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The Crystal and Molecular Structure of Chlorobis-(*N*-n-propylsalicylaldiminato)iron(III)

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$C_{20}H_{24}N_2O_2ClFe$ crystallizes in space group $C2/c$ with $a=16{\cdot}82$, $b=10{\cdot}90$, $c=11{\cdot}56$ Å, $\beta=104{\cdot}0°$; $Z=4$. The structure has been determined by Patterson and Fourier methods and refined to an R value of 0·116 using visual data. The structure consists of discrete monomeric units, each with symmetry 2. The iron atom is five-coordinate, intermediate between trigonal bipyramidal and square pyramidal. The bond distances Fe–Cl, Fe–O and Fe–N are respectively 2·23, 1·89 and 2·09 Å.

Introduction

The reaction of anhydrous iron(II) chloride and an appropriate salicylaldimine in benzene in the presence of at least two equivalents of triethylamine, yields complexes of the type Fe(*N*-R-salicylaldiminato)$_2$Cl. The preparation and properties of these complexes were first reported by van den Bergen, Murray, O'Connor, Rehak & West (1968). On the basis of mass spectral, solution molecular weight, and magnetic data these workers suggested a monomeric, and possibly trigonal bipyramidal configuration for these complexes. In contrast, the analogous *N,N'*-ethylenebis(salicylideneiminato)-iron(III) chloride is dimeric in the solid state, although in the presence of a large excess of solvent, the complex has been isolated as a monomer (Gerloch & Mabbs, 1967). The present crystallographic analysis was undertaken to ascertain the configuration of the title compound in the solid state. A brief account of this structure has been published elsewhere (Davies & Gatehouse, 1970).

Experimental

Unit cell and space group

The crystals of chloro bis-(*N*-n-propylsalicylaldiminato)iron(III) used in this study were kindly supplied by Dr K. S. Murray of the Chemistry Department, Monash University. The compound had been crystallized from a benzene/light petroleum mixture as dark red prisms of centrosymmetric cross section.

Weissenberg photographs showed systematic absences hkl for $h+k$ odd, and $h0l$ for l odd, characteristic of the monoclinic space groups $C2/c$, Cm and Cc. Subsequent solution of the structure has proved the space group to be $C2/c$. No test for the piezoelectric effect was carried out.

The lattice parameters as measured from $h0l$ and $hk0$ Weissenberg photographs taken with Ni-filtered Cu $K\alpha$ radiation ($\lambda=1{\cdot}5418$ Å) at 25 °C are $a=16{\cdot}82\pm0{\cdot}02$, $b=10{\cdot}90\pm0{\cdot}02$, $c=11{\cdot}56\pm0{\cdot}02$ Å, $\beta=104{\cdot}0\pm0{\cdot}2°$. An accurate experimental density determination was not possible as the compound dissolved readily in any liquid mixtures of the correct density. A value of $1{\cdot}4\pm0{\cdot}1$ g.cm^{-3} was obtained by flotation in a mixture of bromobenzene and chlorobenzene. This is consistent with four formula units in the cell ($D_{\rm calc}$ for $Z=4$ is 1·34 gm.cm^{-3}).

Collection and reduction of intensity data

Intensity data were collected from a prism-shaped crystal of approximate dimensions $0{\cdot}04\times0{\cdot}04\times0{\cdot}10$ mm using equi-inclination Weissenberg geometry and Mn-filtered Fe $K\alpha$ radiation ($\lambda=1{\cdot}937$ Å). To minimize absorption effects, the crystal was mounted so that the prism axis was parallel to the rotation axis. This axis is the a axis of the non-standard space group $I2/a$. 571 independent non-zero reflexions were visually estimated from the levels $0kl$–$10kl$ of $I2/a$. After correcting the intensities for Lorentz and polarization effects, the indices of the reflexions were transformed to those of the standard space group $C2/c$ using the following transformation matrix

$$\begin{pmatrix} 1 & 0 & 1 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{pmatrix}$$

All subsequent calculations were performed in space group $C2/c$.

Neither extinction nor absorption corrections were applied to these data. The linear absorption coefficient (μ) for Fe $K\alpha$ radiation is 45·4 cm^{-1}. For rotation about the prism axis, $\mu R=0{\cdot}2$ and the estimated maximum effect on the intensities caused by neglect of absorption is less than 2%. For a prismatic crystal, absorption corrections to particular intensities may be large for rotation about any axis other than the prism axis, even though the 'average' value of μR is small (about 0·4 here). For this reason, no attempt was made to cross-correlate the data and separate scale factors were carried for the 11 zones $0kl$ through $10kl$. ($I2/a$ indexing). The structure amplitudes were not scaled initially. The final scaling (Table 3) is based upon the calculated structure amplitudes.

