because all the PT moieties exist as cation radicals possessing unit charge, and the mean interplanar distances are somewhat large compared with that of graphite (3·35 Å). The polarized absorption spectrum parallel to the *c* axis of a single crystal of (II) shows the characteristic peaks of the PT cation radical, and it also shows that the PT moieties have some mutual interaction, since there is a broad band around 1000 nm which one may assign to the charge-transfer band between PT cation radicals (Uchida, Seki, Ito, Nakano, Hoshizaki & Kozawa, 1986).

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Ten-Vertex Metallaborane Chemistry. Structure of $3\text{-}(OC_2H_5)\text{-}4,9\text{-}(PPh_3)_2$-$7,7\text{-}Cl(Ph_2P$-*ortho*-$C_6H_4)$-*isonido*-$7\text{-}IrB_9H_5\text{-}10\text{-}.CH_2Cl_2$

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Abstract. 7-Chloro-7,10-μ-(*ortho*-diphenylphosphinophenyl-*P*,*C*)-3-ethoxy-4,9-bis(triphenylphosphino)-7-irida-*isonido*-decaborane(5) dichloromethane solvate, $C_{56}H_{54}B_9ClIrOP_3.CH_2Cl_2$, $M_r = 1245{\cdot}87$, monoclinic, $P2_1/n$, $a = 19{\cdot}543\ (3)$, $b = 13{\cdot}199\ (3)$, $c = 23{\cdot}258\ (3)$ Å, $\beta = 107{\cdot}93\ (1)°$, $V = 5708$ Å^3, $Z = 4$, $D_x = 1{\cdot}45$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 2{\cdot}480$ mm^{-1}, $F(000) = 2496$, room temperature, $R = 0{\cdot}0349$, 5954 unique reflections with $F > 4{\cdot}0\sigma(F)$. The IrB_9 cluster structure is of the rare *isonido* ten-vertex geometry that is formally derived from an eleven-vertex *closo* geometry by removal of a four-connected vertex.

Introduction. As part of our investigation of the chemical and thermolytic reactions of unsubstituted and phosphine-substituted *nido*-iridadecaboranes, we have determined the crystal and molecular structure of the title compound which was isolated in about 0·1% yield as the only identified metallaborane prod-

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uct (reaction scale 25 μmol) of the thermolysis of 5,7,5,5-H$(PPh_3)_2(Ph_2P$-*ortho*-$C_6H_4)$-*nido*-5-IrB_9H_{10}-2-.

