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# Preparation and Structure of Chloro(bis{salicylideniminephenyl}disulfido)iron(III)

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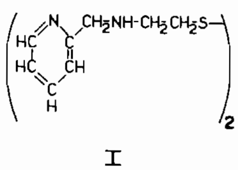
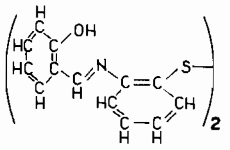
*Chloro(bis{o-salicylideniminephenyl}disulfido)iron (III), $Fe(C_{26}H_{18}N_2S_2O_2)Cl$, has been prepared and its crystal structure determined from precession camera data. The compound crystallized in space group $P2_1/c$ ($a = 10.02(2)$ Å, $b = 21.76(4)$ Å, $c = 11.06(2)$ Å, and $\beta = 96.17(10)°$ at 25° C using Zr filtered Mo Kα radiation) with four formula units per unit cell ($\varrho_{calcd} = 1.52\ gcm^{-3}$; $\varrho_{obsd} = 1.55(2) gcm^{-3}$). The structure, which was refined to a conventional R value of 0.096 by full-matrix least-squares refinement, consists of monomeric iron(III) complexes with octahedral coordination. The disulfide functions as a pentadentate ligand and is coordinated through two nitrogens, two oxygens, and one sulfur atom; the chloride ion occupies the sixth coordination position. The magnetic moment, 5.90 B.M., is normal for a high-spin octahedral iron(III) complex.*

## Introduction

As part of a general study of complexes of polydentate Schiff's base ligands, we became interested in ligands that contained sulfur donors and prepared a series of imines of salicylaldehyde with various aminothiols and aminodisulfides. Several examples of complexes with salicylaldimines of aminothiols[1,2] have been reported but no complexes of salicylaldimines of aminodisulfides have been characterized. In fact, there have been few structure studies of complexes containing any type of coordinated disulfide. The structure of a copper(I) chloride complex of diethyl disulfide has been determined[3] and the disulfide bridges two copper(I) ions; the structure of a monomeric iridium disulfide complex has also been determined.[4] A dinuclear cobalt carbonyl complex of di(perfluorophenyl)disulfide has been isolated[5] and evidence of disulfide coordination with chelate ligands has been obtained from solution studies,[6] particularly with amino acids such as cystine. In a recent paper, the structure of a nickel complex of bis(2-{2-pyridyl-methylamino}ethyl disulfide (I) was reported.[7] In that structure, only one sulfur of the disulfide group was coordinated to the metal ion. In order to obtain more information about the coordination of chelate ligands containing a disulfide group, we have investigated complexes of the imine (II) formed from salicylaldehyde and 2,2′-diaminodiphenyl disulfide. In this paper, we report the preparation, properties. and the structure of the iron(III) complex of the dianion of (II).


I


II

## Experimental

### *Preparation*

The ligand was formed by adding 0.02 moles of salicylaldehyde to a methanol solution containing 0.02 moles of 2,2′-diaminodiphenyldisulfide. To the ligand solution was added 0.02 moles of lithium dissolved in methanol and then a methanol solution containing 0.01 mole of $FeCl_3 \cdot H_2O$ was added dropwise to the stirred solution. After addition of the ferric chloride solution, stirring was continued and the solution was heated to its boiling point. The solution was filtered while hot and cooling the filtrate produced a black microcrystalline solid which was separated from the solution by filtration. Crystals continued to form over a period of several days and another filtration yielded crystals suitable for single-crystal X-ray diffraction studies.

Anal. Calcd. for $FeC_{26}H_{18}N_2S_2O_2Cl$ : Fe, 10.23; C, 57.21; H, 3.33; N, 5.13; S, 11.74. Found: Fe, 10.24; C, 56.16; H, 3.18; N, 5.06; S, 11.26.

### *Magnetic Susceptibility*

The magnetic susceptibility of the compound was determined by the Faraday method using $HgCo(NCS)_4$ as a calibrant, at 298° K and at 196° K. Diamagnetic corrections were made using published atomic values.[8] The values obtained, with the 298° K result listed first in each case, are $\chi_g = 26.0 \times 10^{-6}$, $40.2 \times 10^{-6}$; $\chi_M = 14{,}450 \times 10^{-6}$, $22{,}250 \times 10^{-6}$. The same value of $\mu_{eff}$ was obtained at both temperatures, 5.90 B.M.

TABLE I. Final Positional and Thermal Parameters for $Fe(C_{20}H_{18}N_2S_2O_2)Cl$.

