Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($Å^2 \times 10^3$)

	x	y	z	U_{eq}*
Ni	0	698 (1)	2500	32 (1)
F(1)	1756 (1)	1198 (5)	−17 (3)	84 (1)
F(2)	2045 (2)	−530 (5)	1465 (5)	112 (2)
F(3)	2406 (2)	1886 (6)	1701 (4)	118 (2)
F(4)	836 (3)	5504 (4)	4859 (5)	128 (2)
F(5)	827 (3)	3622 (6)	6315 (3)	129 (2)
F(6)	1679 (2)	4333 (5)	5544 (5)	108 (2)
†F(1A)	1805 (14)	121 (42)	448 (36)	49 (9)
†F(2A)	2271 (14)	260 (40)	2220 (31)	44 (8)
†F(3A)	2151 (21)	2296 (48)	1108 (41)	67 (11)
†F(4A)	590 (12)	4626 (36)	5672 (30)	37 (8)
†F(5A)	1404 (23)	3577 (50)	6257 (39)	71 (11)
†F(6A)	1349 (20)	5209 (47)	4842 (36)	58 (9)
O(1)	828 (1)	703 (2)	1630 (3)	38 (1)
O(2)	365 (1)	2351 (3)	3903 (2)	45 (1)
O(3)	−339 (1)	−1149 (3)	1215 (2)	46 (1)
C(1)	1901 (2)	1003 (5)	1351 (4)	51 (1)
C(2)	1326 (1)	1406 (4)	2117 (3)	37 (1)
C(3)	1408 (2)	2461 (4)	3224 (3)	45 (1)
C(4)	923 (2)	2866 (4)	4021 (3)	39 (1)
C(5)	1073 (2)	4094 (5)	5203 (5)	61 (2)

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

† Alternate positions for disordered F atoms.

metals. The intended product – a dimer containing bridging nitroxyl radicals – was not obtained, although it may have been present and subsequently cleaved by adventitious molecules of water during the process of crystallization. Evidence for dimeric transition-metal complexes containing bridging nitroxyl radicals has appeared, and further efforts are underway to establish the nature of the starting material (Caneschi, Gatteschi, Langier, Rey & Sessoli, 1988).

Table 2. *Bond lengths* (Å) *and angles* (°)

Ni—O(1)	2.026 (3)	Ni—O(2)	2.027 (2)
Ni—O(3)	2.054 (3)	O(1)—C(2)	1.254 (4)
O(2)—C(4)	1.250 (4)	C(1)—C(2)	1.530 (5)
C(2)—C(3)	1.382 (5)	C(3)—C(4)	1.391 (5)
C(4)—C(5)	1.541 (5)		

O(1)—Ni—O(2)	89.6 (1)	O(1)—Ni—O(3)	90.6 (1)
O(2)—Ni—O(3)	174.4 (1)	O(1)—Ni—O(1A)	179.8 (2)
O(2)—Ni—O(1A)	90.2 (1)	O(3)—Ni—O(1A)	89.6 (1)
O(2)—Ni—O(2A)	95.5 (1)	O(3)—Ni—O(2A)	90.2 (1)
O(3)—Ni—O(3A)	84.2 (1)	Ni—O(1)—C(2)	125.0 (2)
Ni—O(2)—C(4)	125.6 (2)	O(1)—C(2)—C(1)	113.4 (3)
O(1)—C(2)—C(3)	128.3 (3)	C(1)—C(2)—C(3)	118.4 (3)
C(2)—C(3)—C(4)	123.1 (3)	O(2)—C(4)—C(3)	127.8 (3)
O(2)—C(4)—C(5)	114.3 (3)	C(3)—C(4)—C(5)	117.9 (3)

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Structures of Adducts formed by Indium(III) Halides and Phosphine Ligands: Tris[1,2-bis(diphenylphosphino)ethane]bis[triiodoindium(III)] and Trichlorobis(trimethylphosphine)indium(III)