Experimental. A dry, deoxygenated solution of 5,7,5,5-H$(PPh_3)_2(Ph_2P$-*ortho*-$C_6H_4)$-*nido*-5-IrB_9H_{10}-2- (27 mg, 25 μmol, prepared as in Bould, Crook, Greenwood, Kennedy & Thornton-Pett, 1990) in benzene (20 cm^3) was heated under reflux (N_2 atmosphere) for 15 h, after which time the benzene was removed on a rotary evaporator. The residue was taken up in dichloromethane (*ca* 2 cm^3), and the solution/suspension applied to a preparative TLC plate (200 × 200 × 1 mm on a glass former, silica gel G, Fluka type GF254, water slurry, dried at 353 K). Development using dichloromethane/pentane (75:25) as the eluting medium resulted in only one mobile component, a pale red band (R_f 0·5), and revealed a considerable amount of dark non-mobile intractible material. The red component was further purified *via* HPLC (silica column, Knauer, 250 mm × 16 mm diameter, Lichrosorb Si60, 7 μm) with 100% dichloromethane as the eluting medium. The resulting small amount of red material was dissolved in dichloromethane, and pentane diffusion into this solution then gave the entire product (*ca* 35 μg, 0·028 μmol) as a single red crystal of suitable dimensions (0·6 × 0·4 × 0·1 mm) for a single-crystal X-ray diffraction analysis. All measurements were made on a Nicolet *P*3/*F* diffractometer; 25 reflections centred ($35{\cdot}0 < 2\theta < 40{\cdot}0°$), graphite-monochromated Mo *Kα* radiation (0·71069 Å), ω–2θ scans, ω scan widths 2° + α-doublet splitting, scan speeds 2·0–29·3° min^{-1} subject to a fast prescan. Complete data set ($h\ 0 \rightarrow 21$, $k\ 0 \rightarrow 14$, $l\ -24 \rightarrow 24$), $2\theta_{max} = 45°$, measured over 260 exposure hours with no significant decay of a standard reflection (measured every 50 reflections); 7998 unique data, 5954 with $F > 4{\cdot}0\sigma(F)$ considered observed and used in structure solution and refinement. Structure solution by Patterson synthesis (for the iridium atom) and difference Fourier methods; empirical absorption correction (Walker & Stuart, 1983), max., min. and average absorption correction factors 0·880, 1·273 and 1·018; full-matrix least-squares refinement with anisotropic thermal parameters for all non-hydrogen atoms except those in a disordered dichloromethane solvate molecule which were assigned isotropic thermal parameters. All phenyl groups were treated as rigid bodies with idealized hexagonal symmetry (C—C = 1·395 Å). Phenyl hydrogen atoms were included in calculated positions (C—H = 1·08 Å) and were assigned to an overall isotropic thermal parameter. The borane hydrogen atoms were located in a Fourier difference synthesis but these tended to move to unreasonable positions when refined so they were assigned to a fixed isotropic thermal parameter and their positional parameters were not included in the refinement. Scattering factors and anomalous-dispersion corrections were from *International Tables for X-ray Crystallography* (1974). Refinement converged to $R = 0{\cdot}0349$, $wR = 0{\cdot}0357$, $w^{-1} = \sigma^2(F_o) + 0{\cdot}0006(F_o)^2$, difference synthesis showed max. density of 0·88 e $Å^{-3}$ (min. −0·64 e $Å^{-3}$); max. $\Delta/\sigma = 0{\cdot}15$ in U_{12} of C(113). All calculations were performed on an Amdahl 5850 computer. Programs *SHELX*76 (Sheldrick, 1976), *PARST* (Nardelli, 1983) and *ORTEP*II (Johnson, 1976).

Discussion. Final fractional coordinates and equivalent isotropic thermal parameters are listed in Table 1, and the molecular structure and its numbering scheme are shown in Fig. 1. Selected interatomic distances and angles are in Table 2.* The compound is revealed to be 3-(OC_2H_5)-4,9-$(PPh_3)_2$-7,7-Cl-$(Ph_2P$-*ortho*-$C_6H_4)$-*isonido*-7-IrB_9H_5-10-.CH_2Cl_2. The basic ten-vertex *isonido* IrB_9 cluster may be regarded as being formally derived from an eleven-vertex *closo*-type geometry, as represented by the compound $(PPh_3)_2RuB_{10}H_8(OEt)_2$ (Crook, Elrington, Greenwood, Kennedy, Thornton-Pett & Woollins, 1985), by the removal of a four-connected vertex rather than by the removal of the six-connected vertex that would produce the far more common *nido*-type cluster as exemplified by $(PPh_3)_2HIrB_9H_{13}$ (Boocock, Bould, Greenwood, Kennedy & McDonald, 1982). The notably short Ir(7)—B(3) distance is mirrored by correspondingly short *M*(1)—B(2) and *M*(1)—B(3) distances in the eleven-vertex *closo*-type metallaboranes. The analysis reveals the presence of iridium-bound chlorine and boron-bound ethoxy moieties, presumably arising from interactions with CH_2Cl_2 solvent and fortuitous traces of ethanol respectively, the latter probably arising in the chromatographic work. Thermolysis in the presence of ethanol under otherwise similar conditions resulted in a (more rapid) general degradation and no metallaborane products.

