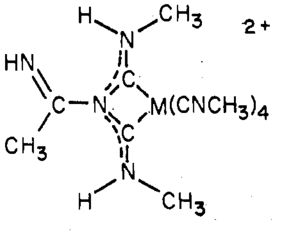
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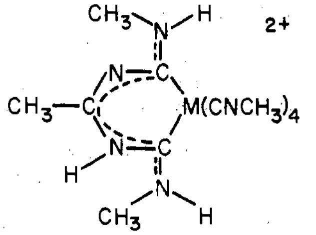
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readily accounted for as ν(C=N) in structure 8 but is of an energy somewhat higher than expected for the six-membered chelate ring in 9. Furthermore, the chemical shifts for the various pmr resonances are very similar to those of the complex 1 which also contains a four-membered chelate ring. The pmr data can only be rationalized in terms of structure 9 if rapid proton exchange is occurring between the two nitrogens in the chelate ring. This proton exchange would serve to make the two terminal NH(CH_3) groups and the two in-plane isocyanide ligands equivalent. A similar phenomenon has been observed for the deprotonated form of **3a** and **3b**[7] except that in these platinum and palladium complexes the proton moves between immediately adjacent nitrogen atoms. At lower temperatures the pmr spectrum of the ruthenium acetamidine adduct undergoes only one change. The terminal N-H resonance at τ 1.82 broadens and splits into two resonances. In acetone-d_6 at −80° these resonances occur at τ 0.64 and 0.12. This indicates that at low temperatures the terminal NH(CH_3) groups become nonequivalent. Presumably, the effect on the chemical shift of the associated methyl resonance is too small to detect. This nonequivalence can be explained in terms of structure 8 if rotation of the $CH_3C(NH)$ group about the C-N (chelate) bond is restricted at low temperatures. However, the low-temperature spectrum is also consistent with structure 9 if the proton exchange described above is slowed sufficiently at low temperatures to reflect the intrinsic asymmetry of the six-membered chelate ring.

Attempted Reactions with 2-Aminopyridine. Refluxing methanolic solutions of 2-aminopyridine with $Fe(CNCH_3)_6^{2+}$ or $Ru(CNCH_3)_6^{2+}$ under conditions more than adequate to effect the additions described above, leads to no reaction. The failure of 2-aminopyridine to add to these complexes although it does add to $Pt(CNCH_3)_4^{2+}$ may be ascribed to a combination of the following effects: the weaker basicity of 2-aminopyridine compared to the other amines, the greater bulk of this amine, and the inability of the pyridine nitrogen to replace an isocyanide ligand in the more substitutionally inert iron and ruthenium complexes.

Acknowledgment. This research was sponsored by grant GM 18357 from the National Institutes of Health.

Registry No. $[Ru(CNCH_3)_6][PF_6]_2$, 49631-67-6; $[Ru(CNCH_3)_6][BF_4]_2$, 49631-68-7; $[Fe(o\text{-phen})_2(CNCH_3)_2][PF_6]_2$, 49664-77-9; $[Fe(o\text{-phen})_2(CNCH_3)_2][BF_4]_2$, 49627-00-1; $[Ru(CNCH_3)_5\{C(NHCH_3)_2\}][PF_6]_2$, 49631-69-8; $[Ru(CNCH_3)_4\{C(NHCH_3)_2\}_2][PF_6]_2$, 49631-70-1; $[Fe(CNCH_3)_5\{C(NHCH_3)NH_2\}][PF_6]_2$, 49716-69-0; $[Ru(CNCH_3)_4\{C_4H_{10}N_4\}][PF_6]_2$, 49849-46-9; $[Ru(CNCH_3)_2\{C_4H_{10}N_4\}_2][PF_6]_2$, 49631-71-2; $[Fe(o\text{-phen})_2\{C_4H_{10}N_4\}][BF_4]_2$, 49631-72-3; $[Fe(CNCH_3)_4(C_6H_{12}N_4)][PF_6]_2$, 49631-73-4; $[Ru(CNCH_3)_4(C_6H_{12}N_4)][PF_6]_2$, 49631-74-5; $[Fe(CNCH_3)_4\{C_5H_{11}N_3\}][PF_6]_2$, 41654-40-4; $[Ru(CNCH_3)_6][HSO_4]_2$, 49627-01-2; $[Fe(CNCH_3)_6][HSO_4]_2$, 15334-25-5; $K_4Ru(CN)_6$, 15002-31-0; $Fe(o\text{-phen})_2(CN)_2$, 15362-08-0; methylamine, 74-89-5; ammonia, 7664-41-7; hydrazine, 302-01-2; acetamidine, 143-37-3.

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Spectral and Structural Studies of Iron(III) Salicylaldimine Complexes. A Six-Coordinate Dinuclear Complex

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The dimeric, five-coordinate complex $[Fe(SALPA)Cl]_2$ (SALPA = the dianion of the potentially tridentate Schiff-base ligand formed from salicylaldehyde and 3-aminopropanol) reacts readily with sodium peroxide to give a red crystalline product, $[Fe_2(SALPA)_2(SALPAH)_2]$, which exhibits antiferromagnetic behavior (μ_{eff}/g-atom of iron = 4.45 and 2.34 BM at 298 and 77°K, respectively). The three-dimensional X-ray crystal structure analysis of the toluene solvate of the complex revealed the presence of six-coordinate iron atoms linked closely by bridging propoxide groups into dinuclear units having a planar, four-membered Fe–O–Fe–O ring (Fe–O = 1.93 (2), 2.00 (2) Å; O–Fe–O = 70.9 (8), 70.2 (8)°; Fe–O–Fe = 108.2 (9), 110.6 (9)°). Hydrogen bonding between the uncoordinated alcohol groups and the phenolic oxygens connects the dimeric units into infinite, doubly linked chains. The iron atoms have virtually identical geometries (distorted octahedron with trans nitrogens, two bridging cis μ_2 oxygens, two terminal oxygens); the iron atoms are nevertheless nonequivalent since the two bridging propoxide groups are chelated to the same iron atom. The preparation of $Fe_2(SALPA)_2(SALPAH)_2$ is discussed and the structure is correlated with the magnetic and spectral properties of several mononuclear and dinuclear iron(III) complexes. Crystal data for $[Fe_2(SALPA)_2(SALPAH)_2]\cdot$toluene: *Pcab*, $a = 20.050$ (3) Å, $b = 24.269$ (4) Å, $c = 18.608$ (3) Å, $Z = 8$, $\rho_{\text{calcd}} = 1.34$ g cm^{-3}, $\rho_{\text{obsd}} = 1.35$ g cm^{-3}. Final agreement factors are $R = 0.098$ and $R_w = 0.071$ for 1160 reflections above background.

