Registry No. $OsCl_4$, 10026-01-4.

Supplementary Material Available: Listing of structure factor amplitudes (1 page). Ordering information is given on any current masthead page.

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Cesium Octachlorodirhenate(III) Hydrate. The Correct Structure and Its Significance with Respect to the Nature of Metal-Metal Multiple Bonding

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The crystal structure of $Cs_2Re_2Cl_8 \cdot H_2O$ has been redetermined. The unit cell contains four $Re_2Cl_8^{2-}$ anions, two of which are anhydrous and have a Re–Re distance of 2.237 (2) Å and the other two of which are coordinated in both axial positions by water molecules, with Re–O = 2.66 (3) Å, and have an Re–Re distance of 2.252 (2) Å. This result is in harmony with generally accepted views about the nature of the quadruple bond and corrects the erroneous claim made earlier by others that the hydrated ion had the shorter Re–Re distance. The compound crystallizes in space group $P2_1/c$ with the following unit cell parameters: $a = 9.323$ (3) Å, $b = 13.377$ (2) Å, $c = 11.979$ (2) Å, $\beta = 95.13$ (2) Å, $V = 1488.1$ (6) Å^3. For $Z = 4$ and a formula weight of 939.85 the calculated density is 4.19 g cm^{-3}.

Introduction

For several years, work has been conducted in this laboratory to establish a fundamental and important feature of strong M–M multiple bonds, namely, that the strength of such a bond (as evidenced, inversely, by the M–M distance) is reciprocally related to the strength of metal–ligand interactions along the extensions of the M–M axis. Ligands in these positions are called axial ligands, L_{ax}, and we have proposed, on the basis of structural evidence, and rationalized, in terms of the orbital overlaps involved, the rule that, other factors being held constant

$$\mathrm{d}r_{\mathrm{MM}}/\mathrm{d}r_{\mathrm{ML_{ax}}} < 0$$

Empirical evidence for this rule was first discussed by Cotton and Norman[1] who dealt with $Mo_2(O_2CR)_4L_2$ systems, employing both crystallographic and Raman data. It was demonstrated that when there is a very strong M–M quadruple bond, the binding of axial ligands is very weak. Nevertheless, even within this regime of strong M–M bonding and weak M–L_{ax} bonding it was shown that attachment of L_{ax} lengthens the M–M bond. Thus r_{MoMo} in $Mo_2(O_2CCF_3)_4$ is 2.090 Å and ν_{MoMo} is 397 cm^{-1} whereas in $Mo_2(O_2CCF_3)_4(py)_2$, $r_{MoMo} =$ 2.129 Å and ν_{MoMo} is 367 cm^{-1}.

Raman spectroscopy was also shown[1] to be a powerful tool for following this behavior, since the ν_{Mo-Mo} frequencies for $Mo_2(O_2CCF_3)_4$ were shown to decrease as the donor capacity (Lewis basicity) of the solvent in which the spectrum was run was increased. Similar results were published a little later by Ketteringham and Oldham.[2]

More recently, this relationship has been discussed further.[3,4] It has been specifically observed that since the binding of axial ligands probably depends primarily on the use of the metal d_{z^2} orbitals, which are also primarily responsible for the σ component of the M–M bond, increasing strength of the M–L_{ax} bonds must be correlated with decreasing strength of the M–M bond and thus with increasing M–M bond length.

In view of this line of thinking, we were surprised, and incredulous, to read[5] that in $Cs_2Re_2Cl_8 \cdot H_2O$, there are two crystallographically independent $Re_2Cl_8^{2-}$ ions, one having axially coordinated H_2O ligands and $r_{ReRe} = 2.210$ Å and the other lacking the axial ligands and having $r_{ReRe} = 2.226$ Å. It seemed evident from the discussion that this result was intended to be taken seriously. The structure was said to have been refined by least squares but the final discrepancy index was only 14.6%, the data used were measured photographically, and *no absorption corrections were applied even though a large crystal* (0.35 × 0.20 × 0.25 mm) *of a substance with*

Table I. Positional Parameters for All Atoms[a]