| Atom | x | y | z | B, Å$^2$ |
|---|---|---|---|---|
| Fe | 0.2441(1) | 0.1142(1) | 0.0549(2) | [a] |
| Cl | 0.2143(3) | 0.0583(2) | –0.1239(3) | [a] |
| S(1) | 0.0911(3) | 0.0326(2) | 0.1284(3) | [a] |
| S(2) | 0.0591(4) | 0.0464(2) | 0.3059(4) | [a] |
| O(1) | 0.3890(8) | 0.1666(4) | 0.0380(8) | [a] |
| O(2) | 0.1030(8) | 0.1707(4) | 0.0056(8) | [a] |
| N(1) | 0.3885(9) | 0.0440(5) | 0.1302(10) | [a] |
| N(2) | 0.2325(10) | 0.1519(6) | 0.2348(10) | [a] |
| C(1) | 0.5132(11) | 0.0581(6) | 0.1574(12) | 3.70(37) |
| C(2) | 0.2080(14) | 0.2131(7) | 0.2494(15) | 4.74(47) |
| RlCl | 0.5131(11) | 0.1689(6) | 0.0906(11) | 3.41(36) |
| R1C2 | 0.5791(11) | 0.1156(6) | 0.1461(11) | 3.54(37) |
| R1C3 | 0.7182(13) | 0.1176(7) | 0.1931(13) | 4.41(43) |
| R1R4 | 0.7878(15) | 0.1704(7) | 0.1800(15) | 5.10(50) |
| R1C5 | 0.7261(16) | 0.2255(8) | 0.1270(16) | 5.48(53) |
| R1R6 | 0.5867(16) | 0.2238(8) | 0.0860(15) | 5.49(53) |
| R2C1 | 0.0847(11) | 0.2284(6) | 0.0421(12) | 3.41(37) |
| R2C2 | 0.1434(11) | 0.2503(6) | 0.1583(11) | 3.61(39) |
| R2C3 | 0.1204(16) | 0.3103(8) | 0.1856(16) | 5.68(56) |
| R2C4 | 0.0404(15) | 0.3503(8) | 0.1139(15) | 5.29(52) |
| R2C5 | –0.0155(14) | 0.3283(7) | 0.0036(15) | 4.97(50) |
| R2C6 | 0.0045(12) | 0.2678(6) | –0.0340(12) | 4.02(42) |
| R3C1 | 0.3451(10) | –0.0176(15) | 0.1440(11) | 2.94(32) |
| R3C2 | 0.2069(9) | –0.0289(5) | 0.1404(10) | 2.64(30) |
| R3C3 | 0.1593(12) | –0.0890(6) | 0.1480(13) | 3.87(39) |
| R3C4 | 0.2469(12) | –0.1370(6) | 0.1578(12) | 3.81(40) |
| R3C5 | 0.3815(11) | –0.1271(6) | 0.1669(11) | 3.41(37) |
| R3C6 | 0.4316(10) | –0.0664(5) | 0.1530(11) | 3.20(35) |
| R4C1 | 0.2838(15) | 0.1203(8) | 0.3400(14) | 4.97(48) |
| R4C2 | 0.2176(13) | 0.9687(7) | 0.3775(13) | 4.42(44) |
| R4C3 | 0.2654(18) | 0.0343(9) | 0.4828(17) | 6.47(62) |
| R4C4 | 0.3841(20) | 0.0519(10) | 0.5449(19) | 7.03(68) |
| R4C5 | 0.4594(20) | 0.1050(10) | 0.5128(20) | 7.25(71) |
| R4C6 | 0.4087(16) | 0.1367(8) | 0.4051(15) | 5.43(53) |

Anisotropic Thermal Parameters[b] for Iron, Sulfur, and Atoms Coordinated to Iron:

| Atom | $10^4\beta_{11}$ | $10^4\beta_{22}$ | $10^4\beta_{33}$ | $10^4\beta_{12}$ | $10^4\beta_{13}$ | $10^4\beta_{23}$ |
|---|---|---|---|---|---|---|
| Fe | 65(1) | 15.8(3) | 71(2) | 5(1) | –2(1) | –2(1) |
| Cl | 76(2) | 20.0(6) | 71(3) | 2(1) | 0(2) | –5(2) |
| S(1) | 59(2) | 21(7) | 92(3) | 1(1) | 7(2) | 4(2) |
| S(2) | 104(3) | 31(1) | 108(4) | 6(2) | 45(3) | 6(2) |
| O(1) | 94(8) | 20(2) | 85(8) | –4(3) | –4(7) | –2(5) |
| O(2) | 91(8) | 21(2) | 70(7) | 13(3) | –17(6) | 7(5) |
| N(1) | 79(8) | 21(2) | 76(9) | 3(4) | 7(7) | –2(6) |
| N(2) | 100(11) | 28(3) | 81(10) | 3(5) | 2(9) | 4(7) |

[a] Atoms refined anisotropically. [b] The form of the anisotropic thermal ellipsoid is $\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+\beta_{12}hk+\beta_{13}hl+\beta_{23}kl)]$.

### *Collection and Reduction of X-ray Data*

A crystal of approximate dimensions 0.15 × 0.15 × 0.95 mm was mounted along the long dimension (a*-axis) on a glass fiber. Precession photographs were taken, using Zirconium-filtered molybdenum K$\alpha$ radiation, $\lambda$ = 0.7106 Å. The crystal was found to be monoclinic with a = 10.02(2) Å[9], b = 21.76(4) Å, c = 11.06 (2) Å, and $\beta$ = 96.17(10)° at 25° C. The density calculated on the basis of 4 formula units per unit cell, 1.52 g/cm$^3$, agrees well with the experimental value, 1.55(2) g/cm$^3$, obtained by the flotation method using a mixture of heptane and carbon tetrachloride.

TABLE II. Observed and Calculated Structure Factors (in Electrons) for Fe($C_{26}H_{18}N_2S_2O_2$)Cl.

