increased. This behavior indicates that the coordinated OCH_3 groups exchange with CH_3OH molecules in solution. At CH_3OH:Nb ratios of 1:1–5:1 only one OCH_3 is involved in exchange. At larger mole ratios exchange of both coordinated OCH_3 groups occurs. This stepwise behavior in the exchange of coordinated OCH_3 suggests one methoxide group is more easily exchanged than the other.

In a similar study benzyl alcohol was treated with $NbCl(OCH_3)_2(dedtp)_2$ in chloroform. The nmr spectrum of the solutions indicated that there was a rapid reaction (complete in less than 1 min) in which coordinated methoxide groups were displaced by the benzyl alcohol.

A very characteristic reaction of all the new compounds is their decomposition which occurs with the loss of a coordinated alkoxide group (probably as the alcohol). On standing the crystalline pale purple compounds turn yellow-green. This occurs in the air, in a drybox, or under vacuum. For some compounds appreciable change occurs in 1 day; for others the reaction is slower. The same color change occurs rapidly when the compounds are dissolved in solvents such as acetone, chloroform, or benzene. Ultimately yellow-green solids precipitate from these solutions. The purple color can be restored by dissolving the yellow-green compounds in an alcohol or in some cases simply by exposing these materials to alcohol vapors. The compounds can be stored in the presence of the vapors of the appropriate alcohol. If an nmr spectrum is taken of the yellow compound obtained by placing a sample of $NbCl(OCH_3)_2(dedtp)_2$ under vacuum for several days, one observes a spectrum similar to that of the parent niobium compound, containing, however, only one OCH_3 group. The spectrum after restoring the purple-pink color in methanol is identical with that of the $NbCl(OCH_3)_2(dedtp)_2$ compound with two OCH_3 groups. The loss of alkoxide accounts for the low carbon analyses reported earlier, since appreciable decomposition of the ethanol and 2-propanol derivatives would be expected during their trip to the analyst.

We are uncertain as to what happens during this decomposition. The analyses, molecular weights, diamagnetism, and nmr spectra are consistent with our formulation of the pale purple compounds. The conversion to a yellow-green material presumably is a hydrolysis or condensation reaction. This is consistent with the limited solubility of the yellow-green materials, which on extended standing become insoluble.

Summary

The results discussed in this paper indicate that the compounds we have prepared are monomers with the stoichiometry $NbX(OR)_2(S_2P(OR')_2)_2$. Since the dithiophosphate ligands seem to be coordinated as bidentate ligands, the compounds probably contain niobium coordinated to seven donor atoms. The compounds give stable highly conducting solutions in methanol. It seems probable that these solutions contain solvated $Nb(S_2P(OR)_2)_2{}^{3+}$ cations. Exchange of the coordinated alkoxide groups is readily achieved by recrystallization from the desired alcohol. The compounds slowly decompose in the solid state and in solvents such as $CHCl_3$ and CH_3CN with the formation of less soluble substances and the loss of one coordinated alkoxide. In many cases the compounds can be regenerated by exposure to the vapors of the appropriate alcohol.

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Magnetic Properties of Some Polynuclear Hydroxy-Bridged Iron(III) Sulfate Complexes

BY R. W. CATTRALL,*[1] K. S. MURRAY, AND K. I. PEVERILL

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Our recent studies of the extraction of iron(III) from aqueous sulfate solutions with high molecular weight primary[2] or secondary amines[3] have yielded compounds of stoichiometry $(R(R')NH_3)_2FeOH(SO_4)_2$ and $(R_2NH_2)_2FeOH(SO_4)_2$ where R is 3,5,5-trimethylhexyl and R′ is *n*-dodecyl. The compounds obtained with the primary amines were crystallized from ethanol solution with 1 mol of ethanol attached. All of these compounds had magnetic moments in the region of 3.6 BM, and preliminary studies suggested hydroxy bridging of dimeric or trimeric aggregates.

