Table II. Statistical and Observed Proportions in Equilibrium Mixtures of GaX_{4-n}^- Anions in Dichloromethane

	GaX_4^-	GaX_3Y^-	$GaX_2Y_2^-$	$GaXY_3^-$	GaY_4^-
statistical ratio	1	4	6	4	1
proportions	0.062	0.250	0.375	0.250	0.062
X = Cl, Y = Br	0.09	0.29	0.37	0.20	0.05
X = Br, Y = I	0.11	0.30	0.34	0.20	0.05
X = Cl, Y = I	0.12	0.21	0.29	0.23	0.15

Equilibrium Ratios. The relative proportions of the various gallium halide complexes present at equilibrium, as represented by the resonance signals in Figure 1B–D, require some comment. The amounts of each species, at a total gallium concentration of 0.4 M, were computed from integral traces and confirmed by measuring the areas under the peaks to give the results in Table II. The proportions deviate from the figures for statistical redistribution of the halide ligands, particularly for $GaBr_4^-/GaI_4^-$ and $GaCl_4^-/GaI_4^-$ mixtures. This result suggests that the GaI_4^- complex is kinetically the most stable of the tetrahalogenogallium anions under the conditions of these experiments, and as with the other results reported, the greater covalent contribution to bonding in the case of Ga–I bonds provides a probable explanation.

Hexahalogenogallate(II) Anions. The $Ga_2X_6^{2-}$ anions (X = Cl, Br, or I) have been prepared, as salts of various cations, and shown by vibrational spectroscopy and X-ray crystallography to have a staggered ethane-like configuration in the solid state and in solution.[13,22] We attempted to observe the ^{71}Ga spectra of concentrated solutions of $[(n\text{-}C_4H_9)_4N]_2[Ga_2X_6]$ (X = Cl, Br) in dichloromethane. No resonance was detected in the case of bromide, despite the fact that the presence of $Ga_2Br_6^{2-}$ ion in solution was confirmed by Raman spectroscopy. A solution of the analogous $Ga_2Cl_6^{2-}$ salt containing (identified) additional $GaCl_4^-$ gave only a strong single resonance due to this latter ion. No other signals could be observed even after collecting more than 100 times as many pulses as were necessary to detect $GaCl_4^-$ and even though the concentration of $Ga_2Cl_6^{2-}$ was approximately 5 times that of $GaCl_4^-$.

We conclude that the gallium resonance is broadened beyond the limit of detection by the electric field gradient which results from the unsymmetrical environment of the ^{71}Ga nucleus when the atom is bonded to another gallium (either ^{71}Ga or ^{69}Ga, both of which have spin $I = {}^3/_2$) as well as to halogen atoms. Equally, we find no evidence for the disproportionation

$$M_2X_6^{2-} \rightleftharpoons MX_2^- + MX_4^-$$

which was observed with $In_2X_6^{2-}$ anions.[7]

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Registry No. $GaCl_4^-$, 15201-06-6; $GaCl_3Br^-$, 44006-78-2; $GaCl_2Br_2^-$, 57300-64-8; $GaClBr_3^-$, 44006-76-0; $GaBr_4^-$, 17611-23-3; $GaBr_3I^-$, 44006-77-1; $GaBr_2I_2^-$, 62938-85-6; $GaBrI_3^-$, 44006-79-3; GaI_4^-, 19468-10-1; $GaCl_3I^-$, 44007-68-3; $GaCl_2I_2^-$, 62883-26-5; $GaClI_3^-$, 44007-69-4; $GaCl_2BrI^-$, 77320-92-4; $GaClBr_2I^-$, 77320-93-5; $GaClBrI_2^-$, 77320-94-6.

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[22] Oldham, C.; Khan, M.; Taylor, M. J.; Tuck, D. G. *Inorg. Nucl. Chem. Lett.* **1980**, *16*, 469.

