SHORT-FORMAT PAPERS

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Structure of Bromopentacarbonylrhenium(I)

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Abstract. [Re(CO)$_5$Br], $M_r = 406$, orthorhombic, *Pnma*, $a = 11.886$ (2), $b = 11.644$ (2), $c = 6.1888$ (10) Å, $V = 856.5$ (2) Å^3, $Z = 4$, $D_x = 3.150$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 18.982$ mm^{-1}, $F(000) = 720$, $T = 298$ K, $R = 0.0483$ and $wR = 0.0682$ for 61 variable parameters and 527 reflections with $F > 2\sigma(F)$. The Re atoms possess octahedral coordination geometry. Bonds to the carbonyl groups range from 1.889 (5) to 1.991 (6) Å and the Re—Br bond measures 2.619 (2) Å. A layered type of packing motif results from the organization of discrete Re octahedra which form planes having a stacking direction perpendicular to the crystallographic *b* axis.

Experimental. A small regularly shaped pale yellow crystal of approximate dimensions 0.20 × 0.20 × 0.30 mm was selected and mounted on the end of a glass fiber in a random orientation. Orthorhombic symmetry suggested on the basis of a Delaunay reduction and axial rotation photographs which all displayed *m* symmetry. Refined cell parameters obtained from the setting angles of 25 reflections with $30 < 2\theta < 35°$. Data collection carried out at ambient temperature on a Nicolet *R3m/E* diffractometer (graphite-monochromated Mo $K\alpha$ radiation) using the ω-scanning technique in bisecting geometry. Scan rate variable, 4–20° min^{-1}; scan range 1.8° in ω. Intensities measured for 583 reflections ($+h$, $+k$, $+l$; $h_{\max} = 12$, $k_{\max} = 12$, $l_{\max} = 6$) with $0 < 2\theta < 45°$ [$(\sin\theta/\lambda_{\max}) = 0.538$ Å^{-1}]. Three standards ($1\bar{1}2$, $\bar{1}31$, $0\bar{1}1$) measured every 100 data showed no significant variation over the period of data collection. The data were corrected for absorption, Lorentz and polarization effects. Empirical absorption corrections applied on the basis of azimuthal scans of seven strong reflections spanning a range of 2θ values (minimum and maximum transmission factors, 0.047 and 0.167, respectively). Structure solution carried out using the *SHELXTL* collection of crystallographic software (Sheldrick, 1978) and refined using the *SHELXTL-PC* crystallographic software package (Sheldrick, 1990). Re-atom position determined from a sharpened Patterson map; remaining atoms located on difference Fourier maps. All atoms were refined with anisotropic temperature factors. Scattering factors, including terms for anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Refinement based on F using weights of the form $w^{-1} = [\sigma^2(F) + 0.0037(F^2)]$. Convergence to conventional R values of $R = 0.0483$ and $wR = 0.0682$ with a goodness-of-fit of 1.03 obtained using 61 variable parameters and 527 reflections with $F > 2\sigma(F)$. No reflections had intensities beyond the range for valid coincidence correction. For final cycle, maximum $\Delta/\sigma = 0.038$ with minimum and maximum residual electron densities of +1.57 and −1.75 e Å^{-3} in the vicinity of the Re atom. A view of the structure illustrating the atomic numbering scheme is given in Fig. 1. In Fig. 2 is presented a stereoview packing diagram projected down the crystallographic *a* axis. The final positional and thermal parameters are given in Table 1† and selected interatomic distances and angles are listed in Table 2.

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† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54730 (4 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

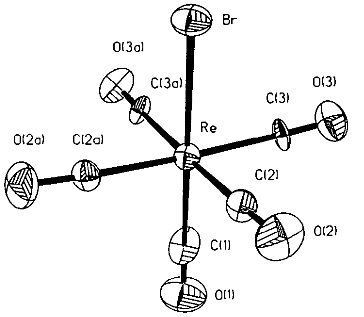

Fig. 1. A perspective view of the bromopentacarbonylrhenium(I) structure illustrating the atomic numbering scheme. Thermal ellipsoids have been drawn at the 50% probability level.