In order to obtain a more detailed knowledge of the degree of aggregation and possibly the mode of bridging in the primary amine complexes, we have carried out magnetic susceptibility studies over the temperature range 80–300°K and have fitted the results to various theoretical models. Measurements have also been made on the known[4] hydroxy-bridged compound $FeOHSO_4$ for comparison. This latter hydrolyzed species is suggested to be the one extracted from aqueous solution by the amine sulfate salts.[2,3]

Although significant advances have been made recently through magnetic,[5-7] spectroscopic,[8-10] and X-ray studies[11-14] toward an understanding of oxygen

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bridging in dimeric and trimeric[15] iron(III) systems, there has been little definitive work on hydroxy-bridged aggregates.[16,17] Both the dihydroxy-bridged species $Fe_2(OH)_2^{4+}$ and the trimeric species $Fe_3(OH)_4^{5+}$ have recently been suggested in various aqueous iron(III) systems.[18,19] Hydroxy bridging also has particular relevance in biological systems.[19] It is interesting to note that magnetic moments of about 3.6 BM have been observed in some aqueous[18,20] and biological systems[19,21] and have been associated with $Fe_2(OH)_2^{4+}$ bridging and more recently with hydroxyiron high polymer formation.[19,22]

Experimental Section

Preparations.—The iron(III) complexes with 3,5,5-trimethylhexylammonium sulfate and *n*-dodecylammonium sulfate were prepared by solvent extraction as described previously.[2] The complexes obtained by evaporation of the organic solvent had the stoichiometries $(RNH_3)_2FeOH(SO_4)_2$ and $(R'NH_3)_2FeOH(SO_4)_2$ where R = 3,5,5-trimethylhexyl and R′ = *n*-dodecyl. The complexes were found to absorb water on standing in air which could be removed by heating at 100° under vacuum. The amount of water absorbed corresponded to 1 mol and 0.5 mol, respectively, added to the above stoichiometric formulas. On recrystallization of the two anhydrous compounds from ethanol, orange-red crystals were obtained which had the above stoichiometries with 1 mol of ethanol attached, respectively. These two ethanolates absorbed water on standing in air which could be removed on heating at 100° under vacuum. The amount of water absorbed again corresponded to 1 mol and 0.5 mol, respectively, added to the stoichiometric formulas. It was not possible to remove the ethanol on heating without decomposing the complexes.

The compound $FeOHSO_4$ was prepared using the procedure described by Posjnak and Merwin.[23] *Anal.* Calcd for $FeOHSO_4$: Fe, 33.1. Found: Fe, 33.0.

Magnetic Measurements.—The magnetic measurements were made using the Gouy method with aqueous nickel chloride solution as calibrant. Measurements on the anhydrous complex were made using a Gouy tube sealed under an atmosphere of dry nitrogen.

Results and Discussion

The susceptibilities of the following iron(III)–amine–sulfate complexes in the range 80–300°K are given in Table I: $(RNH_3)_2FeOH(SO_4)_2 \cdot C_2H_5OH \cdot H_2O$ (complex A), $(RNH_3)_2FeOH(SO_4)_2 \cdot H_2O$ (complex B), $(R'NH_3)_2FeOH(SO_4)_2 \cdot C_2H_5OH \cdot H_2O$ (complex C), $(R'NH_3)_2FeOH(SO_4)_2 \cdot H_2O$ (complex D), and $(R'NH_3)_2FeOH(SO_4)_2 \cdot C_2H_5OH$ (complex E), where R = C_9H_{19} and R′ = $C_{12}H_{25}$. The susceptibility of the compound $FeOHSO_4$ is also included in Table I. The susceptibilities were independent of field strength (3000–6000 G) in all cases.

The values of the susceptibilities at 300°K are in the region expected for an $S = {}^3/_2$ value and are considerably lower than that expected for an $S = {}^5/_2$ value. The temperature dependences of the reciprocal susceptibilities of the various complexes deviate widely from Curie–Weiss behavior and in most cases show distinct curvature. These results are highly suggestive of polynuclear structures.