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Hydrogen Fluoride Containing Isostructural Hydrates of Hexafluorophosphoric, Hexafluoroarsenic, and Hexafluoroantimonic Acids[1]

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The similar X-ray diffraction patterns of the solid "hexahydrates" of HPF_6, $HAsF_6$, and $HSbF_6$ indicate that these hydrates are probably isostructural, with the same cubic *Im*3*m* space group. The ^{19}F NMR spectra clearly show the presence of one molecule of HF for each hexafluoride anion. These solids appear to form a class of clathrate-like hydrates in which the disordered MF_6^- ions occupy tetradecahedral cages in a fully hydrogen-bonded host lattice consisting of H_2O, H_3O^+, and HF molecules, randomly distributed on the sites forming the cages.

It is generally assumed that the common crystal hydrates formed by HPF_6, $HAsF_6$, and $HSbF_6$ are hexahydrates.[2a] By an X-ray study Bode and Teufer[2b] found that the HPF_6 hydrate crystallizes in the cubic space group *Im*3*m* with the O atoms occupying the apices of a four-coordinated network forming the space-filling truncated octahedra of Fedorov. The PF_6^- anions occupy these 14-sided voids. This structure for $HPF_6 \cdot 6H_2O$ failed to fit the extra proton into a fully four-coordinated hydrogen-bonding scheme for the water molecules. It was observed by Davidson and Garg,[3] on the basis of ^1H and ^{19}F wide-line NMR and chemical composition studies, that the hydrate formed by addition of water to the commercial 65% HPF_6 solution is really $HPF_6 \cdot HF \cdot 5H_2O$. The presence of one HF molecule for each H_3O^+ ion in the lattice matches the number of H atoms to the number of hydrogen bonds and makes full hydrogen bonding possible. We now confirm this result and show that the "hexahydrates" of $HAsF_6$ and $HSbF_6$ appear to have the same clathrate structure and to incorporate HF in the lattice in the same way as the HPF_6 hydrate.

Experimental Methods

The HPF_6 hydrate crystals were prepared by the addition of water to commercial 65% HPF_6 as before.[3] The $HAsF_6$ hydrate used consisted of colorless crystals of Ozark-Mahoning "$HAsF_6 \cdot 6H_2O$".

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(1) Issued as NRCC No. 18823.

(2) (a) "Kirk-Othmer Encyclopedia of Chemical Technology", Vol. 9, 2nd ed., Interscience Publishers, New York, 1966, pp 550, 636; (b) H. Bode and G. Teufer, *Acta Crystallogr.*, **8**, 611 (1955); JCPD 13-569. (b) H. Bode and G. Teufer, *Acta Crystallogr.*, **8**, 611 (1955); JCPD 13-569.

(3) D. W. Davidson and S. K. Garg, *Can. J. Chem.*, **50**, 3515 (1972).

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Table I. X-ray Diffraction Patterns

	$HPF_6 \cdot HF \cdot 5H_2O^{a,b}$		$HAsF_6 \cdot HF \cdot 5H_2O^{c,d}$		$HSbF_6 \cdot HF \cdot 5H_2O^{c,e}$	
hkl	*d*, Å	*I*	*d*, Å	*I*	*d*, Å	*I*
110	5.43	80	5.43	100	5.51	100
200	3.84	55	3.85	40	3.88	50
211	3.125	100	3.125	80	3.180	80
220	2.714	4			2.752	10
					2.607	3
310	2.428	25			2.444	3
222	2.217	20				
321	2.052	35	2.056	60	2.078	70
400	1.919	8	1.921	10	1.944	10
411, 330	1.810	4	1.811	10	1.836	20
420	1.717	8	1.713	20	1.736	20
332	1.637	14	1.630	20	1.656	20
422	1.567	<1	1.567	5	1.584	10
510, 431	1.506	4	1.501	20	1.522	20
521	1.402	12	1.403	20	1.417	20
440	1.359	12	1.359	10	1.369	10
530, 433	1.317	<1	1.318	10	1.332	10
600, 442	1.280	6	1.281	10	1.293	10
611, 532	1.246	8	1.247	20	1.257	20
620	1.214	<1			1.227	3
541	1.185	2			1.197	10
622	1.158	<1				
631	1.132	4			1.143	10
444	1.108	2				
640	1.065	2			1.097	10
721, 633	1.045	6	1.044	20	1.056	10
732, 651	0.975	4			0.985	10
800	0.960	2				

[a] See ref 2. [b] $T = 291$ K, $a = 7.678$ Å. [c] This work, intensities roughly estimated. [d] $T = 170$ K, $a = 7.678 \pm 0.003$ Å. [e] $T = 170$ K, $a = 7.760 \pm 0.002$ Å.