Structure solution and refinement

The structure was solved by standard heavy-atom methods. In the least-squares calculations discussed here, the function $\sum w(|F_o|-|F_c|)^2$ was minimized where $|F_o|$ and $|F_c|$ are the observed and calculated structure amplitudes and w is the weight of each individual reflexion. The scattering factors were those tabulated by Ibers (1962). The major programs used during the refinement were modified versions of the full matrix least-squares program of Busing, Martin & Levy (1962) and the Fourier synthesis program of White (1965). All calculations were performed on the Monash University CDC 3200 computer.

Strong vector concentrations on the line $0,y,\frac{1}{2}$ and on the plane $x,0,z$ in the three-dimensional Patterson map indicated that the space group is $C2/c$. With only four formula units in the cell this implies that the molecule has point symmetry 2 ($\bar{1}$ point symmetry being impossible for the expected 5 coordinate iron complex). Interpretation of the Harker line $0,y,\frac{1}{2}$ led to the positions of the iron and chlorine atoms on the twofold axis at $0,y,\frac{1}{4}$. Two structure-factor calculations, each followed by a difference synthesis, led to the positions of all non-hydrogen atoms.

The refinement by least squares was based upon all of the observed reflexions each given unit weight, on separate scale factors for the individual zones and individual isotropic thermal parameters for all atoms. This refinement converged after several cycles and led to values of R_1 and R_2 of 0·116 and 0·129 respectively where

$$R_1=\sum||F_o|-|F_c||/\sum|F_o|$$
$$R_2=(\sum w(|F_o|-|F_c|)^2/\sum w|F_o|^2)^{1/2}$$

Anisotropic thermal parameters were not refined as these are very sensitive to systematic errors in the data, particularly when the data are collected about only one axis. No correction was applied for anomalous dispersion.

Although the data are not sufficiently accurate to resolve the hydrogen atoms, their positions were estimated to allow a better study of the molecular packing. The positions of the hydrogens bonded to atoms C(2), C(3), C(4), C(5), C(7), C(8) and C(9) were calculated assuming the appropriate trigonal or tetrahedral geometry at the carbon atom. The positions of the three methyl hydrogen atoms of C(10) cannot be estimated accurately because of the possibility of rotation about the C(9)–C(10) direction. The positions of these three hydrogens were predicted assuming tetrahedral geometry and a staggered conformation with respect to the hydrogens at C(9). Although this assumption gives chemically reasonable intermolecular distances, the positions of these three hydrogen atoms remain in doubt. In each case, a carbon–hydrogen distance of 1·05 Å was assumed which is typical of values found in direct X-ray determinations. A final structure-factor calculation with all but the methyl hydrogen atoms included and each given a temperature factor 1 Å^2 greater than the B of the bonded carbon atom gave $R_1=0·110$, $R_2=0·116$.

The final values of the positional and vibrational parameters are listed in Table 1, together with their e.s.d.'s derived from the inverse least squares matrix. Table 2 lists the predicted hydrogen atom positions. Table 3 lists, for each reflexion, the observed structure factor and that calculated for all non-hydrogen atoms.

Table 1. *Atomic positional and isotropic thermal parameters*

Numbers in parentheses here and in succeeding tables are estimated standard deviations in the least significant digits.

Positional parameters are × 10^4.

	x/*a*	*y*/*b*	*z*/*c*	*B*(Å^2)
Fe	0 (0)	2523 (5)	2500 (0)	3·1 (2)
Cl	0 (0)	0475 (8)	2500 (0)	3·5 (2)
O	1012 (11)	3203 (13)	2403 (15)	4·1 (4)
N	379 (12)	2763 (16)	4350 (15)	3·1 (4)
C(1)	1664 (17)	3603 (23)	3181 (22)	4·0 (5)
C(2)	2356 (18)	4027 (24)	2725 (24)	5·1 (6)
C(3)	3029 (17)	4477 (24)	3556 (25)	5·0 (6)
C(4)	3087 (16)	4550 (24)	4776 (23)	5·0 (6)
C(5)	2449 (18)	4128 (21)	5220 (22)	3·5 (5)
C(6)	1746 (15)	3687 (21)	4425 (21)	3·8 (5)
C(7)	1091 (17)	3187 (20)	4984 (21)	3·8 (6)
C(8)	−161 (13)	2260 (19)	5145 (18)	3·1 (5)
C(9)	−572 (16)	3440 (23)	5567 (21)	4·4 (6)
C(10)	−1237 (17)	2882 (24)	6220 (23)	5·5 (7)

Table 2. *Idealized positional coordinates for the hydrogen atoms* (× 10^4)

A C–H distance of 1·05 Å was assumed.

	x/*a*	*y*/*b*	*z*/*c*
H(C2)	2342	3991	1813
H(C3)	3531	4785	3245
H(C4)	3612	4923	5352
H(C5)	2491	4138	6141
H(C7)	1204	3174	5918
H(C8*A*)	−612	1667	4657
H(C8*B*)	198	1791	5883
H(C9*A*)	−133	3968	6164
H(C9*B*)	−859	3982	4833
H(C10*A*)	−1543	3602	6532*
H(C10*B*)	−1663	2348	5611*
H(C10*C*)	−937	2333	6942*

* Predicted assuming a staggered conformation with respect to the hydrogens at C(9).

