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Registry No. Copper, 7440-50-8.

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Physical Properties of Linear-Chain Systems. 6. Single-Crystal Absorption Spectra of $RbCrCl_3$ and $CsCrCl_3$[1]

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$RbCrCl_3$ and $CsCrCl_3$ show the same crystal structure as a number of ABX_3 compounds (with A = a univalent alkali metal cation, B = a divalent first-row transition metal ion, and X = Cl, Br, or I).[1] This structural type contains $[BX_6]$ octahedra sharing faces to form linear chains; the chains are sufficiently separated by the A cations for the magnetic interactions between chains to be very weak. Thus the major interactions are one-dimensional, along the chains. These interactions produce very significant effects on the magnetic and spectroscopic properties in comparison to those of isolated BX_6^{4-} octahedra.[3] The spectrum of $CsCrCl_3$ has previously been examined at 77 K,[4] and an anomalous absorption was reported at 22 000 cm^{-1}, an energy approximately twice that observed for the $^5E \rightarrow {}^5T_2$ transition. This absorption was attributed to a pairwise excitation of $^5E \rightarrow {}^5T_2$ transitions associated with adjacent Cr^{2+} ions, (i.e., $2(^5E \rightarrow {}^5T_2)$). As this particular form of interaction has not otherwise been observed in the ABX_3 compounds, the optical spectrum of $RbCrCl_3$ has been examined and that of $CsCrCl_3$ reexamined to 4.2 K.

Experimental Section

Preparations. Anhydrous CsCl, RbCl, and $CrCl_2$ were purchased from ROC/RIC. The compounds $CsCrCl_3$ and $RbCrCl_3$ were prepared by mixing equimolar quantities of CsCl or RbCl and $CrCl_2$, sealing the mixture in an evacuated quartz ampule, and refining in a Bridgman furnance. The resulting single-crystal boules ~5 cm in length by 1.5 cm in diameter could readily be cleaved (along 1120) to give flakes $\parallel c$, suitable for σ and π spectroscopic measurements. Both crystals show strong pleiochroism, appearing red-brown in light polarized parallel to *c* and yellow in light polarized perpendicular to *c*. We were not able to obtain suitable crystals for measurement of the axial spectrum.

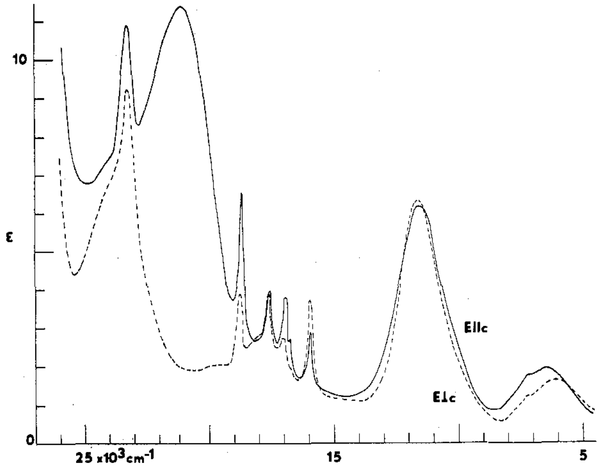

Figure 1. General spectrum of $CsCrCl_3$, 5000–26 000 cm^{-1}. The region 15 000–20 000 cm^{-1} is shown only in outline. ϵ is in units of l. mol^{-1} cm^{-1} throughout.

Crystal Data. The crystal structure of $CsCrCl_3$ has been determined[4] and is similar to that of the type of compound $CsNiCl_3$.[5] While the cell is hexagonal, $P6_3mc$, with $a = 7.256$ (3) Å, $c = 6.224$ (3) Å, $CsCrCl_3$ differs however in that the site symmetry of the Cr^{2+} ion is C_{3v} ($3m$) and not D_{3d} ($\bar{3}$m).

