Crystal and Molecular Structure of an Octahedral Iron(III) Complex with a Sulphur-containing Schiff-base Ligand: Bis(2-aminoethylthiosalicylideneiminato)iron(III) Chloride

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The crystal and molecular structure of the title compound (1) has been determined from diffractometer data by three-dimensional Patterson and Fourier methods and refined by block-diagonal least-squares to R 0·077 for 1302 independent non-zero reflections. Crystals are monoclinic, space group $P2_1/c$, with a = 8·75(1), b = 21·92(2), c = 11·07(1) Å, β = 114·0(2)°, and Z = 4. The iron atom is octahedrally co-ordinated by two terdentate Schiff-base ligands, with Fe–S(1) 2·21(1), Fe–N(1)(amine) 2·08(3), Fe–N(2)(imine) 1·93(2) Å; Fe · · · Cl⁻ is 4·51(1) Å.

CONSIDERABLE attention has recently been given to syntheses of iron(III) chelates of sulphur-containing compounds,[1-4] owing to their relationship to the iron-sulphur centre in biologically active iron-containing proteins. In relation to an extensive study of iron(III) chelates of sulphur-Schiff bases[5] we now report the X-ray crystal structure of the title compound which provides the first structural data for such a molecule.

EXPERIMENTAL

Attempts to synthesize NN'-ethylenebis(thiosalicylideneiminato)iron(III) chloride, [Fe(Ssalen)]Cl {the sulphur analogue of [Fe(salen)]Cl}, by the reaction of iron(III) trichloride with a solution containing ethylenediamine and o-mercaptobenzaldehyde gave deep red air-stable crystals. Though the analytical figures were not in accord with the [Fe(Ssalen)]Cl formulation ($C_{16}H_{14}N_2S_2FeCl$), poor solubility prevented measurement of molecular weight and determination of n.m.r. spectra, which could potentially distinguish between the one quadridentate {[Fe(Ssalen)(en)]Cl} or two terdentate ligand alternatives $\{[\mathrm{Fe}(\mathrm{Ssal{:}N}\cdot[\mathrm{CH}_2]_2\cdot\mathrm{NH}_2)_2]\mathrm{Cl}\}$, bis(2-aminoethylthiosalicylideneiminato)iron(III) chloride ($C_{18}H_{22}N_4S_2FeCl$). Analytical data were in agreement with this empirical formula. Preliminary magnetic measurements indicated that the compound contained low-spin iron(III) ($\mu_{\text{eff.}}$ 2·00 B.M.).[5]

Rotation, oscillation, and Weissenberg photographs taken with Cu-K_α radiation showed that the crystals were monoclinic, space group $P2_1/c$. Unit-cell parameters were determined by use of a standard Philips PW 1100 X-ray-diffractometer computer program. Crystals of (1) were dark red and acicular, with a diamond cross-section.

Crystal Data.—$C_{18}H_{22}ClFeN_4S_2$, M = 449·9, Monoclinic, a = 8·75(1), b = 21·92(2), c = 11·07(1) Å, β = 114·0(2)°, U = 1941 Å^3, D_m = 1·53(1) (by flotation), Z = 4, D_c = 1·54 g cm^{-3}, $F(000)$ = 932. Cu-K_α radiation, λ = 1·5418 Å: $\mu(\text{Cu-}K_\alpha)$ = 96·1 cm^{-1}. Space group $P2_1/c$ (C^5_{2h}, No. 14) from systematic absences: $0k0$ k odd, $h0l$ l odd.

Intensity Measurements.—Intensity data were measured on a Philips PW 1100 automatic X-ray diffractometer, with graphite-monochromated Cu-K_α radiation (a molybdenum tube was not available at the time), from a crystal of dimensions 0·04 × 0·10 × 0·08 mm. 3155 Unique reflections with $6{\cdot}0 < 2\theta(\text{Cu-}K_\alpha) \leqslant 130{\cdot}0°$ were collected, of which 1302 having $I > 3\sigma(I)$ were used in the subsequent refinement. Three, nearly orthogonal, standard reflections, monitored every 2 h throughout data collection, showed a 3·5% drop in intensity, which was allowed for in the calculation of $|F_o|$.

