

# Synthesis and Structural Investigation of Co-Ordination Compound of Chromium I with Uracil, Uracil -4 - Carboxylic Acid

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**Abstract:** The investigation on mixed-ligand cyanonitrosyl complexes of chromium in its unusual oxidation state, from-II to +VI among the VI group transition elements. The physico-chemical properties and elucidation of structural aspects of the synthesized compounds have been characterized by different physical methods i.e., conductance, magnetic and Infrared spectral measurement C,H,N and S were estimated microanalytically, chromium estimated as chromic oxide (CrO<sub>3</sub>). Co-ordination compounds of Uracil and their substituted of CrI are synthesized in this paper.

**Keywords:** Ligands, Transition elements, Cyanonitrosyl, chromic oxide, Uracil

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## GLOSSARY OF SYMBOLES

- NO – Nitric oxide
- DMSO – Dimethyl sulphoxide
- DMF – Dimethyl formamide
- L – ligand
- NOS – Nitric Oxide synthase
- NMR – Nuclear magnetic resonance
- Ur1 – Uracil
- Ur1-4CA – Uracil 4 -Carboxylic Acid

## I. Introduction

Co-ordination compounds of Uracil and their substituted of chromium (I) are synthesized and characterized in this paper. Complex, monovalent chromium are very rare<sup>(1-4)</sup>, A survey of the literature of neutral mixed ligands nitrosyl complexes of Cr (I) reveals few reports on such compounds<sup>(4,8,10)</sup> Although considerable importance is involve in the study of mixed ligand cyano nitrosyl of chromium, cyanonitrosyl complexes of chromium {CrNO<sup>5</sup>} having {CrNO<sup>5</sup>} electronic configuration with Uracil, Uracil -4-carboxylic acid and have been rarely described. We therefore, report here the first synthesis of neutral mixed ligand cyanonitrosyl complexes of chromium with uracil and uracil-4-carboxylic acid. The structure is shown in fig. 1

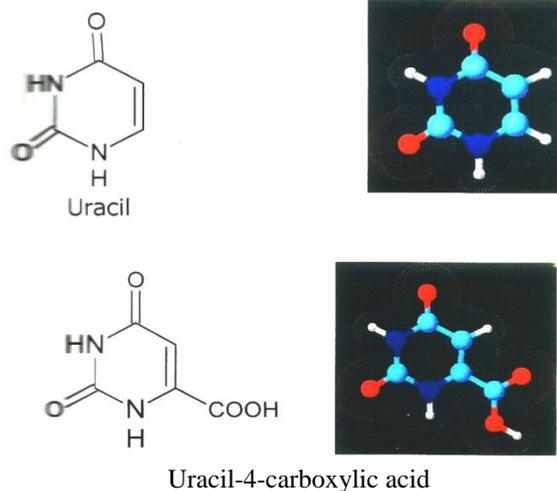


Fig. 1

## II. Structure And Physical Properties

In solid state, uracil exists as the dioxotautomer, which has been shown with the aid of refined X-ray analyses from which the position of hydrogen atom were directly determined. The following list shows some parameters for the monoclinic cell.

$$a = 11.938 \pm 0.001 \text{ \AA}$$

$$b = 12.376 \pm 0.009$$

$$c = 3.6552' \pm 0.003$$

$$\beta = 120.054' \pm 0.4'$$

$$\rho(\text{MoK}\alpha) = 0.71069 \text{ \AA}^{-3}$$

This dioxo form is further supported by other spectroscopic data for instance, UV<sup>(4)</sup> and Raman spectroscopy<sup>(4)</sup> indicated that the same dioxo tautomer predominates in solution.

As the chemical shift of C-5 and C-6<sup>13</sup>C-NMR spectroscopy reveal the 5,6 double bond is highly polarized as expected for a heterocyclic enamino carboxyl compound.

## III. Naturally Occurring Uracil; Uracil as Active Principles

Uracil have represented for more than 90 years, A class of compounds that continually attract organic chemists, bio chemists, medicinal chemists and photobiologists. Uracils were first detected as constituents of ribonucleic acid from which they were prepared by hydrolysis. Uracil can also be generated from cytosine by oxidative deamination using sodium hydrogensulfite. Several uracil derivative have been developed as drugs.

Naturally occurring hetero condensed uracil derivatives are methylxanthines, e.g. caffeine<sup>(5)</sup> theophylline<sup>(5)</sup> and theobromine<sup>(5)</sup> (Vitamin B<sub>2</sub>) acts as a coenzyme in bio-redox reactions<sup>(6)</sup>.

