THE CRYSTAL AND MOLECULAR STRUCTURE
OF THE CHLORO-BIS(PENTAMETHYLENEDITHIOCARBAMATE)
IRON(III)–CHLOROFORM (1 : 1) COMPLEX

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The crystal and molecular structure of $FeCl(S_2CN(CH_2)_5)_2CHCl_3$ were solved by the x-ray structural analysis method and refined to R 0·073 for 1996 observed independent reflections. The compound crystallizes in a space group of symmetry $P\bar{1}$ with the following unit cell dimensions: $a = 0·6384$ (5), $b = 1·475$ (1), $c = 1·1966$ (6) nm, $\alpha = 86·20$ (5), $\beta = 95·24$ (6), $\gamma = 97·45$ (6)°. The experimentally measured density of the crystals of the substance is 1·60 Mgm^{-3} and the value calculated for $Z = 2$ is 1·59 Mgm^{-3}. The basic skeleton structure is formed by the complex molecules forms closed cavities filled with chloroform molecules. The central Fe(III) ion is characterized by a quartet ground state ($S = 3/2$) and is pentacoordinated by a chlorine atom and 4 sulphur atoms in a tetragonal bipyramidal arrangement. The complex-chloroform interaction depends on formation of a double-branched hydrogen bond of the chloroform proton with two sulphur atoms in the coordination polyhedron.

It is known that the trigonally deformed octahedral complexes[1] $\left[Fe(S_2CNR_2)_3\right]$ exist in thermal equilibrium as a result of the relative populations of two equi-energy ground states of the Fe(III) ion characterized by spin quantum numbers of $S = 1/2$ and 5/2. In contrast, structural results for complexes of the type $\left[Fe(III)X(S_2CNR_2)_2\right]$ $(X = Cl)$ indicated[2,3] that the Fe(III) ion exists in an unusual ground state with transient spin $S = 3/2$, reflected in unusual coordination geometry and character of the bonds in the coordination sphere.

This work was carried out in order to study in detail the stereochemistry of a further complex of this type $(X = Cl, R_2 = (CH_2)_5)$ and to determine the type of complex––chloroform interaction.

EXPERIMENTAL

The $[FeClS_2CN(CH_2)_5]_2$ complex was provided by Dr J. Kúdela[4]. The magnetic susceptibility of the powder sample was measured by the Gouy method on a commercial apparatus (Newport Instruments Ltd.) at room temperature, $T = 293·1$ K. The measurement was carried out at three different field intensities, using the $HgCo(SCN)_4$ complex as a reference substance. The measured effective magnetic moment $\mu_{eff} = 3·89$ B.M. corresponds to the pure spin value for the quartet ground state ($S = 3/2$) of the Fe(III) ion, similarly as observed for the pentacoordinated $[FeCl[S_2CN(C_2H_5)_2]_2]$ and $[FeI(S_2CN(C_2H_5)_2)_2]$ complexes[2,3].

The dark brown plate single crystals suitable for x-ray analysis were prepared by slow evaporation of a saturated chloroform solution of the powder sample of $[FeCl(S_2CN(CH_2)_5)_2]$. It was later found that the growth of these crystals was made possible by the formation of the stable chloroform solvated complex.

TABLE I

Coordinates of the atoms in the crystal structure of $[FeCl(S_2CN(CH_2)_5)_2].CHCl_3$ (standard deviations given in brackets)