| H | K | FO | FC |
|---|---|---|---|
| L=0 | | | |
| 0 | 6 | 22 | 23 |
| 0 | 10 | 111 | 109 |
| 0 | 12 | 95 | 91 |
| 0 | 14 | 84 | 80 |
| 0 | 18 | 61 | 63 |
| 0 | 20 | 22 | 23 |
| 0 | 22 | 20 | 22 |
| 1 | 4 | 108 | 108 |
| 1 | 6 | 17 | 18 |
| 1 | 7 | 40 | 35 |
| 1 | 8 | 30 | 33 |
| 1 | 9 | 24 | 28 |
| 1 | 10 | 27 | 35 |
| 1 | 13 | 27 | 24 |
| 1 | 14 | 32 | 31 |
| 1 | 15 | 66 | 73 |
| 1 | 16 | 26 | 26 |
| 1 | 17 | 33 | 32 |
| 1 | 18 | 21 | 17 |
| 1 | 19 | 15 | 17 |
| 1 | 21 | 26 | 27 |
| 1 | 22 | 11 | 9 |
| 1 | 23 | 16 | 12 |
| 2 | 2 | 36 | 33 |
| 2 | 4 | 91 | 98 |
| 2 | 5 | 90 | 94 |
| 2 | 6 | 44 | 47 |
| 2 | 7 | 23 | 24 |
| 2 | 8 | 73 | 76 |
| 2 | 9 | 45 | 39 |
| 2 | 10 | 53 | 51 |
| 2 | 11 | 23 | 17 |
| 2 | 12 | 51 | 46 |
| 2 | 15 | 14 | 17 |
| 2 | 16 | 35 | 33 |
| 2 | 18 | 21 | 21 |
| 2 | 22 | 31 | 30 |
| 3 | 3 | 74 | 70 |
| 3 | 5 | 43 | 40 |
| 3 | 6 | 38 | 40 |
| 3 | 7 | 154 | 146 |
| 3 | 8 | 50 | 49 |
| 3 | 9 | 61 | 58 |
| 3 | 11 | 31 | 32 |
| 3 | 12 | 26 | 21 |
| 3 | 13 | 36 | 35 |
| 3 | 15 | 27 | 28 |
| 3 | 16 | 14 | 10 |
| 3 | 17 | 15 | 15 |
| 3 | 19 | 45 | 51 |
| 3 | 21 | 37 | 40 |
| 3 | 22 | 11 | 10 |
| 4 | 0 | 82 | 80 |
| 4 | 1 | 21 | 18 |
| 4 | 3 | 23 | 21 |
| 4 | 4 | 93 | 98 |
| 4 | 5 | 27 | 31 |
| 4 | 6 | 46 | 43 |
| 4 | 7 | 43 | 51 |
| 4 | 8 | 38 | 38 |
| 4 | 9 | 37 | 34 |
| 4 | 10 | 13 | 16 |
| 4 | 11 | 16 | 14 |
| 4 | 12 | 29 | 30 |
| 4 | 13 | 17 | 15 |
| 4 | 16 | 26 | 25 |
| 4 | 17 | 15 | 14 |
| 4 | 21 | 26 | 26 |
| 4 | 22 | 23 | 21 |
| 4 | 23 | 20 | 11 |
| 5 | 0 | 58 | 54 |
| 5 | 1 | 31 | 40 |
| 5 | 3 | 113 | 110 |
| 5 | 4 | 29 | 35 |
| 5 | 6 | 25 | 24 |
| 5 | 7 | 21 | 24 |
| 5 | 8 | 13 | 12 |
| 5 | 9 | 26 | 29 |
| 5 | 11 | 45 | 51 |
| 5 | 14 | 24 | 26 |
| 5 | 15 | 19 | 21 |
| 5 | 16 | 15 | 16 |
| 5 | 17 | 19 | 19 |
| 5 | 18 | 23 | 19 |
| 5 | 19 | 11 | 10 |
| 5 | 21 | 11 | 12 |
| 6 | 0 | 141 | 142 |
| 6 | 1 | 26 | 27 |
| 6 | 2 | 34 | 38 |
| 6 | 3 | 56 | 56 |
| 6 | 4 | 49 | 50 |
| 6 | 5 | 37 | 42 |
| 6 | 7 | 14 | 13 |
| 6 | 8 | 25 | 28 |
| 6 | 11 | 28 | 31 |
| 6 | 12 | 61 | 64 |
| 6 | 13 | 44 | 43 |
| 6 | 14 | 29 | 30 |
| 6 | 17 | 20 | 22 |
| 6 | 18 | 23 | 22 |
| 6 | 20 | 11 | 10 |
| 6 | 21 | 20 | 19 |
| 6 | 22 | 11 | 12 |
| 7 | 0 | 67 | 67 |
| 7 | 1 | 47 | 48 |
| 7 | 2 | 67 | 61 |
| 7 | 3 | 42 | 48 |
| 7 | 8 | 40 | 40 |
| 7 | 10 | 33 | 34 |
| 7 | 11 | 19 | 24 |
| 7 | 12 | 22 | 22 |
| 7 | 13 | 30 | 26 |
| 7 | 15 | 23 | 21 |
| 7 | 17 | 11 | 13 |
| 7 | 18 | 11 | 14 |
| 7 | 19 | 16 | 16 |
| 8 | 0 | 44 | 40 |
| 8 | 1 | 29 | 27 |
| 8 | 2 | 25 | 24 |
| 8 | 3 | 29 | 30 |
| 8 | 4 | 41 | 43 |
| 8 | 5 | 27 | 25 |
| 8 | 8 | 34 | 32 |
| 8 | 10 | 16 | 15 |
| 8 | 14 | 16 | 18 |
| 8 | 16 | 16 | 12 |
| 9 | 5 | 23 | 26 |
| 9 | 7 | 43 | 46 |
| 9 | 11 | 16 | 18 |
| 10 | 4 | 31 | 27 |
| 10 | 6 | 16 | 13 |
| 10 | 7 | 16 | 18 |
| 10 | 8 | 23 | 29 |
| 10 | 10 | 23 | 20 |
| 11 | 4 | 16 | 16 |
| 11 | 5 | 16 | 12 |
| 11 | 7 | 16 | 16 |
| 11 | 11 | 19 | 17 |
| 12 | 3 | 15 | 11 |
| 12 | 8 | 28 | 19 |
| 12 | 10 | 14 | 9 |
| H | K | FO | FC |
| L=1 | | | |
| 0 | 5 | 13 | 14 |
| 0 | 6 | 35 | 29 |
| 0 | 8 | 19 | 19 |
| 0 | 9 | 21 | 28 |
| 0 | 10 | 76 | 68 |
| 0 | 11 | 16 | 14 |
| 0 | 12 | 9 | 9 |
| 0 | 13 | 39 | 41 |
| 0 | 15 | 28 | 34 |
| 0 | 17 | 39 | 43 |
| 0 | 18 | 22 | 18 |
| 0 | 19 | 19 | 12 |
| 0 | 21 | 31 | 36 |
| 0 | 22 | 16 | 19 |
| 0 | 24 | 13 | 13 |
| 1 | 4 | 75 | 74 |
| 1 | 5 | 13 | 9 |
| 1 | 6 | 23 | 22 |
| 1 | 7 | 51 | 44 |
| 1 | 8 | 23 | 22 |
| 1 | 10 | 27 | 29 |
| 1 | 11 | 31 | 30 |
| 1 | 12 | 25 | 31 |
| 1 | 14 | 27 | 27 |