Data were collected with a θ—2θ scan and a symmetric-scan range of ±1·30° in 2θ from the calculated Bragg scattering angle at a scan rate of 0·02° s^{-1}. No reflection was sufficiently strong to require the insertion of an attenuation filter.

Intensity data were processed with a modified program of Hornstra and Stubbe.[6] Background-corrected intensities were assigned standard deviations according to: $\sigma(I)$ =

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[1] B. A. Averill, T Herskovitz, R. H. Holm, and James A. Ibers, *J. Amer. Chem. Soc.*, 1973, **95**, 3523.

[2] L. Que, jun., M. A. Bobrik, James A. Ibers, and R. H. Holm, *J. Amer. Chem. Soc.*, 1974, **96**, 4168, and refs. therein.

[3] R. Mason and J. A. Zubieta, *Angew. Chem. Internat. Edn.*, 1973, **12**, 390.

[4] Stephen J. Lippard, *Accounts Chem. Res.*, 1973, **6**, 280.

[5] K. S. Murray, R. M. Slade, and B. O. West, unpublished results.

[6] J. Hornstra and B. Stubbe, PW 1100 Data Processing Program, Philips Research Laboratories, Eindhoven, Holland.

$[CT + (t_c/t_b)^2(B_1 + B_2) + (pI)^2]^{\frac{1}{2}}$, where CT is the total integrated peak count obtained in a scan time t_c, B_1 and B_2 are background counts each obtained in time $\frac{1}{2}t_b$, and $I = CT - (t_c/t_b)(B_1 + B_2)$. The value of p was selected as 0·04 and introduces a 4% uncertainty to account for ' machine errors '. The values of I and $\sigma(I)$ were then corrected for Lorentz and polarization effects. Neither extinction nor absorption corrections were applied.

Structure Solution and Refinement.—In the least-squares calculations, the function $\Sigma w(|F_o| - |F_c|)^2$ was minimized, where w is the weight, $1/\sigma^2(F)$, of each individual reflection.

The position of the iron atom was derived from a three-dimensional Patterson map. Two structure-factor calculations, each followed by a difference-Fourier synthesis, led to the positions of all non-hydrogen atoms. Four cycles of full-matrix least-squares refinement, varying alternately the positional parameters, then isotropic thermal parameters of all atoms, gave R 0·092 and R' 0·095, where $R' = [\Sigma w(|F_o| - |F_o|)^2/\Sigma w|F_o|^2]^{\frac{1}{2}}$.

To enable simultaneous refinement of all parameters with the limited computer store available, final refinement was by the block-diagonal least-squares method. Six cycles of refinement, with anisotropic thermal parameters for iron and chlorine, gave R 0·085 and R' 0·091. Idealized positions of all hydrogen atoms (Table 1) were then calculated assuming the appropriate trigonal or tetrahedral geometry and C–H 0·95 and N–H 0·87 Å.[7] A final refinement cycle, with the hydrogen atoms included as fixed contributions and each given a temperature factor 1·0 Å^2 greater than B of the atom to which they are bonded, gave R 0·077 and R' 0·075. A final difference synthesis had no major characteristics greater than 0·4 eÅ^{-3}. Final observed and calculated structure factors are given in Supplementary Publication No. SUP 21288 (16 pp., 1 microfiche).*

TABLE 1

Idealized hydrogen-atom positions (×10^3), numbered according to the carbon atom to which they are attached, except where indicated

	x/a	y/b	z/c
H(1A) *	120	448	422
H(1B) *	−20	418	335
H(3)	341	477	−115
H(4)	470	413	−213
H(5)	573	318	−127
H(6)	562	285	71
H(7)	280	503	58
H(8A)	251	526	315
H(8B)	146	542	168
H(9A)	−30	520	281
H(9B)	−66	476	163
H(3A) †	395	440	495
H(3B) †	510	423	440
H(12)	62	148	290
H(13)	−103	105	85
H(14)	−218	165	−105
H(15)	−177	272	−88
H(16)	168	230	426
H(17A)	254	352	566
H(17B)	337	288	581
H(18A)	541	367	633
H(18B)	540	328	516

* Attached to N(1). † Attached to N(3).

