Spectra of Tetrahedral Complexes of Transition Metals. Jahn–Teller Effect in the Tetrabromoferrate(III) Ion

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The single-crystal spectra of $RFeBr_4$ (R = 4-ethylpyridinium, Me_4N^+, Me_3NH^+, $Me_2NH_2^+$, and $MeNH_3^+$) have been recorded at room temperature and *ca.* 80 K. The fine structure resolvable in one transition has been analysed as vibrational progressions of non-totally symmetric t_2 modes. The consequences of this observation are discussed in terms of a Jahn–Teller distortion of the excited electronic state. It is shown that a consistent assignment of the excited states is possible with the aid of a crystal field energy calculation. The spin–orbit splittings of the lowest-lying excited quartet and doublet states are calculated from the Tanabe–Sugano–Schroeder–Tree matrices. It is suggested that the Jahn–Teller effect is responsible for a quenching of the expected spin–orbit splittings. Finally, the spectral differences between the isoelectronic $MnBr_4^{2-}$ and $FeBr_4^-$ complexes are discussed.

A RELATIVELY large number of papers[1] have appeared on the optical spectroscopy of the $FeCl_4^-$ complex. With few exceptions the work has been confined to room-temperature solution studies which produced broad, diffuse bands, which were not readily analysable. Recently we reported[1] our investigation of the low-temperature, single-crystal spectra of a number of alkylammonium tetrachloroferrate(III) systems. Fine structure was resolved and analysed for one of the bands in this study and a consistent assignment of the entire spectrum achieved by means of crystal field spin–orbit computations. Comparison of the spectra of the isoelectronic $FeCl_4^-$ and $MnCl_4^{2-}$ complexes[2] shows, however, that there are fundamental differences between the

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[1] M. Vala and P. J. McCarthy, *Spectrochim. Acta*, 1970, **28**, *A*, 2183, and references therein.

[2] M. Vala, C. J. Ballhausen, R. Dingle, and S. L. Holt, *Mol. Phys.*, 1972, **23**, 217.

two. For the $MnCl_4^{2-}$ complex the band positions, shapes, and vibrational fine structure are what one would expect; this is not true for the $FeCl_4^-$ complex. In order to understand these differences better we have extended our previous study to the $FeBr_4^-$ complex. A later report will deal with the mixed halides. In this paper we report the low-temperature, single-crystal spectra of the tetrabromoferrate(III) complex with a number of different cations. The 17—18 kK region in these crystals displays fine structure which is analysed as progressions in two non-totally symmetric vibrational modes. This observation together with a crystal-field calculation is used to assign the state and to show that the excited electronic state is distorted *via* Jahn–Teller forces. The quenching of the spin–orbit splitting in this state as the result of the Jahn–Teller effect is also suggested. Finally, some of the spectral differences between $MnBr_4^{2-}$ and $FeBr_4^-$ (and by analogy, $MnCl_4^{2-}$ and $FeCl_4^-$) are discussed.

EXPERIMENTAL

$[Me_4N][FeBr_4]$ was prepared from stoicheiometric amounts of anhydrous $FeBr_3$ and Me_4NBr in 5M-HBr. Small, flat, irregular, deep red crystals were grown by slow solvent evaporation at room temperature. They exhibited no noticeable dichroism, but did extinguish under crossed polarizers.

Each of the four compounds $RFeBr_4$ [R = Me_3NH^+, $Me_2NH_2^+$, $MeNH_3^+$, and ethylpyridinium ($EtpyH^+$)] was prepared similarly from an aqueous solution of the appropriate amine; the final solution was 4—5M in HBr. The Me_3NH^+ compound formed deep-red, irregular plates. The $Me_2NH_2^+$ compound produced either deep red, flat hexagons which showed complete extinction or long prisms with extinction along the major elongation axis. The $MeNH_3^+$ compound separated as deep-red rectangular plates which exhibited extinction parallel to the major crystal axes. Very thin plates of the latter could be grown. The $EtpyH^+$ compound formed irregularly shaped flat crystals which extinguished under crossed polarizers.