A sample of "$HSbF_6 \cdot 6H_2O$" from the same source was not crystalline at room temperature and was found from its ^{19}F NMR spectrum to contain substantial quantities of SbF_5 as well as $HSbF_6$. Hydrate crystals were obtained by addition of HF in sufficient quantity to replace the HF which had been lost in the commercial sample.

Powder X-ray diffraction patterns, corrected for film shrinkage,[5] were obtained at ~170 K for the $HAsF_6$ and $HSbF_6$ hydrates with a flat-plate camera[4] using Zr-filtered Mo radiation of wavelength 0.710688 Å.

^{19}F NMR spectra of hydrates between ~220 K and the melting points were obtained at 169.38 MHz with a Bruker CXP-180 Fourier transform spectrometer. Samples were contained in Delrin tubes. Typically, 100 free-induction decay curves were accumulated at 10-s intervals after it was established that this pulse interval was long enough to avoid saturation; sample spinning was not employed. Spectra of the same samples were recorded immediately after melting. ^{19}F chemical shielding was measured relative to external CF_3COOH and converted to the more usual $CFCl_3$ shielding scale by addition of 78.5 ppm. A few ^{31}P and ^{75}As spectra were also recorded.

Results

The lines observed in the powder X-ray diffraction patterns of the $HAsF_6$ and $HSbF_6$ hydrates at ~170 K are compared in Table I with those found for the HPF_6 hydrate at 291 K by Bode and Teufer.[2] There is close correspondence in both spacings and estimated intensities in the three patterns which may all (except for one weak line of the $HSbF_6$ hydrate specimen studied) be assigned to the cubic space group *Im*3*m* found by Bode and Teufer.[2b] With allowance for temperature differences the unit cell dimension is about 0.5% larger for the $HAsF_6$ hydrate and about 1.6% larger for the $HSbF_6$ hydrate than for the HPF_6 hydrate at a similar temperature.

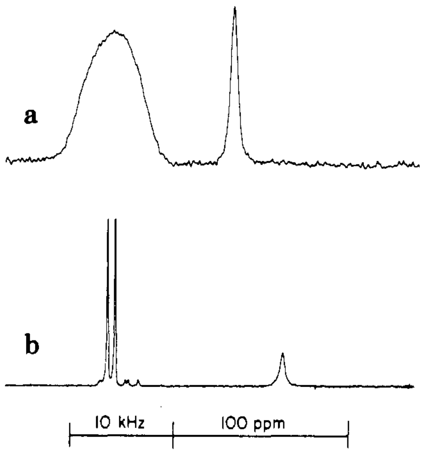

Figure 1. ^{19}F spectra of $HPF_6 \cdot HF \cdot 5H_2O$: (a) solid at 273 K, (b) liquid at 300 K.

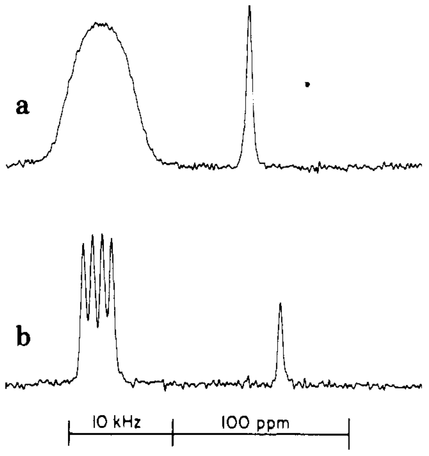

Figure 2. ^{19}F spectra of $HAsF_6 \cdot HF \cdot 5H_2O$: (a) solid at 298 K, (b) liquid at 315 K.

