Table 1. *Fractional coordinates and isotropic or equivalent isotropic thermal parameters*

The thermal parameters are of the form $B = 8\pi^2U$ and $B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B/B_{eq}(Å^2)
Ru	0·000	0·000	0·000	0·987 (4)
S	−0·13953 (7)	0·26052 (6)	0·06300 (4)	1·253 (7)
O(1)	−0·3144 (3)	0·3833 (2)	−0·0575 (1)	2·03 (2)
O(2)	0·0505 (3)	0·3998 (2)	0·1383 (1)	1·89 (3)
O(3)	−0·2957 (2)	0·2198 (2)	0·1256 (1)	2·07 (3)
N(1)	0·3121 (4)	0·0071 (2)	0·1751 (2)	1·77 (4)
N(2)	0·1701 (3)	0·1817 (3)	−0·0834 (1)	1·54 (3)
H	−0·238 (5)	0·435 (5)	−0·082 (3)	3·6 (7)*
H(11)	0·300 (6)	0·101 (6)	0·204 (3)	6 (1)*
H(12)	0·311 (5)	−0·073 (5)	0·219 (3)	4·2 (8)*
H(13)	0·430 (6)	−0·010 (3)	0·159 (3)	2·8 (8)*
H(21)	0·184 (5)	0·126 (5)	−0·146 (3)	4·1 (8)*
H(22)	0·303 (5)	0·199 (5)	−0·032 (3)	4·6 (8)*
H(23)	0·099 (5)	0·284 (4)	−0·105 (2)	3·7 (7)*

* Refined isotropically.

Table 2. *Selected bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Ru—S	2·2785 (5)	S—O(2)	1·496 (1)
Ru—N(1)	2·141 (2)	S—O(3)	1·474 (2)
Ru—N(2)	2·142 (2)	O(1)—H	0·75 (4)
S—O(1)	1·617 (1)	N—H	0·81 (ave)
		N—H	0·76–0·87 (range)
S—Ru—N(1)	88·93 (6)	O(1)—S—O(3)	101·74 (8)
S—Ru—N(2)	90·03 (5)	O(2)—S—O(3)	110·54 (8)
N(1)—Ru—N(2)	90·39 (7)	S—O(1)—H	107 (3)
Ru—S—O(1)	110·41 (6)	Ru—N—H	108 (mean)
Ru—S—O(2)	113·87 (6)	Ru—N—H	103–111 (range)
Ru—S—O(3)	115·39 (6)	H—N—H	110 (mean)
O(1)—S—O(2)	103·53 (8)	H—N—H	105–119 (range)

tions from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*. Table 1 gives the atomic coordinates and Table 2 gives selected bond distances and angles.* Fig. 1 shows the complex with the numbering scheme.

Related literature. Previous structural reports of complexes containing the bisulfite ion as a ligand were of [Ru(bpy)$_2$(py)(HSO$_3$)]PF$_6$ and Ru(bpy)$_2$(HSO$_3$)$_2$ (Allen, Jeter, Cordes & Durham, 1988) and Na$_4$[Ru(SO$_3$)$_2$(HSO$_3$)$_2$(NH$_3$)$_2$] (Johnson, Jeter & Cordes, 1987). The related structural aspects of SO$_2$ as a ligand have been reviewed by Ryan, Kubas, Moody & Eller (1981).

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Structure of *cis*-Tetrachlorobis(tetrahydrofuran)hafnium(IV)

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Abstract. [HfCl$_4$(C$_4$H$_8$O)$_2$], $M_r = 464{\cdot}52$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}914$ (2), $b = 12{\cdot}806$ (3), $c = 14{\cdot}357$ (3) Å, $V = 1455$ (1) Å^3, $Z = 4$, $D_x = 2{\cdot}120$ g cm^{-3}, Mo $K\alpha$ ($\lambda = 0{\cdot}71073$ Å), $\mu = 78{\cdot}3$ cm^{-1}, $F(000) = 220$, $T = 298$ K, $R = 0{\cdot}0359$ for 1494 reflections with $I \geq 3\sigma(I)$ and 137 variables. The HfCl$_4$(THF)$_2$ compound has a distorted *cis* octahedral stereochemistry. The bond angles about the Hf atom are consistent with this structure type. The important bond distances are: Hf—Cl 2·363 (4), 2·377 (4) Å (*trans* to THF's), 2·401 (3), 2·388 (4) Å (*cis* to THF's) and Hf—O 2·197 (8), 2·194 (8) Å.

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Experimental. All operations were performed under an inert atmosphere employing standard Schlenk techniques and a double manifold vacuum line. Solids were manipulated in a Vacuum Atmospheres Co. glovebox equipped with an HE-493 dri-train. THF was freshly distilled from benzophenone ketyl, and CH_2Cl_2 was freshly distilled from P_2O_5 prior to use. Solutions were transferred *via* stainless steel cannulae and/or syringes. The air-sensitive $HfCl_4(THF)_2$ complex was prepared according to the literature method (Manzer, 1982) by reacting $HfCl_4$ with THF in CH_2Cl_2. Single crystals were grown by dissolving $HfCl_4(THF)_2$ in THF, filtering the solution through Celite into a Schlenk tube, and then placing the filtrate in a cold room at 278 K for several days.