TABLE I
EXPERIMENTAL SUSCEPTIBILITIES FOR THE IRON(III) SULFATE COMPLEXES

Temp, °K	$10^6\chi_{Fe}$, cgsu/mol	Temp, °K	$10^6\chi_{Fe}$, cgsu/mol
Complex A (386×10^{-6})[a]		Complex C (445×10^{-6})	
305.0	5426	303.9	6166
294.0	5579	291.3	6233
260.9	5781	275.5	6289
245.8	5843	261.5	6402
218.0	6028	242.9	6580
187.3	6239	226.1	6732
166.4	6343	202.0	6845
143.0	6570	175.0	7158
115.0	6960	157.8	7336
95.4	7500	127.1	7846
83.5	8029	103.0	8478
		89.5	8935
Complex B (352×10^{-6})		Complex D (417×10^{-6})	
302.0	4783	301.0	5660
291.0	4818	280.0	5744
248.2	5012	256.3	5820
207.0	5116	232.7	5970
170.5	5202	209.0	6032
156.7	5170	185.5	6092
144.3	5202	162.6	6142
127.5	5127	140.0	6220
121.4	5156	121.0	6280
114.0	5129	95.0	6440
98.7	5240	83.0	6670
85.0	5310		
Complex E (440×10^{-6})		$FeOHSO_4$ (52×10^{-6})	
299.0	6444	302.4	5490
285.0	6564	286.6	5557
265.4	6653	254.9	5680
238.5	6821	221.0	5770
206.3	7050	205.6	5802
181.5	7236	182.0	5807
162.8	7425	161.0	5812
128.6	7957	137.3	5809
113.0	8321	114.2	5730
90.3	9020	103.0	5702
		89.0	5698
		84.8	5752

[a] Values in parentheses are diamagnetic corrections for ligands.

In order to obtain some information on the spin state of the iron(III) atom and the nature of the polynuclear metal aggregate, the susceptibility results have been fitted, using a least-squares computer program, to the theoretical susceptibility expressions of a number of simple cluster models. It is recognized that, in the absence of X-ray structural data, caution must be exercised in predicting structures from powder susceptibilities measured over the temperature range 80–300°K. The models used were as follows: (a) $S = {}^5/_2$, dimer,[24,25] linear trimer,[24,26] equilateral triangular trimer,[27] isosceles triangular trimer,[26] and infinite linear chain (Fisher);[28,29] (b) $S = {}^3/_2$, dimer[25] and equilateral trimer.[27] Each model assumes nearest-neighbor antiferromagnetic exchange under the Heisenberg Hamiltonian $-2J\Sigma\bar{S}_i \cdot \bar{S}_j$, where J is negative for antiferro-

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magnetism. All the models are exact solutions using the Heisenberg Hamiltonian except for the Fisher linear-chain model which is exact only in the classical limit of infinite spin.[28] In all cases, the g value and temperature-independent paramagnetic term $N\alpha$ were held at 2.0 and 0, respectively. This is correct for $S = {}^5/_2$ and is a reasonable approximation which is generally used for $S = {}^3/_2$. The crystal structure of $FeOHSO_4$ shows that it possesses infinite chains of octahedrally coordinated iron atoms linked by single hydroxy bridges, –Fe–OH–Fe–OH–, with a large distance between chains. This compound therefore appears to provide a suitable system to compare with the Fisher infinite-chain model.

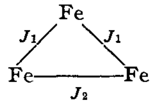
Using the $S = {}^5/_2$ spin state, very poor agreement was obtained for the iron(III)–amine–sulfate complexes for the dimer model (diamagnetic ground state); however, much better agreement was obtained for the trimer models (paramagnetic ground state). Despite the closeness of the magnetic moments to an $S = {}^3/_2$ value, comparison with the $S = {}^3/_2$ models gave very poor agreement for all cases and hence it seems certain that the spin state of the iron(III) centers in the complexes is $S = {}^5/_2$.

The magnetic parameters are summarized in Table II and typical results of fitting the experimental figures to the various models are shown in Figures 1 and 2.