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Abstract. {μ-[1,2-Bis(diphenylphosphino)ethane]-1κP:2$\kappa P'$}-bis[1,2-bis(diphenylphosphino)ethane]-1κP,2κP-bis[triiodoindium(III)], $[(InI_3)_2(C_{26}H_{24}P_2)_3]$, (1), $M_r = 2186.4$, triclinic, $P\bar{1}$, $a = 12.583$ (7), $b = 12.667$ (8), $c = 13.080$ (8) Å, $\alpha = 76.59$ (4), $\beta = 86.72$ (4), $\gamma = 79.34$ (4)°, $V = 1993$ (2) Å^3, $Z = 1$, $D_x = 1.82$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 30.3$ cm^{-1}, $F(000) = 1046$, $T = 290$ K, $R = 0.066$ for 5203 unique observed $[I/\sigma(I) \geq 2.0]$ reflections. $[InCl_3(C_3H_9P)_2]$, (2), $M_r = 373.3$, orthorhombic, *Pnma*, $a = 10.418$ (8), $b = 10.769$ (9), $c = 13.808$ (9) Å, $V = 1549$ (2) Å^3, $Z = 4$, $D_x =$

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1.60 g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu =$ 21.9 cm^{-1}, $F(000) = 736$, $T = 290$ K, $R = 0.043$ for 1247 unique observed $[I/\sigma(I) \geq 2.0]$ reflections. The In atoms in both (1) and (2) have five-coordinate, approximately trigonal bipyramidal, environments. (1) contains 1,2-bis(diphenylphosphino)ethane ligands acting in two ways: one ligand bridging between the two In atoms (bidentate) and two ligands bonded to only one In atom each (monodentate). (1) is the first example of a trigonal bipyramidal type structure for an InI_3 complex. The two trimethylphosphine ligands in (2) occupy the axial positions.

Introduction. Indium(III) halide complexes of formulation $InX_3.L_2$ (X = halide, L = monodentate ligand) are believed to have two possible solid-state structures: either five-coordinate trigonal bipyramidal type structures or ionic dimeric type structures $[InX_2L_4]^+[InX_4]^-$. Complete X-ray structure determinations have been published for $InCl_3(PPh_3)_2$ (Veidis & Palenik, 1969) and $InCl_3(NMe_3)_2$ (Karia, Willey & Drew, 1986). Both complexes were found to have *trans* trigonal bipyramidal type structures. Only one X-ray structure determination has been published for an indium triiodide complex. $InI_3(Me_2SO)_2$ was found to have an ionic dimeric type structure, $[InI_2(Me_2SO)_4]^+[InI_4]^-$ in which the $[InI_4]^-$ anion is tetrahedral and the $[InI_2(Me_2SO)_4]^+$ cation has an octahedral core geometry (Einstein & Tuck, 1970).

We have recently undertaken X-ray structure determinations of the complexes $InI_3(HPPh_2)$ and $InI_3(HP^tBu_2)$ (I. A. Degnan, N. W. Alcock, S. M. Roe & M. G. H. Wallbridge, unpublished work), both of which show tetrahedral geometry. These two complexes and $InCl_3(PPh_3)_2$ are the only phosphine complexes of indium trihalides for which X-ray structure determinations have already been determined. In this work we now report X-ray structure determinations for two more such complexes: $[(InI_3)_2(diphos)_3]$ [diphos = $Ph_2P(CH_2)_2PPh_2$] (1) and $InCl_3(PMe_3)_2$ (2). Both complexes have In atoms in near trigonal bipyramidal environments with phosphine ligands occupying *trans* positions. The structure of (1) is the first such example for any InI_3 complex. The preparations of the related complexes $InI_3(diphos)$ (3) and $InI_3(diphos)_2$ (4) are also described.

Experimental. Owing to the moisture sensitivity of the products, all procedures were carried out under an atmosphere of dry nitrogen using standard Schlenk and glove-box techniques. Solvents used were carefully dried before use. Compound (2) has previously been reported but no preparative details were included (Carty, Hinsperger & Boorman, 1970); the preparative method used in the present work is described below.