Introduction

Schiff base complexes of iron(III) are of considerable interest because of the diversity of coordination geometries and the unusual magnetic properties which have been observed.[1-7] Thus, mononuclear and polynuclear complexes have been found to have distorted trigonal-bipyramidal,[1,2] square-pyra-

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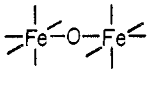
midal,[1,2,7] octahedral,[6] and pentagonal-bipyramidal[5] coordination geometries; polynuclear complexes have been found with a single μ_2-oxo bridge,[5,6] I, or with two μ_2-alkoxo bridges forming a four-membered iron–oxygen ring, II.[1-4,7] These oxygen-bridged polynuclear complexes have generally shown antiferromagnetic properties but the coupling constants have been found to differ considerably for the different types of structures. The polynuclear iron complexes are of additional interest due to the known occurrence of iron "clusters" in several enzymes involved in biochemical redox reactions and occurrence in oxygen carrying proteins.[8]



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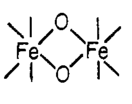

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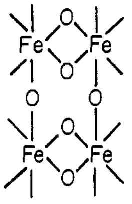

III

Our interest in preparations, chemical and magnetic properties, and structures of polynuclear complexes led us to attempt the preparation of complexes containing both types of μ_2-bridging species, III. Accordingly, we have studied the product of the reaction of the iron(III) dimer $[Fe(SALPA)Cl]_2$, where SALPA = *N*-(3-hydroxypropyl)salicylaldimine, with various bases in ethanol.[9] Instead of the expected $[Fe(SALPA)]_4(O)_2$ complex, a product of approximate empirical formula $Fe(SALPA)_2$ was obtained when sodium peroxide or sodium hydroxide was used. Magnetic susceptibility measurements showed the complex to be strongly antiferromagnetically coupled and, hence, indicated a polynuclear structure. To elucidate the molecular structure, the three-dimensional X-ray structure determination reported in this paper was undertaken. We also report correlations of electronic spectral data for several related mononuclear and dinculear iron(III) salicylaldimine complexes and attempt to correlate the spectral data with their structures and magnetic properties.

Experimental Section

Preparation of $[Fe_2(SALPA)_2(SALPAH)_2]$·toluene. The method of Breece, *et al.*, was used to prepare $[Fe(SALPA)Cl]_2$.[1]

Sodium peroxide (0.78 g, 10 mmol) was added in portions to a stirred suspension of $[Fe(SALPA)Cl]_2$ (1.34 g, 5 mmol) in 50 ml of ethanol. Within a few minutes the black-brown suspension had changed to a bright orange slurry. After stirring for 1.5 hr, the mixture was filtered to give a bright orange solid and an orange filtrate. The residue was recrystallized from toluene to give large red platelets of the solvated compound suitable for single-crystal X-ray study; yield *ca.* 0.8 g (80%); mp 142-145°. *Anal.* Calcd for $Fe_2C_{47}H_{54}N_4O_8$ (mol wt 914.68): C, 61.72; H, 5.95; N, 6.13; Cl, 0.00. Found: C, 61.97; H, 5.95; N, 6.10; Cl, 0.00.

Magnetic Susceptibility. Magnetic measurements were performed by the Faraday method using $Hg[Co(SCN)_4]$ as calibrant. Diamagnetic corrections were applied using Pascal's constants.[10] The experimental moments, BM (°K in parentheses), are 4.45 (298), 3.82 (195), and 2.34 (77).

Spectral Studies. Electronic spectra were determined at room temperature with a Cary 14 spectrophotometer using *ca.* 10^{-3} *M* solutions in 0.1- and 1-cm quartz cells.

Collection and Reduction of the Intensity Data. A red platelet of dimensions 0.044 × 0.293 × 0.611 mm was selected from the preparative batch and mounted on a glass fiber normal to the large face (010). Precession photographs of the reciprocal lattice levels *h0l*, *h1l*, *h2l*, *hk0*, and *hk1* revealed lattice symmetry *mmm* and the systematic absences *hk0* ($k = 2n + 1$), *h0l* ($h = 2n + 1$), and *0kl* ($l = 2n + 1$), were uniquely consistent with the orthorhombic space group *Pcab*, a nonstandard setting of *Pbca*-D_{2h}(No. 61).[11] *Pcab* is related to *Pbca* by an interchange of the *a* and *c* axes and reversal of the direction of all axes.

The crystal was transferred to a Picker automated diffractometer and 16 accurately centered reflections were used to refine cell parameters by a least-squares procedure. The cell parameters obtained are[12] $a = 20.050$ (3), $b = 24.269$ (4), and $c = 18.608$ (3) Å. The calculated density (1.34 g/cm³) and the experimental density (1.35 g/cm⁻³, determined by flotation in a CCl_4-hexane mixture) agree well for eight dinuclear units and eight molecules of toluene in the unit cell. Thus, the structural problem required the determination of all 61 nonhydrogen atoms in the dinuclear unit and the toluene group.

The X-ray intensity measurements were made, using the same crystal as used for the cell measurements, with Zr-filtered molybdenum Kα radiation. The intensities were measured with a scintillation counter mounted 26.5 cm from the crystal and a source–crystal distance of 21.0 cm; the intensities were collected by the θ-2θ scan technique with a takeoff angle of 1.9° and a scan rate of 1° min⁻¹. A symmetrical scan of 2.0° was taken about the calculated position for each reflection out to a maximum 2θ angle of 50° (λ 0.70926 Å). Stationary background counts of 20 sec were taken at the beginning (bgd1) and at the end (bgd2) of the scan. Calibrated copper attenuators were used in the collection of data. The threshold point was set so that the counting rate would not exceed 10,000 counts/sec. The pulse height analyzer was set for approximately a 90° window centered on the Mo Kα peak. Three orthogonal reflections (400, 080, 004) and a null reflection (700) were monitored after every 150 reflections as a check on crystal and instrument stability. No systematic drifts in the intensities of these standards was observed. A total of 2978 unique reflections were collected in a full octant of reciprocal space. Corrected intensities (CI) were obtained by subtracting three times the actual measured background from the total integrated peak count (CT).

$$\mathrm{CI} = \mathrm{CT} - 3(\mathrm{bgd1} + \mathrm{bgd2})$$

The factor of 3 in the calculation arises from the peak scan time being 3 times as long as the background counting time. The corrected intensities were assigned standard deviations according to the formula[13]

$$\sigma(I) = [\mathrm{CT} + 0.25(t_c/t_b)^2(\mathrm{bgd1} + \mathrm{bgd2}) + (pI)^2]^{1/2}$$

where t_c is the scan time and t_b is the counting time of each background either bgd1 or bgd2. A total of 1160 reflections were accepted as statistically above background on the basis that $\sigma(I)/\mathrm{CI}$ be less than 0.5 with p set at 0.02. Lorentz and polarization corrections were applied in the usual way.

Solution and Refinement of the Structure. Computations were carried out on the Burroughs 5500 and the Univac 1108 computers. Programs employed include Carter's cell parameter and diffractometer setting angle program, Zalkin's FORDAP Fourier summation program, Iber's NUCLS modification of the Busing-Martin-Levy ORFLS full-matrix least-squares program, the Martin-Busing-Levy ORFFE function and error program, Johnson's ORTEP plotting program, Stewart's ABSORB absorption correction program from the X-ray-72 system, and various locally written programs. In the structure factor calculations, the scattering factors were taken from Cromer and Waber's tabulation[14] for all atoms except hydrogen; Stewart's hydrogen atom scattering factors were used.[15] The scattering factors for iron and chlorine were corrected for the real and imaginary anomalous dispersion components, using the dispersion factors tabulated by Cromer.[16] The agreement factors are defined in the usual way as $R = (\Sigma||F_o| - |F_c||)/(\Sigma|F_o|)$ and $R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w(|F_o|)^2]^{1/2}$. In all least-squares refinements, the quantity minimized was $\Sigma w(|F_o| - |F_c|)^2$. Atomic coordinates for the two independent

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(8) R. F. Gould, Ed., "Bioinorganic Chemistry," ACS Monograph No. 100, American Chemical Society, Washington, D. C., 1971.