Atom	x/a	y/b	z/c
Fe	0·2524(2)	0·2506(1)	0·4590(1)
Cl	0·2692(4)	0·2587(1)	0·6497(2)
Ligand (1)			
S(1)	0·0633(4)	0·3858(1)	0·4051(2)
S(2)	0·5026(4)	0·3301(1)	0·4109(2)
C(1)	0·3018(4)	0·4165(6)	0·3816(8)
N(1)	0·3302(13)	0·4958(5)	0·3418(7)
C(11)	0·5342(13)	0·5166(6)	0·3149(7)
C(12)	0·5263(19)	0·5262(7)	0·1874(10)
C(13)	0·3363(19)	0·6004(8)	0·1514(9)
C(14)	0·1313(19)	0·5780(8)	0·1855(10)
C(15)	0·1532(16)	0·5665(7)	0·3098(9)
Ligand (2)			
S(3)	0·4084(4)	0·1093(1)	0·4074(2)
S(4)	−0·0299(4)	0·1659(2)	0·4044(2)
C(2)	0·1617(15)	0·0742(6)	0·3809(8)
N(2)	0·1231(13)	−0·0075(5)	0·3441(7)
C(21)	−0·0921(16)	−0·0299(7)	0·3154(9)
C(22)	−0·1186(17)	−0·0600(8)	0·1948(9)
C(23)	0·0578(19)	−0·1342(8)	0·1654(10)
C(24)	0·2774(18)	−0·1139(7)	0·2011(10)
C(25)	0·2894(17)	−0·0828(6)	0·3256(9) .
Chloroform			
C	0·4312(32)	0·2216(11)	0·1357(15)
H^a	0·3779	0·2291	0·2244
Cl(1)	0·4040(12)	0·1097(4)	0·0796(5)
Cl(2)	0·2590(18)	0·2997(5)	0·0612(7)
Cl(3)	0·6884(11)	0·2434(5)	0·1061(6)

^{a} The position of the hydrogen atom in chloroform is calculated.

A plate crystal with dimensions of $0.4 \times 0.45 \times 0.1 \text{ mm}^3$ was chosen for determination of the basic data and measurement of the intensities. Preliminary oscillation and Weissenberg patterns indicated the presence of a triclinic crystal system. The lattice parameters were refined on a Syntex $P2_1$ diffractometer by the least squares method on the basis of 8 centred reflections:

TABLE II

Anisotropic temperature coefficients ($\times 10^4$) from the expression $T = \exp -(B11 \, h^2 + \ldots + B12 \, hk + \ldots)$

Atom	B11	B22	B33	B12	B13	B23
Fe	171(4)	32(1)	65(1)	− 22(2)	39(1)	−22(1)
Cl	273(8)	43(1)	70(2)	− 37(5)	63(7)	3(3)
			Ligand (1)			
S(1)	177(8)	47(1)	119(3)	− 12(5)	67(8)	23(3)
S(2)	176(8)	37(1)	95(2)	− 16(5)	39(7)	6(3)
C(1)	189(30)	37(5)	75(10)	− 22(19)	99(26)	− 2(10)
C(1)	280(27)	33(4)	99(8)	− 45(17)	58(23)	18(9)
C(11)	80(27)	65(6)	97(10)	− 65(20)	65(26)	30(12)
C(12)	390(39)	66(7)	93(11)	−117(25)	0(31)	7(13)
C(13)	341(39)	66(8)	100(11)	− 75(26)	− 34(32)	20(14)
C(14)	361(38)	72(7)	84(11)	−106(25)	− 21(31)	11(13)
C(15)	235(33)	39(5)	98(10)	13(20)	− 12(28)	51(12)
			Ligand (2)			
S(3)	160(8)	43(1)	84(2)	−30(5)	18(7)	− 8(3)
S(4)	168(8)	42(1)	96(3)	−23(5)	47(7)	− 1(3)
C(2)	190(29)	33(5)	96(10)	3(19)	21(26)	−10(11)
N(2)	223(26)	45(4)	108(8)	−69(18)	43(23)	−18(10)
C(21)	82(28)	75(7)	93(11)	−59(22)	35(27)	7(13)
C(22)	159(32)	73(7)	75(10)	− 8(23)	−26(28)	− 7(13)
C(23)	281(31)	91(7)	107(12)	−67(26)	−81(32)	−34(24)
C(24)	134(31)	75(7)	113(11)	−65(23)	34(29)	12(13)
C(25)	196(31)	49(6)	79(10)	25(21)	46(27)	−60(11)
			Chloroform			
C	959(83)	96(13)	119(20)	− 24(46)	− 81(57)	75(22)
Cl(1)	1 430(34)	110(4)	180(7)	−216(17)	−137(33)	20(7)
Cl(2)	829(27)	138(5)	400(9)	−109(17)	−242(23)	− 1(10)
Cl(3)	715(28)	180(6)	150(6)	− 75(17)	134(19)	10(8)

