Asian Journal of Chemistry; Vol. 25, No. 6 (2013), 3515-3516

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2013.13747

NOTE

Synthesis and Crystal Structure of *tris*-(1,10-Phenanthroline)-Iron(II) Dichromate Tetrahydrate, $Fe(C_{12}H_8N_2)_3Cr_2O_7{\cdot}4H_2O$

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(*Received*: 17 March 2012; *Accepted*: 29 December 2012) AJC-12632

In this report, $Fe(C_{12}H_8N_2)_3Cr_2O_7{\cdot}4H_2O$, was prepared by the reaction of $FeCl_2$, 1,10-phenanthroline and potassium chromate. The asymmetric unit is composed of a $[Fe(phen)_3]^{2+}$ cation, a $Cr_2O_7^{2-}$ anion and four water molecules. The metal centre is coordinated in a distorted octahedral mode by six N atoms from three 1,10-phenanthroline. The $Cr_2O_7^{2-}$ anion is in a staggered conformation. The crystal packing is stabilized by intermolecular O-H...O and O-H...N hydrogen bonds and π-π interaction.

Key Words: Crystal structure, $Fe(C_{12}H_8N_2)_3Cr_2O_7{\cdot}4H_2O$.

Metal complexes containing diimine ligands such as 1,10-phenanthroline and bipyridine are very important and widely used in analytical chemistry, catalysis, electrochemistry, ring-opening metathesis polymerization and biochemistry[1-5]. 1,10-Phenanthroline, which is the parent for important class chelating agents, has been widely used in the construction of supramolecular architectures. Lots of phenanthroline complexes have been synthesized and reported[6-9]. Chromate (VI) complexes have been known as causing genotoxic and mutagenic effects in living cells, leading to development of cancer in humans. Considering the redox pathways of the carcinogenic Cr(VI) anion and the interaction of its metabolites Cr(V), Cr(IV) and Cr(III) with DNA, the lowering of its mutagenic activity has been explained by the mode of the chromate ion binding to the metal-organic ligand core[9].

All commercially obtained reagent-grade chemicals were used without further purication. A mixture of $FeCl_2$ (0.1 mmol, 0.02 g), phenanthroline (0.1 mmol, 0.02 g), K_2CrO_4 (0.1 mmol, 0.02 g) and boric acid were added into 25 mL water with 20 % (v/v) methanol and heated for 8 h at 353 K. The solution was obtained by filtration after cooling the reaction to room temperature. Red sheet single crystals suitable for X-ray measurements were obtained after a few days.

The crystal structure of $Fe(C_{12}H_8N_2)_3Cr_2O_7{\cdot}4H_2O$ (Fig. 1) is build up of one $[Fe(phen)_3]^{2+}$ cation, one dichromate anion and four dissociative water molecules. The packing diagram of $Fe(C_{12}H_8N_2)_3Cr_2O_7{\cdot}4H_2O$ is shown in Fig. 2. The crystal data and structure refinement is shown in Table-1. The Cr atom is coordinated by four O atoms forming an tetrahedron. The bond lengths of Cr-O are in the range of 1.615-1.800 Å. The torsion angles of O4-Cr1-O1-Cr2, O7-Cr2-O1-Cr1 are 18.9(6)º and 85.2(6)º, respectively. The Fe atom is coordinated by six nitrogen atoms from three 1,10-phenanthroline molecules. Six Fe-N distances range from 2.008(6) Å to 2.023(6) Å. Some bond lengths and angle are shown in Table-2.

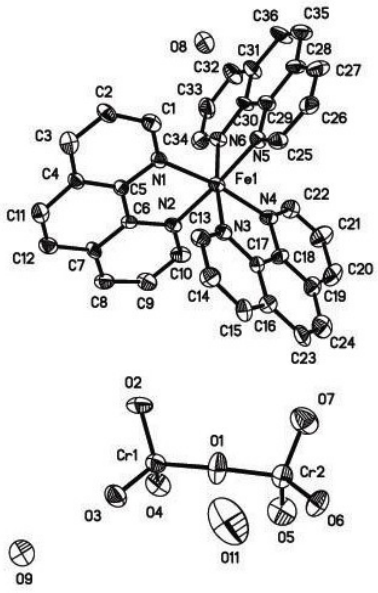

Fig. 1. Molecular structure of $Fe(C_{12}H_8N_2)_3Cr_2O_7{\cdot}4H_2O$ with atom-labling scheme

