termination of D. The data in the present case are not sufficient to define g_x, g_y, and E separately. Equally good fits can be obtained with the following parameter sets for $Co[H_2B(pz)_2]_2$, in the one case holding $E = 0$ and in the other holding g_x and g_y equal to g_z.

$D = 0.43\ cm^{-1}$	$D = 0.44\ cm^{-1}$
$E = 0$	$E = -0.19\ cm^{-1}$
$g_x = 2.10$	$g_x = 2.30$
$g_y = 2.50$	$g_y = 2.30$
$g_z = 2.30$	$g_z = 2.30$

The calculated values of g_x', g_y', and g_z' are 0.95, 1.13, and 6.90, respectively. Similar ambiguity is, of course, found for $\{H_2B[3,5\text{-}(CH_3)_2(pz)_2]_2\}_2Co$.

Figure 7 shows the Calcomp outputs for the two molecules when the H_0 field is in the x direction. For the first compound there is only one possible high-field transition; for the second compound the calculated zero-field splitting is an order of magnitude smaller and there are six potentially observable transitions. Under these latter conditions it is difficult to see why only one transition is observed. The levels are heavily mixed, they are all thermally populated at 4.2°K, and one might anticipate similar relaxation times and transition probabilities for the possible transitions.

The interpretation given above appears to be the most likely on the basis of the data currently available but cannot be regarded as unambiguous. Experiments which might help to resolve this problem are (a) epr studies below 4.2°K where T_{1e} for the $\pm^1/_2$ doublet may be shortened enough to allow detection of the $\pm^1/_2$ resonance, (b) studies of the temperature dependence and anisotropy of the magnetic susceptibility near 4.2°K, and (c) epr studies at K-band frequencies. We are not currently equipped to perform these experiments but hope eventually to do some or all of them.

Conclusions

Single-crystal X-ray data, including the molecular and crystal structures, have been presented for $[H_2B(pz)_2]_2Co$. The Co coordination is essentially tetrahedral, but the distortions from a regular tetrahedron are significant (96–122° N–Co–N angles). The overall molecular symmetry is idealized $C_2(2)$. There are no unusual intermolecular contacts. Individual structural parameters are compared with related compounds.

The epr studies illustrate some of the difficulties which may be encountered in using this approach to define the magnetic properties of the central metal ion and seem to represent a unique case in tetrahedral Co^{2+} where a forbidden transition is observed but the principal allowed transition is not.

The quite large variation in the apparent g values and in the zero-field splittings in the substituted complexes also suggest that caution must be exercised in transferring magnetic data obtained in the solid state to the analysis of solution properties such as isotropic resonance shifts. It is possible that these changes in g values are direct effects of the substituents at the molecular level, but it is also possible that they are due to crystal packing effects. A clearer definition of the magnetic properties of the central metal ion in these complexes will be required before detailed interpretation of the isotropic nuclear resonance shifts of the ligand protons can be undertaken.

Registry No. $[H_2B(pz)_2]_2Co$, 37668-73-8.

Acknowledgment. We wish to thank Professor F. A. Cotton for communicating his results prior to publication.

Contribution from Ames Laboratory–USAEC and the Department
of Chemistry, Iowa State University, Ames, Iowa 50010

Crystal Structure of Hexaamminecobalt Nonafluoroantimonate(III)