The distortion takes the form of a small displacement of the Cr along the chain axis so that three Cr–Cl distances are 2.419 (5) Å and three are 2.618 (6) Å, with corresponding Cr–Cr–Cl angles within each group of 90.1 (2) and 81.7 (2)°, respectively. This distortion is presumably connected with the Jahn–Teller effect but does not of itself split the degenerate E level; this can only occur in this compound by a vibrational mechanism for which there is some evidence from anisotropic motion of the chloride ions parallel to the *c* axis.

The structure of $RbCrCl_3$ has not been determined, but preliminary x-ray photographs indicate a similar unit cell to that of $CsCrCl_3$, with $a = 7.06$ (3) Å and $c = 6.24$ (3) Å.

No complete magnetic data exist for either compound although $CsCrCl_3$ is reported to be antiferromagnetic below 108 K.[9]

Spectroscopic Measurements. The σ and π single-crystal absorption spectra of $CsCrCl_3$ and $RbCrCl_3$ were measured at various temperatures between 300 and ~4 K in the spectral range 5000–30 000 cm^{-1}, using techniques described in ref 1. Oscillator strengths were determined as in ref 1; while internal consistency is probably ~5%, due to inaccuracies in the measurement of crystal thickness and estimation of band areas, the absolute values are not likely to be accurate to better than 15–20%.

Results

The spectrum of $CsCrCl_3$ at 75 K is similar to that previously reported,[4] but there are significant changes at 4 K (Figures 1–3); the spectrum of $RbCrCl_3$ is generally similar to this but shows some unexpected variations in detail (Figures 2–5). The observed energy levels and oscillator strengths are listed in Table I.

The spectra can best be described in terms of four regions of absorption. From 5000 to 15 000 cm^{-1} there are two strong broad unstructured absorptions, attributed (see below) to the $^5E \rightarrow {}^3T_1$ and $^5E \rightarrow {}^5T_2$ transitions; from 15 000 to 19 000 cm^{-1} there is a complex group of weak bands; 19 000–25 000 cm^{-1} is occupied by the double exciton; the charge-transfer absorption occurs beyond about 25 000 cm^{-1}. In region I, the main bands at about 11 500 cm^{-1} are entirely structureless. The weaker bands, whose peak positions depend slightly on

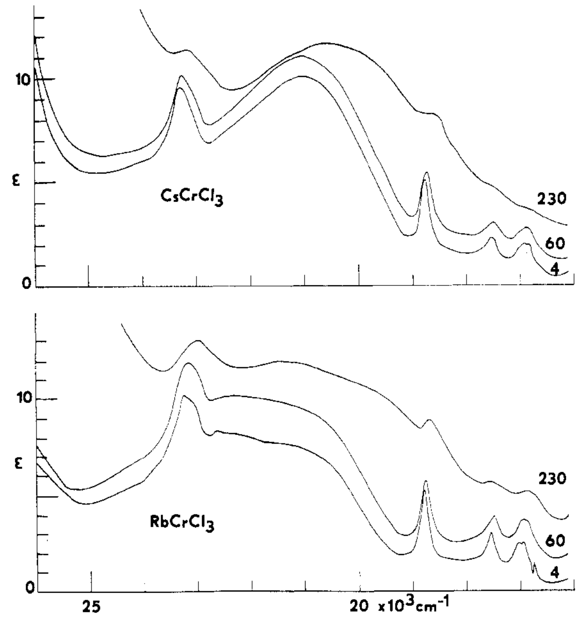

Figure 2. Spectra of $CsCrCl_3$ and $RbCrCl_3$, in the range 16 000–26 000 cm^{-1}, at 4, 60, and 230 K. The spectra are arbitrarily displaced upward; $E \parallel c$.