| 1 | 15 | 10 | 8 |
| 1 | 16 | 47 | 53 |
| 1 | 20 | 20 | 21 |
| 1 | 21 | 9 | 10 |
| 1 | 24 | 13 | 15 |
| 1 | 25 | 10 | 9 |
| 2 | 2 | 46 | 45 |
| 2 | 3 | 25 | 25 |
| 2 | 4 | 128 | 120 |
| 2 | 5 | 76 | 74 |
| 2 | 6 | 121 | 117 |
| 2 | 7 | 12 | 11 |
| 2 | 8 | 65 | 56 |
| 2 | 9 | 78 | 77 |
| 2 | 10 | 36 | 38 |
| 2 | 11 | 40 | 37 |
| 2 | 12 | 17 | 17 |
| 2 | 13 | 49 | 52 |
| 2 | 14 | 13 | 13 |
| 2 | 15 | 29 | 33 |
| 2 | 16 | 16 | 13 |
| 2 | 17 | 20 | 20 |
| 2 | 19 | 15 | 18 |
| 2 | 20 | 12 | 10 |
| 2 | 23 | 13 | 10 |
| 2 | 24 | 13 | 11 |
| 3 | 1 | 123 | 118 |
| 3 | 2 | 52 | 58 |
| 3 | 3 | 80 | 83 |
| 3 | 5 | 24 | 33 |
| 3 | 6 | 117 | 110 |
| 3 | 7 | 63 | 59 |
| 3 | 8 | 65 | 64 |
| 3 | 9 | 105 | 102 |
| 3 | 10 | 24 | 25 |
| 3 | 11 | 43 | 47 |
| 3 | 13 | 10 | 9 |
| 3 | 14 | 35 | 45 |
| 3 | 15 | 21 | 21 |
| 3 | 17 | 14 | 15 |
| 3 | 18 | 12 | 16 |
| 3 | 20 | 41 | 42 |
| 3 | 21 | 9 | 9 |
| 3 | 22 | 16 | 17 |
| 3 | 23 | 9 | 8 |
| 4 | 1 | 29 | 33 |
| 4 | 2 | 62 | 64 |
| 4 | 3 | 72 | 70 |
| 4 | 4 | 52 | 44 |
| 4 | 5 | 96 | 100 |
| 4 | 6 | 29 | 30 |
| 4 | 7 | 18 | 15 |
| 4 | 8 | 32 | 36 |
| 4 | 9 | 69 | 75 |
| 4 | 11 | 18 | 17 |
| 4 | 12 | 24 | 24 |
| 4 | 13 | 8 | 7 |
| 4 | 14 | 21 | 20 |
| 4 | 15 | 35 | 41 |
| 4 | 17 | 15 | 15 |
| 4 | 18 | 15 | 16 |
| 4 | 19 | 15 | 15 |
| 4 | 21 | 9 | 11 |
| 4 | 22 | 13 | 16 |
| 4 | 23 | 17 | 18 |
| 5 | 4 | 42 | 45 |
| 5 | 5 | 26 | 29 |
| 5 | 6 | 72 | 67 |
| 5 | 7 | 13 | 9 |
| 5 | 8 | 49 | 54 |
| 5 | 10 | 18 | 14 |
| 5 | 11 | 21 | 22 |
| 5 | 12 | 37 | 37 |
| 5 | 15 | 21 | 19 |
| 5 | 19 | 16 | 14 |
| 5 | 20 | 19 | 19 |
| 6 | 1 | 76 | 76 |
| 6 | 2 | 16 | 17 |
| 6 | 3 | 45 | 50 |
| 6 | 6 | 11 | 11 |
| 6 | 7 | 16 | 12 |
| 6 | 8 | 23 | 28 |
| 6 | 9 | 48 | 51 |
| 6 | 10 | 38 | 47 |
| 6 | 11 | 30 | 29 |
| 6 | 12 | 26 | 24 |
| 6 | 13 | 43 | 40 |
| 6 | 14 | 12 | 14 |
| 6 | 15 | 29 | 31 |
| 6 | 17 | 21 | 20 |
| 6 | 20 | 13 | 16 |
| 6 | 21 | 17 | 17 |
| 6 | 22 | 14 | 14 |
| 7 | 2 | 67 | 68 |
| 7 | 3 | 12 | 14 |
| 7 | 4 | 15 | 17 |
| 7 | 5 | 21 | 23 |
| 7 | 6 | 12 | 10 |
| 7 | 7 | 17 | 17 |
| 7 | 8 | 19 | 17 |
| 7 | 12 | 24 | 28 |
| 7 | 14 | 9 | 6 |
| 7 | 16 | 30 | 32 |
| 7 | 18 | 13 | 12 |
| 7 | 20 | 9 | 7 |
| 7 | 22 | 10 | 10 |
| 8 | 2 | 22 | 24 |
| 8 | 3 | 22 | 24 |
| 8 | 4 | 18 | 16 |
| 8 | 12 | 9 | 5 |
| 8 | 1 | 29 | 33 |
| 8 | 5 | 13 | 14 |
| 8 | 6 | 16 | 17 |
| 8 | 7 | 29 | 34 |
| 8 | 9 | 37 | 38 |
| 8 | 10 | 26 | 26 |
| 8 | 13 | 33 | 29 |
| 8 | 14 | 13 | 11 |
| 8 | 15 | 13 | 17 |
| 0 | 16 | 47 | 55 |
| 8 | 16 | 9 | 8 |
| 8 | 20 | 10 | 7 |
| 8 | 21 | 14 | 15 |
| 9 | 1 | 27 | 25 |
| 9 | 3 | 28 | 30 |
| 9 | 4 | 21 | 22 |
| 9 | 5 | 13 | 12 |
| 9 | 6 | 57 | 58 |
| 9 | 8 | 30 | 32 |
| 9 | 9 | 16 | 17 |
| 9 | 10 | 13 | 12 |
| 9 | 11 | 9 | 11 |
| 9 | 12 | 27 | 23 |
| 9 | 13 | 13 | 13 |
| 9 | 7 | 9 | 8 |
| 10 | 1 | 17 | 17 |
| 10 | 2 | 9 | 10 |
| 10 | 3 | 26 | 25 |
| 10 | 5 | 29 | 28 |
| 10 | 7 | 9 | 13 |
| 10 | 9 | 37 | 41 |
| 10 | 11 | 14 | 14 |
| 11 | 1 | 14 | 15 |
| 11 | 2 | 10 | 7 |
| 11 | 3 | 10 | 8 |
| 11 | 4 | 14 | 9 |
| 11 | 6 | 31 | 30 |
| 11 | 8 | 17 | 16 |
| 11 | 9 | 14 | 11 |
| 11 | 10 | 14 | 12 |
| 11 | 12 | 14 | 9 |
| 12 | 1 | 14 | 12 |
| 12 | 3 | 14 | 12 |
| 12 | 5 | 14 | 13 |
| 12 | 6 | 9 | 10 |
| 12 | 7 | 9 | 8 |
| 12 | 8 | 14 | 12 |
| 12 | 9 | 19 | 18 |
| 12 | 10 | 9 | 3 |
| 12 | 13 | 13 | 11 |
| 13 | 2 | 13 | 13 |
| 13 | 10 | 12 | 13 |
| 13 | 12 | 15 | 12 |
| -1 | 4 | 68 | 62 |
| -1 | 5 | 46 | 53 |
| -1 | 6 | 105 | 97 |
| -1 | 7 | 55 | 54 |
| -1 | 8 | 42 | 39 |
| -1 | 9 | 57 | 53 |
| -1 | 10 | 27 | 27 |
