Crystal Structure of Diphenyl Pyraline Tetrachloroferrate

Neratur K. LOKANATH,* Sridhar M. ANANDALWAR,* Javaregowda Shashidhara PRASAD,*[†]
and Puttaramappa G. RAMAPPA**

*Department of Studies in Physics, University of Mysore, Manasagangotri, Mysore 570 006, India

**Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India

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The metal complexes of diphenyl pyraline hydrochloride (DPH), an anti-histamine, were found to have a higher drug activity than DPH. To achieve a better understanding of the nature of the drug reaction, we have undertaken some crystal structure studies of the metal complexes of diphenyl pyraline hydrochloride. In this article, we report the structural details of diphenyl pyraline tetrachloroferrate complex (DPTC). The schematic diagram of DPTC is given in Fig. 1. The complex was synthesized by mixing a hot ethanolic solution of ferric chloride with diphenyl pyraline hydrochloride (DPH). The solid which formed under slow evaporation was filtered and washed in ethanol and then dried completely in vacuum. Good single crystals were obtained from aqueous ethanol solution by

slow evaporation.

Hydrogen atoms were placed at chemically acceptable positions. The coordinates of the hydrogen atoms ride on those of the parent atoms. Thermal ellipsoids of the molecule at the 50% probability level are given in Fig. 2. Crystal and experimental data are listed in Table 1. Table 2 gives the fractional atomic coordinates and equivalent thermal parameters for all the refined atoms. Tables 3 and 4 give the bond distances and angles of non-hydrogen atoms. The packing diagrams of the molecules show independent stacking of the ligands and the metal groups. The bond lengths and angles are in good agreement with those of similar complexes.¹ The stacking of the ligand and the metal explains the semiconducting nature of the compound.² The pyridine ring has chair conformation. The molecule exhibits intermolecular hydrogen bonds of the type CH...O and CH...Cl. The intermolecular hydrogen bonds between atoms are given below: C5-H5...Cl2 has length

Fig. 1 Schematic diagram of DPTC.

Table 1 Crystal and experimental data

Empirical formula: C ₁₉ H ₂₅ Cl ₂ Fe _{0.5} NO
Formula weight: 380.21
Temperature: 293(2)K
Crystal system: monoclinic
Space group: <i>P</i> 2 ₁ / <i>n</i> <i>Z</i> =4
<i>a</i> = 8.745(1)Å
<i>b</i> = 14.699(2)Å <i>β</i> = 90.47(2)°
<i>c</i> = 14.605(1)Å
<i>V</i> = 1877.3(4)Å ³
<i>D_x</i> = 1.345 Mg/m ³
<i>R</i> 1 = 0.0557
No. of reflections used = 3307
Measurement: Rigaku AFC7S
Program system: teXsan
Structure determination: DIRDIF-97
Refinement: full matrix

[†] To whom correspondence should be addressed.

E-mail: mysxrd@giasbg01.vsnl.net.in

Table 2 Atomic coordinates and equivalent thermal parameters of the non-hydrogen atoms