DONALD R. SCHROEDER and ROBERT A. JACOBSON*

Received August 15, 1972

The crystal structure of hexaamminecobalt nonafluoroantimonate(III), $Co(NH_3)_6Sb_2F_9$, has been solved by single-crystal X-ray diffraction techniques at room temperature (25 ± 2°) using three-dimensional scintillation counter data and a full-matrix anisotropic least-squares refinement procedure ($R = 0.060$). The compound crystallizes in the monoclinic crystal system of space group $P2_1/c$, with cell lattice parameters $a = 7.223$ (5), $b = 7.298$ (3), $c = 12.432$ (7) Å and $\beta = 93.15$ (8)°. The asymmetric unit consists of one octahedral $Co(NH_3)_6{}^{3+}$ ion and one distorted $Sb_2F_9{}^{3-}$ ion. The $Sb_2F_9{}^{3-}$ ion resembles two octahedra sharing a corner with each octahedron possessing a lone pair. It was also found that the bridging fluorine in this compound is statistically disordered. The bridge is asymmetric with Sb–F bonds of 2.249 (12) and 2.449 (4) Å. The Sb–F bond length opposite the lone pair is 1.948 (7) Å while the Sb–F distance opposite the bridging fluorine is 2.015 (5) Å. The remaining two Sb–F bonds are trans to one another with distances of 2.080 (11) and 2.078 (8) Å, respectively. The mean Co–N distance is 1.990 (5) Å.

Introduction

The crystal structure investigation of hexaamminecobalt nonafluoroantimonate(III) was undertaken as part of a series of investigations of halo-coordinated antimony compounds being performed in this laboratory.[1-6] The stereochemistry and the role, if any, the lone pair plays in the coordination sphere are of particular interest in these studies.

---

(1) C. R. Hubbard and R. A. Jacobson, *Proc. Iowa Acad. Sci.*, **75**, 85 (1968).

(2) S. L. Lawton and R. A. Jacobson, *Inorg. Chem.*, **7**, 2124 (1968).

(3) S. L. Lawton and R. A. Jacobson, *Inorg. Chem.*, **5**, 743 (1966).

(4) C. R. Hubbard and R. A. Jacobson, *Inorg. Chem.*, **11**, 2247 (1972).

(5) S. K. Porter and R. A. Jacobson, *J. Chem. Soc. A*, 1356 (1970).

(6) W. Pflaum and R. A. Jacobson, to be submitted for publication.

Antimony trifluoride is typical of the other antimony halides in the sense of forming a variety of cation–antimony(III) halide complexes. Numerous structural studies have been carried out on the bromide complexes with fewer studies of the corresponding chlorides or fluorides having been reported. Crystallographic structural results on some fluoride complexes have been reported by Bystrom and coworkers[7-9] although their conclusions have recently been questioned by Ryan.[10] In this laboratory structures of $[(CH_3)_4N]_3Sb_2Br_9{\cdot}Br_2$,[4] $(C_5H_5NH)_5Sb_2Br_9{\cdot}Br_2$,[5] and $Cs_3Sb_2Cl_9$,[6] all of which contain $Sb_2X_9{}^{3-}$ ions, have recently been investigated by X-ray diffraction techniques. It is worthwhile to note the different bridging observed in the three cases. In $(C_5H_5NH)_5Sb_2Br_9{\cdot}Br_2$, the $Sb_2Br_9{}^{3-}$ anion was found to consist of two $SbBr_6$ octahedra sharing a face. In $[(CH_3)_4N]_3Sb_2Br_9{\cdot}Br_2$, the same anion is found but in this case the anions are bridged by Br_2 molecules ($Br-Br_2$ distance of 2.89 (1) Å). However in $Cs_3Sb_2Cl_9$, although three chlorines are of the bridging type, all form linear bridges producing a three-dimensional network in the crystal. Since a fluorine complex with formula $Co(NH_3)_6Sb_2F_9$ has been recently prepared, we felt a structural investigation of this complex would form a significant addition to the $Sb_2X_9{}^{3-}$ series.

Experimental Section

A sample of the compound was kindly supplied by Dr. Birchall[11] of McMaster University. The crystals obtained were of good quality and no recrystallization was necessary. Microscopic examination revealed that the crystals were cubic in shape with sharply defined faces and exhibited a bright orange color. They were also found to be stable in the atmosphere; therefore the crystal used for data collection was mounted on a glass fiber.