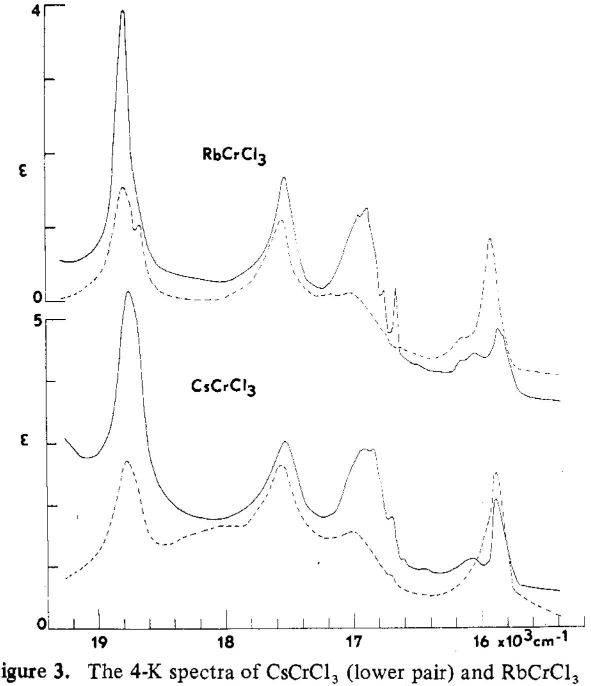

Figure 3. The 4-K spectra of $CsCrCl_3$ (lower pair) and $RbCrCl_3$ (upper pair) in the range 15 400–19 200 cm^{-1}: $E \parallel c$, solid lines; $E \perp c$, dashed lines. (ϵ is at somewhat different scales for the lower and upper curves.)

the polarization direction, do show a small shoulder at ~7200 Å, strongest for $RbCrCl_3$ ($\perp$). There is some beam imbalance due to the prisms in this region, but the baseline does not duplicate this, and this peak seems too large to be spurious. However, care should be exercised in attaching significance to this absorption. The temperature dependence of the bands has been measured in $CsCrCl_3$ (Figure 6) and shows the pattern expected for a vibronically assisted electronic transition. Qualitatively, the other bands show the same behavior.

For $CsCrCl_3$, the region 19 000–26 000 cm^{-1} (Figure 2) is fairly simple, with one main band at 21 000 cm^{-1}, the double exciton, which is observed only with $E \parallel c$, and a sharp strong peak at 23 300 cm^{-1} in both polarizations. There is also a very weak shoulder at 24 400 cm^{-1} ($E \parallel c$) and 24 150 cm^{-1} ($E \perp c$) which is rather more intense with $E \perp c$. Two weak absorptions occur in the $E \perp c$ spectrum at 22 400 and 19 900 cm^{-1}. For $RbCrCl_3$, however, this area is much more complex. The sharp peak at 23 250 cm^{-1} remains a constant feature, but the high-frequency shoulder (23 980 cm^{-1}) is much more intense with $E \perp c$, though still only just detectable with $E \parallel c$. There is also a weak band at 21 050 cm^{-1} with $E \perp c$, in the region of the double exciton. The latter, again only present with $E \parallel c$, is clearly structured, with a maximum at 22 670 cm^{-1} and a strong shoulder at 21 280 cm^{-1}.

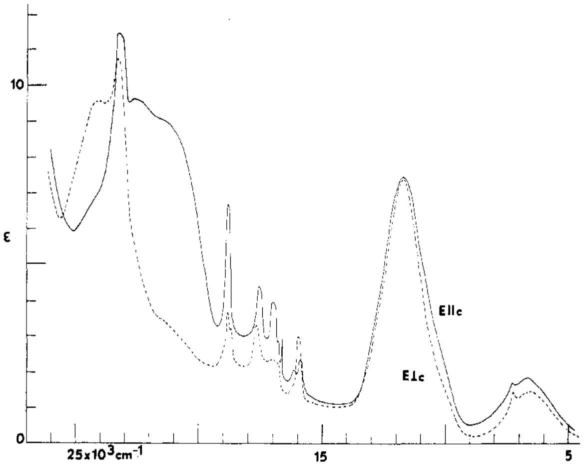

Figure 4. General spectrum of $RbCrCl_3$, as in Figure 1.

