

C_6H_4)IrC(OH)B₈H₆(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 IrB₉ 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 iridacarbadeborane. The larger angle of fold of the open face of the iridacarbadeborane (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(BF₄)₂(C₈H₁₄N₂)₄], *M_r* = 785.43, monoclinic, *C*2/*c*, *a* = 27.511 (11), *b* = 11.729 (5), *c* = 15.880 (5) Å, β = 120.46 (2)°, *V* = 4417 (3) Å³, *Z* = 4, *D_x* = 1.181 Mg m⁻³, λ(Mo *K*α) = 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 BF₄⁻ ligands and four equatorial pyrazole ligands. The coordination of the BF₄⁻ 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 & Schlöter, 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 mono-

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dentate BF₄⁻ 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 CoL₄(BF₄)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 H₂. (1) was isolated from this reaction mixture as light pink solid in 20% yield. ¹H NMR (CDCl₃): 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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(s), 761 (s), 720 (s) cm⁻¹; m.p. 511–518 K (dec.). Analytical calculation (%) for C₃₂H₅₆B₂F₈N₈Co: C 48.93; H 7.20; N 14.25. Found: C 49.18; H 7.45; N 14.49. Single crystals of (1) were grown from toluene/ether solution at 243 K. A pink parallelepiped (0.2 × 0.4 × 0.4 mm) was mounted in a glass capillary. The unit-cell parameters were derived from a least-squares fit of 24 reflections. Data were collected (2θ–θ) using a Syntex P2₁ diffractometer through the hkl ranges 0 to 32, 0 to 13, and –18 to 16 respectively, with 4212 reflections measured, 3911 independent reflections processed ($R_{\text{int}} = 2.57\%$), 2227 reflections observed [$F > 4.0\sigma(F)$], 2θ range 0.0–50.0°. Three standard reflections were measured every 50 reflections. The structure was solved using the Nicolet SHELXTL-PLUS system (Sheldrick, 1987) on a MicroVAXII. The Co atom was located from the Patterson map. Further difference Fourier syntheses located the remaining non-H atoms. The H atoms were placed in idealized positions prior to refinement (fixed isotropic U). Full-matrix least-squares refinement minimized $\sum w(F_o - F_c)^2$. Refinement converged with $R = 0.077$ and $wR = 0.090$, GOF = 2.12, (Δ/σ) = 3.002, ($\Delta\rho$)_{max} = 0.42 e Å⁻³. Scattering factors used were those in SHELXTL-PLUS.

Discussion. The crystal structure of (1) is shown in Fig. 1. Atomic positions are given in Table 1.* The Co atom sits on a crystallographic C₂ axis which

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52634 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

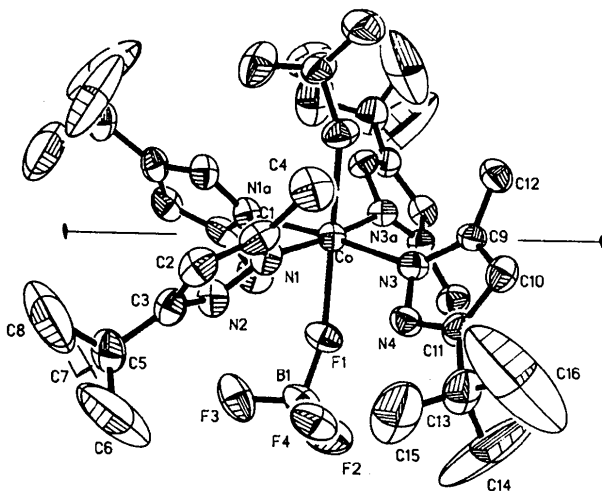


Fig. 1. Molecular structure and labelling scheme for the non-H atoms in [Co(C₈H₁₄N₂)₄(BF₄)₂] with 30% probability thermal ellipsoids. The twofold axis of rotation is indicated.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