(9) $SALPAH_2$, SALPAH, and SALPA refer respectively to the neutral, anionic, and dianionic forms of the Schiff base 3-hydroxypropylsalicylaldimine.

(10) B. N. Figgis and J. Lewis, "Modern Coordination Chemistry," Wiley-Interscience, New York, N. Y., 1960, p 403.

(11) N. F. M. Henry and K. Lonsdale, Ed., "International Tables for X-Ray Crystallography," Vol. I, Kynoch Press, Birmingham, England, 1965.

(12) Numbers in parentheses here and elsewhere in this paper indicate estimated standard deviations in the least significant digits.

(13) P. W. R. Corfield, R. J. Doedens, and J. A. Ibers, *Inorg. Chem.*, 6, 197 (1967).

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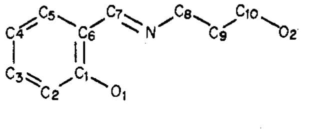
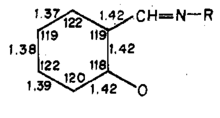
(15) R. F. Stewart, E. R. Davidson, and W. T. Simpson, *J. Chem. Phys.*, 42, 3175 (1965).

(16) D. T. Cromer, *Acta Crystallogr.*, 18, 17 (1965).

iron atoms were deduced from a three-dimensional Patterson synthesis and refined to give the initial residuals $R = 0.422$ and $R_w = 0.505$. Oxygen, nitrogen, and carbon atoms were located by means of subsequent difference Fourier syntheses and least-squares refinements. Because of the large size of the structural problem (61 nonhydrogen atoms), the toluene group and the phenyl portions of the four salicylaldehyde groups were refined as rigid groups of fixed geometry. D_{6h} symmetry (C-C = 1.37 Å) was assumed for the phenyl portion of the toluene group and the methyl-phenyl carbon distance was defined as 1.53 Å.[17] The geometry of the phenyl portion of the salicylaldehyde group is well known from a number of previous structure determinations;[17-22] an idealized structure based on an average of previously observed distances and angles is given below together with the numbering scheme used for each of the SALPA ligands in this structure





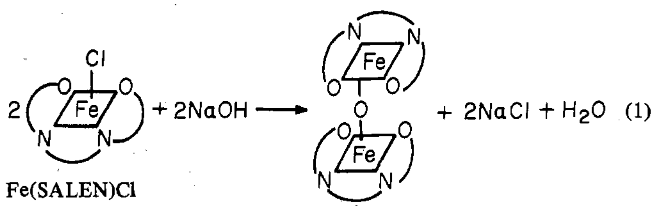
In later least-squares refinements an overall temperature factor, three translational parameters, and the three angular parameters were refined for each of the five groups. Using the above molecular model, a least-squares refinement (156 variables) with reflections weighted according to their estimated variances ($w = 4I/\sigma(I)^2$) gave the agreement factors $R = 0.1100$ and $R_w = 0.0802$. The six bounding faces of the crystal were identified by optical methods as {100}, {010}, and {001} and absorption corrections were applied by the gaussian quadrature method ($\mu = 7.23$ cm^{-1}). Correction factors for F^2 ranged between 1.09 and 1.51. Refinement after absorption improved the agreement slightly, $R = 0.103$ and $R_w = 0.076$. Refinement with anisotropic thermal parameters for the two iron atoms reduced R to 0.101 and R_w to 0.074.

At this point in the refinement, it was evident from the high temperature factors for the terminal atoms in the uncoordinated propyl group C (for atoms C9C, C10C, and O2C, B = 15.8, 10.5, and 13.6 Å^2, respectively) that this portion of the structure was affected by either disorder or severe libration. Careful examination of a series of electron density maps led to the conclusion that disorder, if any, was slight for propyl group C and that the high-temperature factors were due principally to librational effects. Refinement with anisotropic temperature factors for C9C, C10C, and O2C did indeed give a highly significant drop in R factors ($R = 0.098$, $R_w = 0.071$) and a marked improvement in geometry, but the temperature factors for C10C became nonpositive definite. Thus in the final refinement ($R = 0.098$, $R_w = 0.071$) anisotropic temperature factors were refined for C9C and O2C, and the isotropic temperature factor was refined for C10C (176 variables, 1160 reflections). In the final cycle of refinement, the maximum parameter shifts were $0.26\sigma(\beta_{33}$ for O2C) and $0.12\sigma(z$ coordinate for C10C). The major feature on a final electronic density map was a peak of 0.54 e/Å^3 near O2C, which compares to a value of *ca.* 2.0 e/Å^3 for a typical carbon atom in this structure. No attempts were made to locate the hydrogen atoms.

Final atomic parameters are listed in Table I; final observed and calculated structure factors are available.[23]

Discussion

Preparation and Properties of [Fe(SALPA)(SALPAH)]$_2$·toluene. The reactions of oxygen-containing bases with monomeric Schiff base complexes often yield magnetically coupled, dinuclear complexes containing nearly linear μ_2-oxo bridges.[6,7] For example, the pentacoordinate monomer Fe(SALEN)Cl (SALEN = 1,2-bis(salicylaldimine)ethane), reacts as shown schematically in eq 1. In attempting to

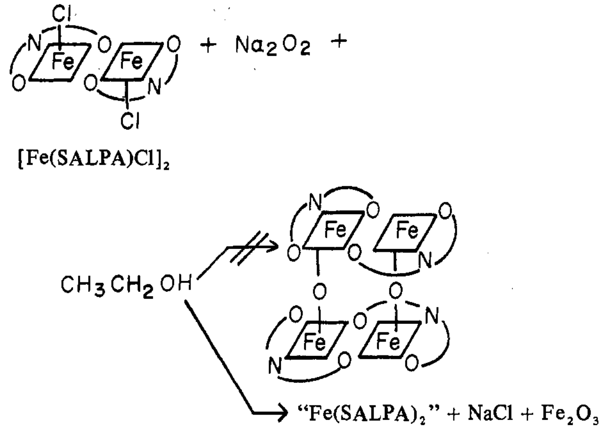


(1)

Fe(SALEN)Cl

[Fe(SALEN)]$_2$O

form, *via* an analogous reaction, a tetranuclear iron(III) complex containing both oxo bridges (as in [Fe(SALEN)]$_2$O) and four-membered Fe-O-Fe-O rings (as in [Fe(SALPA)Cl]$_2$),[1] we have studied the reaction of the pentacoordinate dimer [Fe(SALPA)Cl]$_2$ with various oxygen-containing bases such as hydroxide and peroxide. Although no oxo compounds have as yet been identified, the reaction with sodium hydroxide or sodium peroxide in ethanol did produce a compound having the unexpected tridentate ligand:metal ratio 2:1. Thus



[Fe(SALPA)Cl]$_2$

(2)

"Fe(SALPA)$_2$" + NaCl + Fe$_2$O$_3$

Ligand dissociation has obviously occurred and half the iron atoms in the starting complex have been converted to ferric oxide, leaving sufficient tridentate ligand to form the 2:1 complex. The great insolubility and thermodynamic stability of Fe$_2$O$_3$ undoubtedly provides a strong driving force for the reaction.