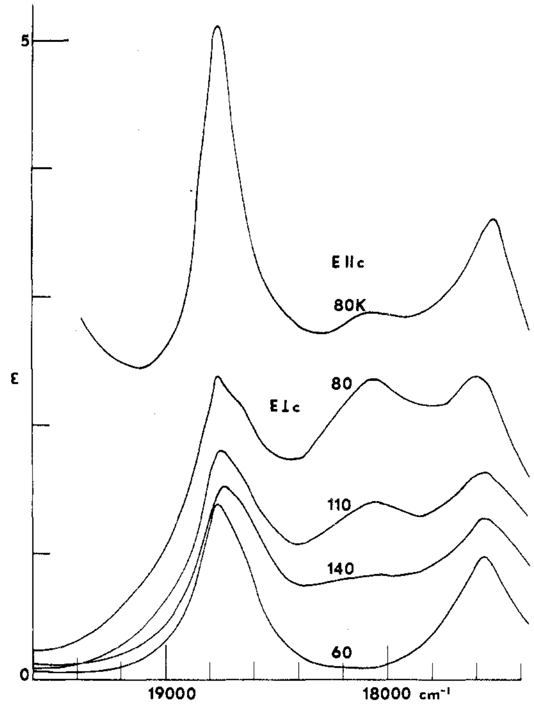

Figure 5. Spectrum of $RbCrCl_3$, in the range 17 400–19 600 cm^{-1}. $E \perp c$ at 60, 80, 110, and 140 K; $E \parallel c$ at 80 K.

The temperature dependence of the $CsCrCl_3$ double exciton band is shown in Figure 6. Although there are considerable difficulties in assigning a meaningful baseline, both because of the charge-transfer band and because of the overlap with the peak at 23 300 cm^{-1}, the constant intensity of the band can be established to very satisfactory accuracy because the entire band profile shows no change over a substantial temperature range. For $RbCrCl_3$, the intensity variation could not be established satisfactorily because of the structure in the band and because of the much greater contribution from the peak at 23 250 cm^{-1}.

Table I. Frequencies (cm^{-1}) and Oscillator Strengths ($l.\ mol^{-1}\ cm^{-2}$) of Absorptions in $CsCrCl_3$ and $RbCrCl_3$ at 4 K[a]

$CsCrCl_3$				$RbCrCl_3$			
$E \parallel c$		$E \perp c$		$E \parallel c$		$E \perp c$	
ν	$10^5 f$	ν	$10^5 f$	ν	$10^5 f$	ν	$10^5 f$
6 490 (80)	1.11	6 250 (80)	0.95	6 710 (80)	1.31	6 580 (80)	1.22
7 220 (40) w sh		7 220 (40) w sh		7 220 (40) w sh		7 220 (40) w sh	
11 500 (50)	4.84	11 630 (50)	3.91	11 730 (50)	6.06	11 700 (50)	5.64
						15 060 w?	
						15 418 w?	
				15 818 (20) sh	0.09		
15 893 (20)	0.082	15 893 (20)	0.20	15 886 (20)		15 936 (10)	0.15
16 090 (20)	0.016	16 000 (20) w sh		16 077 (20)		16 155 w sh	
16 434 (10)	0.003			16 181 (20)			
16 608 (10) sh	0.23			16 515 (20) w			
16 706 (10) sh				16 772 (5)	0.008	16 666 (20) w sh	
16 801 (10) sh		16 708 (20) sh		16 773 (20) sh	0.21	16 750 (20) w sh	0.07*
16 814 (10)				16 829 sh?			
			0.08*	16 871 (20) sh			
				16 906 (5)			
16 915 (10)				16 964 (5)		17 021 (20) sh	
		17 007 (20)		17 094 sh?		17 197 (20) w	
				17 241 (20) w			
17 534 (10)	0.17	17 569 (10)	0.12*	17 528 (10)	0.14	17 553 (10)	0.13*
				17 794 sh?			
		18 067 (20) w	0.015*	18 622 w sh		18 764 (20) sh	0.17
18 755 (10)	0.34	18 772 (10)	0.16	18 797 (5)	0.31	18 797 (5)	
		19 920 (15) w	0.05*				
21 010 (100)	5.72*			21 277 sh	5.0*	21 050 (70) w sh	0.07*
		22 421 (20) w sh		22 676 (50)			
			4.54	23 095 (50) sh			
23 310 (50)	4.5**	23 228 (50)		23 256 (50)	4.2**	23 255 (50)	4.2*
24 390 (100) w sh	0.06*	24 154 (50) w sh	0.12*	24 390 (60) w sh	0.07*	23 980 (50) sh	0.60*