Crystal Data. Crystals of $Co(NH_3)_6Sb_2F_9$, examined by precession and Weissenberg film techniques, were found to exhibit $2/m$ Laue symmetry indicating a monoclinic space group. The following systematic absences were observed: $h0l$ when $l = 2n + 1$ and $0k0$ when $k = 2n + 1$. These absences are consistent only with the space group $P2_1/c$. The unit cell parameters at 25° are $a = 7.223$ (5), $b = 7.298$ (3), $c = 12.432$ (7) Å, and $\beta = 93.15$ (8)°. These parameters and their standard deviations were obtained by a least-squares fit to 12 independent reflection angles whose centers were determined by left–right, top–bottom beam splitting on a previously aligned Hilger-Watts four-circle diffractometer (Mo Kα radiation, λ 0.71069 Å). Any error in the instrumental zero was eliminated by centering the reflection at both $+2\theta$ and -2θ. A calculated density of 2.89 g cm^{-3} for 2 molecules/unit cell agrees quite well with the observed density of 2.84 g cm^{-3} which was determined by flotation techniques.

For data collection, a crystal having approximate dimensions 0.081 × 0.104 × 0.095 mm along the a, b, and c crystal axes, respectively, was mounted such that the b axis was coincident with the spindle axis.

Data were collected at room temperature utilizing a Hilger-Watts four-circle diffractometer equipped with scintillation counter and using Zr-filtered Mo Kα radiation. Within a 2θ sphere of 50°, all data in each of two octants were recorded using the θ–2θ scan technique with a takeoff angle of 3°. Symmetric scan ranges of 1.00° in 2θ at low 2θ values to 2.20° at the high 2θ limit were used. Stationary-crystal, stationary-counter background measurements were made at the beginning and end of each scan for half the total scan time. The counting rate used was 0.2048 sec/step of 0.02° in 2θ. The rest of the experimental arrangement used has already been discussed in some detail.[12] A total of 1317 reflections were measured in this way of which 1037 were found to be $\geqslant 3\sigma(F)$ and therefore considered observed.

Table I. Final Positional Parameters for $Co(NH_3)_6Sb_2F_9{}^{a,b}$

Atom	Position	x	y	z
Sb	4e	0.03496 (11)	0.01997 (9)	0.32524 (6)
Co	2b	0.5*	0.0*	0.0*
F(1)	4e	0.3163 (15)	0.0077 (13)	0.3721 (14)
F(2)	4e	0.1314 (12)	0.1066 (11)	0.1864 (6)
F(3)	4e	0.7878 (10)	0.1425 (11)	0.2688 (7)
F(4)	4e	0.0832 (9)	0.2674 (9)	0.3771 (6)
F(5)	4e	0.0988 (33)	0.5315 (26)	0.0175 (14)
N(1)	4e	0.7595 (13)	0.0857 (13)	0.0137 (8)
N(2)	4e	0.4171 (14)	0.2361 (13)	0.0576 (8)
N(3)	4e	0.4771 (13)	0.3991 (14)	0.3496 (7)

[a] In this and succeeding tables, numbers in parentheses indicate standard deviations in least significant digits. [b] An asterisk indicates an atomic coordinate fixed by symmetry.

As a general check on electronic and crystal stability, the intensities of three standard reflections were remeasured every 25 reflections during the data collection period. These reflections showed no decrease in intensity over the entire period of data collection.

The intensity data were also corrected for Lorentz–polarization effects. The estimated error in each intensity was calculated by $[\sigma(I)]^2 = [C_t + C_b + (0.03C_t)^2 + (0.05C_b)^2]$ where C_t and C_b are the total count and background count, respectively. The estimated standard deviation in each structure factor, $\sigma(F_o)$, was calculated by the finite-difference method.[13] These standard deviations were used during the least-squares refinement to weight the observed structure factors, where w, the individual weighting factor, was defined as $1/\sigma(F_o)^2$. Transmission factors ranged from 0.67 to 0.62 based on a linear absorption coefficient of 53.32 cm^{-1}; no absorption corrections were therefore applied.

Solution and Refinement of Structure

Examination of the Patterson function, which was calculated from sharpened data,[14] coupled with restrictions imposed by the space group led to ready location of the heavy atoms. Since there are four general positions but only two molecules per unit cell, the cobalt atoms must lie on positions of $\bar{1}$ site symmetry while the two antimony atoms must be related by $\bar{1}$ symmetry. The remaining atoms were found by successive structure factor and electron density map calculations. No fluorine atom was found on a center of symmetry site; however, two statistically disordered bridging fluorines were found and given half-occupancy.