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

	x	y	z	U_{eq}
Co	0	2390 (1)	2500	58 (1)
N(1)	-444 (3)	3735 (5)	2727 (4)	67 (3)
N(2)	-678 (3)	4583 (5)	2053 (4)	70 (3)
C(1)	-563 (3)	4024 (8)	3428 (5)	76 (4)
C(2)	-849 (3)	5055 (7)	3186 (6)	82 (5)
C(3)	-928 (3)	5394 (6)	2294 (6)	73 (4)
C(4)	-382 (4)	3300 (8)	4302 (6)	102 (5)
C(5)	-1212 (5)	6388 (8)	1674 (8)	96 (6)
C(6)	-1791 (8)	6329 (17)	1210 (15)	394 (28)
C(7)	-1105 (8)	6601 (14)	915 (12)	327 (25)
C(8)	-1057 (9)	7389 (11)	2234 (12)	383 (25)
N(3)	-505 (2)	1209 (5)	2694 (4)	63 (3)
N(4)	-1056 (2)	1459 (5)	2292 (4)	67 (3)
C(9)	-416 (3)	343 (6)	3311 (5)	60 (3)
C(10)	-913 (3)	80 (7)	3295 (5)	72 (5)
C(11)	-1317 (3)	801 (6)	2633 (6)	71 (4)
C(12)	155 (3)	-187 (7)	3899 (5)	82 (4)
C(13)	-1927 (4)	939 (9)	2302 (9)	109 (7)
C(14)	-2255 (6)	404 (15)	1437 (15)	499 (31)
C(15)	-2123 (5)	2049 (12)	2169 (14)	231 (16)
C(16)	-2047 (7)	368 (19)	2934 (15)	582 (41)
B(1)	-1155 (5)	2778 (10)	135 (7)	87 (6)
F(1)	-641 (2)	2385 (4)	945 (3)	86 (2)
F(2)	-1282 (3)	2151 (5)	-648 (4)	155 (4)
F(3)	-1062 (2)	3904 (5)	-5 (3)	124 (4)
F(4)	-1542 (2)	2754 (5)	439 (4)	122 (3)

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

Co—N(1)	2.136 (7)	Co—N(3)	2.093 (7)
Co—F(1)	2.190 (3)	N(1)—N(2)	1.360 (8)
N(1)—C(1)	1.352 (13)	N(2)—C(3)	1.339 (12)
C(1)—C(2)	1.387 (12)	C(1)—C(4)	1.482 (12)
C(2)—C(3)	1.378 (14)	C(3)—C(5)	1.469 (12)
C(5)—C(6)	1.377 (22)	C(5)—C(7)	1.401 (28)
C(5)—C(8)	1.402 (18)	N(3)—N(4)	1.344 (9)
N(3)—C(9)	1.346 (9)	N(4)—C(11)	1.342 (12)
C(9)—C(10)	1.389 (13)	C(9)—C(12)	1.494 (10)
C(10)—C(11)	1.368 (10)	C(11)—C(13)	1.492 (14)
C(13)—C(14)	1.356 (22)	C(13)—C(15)	1.384 (17)
C(13)—C(16)	1.378 (30)	B(1)—F(1)	1.422 (10)
B(1)—F(2)	1.330 (13)	B(1)—F(3)	1.385 (13)
B(1)—F(4)	1.373 (17)		
N(1)—Co—N(3)	89.1 (3)	N(1)—Co—F(1)	89.4 (2)
N(3)—Co—F(1)	87.1 (2)	N(1)—Co—N(1A)	84.7 (4)
N(3)—Co—N(1A)	173.5 (2)	F(1)—Co—N(1A)	90.8 (2)
N(3)—Co—N(3A)	97.1 (4)	F(1)—Co—N(3A)	92.7 (2)
F(1)—Co—F(1A)	179.7 (2)	Co—N(1)—N(2)	119.3 (6)
Co—N(1)—C(1)	136.2 (5)	N(2)—N(1)—C(1)	104.5 (6)
N(1)—N(2)—C(3)	113.6 (7)	N(1)—C(1)—C(2)	109.3 (8)
N(1)—C(1)—C(4)	121.5 (8)	C(2)—C(1)—C(4)	129.3 (10)
C(1)—C(2)—C(3)	108.0 (9)	N(2)—C(3)—C(2)	104.6 (7)
N(2)—C(3)—C(5)	123.3 (10)	C(2)—C(3)—C(5)	132.1 (10)
C(3)—C(5)—C(6)	113.4 (12)	C(3)—C(5)—C(7)	116.5 (12)
C(6)—C(5)—C(7)	103.8 (14)	C(3)—C(5)—C(8)	110.6 (9)
C(6)—C(5)—C(8)	106.2 (16)	C(7)—C(5)—C(8)	105.5 (14)
Co—N(3)—N(4)	117.3 (4)	Co—N(3)—C(9)	135.8 (4)
N(4)—N(3)—C(9)	104.9 (7)	N(3)—N(4)—C(11)	112.8 (6)
N(3)—C(9)—C(10)	109.8 (6)	N(3)—C(9)—C(12)	121.0 (8)
C(10)—C(9)—C(12)	129.2 (7)	C(9)—C(10)—C(11)	106.7 (8)
N(4)—C(11)—C(10)	105.8 (8)	N(4)—C(11)—C(13)	122.2 (7)
C(10)—C(11)—C(13)	132.1 (10)	C(11)—C(13)—C(14)	111.0 (13)
C(11)—C(13)—C(15)	115.9 (10)	C(14)—C(13)—C(15)	105.9 (11)
C(11)—C(13)—C(16)	109.3 (10)	C(14)—C(13)—C(16)	103.4 (14)
C(15)—C(13)—C(16)	110.5 (17)	F(1)—B(1)—F(2)	109.5 (9)
F(1)—B(1)—F(3)	105.9 (8)	F(2)—B(1)—F(3)	111.1 (9)
F(1)—B(1)—F(4)	105.6 (8)	F(2)—B(1)—F(4)	115.8 (9)
F(3)—B(1)—F(4)	108.4 (10)	Co—F(1)—B(1)	151.9 (6)