$a = 0\cdot6384$ (5), $b = 1\cdot4751$ (13), $c = 1\cdot1966$ (6) nm, $\alpha = 86\cdot20$ (5), $\beta = 95\cdot24$ (6), $\gamma = 97\cdot46(6)^\circ$ $V = 1\cdot113$ nm^3. The crystal density $D_0 = 1\cdot60$ Mgm^{-3} was measured by the flotation method in a bromoform-ethanol mixture. The measured density is in good agreement with the assumed presence of two formula units per unit cell, $Z = 2$, where $Z = [\text{FeCl}(\text{S}_2\text{CN}(\text{CH}_2)_5)_2]\cdot\text{CHCl}_3$ (molecular weight 53·1 2); $D\cdot = 1\cdot59$ Mgm^{-3}.
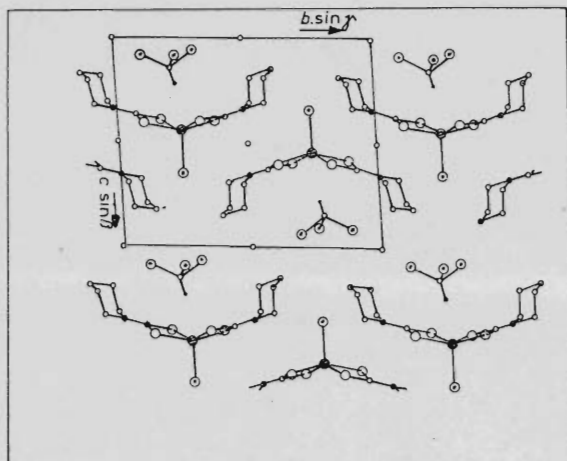

FIG. 1

Projection of a unit cell of the FeCl(S$_2$CN(CH$_2$)$_5$)$_2$.CHCl$_3$ structure onto the (100) plane. Fe ●, S ○, Cl ⊙, C ○, N ●, H·
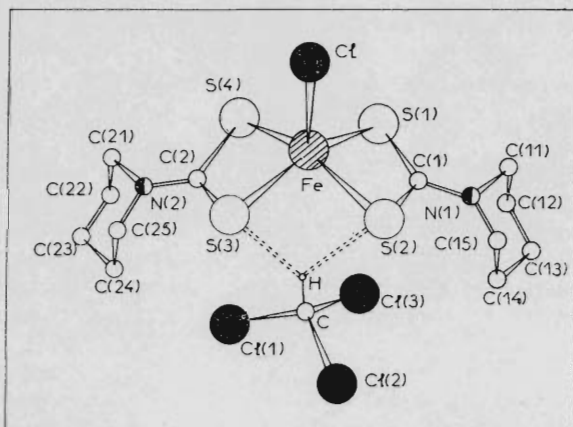
FIG. 2

Schematic representation of the molecular structure of the solvated [FeCl(S$_2$CN(CH$_2$)$_5$)$_2$]. .CHCl$_3$ complex with designation of the atoms

The integral intensity was measured on a Syntex P2$_1$ diffractometer using graphite monochromated MoK$_\alpha$ radiation ($\lambda = 0.071069$ nm) and the $\theta - 2\theta$ scanning technique in the interval $0 <$ $< 2\theta \leqq 55°$. The background was measured at the end of each scan for a time period equal to one half of the reflection scanning time. Of the total of 5601 recorded independent reflections, only 2131 with $I \geqq 1.95\sigma(I)$ were classified as observed (38.0% observed reflections in the given diffraction range). All the intensities were corrected for the Lorentz and polarization factors and converted to the absolute scale by the Wilson method ($B = 0.0236$ nm^2). No correction was made for absorption ($\sigma = 1.79$ mm^{-1}). For final structure solution, 1 996 observed reflections which were not accompanied by an intensity measurement error were used.