These positions were then refined by full-matrix least-squares techniques[15] with isotropic thermal parameters to a conventional discrepancy factor of $R = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 10.3\%$ and a weighted R factor $wR = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2} = 11.5\%$. The scattering factors were those of Hanson, *et al.*,[16] with antimony and cobalt modified for the real and imaginary parts of anomalous dispersion.[17] A difference electron density map at this stage illustrated that all nonhydrogen atoms had been accounted for but that some anisotropic motion, particularly of the heavier atoms, was quite evident. Anisotropic refinement of all atoms heavier than hydrogen was begun and after six cycles of least-squares the discrepancy factor converged at 0.060 ($wR = 0.072$). On the final cycle the shift in each positional and thermal parameter averaged 0.004 times its own σ. A final electron density map showed no peaks higher than 0.5 e/Å^3. A final statistical analysis of the $|F_o|$ and $|F_c|$ values as a function of the indices, the scattering angle, and the magnitude of F_o showed no unusual trends and suggests that the relative weighting scheme used is a reasonable one. The final positional parameters are given in Table I and the final thermal parameters in Table II, along with their standard deviations

---

(7) K. Wilhelmi and A. Bystrom, *Ark. Kemi*, 3, 373 (1951).

(8) K. Wilhelmi and A. Bystrom, *Ark. Kemi*, 3, 17 (1950).

(9) K. Wilhelmi, S. Backlund, and A. Bystrom, *Ark. Kemi*, 6, 77 (1952).

(10) S. H. Mastin and R. R. Ryan, *Inorg. Chem.*, 10, 1757 (1971).

(11) T. Birchall, Department of Chemistry, McMaster University, Hamilton, Ontario, Canada, personal communication.

(12) W. Ozbirn and R. A. Jacobson, *Inorg. Chim. Acta*, 4, 377 (1970).

(13) D. E. Williams and R. E. Rundle, *J. Amer. Chem. Soc.*, **86**, 1660 (1964).

(14) R. A. Jacobson, J. A. Wunderlich, and W. N. Lipscomb, *Acta Crystallogr.*, 14, 598 (1961).

(15) W. R. Busing, K. O. Martin, and H. A. Levy, "ORFLS, a Fortran Crystallographic Least-Squares Program," Report ORNL-TM-305, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1962.

(16) H. P. Hanson, F. Herman, J. D. Lea, and S. Skillman, *Acta Crystallogr.*, 17, 1040 (1964).

(17) "International Tables for X-Ray Crystallography," Vol. III, Kynoch Press, Birmingham, England, 1962, pp 215-216.

Table II. Final Anisotropic Thermal Parameters (×10^4) for $Co(NH_3)_6Sb_2F_9$ [a]

Atom	Position	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Sb	4e	131 (2)	55 (1)	25 (0)	−11 (1)	0 (0)	0 (0)
Co	2b	74 (4)	62 (3)	23 (1)	−4 (3)	5 (1)	0 (1)
F(1)	4e	243 (27)	252 (27)	348 (25)	135 (21)	−193 (22)	−185 (20)
F(2)	4e	336 (26)	156 (17)	56 (6)	14 (17)	73 (10)	−3 (9)
F(3)	4e	129 (17)	219 (20)	103 (8)	19 (15)	−4 (9)	−35 (10)
F(4)	4e	148 (15)	87 (13)	60 (6)	−6 (11)	−14 (7)	−15 (7)
F(5)	4e	605 (91)	210 (47)	41 (13)	71 (53)	63 (29)	−6 (19)
N(1)	4e	102 (21)	100 (19)	42 (7)	−26 (16)	18 (10)	0 (10)
N(2)	4e	158 (23)	79 (18)	43 (7)	−9 (17)	23 (11)	3 (10)
N(3)	4e	118 (21)	140 (22)	25 (6)	−5 (17)	25 (9)	−1 (10)

[a] The form of the anisotropic temperature factor expression is $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$.