Atom	x	y	z
Re(1)	0.0736 (1)	−0.00119 (7)	0.07901 (8)
Re(2)	0.4356 (1)	0.06192 (7)	0.45604 (8)
Cs(1)	0.3809 (2)	0.3961 (2)	0.3240 (2)
Cs(2)	0.1175 (2)	−0.3131 (1)	−0.0456 (1)
Cl(1)	0.2775 (7)	−0.0799 (4)	0.0192 (5)
Cl(2)	−0.0577 (8)	0.0757 (5)	0.2110 (5)
Cl(5)	0.2203 (7)	0.0564 (4)	0.5429 (5)
Cl(6)	0.3159 (7)	−0.0246 (4)	0.3056 (5)
Cl(7)	0.4982 (7)	0.2012 (4)	0.5660 (5)
Cl(4)	0.1978 (7)	0.1484 (4)	0.0706 (5)
Cl(3)	0.0166 (7)	−0.1522 (4)	0.1619 (5)
Cl(8)	0.5887 (7)	0.1235 (5)	0.3295 (5)
O(1)	0.272 (4)	0.194 (2)	0.338 (3)

[a] The numbers in parentheses in this and other tables are the estimated standard deviations in the least significant digits.

a huge absorption coefficient had been used. Moreover, there were shockingly large ranges in the lengths of what should have been chemically similar bonds. For example, the reported Re–Cl bond lengths range from 2.18 to 2.36 Å. The estimated standard deviation in each of the Re–Re distances was stated to be 0.008 Å; thus, the difference between them, 0.016 Å, is not actually significant.

It was our conviction that if this structure were to be determined with sufficient accuracy, the Re–Re distance in $Re_2Cl_8(H_2O)_2^{2-}$ would be seen to be greater than that in $Re_2Cl_8^{2-}$. We have therefore redetermined this structure, taking the precautions required to obtain meaningful accuracy.

Procedure

Preparation of Crystals. $Cs_2Re_2Cl_8 \cdot H_2O$ was made by reacting $Re_2(O_2CCH_3)_4Cl_2$ and CsCl in a manner analogous to that described elsewhere[6] for preparing $(AsPh_4)_2Re_2Cl_8$. A saturated solution in boiling 6 N HCl was then cooled slowly over a period of 12 h. The resulting crystals were suitable for x-ray analysis and one of approximate dimensions 0.05 × 0.05 × 0.15 mm was chosen for this study.

Data Collection. The space group was found to be $P2_1/c$ from the systematic absences, and ω scans of several peaks showed widths at half-height of <0.15°. Accurate cell constants were obtained by centering on 15 intense reflections in the range $20 < 2\theta < 28°$. The cell constants and calculated volume are $a = 9.323$ (3) Å, $b = 13.377$ (2) Å, $c = 11.979$ (2) Å, $\beta = 95.13$ (2)°, and $V = 1488.1$ (6) Å^3. For $Z = 4$ and a formula weight of 939.85 the calculated density is 4.19 g/cm^3.

Data were collected on a Syntex $P\bar{1}$ diffractometer operated at 22 ± 2 °C using Mo Kα radiation. The θ–2θ scan technique was employed using a variable scan rate from 4.0 to 24.0°/min with a scan range from 0.9° below Kα_1 to +0.9° above Kα_2. The intensities of three standard reflections measured each 100 reflections showed no change with time. Data were collected in the range $0 \leq 2\theta < 42°$, giving a total of 2412 independent reflections of which 1430 had intensity $>3\sigma$. Lorentz and polarization corrections were applied to the data; the p factor[7] was set at 0.07. The linear absorption coefficient for Mo Kα radiation is 234.2 and, of necessity, an absorption correction was made. No extinction corrections were made.

Structure Solution.[7] The positions of the Cs and Re atoms were determined from the three-dimensional Patterson function and refined by least-squares methods. The eight chlorine atoms were then located by difference Fourier maps and these twelve atoms refined to give values of $R_1 = 0.098$ and $R_2 = 0.119$. At this point the absorption correction was applied and followed by several least-squares cycles. Another difference Fourier map revealed the oxygen atom of the coordinated water molecule. Isotropic refinement was then continued to give $R_1 = 0.051$ and $R_2 = 0.071$.