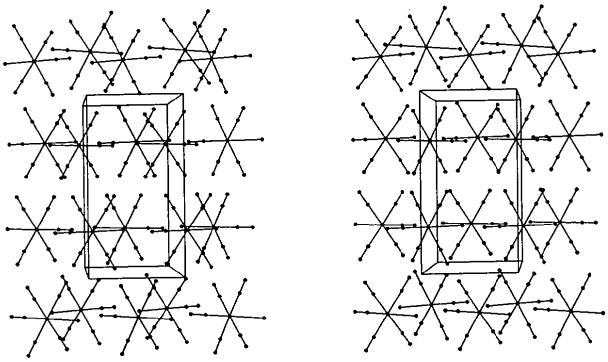

Fig. 2. A stereoview packing diagram projected down the crystallographic *a* axis.

Related literature. The reaction of Re dimers possessing *M*—*M* bonds with CO under pressure frequently results in the formation of monomeric octahedral complexes. The structure of the complex described here has been reported previously (Couldwell & Simpson, 1977). It is similar to that of an $ReCl_4(PMe_2Ph)_2$ complex (Aslanov, Mason, Wheeler & Whimp, 1970) and a related $ReCl_4(PEt_3)_2$ complex (Bucknor, Cotton, Falvello, Reid & Schmulbach, 1986).

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($Å^2 \times 10^3$)

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

	x	*y*	*z*	U_{eq}
Re	1235 (1)	2500	9473 (1)	23 (1)
Br	2550 (2)	2500	6076 (3)	38 (1)
O(1)	−267 (5)	2500	13431 (5)	58 (1)
O(2)	−167 (5)	4443 (5)	7321 (5)	55 (1)
O(3)	2740 (5)	4404 (5)	11445 (5)	54 (1)
C(1)	280 (5)	2500	11913 (5)	33 (1)
C(2)	330 (5)	3719 (5)	8094 (5)	29 (1)
C(3)	2206 (5)	3718 (5)	10748 (5)	27 (1)

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

Re—Br	2.619 (2)	Re—C(1)	1.889 (5)
Re—C(2)	1.975 (5)	Re—C(3)	1.991 (6)
O(1)—C(1)	1.143 (6)	O(2)—C(2)	1.135 (8)
O(3)—C(3)	1.108 (8)		
Br—Re—C(1)	179.7 (2)	Br—Re—C(2)	88.7 (1)
C(1)—Re—C(2)	91.0 (2)	Br—Re—C(3)	88.4 (1)
C(1)—Re—C(3)	91.8 (2)	C(2)—Re—C(3)	88.6 (2)
C(2)—Re—C(2*A*)	91.9 (3)	C(3)—Re—C(2*A*)	177.1 (2)
C(3)—Re—C(3*A*)	90.8 (3)	Re—C(1)—O(1)	177.8 (5)
Re—C(2)—O(2)	178.0 (5)	Re—C(3)—O(3)	179.3 (5)

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Structure of [(1,2-η²)-1,3,5,7-Cycloheptatetraene]bis(triphenylphosphine)platinum(0)

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Abstract. $[Pt\{(1,2\text{-}\eta^2)\text{-}C_7H_6\}\{P(C_6H_5)_3\}_2]$, (I), $M_r = 809.82$, monoclinic, $P2_1/c$, $a = 13.664\ (7)$, $b = 16.783\ (8)$, $c = 16.702\ (8)$ Å, $\beta = 111.68\ (4)°$, $V = 3559\ (3)$ Å^3, $Z = 4$, $D_x = 1.51$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 41.00$ cm^{-1}, $F(000) = 1608$, $T = 298$ K, $R = 0.0329$ and $wR = 0.0366$ for 3807 reflec-

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