[a] The errors in frequency are the maximum errors in locating the peak. The absolute errors in oscillator strength are of the order of 15–20% but the relative errors for peaks that are not overlapped are about 10%. Values marked with one or two asterisks are less or much less accurate than this; sh = shoulder, w = weak, ? = possible peak.

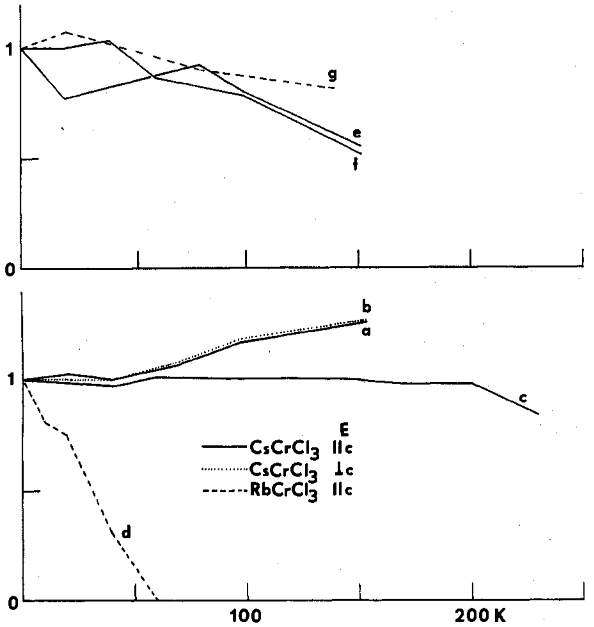

Figure 6. Temperature dependences. All values have been normalized to 1.0 for the oscillator strength at 4 K: (a) $CsCrCl_3$, 11 500 cm^{-1}, $E \parallel c$; (b) $CsCrCl_3$, 11 600 cm^{-1}, $E \perp c$; (c) $CsCrCl_3$ "pairwise excitation", 21 000 cm^{-1}, $E \parallel c$; (d) $RbCrCl_3$, 16 650 cm^{-1}, $E \parallel c$; (e) $CsCrCl_3$, 18 700 cm^{-1}, $E \parallel c$, thick crystal; (f) $CsCrCl_3$, 18 700 cm^{-1}, $E \parallel c$, thin crystal; (g) $RbCrCl_3$, 18 700 cm^{-1}, $E \parallel c$.

The 15 000–19 000-cm^{-1} region (Figure 3) is much more complex at 4 K than was observed at 77 K. The spectra of $CsCrCl_3$ and $RbCrCl_3$ are generally similar but contain some significant differences. There are four main manifolds, superimposed on a rather uneven background that may contain further unresolved bands. From high energy, the first peak is at 18 750–18 800 cm^{-1}. It is present in both polarizations but is considerably stronger for $E \parallel c$; the analogous band in $RbCrCl_3$ with $E \perp c$ has a shoulder at 18 675 cm^{-1}. The next band, at 17 520–17 550 cm^{-1}, has a somewhat unsymmetrical profile and is slightly more intense for $E \parallel c$. There is a weak intermediate peak (for $CsCrCl_3$, $E \perp c$ only) at 18 070 cm^{-1}. The third band (17 010–17 020 cm^{-1}) appears only as a shoulder with $E \perp c$, with very weak and uncertain shoulders at 17 200, 16 750, 16 670 (Rb) and 16 710 cm^{-1} (Cs). With $E \parallel c$ however, it is strong and has considerable structure including a doubling of the peak itself. The spacings of these fine-structure peaks are possibly consistent with vibrational progressions of 100 cm^{-1} for $CsCrCl_3$ and 50 cm^{-1} for $RbCrCl_3$. The strongest peak in this group is a very sharp one at 16 670 cm^{-1} for $RbCrCl_3$. It shows a marked temperature dependence (Figure 6) decreasing in intensity very rapidly as the temperature is raised, until it is almost nonexistent at 40 K. The final band at 15 900 cm^{-1} is substantially stronger with $E \perp c$ than with $E \parallel c$ and is also somewhat structured. For $RbCrCl_3$ with $E \perp c$, there is a shoulder at 16 150 cm^{-1} and a similarly placed peak also occurs in both parallel spectra (16 080–16 090 cm^{-1}). There is also a shoulder at 15 820 cm^{-1} for $RbCrCl_3$, $E \parallel c$.

