

Structure of Dicarbonyliodo(η^5 -pentamethylcyclopentadienyl)iridium(III) Tetrafluoroborate [Cp*Ir(CO)₂I]BF₄

BY FREDERICK W. B. EINSTEIN, XIAOQIAN YAN AND DEREK SUTTON

Department of Chemistry, Simon Fraser University, Burnaby, British Columbia, Canada V5A 1S6

(Received 19 November 1990; accepted 19 March 1991)

Abstract. [Ir(C₁₀H₁₅)(I)(CO)₂][BF₄], $M_r = 597.17$, orthorhombic, $Pca2_1$, $a = 13.035$ (4), $b = 10.517$ (3), $c = 12.013$ (4) Å, $V = 1646.9$ Å³, $Z = 4$, $D_x = 2.41$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 99.74$ cm⁻¹, $F(000) = 1095.75$, $T = 296$ K, $R = 0.044$, $wR = 0.049$ for 1014 observed reflections. The Ir atom in the cation is coordinated to an η^5 -pentamethylcyclopentadienyl group, two carbonyl groups and an iodine. Selected bond distances and angles are: Ir—C₅Me₅ centroid 1.88, Ir—C(1) 1.89 (3), Ir—C(2) 1.81 (4), Ir—I 2.717 (3) Å; C(1)—Ir—C₅Me₅ centroid 126, C(2)—Ir—C₅Me₅ centroid 132, I—Ir—C₅Me₅ centroid 120, C(1)—Ir—C(2) 92 (2), C(1)—Ir—I 90 (2), C(2)—Ir—I 84 (1)°.

Experimental. Compound isolated from reaction of Cp*Re(CO)₂I₂ and Cp*Ir(CO)₂ (Cp* = η^5 -C₅Me₅) with AgBF₄ in ethanol; crystals obtained from CH₂Cl₂–hexane (Zhang & Sutton, 1990). A pale-green crystal 0.39 × 0.24 × 0.12 mm was mounted on a glass fibre. Intensity data were collected at 296 (1) K with an Enraf–Nonius CAD-4F diffractometer using graphite-monochromatized Mo $K\alpha$

radiation. Lattice parameters were determined from 25 reflections ($15 \leq \theta \leq 18^\circ$). 1678 independent reflections were measured ($3 \leq 2\theta \leq 50^\circ$; $h: 0 \rightarrow 15$; $k: 0 \rightarrow 12$; $l: 0 \rightarrow 14$) using ω - 2θ scans; 1014 observed reflections [$I_o \geq 2.5\sigma(I_o)$]; scan range ($1.10 + 0.35\tan\theta$)°; scan speed 0.19–5.49° min⁻¹; intensities of three standards (042, 121, 211) were measured every 80 min of acquisition time and showed no long term change and had an r.m.s. deviation of < 1.4%; data reduction included intensity scaling, Lorentz and polarization corrections and an analytical absorption correction, ψ -scan checked, transmission 0.240–0.545 (Alcock, 1969). Structure solved by Patterson synthesis, and refined by full-matrix least squares and Fourier synthesis methods. The assumption that the space group is non-centrosymmetric $Pca2_1$ was confirmed by the successful refinement. The positions of some methyl-group H atoms were located in difference Fourier maps ($\sin\theta/\lambda < 0.3$ Å⁻¹) and were used to calculate remaining methyl H-atom positions [$d(\text{C}—\text{H}) = 0.95$ Å]. Final refinement included anisotropic thermal parameters for Ir and I, and isotropic thermal parameters for

Table 1. Positional parameters and $B_{\text{iso}}/B_{\text{eq}}$ values (Å²) for [Cp*Ir(CO)₂I]BF₄