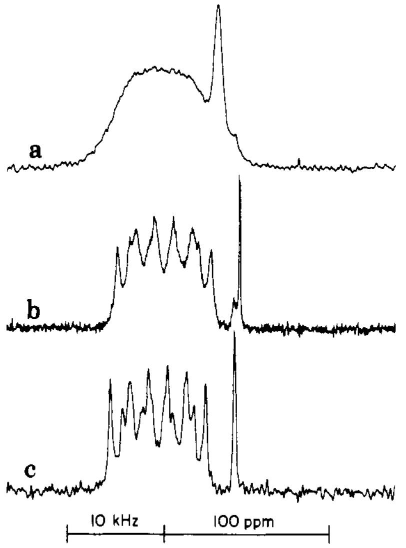

Figure 3. ^{19}F spectra of $HSbF_6 \cdot HF \cdot 5H_2O$: (a) solid at 283 K, (b) liquid at 308 K, (c) solution in dimethyl sulfoxide at 300 K.

^{19}F NMR spectra of the three hydrates and their fresh melts are shown in Figures 1–3. For each hydrate the two bands may be identified with the hexafluoride ion and HF. The ^{19}F nuclear shielding values (σ, Table II) of the hexafluoride ions in the hydrates are hardly changed in the melts, where the splitting patterns are those characteristic of octahedral PF_6^-, AsF_6^-, and SbF_6^- groups with *J* coupling to ^{31}P ($S = ^1/_2$), ^{75}As ($S = ^3/_2$), and the two isotopes ^{121}Sb ($S = ^5/_2$) and ^{123}Sb ($S = ^7/_2$), respectively. The splittings are given in Table II. In

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(4) J. E. Bertie, L. D. Calvert, and E. Whalley, *J. Chem. Phys.*, **38**, 840 (1963).

(5) D. F. Sargent and L. D. Calvert, *J. Phys. Chem.*, **70**, 7689 (1966).

Table II. ^{19}F NMR Lines in Hydrates and Melts[a]

	hydrate		melt			lit		
	T, K	σ	T, K	σ	J	σ	J	ref
PF_6^-	273	73.5	300	72.3	710	73.0	710	7
HF	273	141.8	300	169.7				
	253	141.0						
AsF_6^-	298	65.0	315	64.4	930	65.2	933	14
HF	298	149.9	315	167.7				
	253	147.7						
SbF_6^-	283	119.6	308	122.2	1950[b] 1053[c]	120.1	1946[b] 1053[c]	15
HF	283	153.8	308	168.1				

[a] σ is shielding in ppm relative to $CFCl_3$; J is multiplet splitting in Hz. [b] Coupling to ^{121}Sb. [c] Coupling to ^{123}Sb.

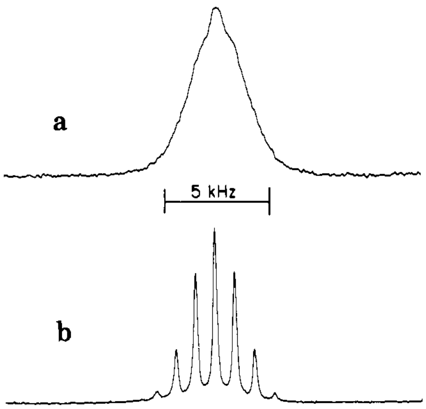

Figure 4. ^{75}As spectra of $HAsF_6{\cdot}HF{\cdot}5H_2O$: (a) solid at 298 K, (b) liquid at 315 K.

Table III. ^{19}F Second Moments of MF_6^- Ions in $HMF_6{\cdot}HF{\cdot}5H_2O$ at 290 K

		second moments, G²			
		contribution		total	
anion	a, Å	inter MF_6^-	J coupling	calcd	exptl
PF_6^-	7.678	0.273	0.008	0.28	0.27 (273 K)
AsF_6^-	7.719[a]	0.264	0.067	0.33	0.32 (298 K)
SbF_6^-	7.802[a]	0.256	0.551	0.81	0.74 (273 K)

[a] 0.53% larger than value measured at 170 K; expansivity assumed to be similar to that of ice.

the SbF_6^- case the 14-line pattern is somewhat better defined when the hydrate is dissolved in dimethyl sulfoxide (Figure 3c). In the melts the σ values of HF are similar to those found previously in aqueous solution (e.g., about 163 ppm relative to $CFCl_3$ for 10–20 wt % HF[6]). In the hydrates the HF lines are shifted appreciably down field, presumably because of the reduced shielding resulting from more extensive hydrogen bonding. These shifts serve the useful purpose of ensuring that the HF present in the hydrate crystals is not the result of occluded or adhering liquid.