TABLE II
PARAMETERS OBTAINED FROM THE BEST FIT OF MAGNETIC DATA (J IN CM^{-1})

	μ_{eff}, BM		Spin $^5/_2$ (g = 2.0)			
			Equil trimer	Isos trimer[a]		Infinite chain
Compound	295°K	85°K	$-J$	$-J_1$	$-J_2$	$-J$
$(C_9H_{19}NH_3)_2FeOH(SO_4)_2\cdot C_2H_5OH\cdot H_2O$ (A)	3.62	2.32	21	21	27	
$(C_9H_{19}NH_3)_2FeOH(SO_4)_2\cdot H_2O$ (B)	3.37	1.90	30			
$(C_{12}H_{25}NH_3)_2FeOH(SO_4)_2\cdot C_2H_5OH\cdot 0.5H_2O$ (C)	3.82	2.53	18	17	23	
$(C_{12}H_{25}NH_3)_2FeOH(SO_4)_2\cdot H_2O$ (D)	3.67	2.11	23			
$(C_{12}H_{25}NH_3)_2FeOH(SO_4)_2\cdot C_2H_5OH$ (E)	3.91	2.55	18	17	21	
$FeOH(SO_4)$	3.61	1.98				18

[a] The model is as given below



Using the trimeric formulation for the iron–amine–sulfate complexes as a basis for discussion, the agreement shown by the ethanolated complexes (A and C) for a linear or equilateral triangular model ($J_{equil} = {}^2/_3J_{linear}$) is improved by assuming that one iron atom is different from the other two, *i.e.*, in an isosceles triangular arrangement. This, of course, leads to two different J values (Table II).

The susceptibilities of the complexes without ethanol (B and D) were less dependent on temperature than those of the ethanolated complexes A and C. The results, however, were still in good agreement with the equilateral triangular or linear models although they could not be fitted to the isosceles triangular model. The J values from the equilateral triangular model are higher than those in the parent ethanolated compounds. Removal of water from the ethanolated complex C gave only a slight increase in the magnetic moment, and the resulting J values were virtually the same. These results, though not definitive, suggest that ethanol might be involved in bonding within the polynuclear cluster whereas water is possibly not.

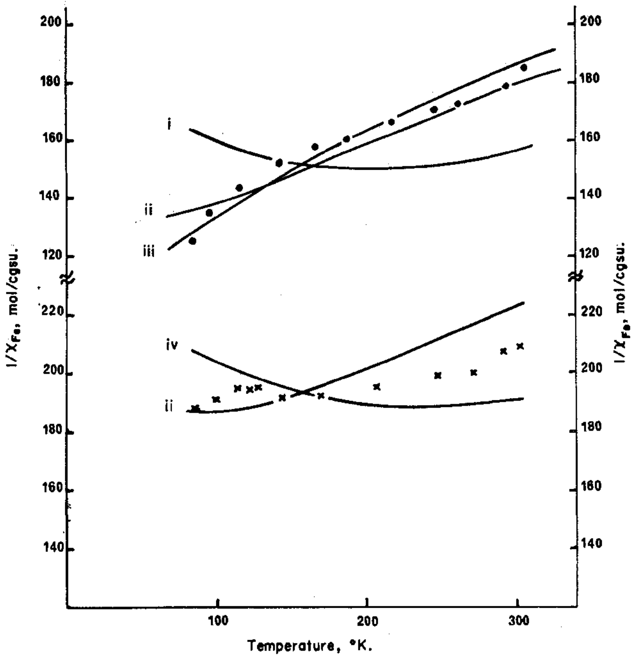


Figure 1.—Experimental and calculated best fit values of $1/\chi_{Fe}$ *vs.* temperature for iron(III) hydroxysulfate complexes: ●, complex A; ×, complex B. Full curves are best fit calculated values corresponding to parameters in Table II. Theoretical $S = {}^5/_2$ models: i, dimer; ii, equilateral triangular (linear) trimer; iii, isosceles triangular trimer; iv, Fisher infinite chain.

