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Structure of (η^5-Cyclopentadienyl)(η^6-tetraphenylborato)iron, $[Fe(C_5H_5)\{B(C_6H_5)_4\}]$

By Robin D. Rogers*

Department of Chemistry, Northern Illinois University, DeKalb, IL 60115, *USA*

and Duane C. Hrncir

Gulf Research and Development Company, PO Drawer 2038, *Pittsburgh, PA* 15230, *USA*

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Abstract. $M_r = 440{\cdot}2$, monoclinic, $P2_1/c$, $a = 9{\cdot}537\ (3)$, $b = 15{\cdot}432\ (4)$, $c = 15{\cdot}390\ (4)$ Å, $\beta = 106{\cdot}92\ (3)°$, $V = 2167{\cdot}0$ Å^3, $Z = 4$, $D_x = 1{\cdot}35$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 7{\cdot}3$ cm^{-1}, $F(000) = 920{\cdot}0$, $T = 295$ K, final $R = 0{\cdot}036$ for 1174 observed [$I > 3\sigma(I)$] reflections. The title compound is isostructural with $(\eta^5\text{-}C_5H_5)Ru(\eta^6\text{-}C_6H_5)B(C_6H_5)_3$ [Kruger, duPreez & Haines (1974). *J. Chem. Soc. Dalton Trans.* pp. 1302–1305]. The average Fe—C(η^5-C_5H_5) distance is 2·05 (1) Å, the average Fe—C[(η^6-C_6H_5)B$(C_6H_5)_3$] separation 2·09 (4) Å. The Fe atom is 1·66 Å from the plane defined by the five cyclopentadienyl C atoms, and 1·537 Å from the plane defined by the C atoms of the η^6-bonded phenyl ring.

Introduction. Through the use of ligand-substitution reactions, new organometallic compounds can be prepared without an accompanying change in the oxidation state of the metal. This type of reaction has been used to prepare a series of organoiron compounds having the general formula $(\eta^5\text{-}C_5H_5)Fe(\eta^6\text{-}C_6H_4R)B(C_6H_5)_3$ where R = H, *p*-tolyl, *m*-tolyl, and *p*-methoxy (Slocum *et al.*, 1981; Owen *et al.*, 1980). The compounds were prepared by reacting $(\eta^5\text{-}C_5H_5)Fe(CO)_2I$ with the appropriate borate salt. Preliminary cell constants (Owens *et al.*, 1980) *via* X-ray diffraction showed the title compound to be isostructural with $(\eta^5\text{-}C_5H_5)Ru(\eta^6\text{-}C_6H_5)B(C_6H_5)_3$ (Kruger *et al.*, 1974). In an effort to compare the metal environments in these two structures, a complete single-crystal X-ray diffraction study of the title compound was undertaken.

Experimental. Yellow-orange parallelepipeds grown from dichloromethane at room temperature (Owen *et al.*, 1980). D_m not determined. Crystal 0·30 × 0·45 × 0·55 mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$. Cell constants from setting angles of 15 reflections ($\theta > 10°$). Correction for Lorentz–polarization effect; no absorption correction. $\theta_{max} = 18°$; h 0 to 8, k 0 to 13, l −13 to 13. 1774 reflections measured, 1174 independent observed reflections [$I > 3\sigma(I)$]. H atoms placed at 1·08 Å from the bonded C atom with a fixed isotropic thermal parameter $B = 5{\cdot}5$ Å^2. Scattering factors for non-H atoms from Cromer & Waber (1965), for H from *International Tables for X-ray Crystallography* (1974). Anomalous-dispersion corrections from Cromer & Liberman (1970). Structure solved with *SHELX76* (Sheldrick, 1976). $\sum w(|F_o| - |F_c|)^2$ minimized, unit weights, 280 parameters varied. $R = 0{\cdot}036$, $R_w = 0{\cdot}038$, $S = 1{\cdot}59$. Δ/σ in final least-squares refinement cycle <0·1. No feature greater than 0·3 e Å^{-3} in final difference Fourier map.