4-Ethylpyridinium tetrabromoferrate belongs to space-group $P2_1/c$ with unit-cell parameters: $a = 7{\cdot}7068$ Å, $b = 14{\cdot}1673$ Å, $c = 13{\cdot}0414$ Å, $\beta = 84{\cdot}19°$, $Z = 4$. The $FeBr_4^-$ tetrahedra are slightly distorted with the Fe–Br bond lengths ranging from 2·309 to 2·345 Å and the BrFeBr angles from 107·7° to 110·9°. The $FeBr_4^-$ groups are nearly aligned with their threefold axes almost coincident in the *c* direction.[3]

The absorption spectra were obtained on a Cary 14 spectrophotometer with a cold-finger liquid-nitrogen Dewar flask. The samples were mounted with Apiezon grease over small holes in a copper plate; the plate was attached to the cold finger. Sample temperatures were not monitored, but are estimated to be *ca.* 80 K. Compensation for the small sample size was made in the Cary reference compartment by a small adjustable iris diaphragm. Polarized spectra were obtained with a Glan–Thompson prism polarizer (Karl Lambrecht).

RESULTS AND DISCUSSION

Crystal Spectra.—The low-temperature optical spectrum of the $FeBr_4^-$ complex in an amorphous, thin film has been reported by Ginsburg and Robin.[4] A spectral analysis was attempted, but because of the diffuse nature of the observed bands, no conclusive assignment was possible.

The crystal spectra of the compounds which we have investigated are given in Figure 1 and the band positions in Table 1. The spectra are similar in broad outline to that reported by Ginsburg and Robin, but exhibit greater fine structure, particularly in the 17—18 kK region. In general, the spectra contain a broad, diffuse low-energy band between 11 and 12 kK, followed by three somewhat more intense peaks between 12·2 and 14·6 kK. Of the latter peaks the one at the highest energy is considerably broader than the other two and in some cases exhibits fine structure. Two closely spaced bands fall between 15·6 and 16·0 kK and are followed by a slightly more intense one at 16·4 kK. A progression of well-defined bands is easily discernible in the 17·0—18·0 kK range of all spectra. Although the general features of the spectra are similar, there are details in the band structure which depend on the particular cation.

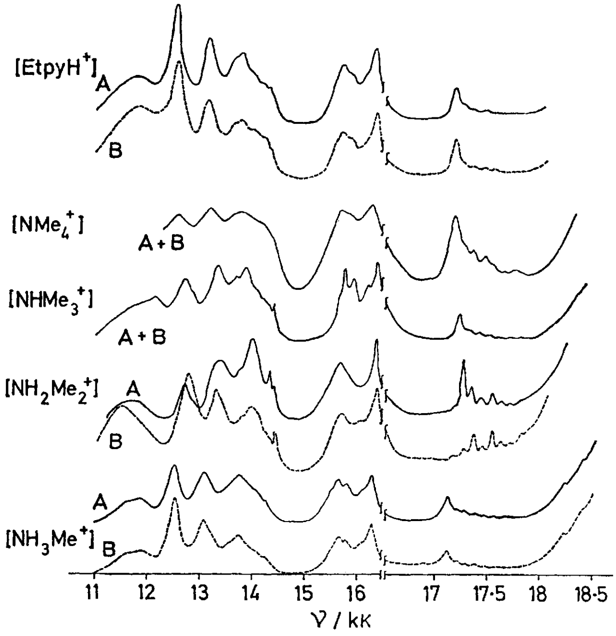

FIGURE 1 The single-crystal spectra of 4-ethylpyridinium $(EtpyH)FeBr_4$, Me_4NFeBr_4, $Me_3NHFeBr_4$, $Me_2NH_2FeBr_4$, and $MeNH_3FeBr_4$ at *ca.* 80 K. The *A* polarizations refer to the electric vector direction perpendicular to the main elongation axis of the crystal and the *B* polarization to the parallel direction

The spectra of the 4-ethylpyridinium salt are slightly different in the two polarizations, as expected from the distortion of the $FeBr_4^-$ tetrahedra from T_d symmetry. Similar differences are observed for the spectra of the

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[3] M. L. Hackert and R. A. Jacobson, *Acta Cryst.*, 1971, **B27**, 1658.