Discussion of the structure

Fig. 1 shows a stereoscopic view of the molecule and defines the labelling scheme for the atoms. This diagram was drawn using the program *ORTEP* (Johnson, 1965), as were all subsequent diagrams. Table 4 gives pertinent intramolecular distances and angles calculated with the program *ORFFE* (Busing, Martin & Levy, 1964). In the discussion which follows, any symmetry transformation to be applied to the coordinates of an atom in Tables 1 and 2 will be stated in parentheses after the atom name.

The iron is atom five coordinate, intermediate between square pyramidal (with the chlorine apical) and trigonal bypyramidal (with the two nitrogens apical). Bond lengths within the molecule are consistent with those previously reported for comparable situations. The Fe–Cl distance of 2·23 Å is very similar to the values

Table 3. *Observed and calculated structure factors*

F_o and F_c are ×10. Unobserved reflexions are indicated by an asterisk.

H	L	FO	FC
<<<<< K = 0 >>>>>			
0	-10	254	282
2	-10	203	144
4	-10	214	257
6	-10	118	135
8	-10	93	77
-2	-8	295	254
0	-8	387	390
2	-8	262	264
4	-8	109	128
6	-8	259	246
-4	-6	314	318
-2	-6	504	480
0	-6	414	417
2	-6	705	694
4	-6	393	394
-6	-4	311	382
-4	-4	680	861
-2	-4	682	751
0	-4	1196	1186
2	-4	430	397
-8	-2	221	266
-4	-2	116	146
-2	-2	69	68
0	-2	76	151
-10	0	441	430
-8	0	518	499
-6	0	565	566
-4	0	711	734
-12	2	360	331
-10	2	525	512
-8	2	867	850
-6	2	394	383
-4	2	617	573
-2	2	916	913
-14	4	231	100
-12	4	391	358
-10	4	462	435
-8	4	515	514
-6	4	60	46*
-4	4	470	390
-14	6	274	226
-12	6	202	196
-10	6	191	193
-8	6	581	468
-6	6	576	517
-14	8	143	132
-12	8	162	165
-10	8	528	472
-8	8	478	456
-10	10	271	231
<<<<< K = 1 >>>>>			
1	-11	165	196
3	-11	79	78
5	-11	193	194
7	-11	222	217
9	-11	163	173
1	-10	60	58*
3	-10	60	77*
7	-10	85	68
-1	-9	211	169
1	-9	192	110
3	-9	117	139
5	-9	108	124
7	-9	78	34*
-1	-8	66	112*
1	-8	163	163
3	-8	150	120
5	-8	176	142
7	-8	189	203
-3	-7	348	343
-1	-7	342	321
1	-7	331	334
3	-7	336	357
5	-7	279	260
-3	-6	192	176
-1	-6	154	99
1	-6	61	2*
3	-6	216	183
5	-6	58	72*
-5	-5	271	311
-3	-5	336	266
-1	-5	416	347
1	-5	721	783
3	-5	209	207
-5	-4	233	216
-3	-4	77	73
-1	-4	195	168
1	-4	88	65
3	-4	497	491
-7	-3	33	61*
-5	-3	131	106
-3	-3	405	396
-1	-3	831	828
1	-3	590	564
-7	-2	378	351
-5	-2	24	48*
-3	-2	349	348
-1	-2	569	674
1	-2	184	169
-9	-1	293	335
-7	-1	232	285
-5	-1	391	443
-3	-1	594	565
-1	-1	334	402
-9	0	144	142
-7	0	181	139
-5	0	495	526
-3	0	319	326
-1	0	117	56
-11	1	336	313
-9	1	356	308
-7	1	403	412
-5	1	435	401
-3	1	184	225
-1	1	1010	1214
-11	2	61	45*
-9	2	167	140
-7	2	50	20
-5	2	245	219
-3	2	401	438
-13	3	283	245
-11	3	252	234
-9	3	611	531
-7	3	695	587
-5	3	307	228
-3	3	175	144
-11	4	160	157
-9	4	95	63
-7	4	454	400
-5	4	280	234
-13	5	77	49*
-11	5	205	181
-9	5	379	364
-7	5	141	172
-5	5	92	131
-13	6	108	117
-11	6	151	109
-9	6	142	129
-7	6	125	94
-15	7	1 3	143
-13	7	230	209
-11	7	278	217
-9	7	473	449
-7	7	375	344
-13	8	56	23*
-11	8	130	173
-9	8	160	171
-11	9	128	178
-9	9	208	176
<<<<< K = 2 >>>>>			
2	-11	110	134
0	-10	92	68
2	-10	98	104
4	-10	73	18*
8	-10	62	29*
0	-9	65	32*
2	-9	66	51*
4	-9	73	3*
6	-9	69	33*
8	-9	122	132
-2	-8	214	196
0	-8	157	110
2	-8	83	75*
4	-8	78	30*
6	-8	82	5*
-2	-7	64	44*
0	-7	63	49*
2	-7	71	38*
4	-7	195	182
6	-7	208	213
-4	-6	66	69*
-2	-6	67	119*
0	-6	297	283
2	-6	66	94*
4	-6	226	204
-4	-5	163	137
-2	-5	174	192
0	-5	112	51
2	-5	55	37*
4	-5	248	274
-6	-4	298	272
-4	-4	261	274
-2	-4	494	521
0	-4	277	303
2	-4	309	270
-6	-3	375	437
-4	-3	37	69*
-2	-3	41	19*
0	-3	226	142
2	-3	238	213
-8	-2	48	28*
-6	-2	149	53
-4	-2	41	8*
-2	-2	585	510
0	-2	100	25
-8	-1	217	142
-6	-1	36	16*
-4	-1	81	65
-2	-1	457	544
0	-1	421	484
-10	0	151	136
-8	0	331	343
-6	0	135	156
-4	0	165	76
-2	0	1033	1076
0	0	826	772
-10	1	57	29*
-8	1	40	32
-6	1	234	173
-4	1	321	288
-2	1	387	332
-12	2	257	222
