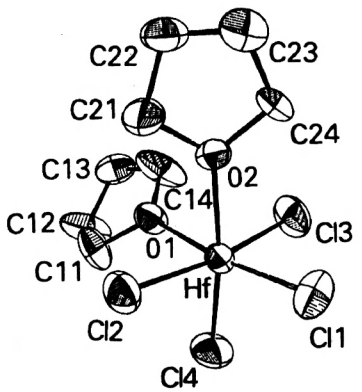

Fig. 1. *ORTEP* (Johnson, 1976) drawing of the [*cis*-$HfCl_4(THF)_2$] molecule. Thermal ellipsoids are drawn at the 40% probability level.

Acknowledgement is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society for support of this research. We also thank Cleveland State University for the Research Creative Activity Award and the Research Challenge grant, approved by the Ohio Board of Regents for partial support of this work.

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

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(*Received* 27 *June* 1989; *accepted* 12 *December* 1989)

Abstract. [(η-C_5H_5)Mo(CO)$_3$Br], $M_r = 324{\cdot}97$, monoclinic, $P2_1/n$, $a = 8{\cdot}186$ (3), $b = 10{\cdot}536$ (3), $c = 11{\cdot}391$ (3) Å, $\beta = 99{\cdot}58$ (2)°, $V = 968{\cdot}7$ (6) Å^3, $Z = 4$, $D_x = 2{\cdot}228$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 56{\cdot}68$ cm^{-1}, $F(000) = 616$, $T = 113$ (1) K, final $R = 0{\cdot}0261$ 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, $4{\cdot}0 < 2\theta < 45°$; 1534 reflections were measured, 1271 unique ($R_{int} = 0{\cdot}028$) reflections were obtained, systematic absences were found at $k = 2n + 1$ in $0k0$, $l = 2n + 1$ in $h0l$. I_o

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0108-2701/90/050892-02$03.00

B_{eq}*

$$*B_{eq} = \tfrac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \mathbf{a}_j.$$

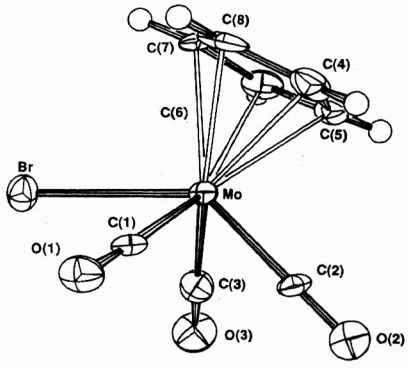

Fig. 1. Molecular structure of $(\eta\text{-}C_5H_5)Mo(CO)_3Br$.