[4] A. P. Ginsburg and M. B. Robin, *Inorg. Chem.*, 1963, **2**, 817.

other compounds,* except for those of Me_4NFeBr_4, for which no polarization behaviour was observed. (This point is important and was carefully checked.) Since for a regular tetrahedral complex all components of the electric dipole transition moment transform as T_2, no polarization should be observed in the spectra. It is a reasonable assumption then that the chromophore in Me_4NFeBr_4 is undistorted.

TABLE 1

Observed band positions/kK and assignments for single-crystal spectra of tetrabromoferrate(III) anion (all data refer to both polarizations unless otherwise noted)

	$EtpyH^+$		Me_4N^+	Me_3NH^+	$Me_2NH_2^+$		$MeNH_3^+$
Assignment	Pol. A	Pol. B	Pol. A and B	Pol. A and B	Pol. A	Pol. B	Pol. A and B
$^4T_1{}^1$	11·80	11·86		11·80	—	11·51	11·60
	—	—		12·15	11·69	—	11·90
	12·55	12·58	12·59	12·72, 12·86	12·58, 12·73	12·78	12·53
	13·13	13·15	13·20	13·33	12·85, 13·41	13·30, 13·42	13·09, 13·19 (B)
	13·66, 13·79	13·65, 13·80	13·61, 13·78	13·70, 13·87	13·81, 14·00	13·80, 13·97	13·60, 13·75
						14·10	13·91 (A)
$^4T_2{}^1$	13·99	14·01	14·19	14·05, 14·21	14·28, 14·35	14·25, 14·34	14·06
	14·21	14·22	14·33	14·30, 14·40	14·44	14·44	14·23
	14·30	14·33	14·54	14·58	14·53, 14·63	14·53, 14·63	—
$^4A_1$, $^4E^1$	—	—	—	15·66	—	—	15·54
	15·71	15·71	15·70	15·78	15·69	15·70	15·66
	15·87	15·86	15·86	15·94	15·82	—	15·81
	16·12	—	—	16·22	—	16·06	16·12
	16·33	16·35	16·30	16·40	16·39	16·37, 16·46	16·28
$^4T_2{}^2$, $-\nu_3$	—	—	—	—	—	16·960	—
$-\nu_3 + \nu_4$	—	—	16·973	17·046	17·062	17·062	16·904
$-\nu_4$	—	—	—	17·155	17·177	17·177	17·023 (A)
$0 - 0$	17·174	17·169	17·182	17·244	17·263	17·263	17·115
$+\nu_4$	17·274	17·267	17·279	17·332	17·355	17·355	17·203
$+2\nu_4$	17·358	17·358	17·363	17·422	17·443	17·443	17·293
$+\nu_3$	17·464	17·460	17·475	17·531	17·546	17·546	17·402
$+\nu_3 + \nu_4$	17·565	17·559	17·561	17·616	17·632	17·632	17·492
$+\nu_3 + 2\nu_4$	—	—	17·661	17·705	17·724	17·724	17·572
$+2\nu_3$	—	—	17·770	17·820	17·827	17·827	17·687
$+2\nu_3 + \nu_4$	—	—	—	17·892	17·913	17·913	—
$^4E^2$	—	—	—	18·36	—	—	18·24, 18·41