Table III. Selected Bond Distances and Angles in $Co(NH_3)_6Sb_2F_9$

(a) Distances, Å

Atoms	Length	Atoms	Length
Sb-F(1)	2.080 (11)	Sb-F(5′)	2.449 (4)
Sb-F(2)	2.015 (5)	Sb-Sb′	4.444 (0)
Sb-F(3)	2.078 (8)	Co-N(1)	1.973 (3)
Sb-F(4)	1.948 (7)	Co-N(2)	1.980 (9)
Sb-F(5)	2.249 (12)	Co-N(3)	2.017 (4)

(b) Angles, Deg

Atoms	Angle	Atoms	Angle
F(1)-Sb-F(2)	82.5 (5)	F(3)-Sb-F(4)	80.9 (3)
F(2)-Sb-F(3)	84.8 (4)	F(4)-Sb-F(5′)	78.3 (5)
F(3)-Sb-F(5)	82.6 (6)	F(4)-Sb-F(5)	75.6 (6)
F(3)-Sb-F(5′)	120.1 (6)	F(1)-Sb-F(5)	102.8 (7)
F(3)-Sb-F(4)	80.9 (3)	F(1)-Sb-F(5′)	66.3 (7)
F(2)-Sb-F(5)	159.1 (6)	N(1)-Co-N(2)	89.9 (4)
F(2)-Sb-F(5′)	147.3 (6)	N(1)-Co-N(3)	89.1 (4)
F(1)-Sb-F(4)	78.2 (4)	N(2)-Co-N(3)	89.3 (4)
F(2)-Sb-F(4)	86.0 (3)	Sb-F(5)-Sb′	142.1 (8)

Table IV. Equations of Atomic Planes and Distances (Å) of Atoms from These Planes[a]

(a) Plane[b] through F(1), F(2), F(3), F(5)

$-0.171X + 0.958Y - 0.231Z - 3.561 = 0$

Sb	−0.37	F(2)	−0.03	F(5)	−0.02
F(1)	0.02	F(3)	0.03		

(b) Plane through F(1), F(2), F(3), F(5′)

$-0.169X + 0.952Y - 0.255Z - 3.464 = 0$

Sb	−0.35	F(2)	−0.05	F(5′)	−0.04
F(1)	0.05	F(3)	0.03		

[a] The coordinate system used for the least-squares planes: $X = xa + zc \cos \beta$; $Y = by$; $Z = zV/ab$. [b] The atoms defining the planes were assigned unit weights.

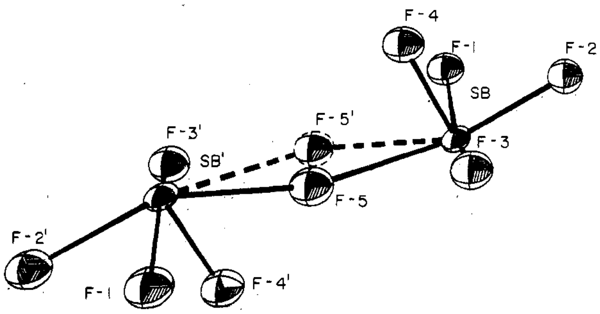

Figure 1. $Sb_2F_9^{3-}$ anion in $Co(NH_3)_6Sb_2F_9$.

as derived from the inverse matrix of the final least-squares cycle.[18] A table of observed and calculated structure factors is available.[19]

Discussion of the Structure

The unit cell consists of $Co(NH_3)_6^{3+}$ cations and $Sb_2F_9^{3-}$ anions. In the cation, the coordination of the NH_3 ligands around the cobalt atom is approximately octahedral and the distances and angles, presented in Table III, agree quite well with those found for the same cation in $Co(NH_3)_6SbCl_6$.[20]

The $Sb_2F_9^{3-}$ anion is shown in Figure 1 and could be described as two square pyramids sharing a corner. A stereoview of the structure is illustrated in Figure 2, produced by Johnson's thermal ellipsoid plot program.[21] In the pyramid, the antimony atom is displaced below the least-squares plane formed by the four basal fluorines by 0.37 Å when the plane is calculated using the shorter bridging fluorine atom and by 0.35 Å when the longer bridging fluorine is used (Table IV). The F(axial)-Sb-F(basal) angles range from 78.2 (4) to 86.0 (3) Å. It appears, therefore, that this antimony fluoride complex can be best described as consisting of two distorted octahedral units sharing a vertex, with a lone pair occupying the sixth position in each octahedron.