Discussion. Fractional coordinates and U_{eq} values are given in Table 1,† selected distances in Table 2.

$(\eta^5\text{-}C_5H_5)Fe(\eta^6\text{-}C_6H_5)B(C_6H_5)_3$ is isostructural with its Ru analogue (Kruger *et al.*, 1974). The Fe atom is sandwiched between the cyclopentadienyl moiety and one of the phenyl rings of the tetraphenylborate group. The bonding of the cyclopentadienyl ring appears normal with average C—C distances and Fe—C separations of 1·41 (1) and 2·05 (1) Å respectively. The Fe atom resides 1·66 Å from the plane defined by the five cyclopentadienyl C atoms. These values compare favorably to the average Fe—C distance of 2·040 Å found for other cyclopentadienyl–iron complexes (Johnson & Treichel, 1977).

As in the isostructural Ru complex, steric interactions lead to a less symmetrical bonding of the Fe atom to the tetraphenylborate moiety. The Fe—C(41) (the C atom bonded to B) distance is 2·156 (6) Å, which is significantly longer than the average of the other five Fe—C separations [2·07 (1) Å]. A slight lengthening of the C(41)—C(46), C(41)—C(42), and C(45)—C(46) bond lengths [average 1·429 (9) Å] over the other three

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* To whom correspondence should be addressed.

† Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, best-planes results, bond distances and angles, and a figure of the molecular structure and a stereoview of the unit-cell packing have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39351 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters for non-*H *atoms*

$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	*y*	*z*	U_{eq}(Å^2)
Fe	0·2604 (1)	0·23211 (6)	0·42096 (7)	0·048
B	0·6478 (8)	0·1967 (5)	0·4816 (5)	0·035
C(11)	0·6388 (6)	0·1535 (5)	0·3837 (4)	0·038
C(12)	0·6773 (7)	0·1989 (5)	0·3150 (6)	0·049
C(13)	0·6778 (8)	0·1607 (6)	0·2337 (6)	0·060
C(14)	0·6410 (8)	0·0758 (7)	0·2156 (5)	0·059
C(15)	0·6028 (8)	0·0277 (5)	0·2810 (6)	0·062
C(16)	0·6036 (7)	0·0660 (5)	0·3624 (5)	0·052
C(21)	0·7203 (8)	0·2929 (4)	0·4930 (5)	0·039
C(22)	0·8530 (8)	0·3138 (5)	0·5573 (5)	0·042
C(23)	0·9104 (7)	0·3964 (6)	0·5680 (5)	0·051
C(24)	0·837 (1)	0·4646 (5)	0·5149 (6)	0·062
C(25)	0·7059 (9)	0·4469 (5)	0·4504 (5)	0·055
C(26)	0·6520 (8)	0·3635 (6)	0·4402 (5)	0·050
C(31)	0·7447 (7)	0·1314 (4)	0·5621 (5)	0·037
C(32)	0·7136 (7)	0·1133 (5)	0·6429 (5)	0·046
C(33)	0·806 (1)	0·0621 (5)	0·7109 (5)	0·051
C(34)	0·9352 (9)	0·0314 (5)	0·6996 (5)	0·048
C(35)	0·9702 (7)	0·0476 (5)	0·6213 (6)	0·051
C(36)	0·8750 (8)	0·0978 (5)	0·5529 (4)	0·040
C(41)	0·4826 (7)	0·2024 (5)	0·4982 (4)	0·035
C(42)	0·3858 (8)	0·1295 (4)	0·4860 (4)	0·040
C(43)	0·2530 (8)	0·1304 (5)	0·5065 (5)	0·053
C(44)	0·2065 (8)	0·2066 (7)	0·5395 (5)	0·069
C(45)	0·2935 (9)	0·2810 (5)	0·5514 (5)	0·061
C(46)	0·4297 (7)	0·2786 (5)	0·5302 (4)	0·042
C(51)	0·201 (1)	0·1902 (6)	0·2894 (5)	0·071
C(52)	0·073 (1)	0·2084 (9)	0·3155 (7)	0·086
C(53)	0·082 (1)	0·2972 (8)	0·3406 (7)	0·083
C(54)	0·211 (1)	0·3318 (6)	0·3306 (7)	0·075
C(55)	0·2847 (9)	0·2667 (9)	0·2979 (5)	0·073

C—C distances [1·40 (1) Å] is also found. The C—C bond distances average 1·39 (1) Å in the other three phenyl rings.

