4)	1567(3)	54(2)
C(113)	–928(6)	–61(5)	1431(4)	89(3)
C(114)	–3082(6)	397(5)	839(3)	86(3)
C(115)	–2680(6)	297(5)	2186(3)	67(3)
C(121)	719(5)	2011(5)	3719(3)	55(2)
C(122)	121(5)	2511(5)	4390(3)	55(2)
C(123)	–111(7)	1551(6)	4858(3)	90(3)
C(124)	–1283(5)	2762(6)	4119(3)	74(3)
C(125)	1133(6)	3687(5)	4887(3)	74(3)
C(211)	2057(6)	5922(5)	1289(3)	75(3)
C(212)	2292(7)	7032(6)	1015(3)	74(3)
C(213)	3653(8)	7774(9)	1129(5)	186(6)
C(214)	1591(12)	7851(8)	1538(5)	182(7)
C(215)	1430(8)	6863(7)	232(3)	123(4)
C(221)	4809(5)	7192(4)	3140(3)	51(2)
C(222)	4695(6)	8129(5)	3816(3)	59(2)
C(223)	6081(7)	9118(6)	4080(3)	105(3)
C(224)	3567(7)	8723(6)	3600(3)	93(3)
C(225)	4377(7)	7535(6)	4453(3)	89(3)
C(311)	3167(5)	2676(4)	1379(3)	54(2)
C(312)	3227(7)	1368(5)	1352(3)	89(3)
C(313)	4403(6)	3360(6)	1116(3)	91(3)
C(314)	1824(6)	2615(5)	837(3)	69(3)
C(321)	4517(5)	3437(5)	3116(3)	51(2)
C(322)	5959(5)	3966(6)	2976(3)	81(3)
C(323)	4410(6)	2143(5)	3232(3)	74(3)
C(324)	4346(5)	4203(5)	3832(3)	62(2)

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

Refinement converged with R = 2.51% and R_w = 2.52% for 344 parameters refined against those 3747 reflections with $|F_o| > 6\sigma(|F_o|)$, and R = 4.51% for all data. During refinement the structure was corrected for the effects of secondary extinction. This was done by refining the parameter χ in the expression $F^* = F[1 + 0.002\chi F^2/(\sin 2\theta)]^{-1/4}$; the final value for χ was $[1.4(4) \times 10^{-4}$. Following refinement, the extreme features left on the difference-Fourier map were a peak of 0.94 e Å^{-3} and a negative feature of –0.68 e Å^{-3}. Final atomic coordinates appear in Table IV.

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Supplementary Material Available: Complete listings of interatomic distances and angles, anisotropic thermal parameters, and calculated positions for all hydrogen atoms (5 pages). Ordering information is given on any current masthead page.

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