Final parameters for all non-hydrogen atoms are listed in Table 2 together with their estimated standard deviations, which were derived from the inverse least-squares matrix. Atomic scattering factors used were taken from ref. 8.

TABLE 2

Atomic positional (×10^4) and isotropic thermal parameters (×10)

	x/a	y/b	z/c	B/Å^2
Fe	2255(5)	3825(2)	2812(4)	*
Cl	7094(9)	4860(3)	3945(7)	*
S(1)	4061(8)	3294(3)	2308(7)	27(1)
N(1)	687(25)	4353(9)	3389(21)	33(5)
N(2)	2403(21)	4557(9)	1903(19)	23(4)
C(1)	4267(29)	3600(11)	932(25)	27(6)
C(2)	3667(29)	4186(10)	416(25)	24(5)
C(3)	3824(31)	4377(12)	−765(27)	32(6)
C(4)	4584(33)	4006(12)	−1345(28)	41(7)
C(5)	5213(32)	3447(12)	−833(28)	39(7)
C(6)	5103(32)	3249(12)	324(27)	36(6)
C(7)	2910(31)	4625(11)	964(27)	32(6)
C(8)	1727(30)	5094(11)	2311(26)	32(6)
C(9)	206(33)	4886(12)	2485(28)	35(6)
S(2)	83(9)	3532(3)	1019(7)	32(2)
N(3)	4231(23)	4097(9)	4557(21)	28(5)
N(4)	2148(24)	3132(9)	3882(21)	30(5)
C(10)	−176(29)	2736(11)	1074(24)	26(5)
C(11)	493(28)	2385(12)	2209(25)	30(6)
C(12)	162(33)	1743(12)	2120(27)	37(6)
C(13)	−821(31)	1496(12)	927(27)	36(6)
C(14)	−1480(33)	1839(13)	−206(29)	39(7)
C(15)	−1194(28)	2468(12)	−108(26)	29(6)
C(16)	1497(28)	2590(12)	3535(24)	28(6)
C(17)	3129(30)	3251(12)	5304(26)	31(6)
C(18)	4719(30)	3558(11)	5432(26)	31(6)

* For Cl and Fe anisotropic temperature factors were employed of the form: $T = \exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$, with parameters (×10^4)

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Fe	129(7)	14(1)	65(4)	1(3)	42(5)	2(2)
Cl	162(14)	16(2)	78(9)	−5(4)	72(10)	−1(3)

Calculations.—The major calculations during refinement were carried out by use of modified versions of the full-matrix least-squares refinement program of Busing, Martin, and Levy,[9] the block-diagonal least-squares program of Shiono,[10] and the Fourier program of White.[11] All diagrams were drawn by use of the program ORTEP.[12] All calculations were performed on the Monash University CDC 3200 computer.

DESCRIPTION AND DISCUSSION OF STRUCTURE

The crystallographic asymmetric unit contains $[Fe(S\text{-}sal\colon N\cdot[CH_2]_2\cdot NH_2)_2]Cl$. The structural configuration of the cation is shown in Figure 1.

The terdentate nature of the Schiff-base ligands is confirmed, the meridional conformation being similar to

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* See Notice to Authors No. 7 in *J.C.S. Dalton*, 1974, Index issue.

[7] M. R. Churchill, *Inorg. Chem.*, 1973, **12**, 1213.

[8] ' International Tables for *X*-Ray Crystallography,' vol. III, Kynoch Press, Birmingham, 1962.

[9] W. R. Busing, K. O. Martin, and H. A. Levy, ORFLS, A Fortran Crystallographic Least-Squares Program, Oak Ridge National Laboratory, Tennessee, Report ORNL TM 305, 1962.

[10] R. Shiono, Department of Crystallography, University of Pittsburgh, U.S.A.

[11] J. C. B. White, Melbourne University Fourier Program, MUFR3. See J. S. Rollett in ' Computing Methods and the Phase Problem in *X*-Ray Crystal Analysis,' eds. R. Pepinsky, J. M. Robertson, and J. C. Speakman, Pergamon Press, Oxford, 1961, p. 87.