When all the atoms had been put in the proper asymmetric unit, four cycles of full-matrix refinement were run in which all atoms but the oxygen were treated anisotropically. This was sufficient to converge the structure, as no parameter shifted by more than half a standard deviation. The final values of R_1 and R_2 were 0.037 and 0.048, respectively, and the error in an observation of unit weight was 1.01. Anomalous dispersion effects were included in the calculated structure factors for all atoms in the final four cycles of refinement. A table of structure factor amplitudes is available.[9]

Results

The positional parameters of all atoms are listed in Table I and the thermal parameters in Table II. Figure 1 shows the contents of one unit cell, projected on the *ab* plane (the same view shown in ref 5) and also defines the numbering scheme. The $Re_2Cl_8^{2-}$ ions lie on the inversion centers at 0, 0, 0 and 0, $^1/_2$, $^1/_2$, while the $Re_2Cl_8(H_2O)_2^{2-}$ ions lie on the inversion centers at $^1/_2$, 0, $^1/_2$ and $^1/_2$, $^1/_2$, 0. The atoms in the former are labeled Re(1) and Cl(1)–Cl(4), while those in the latter are Re(2) and Cl(5)–Cl(8).

The bond distances and angles are listed in Tables III and IV, respectively. Figures 2 and 3 show the $Re_2Cl_8^{2-}$ and the $Re_2Cl_8(H_2O)_2^{2-}$ ions, respectively, with their average bond lengths and angles.

Discussion

We have found the structure to be grossly as reported in ref 5. However, we find that with respect to the most important feature, indeed the only one which makes this structure of any interest in view of the fact that there have been several other structural studies[8,10,11] of the $Re_2Cl_8^{2-}$ ion, the result reported in ref 5 is quite the opposite of what is correct—and reasonable. The Re–Re distance in the $Re_2Cl_8(H_2O)_2^{2-}$ ion, 2.252 (2) Å, is *longer* than that in the $Re_2Cl_8^{2-}$ ion, 2.237 (2) Å, by an amount, 0.015 Å, that is better than 99% certain to be significant since it is about 4 times greater than the sum of the esd's.

The bond lengths and bond angles pertinent to Re–Cl bonding found in this study agree in a very satisfactory way

Table II. Thermal Parameters[a]

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Re(1)	0.0046 (1)	0.00294 (4)	0.00353 (6)	−0.0008 (1)	0.0005 (1)	−0.0002 (1)
Re(2)	0.0049 (1)	0.00264 (4)	0.00456 (6)	0.0005 (1)	0.0019 (1)	0.0005 (1)
Cs(1)	0.0097 (2)	0.00993 (14)	0.0068 (1)	0.0024 (3)	0.0049 (3)	0.0037 (2)
Cs(2)	0.0082 (2)	0.00459 (9)	0.0080 (1)	0.0015 (3)	0.0001 (3)	−0.0029 (2)
Cl(1)	0.0059 (8)	0.0050 (4)	0.0068 (5)	0.0028 (9)	0.001 (1)	−0.0014 (7)
Cl(2)	0.0104 (9)	0.0054 (4)	0.0049 (5)	0.0007 (11)	0.004 (1)	−0.0019 (7)
Cl(3)	0.0062 (7)	0.0041 (3)	0.0074 (5)	0.0015 (9)	0.004 (1)	−0.0006 (7)
Cl(4)	0.0088 (8)	0.0050 (4)	0.0047 (4)	−0.0021 (10)	−0.001 (1)	0.0011 (6)
Cl(5)	0.0083 (8)	0.0033 (3)	0.0070 (5)	0.0007 (9)	0.000 (1)	−0.0004 (7)
Cl(6)	0.0075 (8)	0.0035 (3)	0.0062 (5)	−0.0038 (9)	0.000 (1)	−0.0002 (7)
Cl(7)	0.0101 (9)	0.0033 (3)	0.0052 (5)	−0.0028 (9)	0.000 (1)	0.0020 (6)
Cl(8)	0.0100 (9)	0.0053 (4)	0.0056 (5)	−0.0039 (10)	0.005 (1)	0.0029 (7)
O(1)	$B_{iso} = 9.6$ (8) Å^2					

[a] The form of the anisotropic ellipsoid is $\exp[-10^4(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$.