-10	2	300	267
-8	2	235	189
-6	2	241	229
-4	2	171	36
-2	2	193	172
-12	3	134	120
-10	3	214	210
-8	3	225	193
-6	3	481	375
-4	3	219	229
-14	4	71	48*
-12	4	112	89
-10	4	254	207
-8	4	68	72*
-6	4	64	7*
-4	4	84	40
-14	5	104	68
-12	5	66	66*
-10	5	129	108
-8	5	66	2*
-6	5	140	113
-14	6	65	42*
-12	6	81	50*
-10	6	78	85*
-8	6	195	131
-6	6	264	244
-14	7	49	39*
-12	7	68	40*
-10	7	100	93
-8	7	94	97
-12	8	63	45*
-10	8	240	183
-8	8	290	267
-12	9	116	120
-10	9	134	127
<<<<< K = 3 >>>>>			
3	-11	131	141
5	-11	83	66
1	-10	55	48*
3	-10	55	39*
-1	-9	71	80*
1	-9	75	29*
3	-9	80	27
5	-9	73	3*
7	-9	73	46*
-1	-8	67	45*
1	-8	67	74*
3	-8	75	64*
5	-8	102	70
7	-8	93	98
-3	-7	74	98*
-1	-7	109	86
1	-7	163	185
3	-7	186	190
5	-7	253	220
-3	-6	175	204
-1	-6	148	142
1	-6	257	255
3	-6	225	217
5	-6	100	87
-5	-5	227	181
-3	-5	113	119
-1	-5	413	402
1	-5	89	116
3	-5	380	359
-5	-4	52	39*
-3	-4	212	171
-1	-4	322	304
1	-4	215	220
3	-4	206	157
-7	-3	288	242
-5	-3	135	63
-3	-3	210	230
-1	-3	123	170
1	-3	76	108
-7	-2	48	78*
-5	-2	87	77
-3	-2	45	6*
-1	-2	85	50
1	-2	235	273
-9	-1	124	119
-7	-1	137	79
-5	-1	105	108
-3	-1	63	[illegible]
-1	-1	696	714
-9	0	112	75
-7	0	48	9*
-5	0	47	18*
-3	0	465	371
-1	0	880	955
-11	1	205	160
-9	1	165	138
-7	1	152	104
-5	1	307	282
-3	1	671	648
-1	1	411	240
-11	2	65	70*
-9	2	293	305
-7	2	303	288
-5	2	100	36
-3	2	460	370
-13	3	197	184
-11	3	221	194
-9	3	270	235
-7	3	395	321
-5	3	105	119
-3	3	186	52
-13	4	65	51*
-11	4	164	133
-9	4	203	190
-7	4	89	51
-5	4	266	235
-13	5	103	54
-11	5	84	70
-9	5	76	2*
-7	5	76	89
-5	5	227	152
-13	6	58	51*
-11	6	74	33*
-9	6	72	70*
-7	6	215	206
-13	7	92	87
-11	7	270	236
-9	7	162	127
-7	7	295	289
-13	8	48	14*
-11	8	134	115
-9	8	296	261
-11	9	56	78*
-9	9	67	27*
<<<<< K = 4 >>>>>			
0	-10	108	111
2	-10	187	173
4	-10	139	128
6	-10	95	99
8	-10	172	170
0	-9	104	80
2	-9	61	104*
4	-9	96	112
8	-9	131	163
-2	-8	127	131
0	-8	292	277
2	-8	235	222
4	-8	154	132
6	-8	313	309
-2	-7	212	232
0	-7	134	103
2	-7	75	54*
4	-7	72	46*
6	-7	67	61*
-4	-6	105	124
-2	-6	335	344
0	-6	252	241
2	-6	166	180
4	-6	382	354
-4	-5	88	97
-2	-5	60	69*
0	-5	322	362
2	-5	310	315
4	-5	61	64*
-6	-4	410	423
-4	-4	348	364
-2	-4	488	472
0	-4	336	240
2	-4	504	498
-4	-3	53	11*
-2	-3	349	372
0	-3	316	356
2	-3	71	73
-6	-2	255	226
-6	-2	294	282
-4	-2	62	91*
-2	-2	390	303
0	-2	238	243
-8	-1	155	130
-6	-1	52	3*
-4	-1	215	151
-2	-1	199	223
0	-1	220	244
-10	0	344	342
-8	0	347	347
-6	0	517	450
-4	0	487	492
-2	0	762	820
0	0	255	275
-10	1	183	176
-8	1	216	208
-6	1	248	243
-4	1	52	64*
-2	1	636	603
-12	2	213	236
-10	2	405	354
-8	2	266	245
-6	2	224	211
-4	2	502	489
-2	2	257	229
-12	3	66	27*
-10	3	188	164
-8	3	140	121
-6	3	223	211
-4	3	246	212
-14	4	126	141
-12	4	243	198
-10	4	205	182
-8	4	183	149
-6	4	497	439
-4	4	408	358
-14	5	52	26*
-12	5	176	133
-10	5	239	201
-8	5	234	226
-6	5	63	24*
-14	6	153	123
-12	6	209	184
-10	6	241	208
-8	6	269	242
-6	6	331	274
-12	7	60	9*
-10	7	163	185
-8	7	132	113
-12	8	208	210
-10	8	135	122
-8	8	185	141
-10	9	64	50
<<<<< K = 5 >>>>>			
1	-10	47	47*
-1	-9	120	113
1	-9	160	161
3	-9	69	140
5	-9	89	91
7	-9	211	221
-1	-8	62	86*
1	-8	63	37
3	-8	101	75
5	-8	164	159
7	-8	118	116
-3	-7	74	49*
-1	-7	314	300
1	-7	237	233
3	-7	296	363
5	-7	460	508
-3	-6	67	93*
-1	-6	94	148
1	-6	74	54*
3	-6	71	45*
5	-6	94	103
-5	-5	336	322
-3	-5	425	451
-1	-5	381	389
1	-5	375	416
3	-5	354	345
-5	-4	64	37*
-3	-4	62	12*
-1	-4	135	122
1	-4	91	82
3	-4	60	41*
-7	-3	355	387
-5	-3	446	486
-3	-3	231	243
-1	-3	327	361
1	-3	289	265
-7	-2	63	54*
-5	-2	83	37
-3	-2	88	92
-1	-2	58	27*
1	-2	53	57*
-9	-1	336	310
-7	-1	321	273
-5	-1	143	169
-3	-1	695	756
-1	-1	301	303
-9	0	66	70*
-7	0	61	5*
-5	0	322	333
-3	0	57	12*
-1	0	184	233
-11	1	185	153
-9	1	284	254
-7	1	474	442
-5	1	632	622
-3	1	655	686
-1	1	483	453
-11	2	164	144
-9	2	65	112*
-7	2	70	15*
-5	2	109	66
-3	2	110	96
-13	3	230	246
-11	3	479	422
-9	3	349	323
-7	3	377	357