Iridium–boron, iridium–phosphorous and inter-boron distances are all within the normal range (Kennedy, 1986, and references therein) for iridaboranes. The boron–phosphorus distances of the boron-bound phosphines are also within the usual range although that for the phosphine attached to the open face of the cluster [1·866 (9) Å] is slightly but significantly shorter than for the phosphine away from the open face [1·931 (8) Å]. As mentioned

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* Lists of structure factors, anisotropic thermal parameters, H-atom and solvent parameters and complete lists of bond distances and angles, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52654 (44 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atom coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors*

	x	*y*	*z*	U_{eq}*(Å^2)
Ir(7)	2638·6 (1)	1788·2 (2)	140·7 (1)	34·2 (1)
Cl(1)	2192 (1)	2705 (1)	822 (1)	55·8 (7)
P(1)	2046 (1)	2724 (1)	−710 (1)	45·5 (6)
P(2)	3899 (1)	678 (1)	1623 (1)	40·1 (6)
P(3)	1762 (1)	−1644 (1)	−343 (1)	40·6 (6)
C(111)	1696 (2)	1854 (3)	−1313 (2)	47 (2)
C(112)	1914 (2)	848 (3)	−1196 (2)	43 (2)
C(113)	1719 (2)	132 (3)	−1659 (2)	57 (3)
C(114)	1306 (2)	421 (3)	−2239 (2)	74 (4)
C(115)	1088 (2)	1427 (3)	−2355 (2)	77 (4)
C(116)	1283 (2)	2143 (3)	−1892 (2)	70 (3)
C(121)	1304 (2)	3581 (3)	−774 (2)	50 (3)
C(122)	794 (2)	3282 (3)	−501 (2)	66 (3)
C(123)	177 (2)	3863 (3)	−575 (2)	84 (4)
C(124)	70 (2)	4744 (3)	−922 (2)	80 (4)
C(125)	580 (2)	5044 (3)	−1196 (2)	80 (4)
C(126)	1197 (2)	4462 (3)	−1122 (2)	61 (3)
C(131)	2716 (2)	3476 (3)	−896 (2)	54 (3)
C(132)	3097 (2)	4190 (3)	−477 (2)	71 (3)
C(133)	3675 (2)	4704 (3)	−574 (2)	103 (5)
C(134)	3873 (2)	4504 (3)	−1090 (2)	118 (6)
C(135)	3493 (2)	3790 (3)	−1509 (2)	119 (7)
C(136)	2914 (2)	3275 (3)	−1412 (2)	93 (5)
C(211)	3985 (2)	1870 (3)	2018 (2)	44 (2)
C(212)	4189 (2)	1850 (3)	2647 (2)	64 (3)
C(213)	4349 (2)	2751 (3)	2975 (2)	76 (4)
C(214)	4304 (2)	3673 (3)	2672 (2)	76 (4)
C(215)	4100 (2)	3694 (3)	2042 (2)	76 (4)
C(216)	3940 (2)	2792 (3)	1715 (2)	54 (3)
C(221)	4817 (2)	268 (3)	1731 (2)	43 (2)
C(222)	5378 (2)	962 (3)	1929 (2)	59 (3)
C(223)	6068 (2)	703 (3)	1923 (2)	73 (4)
C(224)	6197 (2)	−250 (3)	1719 (2)	65 (3)
C(225)	5635 (2)	−945 (3)	1521 (2)	66 (3)
C(226)	4945 (2)	−686 (3)	1527 (2)	59 (3)
C(231)	3504 (3)	−125 (4)	2061 (2)	56 (3)
C(232)	3869 (3)	−915 (4)	2428 (2)	84 (4)
C(233)	3523 (3)	−1486 (4)	2761 (2)	123 (6)
C(234)	2814 (3)	−1269 (4)	2727 (2)	117 (6)
C(235)	2449 (3)	−479 (4)	2361 (2)	110 (5)
C(236)	2794 (3)	93 (4)	2028 (2)	79 (4)
C(311)	1878 (2)	−2575 (3)	249 (2)	46 (2)
C(312)	1967 (2)	−2237 (3)	836 (2)	63 (3)
C(313)	2008 (2)	−2934 (3)	1296 (2)	76 (4)
C(314)	1960 (2)	−3969 (3)	1170 (2)	79 (4)
C(315)	1872 (2)	−4307 (3)	583 (2)	77 (4)
C(316)	1831 (2)	−3610 (3)	123 (2)	64 (3)
C(321)	837 (2)	−1279 (4)	−529 (2)	51 (3)
C(322)	643 (2)	−259 (4)	−611 (2)	61 (3)
C(323)	−81 (2)	16 (4)	−777 (2)	86 (4)
C(324)	−610 (2)	−727 (4)	−862 (2)	103 (5)
C(325)	−416 (2)	−1746 (4)	−780 (2)	100 (5)
C(326)	308 (2)	−2022 (4)	−614 (2)	70 (3)
C(331)	1852 (2)	−2343 (3)	−986 (2)	47 (2)
C(332)	2528 (2)	−2742 (3)	−940 (2)	62 (3)
C(333)	2643 (2)	−3227 (3)	−1435 (2)	83 (4)
C(334)	2083 (2)	−3313 (3)	−1976 (2)	88 (4)
C(335)	1407 (2)	−2913 (3)	−2022 (2)	81 (4)
C(336)	1292 (2)	−2428 (3)	−1527 (2)	61 (3)
O(1)	4237 (2)	2244 (3)	510 (2)	52 (2)
C(1)	4789 (5)	2310 (8)	212 (5)	121 (6)
C(2)	5369 (6)	2878 (11)	540 (6)	161 (7)
B(1)	3855 (4)	226 (6)	238 (3)	41 (3)
B(2)	3310 (4)	991 (6)	−377 (3)	43 (3)
B(3)	3719 (4)	1501 (5)	342 (3)	39 (3)
B(4)	3384 (4)	651 (5)	769 (3)	38 (3)
B(5)	3214 (4)	−587 (5)	427 (3)	38 (3)
B(6)	3149 (4)	−345 (5)	−362 (3)	40 (3)
B(8)	2485 (3)	256 (5)	456 (3)	39 (3)
B(9)	2384 (4)	−537 (5)	−144 (3)	37 (2)
B(10)	2412 (4)	549 (5)	−534 (3)	41 (3)