| -1 | 11 | 25 | 25 |
| -1 | 12 | 23 | 23 |
| -1 | 13 | 39 | 42 |
| -1 | 14 | 23 | 23 |
| -1 | 15 | 20 | 18 |
| -1 | 16 | 44 | 47 |
| -1 | 17 | 39 | 43 |
| -1 | 18 | 11 | 10 |
| -1 | 19 | 15 | 17 |
| -1 | 20 | 33 | 39 |
| -1 | 21 | 9 | 6 |
| -1 | 24 | 9 | 9 |
| -2 | 2 | 88 | 90 |
| -2 | 3 | 129 | 119 |
| -2 | 4 | 71 | 76 |
| -2 | 5 | 83 | 80 |
| -2 | 6 | 88 | 82 |
| -2 | 7 | 52 | 48 |
| -2 | 8 | 64 | 60 |
| -2 | 10 | 57 | 53 |
| -2 | 12 | 12 | 15 |
| -2 | 13 | 21 | 21 |
| -2 | 14 | 10 | 7 |
| -2 | 15 | 24 | 29 |
| -2 | 17 | 49 | 52 |
| -2 | 18 | 19 | 22 |
| -2 | 19 | 17 | 17 |
| -2 | 20 | 22 | 21 |
| -2 | 22 | 9 | 8 |
| -2 | 23 | 16 | 18 |
| -2 | 24 | 17 | 14 |
| -2 | 25 | 10 | 4 |
| -2 | 26 | 10 | 8 |
| -3 | 2 | 75 | 80 |
| -3 | 3 | 122 | 116 |
| -3 | 4 | 107 | 108 |
| -3 | 5 | 17 | 23 |
| -3 | 6 | 66 | 70 |
| -3 | 7 | 26 | 25 |
| -3 | 8 | 81 | 86 |
| -3 | 10 | 27 | 29 |
| -3 | 11 | 21 | 22 |
| -3 | 13 | 10 | 11 |
| -3 | 14 | 74 | 76 |
| -3 | 16 | 28 | 30 |
| -3 | 17 | 12 | 11 |
| -3 | 18 | 12 | 8 |
| -3 | 19 | 8 | 7 |
| -3 | 20 | 24 | 25 |
| -3 | 22 | 21 | 23 |
| -3 | 23 | 13 | 10 |
| -4 | 1 | 60 | 57 |
| -4 | 2 | 28 | 34 |
| -4 | 3 | 38 | 34 |
| -4 | 4 | 9 | 12 |
| -4 | 5 | 66 | 66 |
| -4 | 6 | 40 | 42 |
| -4 | 8 | 31 | 31 |
| -4 | 11 | 34 | 33 |
| -4 | 13 | 66 | 69 |
| -4 | 14 | 21 | 26 |
| -4 | 15 | 20 | 16 |
| -4 | 16 | 12 | 11 |
| -4 | 17 | 39 | 44 |
| -4 | 19 | 22 | 27 |
| -4 | 20 | 20 | 25 |
| -4 | 22 | 13 | 14 |
| -4 | 23 | 13 | 12 |
| -5 | 1 | 40 | 43 |
| -5 | 2 | 45 | 49 |
| -5 | 4 | 92 | 84 |
| -5 | 5 | 39 | 44 |
| -5 | 9 | 7 | 4 |
| -5 | 10 | 39 | 42 |
| -5 | 11 | 19 | 20 |
| -5 | 12 | 21 | 25 |
| -5 | 13 | 37 | 38 |
| -5 | 14 | 27 | 28 |
| -5 | 15 | 12 | 14 |
| -5 | 16 | 21 | 22 |
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| 5 | 9 | 16 | 16 |
| 5 | 1 | 10 | 10 |
| 6 | 7 | 16 | 17 |
| 6 | 8 | 16 | 22 |
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| 8 | 8 | 14 | 18 |
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| 9 | 5 | 17 | 21 |
| 9 | 6 | 14 | 14 |
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| 11 | 6 | 15 | 14 |
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| -10 | 5 | 14 | 15 |
| -10 | 7 | 14 | 12 |
| 1 | 4 | 29 | 34 |
| 1 | 5 | 20 | 26 |
| 1 | 6 | 12 | 11 |
| 1 | 7 | 43 | 47 |
| 1 | 8 | 31 | 32 |
| 1 | 9 | 15 | 15 |
| 1 | 0 | 20 | 16 |
| 1 | 1 | 22 | 21 |
| 1 | 2 | 18 | 17 |
| 2 | 3 | 49 | 45 |
| 2 | 4 | 7 | 11 |
| 2 | 5 | 11 | 15 |
| 2 | 6 | 13 | 15 |
| 2 | 7 | 17 | 17 |
| 2 | 8 | 10 | 5 |
| 2 | 9 | 16 | 18 |
| 2 | 1 | 12 | 12 |
| 2 | 2 | 19 | 19 |
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| 3 | 5 | 41 | 49 |
| 3 | 6 | 31 | 34 |
| 3 | 7 | 23 | 24 |
| 3 | 8 | 27 | 28 |
| 3 | 9 | 24 | 23 |
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| 3 | 2 | 13 | 8 |
| 4 | 6 | 10 | 10 |
| 4 | 7 | 11 | 8 |
| 4 | 8 | 16 | 15 |
| 4 | 9 | 21 | 17 |
| 4 | 0 | 18 | 15 |
| 4 | 1 | 19 | 13 |
| 5 | 5 | 35 | 40 |
| 5 | 6 | 46 | 45 |
| 6 | 0 | 51 | 42 |
| 6 | 6 | 38 | 45 |
| 6 | 8 | 13 | 13 |
| 6 | 9 | 27 | 27 |
| 7 | 5 | 22 | 28 |
| 7 | 6 | 18 | 21 |
| 7 | 7 | 23 | 27 |
| 7 | 8 | 27 | 25 |
| 8 | 5 | 18 | 18 |
| 8 | 9 | 24 | 24 |
| 9 | 5 | 19 | 17 |
| 9 | 6 | 20 | 21 |
| 9 | 7 | 14 | 14 |
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| -5 | 5 | 40 | 37 |
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| -8 | 9 | 33 | 30 |
| -12 | 5 | 23 | 21 |
| 1 | 5 | 24 | 22 |
| 1 | 6 | 67 | 58 |
| 1 | 7 | 59 | 58 |
| 1 | 8 | 22 | 24 |
| 1 | 9 | 34 | 33 |
| 1 | 0 | 18 | 13 |
| 1 | 1 | 19 | 15 |