Since each ligand contains two ionizable protons (one phenolic and one alcoholic), the resulting complex could, depending on the number of protons lost, be neutral, anionic, or cationic; complexes of all three types have been reported for the reaction of iron(III) with salicylaldehyde semicarbazone.[24] Although an excess of base was employed in this study, only the neutral complex was isolated and only three of the four ionizable protons from the two ligands were removed. Since alcohols are generally less acidic than phenols, the remaining proton would be expected to be one of the alcohol protons.

Magnetic susceptibility measurements showed the compound to have marked antiferromagnetic behavior, requiring the molecular formulation to be at least dinuclear. Thus, the magnetic moment per iron atom at 298°K (4.45 BM) is well below the spin-only value for five unpaired electrons (6.0 BM) and the moment drops considerably as the temperature is reduced to 195°K (3.82 BM) and 77°K (2.34 BM). The approximate coupling constant represented by this behavior is typical for a polynuclear iron(III) complex containing a

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[17] L. Wei, R. M. Stogsdill, and E. C. Lingafelter, *Acta Crystallogr.*, 17, 1058 (1964).

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[19] E. C. Lingafelter, G. L. Simmons, B. Morosin, C. Scheringer, and C. Freiburg, *Acta Crystallogr.*, 14, 1222 (1961).

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[21] P. L. Orioli, E. C. Lingafelter, and B. W. Brown, *Acta Crystallogr.*, 17, 1113 (1964).

[22] M. R. Fox, P. L. Orioli, E. C. Lingafelter, and L. Sacconi, *Acta Crystallogr.*, 17, 1159 (1964).

[23] See paragraph at end of paper regarding supplementary material.

[24] N. M. Samus and V. G. Chebanu, *Russ. J. Inorg. Chem.*, 14, 1097 (1969).

Table I. Final Atomic Parameters for $[Fe_2(SALPA)_2(SALPAH)_2]\cdot$toluene

(A) Individual Positional and Isotropic Thermal Parameters, with Esd's[a]

Atom[b]	*x*	*y*	*z*	*B*, Å^2	Atom[b]	*x*	*y*	*z*	*B*, Å^2
Fe1	0.1866 (2)	0.1715 (2)	0.1646 (3)	*c*	O2B	0.1192 (10)	0.1364 (8)	0.1025 (11)	5.4 (6)
Fe2	0.0582 (2)	0.1953 (2)	0.0656 (3)	*c*	O1C	0.0180 (9)	0.2634 (7)	0.0356 (10)	4.3 (5)
O1A	0.2460 (11)	0.1070 (6)	0.1688 (12)	5.2 (5)	C7C	0.1067 (15)	0.2374 (13)	−0.0890 (17)	4.7 (8)
C7A	0.2944 (15)	0.1675 (13)	0.0482 (18)	4.6 (8)	NC	0.1058 (12)	0.1969 (11)	−0.0382 (12)	5.3 (7)
NA	0.2623 (10)	0.2025 (8)	0.0902 (12)	2.7 (5)	C8C	0.1371 (20)	0.1391 (15)	−0.0583 (24)	6.9 (11)
C8A	0.2733 (12)	0.2620 (10)	0.0834 (15)	2.9 (7)	C9C	0.1676 (35)	0.1367 (16)	−0.1221 (37)	*c*
C9A	0.2080 (14)	0.2922 (11)	0.0606 (19)	4.7 (8)	C10C	0.1932 (28)	0.0741 (21)	−0.1243 (31)	11.2 (16)
C10A	0.1529 (15)	0.2870 (12)	0.1197 (19)	4.9 (9)	O2C	0.2265 (20)	0.0732 (11)	−0.1806 (23)	*c*
O2A	0.1275 (11)	0.2288 (7)	0.1225 (11)	3.6 (5)	O1D	−0.0018 (11)	0.1414 (8)	0.0235 (12)	5.2 (5)
O1B	0.2268 (9)	0.2172 (7)	0.2368 (11)	3.6 (5)	C7D	−0.0676 (14)	0.1690 (10)	0.1612 (17)	3.6 (6)
C7B	0.1210 (15)	0.1668 (13)	0.3110 (17)	4.8 (8)	ND	−0.0101 (12)	0.1965 (10)	0.1561 (13)	4.3 (6)
NB	0.1316 (12)	0.1388 (10)	0.2582 (16)	4.7 (6)	C8D	0.0037 (14)	0.2366 (11)	0.2166 (16)	3.3 (7)
C8B	0.1085 (14)	0.0785 (12)	0.2546 (21)	4.8 (8)	C9D	−0.0178 (16)	0.2949 (12)	0.2077 (17)	5.2 (9)
C9B	0.0665 (19)	0.0690 (12)	0.1834 (22)	6.8 (9)	C10D	−0.0956 (14)	0.2989 (12)	0.1825 (17)	3.9 (7)
C10B	0.1004 (16)	0.0730 (13)	0.1149 (20)	4.9 (8)	O2D	−0.1349 (10)	0.2729 (7)	0.2399 (12)	5.0 (6)

(B) Anisotropic Thermal Parameters[d]

Atom	$10^5\beta_{11}$	$10^5\beta_{22}$	$10^5\beta_{33}$	$10^5\beta_{12}$	$10^5\beta_{13}$	$10^5\beta_{23}$
Fe1	0.0016 (1)	0.0016 (1)	0.0028 (2)	0.0001 (1)	0.0002 (2)	−0.0001 (1)
Fe2	0.0019 (1)	0.0016 (1)	0.0019 (2)	0.0001 (1)	−0.0000 (2)	−0.0003 (1)
C9C	0.0219 (50)	0.0013 (9)	0.0143 (43)	0.0030 (17)	0.0055 (38)	0.0011 (15)
O2C	0.0143 (25)	0.0029 (7)	0.0127 (23)	0.0013 (9)	0.0053 (20)	0.0031 (11)

(C) Rigid-Group Isotropic Temperature Factors and Derived Atomic Coordinates

Group	*B*, Å^2	Atom	*x*	*y*	*z*
Toluene	10.5 (6)	C1T	0.1686 (12)	0.4250 (37)	0.2008 (21)
		C2T	0.0930 (11)	0.4331 (18)	0.2079 (12)
		C3T	0.0564 (13)	0.4496 (15)	0.1495 (11)
		C4T	−0.0113 (13)	0.4568 (10)	0.1559 (15)
		C5T	−0.0423 (11)	0.4476 (19)	0.2205 (18)
		C6T	−0.0057 (13)	0.4311 (15)	0.2789 (14)
		C7T	0.0620 (13)	0.4239 (9)	0.2725 (11)
Benzene A	6.7 (4)	C1A	0.0820 (11)	0.2763 (18)	0.1119 (13)
		C2A	0.2828 (13)	0.0253 (10)	0.1204 (11)
		C3A	−0.0057 (7)	0.3139 (25)	0.0689 (14)
		C4A	0.0238 (11)	0.3395 (19)	0.0057 (12)
		C5A	0.3311 (12)	0.0805 (10)	0.0010 (12)
		C6A	0.1092 (7)	0.3000 (24)	0.0534 (15)
Benzene B	5.9 (4)	C1B	0.2005 (12)	0.2437 (8)	0.2934 (10)
		C2B	0.2242 (10)	0.2928 (9)	0.3198 (12)
		C3B	0.1931 (14)	0.3176 (8)	0.3782 (12)
		C4B	0.1392 (13)	0.2951 (9)	0.4115 (10)
		C5B	0.1143 (10)	0.2468 (10)	0.3872 (13)
		C6B	0.1426 (13)	0.2196 (8)	0.3291 (13)
Benzene C	4.7 (4)	C1C	0.0308 (16)	0.3031 (7)	−0.0093 (10)
		C2C	0.0057 (10)	0.3557 (9)	−0.0065 (10)
		C3C	0.0225 (17)	0.3937 (7)	−0.0595 (12)
		C4C	0.0635 (15)	0.3811 (7)	−0.1153 (10)
		C5C	0.0894 (9)	0.3298 (9)	−0.1200 (10)
		C6C	0.0749 (17)	0.2897 (7)	−0.0692 (12)
Benzene D	5.6 (4)	C1D	−0.0558 (15)	0.1171 (18)	0.0403 (13)
		C2D	−0.0825 (12)	0.0769 (10)	−0.0030 (10)
		C3D	−0.1421 (18)	0.0516 (12)	0.0161 (12)
		C4D	−0.1762 (14)	0.0648 (17)	0.0768 (14)
		C5D	−0.1516 (11)	0.1042 (10)	0.1209 (10)
		C6D	−0.0924 (18)	0.1313 (13)	0.1055 (12)