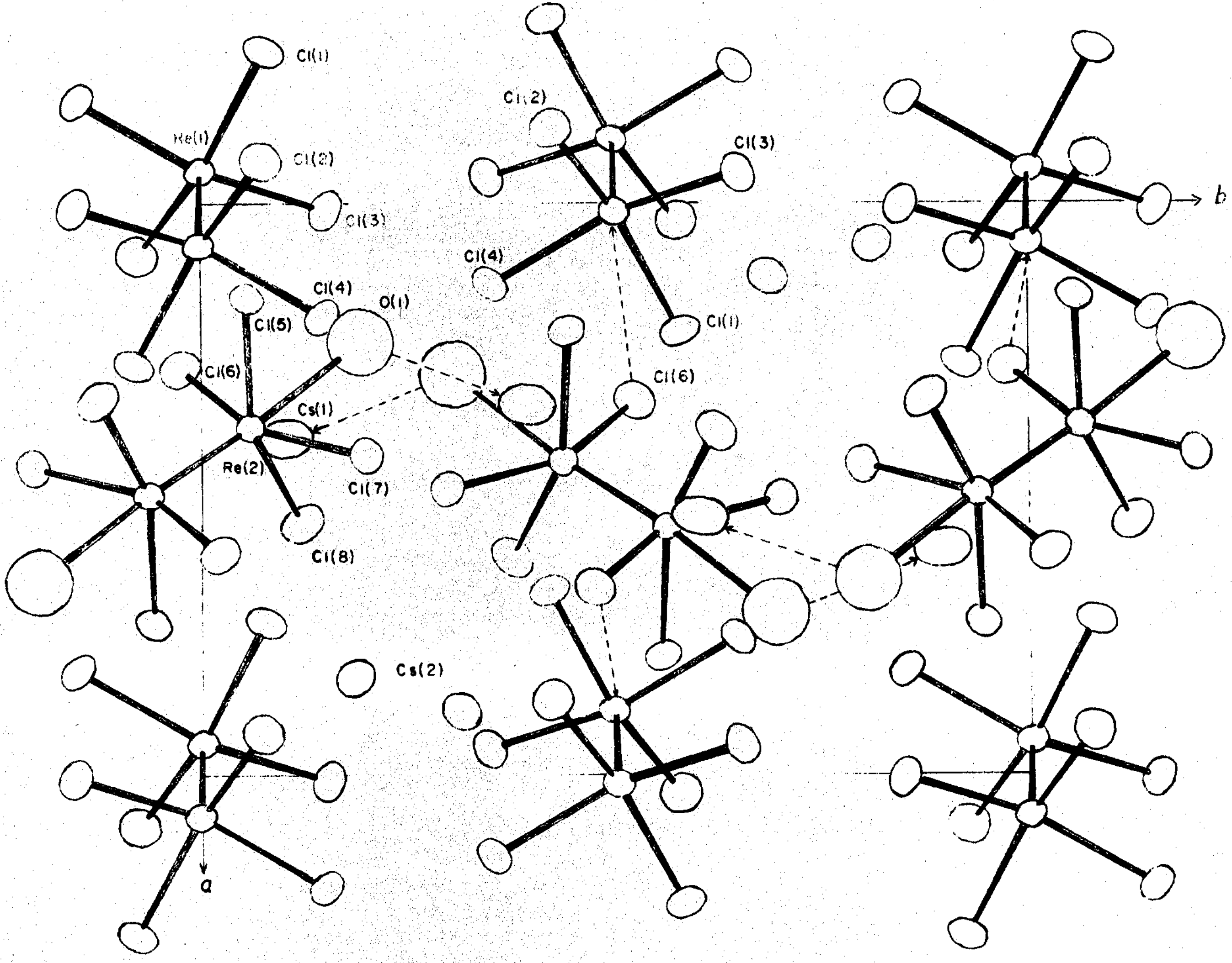

Figure 1. A projection of the unit cell on the *ab* plane to show the packing and the axial interactions. The water molecules, O(1), coordinated to Re(2) atoms are evident on the three anions running along the $a - {}^1/_2$ line. Dashed lines show O(1) to Cs(1) coordination and the very long Cl(6)···Re(1) contacts.

Table III. Bond Distances (Å)[a]

Re(1)-Re(1)′	2.237 (2)	Re(2)-Re(2)′	2.252 (2)
Re(1)-Cl(1)	2.341 (6)	Re(2)-Cl(5)	2.345 (6)
Re(1)-Cl(2)	2.326 (6)	Re(2)-Cl(6)	2.342 (6)
Re(1)-Cl(3)	2.334 (6)	Re(2)-Cl(7)	2.329 (6)
Re(1)-Cl(4)	2.334 (6)	Re(2)-Cl(8)	2.324 (6)
Re(1)-Cl(6)	3.386 (6)	Re(2)-O(1)	2.66 (3)
		Cs(1)-O(1)	2.90 (3)

[a] Other Cs(1) or Cs(2) contacts are <3.4 Å.

