on the 27 365-cm^{-1} $^4E(D)$ origin. The weak shoulders are apparently components of the same energy progression based on a false origin or a second spin–orbit component some 45 cm^{-1} above the 27 365-cm^{-1} origin. The oscillator strength of this manifold is constant between 150 and 4.2 K, Figure 2.

With $E \parallel c$ the absorption is very weak and appears as three very weak, strongly overlapped maxima at ~27 370, ~27 500, and ~27 650 cm^{-1}. These appear to be the $E \parallel c$ components of the intense members of the 145-cm^{-1} progression observed with $E \perp c$.

$^6A_1(S) \rightarrow {}^4T_1(P)$. This absorption is most intense with $E \perp c$ and appears as a weak maximum at 29 640 cm^{-1} followed by a more intense manifold exhibiting a shoulder at ~29 890 cm^{-1} and band maximum at 30 030 cm^{-1}. With $E \parallel c$ the absorption is somewhat less intense and appears as a sharp, weak peak at 29 498 cm^{-1}, a weak broad peak at 29 638 cm^{-1}, and a more intense manifold with a shoulder at ~29 890 cm^{-1} and maximum at 30 030 cm^{-1}. This state is quite sensitive to the effect of spin–orbit coupling[13] and to a trigonal field. These effects most likely account for the observed structure but the lack of resolution and absence of clear-cut polarization behavior makes quantitative analysis on this basis impossible.

$^6A_1(S) \rightarrow {}^4A_2(F)$. This absorption appears as a weak shoulder on the charge-transfer absorption with $E \perp c$ and as a pronounced shoulder with $E \parallel c$. In both cases the band is observed at ~34 600 cm^{-1}. The strong overlap with the charge-transfer absorption prevents useful analysis of this absorption.

Summary and Conclusion

While the optical spectrum of $RbMnBr_3$ is superficially that of Mn^{2+} in a trigonally distorted octahedral environment, it displays oscillator strengths which are 2–3 orders of magnitude greater than the analogous transitions in isolated manganese(II) complexes. Comparing the spectrum of $RbMnBr_3$ to the spectrum of the structurally analogous $CsMnBr_3$[6] we note a commonality of polarization behavior. In all cases except where a $^6A_1 \rightarrow {}^4A_1$ transition occurs the more allowed process occurs perpendicular to the chain axis. In the case of the $^6A_1 \rightarrow {}^4A_1$ transitions the sense of polarization is the opposite. Day and Dubicki[7] have derived the selection rules for electronic transitions involving antiferromagnetically coupled Mn^{2+} pairs in TMMC. Since the site symmetry of the Mn^{2+} ions in $RbMnBr_3$ is the same as that for the Mn^{2+} ions in TMMC, the same selection rules apply. These are (for D_{3d} symmetry)

$$^6A_{1g} \xrightarrow{E \parallel c} {}^4A_{1g}$$

$$^6A_{1g} \xrightarrow{E \perp C} {}^4E_g$$

$$^6A_{1g} \nrightarrow {}^4A_{2g}$$

Since states of T_{1g} symmetry in the O_h group become states of $A_{2g} + E_g$ symmetry in the D_{3d} group and since

$$T_{2g} \xrightarrow{D_{3d}} A_{1g} + E_g$$

we should expect one band, allowed $E \perp c$, for the two $^6A_{1g} \rightarrow {}^4T_{1g}$ transitions, Figure 1, and two manifolds for each of the two $^4T_{2g}$ parent states. The behavior of the lowest $^6A_{1g} \rightarrow {}^4T_{1g}$ transitions as well as the behavior of the lowest $^6A_{1g} \rightarrow {}^4E_g$ transition would support the use of the simple pair model selection rules. It is clear however, from the behavior of the $^6A_{1g} \rightarrow {}^4A_{1g}$, ^4E_g, the $^6A_{1g} \rightarrow {}^4T_{2g}(D)$, the $^6A_{1g} \rightarrow {}^4T_{1g}(P)$, and the $^6A_{1g} \rightarrow {}^4A_{2g}$ transitions that a significant modification of this approach is necessary to accurately describe the spectra of $RbMnBr_3$ and $CsMnBr_3$.