Colorless block-like crystals (0·4 × 0·4 × 0·3 mm) were sealed with epoxy cement in a glass capillary at room temperature and mounted on an Enraf–Nonius CAD-4 X-ray diffractometer equipped with a graphite monochromator (Mo $K\alpha$ radiation, λ = 0·71073 Å). Cell parameters were determined from accurately measured goniometer settings of 25 reflections, $9 < 2\theta < 36°$. Systematically absent reflections ($h00$ for h odd, $0k0$ for k odd, and $00l$ for l odd) uniquely determine the space group. 2020 reflections measured, 1922 unique, 1494 of which were considered observed [$I > 3\sigma(I)$] using (ω–2θ) scans over index ranges of $0 \le h \le 10$, $0 \le k \le 16$, $0 \le l \le 18$. Three intensity controls were measured at 1 h intervals and showed 11·1% decay over 23·6 h exposure. An empirical absorption correction was made using ψ scans for nine reflections with χ near 90°; reflections were measured at 10° intervals from 0 to 360°. The minimum and maximum relative absorption corrections were 1·00 and 1·17. The structure was solved using the Patterson function. All non-H atoms were located and refined by a series of least-squares refinements on F and difference Fourier syntheses. The H atoms were not located. $F_{\rm obs}$ were corrected for Lorentz, polarization, absorption and decay effects. The final refinement parameters are: R = 0·0359, wR = 0·0455, $w = 1/\sigma^2(F_o)$, S = 1·232, $(\Delta\sigma)_{\rm max}$ = 0·01. All peaks in the final difference Fourier map were below 0·75 e Å^{-3}. The most negative peak was −0·66 e Å^{-3}. A secondary-extinction correction of the form $F_c = F_c/(1 + gI_c)$ was applied. The refined extinction coefficient, g, was 3·27 × 10^{-7}. Both enantiomorphs were refined under identical conditions and the one giving lower residuals is reported here (the other enantiomorph gave R = 0·0478, wR = 0·0633, S = 1·717). Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed with the Enraf–Nonius (1983) *SDP PLUS* on a PDP-11 computer in the Department of Chemistry at Cleveland State University.

Table 1. *Positional and equivalent isotropic displacement parameters and their e.s.d.'s for* [$HfCl_4(THF)_2$]

$B_{\rm eq} = (1/3)\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$B_{\rm eq}$(Å^2)
Hf	0·90793 (7)	0·98788 (3)	0·90469 (3)	3·416 (7)
Cl1	1·1545 (5)	0·9440 (4)	0·8205 (3)	5·87 (9)
Cl2	0·8148 (5)	1·1139 (3)	0·7921 (2)	4·75 (7)
Cl3	0·9565 (6)	0·8523 (3)	1·0157 (2)	6·11 (9)
Cl4	1·0251 (6)	1·1199 (3)	1·0014 (3)	6·67 (9)
O1	0·663 (1)	1·0102 (6)	0·9744 (5)	4·3 (2)
O2	0·760 (1)	0·8739 (6)	0·8246 (5)	3·7 (2)
C11	0·584 (2)	1·114 (1)	0·9887 (9)	5·7 (3)
C12	0·475 (3)	1·104 (1)	1·067 (1)	8·1 (4)
C13	0·425 (2)	0·983 (1)	1·071 (1)	6·0 (3)
C14	0·567 (2)	0·930 (1)	1·024 (1)	7·5 (4)
C21	0·601 (2)	0·899 (1)	0·7715 (9)	5·5 (3)
C22	0·528 (2)	0·792 (1)	0·752 (1)	5·6 (3)
C23	0·676 (2)	0·724 (1)	0·740 (1)	6·2 (4)
C24	0·803 (2)	0·7620 (9)	0·814 (1)	5·3 (3)

Table 2. *Important bond distances* (Å) *and angles* (°) *for* [$HfCl_4(THF)_2$] *with e.s.d.'s in parentheses*

Hf—Cl(1)	2·363 (4)	Hf—O(2)	2·194 (8)
Hf—Cl(2)	2·401 (3)	O(1)—C(11)	1·48 (2)
Hf—Cl(3)	2·388 (4)	O(1)—C(14)	1·47 (2)
Hf—Cl(4)	2·377 (4)	O(2)—C(21)	1·51 (2)
Hf—O(1)	2·197 (8)	O(2)—C(24)	1·482 (14)

Cl(1)—Hf—Cl(2)	94·0 (1)	Cl(3)—Hf—O(1)	86·2 (2)
Cl(1)—Hf—Cl(3)	92·0 (2)	Cl(3)—Hf—O(2)	87·2 (2)
Cl(1)—Hf—Cl(4)	98·4 (2)	Cl(4)—Hf—O(1)	89·1 (2)
Cl(1)—Hf—O(1)	172·3 (2)	Cl(4)—Hf—O(2)	170·8 (2)
Cl(1)—Hf—O(2)	90·8 (2)	O(1)—Hf—O(2)	81·7 (3)
Cl(2)—Hf—Cl(3)	171·1 (2)	Hf—O(1)—C(11)	123·6 (8)
Cl(2)—Hf—Cl(4)	92·0 (1)	Hf—O(1)—C(14)	126·0 (8)
Cl(2)—Hf—O(1)	87·1 (2)	Hf—O(2)—C(21)	124·4 (7)
Cl(2)—Hf—O(2)	86·0 (2)	Hf—O(2)—C(24)	125·2 (8)
Cl(3)—Hf—Cl(4)	93·7 (1)		