The position of the Fe atom was found from the three-dimensional Patterson synthesis and the Fourier synthesis of the electron density phased from the contribution of this atom indicated the positions of 5 atoms in the coordination sphere. A space group of $P\bar{1}$ was assumed here and was confirmed at the end of the structural refinement. The positions of the other nonhydrogen atoms were found by further gradual application of Fourier synthesis of the electron density. Differential Fourier synthesis of the residual electron density confirmed the presence of a chloroform molecule. The structure was refined in two cycles by the least squares method using a full matrix, isotropic thermal parameters for all the atoms and individual weights for all the 1 996 observed reflections, to give $R = 0.14$, $R = \Sigma||F_0| - |F_c||/$ $/\Sigma|F_0|$. In a further 6 refinement cycles, anisotropic thermal vibrations of all the atoms were assumed and, together with the positional parameters, were refined by the least squares method in a 9×9 block diagonal approximation to a final value of $R = 0.073$. The correct choice of the weights of the individual reflections in the minimized quantity $\Sigma w(|F_0) - |F_c|)^2$ according to the Hughes scheme ($\sqrt{w} = |F_0|/a$, where $|F_0| \leqq a$, or $\sqrt{w} = a/|F_0|$, where $|F_0| > a$ and $a = 45$, was confirmed by the approximately symmetrical distribution of the values of this quantity throughout the sin θ/λ and $|F_0|$ intervals. In the final cycle, the shifts of all the parameters were less than 0.1σ. Differential Fourier synthesis of the residual electron density did not indicate significant fluctuation with the highest maximum 900 enm^{-3}. The refined positional and anisotropic thermal parameters are given in Tables I and II. The composition of the unit cell projected onto the (100) plane is schematically depicted in Fig. 1.

DISCUSSION

The designation of the atoms and molecular structure of $[\text{FeCl}(S_2CN(CH_2)_5)_2]$. .CHCl$_3$ is depicted schematically in Fig. 2 and the intramolecular bond lengths and angles are given in Table III.

The crystal structure consists of monomeric pentacoordinated $[\text{FeCl}(S_2CN.$.$(CH_2)_5)_2]$ complex molecules and chloroform molecules. The coordination of the central Fe atom is tetragonalpyramidal with a chlorine atom in the axial position of the pyramid. Four sulphur donor atoms of the dithiocarbamate ligand form the planar base of the pyramid (deviation of the sulphur atoms from this plane constructed by the least squares method is ± 0.008 nm) and the Fe–Cl vector is almost perpendicular to the plane of the base. The Fe atom, however, does not lie in the plane of the base but deviates by 0.062 nm towards the apex of the pyramid, similarly as in the analogous $[\text{FeCl}(S_2CN(C_2H_5)_2)_2]$ $[\text{FeI}(S_2CN(C_2H_5)_2)_2]$ complexes[2,3] (Table IV) and in other known monomeric complexes of transition metals with pyra-

TABLE III

Intramolecular bonding distances (nm) and angles (deg) in the $FeCl(S_2CN(CH_2)_5)_2.CHCl_3$ complex and their standard deviations

Distances

Fe—Cl	0·2268(3)	N(1)—C(11)	0·144(1)
Fe—S(1)	0·2268(3)	N(1)—C(15)	0·147(1)
Fe—S(2)	0·2289(3)	C(11)—C(12)	0·154(1)
Fe—S(3)	0·2284(3)	C(12)—C(13)	0·157(1)
Fe—S(4)	0·2294(3)	C(13)—C(14)	0·149(1)
S(1)—C(1)	0·1696(9)	C(14)—C(15)	0·150(1)
S(2)—C(1)	0·1715(9)	N(2)—C(21)	0·146(1)
S(3)—C(2)	0·1717(9)	N(2)—C(25)	0·145(1)
S(4)—C(2)	0·1728(9)	C(21)—C(22)	0·148(1)
C(1)—N(1)	0·133(1)	C(22)—C(23)	0·151(1)
C(2)—N(2)	0·130(1)	C(23)—C(24)	0·154(1)
		C(24)—C(25)	0·153(1)

Chloroform

C—Cl(1)   0·176(1)   C—Cl(2)   0·172(2)   Cl(3)   0·178(2)