A second peculiar feature of this anion is the statistical disordering of the fluorine bridge and its asymmetric nature. One Sb-F distance is 2.249 (12) Å while the other is appreciably lengthened, 2.449 (4) Å. Although omitted from the figures for clarity, there is also pronounced anisotropic motion of the bridging fluorines with maximum components in the bridging plane. The angles to the neighboring basal fluorines are 82.6 (6) and 102.8 (7)° (66.3 (7) and 120.1 (6)° if the longer bridge is used). The Sb-F(5)-Sb angle was found to be 142.1 (8)°. It is worthwhile to note that in the compound $CsSb_2F_7$ a bent fluorine bridge is also found (125.3° angle).[22] In the absence of π bonding it would appear that fluorine bridges tend to be bent. However it is difficult to see why such an asymmetric arrangement is preferred over a symmetric one.

A third aspect of interest in this anion is the variation of the Sb-F nonbridging distances that is observed. In order of increasing bond length, the distance opposite the lone pair is the shortest at 1.948 (7) Å, while the Sb-F distance opposite the bridge is next at 2.015 (5) Å. The other two fluorines in the base, trans to one another, have essentially identical distances, 2.078 (8) and 2.080 (11) Å, respectively.

In this salt as in the bromide and chloride salts containing

---

[18] W. R. Busing, K. O. Martin, and H. A. Levy, "ORFFE, a Fortran Crystallographic Function and Error Program," Report ORNL-TM-306, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1964.

[19] A listing of observed and calculated structure factor amplitudes will appear following these pages in the microfilm edition of this volume of the journal. Single copies may be obtained from the Business Operations Office, Books and Journals Division, American Chemical Society, 1155 Sixteenth 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-73-515.

[20] D. R. Schroeder and R. A. Jacobson, *Inorg. Chem.*, 12, 210 (1973).

[21] C. K. Johnson, "ORTEP, a Fortran Thermal-Ellipsoid Plot Program for the Crystal Structure Illustrations," Report ORNL-3744, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1965.

[22] R. R. Ryan, S. H. Mastin, and A. C. Larson, *Inorg. Chem.*, 10, 2795 (1971).

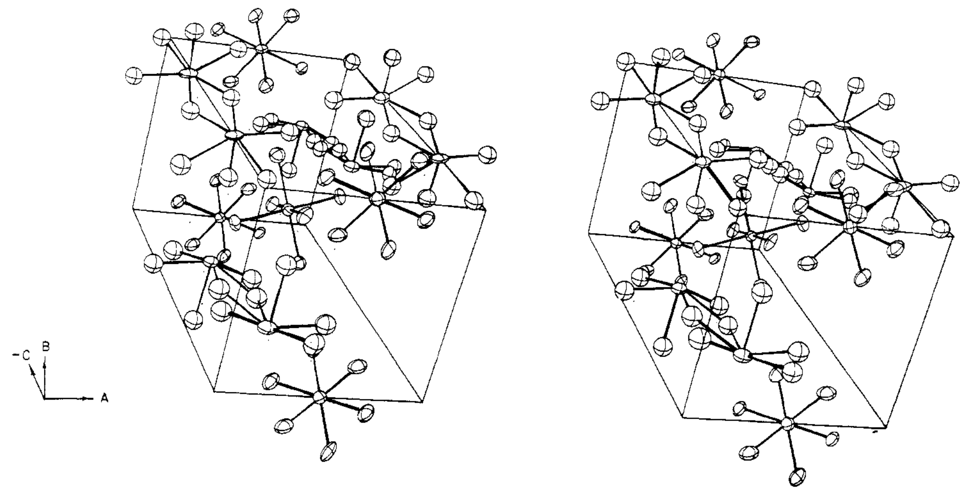

Figure 2. Stereogram of $Co(NH_3)_6Sb_2F_9$ with Co at (1/2, 0, 0).

the $Sb_2X_9^{3-}$ species, the antimony-bridging halogen distance is lengthened with respect to the terminal halogen distance. In the bromide and chloride anions this lengthening was found to be 0.37 and 0.36 Å, respectively. If the asymmetric Sb-F bridge distances are averaged in the present compound, the corresponding lengthening is 0.33 Å and is thus indicative of an effect which is essentially independent of the halogen present.