Crystal-field Computations.—The ferric ion in $FeBr_4^-$ has a high-spin $3d^5$ electronic configuration with a sextet ground state, $^6A_1$, and only quartet and doublet excited states; all transitions are, to first order, spin-forbidden. The assignment of the various bands to specific excited electronic states can be aided by a calculation of the crystal-field energies for a tetrahedrally co-ordinated $3d^5$ metal ion.[5] To do this, we used our BESTFIT programme[1] which employs the Tanabe–Sugano matrices[6] modified to include Tree's correction term.[7] In this programme a range for the crystal-field strength, Dq, and the Racah electron repulsion integrals, B and C, is specified, together with the observed energies. The programme calculates all the quartet state energies for a given set of parameters, determines the r.m.s. deviation from the observed values, and ultimately finds the set of parameters which minimizes the r.m.s. deviation.

In this approach the closeness of the fit of the observed bands to theoretical energies depends obviously on a judicious choice of experimental band positions. If one attempts tentatively to assign the spectrum on the basis of a Tanabe–Sugano–Orgel type diagram, one quickly sees that the expected sharp transitions to the field-independent $^4A_1$,^4E states are not readily identifiable. (The isoelectronic $MnBr_4^{2-}$ complex yields a sharp band which is undoubtedly the $^4A_1$,^4E transition.) To gain a better feeling for the position of the $^4A_1$,^4E band we have investigated the low-temperature crystal spectra of the mixed halide complexes $FeBr_3Cl^-$ and $FeBrCl_3^-$, in addition to the published[1] $FeCl_4^-$. From the correlation of these spectra it is apparent that the bands in the region between 15 kK and 16·5 kK in $FeBr_4^-$ correspond to $^4A_1$,^4E and those in the 17—18 kK range to $^4T_2{}^2$. Thus, the bands below 15 kK in energy must be attributable to the $^4T_1{}^1$ and $^4T_2{}^1$ states. Since the peaks in this region do not admit of any vibrational analysis (see p. 1874), the identification of their respective origins and band systems is very difficult. (A possible reason for this is discussed later.) Rather than attempt a crystal-field calculation fitting three parameters (B, C, and Dq) to two known bands (^4E and $^4T_2{}^2$) we chose to include the approximate centre of gravity for the lower two bands ($^4T_1{}^1$, $^4T_2{}^1$). Table 2 gives the band energies chosen, the computed crystal-field

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* The polarization differences in the Me_3NH^+ cases are slight and are not shown in Figure 1.

[5] Anticipating our results a little, we note that our crystal-field calculation for a rigid tetrahedral geometry is justifiable even when Jahn–Teller distortion occurs, since the potential energy as a function of the nuclear co-ordinates retains the full (T_d) symmetry of the non-distorted point group; see G. Herzberg, 'Electronic Spectra of Polyatomic Molecules,' van Nostrand, Princeton, N.J. 1966, p. 51.

[6] Y. Tanabe and S. Sugano, *J. Phys. Soc. Japan*, 1954, **9**, 753.

[7] R. Stevenson, 'Multiplet Structure of Atoms and Molecules,' W. B. Saunders, Philadelphia, 1965.

energies, the assignments, and the optimum crystal-field parameters. Several combinations of band assignments were tested, the one with the lowest r.m.s. deviation between observed and calculated energies being chosen as the best fit (*i.e.*, assignment 1 in Table 2 with $\alpha = 90$). As in our previous calculation on $FeCl_4^-$ the inclusion of Tree's factor (α) results in much better agreement between observed and calculated values. With an r.m.s. deviation between the four observed and calculated energies of only *ca.* 100 cm^{-1}, we consider the crystal-field model valid in this case and capable of at least semiquantitatively predicting the energies of the different excited quartet states. Justification for this assignment can be obtained from a vibrational analysis of the $^4T_2{}^2 \longleftarrow {}^6A_1$ transition.