| 2 | 4 | 30 | 26 |
| 2 | 5 | 15 | 14 |
| 2 | 7 | 38 | 42 |
| 2 | 8 | 20 | 24 |
| 2 | 9 | 17 | 14 |
| 2 | 2 | 14 | 12 |
| 2 | 3 | 22 | 17 |
| 3 | 4 | 39 | 50 |
| 3 | 5 | 37 | 42 |
| 3 | 6 | 15 | 17 |
| 3 | 9 | 13 | 11 |
| 3 | 1 | 14 | 14 |
| 4 | 5 | 28 | 32 |
| 4 | 9 | 19 | 15 |
| 5 | 6 | 21 | 29 |
| 5 | 7 | 37 | 42 |
| 6 | 5 | 18 | 20 |
| 6 | 7 | 24 | 30 |
| 7 | 6 | 14 | 13 |
| 8 | 5 | 20 | 23 |
| 8 | 6 | 21 | 16 |
| 8 | 7 | 21 | 21 |
| 8 | 8 | 15 | 10 |
| 8 | 0 | 16 | 13 |
| 9 | 5 | 21 | 20 |
| H | L | FO | FC |
| K=7 | | | |
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| 0 | 6 | 41 | 47 |
| 0 | 7 | 25 | 26 |
| 0 | 9 | 21 | 18 |
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| -1 | 9 | 12 | 12 |
| -1 | 0 | 13 | 15 |
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| -3 | 6 | 22 | 13 |
| -3 | 8 | 40 | 42 |
| -3 | 9 | 17 | 23 |
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| -4 | 5 | 13 | 13 |
| -4 | 6 | 37 | 35 |
| -4 | 7 | 30 | 26 |
| -4 | 0 | 23 | 23 |
| -4 | 1 | 14 | 12 |
| -5 | 5 | 29 | 29 |
| -5 | 6 | 23 | 22 |
| -5 | 8 | 18 | 17 |
| -5 | 0 | 31 | 29 |
| -6 | 5 | 21 | 21 |
| -6 | 6 | 47 | 47 |
| -6 | 9 | 14 | 15 |
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| -8 | 5 | 14 | 14 |
| -8 | 8 | 21 | 18 |
| -8 | 9 | 15 | 16 |
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| 1 | 9 | 12 | 12 |
| 1 | 0 | 35 | 39 |
| 2 | 4 | 65 | 69 |
| 2 | 5 | 15 | 10 |
| 2 | 6 | 14 | 13 |
| 2 | 7 | 24 | 25 |
| 2 | 8 | 31 | 33 |
| 2 | 9 | 12 | 12 |
| 2 | 0 | 23 | 24 |
| 3 | 7 | 16 | 15 |
| 3 | 8 | 12 | 10 |
| 3 | 0 | 14 | 10 |
| 3 | 2 | 15 | 12 |
| 4 | 4 | 34 | 43 |
| 4 | 5 | 10 | 10 |
| 4 | 7 | 21 | 23 |
| 4 | 8 | 18 | 20 |
| 4 | 9 | 14 | 13 |
| 5 | 6 | 44 | 52 |
| 5 | 8 | 24 | 25 |
| 5 | 9 | 20 | 21 |
| 6 | 6 | 36 | 37 |
| 7 | 6 | 14 | 13 |
| 7 | 8 | 31 | 29 |
| 8 | 5 | 21 | 19 |
| 8 | 8 | 16 | 19 |
| H | L | FO | FC |
| K=L | | | |
| 0 | 5 | 21 | 27 |
| 0 | 7 | 24 | 26 |
| 0 | 8 | 42 | 42 |
| 0 | 9 | 27 | 36 |
| 0 | 10 | 13 | 12 |
| 0 | 11 | 14 | 15 |
| 0 | 12 | 15 | 13 |
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| -1 | 8 | 8 | 9 |
| -1 | 10 | 9 | 8 |
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| -1 | 12 | 14 | 17 |
| -2 | 5 | 10 | 10 |
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| -3 | 11 | 14 | 18 |
| -4 | 7 | 26 | 26 |
| -4 | 8 | 32 | 31 |
| -4 | 10 | 24 | 21 |
| -5 | 5 | 34 | 28 |
| -5 | 6 | 19 | 20 |
| -5 | 8 | 22 | 19 |
| -5 | 10 | 11 | 7 |
| -6 | 5 | 11 | 10 |
| -6 | 6 | 29 | 29 |
| -6 | 8 | 20 | 18 |
| -6 | 9 | 38 | 37 |
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| -7 | 5 | 25 | 20 |
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| -7 | 11 | 12 | 14 |
| -8 | 5 | 26 | 19 |
| -8 | 6 | 35 | 31 |
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| -9 | 6 | 19 | 14 |
| -9 | 7 | 19 | 17 |
| -9 | 9 | 13 | 9 |
| -10 | 5 | 20 | 15 |
| 1 | 5 | 21 | 26 |
| 1 | 6 | 57 | 58 |
| 1 | 8 | 9 | 14 |
| 1 | 9 | 13 | 12 |
| 1 | 10 | 10 | 9 |
| 2 | 5 | 12 | 15 |
| 2 | 7 | 23 | 25 |
| 2 | 8 | 34 | 41 |
| 2 | 9 | 17 | 18 |
| 2 | 10 | 10 | 6 |
| 3 | 5 | 44 | 49 |
| 3 | 6 | 17 | 17 |
| 3 | 7 | 18 | 15 |
| 3 | 8 | 24 | 26 |
| 3 | 9 | 15 | 20 |
| 3 | 10 | 11 | 10 |
| 4 | 7 | 22 | 25 |
| 4 | 8 | 28 | 28 |
| 4 | 9 | 16 | 17 |
| 5 | 5 | 44 | 40 |
| 5 | 6 | 31 | 29 |
| 5 | 8 | 17 | 16 |
| 6 | 8 | 22 | 17 |
| 6 | 9 | 18 | 15 |
| 7 | 5 | 29 | 28 |
| 7 | 6 | 19 | 13 |
| 7 | 8 | 13 | 16 |
| 8 | 5 | 27 | 18 |
| 8 | 6 | 19 | 16 |
| 9 | 5 | 20 | 17 |
| 9 | 8 | 20 | 19 |
| 11 | 5 | 19 | 15 |