Table 1. *Collection and refinement parameters*

	(1)	(2)
Absences	None	*hk*0, $h = 2n + 1$; 0*kl*, $k + l = 2n + 1$
Index ranges *h*, *k*, *l*	0/15, −16/16, −16/16	0/13, 0/13, 0/17
Crystal size (mm)	Not measured	0.3 × 0.5 × 0.2
Transmission factor range	–	0.66–0.70
No. of reflections		
Measured	7366	1597
Unique	6998	1450
Observed $[I/\sigma(I) \geq 2.0]$	5203	1247
R_{int}	0.022	–
Parameters refined	415	64
Weight parameter, *g*	0.0017	0.00087
Shift/σ in last cycle	0.03	0.10
ΔF map peaks (e Å^{-3})	1.5, −1.0	1.0, −0.9
Final *R*	0.044	0.044
Final *wR*	0.054	0.052
R, all data	0.065	0.052
S	1.0	1.9

Preparation of (2): trichlorobis(trimethylphosphine)indium(III). 1.68 g (7.59 mmol) of $InCl_3$ was dissolved in 20 cm^3 of tetrahydrofuran (THF). To this solution 1.16 g (15.3 mmol) of trimethylphosphine was added dropwise from a syringe. The resulting clear solution was concentrated under reduced pressure to *ca* 5 cm^3, and 10 cm^3 of hexane was added. This gave a microcrystalline colourless solid that was further recrystallized from a THF/hexane mixture. Crystals suitable for X-ray diffraction were obtained by slow diffusion of hexane into a tetrahydrofuran solution of (2) at room temperature.

Preparation of (3): [1,2-bis(diphenylphosphino)ethane)triiodoindium(III). To a solution of 1.76 g (3.35 mmol) of InI_3 in 10 cm^3 of acetonitrile was added dropwise a solution of 1.41 g (3.55 mmol) of diphos in 20 cm^3 of acetonitrile. After concentrating the resulting solution to approximately half of the original volume, 10 cm^3 of hexane was added. This caused the precipitation of a colourless solid which was isolated by filtration and dried *in vacuo*. Yield 2.32 g (73%). Elemental analysis found: C 34.75, H 2.80; calculated for $C_{26}H_{24}P_2InI_3$: C 34.93, H 2.71%. ^{1}H NMR ($CDCl_3$), δ 2.55 (4H, CH_2), 7.51 (20H, C_6H_5); ^{31}P[^{1}H] NMR ($C_6D_5CD_3$), δ −10 (rel. H_3PO_4).

Preparation of (1) *and* (4): bis[1,2-bis(diphenylphosphino)ethane]triiodoindium(III) (4). 1.36 g (2.75 mmol) of InI_3 was reacted with 2.19 g (5.51 mmol) of diphos using the method for (3). Yield 2.79 g (78%). Elemental analysis found: C 47.57, H 3.80; calculated for $C_{52}H_{48}P_4InI_3$: C 48.33, H 3.72%. ^{1}H NMR ($CDCl_3$), δ 2.33 (8H, CH_2), 7.42 (40H, C_6H_5); ^{31}P[^{1}H] NMR ($C_6D_5CD_3$), δ −16. Repeated attempts to obtain crystals of (3) and (4) suitable for X-ray diffraction gave only unsuitable fine needle-shaped crystals. However, attempts to obtain crystals of (3) from acetonitrile solutions layered with hexane (1:4) at

268 K gave not only these needle-shaped crystals but also a very small quantity of well formed cubes. X-ray diffraction studies on these crystals showed them to be to compound (1). Attempts at preparing (1) in high yields have so far proved to be unsuccessful. Recrystallization of a mixture of $\mathrm{InI_3}$ and diphos in a 2:3 ratio yielded mostly impure (3).

Crystal Structure Determination. Data were collected with a Nicolet $P2_1$ four-circle diffractometer in ω–2θ mode, for crystals mounted in sealed Lindemann tubes. Collection and refinement data are given in Table 1. Maximum 2θ was 50° with scan range $\pm 1.3°$ (2θ) around the $K\alpha_1 - K\alpha_2$ angles, scan speed 5–29 [compound (1)] or 4–29° min^{-1} [compound (2)], depending on the intensity of a 2 s pre-scan; backgrounds were measured at each end of the scan for 0.25 of the scan time.