TABLE 2

Results of fitting $FeBr_4^-$ spectrum with ' Bestfit ' programme

	Observed (calc.) * energies/cm^{-1}			R.m.s. deviation/ cm^{-1}		Best parameters (α = 90·0)		
Assignment	$^4T_1{}^{1,2}$	$^4T_2{}^{1,2}$	$^4E^{1,2}$	α = 90·0	α = 0	B	C/B	Dq
1	12,500	14,300	15,690	97	346	470	3·9	380
	(12,434)	(14,426)	(15,665)					
	—	17,263	—					
	(19,151)	(17,178)	(17,695)					
2	11,600	14,300	15,690	256	442	475	3·8	425
	(11,885)	(14,125)	(15,575)					
	—	17,263	—					
	(19,341)	(16,996)	(17,640)					
3	12,500	14,300	16,390	232	629	450	4·4	450
	(12,453)	(14,604)	(16,200)					
	—	17,263	—					
	(20,377)	(17,439)	(18,090)					
4	11,600	14,300	16,390	427	766	475	4·0	450
	(12,206)	(14,488)	(16,050)					
	—	17,263	—					
	(20,104)	(17,429)	(18,115)					

* Calc. values in parentheses for α = 90.

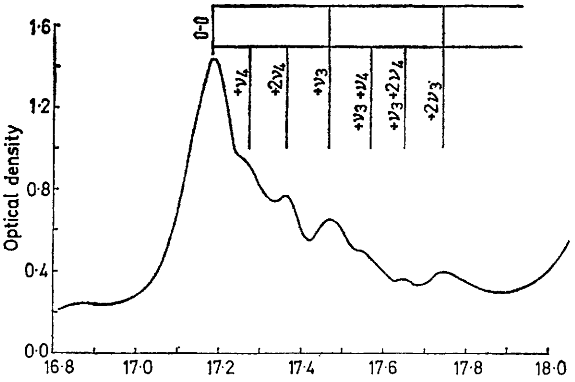

FIGURE 2 Region from 17,000 to 18,000 cm^{-1} of the polarized single crystal spectrum of Me_4NFeBr_4 at *ca.* 80 K

Vibrational Analysis of the 17—18 kK *Region.*—The fine structure in the 17—18 kK region of Me_4NFeBr_4 is shown in detail in Figure 2 together with a vibrational analysis. Two overlapping progressions are observed, one of *ca.* 292 cm^{-1}, the other of *ca.* 93 cm^{-1}.

The vibrational frequencies of the ground state of $FeBr_4^-$ have been studied by a number of workers, but most completely by Avery *et al.*[8] In solid Et_4NFeBr_4 a Raman band at 200 cm^{-1} has been assigned to the ν_1 symmetric breathing mode and two i.r. bands, one at 285 cm^{-1} and the other at 95 cm^{-1}, to the non-totally symmetric tetrahedron-distorting t_2 modes, ν_3 and ν_4. Thus the 93 cm^{-1} and 292 cm^{-1} excited state vibrational modes can be rather clearly assigned to the ν_4 and ν_3 non-totally symmetric t_2 vibrations, respectively.

In general, progressions in single quanta of non-symmetric vibrations do not appear unless the excited state is distorted in such a way that the non-symmetric vibration becomes totally symmetric in the lower symmetry of the distorted structure.[9] Jahn and Teller[10] have shown that the orbital degeneracy of an excited state may be lifted if the symmetric configuration of the state is unstable with respect to any of its non-totally symmetric normal modes. Of the three non-totally symmetric modes of a tetrahedral complex, one is of e and two are of t_2 symmetry. Electronic T_1 or T_2 states are unstable with respect to t_2 and e vibrational modes. We may conclude then that the bands between 17 and 18 kK in Me_4NFeBr_4 represent transitions to vibronic states associated with a Jahn–Teller distorted $^4T_2$ excited state. (Our crystal-field calculation eliminates the $^4T_1$ state from further consideration.)