[a] Estimated standard deviations, esd's, are right-adjusted to the least significant digits of the preceding number. [b] A, B, C, and D label substituents for the individual salicylaldimine ligands. [c] Refined anisotropically. [d] The anisotropic thermal parameter is defined as $\exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - \beta_{12}hk - \beta_{13}hl - \beta_{23}kl)$.

four-membered Fe–O–Fe–O ring ($J \approx 5$–10 cm^{-1}) but is considerably smaller than the coupling constant ($J \approx 75$–100 cm^{-1}) usually observed for μ_2-oxo-bridged complexes.[25,26]

Molecular weight determination by osmometry in toluene at 37° indicates 33% dissociation of the dinuclear species into monomers in a saturated solution (2.7×10^{-3} *M*) and complete dissociation at infinite dilution. If the complex is formulated as a dimer containing a four-membered ring, either the coordination member of iron would have to be 7 or one of the ligands of the 2:1 complex would have to be bidentate rather than tridentate. Since alcohols are generally poor coordinating groups,[27] it seemed probable that the remaining alcohol group would be uncoordinated and the infrared spectrum did, in fact, exhibit a very broad, strong band centered at 3360 cm^{-1} which could be due to an uncoordinated, hydrogen-bonded alcohol group.

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(25) J. Lewis, F. E. Mabbs, and A. Richards, *J. Chem. Soc. A*, 1014 (1967).

(26) M. Gerloch, J. Lewis, F. E. Mabbs, and A. Richards, *J. Chem. Soc. A*, 112 (1968).

(27) J. A. Bertrand, J. A. Kelley, and J. L. Breece, *Inorg. Chim. Acta*, 4, 247 (1970).

Table II. Interatomic Distances (A) for $Fe_2(SALPA)_2(SALPAH)_2$·toluene

About Fe1		About Fe2	
Atoms	Dist	Atoms	Dist
Fe1-Fe2	3.217 (7)		
Fe1-O2A	1.988 (20)	Fe2-O2A	1.926 (21)
Fe1-O2B	1.972 (21)	Fe2-O2B	2.001 (21)
Fe1-O1A	1.970 (19)	Fe2-O1C	1.923 (19)
Fe1-O1B	1.919 (19)	Fe2-O1D	1.942 (21)
Fe1-NA	2.188 (22)	Fe2-NC	2.155 (24)
Fe1-NB	2.209 (28)	Fe2-ND	2.171 (25)
O2A-O2B	2.278 (26)		

Ligand A		Ligand B		Ligand C		Ligand D	
Atoms	Dist	Atoms	Dist	Atoms	Dist	Atoms	Dist
C6A-C7A	1.42 (3)	C6B-C7B	1.40 (3)	C6C-C7C	1.47 (3)	C6D-C7D	1.47 (3)
C7A-NA	1.32 (3)	C7B-NB	1.21 (3)	C7C-NC	1.36 (3)	C7D-ND	1.34 (3)
NA-C8A	1.47 (3)	NB-C8B	1.54 (3)	NC-C8C	1.58 (4)	ND-C8D	1.51 (3)
C8A-C9A	1.56 (3)	C8B-C9B	1.59 (5)	C8C-C9C	1.34 (6)	C8D-C9D	1.49 (4)
C9A-C10A	1.57 (3)	C9B-C10B	1.45 (4)	C9C-C10C	1.60 (6)	C9D-C10D	1.63 (4)
C10A-O2A	1.50 (3)	C10B-O2B	1.60 (3)	C10C-O2C	1.24 (5)	C10D-O2D	1.47 (3)
C1A-O1A	1.36 (3)	C1B-O1B	1.34 (3)	C1C-O1C	1.30 (3)	C1D-O1D	1.27 (3)

Table III. Selected Interatomic Angles (deg) for $[Fe_2(SALPA)_2(SALPAH)_2]$·toluene

(A) About Bridging Atoms

Atoms	Angle	Atoms	Angle
Fe1-O2A-Fe2	110.6 (9)	Fe1-O2B-Fe2	108.2 (9)
Fe1-O2A-C10A	118.0 (17)	Fe2-O2B-C10B	126.3 (16)
Fe2-O2A-C10A	128.5 (18)	Fe1-O2B-C10B	119.5 (17)

(B) Central Coordination Spheres

Atoms	Angle	Atoms	Angle
O2A-Fe1-O2B	70.2 (8)	O2A-Fe2-O2B	70.9 (8)
O2A-Fe1-NB	105.3 (9)	O2A-Fe2-ND	91.3 (8)
O2A-Fe1-NA	85.7 (9)	O2A-Fe2-NC	99.6 (9)
O2A-Fe1-O1B	97.0 (8)	O2A-Fe2-O1C	95.7 (8)
O2A-Fe1-O1A	158.8 (9)	O2A-Fe2-O1D	162.3 (8)
O2B-Fe1-NA	104.7 (8)	O2B-Fe2-ND	97.4 (9)
O2B-Fe1-NB	88.0 (9)	O2B-Fe2-NC	92.9 (9)
O2B-Fe1-O1B	161.3 (8)	O2B-Fe2-O1C	165.8 (8)
O2B-Fe1-O1A	95.4 (9)	O2B-Fe2-O1D	92.1 (8)
NA-Fe1-NB	165.7 (9)	NC-Fe2-ND	167.0 (9)
NA-Fe1-O1A	83.0 (9)	NC-Fe2-O1C	84.8 (9)
NA-Fe1-O1B	87.3 (8)	NC-Fe1-O1D	85.7 (9)
NB-Fe1-O1B	82.3 (9)	ND-Fe2-O1C	87.1 (9)
NB-Fe1-O1A	89.2 (9)	NC-Fe2-O1D	85.7 (9)
O1B-Fe1-O1A	100.3 (8)	O1C-Fe2-O1D	101.6 (8)