Three standard reflections were monitored every 200 reflections, and showed a slight decrease during data collection for each crystal (*ca* 3%). The data were rescaled to correct for this. Unit-cell dimensions and standard deviations were obtained by least-squares fit to 15 reflections ($20 < 2\theta < 22°$). Reflections were processed using profile analysis. Those with $I/\sigma(I) \geq 2.0$ were considered observed. They were corrected for Lorentz, polarization and absorption effects (by the Gaussian method). [Crystal (1) was lost before its dimensions could be recorded, but its approximately cubic shape (*ca* 0.2 mm cube) indicates that absorption was unlikely to produce systematic errors.] Crystal (2) was prismatic, 0.19 × 0.24 × 0.28 mm.

For (1), space group $P\bar{1}$ was assumed, and the structure was solved by direct methods using *SHELXTL-Plus* (Sheldrick, 1986), locating the four heavy atoms. The $(\mathrm{InI_3})_2L_3$ molecule lies on a crystallographic centre of inversion. For (2), the systematic absences indicate either *Pnma* or $Pn2_1a$; the first was assumed and the In atom located by the Patterson interpretation section of *SHELXTL-Plus*, positioned on the mirror plane. For both structures the light atoms were then found on successive Fourier syntheses.

Anisotropic temperature factors were used for all non-H atoms. H atoms were given fixed isotropic temperature factors, $U = 0.07$ Å^2. Those defined by the molecular geometry were inserted at calculated positions and not refined; methyl groups were treated as rigid $\mathrm{CH_3}$ units. Final refinement was on F by least-squares methods. All the largest residual peaks in final difference Fourier syntheses lay near the In or I atoms.

A weighting scheme of the form $w = 1/[\sigma^2(F) + gF^2]$ was used and shown to be satisfactory by a weight analysis. Computations were performed with *SHELXTL-Plus* on a DEC MicroVAX II. Scattering factors in the analytical form and anomalous-dispersion factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final atomic coordinates are given in Tables 2 and 3, and selected bond lengths and angles in Tables 4 and 5.*

Discussion. We found that it was possible to prepare not only a 1:1 complex (3) between $\mathrm{InI_3}$ and diphos, as previously reported (Carty & Tuck, 1966), but also a 1:2 complex (4). Our attempts to obtain single crystals suitable for X-ray diffraction studies of either of these complexes were unsuccessful, though attempted crystallization of (3) gave the previously unknown complex $[(\mathrm{InI_3})_2(\mathrm{diphos})_3]$ (1). The molecular structure and atomic numbering scheme for this structure are shown in Fig. 1.

The structure consists of discrete molecules of $[(\mathrm{InI_3})_2(\mathrm{diphos})_3]$ in which one diphos ligand is acting as a bidentate ligand, bridging between both the In atoms. The other two diphos ligands are acting as monodentate ligands each with one P atom remaining uncoordinated. The core geometry around both In atoms is close to trigonal bipyramidal. The P—In—P angle is 173.7 (1)° which is slightly distorted from the ideal 180°. This can probably be attributed to constraints within the molecule arising from the bridging diphos ligand. This is the first example of an $\mathrm{InI_3}$ complex that has been conclusively proven to have a trigonal bipyramidal type structure. Significant bond lengths and angles are presented in Table 4.

The mean In—I bond distance is 2.73 Å, which is slightly longer than the mean values of 2.67 Å in $\mathrm{InI_3(HPPh_2)}$ and $\mathrm{InI_3(HP^tBu_2)}$ (I. A. Degnan, N. W. Alcock, S. M. Roe & M. G. H. Wallbridge, unpublished work), and 2.64 Å for the terminal In—I bonds in $\mathrm{In_2I_6}$ (Forrester, Zalkin & Templeton, 1964). This lengthening of the In—I bond is consistent with the increased steric requirements around the In atom on increasing the coordination number from four to five.