Table IV. Bond Angles (deg)

Re(1)′-Re(1)-Cl(1)	101.6 (2)	Cl(1)-Re(1)-Cl(3)	87.9 (2)
Re(1)′-Re(1)-Cl(2)	104.4 (2)	Cl(1)-Re(1)-Cl(4)	87.3 (2)
Re(1)′-Re(1)-Cl(3)	103.0 (2)	Cl(2)-Re(1)-Cl(3)	86.6 (2)
Re(1)′-Re(1)-Cl(4)	102.8 (2)	Cl(2)-Re(1)-Cl(4)	86.8 (2)
Cl(1)-Re(1)-Cl(2)	154.0 (2)	Cl(3)-Re(1)-Cl(4)	154.2 (2)
Re(2)′-Re(2)-Cl(5)	102.2 (2)	Cl(6)-Re(2)-Cl(8)	87.2 (2)
Re(2)′-Re(2)-Cl(6)	101.3 (2)	Cl(7)-Re(2)-Cl(8)	87.1 (2)
Re(2)′-Re(2)-Cl(7)	103.2 (2)	Re(2)′-Re(2)-O(1)	174.2 (6)
Re(2)′-Re(2)-Cl(8)	103.5 (2)	Cl(5)-Re(2)-O(1)	77.5 (8)
Cl(5)-Re(2)-Cl(6)	87.7 (2)	Cl(6)-Re(2)-O(1)	72.9 (6)
Cl(5)-Re(2)-Cl(7)	87.3 (2)	Cl(7)-Re(2)-O(1)	82.7 (6)
Cl(5)-Re(2)-Cl(8)	154.4 (2)	Cl(8)-Re(2)-O(1)	77.0 (7)
Cl(6)-Re(2)-Cl(7)	155.6 (2)		

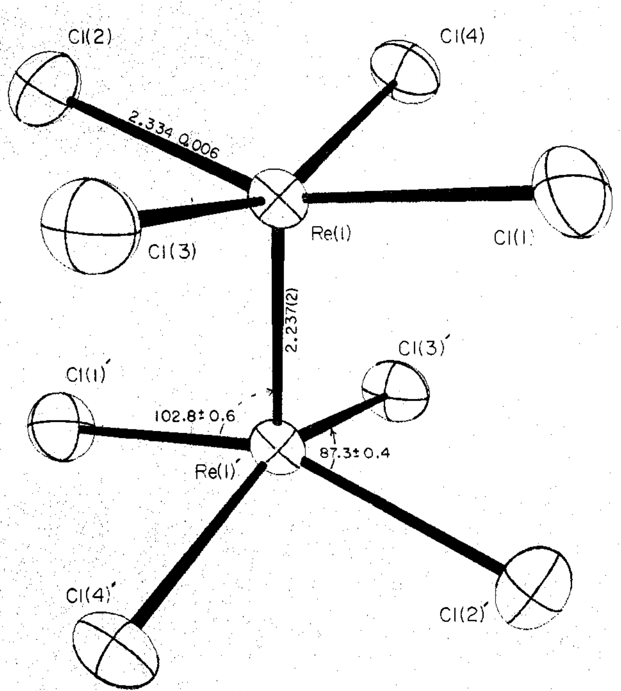

Figure 2. The $Re_2Cl_8^{2-}$ ion with its principal dimensions, averaged where appropriate according to D_{4h} symmetry.

with the most accurate previous values for $Re_2Cl_8^{2-}$, namely, those in $[(\eta\text{-}C_4H_9)_4N]_2Re_2Cl_8$.[8] There is no significant dif-