(C) Within Individual Ligands

Ligand A		Ligand B		Ligand C		Ligand D	
Atoms	Angle	Atoms	Angle	Atoms	Angle	Atoms	Angle
O2A-C10A-C9A	109.8 (23)	O2B-C10B-C9B	107.5 (30)	O2C-C10C-C9C	102.1 (47)	O2D-C10D-C9D	106.1 (25)
C10A-C9A-C8A	111.3 (27)	C10B-C9B-C8B	118.5 (32)	C10C-C9C-C8C	102.1 (46)	C10D-C9D-C8D	111.4 (23)
C9A-C8A-NA	111.1 (20)	C9B-C8B-NB	109.6 (27)	C9C-C8C-NC	115.5 (35)	C9D-C8D-ND	118.4 (25)
C8A-NA-C7A	120.6 (24)	C8B-NB-C7B	121.0 (29)	C8C-NC-C7C	118.0 (30)	C8D-ND-C7D	115.3 (25)
NA-C7A-C6A	129.6 (21)	NB-C7B-C6B	131.0 (25)	NC-C7C-C6C	116.3 (32)	ND-C7D-C6D	123.6 (22)
Fe1-NA-C7A	119.4 (18)	Fe1-NB-C7B	121.6 (27)	Fe2-NC-C7C	129.9 (31)	Fe2-ND-C7D	126.3 (21)
Fe1-NA-C8A	119.8 (21)	Fe1-NB-C8B	117.3 (22)	Fe2-NC-C8C	111.8 (31)	Fe2-ND-C8D	118.1 (18)
Fe1-O1A-C1A	126.4 (23)	Fe1-O1B-C1B	131.4 (22)	Fe2-O1C-C1C	137.8 (30)	Fe2-O1D-C1D	137.8 (21)

In formulating the complex as a dimer, there is also a question as to which groups are bridging since both alkoxide and phenoxide groups are capable of bridging. From previous studies, alkoxide groups seem to be better as bridging groups and would be expected to bridge in this complex.[27]

Description of the Crystal Structure of $[Fe_2(SALPA)_2(SALPAH)_2]$·toluene. Relevant bond distances and angles are given in Tables II and III; least-squares planes are given in Table IV. The structure of $[Fe_2(SALPA)_2(SALPAH)_2]$·toluene consists of well-separated molecules of $Fe_2(SALPA)_2(SALPAH)_2$ and toluene of solvation. As inferred from the chemical data, the metal complex, Figure 1, consists of a dinuclear unit involving two six-coordinate iron atoms linked by an almost symmetric Fe-O-Fe-O ring. One of the iron atoms is coordinated by two tridentate ligands (A and B); each of the ligands chelated to this iron is completely deprotonated and this portion of the complex could be thought of as an anionic complex, $Fe(SALPA)_2^-$. Each of these ligands occupies three planar coordination sites with the nitrogens of the two ligands trans. The two alkoxide groups, which occupy cis-coordination positions, are also coordinated to the second iron atom and provide the bridging atoms for the dimeric unit. The second iron is also coordinated to the remaining two ligands (C and D), each serving as bidentate ligands with uncoordinated alcohol groups. These two ligands are coordinated in such a way that the nitrogens are again trans. The two iron atoms, thus, have virtually identical donor sets and geometries but they are,

Table IV. Least-Squares Planes[a,b] within the $Fe_2(SALPA)_2(SALPAH)_2$ Molecule

Atom	Dev, A	Atom	Dev, A
(A) Four-Membered Iron-Oxygen Ring			
Equation: $0.6005x + 0.1149y - 0.7913z = 0.3176$			
Fe1	0.02	C10A*	−0.56
Fe2	0.04	C10B*	0.60
O2A	−0.05		
O2B	0.01		
(B) Iron Atoms and the Six Coordinated Oxygen Atoms			
Equation: $0.6091x + 0.4998y - 0.6159z = 2.4941$			
Fe1	0.02	O1B	−0.20
Fe2	0.17	O2B	0.56
O1A	0.13	O1C	−0.51
O2A	−0.43	O1D	1.07
(C) Coordination Plane for Fe1			
Equation: $0.6565x + 0.3791y - 0.6522z = 2.0692$			
Fe1	0.03	O1B	−0.04
O1A	−0.11	O2B	0.49
O2A	−0.23		
(D) Coordination Plane for Fe2			
Equation: $0.5421x - 0.0268y - 0.8399z = -0.5369$			
Fe2	−0.02	O1C	0.00
O2A	0.14	O1D	−0.06
O2B	−0.14		
(E) O2A Coordination			
Equation: $0.5459x - 0.1319y - 0.8274z = -1.0940$			
Fe1	−0.05	Fe2	−0.10
O2A	0.13	C10A	−0.01
(F) O2B Coordination			
Equation: $0.5803x - 0.0816y - 0.8103z = -0.6216$			
Fe1	0.03	Fe2	0.08
O2B	−0.19	C10B	0.09

[a] Planes are derived using unit weights for all unmarked atoms and zero weights for all atoms listed with asterisks. [b] The planes are in cartesian coordinates with the x axis coincident to a and the z axis coincident to c^*.

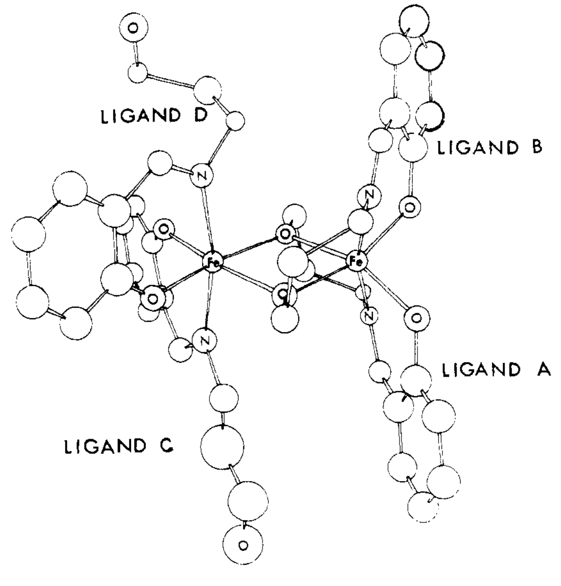

Figure 1. Structure of the $Fe_2(SALPA)_2(SALPAH)_2$ molecule.

in terms of the overall complex, definitely nonequivalent. As implied from the stoichiometry of the complex, two of the four propanol groups are neutral and uncoordinated, but a surprising feature of the structure is the fact that the chelates containing the two propoxide groups are attached to the same iron atoms.

The Fe-O-Fe-O ring is almost planar, the maximum deviation from the plane of the four atoms being 0.05Å (O2A).

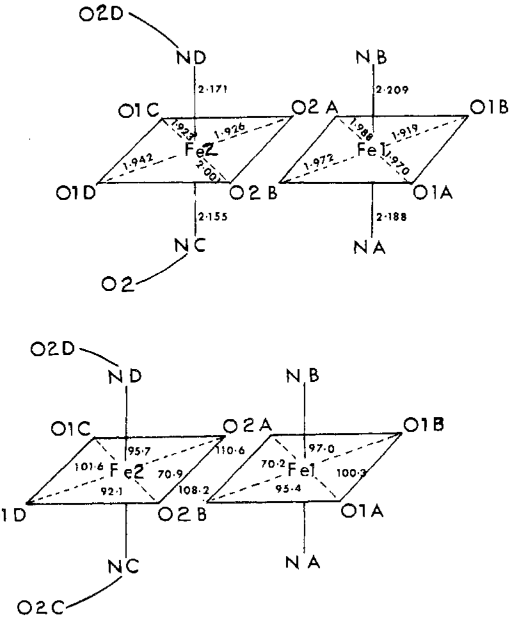

Figure 2. Geometry of the inner coordination sphere of $[Fe_2(SALPA)_2(SALPAH)_2]$·toluene.