The In—P bond distances of 2.798 (3) Å [In(1)—P(2)] and 2.819 (2) Å [In(1)—P(3)] are the longest In—P bond distances that have been reported to date. They are significantly longer than the mean distances of 2.60 Å in $\mathrm{InI_3(HPPh_2)}$, 2.59 Å in $\mathrm{InI_3(HP^tBu_2)}$ (I. A. Degnan, N. W. Alcock, S. M. Roe & M. G. H. Wallbridge, unpublished work), 2.59 Å in $\mathrm{In(P^tBu_2)_3}$, 2.64 Å in $\mathrm{(Et_2InP^tBu_2)_2}$ (Alcock, Degnan, Wallbridge, Powell, McPartlin &

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* Lists of anisotropic thermal parameters, H-atom coordinates, full bond lengths and angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54822 (40 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI0104]

Table 2. *Atom coordinates* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($Å^2 \times 10^3$) *for compound* (1)

Equivalent isotropic U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor

	x	*y*	*z*	U_{eq}
In(1)	3247.3 (4)	7920.3 (4)	3470.6 (4)	41 (1)
I(1)	4635.5 (4)	7143.3 (5)	2011.2 (4)	51 (1)
I(2)	1794.1 (5)	6674.0 (5)	4522.9 (4)	56 (1)
I(3)	3346.2 (6)	9899.4 (4)	3889.6 (5)	65 (1)
P(1)	1013 (2)	6178 (2)	677 (2)	48 (1)
P(2)	1690 (2)	8968 (2)	1947 (2)	39 (1)
P(3)	4629.3 (16)	6772.8 (15)	5142.9 (14)	37 (1)
C(1)	932 (6)	8012 (6)	1586 (6)	46 (3)
C(2)	1698 (6)	7023 (6)	1310 (7)	49 (3)
C(3)	4729 (6)	5267 (6)	5439 (6)	43 (3)
C(11)	1957 (7)	4874 (7)	1024 (7)	58 (3)
C(12)	1631 (9)	3872 (8)	1444 (8)	74 (4)
C(13)	2357 (13)	2907 (9)	1637 (11)	100 (6)
C(14)	3432 (13)	2909 (10)	1414 (11)	106 (7)
C(15)	3798 (10)	3861 (11)	1025 (12)	104 (6)
C(16)	3057 (9)	4862 (9)	824 (9)	81 (5)
C(21)	−110 (7)	5952 (7)	1586 (6)	49 (3)
C(22)	−1146 (7)	6195 (8)	1202 (8)	66 (4)
C(23)	−2021 (9)	6100 (10)	1897 (13)	96 (6)
C(24)	−1877 (12)	5789 (11)	2945 (12)	101 (6)
C(25)	−879 (11)	5561 (10)	3350 (9)	86 (5)
C(26)	9 (9)	5627 (8)	2679 (7)	67 (4)
C(31)	649 (6)	9973 (6)	2385 (6)	42 (3)
C(32)	109 (7)	9640 (8)	3333 (7)	65 (4)
C(33)	−665 (8)	10366 (10)	3719 (7)	81 (5)
C(34)	−927 (9)	11437 (10)	3159 (9)	85 (5)
C(35)	−416 (8)	11782 (8)	2216 (8)	73 (4)
C(36)	371 (7)	11044 (7)	1838 (7)	55 (3)
C(41)	2207 (6)	9713 (6)	705 (5)	38 (2)
C(42)	3100 (7)	10166 (7)	709 (6)	58 (3)
C(43)	3487 (9)	10775 (8)	−211 (8)	74 (4)
C(44)	2995 (8)	10878 (8)	−1129 (7)	64 (4)
C(45)	2107 (8)	10409 (8)	−1158 (6)	66 (4)
C(46)	1712 (7)	9819 (7)	−238 (6)	56 (3)
C(51)	6012 (6)	7042 (6)	5046 (6)	40 (3)
C(52)	6760 (8)	6476 (8)	5773 (7)	64 (4)
C(53)	7792 (8)	6765 (9)	5719 (9)	77 (4)
C(54)	8050 (8)	7577 (9)	4925 (8)	67 (4)
C(55)	7303 (8)	8127 (9)	4202 (7)	74 (4)
C(56)	6284 (7)	7867 (8)	4256 (6)	58 (3)
C(61)	4132 (6)	7119 (6)	6372 (6)	41 (3)
C(62)	3569 (8)	6446 (7)	7120 (7)	61 (4)
C(63)	3193 (8)	6751 (10)	8042 (7)	77 (4)
C(64)	3376 (9)	7713 (10)	8232 (8)	81 (5)
C(65)	3916 (8)	8376 (9)	7497 (8)	73 (4)
C(66)	4308 (7)	8087 (7)	6586 (7)	56 (3)