-5	3	558	548
-3	3	314	307
-13	4	56	51*
-11	4	92	95
-9	4	75	58*
-7	4	70	1*
-5	4	298	314
-13	5	123	150
-11	5	110	118
-9	5	190	147
-7	5	200	200
-5	5	381	368
-13	6	46	25*
-11	6	65	17*
-9	6	69	11*
-7	6	134	53
-13	7	161	181
-11	7	275	287
-9	7	264	277
-7	7	284	259
-9	8	53	33*
<<<<< K = 6 >>>>>			
0	-9	45	14*
4	-9	105	115
-2	-8	61	60*
0	-8	150	163
2	-8	178	154
4	-8	211	219
6	-8	188	205
-2	-7	62	96*
0	-7	64	100*
2	-7	72	91*
4	-7	97	122
6	-7	61	13*
-4	-6	73	139*
-2	-6	191	183
0	-6	118	154
2	-6	110	188
4	-6	163	191
-4	-5	134	132
-2	-5	67	42*
0	-5	75	19*
2	-5	71	44*
4	-5	67	61*
-6	-4	203	227
-4	-4	382	393
-2	-4	116	141
0	-4	255	222
2	-4	194	186
-6	-3	67	92*
-4	-3	207	202
-2	-3	71	19*
0	-3	190	167
2	-3	263	286
-8	-2	151	143
-6	-2	220	235
-4	-2	79	168*
-2	-2	262	283
0	-2	208	184
-8	-1	134	169
-6	-1	130	177
-4	-1	347	362
-2	-1	91	66
0	-1	144	118
-10	0	129	91
-8	0	329	296
-6	0	285	282
-2	0	177	170
0	0	325	301
-10	1	132	102
-8	1	67	75*
-6	1	145	123
-4	1	247	233
-2	1	188	127
-12	2	66	121*
-10	2	245	276
-8	2	168	157
-4	2	251	294
-2	2	234	215
-12	3	57	49*
-8	3	75	65*
-6	3	100	97
-4	3	370	366
-14	4	127	129
-12	4	179	159
-10	4	79	45*
-6	4	269	258
-4	4	466	504
-12	5	50	68*
-10	5	68	78*
-8	5	70	53*
-6	5	189	187
-12	6	124	132
-8	6	152	149
-6	6	254	232
-10	7	53	47*
-8	7	57	80*
-8	8	60	66*
<<<<< K = 7 >>>>>			
-1	-8	45	48*
1	-8	48	9*
3	-8	109	127
-3	-7	58	53*
-1	-7	66	66
1	-7	72	118*
3	-7	134	146
5	-7	96	115
-3	-6	60	1*
-1	-6	69	124
1	-6	71	43*
3	-6	68	14*
5	-6	127	79
-5	-5	69	103*
-3	-5	152	167
-1	-5	82	76*
1	-5	77	78*
3	-5	81	39*
-5	-4	65	50*
-3	-4	67	26*
-1	-4	107	127
1	-4	72	71*
3	-4	134	120
-7	-3	102	134
-5	-3	157	175
-3	-3	84	72*
-1	-3	77	44*
1	-3	81	8*
-7	-2	66	1*
-5	-2	67	22*
-3	-2	75	45*
-1	-2	71	3*
1	-2	65	1*
-7	-1	78	61*
-5	-1	118	159
-3	-1	77	110*
-1	-1	112	103
-9	0	64	65*
-7	0	67	37*
-5	0	150	176
-3	0	172	52
-1	0	112	91
-11	1	127	79
-9	1	107	97
-7	1	84	78*
-5	1	285	331
-3	1	80	73*
-1	1	79	78*
-11	2	97	106
-9	2	63	13*
-7	2	75	62*
-5	2	72	38*
-3	2	66	14*
-13	3	152	140
-11	3	90	83
-9	3	78	85*
-7	3	77	85*
-5	3	62	110*
-3	3	232	221
-11	4	51	55*
-9	4	68	66*
-7	4	140	137
-5	4	66	1*
-11	5	58	53*
-9	5	66	8*
-7	5	76	17*
-5	5	196	195
-9	6	55	23*
-7	6	58	2*
-9	7	52	52*
-7	7	63	107*
<<<<< K = 8 >>>>>			
0	-7	65	40
4	-7	121	158
6	-7	83	40
-4	-6	51	94*
-2	-6	61	16*
0	-6	69	100*
2	-6	65	96*
4	-6	66	79*
-4	-5	110	118
-2	-5	132	135
0	-5	119	163
2	-5	131	159
4	-5	103	93
-6	-4	138	163
-4	-4	100	150
-2	-4	79	81
0	-4	75	31*
2	-4	78	89*
-6	-3	119	104
-4	-3	90	91
-2	-3	73	15*
0	-3	71	55*
2	-3	132	139
-8	-2	63	69*
-6	-2	73	24*
-4	-2	142	174
-2	-2	77	23*
0	-2	115	87
-8	-1	82	23
-6	-1	90	122
-4	-1	105	115
-2	-1	102	86
0	-1	95	64
-8	0	70	32*
-6	0	198	226
-4	0	190	187
-2	0	116	131
-10	1	50	55*
-8	1	120	123
-6	1	235	211
-4	1	226	266
-2	1	164	160
-10	2	59	9*
-8	2	105	122
-6	2	74	143*
-4	2	80	43*
-2	2	116	120
-10	3	48	56*
-8	3	65	56*
-6	3	67	14*
-4	3	92	64
-10	4	55	34*
-8	4	63	19*
-6	4	104	130
-4	4	155	177
-8	5	54	17*
-6	5	79	94
-8	6	74	70
-6	6	62	79*
<<<<< K = 9 >>>>>			
-1	-6	94	95
1	-6	81	118
5	-6	75	104
-3	-5	102	128
-1	-5	121	137
1	-5	58	68*
3	-5	148	168
-5	-4	88	97
-3	-4	51	79*
-1	-4	61	14*
1	-4	60	9*
3	-4	55	72*
-7	-3	90	172
-5	-3	119	159
-3	-3	157	182
-1	-3	68	87*
1	-3	206	215
-7	-2	93	104
-5	-2	55	96*
-3	-2	94	97
-1	-2	132	99
1	-2	87	96
-7	-1	59	108*
-5	-1	248	295
-3	-1	100	157
-1	-1	153	178
-7	0	52	47*
-5	0	186	192
-3	0	66	22*
-1	0	89	72
-9	1	48	37*
-7	1	240	291
-5	1	68	127*
-3	1	150	153
-1	1	189	169
-9	2	129	153
-7	2	82	68
-5	2	62	103*
-3	2	148	182
-9	3	126	136
-7	3	57	108*
-5	3	165	195
-3	3	203	245
-7	4	49	2*
-5	4	127	130
-7	5	164	210
-5	5	113	145
<<<<< K = 10 >>>>>			
-2	-4	165	199
0	-4	104	132
2	-4	162	207
0	-3	65	108
-4	-2	106	147
-2	-2	124	182
0	-2	121	164
-2	-1	54	14*
-6	0	235	259
-4	0	135	174
-2	0	153	200
0	0	212	220
-6	1	90	145
-6	2	90	173
-4	2	137	195
-2	2	148	183
-4	4	111	120

