The *X*-Ray Crystal Structure of Chlorobis-(*N*-n-propylsalicylaldiminato)iron(III)

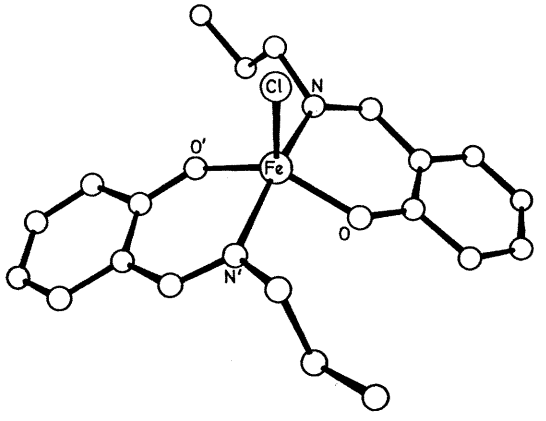
By J. E. DAVIES and B. M. GATEHOUSE*

(*Department of Chemistry, Monash University, Clayton, Victoria, Australia* 3168)

Summary A single-crystal *X*-ray diffraction study has shown that the title compound is monomeric and five-co-ordinate.

THE preparation, magnetic properties, and spectral data of several complexes of the type Fe(*N*-R-salicylaldiminato)$_2$X have been reported previously.[1] The magnetic moments and mass spectra of these complexes indicated that they are monomeric and probably trigonal bipyramidal. We now report the *X*-ray crystal structure analysis of Fe(*N*-n-propylsalicylaldiminato)$_2$Cl which confirms that this complex exists as a five-co-ordinate monomer in the solid state.

Crystal data: Dark-red prisms elongated along the [101] direction, monoclinic, $a = 16{\cdot}82 \pm 0{\cdot}02$, $b = 10{\cdot}90 \pm 0{\cdot}02$, $c = 11{\cdot}56 \pm 0{\cdot}02$ Å, $\beta = 104 \pm 0{\cdot}2°$, D_m *ca.* 1·40 g cm^{-3}, $Z = 4$, $D_c = 1{\cdot}34$ g cm^{-3}. The centrosymmetric space group $C2/c$ was confirmed by the structure analysis.



FIGURE

A total of 571 independent, non-zero reflections were visually estimated from equi-inclination Weissenberg photographs taken about the [101] direction with Mn-filtered Fe-K_α radiation. The initial parameters for the iron and chlorine atoms were deduced from a three-dimensional Patterson synthesis and the positions of the remaining atoms (excluding the hydrogens) were obtained from subsequent difference syntheses. Full-matrix least-squares refinement of the structure with individual isotropic temperature factors has reduced the conventional reliability index to a present value of 0·117. So far no corrections have been made for absorption or anomalous dispersion.

The structure contains both right- and left-handed molecules (related by the *c*-glide) which pack together to form a crystal racemate. The molecular structure is illustrated in the Figure. The iron and chlorine atoms lie on the two-fold axis at $Z = \frac{1}{4}$ and the two bidentate Schiff-base ligands are therefore symmetry related. The stereochemistry about the iron atom can best be described as intermediate between square-pyramidal (with the chlorine apical) and trigonal bipyramidal (with the two nitrogens apical). The relevant bond lengths and angles are given in the Table.

Parameters describing the stereochemistry about the iron atom

Fe–Cl	= 2·234 Å	N–Fe–N′	= 166°
Fe–N	= 2·096 Å	O–Fe–O′	= 134°
Fe–O	= 1·887 Å	O–Fe–N	= 87°
		Cl–Fe–O	= 113°
		Cl–Fe–N	= 97°

The Fe–Cl distance of 2·234 ± 0·009 Å is consistent with the previously reported values of 2·238 Å in *NN*′-bis(salicylideneiminato)iron(III) chloride[2] and 2·218 Å in α-chlorohemin.[3] The bidentate ligand is approximately planar and bond lengths in the ligand are very similar to those reported elsewhere for similar situations.[4]

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