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{iso}}/B_{\text{eq}}^\dagger$
Ir	0.03319 (7)	0.12921 (9)	0	2.59
I	0.1188 (3)	0.0189 (3)	0.1822 (3)	6.41
C(1)	-0.0778 (27)	0.0139 (35)	0.009 (6)	6.1 (8)
O(1)	-0.1450 (20)	-0.049 (3)	0.024 (3)	7.3 (7)
C(2)	0.1107 (32)	0.0155 (42)	-0.076 (3)	5.5 (9)
O(2)	0.1582 (24)	-0.040 (3)	-0.138 (3)	7.4 (8)
C(11)	-0.0471 (26)	0.3055 (32)	0.056 (3)	2.8 (7)
C(12)	0.0537 (28)	0.3198 (36)	0.075 (3)	4.1 (8)
C(13)	0.1162 (20)	0.3131 (24)	-0.015 (3)	3.1 (6)
C(14)	0.0507 (24)	0.3033 (30)	-0.105 (3)	2.6 (6)
C(15)	-0.0557 (32)	0.2929 (40)	-0.060 (4)	2.7 (10)
C(21)	-0.1388 (38)	0.3207 (42)	0.125 (4)	6.0 (11)
C(22)	0.0987 (26)	0.3523 (31)	0.198 (3)	4.7 (7)
C(23)	0.2321 (24)	0.3356 (27)	-0.020 (3)	4.4 (7)
C(24)	0.0765 (26)	0.3000 (34)	-0.226 (3)	4.6 (7)
C(25)	-0.1483 (31)	0.2948 (35)	-0.133 (3)	3.2 (8)
F(1)	0.4618 (16)	0.4148 (18)	1.0242 (26)	6.7 (5)
F(2)	0.5226 (20)	0.2298 (24)	0.9615 (20)	7.8 (7)
F(3)	0.6156 (25)	0.3548 (29)	1.0728 (27)	9.1 (8)
F(4)	0.5956 (20)	0.4077 (24)	0.9024 (22)	2.9 (7)
B	0.5439 (24)	0.347 (3)	0.986 (3)	2.9 (7)

[†] B_{eq} is the mean of the principal axes of the thermal ellipsoid.

Table 2. Selected bond lengths (Å) and bond angles (°) for [Cp*Ir(CO)₂I]BF₄

Ir—I	2.717 (3)	C(11)—C(12)	1.34 (5)
Ir—C(1)	1.89 (3)	C(12)—C(13)	1.35 (5)
Ir—C(2)	1.81 (4)	C(13)—C(14)	1.39 (4)
Ir—C ₅ Me ₅ centroid	1.88	C(14)—C(15)	1.49 (5)
Ir—C(11)	2.23 (3)	C(15)—C(11)	1.41 (6)
Ir—C(12)	2.21 (4)	C(11)—C(21)	1.46 (6)
Ir—C(13)	2.22 (3)	C(12)—C(22)	1.63 (5)
Ir—C(14)	2.23 (3)	C(13)—C(23)	1.53 (4)
Ir—C(15)	2.20 (4)	C(14)—C(24)	1.49 (5)
C(1)—O(1)	1.11 (5)	C(15)—C(25)	1.49 (6)
C(2)—O(2)	1.14 (5)		
I—Ir—C(1)	90 (2)	C(15)—C(14)—C(13)	107 (3)
I—Ir—C(2)	84 (1)	C(12)—C(11)—C(21)	134 (4)
C(1)—Ir—C(2)	92 (2)	C(15)—C(11)—C(21)	120 (3)
Ir—C(1)—O(1)	173 (5)	C(11)—C(12)—C(22)	122 (3)
Ir—C(2)—O(2)	168 (4)	C(13)—C(12)—C(22)	121 (3)
C(1)—Ir—C ₅ Me ₅ centroid	126	C(12)—C(13)—C(23)	128 (3)
C(2)—Ir—C ₅ Me ₅ centroid	132	C(14)—C(13)—C(23)	126 (3)
I—Ir—C ₅ Me ₅ centroid	120	C(13)—C(14)—C(24)	129 (3)
C(11)—C(15)—C(14)	106 (3)	C(15)—C(14)—C(24)	124 (3)
C(12)—C(11)—C(15)	105 (3)	C(14)—C(15)—C(25)	123 (4)
C(13)—C(12)—C(11)	117 (3)	C(11)—C(15)—C(25)	130 (4)
C(14)—C(13)—C(12)	105 (3)		