While there are distinct similarities in the band positions and polarization behavior of $RbMnBr_3$ and $CsMnBr_3$, the temperature dependences of the oscillator strengths of corresponding transitions are considerably different. For transitions in the spectrum of $RbMnBr_3$ the temperature dependence that is observed is primarily that predicted by theory[11,12] for an exchange-induced electric dipole process while for $CsMnBr_3$ a variety of temperature dependences is observed some of which possess a distinct vibronic component.

On the basis of the oscillator strengths of corresponding transitions in $RbMnBr_3$ and $CsMnBr_3$, it does not appear, with the limits of our experimental measurements, that the shorter interchain distance and attendant higher T_N in $RbMnBr_3$ in any way affect the transition probability. Indeed in all cases the transitions in the $CsMnBr_3$ system appear to be approximately equal in intensity or stronger. Whether this is, in fact, a general phenomenon or not awaits the investigation of other systems.

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Some Metal Halide–Phosphorus Halide–Alkyl Halide Complexes. 4. Bis(*tert*-butyltrichlorophosphonio)tri-μ-chloro-hexachloro-*triangulo*-trirhenium(III) Hexachlororhenate(IV)

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The reaction of a metal halide with a *tert*-butyl halide and a phosphorus(III) halide has been used[2-4] to synthesize the anions BCl_4^-, $AlCl_4^-$, $SnCl_5^-$, $NbCl_6^-$, $TaCl_6^-$, WCl_6^-, $Ti_2Cl_9^-$, $Ti_2Br_9^-$, and $Zr_2Cl_9^-$. The analogous reaction for rhenium(V) chloride is now presented in which a single compound containing the tri-μ-chloro-hexachloro-*triangulo*-trirhenium(III) unit, the hexachlororhenate(IV) anion, and the *tert*-butyltrichlorophosphonium cation is formed. Previously bromides[5] containing both Re_3Br_9 and $ReBr_6^{2-}$ have been reported.

Experimental Section

Rhenium(V) chloride (99.5%) was used as supplied and other reagents were as previously described.[2]

Table I. Infrared Spectra (cm^{-1})

295 K	90 K	Assignments	Lit.[2–4,6,8,13]
		t-BuPCl$_3^{\bullet}$	
780 w		A$_1$ ν(P–C)	778–783
483 w		ν(P–Cl)	481–486
232 w, sh	230 w	σ(P–Cl)	229–235
643 s 632 s		E ν(P–Cl)	632–648
	214 w	σ(t-BuP–Cl)	208–214
208 vw, br	208 w		
		ReCl$_6^{2-}$	
651 m, sh		$\nu_1 + \nu_3$	
587 w		$\nu_2 + \nu_3$	
312 m, sh	310 mw	T$_{2g}$ ν_3	320–380
285 vw	284 w	$\nu_5 + \nu_6$?	
170 m	170 m	T$_{2g}$ ν_4	170–180
		Re$_3$Cl$_9$	
375 m	380 m	A$_2''$ ν(Re–Cl) terminal	378 sh (369 sh)
	358 s, sh	E$'$ ν(Re–Cl)	
355 vs	353 s, sh	terminal	359 s
	350 s	E$'$ ν(Re–Cl)	350 sh
345 vs	340 s, sh	ring	
324 m, sh	324 m	E$'$ ν(Re–Cl) ring	322 m
	258 m, sh	E$'$ ν(Re–Cl)	240–300 vw
255 ms	252 ms	ring	220 m
150 m	151 m	2A$''$ + 2E$'$ def	
126 w	128 w		
118 w	117 w		
98 w	100 w		
		Unassigned	
668 w, sh			
616 w, sh			
418 w			
60 w			

Vacuum-line and glovebox techniques were used throughout as the product was water sensitive. Phosphorus(III) chloride was added to rhenium(V) chloride in carbon disulfide and to this was added *tert*-butyl chloride (PCl_3 and t-BuCl were present in a slight excess of an equimolar ratio). A green-brown precipitate formed over 30 min. Anal. Calcd for $C_8H_{18}Cl_{21}P_2Re_4$: Re, 44.72; Cl, 44.70. Found: Re, 44.7; Cl, 44.65. The product, which could not be recrystallized, was stored in sealed glass tubes.