Bond Angles

S(1)—Fe—S(2)	76·2(1)	C(1)—N(1)—C(11)	123·1(8)
S(3)—Fe—S(4)	75·8(1)	C(1)—N(1)—C(15)	123·0(8)
Cl—Fe—S(1)	106·4(1)	C(11)—N(1)—C(15)	113·6(7)
Cl—Fe—S(2)	105·1(1)	C(11)—C(12)—C(13)	109·0(9)
Cl—Fe—S(3)	105·6(1)	C(12)—C(13)—C(14)	111·8(9)
Cl—Fe—S(4)	105·9(1)	C(13)—C(14)—C(15)	108·6(9)
S(1)—Fe—S(3)	148·0(1)	N(1)—C(11)—C(12)	108·5(8)
S(2)—Fe—S(4)	149·0(1)	N(1)—C(15)—C(14)	111·5(9)
S(1)—Fe—S(4)	95·3(1)	S(3)—C(2)—N(2)	125·8(7)
S(2)—Fe—S(3)	95·5(1)	S(4)—C(2)—N(2)	123·9(7)
Fe—S(1)—C(1)	86·9(3)	C(2)—N(2)—C(21)	122·3(8)
Fe—S(2)—C(1)	85·8(3)	C(2)—N(2)—C(25)	123·0(8)
Fe—S(3)—C(2)	87·7(3)	C(21)—N(2)—C(25)	114·7(8)
Fe—S(4)—C(2)	87·1(1)	C(21)—C(22)—C(23)	110·6(9)
S(1)—C(1)—S(2)	111·1(5)	C(22)—C(23)—C(24)	115·8(9)
S(3)—C(2)—S(4)	109·4(5)	C(23)—C(24)—C(25)	108·7(9)
S(1)—C(1)—N(1)	124·8(7)	N(2)—C(21)—C(22)	109·8(8)
S(2)—C(1)—N(1)	124·0(7)	N(2)—C(25)—C(24)	109·2(8)

Chloroform

Cl(1)—C—Cl(2)   110(1)   Cl(1)—C—Cl(3)   106(1)   Cl(2)—C—Cl(3)   105(1)

midal coordination independent of the type of ligand[5-13]. In contrast to the trigonally deformed octahedral Fe(III) tris-dithiocarbamates, the decrease in the symmetry of the coordination sphere in these pyramidal complexes from D_3 to C_{2v} results in preference of the ground state $^4T_{1g}$, split in the C_{2v} symmetry field to orbital singlets $^4A_2$, $^4B_1$ and $^4B_2$, so that a ground state with transient spin $S = 3/2$ (ref.[14]) is formed. Thus the magnetic moment of these complexes should correspond to the pure spin value $15^{1/2}$ B.M., measured for all three known complexes of this type independent of the nature of the halogen and the substituent on the dithiocarbamate ligand (Table IV).

TABLE IV

Some structural and magnetic properties of pyramidal Fe(III) dithiocarbamates

Complex	Fe$-$S^a, nm	Fe$-$X^b, nm	d^c, nm	μ^d, B.M.
[FeCl[S$_2$CN(ethyl)$_2$]$_2$]	0·230	0·226	0·063	3·9
[Fe[S$_2$CN(ethyl)$_2$)$_2$]	0·228	0·259	0·061	3·9
[FeCl(S$_2$CN(CH$_2$)$_5$)$_2$]	0·228	0·227	0·062	3·89

^{a} Average value of the Fe–S bond length; ^{b} X = Cl, I; ^{c} distance of the Fe atom from the plane of the 4 sulphur atoms; ^{d} magnetic moment of the powder sample

TABLE V

Deviation of selected atoms from the plane constructed by the least squares method through the S$_2$CNC$_2$ and S$_2$CN ligand fragments

Fragment	Deviation of the atom, 10^4 nm						
	Fe	S(1,3)	S(2,4)	C(i)	N(i)	C(i1)	C(i5)
S$_2$CNC$_2$							
Ligand (1), $i = 1$	68	18	13	−27	−42	22	18
Ligand (2), $i = 2$	−29	36	−17	−23	−14	39	−21
S$_2$CN							
Ligand (1), $i = 1$	85	3	−4	3	−2	38	11
Ligand (2), $i = 2$	−44	6	2	−4	−4	45	−19

The Fe–S bonds are significantly shorter (average value 0·228 nm) than the same bonds both in high-spin and low-spin Fe(III)tris-dithiocarbamates[15]. It is known that dithiocarbamate ligands are characterized by their strong σ-donor properties[16,17], so that the metal–sulphur bonds have significant covalent character. The decrease in the electron density on the sulphur atoms as a result of the covalent metal $\leftarrow$ sulphur electron transfer compensates the ability of the π-system to transfer charge from the nitrogen atom to the sulphur donor atoms, so that resonance structure *Ic* makes a large contribution to the overall electron structure of the ligand:
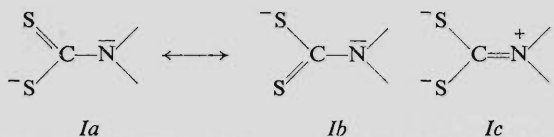