[12] C. K. Johnson, ORTEP, Fortran Ellipsoid Plot Program for Crystal Structure Illustrations, Oak Ridge National Laboratory, Tennessee, Report ORNL 3794, 1965.

that found in (2), bis(2-aminoethylsalicylideneiminato)-chromium(III) iodide $[Cr(sal{:}N{\cdot}[CH_2]_2{\cdot}NH_2)_2]I$,[13] in both cases only one mole of the aromatic aldehyde having condensed with the diamine.

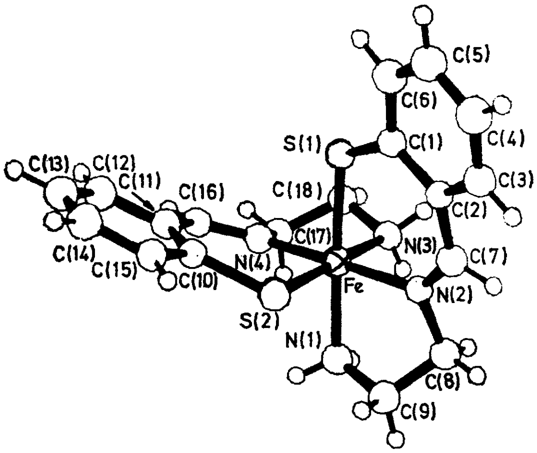

FIGURE 1 Molecular structure and atom-numbering system for the cation

The thiosalicylideneiminato moieties, ($-S-C_6H_4{\cdot}CH{\cdot}N-$), of (1), which in themselves are tolerably planar (Table 3), are inclined at an angle of 21° 45′ and 23° 24′, respectively, with the mean planes formed by Fe,S(1),N(2) and Fe,S(2),N(4), whereas in (2) the salicylideneiminato-moiety forms angles of only 4° 24′ and 9° 42′ with the corresponding Cr co-ordination planes. This twist of the Schiff-base results mainly from the lengthening of the Fe–S with respect to the Cr–O bond by *ca.* 0·3 Å, bond lengths and angles within the co-ordination ring of the ligand remaining constant. This twist also confers on the sulphur atom tetrahedral geometry, the mean Fe–S–C angle being 110·2°, whereas in (2) the mean Cr–O–C angle is 129·7°.

TABLE 3

Equations of least-squares planes in the form $AX + BY + CZ - D = 0$, where X, Y, Z are co-ordinates in Å referred to an orthogonal system of axes having X along the a axis, Y in the (a, b) plane, and Z along the c^* axis, and related to the fractional co-ordinates of Table 2 by the matrix equation:

$$\begin{bmatrix} 8\cdot751 & 0 & -4\cdot498 \\ 0 & 21\cdot920 & 0 \\ 0 & 0 & 10\cdot118 \end{bmatrix} \begin{bmatrix} x \\ y \\ z \end{bmatrix} = \begin{bmatrix} X \\ Y \\ Z \end{bmatrix}$$

Distances (Å) of relevent atoms from the planes are given in square brackets

Plane (1): S(1), C(1)—(7), N(2)

$$-0\cdot7104X - 0\cdot4054Y - 0\cdot5754Z + 6\cdot0922 = 0$$

[S(1) 0·03(1), C(1) −0·01(3), C(2) −0·02(3), C(3) 0·03(3), C(4) 0·04(3), C(5) 0·01(3), C(6) −0·05(3), C(7) −0·08(3), N(2) 0·05(2), N(1) 0·91(2), C(8) −0·12(3), C(9) 0·97(3)]

Plane (2): S(2), C(10)—(16), N(4)

$$0\cdot9678X - 0\cdot1641Y - 0\cdot1908Z + 1\cdot8175 = 0$$

[S(2) −0·02(1), C(10) 0·01(3), C(11) −0·01(3), C(12) 0·00(3), C(13) 0·00(3), C(14) 0·03(3), C(15) −0·01(3), C(16) −0·07(3), N(4) 0·07(2), N(3) 1·06(2), C(17) −0·03(3), C(18) 1·12(3)]