Infrared spectra of Nujol mulls between potassium bromide or polythene plates were obtained using the Grubb-Parson Spectromaster (1000–400 cm^{-1}) and the RIIC FS-720 Interferometer (400–30 cm^{-1}). Raman spectra could not be obtained using laser excitation. Uv and visible spectra of the solid were recorded by reflectance using a Unicam 700C spectrophotometer. Mass spectra were obtained by solid sample insertion using the AEI MS12 instrument.

Results and Discussion

Infrared Spectra (Table I). The Cation. The t-BuPCl$_3^+$ cation spectrum has been well characterized[2–4] so that we subtracted the usually observed vibrations from the experimental spectra and assigned the rest mainly to rhenium–halide modes.

The Anion, ReCl$_6^{2-}$. An O_h anion has the vibrational representation

$$\Gamma_{\mathrm{vib}} = \nu_1\,[\mathrm{A_{1g}}\,(\mathrm{R})] + \nu_2\,[\mathrm{E_g}\,(\mathrm{R})] + \nu_3, \nu_4\,[2\,\mathrm{T_{2g}}\,(\mathrm{ir})] + \nu_5\,[\mathrm{T_{2g}}\,(\mathrm{R})] + \mathrm{T_{2u}}\,(\text{inactive})$$

The two ir-active fundamentals appear[6] at 320 and 173 cm^{-1} in the spectrum of the potassium salt, and the three Raman-active modes, at 346, 283, and 177 cm^{-1}.

Table II

(i) Mass Spectrum[a] for (t-BuPCl$_3$)$_2$(Re$_3$Cl$_9$)(ReCl$_6$)

m/e[b]	% parent peak	Species	m/e	% parent peak	Species
878	100	Re$_3$Cl$_9^+$	514	28.2	Re$_2$Cl$_4^+$
843	24.2	Re$_3$Cl$_8^+$	479	27.7	Re$_2$Cl$_3^+$
808	5.2	Re$_3$Cl$_7^+$	444	11.5	Re$_2$Cl$_2^+$
771	7.0	Re$_3$Cl$_6^+$	409	6.5	Re$_2$Cl$^+$
736	10.4	Re$_3$Cl$_5^+$	372	4.2	Re$_2^+$
701	8.4	Re$_3$Cl$_4^+$	329	3.9	ReCl$_4^+$
666	7.3	Re$_3$Cl$_3^+$	292	28.3	ReCl$_3^+$
629	4.5	Re$_3$Cl$_2^+$	257	16.1	ReCl$_2^+$
594	1.9	Re$_3$Cl$^+$	222	6.1	ReCl$^+$
586	36.2	Re$_2$Cl$_6^+$	187	6.7	Re$^+$
549	55.2	Re$_2$Cl$_5^+$			

(ii) Isotopic Distribution of Parent Peak, Re$_3$Cl$_9^+$

	% abundance			% abundance	
m/e	Calcd[c]	Obsd	m/e	Calcd[c]	Obsd
870	1.5	3.1	884	17.8	17.3
872	12.7	13.3	886	5.0	5.5
874	43.2	44.7	888	1.0	1.6
876	82.9	83.5	890	0.1	0
878	100	100	892	<0.1	0
880	80.8	80.4	894	<<0.1	0
882	45.3	43.1			

[a] Rhenium–chlorine peaks only; ion voltage 70 eV; source temperature 320 °C. [b] Highest intensity peak of each multiplet selected. [c] Calculated using isotopic abundances: ^{35}Cl, 75.53%; ^{37}Cl, 24.47%; ^{185}Re, 37.07%; ^{187}Re, 62.93%.

We have assigned bands at 310 and 170 cm^{-1} to the ir-active fundamentals. The combination bands give ν_1 = 341 cm^{-1} and ν_2 = 277 cm^{-1} in good agreement with published results.[6]

In previous work,[3] the ν_1 mode for WCl$_6^-$ was observed as a very weak band so that there is only poor justification for assigning the strong band at 355 cm^{-1} to ν_1(ReCl$_6^{2-}$) particularly as it splits into two components at low temperature.