* $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

Table 2. *Selected interatomic distances* (Å) *and angles* (°) *for* 3-(OC_2H_5)-4,9-(PPh_3)$_2$-7,7-Cl(Ph_2P-*ortho*-C_6H_4)-*isonido*-7-IrB_9H_5-10- *with estimated standard deviations in parentheses*

Cl(1)—Ir(7)	2·364 (4)	P(1)—Ir(7)	2·316 (4)
B(2)—Ir(7)	2·292 (10)	B(3)—Ir(7)	2·053 (9)
B(4)—Ir(7)	2·280 (8)	B(8)—Ir(7)	2·204 (9)
B(10)—Ir(7)	2·215 (9)	B(4)—P(2)	1·931 (8)
B(9)—P(3)	1·866 (9)		
B(10)—C(112)	1·597 (9)		
C(1)—O(1)	1·452 (14)	B(3)—O(1)	1·377 (9)
C(2)—C(1)	1·376 (16)		
B(2)—B(1)	1·807 (11)	B(3)—B(1)	1·733 (12)
B(4)—B(1)	1·841 (13)	B(5)—B(1)	1·802 (13)
B(6)—B(1)	1·799 (11)		
B(3)—B(2)	1·751 (11)	B(6)—B(2)	1·793 (12)
B(10)—B(2)	1·778 (12)		
B(4)—B(3)	1·753 (13)	B(5)—B(4)	1·803 (11)
B(8)—B(4)	1·762 (11)	B(6)—B(5)	1·827 (12)
B(8)—B(5)	1·827 (12)	B(9)—B(5)	1·753 (10)
B(9)—B(6)	1·739 (13)	B(10)—B(6)	1·809 (12)
B(9)—B(8)	1·708 (12)	B(10)—B(9)	1·705 (12)