Each of the other compounds studied shows a similar vibrational structure in this region with similar frequency intervals: $EtpyH^+$, 290, 95; Me_3NH^+, 288, 87; $Me_2NH_2^+$, 282, 88; $MeNH_3^+$, 281, 88 cm^{-1}. While it is tempting to ascribe these progressions to the Jahn–Teller distortion of a $^4T_2$ state, this is not possible at

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[8] J. S. Avery, C. D. Burbridge, and D. M. L. Goodgame, *Spectrochim. Acta*, 1968, **24**, *A*, 1721, and references therein.

[9] H. C. Longuet-Higgins, U. Öpik, M. H. L. Pryce, and R. A. Sack, *Proc. Roy. Soc.*, 1958, *A*, **244**, 1.

[10] H. A. Jahn and E. Teller, *Proc. Roy. Soc.*, 1937, *A*, **161**, 22.

present since the chromophore symmetry is unknown except for the EtpyH$^+$ salt. Quite possibly the Jahn–Teller effect and not static crystal effects are responsible for the distortion in these salts. This could occur if the static crystal effects reduced the complex symmetry to, say, either C_{3v} or D_{2d}; in both of these cases degenerate orbital symmetry remains. However, further speculation is unwarranted until the symmetry of the $FeBr_4^-$ complex in these salts is determined.

Evidence for the exact nature (geometry, energy, *etc.*) of the other excited electronic states in $FeBr_4^-$ is much weaker than for the $^4T_2{}^2$ state. This is the direct result of two circumstances. First, the presence of a large number of unassigned bands (especially in the 11—15 kK region) render our crystal-field calculations and assignments quite approximate. No convincing explanation for the large number of bands can be made until more information is available on the exact site symmetry of the chromophore and finer spectral resolution is obtained by measurements at lower temperatures. Secondly, when spin–orbit coupling is introduced, as it must be to explain the presence of spin-forbidden transitions, it can be shown that *every* excited quartet and doublet state of $FeBr_4^-$ (except $^2A_1$ and $^2A_2$) contains at least one $G_{3/2}$ spin–orbit component. $G_{3/2}$ states are susceptible to Jahn–Teller distortion by coupling with vibrations of either e or t_2 symmetry.[11] Thus, every quartet and most doublet states of $FeBr_4^-$ are theoretically capable of distortion *via* Jahn–Teller forces.

Not only is spin–orbit coupling important in intensity-gaining mechanisms of spin-forbidden transitions, but it may manifest itself in the splitting of certain origin bands.[2] (This is in contrast to octahedral complexes where parity selection rules prohibit such direct influence and where difficultly-interpretable spin–orbit–vibronic processes must be considered.) In order to determine the extent of spin–orbit splitting in the excited states of the $FeBr_4^-$ complex, the complete spin–orbit crystal field energies were computed by diagonalizing Schroeder's d^5 spin–orbit matrices[12] modified to include Tree's correction factor.[7] The results (Table 3) were obtained by use of the optimum Racah parameters of Table 2, plus $\zeta_{s.o.} = 420$ cm^{-1}, the free-ion spin–orbit interaction parameter. The calculated splittings are largest for the $^4T_2{}^2$ state, spanning a range of *ca.* 260 cm^{-1}. With a predicted splitting of this magnitude one might expect to observe it easily, provided the band width at the origin were sufficiently small. If we assume that the assignments given above are correct, the band width of the origin band of the $^4T_2{}^2$ state at 17,263 cm^{-1} in Me_4NFeBr_4 is *ca.* 100 cm^{-1}. Thus a spin–orbit splitting should be readily observed. It can be seen, however, that the spacing pattern of the observed bands does not fit the predicted spin–orbit splittings, but is analysed nicely as two vibrational progressions. From this one might conclude that this represents a transition to an orbitally non-degenerate state, in which the spin–orbit coupling is always very small.[13] But another conclusion can be drawn.