Final positional parameters and equivalent isotropic displacement parameters are listed in Table 1. Important bond lengths and angles are given in Table 2.* The stereochemistry of the molecule is illustrated by the *ORTEP* (Johnson, 1976) drawing shown in Fig. 1 which also defines the atomic labeling scheme.

Related literature. A reaction of $HfCl_4(NMe_3)_2$ with THF in benzene gives *trans*-$HfCl(THF)_2$ as shown by IR spectroscopy (Wade & Willey, 1979). X-ray structures of other THF adducts of the early transition metals have been reported: *mer*-$M$$Cl_3(THF)_3$, M = Sc (Atwood & Smith, 1974), Ti (Handlowic, Miklos & Zikmund, 1981), V and Cr (Cotton, Duraj, Powell & Roth, 1986). The structure of the cation $[V_2(\mu\text{-}Cl)_3(THF)_6]^+$ has also been determined recently (Cotton, Duraj, Manzer & Roth, 1985).

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* Lists of structure factors, complete bond distances and angles, and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52521 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

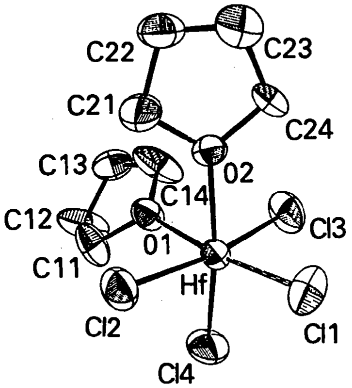

Fig. 1. *ORTEP* (Johnson, 1976) drawing of the [*cis*-$HfCl_4(THF)_2$] molecule. Thermal ellipsoids are drawn at the 40% probability level.

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Structure Determination of Bromotricarbonyl(η-cyclopentadienyl)molybdenum(II)

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Abstract. [$(\eta\text{-}C_5H_5)Mo(CO)_3Br$], $M_r = 324.97$, monoclinic, $P2_1/n$, $a = 8.186\,(3)$, $b = 10.536\,(3)$, $c = 11.391\,(3)$ Å, $\beta = 99.58\,(2)°$, $V = 968.7\,(6)$ Å^3, $Z = 4$, $D_x = 2.228$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 56.68$ cm^{-1}, $F(000) = 616$, $T = 113\,(1)$ K, final $R = 0.0261$ for all 1271 independent reflections. The molecule adopts a distorted square pyramidal coordination environment about the Mo atom. The shorter Mo—C bond of the carbonyl ligand *trans* to the bromide apparently arises from the lack of competition for $d\pi$ bonding electrons. The longer Mo—C bonds for the mutually *trans* carbonyl ligands, and the squashed *trans* C—Mo—C angle (approximately 28° smaller than the Br—Mo—C_{trans} angle) result from sharing one pair of $d\pi$ bonding electrons. Selected bond distances and angles: Mo—Br = 2.651 (1), av. Mo—C_{cis} = 2.020 (6), Mo—C_{trans} = 1.978 (7), Mo—CP_{cent} = 1.993 (1), av. $(C—O)_{cis}$ = 1.132 (5), $(C—O)_{trans}$ = 1.154 (9), Br—Mo—C_{trans} = 136.7 (2), Br—Mo—C_{cis} = 77.0 (3), Br—Mo—CP_{cent} = 112.04 (3), av. C_{trans}—Mo—C_{cis} = 78.1 (2), av. C_{cis}—Mo—CP_{cent} = 125.8 (2), C_{trans}—Mo—CP_{cent} = 111.3 (2).

Experimental. Bromotricarbonyl(η-cyclopentadienyl)molybdenum(II) was isolated as a byproduct from the bromination of bis[(η-cyclopentadienyl)dicarbonylmolybdenum(II)]. Red-brown crystals were grown from cold toluene (250 K).

A crystal of dimensions 0.3 × 0.3 × 0.1 mm was mounted in air on a glass fiber. Lattice parameters were determined by a least-squares fitting of 15 reflections ($15 < 2\theta < 30°$). Experiments were carried out on a Syntex $P2_1$ diffractometer equipped with an LT-1 low-temperature system under the following conditions: graphite-monochromated Mo $K\bar{\alpha}$ radiation, θ–2θ scan mode, scan speed 1.0 to 20° min^{-1}, scan width (1.2° + dispersion), background one-sixth of the total scan at both edges, $4.0 < 2\theta < 45°$; 1534 reflections were measured, 1271 unique ($R_{int} = 0.028$) reflections were obtained, systematic absences were found at $k = 2n + 1$ in $0k0$, $l = 2n + 1$ in $h0l$. I_o

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