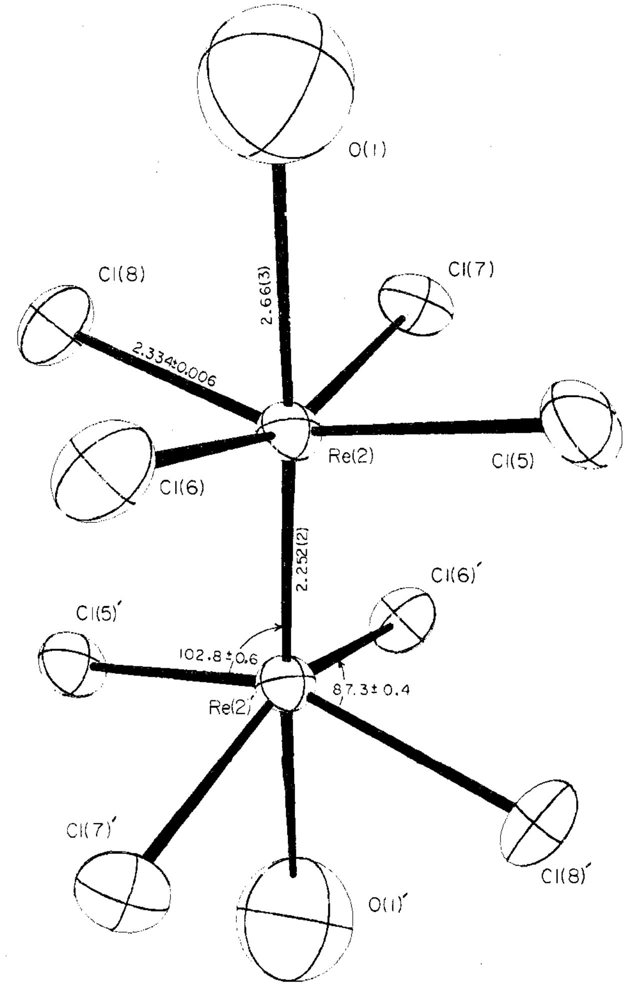

Figure 3. The $Re_2Cl_8(H_2O)_2^{2-}$ ion with its principal dimensions, averaged where appropriate according to D_{4h} symmetry.

Table V. Re–Re Distances in $Re_2X_8^{2-}$ Ions

Compd	Dist, Å	Ref
$(C_5H_6N)_2Re_2Cl_8$	2.244 (15)	10
$K_2Re_2Cl_8\cdot 2H_2O$	2.241 (7)	11
$(N(n\text{-}Bu)_4)_2Re_2Cl_8$	2.222 (2)	8
$Cs_2Re_2Br_8$	2.228 (4)	12

ference between the Re–Cl distances or Re–Re–Cl angles in the $Re_2Cl_8^{2-}$ ion (2.334 (4) Å, 103.0 (8)°) and the $Re_2Cl_8(H_2O)_2^{2-}$ ion (2.335 (9) Å, 102.6 (8)°) in the present case. The appropriate grand averages are listed here for comparison with the average values in the tetra-*n*-butylammonium salt:

	$Cs_4(Re_2Cl_8)_2(H_2O)_2$	$((n\text{-}Bu)_4N)_2Re_2Cl_8$
Re–Cl, Å	2.334 ± 0.006	2.32 ± 0.02
Re′–Re–Cl, deg	102.8 ± 0.6	103.9 ± 0.2

Moreover, the scatter in the crystallographically independent distances and angles of these types is very small, the ranges being 3.326–3.345 Å and 101.6–103.5° for Re–Cl and Re′–Re–Cl, respectively.

It is also appropriate to compare the Re(1)–Re(1)′ distance in this structure to the Re–Re distances found in other structures containing the $Re_2Cl_8^{2-}$ ion and, also, the $Re_2Br_8^{2-}$ ion, all in circumstances where no axial coordination exists. This is done in Table V and it is clear that these are all essentially the same. For the $Re_2Cl_8^{2-}$ ions only they range from 2.222 (2) to 2.244 (15) Å, with a mean value, calculated with each one weighted according to its individual esd, of 2.228 (15) Å. This may be taken as the best available value for the Re–Re distance in $Re_2Cl_8^{2-}$. It is interesting that it is identical with the one Re–Re distance reported for the $Re_2Br_8^{2-}$ ion.