Ia   *Ib*   *Ic*

TABLE VI

Intermolecular interactions $<0·40$ nm observed in the $[FeCl(S_2CN(CH_2)_5)_2].CHCl_3$ crystal structure

Complex–complex interactions

Cl...C(11)[a]	0·368	Cl...C(24)[c]	0·375
Cl...C(12)[a]	0·392	Cl...C(25)[c]	0·358
Cl...C(13)[a]	0·399	S(1)...C(11)[e]	0·377
Cl...C(14)[d]	0·386	S(1)...C(15)[d]	0·378
Cl...C(15)[d]	0·352	S(2)...C(11)[a]	0·386
Cl...C(21)[b]	0·376	S(3)...C(21)[f]	0·377
Cl...C(22)[b]	0·384	S(3)...C(25)[c]	0·359
Cl...C(23)[b]	0·387	S(4)...C(21)[b]	0·396

Chloroform–complex interactions

H...S(2)	0·272
H...S(3)	0·288

Cl(1)...C(24)	0·389	Cl(1)...C(23)[g]	0·396
Cl(1)...C(22)[f]	0·389	Cl(3)...C(13)[h]	0·394
Cl(1)...C(22)[g]	0·368	Cl(3)...C(23)[i]	0·391

Symmetrical transformations: [a] $1 - x, 1 - y, 1 - z$; [b] $-x, -y, 1 - z$; [c] $1 - x, -y, 1 - z$; [d] $-x, 1 - y, 1 - z$; [e] $x - 1, y, z$; [f] $1 + x, y, z$; [g] $-x, -y, -z$; [h] $1 - x, 1 - y, -z$; [i] $1 - x, -y, -z$.

Consequently, shortening of the Fe–S bond cannot be explained by a change in the spin state of the Fe(III) ion, but probably by the fact that the pyramidal coordination geometry provides better conditions for σ-overlapping than strongly trigonally deformed octahedral geometry of the Fe(III) tris complexes. The possible alternative formation of a Fe–S π-bond must be excluded because the highest occupied π-MO of the dithiocarbamate ligand does not have suitable energy and symmetry for overlap with the d orbitals of the metal ion[14,18]. The fact that the π-electron density remains localized on the sulphur atoms as a result of conjugation is also documented by the fact that the observed coordination geometry of the studied complex (similarly as for the other two in Table IV) can be reproduced very precisely from minimization of the electrostatic potential energy assuming the presence of a fractional positive charge on the central Fe atom and fractional negative charges on the atoms of the coordinating ligands[19].

For this type of bidentate coordination, both dithiocarbamate ligands have characteristic stereochemistry with a high degree of planarity of the S_2CNC_2 ligand fragment (Table V). None of the bonds exhibits anomalous geometry and the piperidine rings are in the chair conformation.

Interatomic interactions < 0.40 nm are listed in Table VI. Although the positions of the H-atoms were not found, it is probable that the S$\cdots$C interactions in Table VI should be considered as S$\cdots$H—C type hydrogen bonds, observed in many crystal structures of dithiocarbamate complexes[20]. In this connection, the Cl$\cdots$H—C interactions (Table VI) have a dominant effect on the position of the molecules in the crystal, probably as a result of the higher electronegativity of the Cl atom. The formation of bifurcated hydrogen bonds of the same type as in the $[Cr(S_2CN(CH_2)_5)_3]$. .2 $CHCl_3$ structure[21], i.e. formed by interaction of the chloroform hydrogen atom with the occupied p_π orbitals of two sulphur atoms from two ligands (Fig. 2) has a decisive effect on the formation of the chloroform solvated complex. As a result of the different position of the molecules in the structure, the $[FeCl(S_2CN(CH_2)_5)_2]$. .$CHCl_3$ solvate is much more stable than $[Cr(S_2CN(CH_2)_5)_3]$.2 $CHCl_3$ and does not decompose at normal temperatures.

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