other non-H atoms. H atoms were included in fixed positions in structure factor calculations. Refinement was considered complete when the shift/e.s.d. ratio was less than 0.02; 94 parameters varied. Final residuals $R = 0.044$, $wR = 0.049$; goodness of fit 1.004; quantity minimized $\sum w(F_o - F_c)^2$, where $w = 1$. Highest peak in final electron density map was 3.4 (2) e Å⁻³ at a distance 1.01 Å from Ir atom and ρ_{\min} was -1.3 (2) e Å⁻³. Analytical forms of scattering factors for neutral atoms used (*International Tables for X-ray Crystallography*, 1974, Vol. IV, Tables 2.2B and 2.3.1.); all non-H-atom scattering factors corrected for real and imaginary components of anomalous dispersion. Inspection of $w\Delta^2$ as a function of $\sin\theta/\lambda$, F_o and values of h , k , l showed no unusual features or trends. Positional parameters are listed in Table 1, and bond distances and angles are listed in Table 2.† Programs used were from the *NRC VAX Crystal Structure System* (Gabe, Le Page, Charland, Lee & White, 1989) including their version of *ORTEP* (Johnson, 1965), and were run on a MicroVAX II computer. Fig. 1. shows the cation with the atom-labelling scheme.

Related literature. The structures of the related compounds [Cp*Ir(PMe₃)₂Cl][PF₆] (Kaner, Kouvetakis & Mayorga, 1986) and [Cp*Ir(CO)₂(CH₂Cl)]-[Re₂(CO)₆(μ-Cl)_x(μ-Br_{3-x})] (Einstein, Glavina, Pomeroy & Willis, 1986) have been reported.

† 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 54100 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

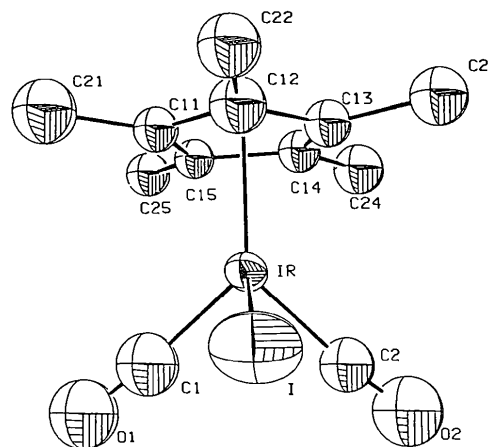


Fig. 1. View of the cation [Cp*Ir(CO)₂I]⁺ with atom labelling.

This work was supported by NSERC Canada through operating and infrastructure grants to FWBE and DS. We thank Johnson Matthey Co. for the generous loan of iridium trichloride.

References

- ALCOCK, N. W. (1969). In *Crystallographic Computing*, edited by F. R. AHMED, p. 271. Copenhagen: Munksgaard.
- EINSTEIN, F. W. B., GLAVINA, P. G., POMEROY, R. K. & WILLIS, A. C. (1986). *J. Organomet. Chem.* **317**, 255–265.
- GABE, E. J., LE PAGE, Y., CHARLAND, J.-P., LEE, F. L. & WHITE, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- KANER, R. B., KOUVETAKIS, J. & MAYORGA, S. G. (1986). *Acta Cryst.* **C42**, 500–501.
- ZHANG, X. & SUTTON, D. (1990). Unpublished results.

Acta Cryst. (1991). **C47**, 1978–1980

Structure of *N*-Ethyl-*m*-fluorophenylsuccinimide

BY WITOLD KWIATKOWSKI* AND JANINA KAROLAK-WOJCIECHOWSKA

Institute of General Chemistry, Technical University, 90-924 Łódź, Zwirki 36, Poland

(Received 31 July 1990; accepted 15 January 1991)

Abstract. 1-Ethyl-3-(3-fluorophenyl)-2,5-pyrrolidinedione, C₁₂H₁₂FNO₂, $M_r = 221.23$, monoclinic, $P2_1/c$, $a = 6.623$ (1), $b = 10.810$ (2), $c = 15.297$ (2) Å, $\beta = 96.82$ (1)°, $V = 1087.6$ (3) Å³, $Z = 4$, $D_x = 1.351$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 0.84$ mm⁻¹, $F(000) = 464$, room temperature, final $R = 0.052$ for 1577 observed reflections (of 1969 unique data). The succinimide fragment of the molecule is non-planar and has an envelope

conformation [the deviation of C1 from the plane N1–C4–C2–C3 is -0.096 (2) Å and the dihedral angle between planes C4–C1–C2 and N1–C4–C2–C3 is 5.8 (2)°]. The phenyl ring is planar. The angle between the best planes of the five-membered ring and the phenyl ring is 87.1 (1)°.

Experimental. The title compound recrystallized from ethanol and gave colourless crystals. Crystal