Table 3. *Atom coordinates* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($Å^2 \times 10^3$) *for compound* (2)

Equivalent isotropic U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor

	x	*y*	*z*	U_{eq}
In(1)	2093.7 (6)	7500.0	5228.8 (4)	45 (1)
Cl(1)	2844.0 (16)	5528.0 (17)	6004.1 (13)	58 (1)
Cl(2)	647 (3)	7500	3827 (2)	88 (1)
P(1)	91 (2)	7500	6322 (2)	53 (1)
P(2)	3981 (3)	7500	4023 (2)	59 (1)
C(11)	514 (13)	7500	7586 (8)	83 (5)
C(12)	−912 (8)	6169 (9)	6189 (8)	102 (4)
C(21)	5608 (12)	7500	4622 (10)	82 (5)
C(22)	3965 (10)	8847 (8)	3288 (6)	88 (3)

Sheldrick, 1989) and 2.71 Å in $InCl_3(PPh_3)_2$ (Veidis & Palenik, 1969). A contributing factor to these long bonds is probably the steric bulk of the three iodide ligands.

$InCl_3(PMe_3)_2$ (2) is isostructural with $InCl_3(PPh_3)_2$ and $InCl_3(NMe_3)_2$ which are the only other similar

Table 4. *Principal bond lengths* (Å) *and angles* (°) *for compound* (1)

In(1)—I(1)	2.742 (2)	In(1)—I(2)	2.739 (2)
In(1)—I(3)	2.713 (2)	In(1)—P(2)	2.798 (3)
In(1)—P(3)	2.819 (2)	P(1)—C(2)	1.846 (10)
P(1)—C(11)	1.826 (9)	P(1)—C(21)	1.814 (8)
P(2)—C(1)	1.829 (9)	P(2)—C(31)	1.817 (8)
P(2)—C(41)	1.829 (8)	P(3)—C(3)	1.838 (7)
P(3)—C(51)	1.826 (8)	P(3)—C(61)	1.811 (8)

I(1)—In(1)—I(2)	119.7 (1)	I(1)—In(1)—I(3)	119.8 (1)
I(2)—In(1)—I(3)	120.4 (1)	I(1)—In(1)—P(2)	91.9 (1)
I(2)—In(1)—P(2)	89.5 (1)	I(3)—In(1)—P(2)	89.7 (1)
I(1)—In(1)—P(3)	92.3 (1)	I(2)—In(1)—P(3)	84.4 (1)
I(3)—In(1)—P(3)	92.2 (1)	P(2)—In(1)—P(3)	173.7 (1)

Table 5. *Principal bond lengths* (Å) *and angles* (°) *for compound* (2)

In(1)—Cl(1)	2.503 (3)	In(1)—Cl(2)	2.453 (4)
In(1)—P(1)	2.575 (3)	In(1)—P(2)	2.576 (3)
P(1)—C(12)	1.783 (10)	P(1)—C(11)	1.801 (12)
P(2)—C(21)	1.885 (13)	P(2)—C(22)	1.771 (9)

Cl(1)—In(1)—Cl(2)	122.0 (1)	Cl(1)—In(1)—P(1)	90.1 (1)
Cl(2)—In(1)—P(1)	88.0 (1)	Cl(1)—In(1)—P(2)	92.2 (1)
Cl(2)—In(1)—P(2)	87.6 (1)	P(1)—In(1)—P(2)	175.6 (1)
Cl(1)—In(1)—Cl(1A)	116.1 (1)		