The Trimeric Unit, Re$_3$Cl$_9$. In rhenium(III) chloride,[7] a planar, chloro-bridged Re$_3$Cl$_3$ ring has two terminal Cl atoms bound to each Re above and below the plane of the ring. One of the out-of-plane Cl atoms is coordinated to a vacant in-plane position of an Re atom of an adjacent Re$_3$Cl$_9$ unit. In the bromides,[5] M$_2$(Re$_3$Br$_9$)(ReBr$_6$), there are no intramolecular Br bridges so that each Re atom of the Re$_3$Br$_9$ cluster is four-coordinate to Br. Both trimeric units are strongly metal–metal bonded.

There was a clear similarity between our spectrum (Table I) and that[8] of Re$_3$Cl$_9$. The ion Re$_3$Cl$_{12}^{3-}$, which has no intraion bridges, also has[9] a related ir spectrum [344 (s), 324 (s), 282 (m), 274 (m), 243 (m), 225–218 (s), 204 (s) cm^{-1}] and absorption in the region 360–345 cm^{-1} seems to be characteristic of authentic trimeric chlororhenium(III) species.[10] We have tentatively assigned the absorptions in D_{3h} symmetry which was a reasonable description of the trimeric unit[5] in M$_2$(Re$_3$Br$_9$)(ReBr$_6$). The vibrational representation is

$$\Gamma_{\mathrm{vib}} = 4\,\mathrm{A}'\,(\mathrm{R}) + \mathrm{A_1}'\,(\text{inactive}) + 2\,\mathrm{A_2}'\,(\text{inactive}) + 3\mathrm{A_2}''\,(\mathrm{ir}) + 6\mathrm{E}'\,(\mathrm{ir, R}) + 4\mathrm{E}''\,(\mathrm{R})$$

The ir-active modes may be divided into A$_2''$ + E$'$ terminal Re–Cl stretching, 3E$'$ ring stretching, and 2 A$_2''$ + 2 E$'$ deformation modes. Some of the absorptions split at low temperature and were assigned to E modes.

Mass Spectrum (Table II). This provided evidence for the presence of Re$_3$Cl$_9$. A large number of groups of lines were observed. The most intense member of each multiplet was selected for Table II. The remaining lines of each group reflected the isotopic abundances of rhenium and chlorine. The

Table III. Electronic Spectrum (cm^{-1}) for $(t\text{-BuPCl}_3)_2(\text{Re}_3\text{Cl}_9)(\text{ReCl}_6)$

295 K	90 K	Assignments[7,8,13]
(5 900)[a]	(6 000)	
(6 600)	(6 600)	
6 700	6 700	ReCl_6^{2-} ($^4A_{2g} \rightarrow {}^2T_{1g}$)?
	(6 800)	
(8 600)	(8 800)	ReCl_6^{2-} ($^4A_{2g} \rightarrow {}^2E_g$)
9 500	9 500	ReCl_6^{2-} ($^4A_{2g} \rightarrow {}^2T_{2g}$)
(10 200)	10 300	Re_3Cl_9
	10 600	
(11 500)	(11 500)	
	(12 100)	
	(12 500)	
	14 200	ReCl_6^{2-} ($^4A_{2g} \rightarrow {}^2T_{2g}$); Re_3Cl_9
15 000	(14 800)	ReCl_6^{2-} ($^4A_{2g} \rightarrow {}^2T_{2g}$)
	(17 500)	
18 300	18 400	Re_3Cl_9
	(21 500)	
	(23 800)	
25 000	25 500	Re_3Cl_9
28 000	27 800	
32 000	32 000	
33 400	33 300	
38 000	37 500	
45 500	45 400	

[a] Parentheses indicate shoulders.

group centred on *m/e* 878 were the strongest (Re_3Cl_9^+ 877.77) so that 878 was used as the parent peak. This group was analyzed in terms of the isotopic abundances and excellent agreement was found between the calculated and observed intensities for *m/e* 870–894.

The mass spectrum and assignments agree well with the reported spectra[11,12] for rhenium(III) chloride (Re_3Cl_9) in which several low molecular weight (201–314) fragments containing oxygen have been observed. These were reproduced on handling $(t\text{-Bu})_2\text{P}_2\text{Re}_4\text{Cl}_{21}$ in the air, but if the sample probe was filled under nitrogen, the oxygen-free spectrum was observed. Bromine-containing fragments were also recognized.