2·238 Å in *N,N'*-ethylenebis(salicylideneiminato)iron-(III) chloride (Gerloch & Mabbs, 1967), 2·213 Å in chlorobispentane-2,4-dionatoiron(III) (Lindley & Smith, 1970), and 2·26 Å in monochlorobis(diethyldithiocarbamato)iron(III) (Hoskins & White, 1970). The three C–C bonds most distant from the metal [C(2)–C(3), C(3)–C(4) and C(4)–C(5)] have an average length of 1·38 Å while the four C–C bonds nearest the metal [C(1)–C(2), C(1)–C(6), C(5)–C(6) and C(6)–C(7)] have an average length of 1·45 Å. This is in agreement with the work of Lingafelter & Braun (1966) who observed similar differences in the 'near' and 'far' sides of the benzene ring in a study of several salicylaldehyde complexes. Lingafelter & Braun have further shown that these differences are in excellent agreement with bond orders calculated by a simple Hückel orbital method.

The N–O distance (the 'bite' of the chelate) is 2·76 (3) Å. Within the errors, this is identical with the average bite observed for the same ligand in μ-oxo-bis[bis-*N*-n-propylsalicylaldiminatoiron(III)] (Davies & Gatehouse, 1972) where each iron atom has a coordination configuration very similar to that observed in the title compound.