Four very weak lines in the ^{19}F spectrum of the HPF_6 hydrate melt (Figure 1b) are possibly due to oxyfluorophosphate ions small enough to fit into the tetradecahedral lattice cavities.[3] Thus the two lines at highest fields appear to correspond to the doublet found by Gutowsky et al.[7] in liquid HPF_2O_2. No impurity lines were observed for the $HAsF_6$ hydrate in either the ^{19}F spectra (Figure 2) or the ^{75}As spectra (Figure 4), which in the melt consisted only of the septet expected for the octahedral AsF_6^- ion with $J = 930$ Hz. A small unidentified ^{19}F line occurred about 11 ppm upfield from the HF line in the $HSbF_6$ hydrate.

Within experimental error the PF_6^-:HF and SbF_6^-:HF ratios of the integrated intensities of the ^{19}F lines of the hydrates were 6:1, while the AsF_6^-:HF ratio was consistently nearer to 7:1.

Discussion

The earlier wide-line NMR study[3] of the HPF_6 hydrate showed a ^{19}F line-narrowing region centered near 45 K, ascribed to PF_6^- reorientation, and ^1H and ^{19}F line narrowing near 135 K attributed[3] to the onset of proton diffusion. Between 170 and 230 K the ^{19}F second moment of 0.7–0.8 G² (at 17 MHz) was attributed to superimposed lines of PF_6^- (calculated second moment 0.49 G²) and HF (calculated second moment 2.02 G²). (The corresponding proton second moment was less than 0.1 G².) The present ^{19}F measurements at 10 times the previous magnetic field show PF_6^- and HF peaks which are partly overlapped at 230 K but which become completely separated as the HF line becomes narrower with rise in temperature. In this region of line narrowing, which is attributed to the onset of diffusion of the F atoms in HF, the HF line width changes from 1.4 G at 235 K to 0.20 G at 273 K.

The experimental ^{19}F second moment of PF_6^- of 0.27 G² at 273 K is close to the value calculated (Table III) for the model in which all direct dipole–dipole contributions have been removed by proton and F atom diffusion except for the interactions between PF_6^- ions isotropically rotating about the cage centers, which are located at *bcc* sites in the lattice.[2] A further contribution[8] of $SM(J) = S(S+1)J_{PF}^2/(3\gamma_F^2)$ arises from J coupling with the ^{31}P nucleus ($S = {}^1/_2$).

Similarly calculated second moments for the isostructural $HAsF_6$ and $HSbF_6$ hydrates are compared in Table III with the experimental values for these hydrates.

The good agreement with the model in all three cases indicates that similar motional processes occur at relatively high temperatures in these hydrates. The ^{19}F line of HF in the $HAsF_6$ and $HSbF_6$ hydrates broadens with cooling below 260 K as it does for the HPF_6 hydrate and the diffusion rates of the F atoms therefore appear to be comparable.

As already observed,[3] the narrowing of the ^{19}F spectrum of the PF_6^- group occurs over a wide temperature range (17–80 K) and takes the form of a gradual increase in intensity of the narrow line from rotating PF_6^- at the expense of the broad line from rigid PF_6^-. As previously indicated,[9] this behavior provides an example of the Resing apparent-phase change effect[10] associated with the presence of a broad distribution of reorientational correlation times. The narrow line does not require isotopic reorientation in the usual sense since any sufficiently rapid reorientational process which results in interchange of the 24 physically indistinguishable configurations (except for spins) of an octahedral group will average the intragroup dipole–dipole interactions to zero, regardless of the number of physically distinct preferred orientations. The distribution of reorientation times must therefore result from differences in the PF_6^- reorientation rate in different cages, that is, from lattice disorder.