P(1)—Ir(7)—Cl(1)	96·3 (2)	B(2)—Ir(7)—Cl(1)	167·5 (2)
B(2)—Ir(7)—P(1)	90·7 (3)	B(3)—Ir(7)—Cl(1)	120·5 (3)
B(3)—Ir(7)—P(1)	120·1 (3)	B(3)—Ir(7)—B(2)	47·1 (2)
B(4)—Ir(7)—Cl(1)	101·6 (3)	B(4)—Ir(7)—P(1)	161·9 (2)
B(4)—Ir(7)—B(2)	71·2 (4)	B(4)—Ir(7)—B(3)	47·4 (3)
B(8)—Ir(7)—Cl(1)	97·4 (3)	B(8)—Ir(7)—P(1)	133·6 (2)
B(8)—Ir(7)—B(2)	85·0 (4)	B(8)—Ir(7)—B(3)	89·5 (4)
B(8)—Ir(7)—B(4)	46·3 (2)	B(10)—Ir(7)—Cl(1)	144·9 (2)
B(10)—Ir(7)—P(1)	81·4 (3)	B(10)—Ir(7)—B(2)	46·4 (3)
B(10)—Ir(7)—B(3)	89·6 (4)	B(10)—Ir(7)—B(4)	85·3 (3)
B(10)—Ir(7)—B(8)	62·6 (4)		
B(3)—O(1)—C(1)	119·8 (7)	C(2)—C(1)—O(1)	112·0 (11)
B(3)—B(1)—B(2)	59·3 (5)	B(4)—B(1)—B(2)	93·8 (6)
B(4)—B(1)—B(3)	58·7 (5)	B(5)—B(1)—B(2)	103·9 (6)
B(5)—B(1)—B(3)	113·3 (6)	B(5)—B(1)—B(4)	59·3 (5)
B(6)—B(1)—B(2)	59·6 (5)	B(6)—B(1)—B(3)	113·6 (6)
B(6)—B(1)—B(4)	103·1 (6)	B(6)—B(1)—B(5)	61·0 (5)
B(1)—B(2)—Ir(7)	97·7 (5)	B(3)—B(2)—Ir(7)	59·3 (4)
B(3)—B(2)—B(1)	58·3 (5)	B(6)—B(2)—Ir(7)	108·0 (6)
B(6)—B(2)—B(1)	60·0 (5)	B(6)—B(2)—B(3)	113·0 (6)
B(10)—B(2)—Ir(7)	64·5 (4)	B(10)—B(2)—B(1)	106·5 (6)
B(10)—B(2)—B(3)	117·0 (7)	B(10)—B(2)—B(6)	60·9 (5)

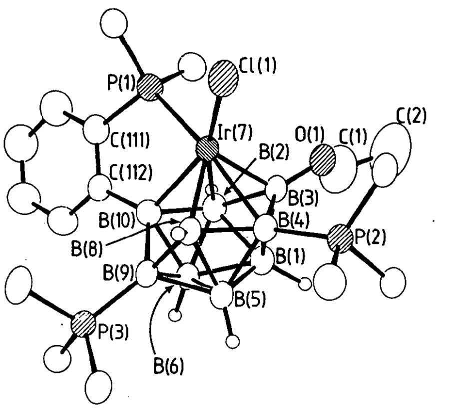

Fig. 1. *ORTEP* drawing of the molecular structure of 3-(OC_2H_5)-4,9-(PPh_3)$_2$-7,7-Cl(Ph_2P-*ortho*-C_6H_4)-*isonido*-7-IrB_9H_5-10-, with phenyl hydrogen atoms and phenyl carbon atoms (apart from the *ipso* ones) omitted for clarity Ir(7)—B(9) and B(8)—B(10) are regarded as non-bonding at 3·147 (7) and 2·30 (1) Å respectively.

above, the most notable interatomic distance is that for Ir(7)—B(3) which, at 2·053 (9) Å, is substantially shorter than the other iridium–boron distances [which fall into two pairs: Ir(7)—B(2),B(4) mean value 2·286 (7), and Ir(7)—B(8),B(10) mean value 2·209 (8) Å], suggesting a greater degree of two-centre two-electron bonding in this region. This apparently mirrors the equivalent distance of 2·08 (4) Å (although the large e.s.d. in this value makes comparison difficult) in the *quasi*-isostructural iridacarbadecaborane (PPh_3)(Ph_2P-*ortho*-

$\overline{C_6H_4)IrC(OH)}B_8H_6(OMe)$ which is the only other example of a ten-vertex *isonido* iridaborane species described in the primary literature (Crook, Greenwood, Kennedy & McDonald 1981).