The Fe atom is 1·537 Å from the plane defined by atoms C(41)–C(46). The angle between the planes defined by the cyclopentadienyl and phenyl rings is 2·28°. Both rings are planar to within 0·013 Å.

Table 2. *Selected interatomic distances* (Å)

Fe—C(41)	2·156 (6)	Fe—C(51)	2·042 (8)
Fe—C(42)	2·060 (6)	Fe—C(52)	2·064 (8)
Fe—C(43)	2·062 (7)	Fe—C(53)	2·050 (8)
Fe—C(44)	2·072 (7)	Fe—C(54)	2·035 (8)
Fe—C(45)	2·081 (7)	Fe—C(55)	2·045 (8)
Fe—C(46)	2·090 (6)		
Fe—Cent4*	1·537	Fe—Cent5	1·660
B—C(11)	1·63 (1)	B—C(21)	1·63 (1)
B—C(31)	1·65 (1)	B—C(41)	1·67 (1)

* Cent4 is the centroid of the plane defined by atoms C(41)–C(46), Cent5 by atoms C(51)–C(55).

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References

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104–109.

International Tables for X-ray Crystallography (1974). Vol. IV, p. 72. Birmingham: Kynoch Press.

JOHNSON, J. W. & TREICHEL, J. W. (1977). *J. Am. Chem. Soc.* **99**, 1427–1436.

KRUGER, G. J., DU PREEZ, A. L. & HAINES, R. J. (1974). *J. Chem. Soc. Dalton Trans.* pp. 1302–1305.

OWEN, D. A., SIEGEL, A., LIN, R., SLOCUM, D. W., CONWAY, B., MORONSKI, M. & DURAJ, S. (1980). *Ann. NY Acad. Sci.* pp. 90–100.

SHELDRICK, G. M. (1976). *SHELX76*. A system of computer programs for X-ray structure determination. Univ. of Cambridge, England.

SLOCUM, D. W., CONWAY, B., HODGMAN, M., KUCHEL, K., MORONSKI, M., NOBLE, R., WEBBER, K., DURAJ, S., SIEGEL, A. & OWEN, D. A. (1981). *J. Macromol. Sci. Chem.* **16**, 357–368.

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Tris(2,6-dimethyl-4*H*-pyran-4-one)dinitratocadmium(II), $[Cd(NO_3)_2(C_7H_8O_2)_3]$

BY A. BANERJEE AND C. J. BROWN

*Department of Metallurgy and Materials Engineering, City of London Polytechnic, Central House, Whitechapel High Street, London E*1 7*PF, England*

AND P. C. JAIN AND P. GAUTAM

Department of Chemistry, University of Kurukshetra, Kurukshetra, Haryana 132 119, *India*

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Abstract. $M_r = 608·8$, monoclinic, $P2_1/n$, $a = 19·372$ (5), $b = 11·345$ (4), $c = 23·023$ (5) Å, $\beta = 93·77$ (5)°, $V = 5048·9$ Å^3, $Z = 8$, $D_m = 1·60$ (1), $D_x = 1·602$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 0·93$ mm^{-1}, $F(000) = 2464$, $T = 293$ K, $R = 0·061$ for 7325 observed reflexions ($I > \sigma I$). The structure comprises two discrete non-equivalent molecules in each of which cadmium is seven-coordinated, to three oxygen atoms of the dimethyl-4-pyrone ligands, and bidentately to four oxygen atoms of two nitrato groups. The pyrone rings are planar and there are no strong intermolecular interactions.

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