Table VI. Some Re to Axial Ligand Distances

Compd	Axial ligand	Dist, Å	Ref
$Re_2(O_2CPh)_4Cl_2$	Cl^-	2.49	13
$(NH_4)_2[Re_2(O_2CH)_2Cl_6]$	Cl^-	2.71	14
$Re_2(Ph_2PCH_2PPh_2)_2Cl_5$	Cl^-	2.58	15
$Re_2(MeSCH_2CH_2SMe)_2Cl_5$	Cl^-	2.49	16
$Re_2(N_2CPh_3)_2Cl_4\cdot C_4H_8O$	C_4H_8O	2.54	4
$Re_2(O_2CCH_3)_2Cl_4\cdot 2H_2O$	H_2O	2.50	17
$Re_2(O_2CC_3H_7)_4(ReO_4)_2$	ReO_4^-	2.18	18
$Re_2(O_2CC_3H_7)_3Cl_2(ReO_4)$	ReO_4^-	2.28	19
$Na_2Re_2(SO_4)_4\cdot 8H_2O$	H_2O	2.28	20

The intermolecular packing is shown in Figure 1; this is closely similar to, and certainly qualitatively the same as, that presented in ref 5. In the present case we have clearly indicated the various intermolecular contacts that are relevant to the question of axial interactions—or the lack thereof. It is evident that the Cl(6)···Re(1) relationship is simply a packing contact. The distance involved, 3.39 Å, as well as the four Cl(6)–Cl(*n*), *n* = 1–4, distances, which range from 3.6 to 3.9 Å, are entirely appropriate to such contacts. It is particularly instructive to compare the Cl(6)···Re(1) distance to those in several carboxylato-bridged dirhenium compounds, where genuine coordination trans to a Re–Re quadruple bond exists. All of the available data are listed in Table VI.

Axially coordinated Cl^- ions lie at distances in the range 2.5–2.7 Å while coordinated oxygen atoms are found at distances from 2.15 to 2.54 Å. These data provide a sound empirical basis for concluding that in $Cs_2Re_2Cl_8\cdot H_2O$, the Re(1)···Cl(6) distance of 3.39 Å does not allow the Cl(6) atom to be considered coordinated to Re(1) while, conversely, the Re(2)–O distance of 2.66 Å permits one to regard the water molecule as coordinated axially to Re(2), albeit with a slightly weaker interaction than those previously observed.

In conclusion, it is abundantly clear that the Re(2)–Re(2)′ distance is very significantly longer than both the Re(1)–Re(1)′ distance here and the weighted mean, mentioned earlier, of all such distances in $Re_2Cl_8^{2-}$ ions that are not axially coordinated. The conclusion, then—which is precisely the opposite of that previously implied—seems very clear: The binding of the axial ligands, H_2O, by $Re_2Cl_8^{2-}$ causes the Re–Re bond to become longer by 0.015 ± 0.004 Å and thus, presumably, a little weaker.

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Supplementary Material Available: Listing of structure factor amplitudes (7 pages). Ordering information is given on any current masthead page.

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Molecular Structure and Absolute Configuration of *cis*-Dichloro((*S*)-methyl *p*-tolyl sulfoxide)(3-methyl-1-butene)platinum(II)

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The molecular structure and absolute configuration of *cis*-dichloro((*S*)-methyl *p*-tolyl sulfoxide)(3-methyl-1-butene)-platinum(II), $PtCl_2[CH_3(O)S(C_6H_4CH_3)][(CH_3)_2CHCH{=}CH_2]$, have been determined by standard Patterson and Fourier techniques using x-ray data collected by counter methods. The molecule crystallizes in the monoclinic space group $P2_1$ with $Z = 2$. The unit cell dimensions are $a = 10.556$ (2) Å, $b = 10.488$ (2) Å, $c = 7.941$ (2) Å, and $\beta = 107.72$ (1)°. The structure has been refined by full-matrix least-squares techniques on F, using 2820 unique reflections for which $F^2 > 2\sigma(F^2)$, to a final agreement factor of 0.0377. The complex exhibits a square-planar coordination geometry with the double bond of the coordinated olefin ligand tilted from the perpendicular by 5.7 (6)°. The absolute configuration at the asymmetric carbon atom of the olefin was determined by the Bijvoet method to be *S*.

Introduction

In the first paper of this series[1] we described the structure of a complex which exhibited a strong asymmetric bias for the coordination of one enantiotopic face of the prochiral olefin styrene. The preferred configuration of the coordinated olefin ligand and the arrangement of the *p*-tolyl group on the asymmetric sulfoxide ligand positioned the two phenyl rings in such a way that an attractive interaction between them could occur. In attributing the preferential formation of one diastereomer to this interligand attraction, we felt it necessary to examine the molecular structure of a similar complex which contained an olefin ligand for which an attractive interaction of comparable magnitude was unlikely. This would involve replacing the aryl olefinic substituent with an alkyl group. The 3-methyl-1-butene complex had been found to exist preferentially as one diastereomer in a ratio of 2:1 at equilibrium.[2] On the basis of circular dichroism spectra the complex was postulated to contain the olefin with the *S* absolute configuration. Suitable crystals were eventually obtained and we report here the results of the single-crystal x-ray structural analysis of *cis*-dichloro((*S*)-methyl *p*-tolyl sulfoxide)(3-methyl-1-butene)platinum(II).