The asymmetric arrangement of the ligands with respect to the idealized mirror plane of symmetry of the IrB_9 cluster [*i.e.* that through Ir(7)B(1)B(3)B(7)] does not seem to be reflected in significant differences in the iridium–boron distances, suggesting that the carbon atom produces most of the differences that have been observed between the corresponding interatomic distances in the iridacarbadecaborane. The larger angle of fold of the open face of the iridacarbadecaborane (as defined by the angle between the planes formed by the cluster vertices 8, 9, 10 and 7, 8, 10), which is 22·1 (2)° compared to 5·4 (5)° in the title compound, is probably also mainly a result of the shorter distances between the cluster carbon atom and its attached atoms.

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trans-Tetrakis(3-*tert*-butyl-5-methylpyrazole)bis(tetrafluoroborato)cobalt(II), a Complex with Two Coordinated Tetrafluoroborate Anions

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Abstract. $[Co(BF_4)_2(C_8H_{14}N_2)_4]$, M_r = 785·43, monoclinic, $C2/c$, a = 27·511 (11), b = 11·729 (5), c = 15·880 (5) Å, β = 120·46 (2)°, V = 4417 (3) Å^3, Z = 4, D_x = 1·181 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 0·448 mm^{-1}, F(000) = 1652, T = 295 K, R = 0·077 for 3911 independent reflections. The complex is octahedral with two axially coordinated BF_4^- ligands and four equatorial pyrazole ligands. The coordination of the BF_4^- ligand results in the expected elongation of the B1—F1 bond compared with the average bond length of the non-coordinated F atoms.

Introduction. While the tetrafluoroborate anion has long been used as a non-coordinating anion, it has also been exploited as a labile ligand in a variety of transition-metal complexes (Beck & Schloter, 1978; Brown, Nuttall, McAvoy & Sharp, 1966; Legzdins & Martin, 1983; Mattson & Graham, 1981; Olgemöller, Bauer, Löbermann, Nagel & Beck, 1982; Rheingold, Wu & Heck, 1987). There have been a number of X-ray structure determinations in which a monodentate BF_4^- anion has been variously described as a 'semi-coordinated' (Brown, Lee & Melsom, 1967), 'weakly coordinated' (Gaughan, Dori & Ibers, 1974), or 'normal' (Tomlinson, Bonamico, Dessy, Fares & Scaramuzza, 1972) ligand, the differentiating criterion being the metal—fluorine bond distance. Here we report the structure of *trans*-tetrakis(3-*tert*-butyl-5-methylpyrazole)bis(tetrafluoroborato)cobalt(II) (1). Analogous complexes of the type $CoL_4(BF_4)_2$ (where L = pyridine or γ-picoline) have been reported (Brown, Nuttall, McAvoy & Sharp, 1966), but not characterized structurally.

Experimental. The reaction of potassium hydridotris(3-*tert*-butyl-5-methylpyrazolyl)borate with hexaaquacobalt(II) tetrafluoroborate in dry tetrahydrofuran results in the hydrolysis of the hydridotris(3-*tert*-butyl-5-methylpyrazolyl)borate ligand and the evolution of H_2. (1) was isolated from this reaction mixture as light pink solid in 20% yield. ^{1}H NMR ($CDCl_3$): 82·0 (*br s*, 1H), 11·9 (*br s*, 9H), −13·3 (*br s*, 3H) p.p.m. IR (Nujol): 3376 (*vs*), 1567 (*s*), 1295 (*s*), 1148 (*s*), 1079 (*s*), 1036 (*s*), 940 (*s*), 827

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