The most likely structure for all three hydrates at very low temperatures is one in which the hexafluoride ions are frozen in disordered orientations and the HF molecules randomly occupy one-sixth of the positions ascribed to water molecules by Bode and Teufer.[2b] A further one-sixth of these positions are occupied by H_3O^+ ions which are expected to be generally

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(6) P. M. Borodin, R. Singh, and V. A. Shcherbakov, *Zh. Strukt. Khim.*, **9**, 1078 (1968).

(7) H. S. Gutowsky, D. W. McCall, and C. P. Slichter, *J. Chem. Phys.*, **21**, 279 (1953).

(8) A. Abragam, "The Principles of Nuclear Magnetism", Clarendon Press, Oxford, 1961, p 438.

(9) H. A. Resing and D. W. Davidson, *Can. J. Phys.*, **54**, 295 (1976).

(10) H. A. Resing, *J. Chem. Phys.*, **43**, 669 (1965).

nearest neighbors of HF molecules in the hydrogen-bonded lattice. The cages have cubic symmetry only on space averaging. At temperatures near 0 °C, the rapid diffusion and interchange of water and HF molecules produces time averaging to cubic symmetry on the NMR time scale.

The bond lengths in the hexafluoride ions of the hydrates are not known. If one assumes P–F, As–F, and Sb–F bond lengths similar to those measured[11-13] for K salts of these ions, 1.58, 1.72, and 1.77 Å, respectively, the corresponding van der Waals ionic diameters are 5.86, 6.14, and 6.24 Å. These values are only slightly in excess of the cage "free diameters", which, with the unit cell parameters in Table III and 2.8 Å for the van der Waals diameter of water, are 5.78, 5.83, and 5.92 Å, respectively.

Registry No. $HPF_6 \cdot HF \cdot 5H_2O$, 39610-10-1; $HAsF_6 \cdot HF \cdot 5H_2O$, 77152-86-4; $HSbF_6 \cdot HF \cdot 5H_2O$, 77152-87-5.

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(11) H. Bode and H. Clausen, *Z. Anorg. Allg. Chem.*, **265**, 229 (1951).
(12) G. Gafner and G. J. Kruger, *Acta Crystallogr., Sect. B*, **B30**, 250 (1974).
(13) H. Bode and E. Voss, *Z. Anorg. Allg. Chem.*, **264**, 144 (1951).
(14) E. W. Lawless, C. J. W. Wiegand, Y. Mizumoto, and C. Weis, *Inorg. Chem.*, **10**, 1084 (1971).
(15) K. O. Christe, C. J. Schack, and R. D. Wilson, *Inorg. Chem.*, **14**, 2224 (1975).

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Photochemistry of the Dinuclear Cobalt(III) Complexes $(CN)_5Co(\mu\text{-}CN)Co(NH_3)_5$ and $(CN)_5Co(\mu\text{-}NC)Co(NH_3)_5$. Intramolecular Energy Transfer between Metal Centers

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Irradiation of aqueous $(NC)_5Co(\mu\text{-}NC)Co(NH_3)_5$ in the wavelength region 254–365 nm leads to photoaquation of the pentacyanocobaltate(III) center, giving $Co(CN)_5H_2O^{2-}$ and $Co(NH_3)_5CN^{2+}$ as products with large quantum yields (0.2–0.3 mol/einstein). These wavelengths represent ligand field (LF) excitation of the pentacyanocobaltate(III) chromophore; hence, efficient photoreaction of that center indicates that in this case energy transfer to the lower LF states of the pentaamminecobalt(III) chromophore is at best competitive with ligand labilization. In contrast, LF excitation of the former chromophore in the linkage isomer dinuclear complex $(NC)_5Co(\mu\text{-}CN)Co(NH_3)_5$ leads to very little reaction at that site. Since $Co(CN)_6^{3-}$ is quite photoactive under these conditions, this result implies that coordination of the $Co(NH_3)_5^{2+}$ moiety to one CN provides a new pathway for rapid deactivation of the $Co(CN)_6^{3-}$ LF states. The differences between the two dinuclear complexes' photochemical behaviors are rationalized in terms of symmetry restrictions and larger Franck–Condon terms inhibiting energy transfer between the two cobalt(III) centers of $(NC)_5Co(\mu\text{-}NC)Co(NH_3)_5$ relative to the linkage isomer. For comparison the complex ion $Co(CN)_5(CH_3CN)^{2-}$ has been synthesized. Ligand field excitation of this species leads to highly efficient photoaquation of CH_3CN exclusively ($\phi \sim 0.4$).