The mean plane through the group of eleven atoms Fe, O, N, C(1) to C(8) was calculated using the procedure of Blow (1960). The plane is defined by the equation

$$-0·3984X+0·9133Y-0·0847Z=2·5298$$

where X, Y and Z are coordinates in Å relative to the orthogonal set of axes a,b,c^*. Fractional coordinates x,y,z in the crystal system are related to the coordinates X,Y,Z in the orthogonal system by the matrix equation

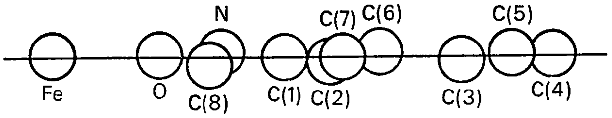

Fig. 2. The atoms Fe, O, N, C(1) to C(8) viewed parallel to their mean plane.

$$\begin{pmatrix} 16·82 & 0 & -2·820 \\ 0 & 10·90 & 0 \\ 0 & 0 & 11·21 \end{pmatrix} \begin{pmatrix} x \\ y \\ z \end{pmatrix} = \begin{pmatrix} X \\ Y \\ Z \end{pmatrix}.$$

Fig. 2 is a diagram of the eleven atoms Fe, O, N, C(1)–C(8) viewed parallel to the mean plane and Table 5 lists the deviations of these atoms from the plane.

Table 5. *Deviations* (Å) *from the weighted least-squares plane of all atoms used to define the plane*

Fe	0·025 (5)	C(4)	0·014 (26)
O	0·022 (15)	C(5)	0·029 (24)
N	0·042 (18)	C(6)	0·047 (23)
C(1)	−0·003 (25)	C(7)	−0·002 (23)
C(2)	−0·052 (27)	C(8)	−0·083 (21)
C(3)	−0·041 (27)		

The structure is a crystal racemate, consisting of the packing of discrete, monomeric molecules. Parallel sheets of right-handed molecules alternate with sheets of left-handed molecules, each sheet being parallel to

Table 4. *Selected intramolecular distances and angles*

Distances (Å)

Fe—Cl	2·23 (1)	C(1)–C(6)	1·41 (4)	C(6)–C(7)	1·51 (4)
Fe—O	1·89 (2)	C(2)–C(3)	1·38 (4)	C(7)–N	1·33 (3)
Fe—N	2·09 (2)	C(3)–C(4)	1·39 (4)	N—C(8)	1·54 (3)
O—C(1)	1·31 (3)	C(4)–C(5)	1·38 (4)	C(8)–C(9)	1·59 (4)
C(1)–C(2)	1·47 (4)	C(5)–C(6)	1·39 (3)	C(9)–C(10)	1·61 (4)