Plane (3): S(1), S(2), N(1), N(3)

$$0\cdot3759X + 0\cdot7881Y - 0\cdot4874Z - 5\cdot4765 = 0$$

[S(1) 0·02(1), S(2) −0·02(1), N(1) 0·02(2), N(2) −0·02(2), Fe 0·01(1)]

Plane (4): S(1), N(1), N(2), N(4)

$$-0\cdot4865X - 0\cdot3419Y - 0\cdot8040Z + 5\cdot5702 = 0$$

[S(1) 0·00(1), N(1) 0·00(2), N(2) 0·00(2), N(4) 0·00(2), Fe 0·07(1)]

Plane (5): S(2), N(2)—(4)

$$0\cdot8215X - 0\cdot4848Y - 0\cdot3000Z + 4\cdot3878 = 0$$

[S(2) 0·01(1), N(2) −0·01(2), N(3) 0·01(2), N(4) −0·01(2), Fe 0·05(1)]

The ethylenediamine bridge in each ligand takes on an asymmetric gauche conformation, similar to that in complexes of the quadridentate Schiff-base dianion, ethylenediaminebis(salicylideneiminate), C(9) and C(18) being 0·40(3) and 0·38(3) Å above, and C(8) and C(17) 0·30(3) and 0·34(3) Å below their associated planes, Fe, N(1), N(2) and Fe, N(3), N(4) (Table 3).

TABLE 4

Bond lengths (Å) and angles (°)

(*a*) Distances			
Fe–S(1)	2·21(1)	Fe–N(2)	1·93(2)
Fe–S(2)	2·21(1)	Fe–N(3)	2·09(2)
Fe–N(1)	2·08(2)	Fe–N(4)	1·95(2)
S(1)–C(1)	1·74(3)	S(2)–C(10)	1·76(2)
C(1)–C(2)	1·42(3)	C(10)–C(11)	1·39(3)
C(2)–C(3)	1·43(4)	C(11)–C(12)	1·43(4)
C(3)–C(4)	1·36(4)	C(12)–C(13)	1·36(4)
C(4)–C(5)	1·37(4)	C(13)–C(14)	1·37(4)
C(5)–C(6)	1·39(5)	C(14)–C(15)	1·40(4)
C(1)–C(6)	1·41(4)	C(15)–C(10)	1·38(3)
C(2)–C(7)	1·44(4)	C(11)–C(16)	1·44(3)
C(7)–N(2)	1·29(4)	N(4)–C(16)	1·30(3)
N(2)–C(8)	1·47(4)	N(4)–C(17)	1·48(3)
C(8)–C(9)	1·49(4)	C(17)–C(18)	1·50(4)
N(1)–C(9)	1·48(3)	N(3)–C(18)	1·48(3)
	Fe · · · Cl	4·51(1)	
(*b*) Angles			
S(1)–Fe–S(2)	93·6(3)	N(1)–Fe–N(3)	87·6(8)
S(1)–Fe–N(2)	95·7(6)	N(1)–Fe–N(4)	92·6(9)
S(1)–Fe–N(3)	88·6(6)	S(2)–Fe–N(2)	89·4(6)
S(1)–Fe–N(4)	87·6(7)	S(2)–Fe–N(4)	94·8(7)
N(1)–Fe–S(2)	90·2(6)	N(3)–Fe–N(2)	92·3(8)
N(1)–Fe–N(2)	83·7(8)	N(3)–Fe–N(4)	83·5(9)
Fe–S(1)–C(1)	111·0(9)	Fe–S(2)–C(10)	109·4(9)
S(1)–C(1)–C(2)	123·3(20)	S(2)–C(10)–C(11)	124·6(20)
C(1)–C(2)–C(7)	126·6(23)	C(10)–C(11)–C(16)	127·5(24)
C(2)–C(7)–N(2)	129·0(25)	C(11)–C(16)–N(4)	126·6(24)
Fe–N(2)–C(7)	129·3(18)	Fe–N(4)–C(16)	130·7(18)
Fe–N(2)–C(8)	112·7(15)	Fe–N(4)–C(17)	111·0(16)
N(2)–C(8)–C(9)	106·7(21)	N(4)–C(17)–C(18)	107·2(21)
C(8)–C(9)–N(1)	107·7(22)	C(17)–C(18)–N(3)	106·7(21)
Fe–N(1)–C(9)	105·8(16)	Fe–N(3)–C(18)	106·8(15)
C(1)–C(2)–C(3)	118·4(23)	C(10)–C(11)–C(12)	119·0(24)
C(2)–C(3)–C(4)	120·2(25)	C(11)–C(12)–C(13)	119·2(26)
C(3)–C(4)–C(5)	121·9(27)	C(12)–C(13)–C(14)	122·3(27)
C(4)–C(5)–C(6)	119·6(27)	C(13)–C(14)–C(15)	118·3(26)
C(5)–C(6)–C(1)	120·9(26)	C(14)–C(15)–C(10)	121·3(25)
C(6)–C(1)–C(2)	118·8(24)	C(15)–C(10)–C(11)	119·7(24)