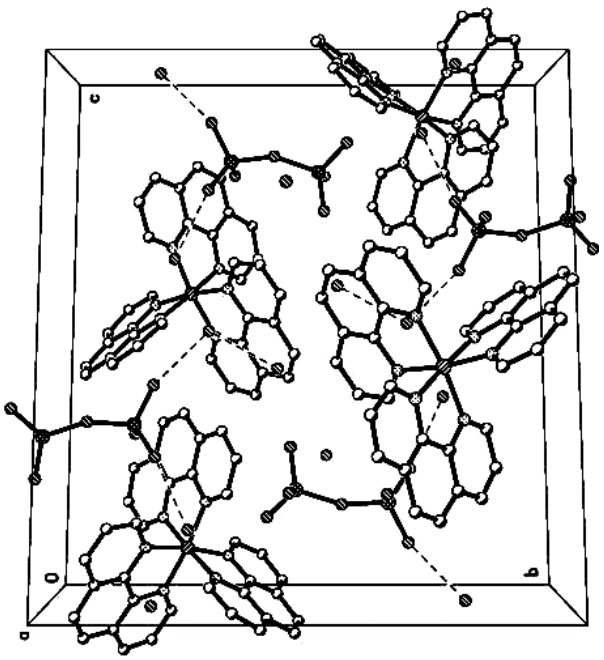

Fig. 2. Packing diagram of the title complex

TABLE-1
CRYSTAL DATA AND STRUCTURE REFINEMENT FOR 081031f

Identification code	081031f
Empirical formula	$C_{36}H_{32}N_6O_{11}Cr_2Fe$
Formula weight	884.53
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 10.4062(17)Å; α = 90°
	b = 18.457(2) Å; β =103.758(2)°
	c = 20.227(3) Å; γ = 90°
Volume	3773.4(9) $Å^3$
Z, Calculated density	4, 1.557 Mg/m^3
Absorption coefficient	1.013 mm^{-1}
F(000)	1808
Crystal size	0.30 × 0.27 × 0.05 mm
Theta range for data collection	1.51 to 25.01°
Limiting indices	-8 <= h <= 12, -21 <= k <= 21, -24 <= l <= 19
Reflections collected/unique	19020 / 6645 [R(int) = 0.1758]
Completeness to θ = 25.01	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9511 and 0.7508
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6645 / 0 / 505
Goodness-of-fit on F^2	0.864
Final R indices [I>2σ(I)]	R1 = 0.0673, wR2 = 0.1173
R indices (all data)	R1 = 0.1920, wR2 = 0.1525
Largest diff. peak and hole	0.423 and -0.453 e. $Å^{-3}$

TABLE-2
SOME BOND LENGTHS (Å) AND ANGLES (º) FOR 081031f

Cr(1)-O(4)	1.615(6)
Cr(1)-O(1)	1.793(5)
Cr(2)-O(5)	1.627(6)
Cr(2)-O(1)	1.800(5)
Fe(1)-N(1)	2.008(6)
Fe(1)-N(2)	2.021(6)
N(1)-C(1)	1.337(8)
N(2)-C(10)	1.348(8)
N(3)-C(13)	1.340(8)
N(4)-C(18)	1.389(8)
N(5)-C(29)	1.375(8)
N(6)-C(34)	1.334(8)
O(8)-H(8C)	0.8501
O(9)-H(9C)	0.8501
C(1)-C(2)	1.414(9)
C(8)-H(8)	0.9300
O(4)-Cr(1)-O(2)	109.7(3)
O(4)-Cr(1)-O(3)	110.5(3)
O(2)-Cr(1)-O(1)	108.5(3)
O(3)-Cr(1)-O(1)	107.7(3)
O(5)-Cr(2)-O(6)	110.0(3)
O(7)-Cr(2)-O(1)	108.2(3)
N(1)-Fe(1)-N(6)	93.8(2)
N(1)-Fe(1)-N(2)	82.3(2)
N(2)-Fe(1)-N(3)	89.9(2)
N(6)-Fe(1)-N(4)	92.3(2)
C(1)-N(1)-C(5)	116.6(6)
C(1)-N(1)-Fe(1)	130.7(5)
C(5)-N(1)-Fe(1)	112.4(5)
C(6)-N(2)-Fe(1)	111.6(5)
C(17)-N(3)-Fe(1)	112.1(4)

Crystal packing is stabilized by O-H...O and O-H...N hydrogen bonds and π- π interaction

ACKNOWLEDGEMENTS

This study were supported by the Natural Science Foundation of Shandong Province (No. ZR2010BL025), State Key Laboratory of Inorganic Synthesis and Preparative Chemistry (Jilin University) (No. 2011-13) and MOE Key Laboratory of Analytical Chemistry for Life Science (Nanjing University) (No. KLACLS1002) and the National Science Foundation of China (No. 21171132).

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