Experimental Section

The crystals of *cis*-dichloro((*S*)-methyl *p*-tolyl sulfoxide)(3-methyl-1-butene)platinum(II), $PtCl_2[CH_3(O)S(C_6H_4CH_3)]-[(CH_3)_2CHCH{=}CH_2]$, kindly provided by H. Boucher and B. Bosnich, appeared as translucent, white, triangular blocks. Preliminary Weissenberg and precession photography showed the crystals to be monoclinic with Lauē symmetry $2/m$. The systematic absences observed, $0k0$ for k odd, and the requirement of an acentric space group for an optically active molecule determined the space group to be $P2_1$, C_2^2, No. 4.[3]

The crystal chosen for data collection was of approximate dimensions 0.23 × 0.20 × 0.10 mm. It was carefully measured on a microscope fitted with a filar eyepiece before application of an absorption correction. The crystal had seven faces, {100}, (110), ($\bar{1}\bar{1}0$), (011), ($01\bar{1}$), and ($0\bar{1}0$), which were identified by optical goniometry. The crystal was mounted on a Picker FACS-1 computer-controlled diffractometer in a random orientation with [010] approximately 28° from coincidence with the ϕ axis. Cell constants and an orientation matrix were obtained from a least-squares refinement of 18 intense, carefully centered reflections with $20 < 2\theta < 50°$. Prefiltered Cu radiation was used, λ 1.540 56 Å, at an ambient temperature of 20 °C. The crystal data are summarized in Table I.

Table I. Crystal Data

$C_{13}H_{20}Cl_2OPtS$	Fw 490.36
$a = 10.556$ (2) Å	Space group $P2_1$
$b = 10.488$ (2) Å	$Z = 2$
$c = 7.941$ (2) Å	Density (obsd)[a] = 1.950 (2) g cm^{-3}
$\beta = 107.72$ (1)°	Density (calcd) = 1.944 g cm^{-3}
Cell vol 837.51 Å^3	$\mu = 187.4$ cm^{-1} for Cu Kα_1

[a] By neutral buoyancy in hexane and 1,2-dibromotetrafluoroethane.

Table II. Experimental Conditions for Data Collection

Radiation: Cu Kα, Ni foil (0.018 mm) prefilter
Takeoff angle: 2.3° (90% of maximum Bragg intensity)
Aperture: 4 × 4 mm, 31 cm from crystal
Data collected: $\pm h, \pm k, l$, for $0 < 2\theta < 130°$
Scan: θ–2θ at 2° min^{-1}, with α_1–α_2 dispersion correction
Scan range: 1.2° symmetric for $2\theta < 70°$, 0.8° below α_1 to 0.6° above α_2 for $70 < 2\theta < 130°$
Background: 10 s stationary crystal, stationary counter measured at limits of scan for $2\theta < 110°$ and 20 s for $2\theta > 110°$
Standards: Six recorded every 175 observations, 020, $\bar{1}00$, $0\bar{2}0$, 100, 001, and $\bar{1}02$

The conditions used for data collection are given in Table II. The measurement of standard reflections over the course of data collection and an examination of ω scans for several intense, low-angle reflections before and after data collection showed no significant degradation of crystal quality had occurred. The standard reflections showed an average increase of 0.46% with the largest change being 3.8%.

The intensity data were processed as previously described.[4] The value of p, the "ignorance factor", was chosen to be 0.02 from an examination of both the variations in the standard reflections[5] and the final weighting scheme. An absorption correction was applied to all 3088 data with $F^2 > 0$.[6] The maximum and minimum transmission coefficients were 0.838 and 0.661, respectively, a variation of 26.8%. There were 211 pairs of symmetry-equivalent reflections, for the planes $hk0$ and $\bar{h}k0$. These were averaged to give a weighted agreement factor of 6.0% based on F^2 before the absorption correction.