bisects the angles N1—Co—N1a and N3—Co—C3a. With the exception of N1a and N3a, only the atoms on the crystallographically unique half of the molecule are labelled and will be used to describe the structure. (1) is an octahedral complex with two axially coordinated tetrafluoroborate ligands and four equatorial pyrazole ligands. Selected bond lengths and angles are given in Table 2. The Co—F bond lengths of 2.190 (3) Å in (1) are only slightly longer than the 2.05 (2) and 2.04 (1) Å found in CoF₂ (Stout & Reed, 1954). The Co—F1—B1 bond angle of 151.9 (6)° falls within the wide range (126–180°) observed for monodentate BF₄⁻ ligands. The coordination of the BF₄⁻ ligand results in the expected elongation of the B1—F1 bond to 1.422 Å compared with the average bond length (B—F_{ave} = 1.363 Å) of the non-coordinated F atoms. Bond angles within the BF₄⁻ ligand are unexceptional, ranging from 105.6 (8) to 115.8 (9)°. These data suggest that the tetrafluoroborate anions in (1) can be described as 'normal' (Tomlinson, Bonamico, Dessy, Fares & Scaramuzza, 1972) BF₄⁻ ligands. An alternative description of the complex as a BF₃ adduct of a cobalt fluoride (Burch, Calabrese & Ittel, 1988) appears inappropriate in this case. The remaining bond lengths and angles about Co show only small deviations from an idealized octahedron. The planes of the pyrazole rings are twisted out of the equatorial plane of the octahedron to minimize steric interactions, but these ligands exhibit no unusual bond lengths or angles.

In conclusion, (1) provides another example of the ability of the tetrafluoroborate anion to act as a

ligand to transition metals. Concerning our original intent in carrying out the reaction leading to the formation of (1), we note that the facile hydrolysis of the tris(pyrazolyl)borate ligand in the presence of only small amounts of water may prove a troublesome feature of this increasingly popular class of tridentate ligands.

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Structure of Chlorodiphenyl[3-(*p*-tolylthio)propyl]tin

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Abstract. [Sn(Cl)(C₆H₅)₂(C₁₀H₁₃S)], *M*_r = 473.6, orthorhombic, *P*2₁2₁2₁, *a* = 8.943 (11), *b* =

10.268 (11), *c* = 23.201 (12) Å, *V* = 2130.5 Å³, *Z* = 4, *D*_x = 1.47 Mg m⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 1.96 mm⁻¹, *F*(000) = 952, *T* = 293 K, *R* = 0.0646 for 2405 observed reflexions. The Sn atom is in a dis-

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