The dihedral angle between the Fe1-O2A-Fe2 and Fe1-O2B-Fe2 planes is 3.4 (9)°. Angles at oxygen (110.6 (9), 108.2 (9)°) and iron (70.9 (8), 70.2 (8)°) are typical for a symmetrical Fe-O-Fe-O ring.[1,5,7] The Fe-O (bridging (1.93 (2)-2.00 (2) Å), the Fe-O(terminal) (1.92 (2)-1.97 (2) Å), and the Fe-N (2.16 (2)-2.21 (3) Å) distances are also in the ranges normally observed for iron(III)-Schiff base complexes.[1,5,7,9,28]

The carbon atoms, C10A and C10B, attached to the bridging oxygens, O2A and O2B, deviate significantly from the plane of the four-membered iron-oxygen ring (deviations of −0.56 and 0.60 Å from the plane). The coordination of the bridging oxygen atoms is, thus, farther from a planar arrangement than the coordination of the bridging oxygen in the structure of the toluene solvate of $[Fe(SALPA)Cl]_2$, in which the carbon atom was 0.28 Å from the plane of the four-membered ring. The coordination of the bridging oxygen in these iron(III) complexes differs significantly from that found for several copper(II) complexes and argues against a π pathway for spin exchange between the iron atoms.

The preference of an Fe-O-Fe-O ring for an O-Fe-O angle close to 71° results in distortions in the remaining angles in the coordination sphere from ideal octahedral symmetry. An increase from the ideal value of 90° occurs for the angles O(bridge)-Fe-O(terminal) (92.1 (8)-97.0 (8)°) and O(terminal)-Fe-O(terminal) (100.3 (8), 101.6 (8)°). Each FeO_4 group varies somewhat from planarity (maximum deviation 0.49 Å) and steric requirements for the tridentate ligands produce variations from 90° for the O-Fe-N angles (83.0 (9)-105.3 (9)°) and variations from 180° for the N-Fe-N linkages (165.7 (9), 167.0 (9)°).

The uncoordinated propyl groups of ligands C and D are involved in hydrogen bonding to phenolic oxygens O1A and O1B and this hydrogen bonding links the dinuclear species into an infinite network. Thus, the distance O2D-O1B′ (where the prime denotes the atom related by a glide operation) is a typical O-O distance (2.79 (3) Å) for hydrogen

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(28) J. E. Davies and B. M. Gatehouse, *Chem. Commun.*, 1166 (1970).

Table V. Electronic Spectra of Several Schiff Base Ligands and Iron(III) Complexes[a-c]

Compd		ν_1	ν_2	ν_3	ν_4	ν_5	ν_6	ν_7
(A) Ligands								
SANE						31.8 (1670)	36.7 (1950)	39.5 (5300)
SALEN						31.8 (1850)	35.8 sh (1240)	39.4 (6210)
SALPNAD						31.9 (2045)	36.2 sh (2700)	39.5 (8050)
SAL						30.9 (3450)		39.5 (11,300)
(B) Six-Coordinate Iron(III) Complexes								
$Fe_2(SO_4)_3 \cdot (NH_4)_2SO_4 \cdot 24H_2O$	Six-coordinate monomer	12.6 (0.05)	18.2 (0.01)	24.2 24.6 (1.3) 25.4	27.7 (1.0)			
$Fe(SAL)_3$	Six-coordinate monomer	9.53 (1.3)	13.7 (2.2)	23.1 (4140)	28.3 (4150)	29.2, 32.5	36.8	42.6
$[Fe(HEDTA)]_2O^{2-}$	μ_2-Oxo-bridged octahedral dimer	11.2 (2.6)	18.2 (40)	21.0 (25)				
$Fe_2(SALPA)_2(SALPAH)_2$	Fe–O–Fe–O bridged octahedral "dimer"	10.5 sh (630)	18.2 (9320)	22.0 sh (10,000)	25.0 (14,400)	32.2 (28,900)	36.4 (33,800)	41.8 sh (46,000)
(C) Five-Coordinate Iron(III) Complexes								
$Fe(SANE)_2Cl$	Distorted square-pyramidal monomer			18.6 (4200)	25.0 (3240)	32.3 (5600)	35.9 (6800)	41.0 sh (11,500)
$Fe(SALEN)Cl$	Square-pyramidal monomer			20.0 (1870)	25.0 sh (3020)	31.0 sh (7000)	36.0 sh (10,500)	
$[Fe(SALPA)Cl]_2$	Fe–O–Fe–O bridged square-pyramidal "dimer"			21.1 (2400)	26.5 sh (3000)	31.8 (7200)	38.9 (20,300)	

[a] Uv and visible spectra were measured on 10^{-4} *M* methanol solutions. Frequencies in kK with extinction coefficients (per formula unit) in parentheses. [b] SANE = *N*-(β-phenylethyl)salicylaldimine, SALPNAD = *N*-(β-hydroxyphenylethyl)salicylaldimine, SALEN = 1,2-bis(salicylaldimine)ethane, SAL = salicylaldehyde. [c] sh = shoulder.

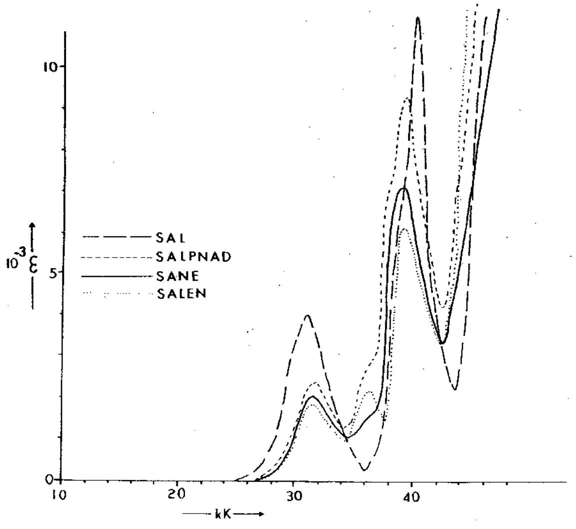

Figure 3. Uv spectra of salicylaldehyde and several salicylaldimine ligands.

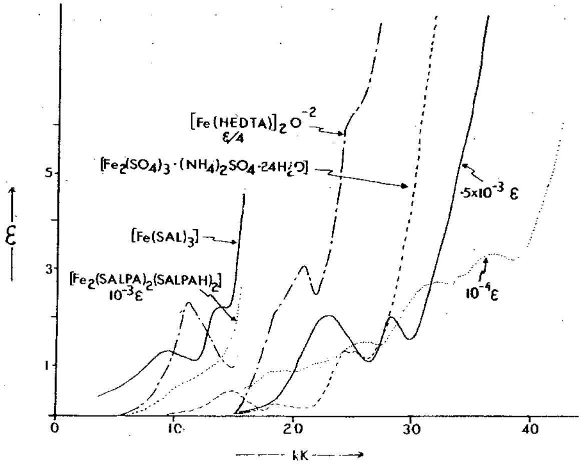

Figure 4. Electronic spectra of six-coordinate mono- and dinuclear Fe(III)–Schiff base complexes. Intensity scale is changed at *ca.* 15,000 kK and multiplied by the indicated factors.