The iron atom is octahedrally co-ordinated by the Schiff-base ligands, being only slightly displaced from the centre of the octahedron towards N(1) and N(4) [planes (3), (4), and (5), Table 3].

Pertinent ligand intramolecular bond distances and angles, listed in Table 4, are in close agreement with those

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[13] A. P. Gardner, B. M. Gatehouse, and J. C. B. White, *Acta Cryst.*, 1971, **B27**, 1505.

documented[14] for oxygen Schiff-base complexes. The S–C bond lengths [1·74(3) and 1·76(2) Å] are close to the single-bond distance (1·78 Å) calculated from the covalent radii of sulphur (1·04 Å) and sp^2 carbon (0·74 Å), and indicate minimal interaction between the π-system of the phenyl group and the *d* orbitals of the sulphur atom.

TABLE 5

Comparison of iron–nitrogen distances in iron–oxygen Schiff-base complexes

Schiff-base complex	Fe–N
$[\{Fe(salen)\}_2O],2py$ [a]	2·089(18), 2·112(18), 2·087(21), 2·059(20)
$[\{ClFe(salen)\}_2]$ [a]	2·098(9), 2·091(10)
$[ClFe(salen)],MeNO_2$ [a]	2·099(11), 2·064(10)
$[\{Fe(salen)\}_2O],CH_2Cl_2$ [b]	2·101(12), 2·116(10), 2·098(12), 2·105(10)
$[Cl(N\text{-}Pr^n\text{-}sal)_2Fe]$ [c]	2·09(2)
$[\{(N\text{-}Pr^n\text{-}sal)_2Fe\}_2O]$ [c]	2·14(2), 2·14(2), 2·13(2), 2·14(2)
$[\{N\text{-}p\text{-}ClC_6H_5\text{-}sal)_2Fe\}_2O]$ [c]	2·11(2), 2·15(2), 2·18(2), 2·18(2)
$[\{Fe(salen)\}_2O]$ [c]	2·12(1), 2·12(1), 2·11(1), 2·12(1)
(1) [d]	2·08(2), 1·93(2), 2·09(2), 1·95(2)

[a] Ref. 14. [b] P. Coggon, A. T. McPhail, F. E. Mabbs, and V. N. McLachlan, *J. Chem. Soc.* (*A*), 1971, 1014. [c] J. E. Davies and B. M. Gatehouse, *Acta Cryst.*, 1972, **B28**, 3641; 1973, **B29**, pp. 1934, 2651; *Cryst. Struct. Comm.*, 1972, **1**, 118. [d] Present work.