The absence of *h*0*l* reflections with $l = 2n+1$ and 0*k*0 reflections with $k = 2n+1$ indicated space group $P2_1/c$. Intensity data were collected on the precession camera, using the same crystal as used for the space group determination. Reflections were estimated visually from the *hkl* ($l = 0$–4), the *hkl* ($k = 0$–7), and *hkk* layers (approximately 70% of the theoretical number of reflections) and a total of 2163 unique non-zero reflections were obtained. Lorentz-polarization corrections were computed but no corrections were made for absorption ($\mu = 9.5\ \text{cm}^{-1}$).

*Solution and Refinement of the Structure*

Computations were carried out on the Univac 1108 computer; programs employed included modified versions of Zalkin's FORDAP Fourier Summation program, the Busing–Martin–Levy XFLS and ORFFE least-squares refinement and function and error analysis programs, Johnson's ORTEP program for crystal structure illustrations, and various locally written programs.

The coordinates of the iron atoms were determined from a three dimensional Patterson synthesis; after least-squares refinement of individual scale factors for each layer of data and the iron coordinates, a Fourier synthesis, phased on the iron atoms, revealed the positions of all non-hydrogen atoms.

A Fourier synthesis phased on these atoms showed definite anisotropic thermal motion of the heavy atoms; anisotropic thermal parameters were therefore assigned to the iron atoms and to all atoms coordinated to the iron. Further full-matrix least-squares refinement (minimizing $(|F_o|-|F_c|)^2$) of all parameters converged to a conventional $R_1$ value $(\Sigma|F_o|-|F_c||/\Sigma|F_o|)$ of 0.096. In the final cycle of refinement, there was no parameter shift greater than 1/10 of one estimated standard deviation. A final difference Fourier had no positive maxima greater than 0.5 e/Å$^3$.

In the structure factor calculations, the scattering factors tabulated by Ibers[9] were employed for all atoms.

The positional and thermal parameters derived from the last cycle of least-squares refinement are presented in Table I; the observed and calculated structure factors are given in Table II.

## Discussion

The structure consists of monomeric iron(III) complexes (Figure 1) with distorted octahedral coordination. Selected bond distances and bond angles are presented in Tables III and IV, respectively. The dianion functions as a pentadentate ligand, coordinating through the two phenolic oxygens, the two imine nitrogens, and one of the sulfur atoms of the disulfide group. In order to achieve this coordination, the ligand forms three six-membered chelate rings and one five-membered chelate ring. Two of the six-membered rings involve phenolic oxygens and imine nitrogens and each of these is a normal, planar ring with a delocalized pi-system similar to the chelate rings in salicylaldehyde and other salicylaldimine complexes. For one of these rings, the imine nitrogen (N1) is also included in the five-membered ring and the phenolic oxygen (O1) is trans to the sulfur (S1) which is involved in the five-membered ring. The second sulfur (S2) is 3.79 Å from the iron atom and is not considered coordinated. The remaining six-membered ring includes both the coordinated and uncoordinated sulfurs and the second imine nitrogen(N2); there is no multiple bonding in this ring and it is distinctly non-planar. The two imine nitrogens occupy *cis* positions and the remaining coordination position, which is *trans* to the second imine nitrogen, is occupied by a chloride ion.

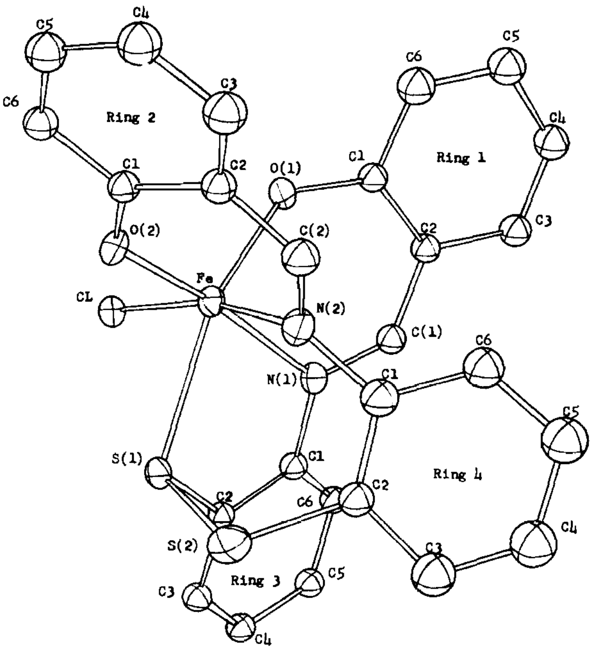

Figure 1. A perspective drawing of the molecular structure of chloro(bis-{*o*-salicylideneiminephenyl}disulfido)iron(III).