Apart from the peak at 16 670 cm^{-1} already mentioned, most of this group show no very marked temperature dependence, becoming broader and less well resolved as the temperature is raised, but with no dramatic changes in intensity. There are two exceptions. For $CsCrCl_3$, the peak with $E \parallel c$ at 18 750 cm^{-1} when examined with a thick crystal (0.042 cm) shows a rapid decline in intensity, losing 20% between 4 and 20 K; however, with a thin crystal (0.0067 cm), the intensity appears to be constant in this range, only falling slowly as the temperature increases (Figure 6). Similarly, for

$RbCrCl_3$ ($E \parallel c$) with a crystal 0.025 cm thick, the intensity does not alter rapidly. However, this apparent discrepancy may be the result of the differences in overlap between overlapping bands in the more intense and less intense spectra.

The other anomaly is for $RbCrCl_3$ with $E \perp c$, where a quite substantial fairly broad peak at 18 080 cm^{-1} appears suddenly as the temperature is raised from 60 to 80 K but disappears by 140 K (Figure 5). A much weaker peak in the same position with the same temperature dependence is also visible with $E \parallel c$.

Discussion

Assignments. Two relevant calculations are available, both using the approximation of octahedral symmetry. Konig and Kremer[6] have presented graphically the results of the calculation of the energy levels of d^4 and d^6 systems including spin–orbit interaction while McPherson et al.[4] have assigned the spectrum of $CsCrCl_3$ on both strong-field and weak-field models, but excluding spin–orbit effects. The most obvious drawback of the second approach is that the strong band at 23 250 cm^{-1} is unassigned (weak field) or assigned to a quintet–singlet transition (strong field). We have reexamined this calculation, using as they did the weak-field matrices of Ferguson, Guggenheim, and Krausz.[7] It is clear that the assignment of the 11 600-cm^{-1} band as the $^5E \rightarrow {}^5T_2$ transition of the 6500-cm^{-1} band $^5E \rightarrow {}^3T_1(a)$ excitation must be correct. This immediately gives the value of $10Dq$ as ~11 600 cm^{-1}. Either by following McPherson,[4] using 90% of the free-ion term energies, or with $B = 570\ cm^{-1}$, $C = 3400\ cm^{-1}$, four bands in the range 15 800–18 800 cm^{-1} can be fitted fairly well to the transitions: $^3T_1(b)$ (15 786 cm^{-1}), $^3E(a)$ (16 440 cm^{-1}), $^3T_2$ (17 982 cm^{-1}), and $^3A_1$ (18 730 cm^{-1}).