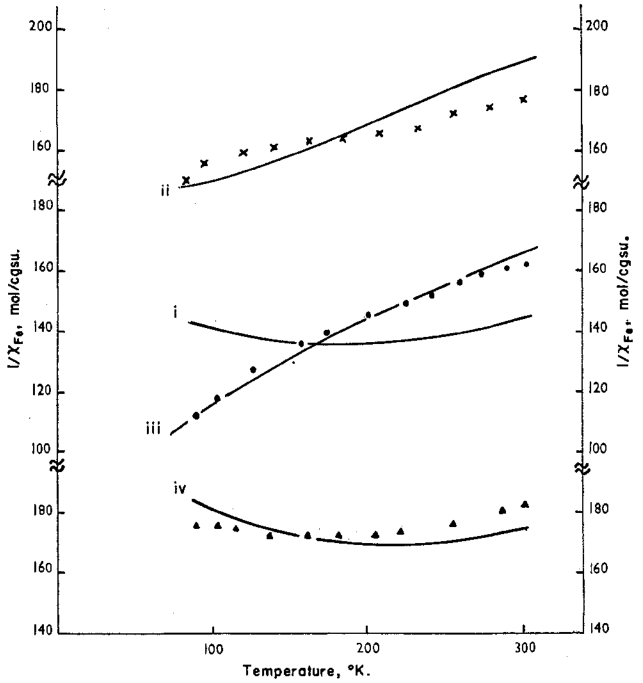


Figure 2.—Experimental and calculated best fit values of $1/\chi_{Fe}$ *vs.* temperature for iron(III) hydroxysulfate complexes: ●, complex C; ×, complex D; ▲, $FeOH(SO_4)$. Full curves are best fit calculated values corresponding to parameters in Table II. Theoretical $S = {}^5/_2$ models: i, dimer; ii, equilateral triangular (linear) trimer; iii, isosceles triangular trimer; iv, Fisher infinite chain.

It is worth pointing out that the J values for the equilateral triangular model are slightly lower than those shown[24] by the basic iron(III) carboxylates which possess the Fe_3O triangular oxy-bridged unit.[27]

The $S = {}^5/_2$ Fisher infinite-chain model gave quite good agreement for the compound $FeOHSO_4$, which is to be expected in view of the crystal structure.[4] Mössbauer effect measurements on this compound show[30] an antiferromagnetic ordering temperature of *ca.* 80°K at which a quadrupole doublet is replaced by a six-line hyperfine spectrum. Application of the Fisher model to the amine sulfate complexes gave moderate agreement for complex B but very poor agreement for the other complexes.

The results of the magnetic studies of the iron(III)–amine–sulfate complexes therefore suggest that the basic unit of the polynuclear structure is possibly a trinuclear cluster of interacting $S = {}^5/_2$ iron(III) ions. On the basis of chemical and physical evidence given previously, together with the relationship to $FeOHSO_4$, it is suggested that the bridging is through hydroxy groups and not through oxy linkages as found with certain other basic ferric complexes.[15] Trinuclear hydroxy-bridged clusters have, in fact, been characterized in some hydrolyzed tin(II) and beryllium(II) species.[31]

In order further to test the applicability of the simple trinuclear cluster model to the present compounds, we are currently extending the susceptibility measurements down to 4°K together with field-dependent magnetization studies.

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The Degradation of Biscarborane

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It has been observed that 1,2-dicarba-*closo*-dodecaborane(12) and its B- and C-substituted derivatives are degraded by ethanolic potassium hydroxide to give the corresponding 7,8-dicarba-*nido*-dodecahydroundecaborate(1−) ion and its B- and C-substituted derivatives with evolution of 1 mol equiv of hydrogen[1-4]

$$1,2\text{-}B_{10}C_2H_{12} + OH^- + 3C_2H_5OH \xrightarrow[15\ \text{hr}]{80°} 7,8\text{-}B_9C_2H_{12}^- + H_2O + B(OC_2H_5)_3 + H_2$$