The bond angles at iron show considerable distortion from an ideal octahedron with ligand–iron–ligand angles between *cis*-positions varying from 77.8(3)° for the sulfur and nitrogen of the five-membered ring to 105.4(3)° for one of the chlorine–oxygen angles. The two Fe–O bond distances are only slightly different (1.87, 1.91 Å) and are similar to those found in other structures,[10–12] the two Fe–N bond distances (2.20 and 2.17 Å) show a similar difference and are slightly longer than the 2.10 Å found in other structures.[10–12] The sulfur–sulfur distance of 2.046(5) Å agrees well with the values found for other structures

TABLE III. Intramolecular Distances (Å) for $Fe(C_{26}H_{18}N_2S_2O_2)Cl$.

| Atoms | Distance | Atoms | Distance |
|---|---|---|---|
| (a) Distances from Iron Atom | | | |
| Fe–Cl | 2.314(4) | Fe–N(1) | 2.204(10) |
| Fe–O(1) | 1.871(8) | Fe–N(2) | 2.166(12) |
| Fe–O(2) | 1.909(7) | Fe–S(1) | 2.536(4) |
| | | Fe–S(2) | 3.798(4) |
| (b) Distances within Chelate Rings | | | |
| S(1)–S(2) | 2.046(5) | N(2)–R4C1 | 1.40(2) |
| N(1)–C(1) | 1.29(1) | C(1)–R1C2 | 1.43(2) |
| N(2)–C(2) | 1.37(2) | C(2)–R2C2 | 1.40(2) |
| O(1)–R1C1 | 1.32(1) | S(1)–R3C2 | 1.77(1) |
| O(2)–R2C1 | 1.34(1) | S(2)–R4C2 | 1.77(1) |
| N(1)–R3C1 | 1.42(2) | | |

(c) Distances within Phenyl Groups

| Atoms | Ring 1 | Ring 2 | Ring 3 | Ring 4 |
|---|---|---|---|---|
| C1–C2 | 1.44(2) | 1.44(2) | 1.40(1) | 1.39(2) |
| C2–C3 | 1.44(2) | 1.37(2) | 1.40(2) | 1.42(2) |
| C3–C4 | 1.36(2) | 1.38(2) | 1.36(2) | 1.36(3) |
| C4–C5 | 1.44(2) | 1.37(2) | 1.36(2) | 1.45(3) |
| C5–C6 | 1.42(2) | 1.40(2) | 1.43(3) | 1.42(3) |
| C6–C1 | 1.41(2) | 1.39(2) | 1.37(2) | 1.42(2) |

TABLE IV. Interatomic Angles (deg) for $Fe(C_{26}H_{18}N_2S_2O_2)Cl$.

| Atoms | Angle | Atoms | Angle |
|---|---|---|---|
| (a) Angles at Iron Atom | | | |
| Cl–Fe–O(1) | 105.4(3) | S(1)–Fe–O(1) | 163.9(3) |
| Cl–Fe–O(2) | 94.1(3) | S(1)–Fe–O(2) | 95.0(3) |
| Cl–Fe–S(1) | 83.0(1) | N(1)–Fe–N(2) | 90.7(4) |
| Cl–Fe–N(1) | 88.8(3) | N(1)–Fe–O(1) | 88.6(4) |
| Cl–Fe–N(2) | 165.7(3) | N(1)–Fe–O(2) | 172.0(4) |
| S(1)–Fe–N(1) | 77.8(3) | N(2)–Fe–O(1) | 88.9(4) |
| S(1)–Fe–N(2) | 82.9(3) | N(2)–Fe–O(2) | 84.7(4) |
| | | O(1)–Fe–O(2) | 97.9(4) |
| (b) Angles at Atoms of the Chelate Rings | | | |
| Fe–O(1)–R1C1 | 133.3(8) | C(1)–R1C2–R1C3 | 115.8(11) |
| Fe–O(2)–R2C1 | 129.8(7) | C(2)–R2C2–R2C1 | 124.5(12) |
| Fe–N(1)–C(1) | 120.2(9) | C(2)–R2C2–R2C3 | 118.1(13) |
| Fe–N(2)–C(2) | 120.3(11) | N(1)–R3C1–R3C2 | 118.4(13) |
| Fe–N(1)–R3C1 | 119.9(10) | N(1)–R3C1–R3C6 | 121.9(14) |
| Fe–N(2)–R4C1 | 121.8(9) | N(2)–R4C1–R4C2 | 120.0(13) |
| Fe–S(1)–S(2) | 111.5(2) | N(2)–R4C1–R4C6 | 121.5(14) |
| Fe–S(1)–R3C2 | 98.1(4) | S(1)–R3C2–R3C1 | 120.4(8) |
| O(1)–R1C1–R1C2 | 121.8(11) | S(1)–R3C2–R3C3 | 119.2(8) |
| O(1)–R1C1–R1C6 | 119.3(12) | S(2)–R4C2–R4C1 | 121.5(11) |
| O(2)–R2C1–R2C2 | 121.7(11) | S(2)–R4C2–R4C3 | 115.5(12) |
| O(2)–R2C1–R2C6 | 119.0(11) | S(1)–S(2)–R4C2 | 104.2(5) |
| C(1)–R1C2–R1C1 | 123.3(11) | | |

TABLE IV. (Cont.)

(c) Angles within Phenyl Groups

| Atoms | Ring 1 | Ring 2 | Ring 3 | Ring 4 |
|---|---|---|---|---|
| C6–Cl–C2 | 118.8(11) | 119.3(11) | 118.7(10) | 118.3(14) |
| Cl–C2–C3 | 120.9(11) | 117.0(15) | 120.4(10) | 122.6(14) |
| C2–C3–C4 | 118.5(12) | 125.4(15) | 120.2(11) | 117.6(17) |
| C3–C4–C5 | 122.9(14) | 116.7(15) | 120.7(12) | 123.5(19) |
| C4–C5–C6 | 118.1(5) | 122.2(14) | 119.8(11) | 116.4(18) |
| C5–C6–C1 | 120.7(15) | 119.4(12) | 120.0(10) | 120.3(16) |

(2.03–2.05 Å).[3,4,7] Bond distances and bond angles obtained for the rest of the ligand are also normal.

The magnetic moment of the compound, 5.90 B.M., is a normal value for high-spin iron(III).

## Acknowledgements

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