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Fe1	0.0000	0.0000	0.0000	0.0319(2)
Cl1	-0.0135(2)	0.0499(1)	-0.1461(8)	0.0836(5)
Cl2	0.1945(2)	0.0962(1)	0.0285(1)	0.1038(7)
C1	0.2132(8)	0.4652(4)	0.9360(4)	0.0823(2)
C2	0.3159(7)	0.4654(4)	0.8650(4)	0.0750(2)
C3	0.3398(5)	0.3878(3)	0.8127(4)	0.0612(1)
C4	0.2622(4)	0.3081(3)	0.8336(3)	0.0476(9)
C5	0.1587(5)	0.3088(4)	0.9045(3)	0.0628(1)
C6	0.1330(7)	0.3864(4)	0.9550(3)	0.0781(2)
C7	0.2882(4)	0.2222(3)	0.7780(3)	0.0460(9)
C8	0.2280(4)	0.2313(2)	0.6798(3)	0.0466(9)
C9	0.0782(5)	0.2050(3)	0.6601(3)	0.0538(1)
C10	0.0183(6)	0.2138(3)	0.5728(4)	0.0680(1)
C11	0.1071(7)	0.2495(4)	0.5033(4)	0.0781(2)
C12	0.2569(7)	0.2760(4)	0.5230(4)	0.0780(2)
C13	0.3154(6)	0.2671(4)	0.6105(3)	0.0660(1)
O14	0.4499(3)	0.2051(2)	0.7800(2)	0.0489(7)
C15	0.4872(4)	0.1143(3)	0.7550(3)	0.0468(9)
C16	0.6500(5)	0.1133(3)	0.7214(3)	0.0534(1)
C17	0.6979(5)	0.0186(3)	0.6953(3)	0.0578(1)
N18	0.6795(4)	-0.0467(2)	0.7726(2)	0.0483(8)
C19	0.5201(5)	-0.0462(3)	0.8077(3)	0.0529(1)
C20	0.4728(5)	0.0485(3)	0.8359(3)	0.0546(1)
C21	0.7239(6)	-0.1410(3)	0.7434(4)	0.0721(1)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* (a_i a_j).$$

Fig. 2 ORTEP with numbering scheme of DPTC at 50% probability.

3.624(1) Å and angle 130(3)° with symmetry code $x, y, 1 + z$, C9-H9...Cl1 has length 3.727(5) Å and angle 151(3)° with symmetry code $x, y, 1 + z$ and C11-H11...O14 has length 3.591(4) Å and angle 170(4)° with symmetry code $-1/2 + x, 1/2 - y, -1/2 + z$.

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Table 3 Bond lengths (Å)

Atoms	Length	Atoms	Length
Fe1-Cl2	2.249(1)	C9-C10	1.381(7)
Fe1-Cl1	2.258(1)	C10-C11	1.386(8)
C1-C2	1.377(9)	C11-C12	1.395(8)
C1-C6	1.384(9)	C12-C13	1.379(7)
C2-C3	1.389(7)	O14-C15	1.422(4)
C3-C4	1.389(6)	C15-C16	1.509(5)
C4-C5	1.381(6)	C15-C20	1.533(5)
C4-C7	1.520(6)	C16-C17	1.505(6)
C5-C6	1.378(7)	C17-N18	1.491(6)
C7-O14	1.437(4)	N18-C19	1.490(5)
C7-C8	1.529(5)	N18-C21	1.503(5)
C8-C13	1.378(6)	C19-C20	1.510(6)
C8-C9	1.393(6)		

Table 4 Bond angles (°)

Atoms	Angle	Atoms	Angle
Cl2-Fe1-Cl1	90.24(5)	C10-C9-C8	121.0(5)
Cl2-Fe1-Cl1	89.76(5)	C9-C10-C11	120.0(5)
C2-C1-C6	119.2(5)	C10-C11-C12	119.1(5)
C1-C2-C3	121.0(6)	C13-C12-C11	120.3(5)
C2-C3-C4	119.7(5)	C8-C13-C12	120.9(5)
C5-C4-C3	118.9(4)	C15-O14-C7	112.8(3)
C5-C4-C7	120.5(4)	O14-C15-C16	108.1(3)
C3-C4-C7	120.5(4)	O14-C15-C20	112.0(3)
C6-C5-C4	121.2(5)	C16-C15-C20	109.2(3)
C5-C6-C1	120.0(5)	C17-C16-C15	110.9(3)
O14-C7-C4	106.6(3)	N18-C17-C16	111.8(4)
O14-C7-C8	111.5(3)	C19-N18-C17	111.4(3)
C4-C7-C8	112.1(3)	C19-N18-C21	110.3(3)
C13-C8-C9	118.7(4)	C17-N18-C21	110.4(4)
C13-C8-C7	122.2(4)	N18-C19-C20	111.0(3)
C9-C8-C7	119.1(4)	C19-C20-C15	110.3(4)

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