We have found that biscarborane[5] {1-[1′-1′,2′-dicarba-*closo*-dodecaborane(12)]-1,2-dicarba-*closo*-dodecaborane(12)} is selectively degraded by ethanolic potassium hydroxide at reflux for 1.5 hr to give initially 1 mol equiv of hydrogen and 7-[1′-1′,2′-dicarba-*closo*-dodecaborano(12)]-7,8-dicarba-*nido*-dodecahydroundecaborate(1−) (I), which may be isolated or further degraded by ethanolic potassium hydroxide at reflux for 120 hr to give a second mole equivalent of hydrogen and 7-(7′-7′,8′-dicarba-*nido*-dodecahydroundecaborato)-7,8-dicarba-*nido*-dodecahydroundecaborate(2−) (II)

$$1\text{-}(1'\text{-}1',2'\text{-}B_{10}C_2H_{11})\text{-}1,2\text{-}B_{10}C_2H_{11} + OH^- + 3C_2H_5OH \xrightarrow[1.5\ \text{hr}]{80°} 7\text{-}(1',1',2'\text{-}B_{10}C_2H_{11})\text{-}7,8\text{-}B_9C_2H_{11}^-\ (I) + H_2O + B(OC_2H_5)_3 + H_2$$

$$I + OH^- + 3C_2H_5OH \xrightarrow[120\ \text{hr}]{80°} 7\text{-}(7'\text{-}7',8'\text{-}B_9C_2H_{11})\text{-}7,8\text{-}B_9C_2H_{11}^{2-}\ (II) + H_2O + B(OC_2H_5)_3 + H_2$$

I and II were conveniently isolated in 80% yield as their cesium or quaternary ammonium salts, which were conveniently purified by recrystallization from hot water or water–ethanol.

The 60-MHz ^1H nmr spectrum of the cesium salt of I in deuterioacetone consisted of two broad singlets of equal intensity at τ 8.3 and 6.1, which were assigned with confidence to the 8 and 2′ carborane protons, respectively, by comparison with the resonances observed in the spectra of biscarborane[5] and Cs-7,8-$B_9C_2H_{12}$.[3] The 80-MHz ^{11}B nmr spectrum of I in acetone is presented in Figure 1. At least ten different types of boron environments (a–j) could be distinguished. Two high-field doublets of intensity 1.0 (i, j) were very similar to features attributed to the 10 and 1 boron atoms in the spectrum of 7,8-$B_9C_2H_{12}^-$ and its C-substituted derivatives. The spectrum contained another high-field doublet of intensity 1.0 (h) and a low-field set of at least seven other doublets of total intensity 16. These ten observed unique boron environments of total intensity 19 correspond nicely to the 15 unique boron en-

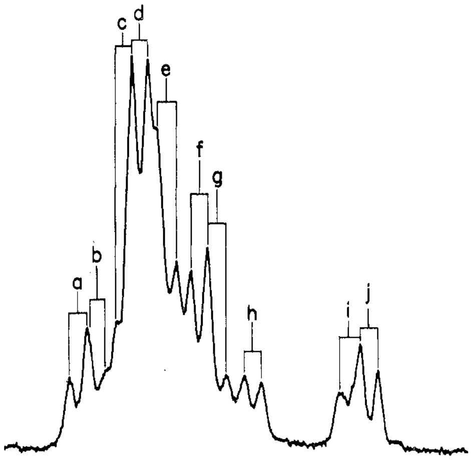

Figure 1.—An 80-MHz ^{11}B nmr spectrum of Cs-7-(1′-1′,2′-$B_{10}C_2H_{11}$)-7,8-$B_9C_2H_{11}$ in acetone. Chemical shifts, relative to external $BF_3 \cdot O(C_2H_5)_2$ (coupling constants, Hz): (a) +5.2 (158), (b) +7.2 (136), (c) +10.0 (138), (d) +11.8 (145), (e) +14.7 (161), (f) +18.1 (152), (g) +20.0 (152), (h) +23.7 (140), (i) +34.0 (140), (j) +36.3 (158).

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