TABLE 3

Calculated spin–orbit crystal-field energies/cm^{-1} for $FeBr_4^-$ ($Dq = 380$ cm^{-1}, $B = 470$, $C/B = 3{\cdot}9$, $\alpha = 90{\cdot}0$, ζ s.o. $= 420$)

State	S.O. Comp.	Energy/cm^{-1}	State	S.O. Comp.	Energy/cm^{-1}
$^4T_1{}^1$	$E_{5/2}$	12,146	$^4T_2{}^2$	$G_{3/2}$	16,674
	$G_{3/2}$	+69		$E_{1/2}$	+41
	$G_{3/2}$	+147		$E_{5/2}$	+179
	$E_{1/2}$	+224		$G_{3/2}$	+261
$^4T_2{}^1$	$E_{5/2}$	14,461	$^2T_2{}^1$	$E_{5/2}$	17,070
	$G_{3/2}$	+58	$^4E^2$	$E_{5/2}$	17,513
	$E_{1/2}$	+189		$G_{3/2}$	+4
	$G_{3/2}$	+208		$E_{1/2}$	+70
$^4A,{}^4E_1$	$G_{3/2}$	15,566	$^2T_2{}^1$	$G_{3/2}$ *	17,750
	$E_{5/2}$	+8	$^4T_1{}^2$	$G_{3/2}$	19,110
	$E_{1/2}$	+47		$E_{5/2}$	+77
	$G_{3/2}$	+47		$G_{3/2}$	+158
				$E_{1/2}$	+208

* Partner to $E_{5/2}$ spin–orbit component of $^2T_2{}^1$ state at 17,070 cm^{-1}.

Ham[14] has shown that the dynamic Jahn–Teller effect can simulate covalent bonding by reducing the expectation values of certain electronic operators. In particular, he demonstrated that spin–orbit interaction, as well as other interactions involving orbital angular momentum operators, in triply degenerate states of cubic systems which couple with doubly and triply degenerate vibrations are quenched to unexpectedly small values. So the absence of any observable spin–orbit splitting of the 17 kK band origin does not negate our previous conclusion that this band represents a transition to a $^4T_2$ state, but rather enforces it in the light of Ham's discovery.

Although we have been able to observe a Jahn–Teller effect in only one of the excited states of $FeBr_4^-$, we suggest (without proof) that other excited states of this ion may also be Jahn–Teller distorted. As noted above, all of the excited states except $^2A_1$ and $^2A_2$ are capable of such distortion. Each electronic state will probably have a different vibronic coupling strength, which will result in bands of differing shape and spacing. This may explain the unusual spacings in the lower energy transitions. We have not been able to analyse these bands in terms of vibrational and/or spin–orbit intervals.

It is interesting to compare the spectrum of $FeBr_4^-$ with that of its isoelectronic partner, $MeBr_4^{2-}$. In the spectrum of the latter complex, all nine of the predicted d–d transitions are observed, some with well-developed progressions in the totally symmetric vibration built upon spin–orbit origins.[2] These excited states are therefore not Jahn–Teller distorted but retain the same tetrahedral symmetry as in the ground state. The fact that the fine structure on the $MnBr_4^{2-}$ bands is much more highly developed than in $FeBr_4^-$ is a further indication of the perturbation from the Jahn–Teller effect in the latter complex. This is not to say that the Jahn–Teller effect is not operative in $MnBr_4^{2-}$, but only

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[11] H. A. Jahn, *Proc. Roy. Soc.*, 1938, *A*, **164**, 117.

[12] K. Schroeder, *J. Chem. Phys.*, 1962, **37**, 1587.

[13] G. Herzberg, ref. 5, p. 52.

[14] F. S. Ham, *Phys. Rev.*, 1965, **138**, *A*, 1727.

that, if it is, it is much smaller than in $FeBr_4^-$. Since the Jahn–Teller effect is the result of a delicate balancing of electron–nuclear and nuclear–nuclear potential energy terms, the increased nuclear charge of the Fe^{3+} species greatly enhances the electron–nuclear interaction, thereby ensuring a larger distortion in the $FeBr_4^-$ complex.

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