Although no hydrogen atoms were clearly indicated in the difference map, weak hydrogen bonding could well be present. The shorter nitrogen-fluorine distances range from 2.84 to 2.94 Å while the corresponding Co-N-F angles range from 97.5 to 120.2°.

Registry No. $Co(NH_3)_6Sb_2F_9$, 37871-78-6.

Contribution from the Department of Chemistry,
Brown University, Providence, Rhode Island 02912

Crystal and Molecular Structure of the Complex Tri-μ-methylmercapto-hexacarbonyldiiron(II) Tetrakis(*cis*-1,2-di(perfluoromethyl)ethylene-1,2-dithiolato)diiron, $[Fe_2(\mu\text{-}SCH_3)_3(CO)_6][Fe_2(S_2C_2(CF_3)_2)_4]$

ARTHUR J. SCHULTZ and RICHARD EISENBERG*

Received July 17, 1972

The crystal and molecular structure of the cation-anion complex $[Fe_2(\mu\text{-}SCH_3)_3(CO)_6][Fe_2(S_2C_2(CF_3)_2)_4]$ has been determined by single-crystal X-ray diffraction methods with data collected by the θ-2θ counter technique. The complex crystallized in space group $P\bar{1}$ of the triclinic system in a cell of dimensions a = 13.64 (1), b = 14.81 (1), c = 17.81 (1) Å; α = 124.27 (3), β = 127.19 (3), γ = 76.47 (3)°; and V = 2336 Å^3. There are two cation-anion pairs in the unit cell (ρ_{calcd} = 2.03, ρ_{exptl} = 2.02 (3) g/cm^3). The structure was solved by direct methods in the completely general space group $P1$ because of an early calculational error, and it has been refined by least-squares procedures to a conventional R factor of 0.058 for 4335 reflections above 2σ. The structure of the previously unreported cation consists of two faced-shared octahedra with methylmercapto groups occupying the bridging positions. The average Fe-S and Fe-C distances are 2.305 (2) and 1.80 (1) Å, respectively. The Fe· · ·Fe separation is 3.062 (4) Å indicating little if any direct metal-metal bonding, and, hence, the cation can be formulated as containing two discrete Fe(II) d^6 metal ions in octahedral coordination geometries. The anion $[Fe_2(S_2C_2(CF_3)_2)_4]^-$ is crystallographically required to be centrosymmetric, and it exhibits a structure characteristic of other iron and cobalt dithiolene dimers in which each metal atom has a square-pyramidal coordination geometry. Dimerization occurs through the formation of two Fe-S linkages of distance 2.311 (5) Å. The average Fe-S, C-S, and C-C bond lengths within the metal-dithiolene chelate rings are 2.190 (6), 1.721 (9) and 1.33 (1) Å, respectively. The Fe-Fe distance is 2.767 (4) Å. The anion is compared structurally with other dithiolene dimers in order to assess the effects of complex charge and the number of valence electrons on the formation of a metal-metal bond and related structural parameters.

Introduction

Everyone has at least one skeleton in his closet and in a sense, this is one of ours. During the last few years we, like many others, have been intrigued by the development and use of metal complexes as model systems for metalloenzymes and related metal-containing proteins,[1] even when these model systems are chemically far removed from the biochemical systems whose properties they are supposed to resemble. One particular area of interest in this regard has been the inorganic chemists' approach to the nonheme iron proteins and,

---

[1] See, for example, *Advan. Chem. Ser.*, No. 100 (1970).