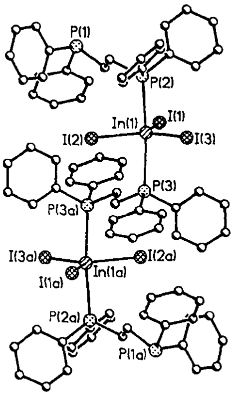

Fig. 1. View of molecular structure of (1) showing principal atomic numbering.

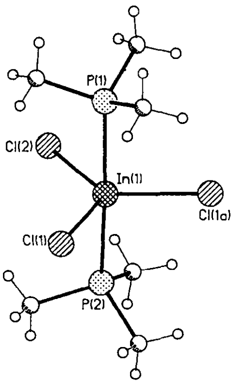

Fig. 2. View of molecular structure of (2) showing principal atomic numbering.

complexes of $InCl_3$ to have been structurally characterized by single-crystal X-ray studies. The structure consists of discrete molecules of (2), in which the environment of the In atom shows only slight distortions from an ideal trigonal bipyramidal geometry [P—In—P angle of 175.6 (1)°]. This is in agreement with the geometry proposed from the powder diffraction study previously carried out on this compound (Beattie, Ozin & Blayden, 1969). The molecular structure and atomic numbering scheme are shown in Fig. 2, with bond distances and angles in Table 5.

The mean In—Cl distance is 2.49 (1) Å and the mean In—P distance is 2.576 (3) Å. These can be compared to the distances in $InCl_3(PPh_3)_2$, which has a shorter mean In—Cl distance of 2.38 Å and a longer mean In—P distance of 2.71 Å. Taken together these differences suggest that PPh_3 is a weaker ligand than PMe_3, with the stronger In—Cl bonds compensating for the weaker In—P bonds. The extra steric bulk of the PPh_3 ligand may contribute to this weakness. PPh_3 has a Tolman cone angle (Tolman, 1970) of 145°, while PMe_3 has a cone angle of 118°. Comparison of the In—P bond distances in (2) with other known In—P distances suggests that the mean value of 2.576 Å found in (2) is closer to a 'normal' single-bond distance than the longer distance in $InCl_3(PPh_3)_2$. For example, it is similar to the mean In—P values in $In(P^tBu_2)_3$, in the various adducts of InI_3 noted above, and in $[Li(THF)_4]^+$ $[In(PPh_2)_4]^-$ (2.58 Å) (Carrano, Cowley, Giolando, Jones, Nunn & Power, 1988). The mean In—Cl bond distance of 2.49 Å is close to the value of 2.45 Å in $[Et_4N]_2[InCl_5]$ (Brown, Einstein & Tuck, 1969) and $[InCl_4(H_2O)_2]^-$ (Zeigler, Schlimper, Nuber, Weiss & Ertl, 1975). It is, however, somewhat longer than the values of 2.38 Å found in $InCl_3(PPh_3)_2$ and 2.37 Å in $InCl_3(NMe_3)_2$. Clearly, the nature of the axial ligand in these trigonal bipyramidal complexes has a significant effect on the lengths of the equatorial In—Cl bonds.

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Structural Characterization of 1,1,1,2,2,2,3,3,3-Nonacarbonyl-1,2-μ-iodo-1,2,3-[μ_3-(phenylpyridylphosphido)-μ-*P*,*N*]-*triangulo*-triruthenium

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Abstract. $C_{20}H_9INO_9PRu_3$, $M_r = 868.4$, monoclinic, $P2_1/c$, $a = 10.058$ (1), $b = 14.182$ (3), $c = 17.988$ (3) Å, $\beta = 97.94$ (2)°, $V = 2541$ Å^3, $Z = 4$, $D_x = 2.269$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 30.40$ cm^{-1}, $F(000) = 1632$, room temperature, final $R = 0.025$ for 3564 unique observed reflections with I

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