Electronic Spectrum (Table III). There was reasonably good agreement between the reflectance spectrum and that expected for ReCl_6^{2-} + Re_3Cl_9.[7,8,13] Most of the low-frequency, spin-forbidden bands for ReCl_6^{2-} were sharp but one (6700 cm^{-1}) was at lower frequency than is usually observed. The spectra[7,8] for Re_3Cl_9 show three or four regions of absorption below 26 000 cm^{-1}. Only the one near 14 000 cm^{-1} was doubtful as ReCl_6^{2-} also absorbs here. There was no evidence to suppose that $\text{Re}_2\text{Cl}_8^{2-}$, Re_2Cl_9^-, or $\text{Re}_2\text{Cl}_9^{2-}$ was present.[14,15] This confirmed the mass and ir spectral assignments.

Preparative Route. The use of phosphorus(III) chloride can result in reduction of the metal ion, e.g., in WCl_6^- prepared from[3] WCl_6 and in our unpublished observations of MoCl_5. In the present work, $\text{Re}_2\text{Cl}_{10}$ was reduced at room temperature to give ReCl_6^{2-} and the Re_3 cluster directly. Thermal methods may be used[16] for the conversion of Re_2X_{10} (X = Cl, Br) to Re_3X_9 which is then followed by reactions which yield other Re_3 cluster compounds.

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Linear, Bent, and Triangular M–O$_2$ Bonding in Some Dioxygen Complexes of Cobalt

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Chemical bonding of dioxygen to five- and four-coordinated metal complexes, using $[\text{Co(CN)}_5\text{O}_2]^{3-}$ (**1**)[1] and $[\text{Co(PR}_3)_4\text{O}_2]^+$ (**2**)[2] as model systems, is explored in this note with the hope of discerning the driving force for bent vs. triangular Co–O$_2$ configurations, respectively. These two geometries are of particular interest in that they are related to the two alternative structures, viz., bent and triangular, first proposed by Pauling[3] and Griffith,[4] respectively, for the Fe–O$_2$ bonding in oxyhemoglobin and oxymyoglobin.

The Fenske–Hall parameter-free MO model[5] was employed in this work. The basis functions were those utilized previously.[5b,c] For phosphorus, only 1s to 3p atomic orbitals are included in the calculations. The pertinent molecular parameters were taken from the known structures of $[\text{Co(CN)}_5\text{O}_2]^{3-}$ [1b] and $[\text{Co(Ph}_2\text{PCH{=}CHPPh}_2)_2\text{O}_2]^+$.[2] In particular, the Co–O and O–O distances chosen are 1.93 and 1.26 Å, and 1.89 and 1.42 Å, for **1** and **2**, respectively. The calculated one-electron molecular orbital energies as a function of the Co–O–O angle (θ) for **1** and **2** are shown in Figures 1 and 2, respectively.

These diagrams indicate that **1** favors a bent structure because of the steep energetic valley of π_x^* at $\theta \approx 132°$ whereas **2** prefers an isosceles triangular structure as a result of the large slope of xz at $\theta \approx 69°$. The reason for this difference is the relative energetic ordering of the metal z^2 orbital with respect to the dioxygen π^* orbitals.[6,7] In **1**, z^2 lies above π^* so that as θ decreases, π_x^* declines rapidly in energy due to both the decrease in its *antibonding* interaction with xz (which lies lower in energy) and the increase in the *bonding* interaction with z^2 (which lies higher in energy).[8] The latter interaction is symmetry forbidden in either linear or triangular configuration. Below $\theta \approx 132°$, π_x^* rises steeply in energy due to the reacquired, enhanced antibonding interaction with xz. The π_y^* orbital exhibits only a slight minimum since π_y^* is perpendicular to the plane of bending. The net result is a Walsh minimum[11a] corresponding to a bent configuration at $\theta \approx 132°$ for $\text{M(d}^6)\text{–O}_2^{(n-2)-}(\pi^{*n})$ where $n = 3$ for [Co-

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