Angles (°)

O–Fe—O'	134 (1)	C(1)–C(2)–C(3)	117 (3)	C(5)–C(6)–C(7)	116 (2)
N–Fe—N'	166 (1)	C(1)–C(6)–C(7)	121 (2)	C(6)–C(7)–N	123 (2)
O–Fe—N	88 (1)	C(2)–C(3)–C(4)	125 (3)	C(7)–N—Fe	128 (2)
Cl–Fe—O	113 (1)	C(2)–C(1)–C(6)	117 (2)	C(7)–N—C(8)	112 (2)
Cl–Fe—N	97 (1)	C(3)–C(4)–C(5)	119 (3)	N—C(8)–C(9)	105 (2)
O–C(1)–C(2)	118 (2)	C(4)–C(5)–C(6)	119 (3)	C(8)–N—Fe	119 (1)
O–C(1)–C(6)	125 (2)	C(5)–C(6)–C(1)	123 (2)	C(8)–C(9)–C(10)	104 (2)

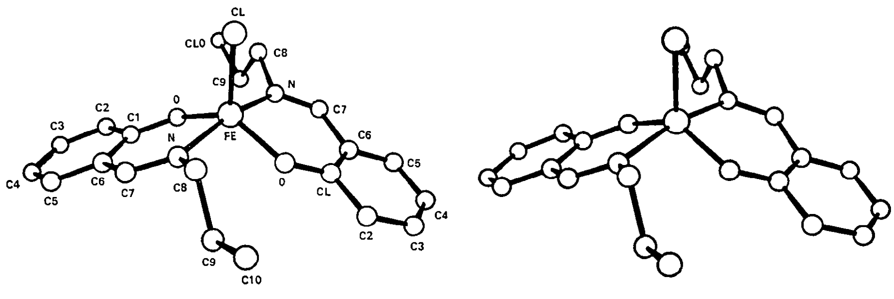

Fig. 1. The molecular geometry of chlorobis-(*N*-n-propylsalicylaldiminato)iron(III). The system for numbering the atoms is shown.

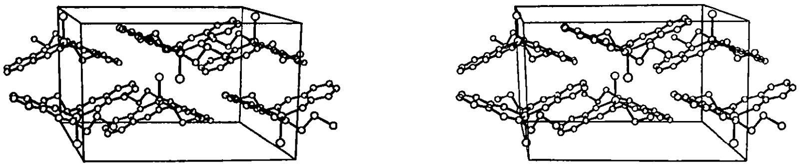

Fig. 3. The unit cell of chlorobis-(*N*-n-propylsalicylaldiminato)iron(III) viewed down the c^* axis.

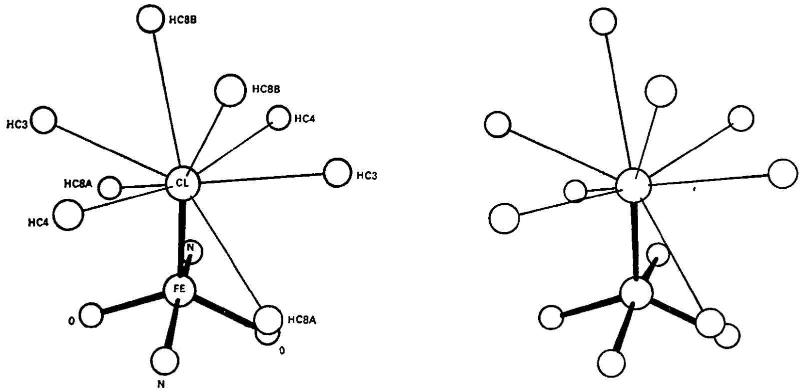

Fig. 4. The hydrogen atom environment of each chlorine atom, based on the predicted hydrogen positions.

the *ab* plane. The two enantiomers are related by the **c** glide and all molecules of the same enantiomer have identical orientations, relative to the crystal axes. Fig. 3 is an illustration of this packing arrangement.

There are no unusual intermolecular contacts. An interesting feature of the packing is that each chlorine atom is completely surrounded by the eight hydrogen atoms H(C3) $(x-\frac{1}{2}, y-\frac{1}{2}, z)$, H(C4) $(\frac{1}{2}-x, \frac{1}{2}-y, 1-z)$, H(C8*A*), H(C8*B*) $(-x, -y, 1-z)$ and the four related by the twofold rotation operation $(-x, y, \frac{1}{2}-z)$. This arrangement is illustrated in Fig. 4. The C–H–Cl angles vary from 110 to 175° and the calculated Cl–H distances vary from 2·9 (1) to 3·2 (1) Å. These distances are not significantly different from the sum of the van der Waals radii (3·0 Å, Pauling, 1960), and by the criteria of Hamilton & Ibers (1968), this effectively discounts any possibility of weak hydrogen bonding.

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