The lengths of the Fe–S bonds [2·21(1) Å, Table 4] are significantly shorter than the sum of the appropriate covalent radii (Fe 1·34 and S 1·04 Å), suggesting π interaction between the sulphur and iron *d* orbitals, similar to that found in the *S*-bonded sulphinates, $[(\pi\text{-}C_5H_5)Fe(CO)_2(SO_2{\cdot}C_6F_5)]$[15] and $[(\pi\text{-}C_5Me_5)Fe(CO)_2(SO_2{\cdot}CH_2{\cdot}CH{:}CH\text{-}Ph)]$,[15] and Fe–SR bond lengths in synthetic analogues of iron–sulphur proteins.[1,2]

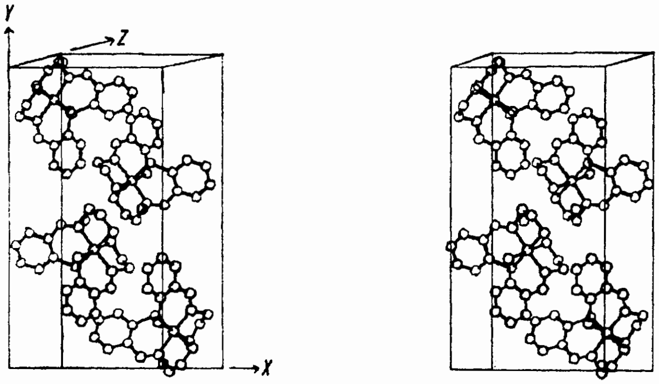

FIGURE 2 Packing of the molecule in the unit cell. Thermal ellipsoids have been assigned the same value and hydrogen atoms are omitted for clarity

Another notable feature of the iron–ligand bonding is the short Fe–N(imine) distances [1·93(2) and 1·95(2) Å] compared with Fe–N(amine) distances [2·08(3) and 2·09(2) Å] and Fe–N(imine) distances of *ca.* 2·1 Å in iron–oxygen Schiff-base complexes (Table 5). This shortening of the Fe–N(imine) distance is similar to that found in the low-spin complex $[Fe(terpy)_3][ClO_4]_3$,[16] in which the central nitrogen of the terpyridine ligand is bound more strongly to the iron.

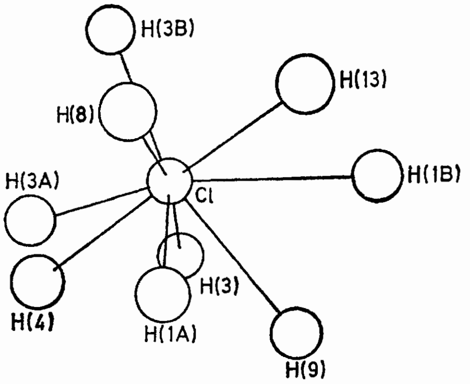

FIGURE 3 The hydrogen-atom environment for each chlorine atom, based on the predicted hydrogen-atom positions. Hydrogen-atom numbering is shown in Table 1

Packing within the unit cell is shown in Figure 2, and differs from that of complex (2),[13] which crystallizes in space group $P\bar{1}$. Strong hydrogen bonds are found between the chloride ion and hydrogen on N(1) and N(3) of several related molecules. Figure 3 shows the nature of the hydrogen bonding to chlorine and Table 6 the A···Cl and A–H···Cl distances and angles; three of the four hydrogen atoms attached to the amine nitrogens are, by the criteria of Hamilton and Ibers,[17] strongly hydrogen bonded to the chloride.

TABLE 6

Chlorine–hydrogen distances (Å) and angles (°)

A···Cl		A–H···Cl		A–H···Cl
$N(3^I)$···Cl	3·30	H(3A)···Cl	2·43	172·5
N(3)···Cl	3·30	H(3B)···Cl	2·43	164·7
N(1)···Cl	3·29	H(1A)···Cl	2·45	157·5
C(13)···Cl	3·70	H(13)···Cl	2·88	140·7
C(4)···Cl	3·63	H(4)···Cl	2·97	127·6
C(3)···Cl	3·68	H(3)···Cl	3·05	124·2
C(8)···Cl	3·84	H(8)···Cl	3·10	134·5
$N(1^I)$···Cl	3·62	H(1B)···Cl	3·13	116·2
C(9)···Cl	3·69	H(9A)···Cl	3·11	119·6

I $1 - x, 1 - y, 1 - z$

Apart from hydrogen-bonding distances there are no unusual intermolecular contacts, the shortest being 3·27 Å between C(15) and C(16).

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