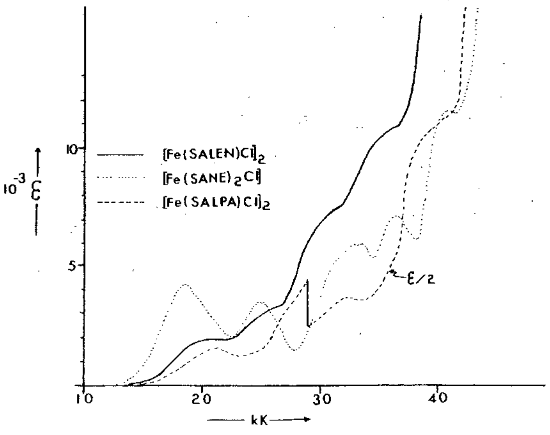

Figure 5. Electronic spectra of pentacoordinate and mono- and dinuclear iron(III)–Schiff base complexes.

bonding. Similarly the O2C-O1A′ distance (2.94 (3) Å) is reasonable for a second hydrogen bond.

With the exception of the terminal atoms in propyl group C, all the distances and angles in the imine and side chain linkages are normal. As discussed in the structure determination section, this portion of the structure appears to suffer from librational effects and the bond angles and distances at C9C, C10C, and O2C scatter somewhat from ideal values.

Spectral Investigations. Electronic spectral data for several salicylaldehyde derivatives are given in Table VA and Figure 3. For salicylaldehyde, two charge-transfer bands are observed at 30.9 and 39.5 kK and are assignable to the $n \rightarrow \pi^*$ and $\pi \rightarrow \pi^*$ transitions involving the conjugated aldehyde chromophore.[29] For the salicylaldimine compounds these two bands are observed at nearly the same energy and a third band, near 36 kK, is also found. A distinct tail into the visible region from the absorption envelope of these bands is responsible for the characteristic yellow color of the imines.

Since high-spin iron(III) complexes contain five unpaired electrons, all d-d transitions are spin forbidden and, hence, should be weak. Although most ferric complexes do show weak d-d absorption bands,[30] electronic spectra for salicylaldehyde and imine complexes of iron(III) exhibit remarkably intense d-d absorptions (Table V; Figures 3-5). For example, both ferric ammonium sulfate and ferric salicylaldehyde contain pseudooctahedral FeO_6 cores, yet extinction coefficients for the bands above 20 kK are several thousand times more intense for the salicylaldehyde complex. Much of the intensity may be ascribed to borrowing from the low-lying charge-transfer bands in the imine ligand, an interpretation supported by the fact that intensities of the bands below 20,000 kK for $Fe(SAL)_3$ are characteristically weak. It is of interest to note that the d-d bands for the FeO_6 Schiff base complexes are not significantly less intense than for d-d bands of the acentric, square-pyramidal complexes listed in Table VC.

The electronic spectra of the complexes containing a pseudooctahedral $Fe^{III}O_6$ core are quite similar, regardless of whether the complex contains isolated or magnetically coupled species. A pattern of four bands is observed and can be assigned, in order of increasing energy, to the ${}^6A_1 \rightarrow {}^4T_1$, ${}^6A_1 \rightarrow {}^4T_2$, ${}^6A_1 \rightarrow ({}^4A_1, {}^4E)$, and ${}^6A_1 \rightarrow {}^4T_2$ transitions, assuming idealized octahedral symmetry.[30] Thus, even though significant electron pairing *via* superexchange coupling (leading to a ground-state singlet[25,26]) has occurred for the oxygen-bridged species (*e.g.*, μ_{eff} at 298°K for $Fe_2(SALPA)_2(SALPAH)_2$ is reduced to 4.52 BM from the free-spin value of 5.93 BM), the electronic spectra may still be interpreted on the basis of isolated, free-spin iron(III) centers. Gray has commented on this remarkable feature of the spectra of polynuclear iron(III) complexes and we have made similar observations with polynuclear magnetically coupled copper(II) complexes.[8,31] Apparently the d-orbital energy levels are coupled sufficiently to allow significant reduction of unpaired spin yet not so strongly coupled as to alter greatly the energy differences between the d orbitals.

A series of relatively intense bands in the ultraviolet region for oxygen-bridged iron(III) complexes containing nonchromophoric ligands has been observed[8] and assigned[32-34] as "simultaneous pair excitations," *i.e.*, transitions involving simultaneous promotion of an electron on each half of the dinuclear complex. Although we had hoped similar bands might be observable in the spectra of the polynuclear SALPA complexes as shoulders on the charge-transfer bands, the ultraviolet spectra of the complexes are, in fact, little changed from that of the free ligands.

Electronic spectra for several five-coordinate Schiff base complexes with square-pyramidal geometries are compared in Figure 5 and Table VC. Although magnetically dilute and spin-coupled species are represented, the spectra of the complexes are quite similar. Two intense d-d absorptions are noted for each of the pentacoordinate complexes near 20 and 25 kK, with probable assignments to the transitions of $d_{x^2-y^2} \rightarrow d_{z^2}$ and to $d_{x^2-y^2} \rightarrow d_{xz,yz}$ character, respectively.

Conclusions

The reaction of $[Fe(SALPA)Cl]_2$ with oxygen-containing bases yields a product with empirical composition Fe(SALPA)(SALPAH). A crystal structure determination has shown the compound to contain a dinuclear $Fe_2(SALPA)_2(SALPAH)_2$ unit with a four-membered Fe-O-Fe-O ring, a structural feature permitting a reduction of the magnetic moments of the iron atoms *via* superexchange coupling. The electronic spectra of several mononuclear and dinuclear Schiff base complexes of iron(III), having five- and six-coordinate structures, have been interpreted. The pentacoordinate complexes show two bands in the visible region whereas the six-coordinate structures exhibit four bands. Although the oxygen-bridged dinuclear species have considerable d-electron pairing *via* superexchange, the spectra are very similar to isolated mononuclear complexes having similar geometry and simultaneous pair excitations are not observed.

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Registry No. $[Fe_2(SALPA)_2(SALPAH)_2]$, 49788-37-6; SANE, 49792-36-1; SALEN, 94-93-9; SALPNAD, 49788-35-4; SAL, 90-02-8; $Fe_2(SO_4)_3 \cdot (NH_4)_2SO_4 \cdot 24H_2O$, 7783-83-7; $Fe(SAL)_3$, 21127-11-7; $[Fe(HEDTA)]_2O^{2-}$, 30230-33-2; $Fe(SANE)_2Cl$, 42294-92-8; Fe(SALEN)Cl, 35828-37-6; $[Fe(SALPA)Cl]_2$, 49788-40-1; Na_2O_2, 1313-60-6.

Supplementary Material Available. A listing of structure factor amplitudes will appear following these pages in the microfilm edition of this volume of the journal. Photocopies of the supplementary material from this paper only or microfiche (105 × 148 mm, 24× reduction, negatives) containing all of the supplementary material for the papers in this issue may be obtained from the Journals Department, American Chemical Society, 1155 16th St., N.W., Washington, D. C. 20036. Remit check or money order for $3.00 for photocopy or $2.00 for microfiche, referring to code number INORG-74-927.

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