Introduction

In recent years there has been considerable interest in sensitization (energy-transfer) and redox (electron-transfer) processes involving metal complex excited states.[1] Much of this interest has derived from schemes proposing to utilize such reactions for the conversion of radiant energy to chemical potential energy, i.e., for solar energy storage. Most studies have focused on bimolecular processes, and a number of these have been demonstrated where both chromophores in the energy or electron transfer are metal complexes. However, the diffusion terms in bimolecular rate laws and the uncertain relative configurations of the interacting centers make comparison of quantitative data a rather challenging exercise. Clearly, to understand in detail the principles governing energy or electron transfer, one must elucidate the dominating structural or spatial parameters. The ideal situation would be to have two chromophores in fixed positions relative to each other, a situation best approximated by placing both the acceptor and donor metal centers in the same complex. A limited number of photochemical studies have been done on such systems; however, in each of these examples a key excited state of at least one center is charge transfer in character.[2-5] Ligand field excited states play important roles in the photoreactions of transition-metal complexes especially with regard to photodegradation of complexes by ligand labilization. As a consequence, it is of interest to examine energy transfer between two metal centers, both having lowest energy ligand field (LF) states. Examples are the dicobalt complexes $(CN)_5Co(\mu\text{-}CN)Co(NH_3)_5$ ("C_6N_6") and $(CN)_5Co(\mu\text{-}NC)\text{-}Co(NH_3)_5$ ("C_5NCN_5"),[6] each possessing a pentacyanocobalt(III) chromophore linked to a pentaaminecobalt(III) center, the sole difference being the configuration of the bridging cyanide ligand.

The spectra of C_6N_6 and C_5NCN_5 in aqueous solution are shown in Figure 1 and the spectra of comparison mononuclear complexes in Figures 2 and 3. The spectra of the mononuclear species show the two spin-allowed, Lapporte-forbidden LF bands expected for a diamagnetic d^6 complex with an approximately octahedral field (${}^1T_1 \leftarrow {}^1A_1$ and ${}^1T_2 \leftarrow {}^1A_1$ in O_h symmetry). Notably the LF bands for the pentaammine species occur at considerably longer wavelengths than in the pentacyano series, owing to the greater ligand field strength of the CN^- ion. The dinuclear ions' LF spectra appear to be the sum of the two types of spectra (Figures 2 and 3) although the onset of charge-transfer absorption occurs at somewhat

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(1) (a) Balzani, V.; Moggi, L; Manfrin, M. F.; Bolletta, F.; Laurence, G. S. *Coord. Chem. Rev.* **1975**, *15*, 321–433. (b) Whitten, D. *Acc. Chem. Res.* **1980**, *13*, 83.
(2) Durante, V. A.; Ford, P. C. *J. Am. Chem. Soc.* **1975**, *97*, 6898.
(3) Malin, J.; Ryan, D. A.; O'Halloran, T. V. *Ibid.* **1978**, *100*, 2097.
(4) Vogler, A.; Kunkely, H. *Ber. Bunsenges. Phys. Chem.* **1975**, *79*, 301.
(5) Gelroth, J. A.; Figard, J. E.; Peterson, J. D. *J. Am. Chem. Soc.* **1979**, *101*, 3649.
(6) (a) deCastello, R. A.; Piriz Mac-Coll, C.; Egen, N.; Haim, A. *Inorg. Chem.* **1969**, *8*, 699. (b) deCastello, R. A.; Piriz Mac-Coll, C.; Haim, A. *Ibid.* **1971**, *10*, 203.

0020-1669/81/1320-2016$01.25/0