It has become apparent from the present work, however, that the 15 800–19 000-cm^{-1} region is considerably more complex than was previously known, and the assumption of only four transitions in this region is not necessarily correct. Also, the spin–orbit calculations[6] show that the $^3T_1(a)$ and $^5T_2$ manifolds are hardly split by spin–orbit interaction but that the 15 000–20 000-cm^{-1} region becomes very complex, sufficiently so to account for the transitions observed. Parameter values $Dq = 1150\ cm^{-1}$, $B = 800\ cm^{-1}$, and $C = 3200\ cm^{-1}$ with the peak field model produce the following values (cm^{-1}): $^3T_1(a)$, 6574; $^5T_2$, 11 500; $^3T_1(b)$, 15 862; $^3E(a)$, 16 724; $^3T_2$, 18 298; $^3A_1$, 20 000; $^3A_2$, 20 029; $^3E(b)$, 23 031. This is our view gives the most satisfactory agreement possible in this very simple approximation, providing an assignment of the 23 000-cm^{-1} peak to $E \rightarrow {}^3E(b)$, with three absorptions between 15 900 and 18 300 cm^{-1}. For any further progress, both spin–orbit coupling and the C_{3v} site symmetry would need to be considered, but given the degree of resolution in our spectra, little unambiguous progress can be made.

Temperature Dependence. While the various intercombination bands display enhanced intensity as has been observed in other magnetically coupled ABX_3 systems, there is only a slight dependence of intensity on temperature (with one or two exceptions). This is in contrast to the results obtained for $CsMnBr_3$[8] and $(CH_3)_4NMnBr_3$.[9] The two exceptions are the manifold at 18 080 cm^{-1} in the spectrum of $RbCrCl_3$ and the peak at 16 670 cm^{-1} in $RbCrCl_3$. The 18 080-cm^{-1} band does not appear until ~60 K and disappears by 140 K. On the basis of earlier experience[1,8,9] we may conclude that this transition is magnon "hot band" assisted but we note that it is surprisingly broad. The second strikingly temperature-dependent feature, the 16 670-cm^{-1} band, is relatively intense at 4.2 K yet is nonexistent at 40 K. It appears that this peak is the 0 → 0, no-phonon origin of the manifold to its high energy side which is comprised of an ~100-cm^{-1} progression of symmetric phonons. The maximum at ~16 935 cm^{-1} which does not fall in this progression may be a component of a second symmetric progression of ~250 cm^{-1} as might the ill-resolved, high-energy shoulder of the band located at 17 220 cm^{-1}. The temperature dependence of the 16 670-cm^{-1} origin is then due to thermal broadening by the contributions of phonon hot bands. This seems to be borne out by the red shift observed in the low-energy absorption edge between 4.2 and 40 K.

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Registry No. $CsCrCl_3$, 13820-84-3; $RbCrCl_3$, 13820-90-1.

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Synthesis and Some Crystal Data of H_xReO_3 ($x = 0.15$)

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Rhenium trioxide is known as a chemically stable compound in air at room temperature.[1] Unidentified x-ray diffraction peaks, however, were found after 1 h of grinding an ReO_3 powder sample in guaranteed reagent grade ethyl alcohol in an agate mortar and they became bigger and clearer with the length of grinding time. While grinding in carbon tetrachloride (guaranteed reagent grade) for 4 h at room temperature yielded no diffraction peaks other than those of ReO_3, the clearly split diffraction peaks from those of ReO_3 were seen in the case of grinding in distilled water for 0.5 h. The above experiments suggested that some chemical reaction of ReO_3 powder with water occurred in the grinding process at room temperature, so we boiled ReO_3 powder in water at 100 °C for several hours and obtained a new compound.

The chemical composition was determined to be H_xReO_3 ($x = 0.15 \pm 0.04$) by means of mass spectroscopy and thermogravimetric analysis (tga).

The purpose of the present paper is to report the synthesis and some crystal data of H_xReO_3.

Experiments and Results

(A) Sample Synthesis. Re metal powder (99.99% purity, Herman C. Stark, Berlin) was employed as a starting material, and ReO_3 was synthesized and identified as a single phase by x-ray diffraction studies.[2] The ReO_3 thus obtained was ground under distilled water in an agate mortar for about 10 h, and 2–3 g of ReO_3 was boiled with about 300 ml of water in a Pyrex glass vessel. The change of the boiling time from 5 h to 1 week did not bring about any change in *d* spacings and relative intensities of x-ray diffraction peaks of the synthesized compound. The gold-colored compound obtained was elec-