## IV. Uracil Synthesis

The classical and primary synthesis route to uracil from formal acetic acid and urea in sulphuric acid is still important<sup>(7)</sup>. Some alternative syntheses use malic acid, urea and PPA<sup>(8)</sup>. The reaction of formylacetate with thio urea is convenient for the synthesis of 2-thiouracil. Another main synthesis involves the reaction of urea with keto ester, di ketone or acid anhydrides<sup>(9)</sup>. A broad choice of heterocondensed uracil are easily and generally accessible from heterocyclic  $\beta$ -enamino esters and isocyanates.

## V. Experimental

We use Hydroxylaminehydrochloride (BDH, England), Potassium Cyanide (**May and Baker**), chromic acid were used as such. Uracil, Uracil-4-carboxylic acid were used as supplied. Distilled water was used in all the operations.

Carbon, Hydrogen, Nitrogen and Sulphur present in the synthesized complex were estimated micro analytically. Then for the estimation of the chromium as chromic oxide (Cr<sub>2</sub>O<sub>3</sub>), the compounds were decomposed by heating with alkali followed by dissolving in nitric acid. Chromium was precipitated as chromic hydroxide by means of dil. ammonium hydroxide, chromic hydroxide, when ignited, was converted in to Cr<sub>2</sub>O<sub>3</sub>. Repeated heating, cooling and weighing were carried out unit constant weight obtained. Then we do measure conductance measurements, Magnetic susceptibility, Infrared measurement, NMR and molecular weight. The conductance were measured in analytical grade dimethyl sulphoxide (DMSO) and di methyl formamide (DMF) using dip type cell on Toshniwal conductivity Bridge at the department of Chemistry, Atarra P.G. College, Atarra. At room temperature, Magnetic susceptibility measurement of the investigated complexes were made by Gouy method. And IR spectra (4000-450 Cm<sup>-1</sup>) of the unco-ordinated ligands and synthesized complexes were recorded in nujol mulls supported between KBr pellets on Perkin Elmer (RXI) Spectrometer (at sophisticated analytical instrument facility, Central Drug Research Institute, Lucknow) then NMR spectra of unco-ordinated ligands and synthesized complexes were recorded on Bruker DRX-300 Mz FTNMR using DMSO as solvent and molecular weight determined by Rast's method.

Then we prepared the parent compound potassium pentacyanonitrocylic chromate (1) mono di hydrate by the method reported by **Wilkinson et. al.**

## VI. Preparation of Complexes

- (A) Preparation of [Cr(NO)(CN)<sub>2</sub>(Ura)<sub>2</sub>H<sub>2</sub>O] A filtered acidified aqueous solution of (50 ml.) Potassium salt of pentacyanonitrocylic chromate(1) monohydrate (.1 mg.) was added into an aq. alcohol solution (1:1 10 ml) of uracil ligand (0.02M). The resulting greenish-brown solution was obtained.
- (B) Preparation of [CrNo(CN)<sub>2</sub>(Ura-4-CA)<sub>2</sub>(H<sub>2</sub>O)] A filtered aq. solution (50 ml) of K salt of the pentacyanonitrocylic chromate (I) monohydrate (0.1M) was added into an aq. alcoholic solution (1:1 10ml) of the uracil-4-carboxylic and ligand (0.02M). The resulting light brownish solution obtained.

The I.U.P.A.C. Name of complexes are given table 1.1

**Table 1.1**

S.N.	Compound	I.U.P.A.C. Name	Electron Configuration
1.	[Cr(NO)(CN) <sub>2</sub> (Uril) <sub>2</sub> (H <sub>2</sub> O)]	Aquadicyano di (Uracil)-nitrosyl chromium (I)	{CrNO} <sup>5</sup>
2.	[Cr(NO)(CN) <sub>2</sub> (Uril4-CA) <sub>2</sub> (H <sub>2</sub> O)]	Aquadicyanodi (Uracil)-4- carboxylicacid)-nitrosyl chromium (1)	{CrNO} <sup>5</sup>

All the complexes are coloured solid; they are stable in air as shown in table 1.2

**Table 1.2**

S.N.	Compound	Colour	Decomposition Temperature (°C)	% yield
1.	[Cr(NO)(CN) <sub>2</sub> (Uril) <sub>2</sub> (H <sub>2</sub> O)]	Greenish Brown	310	56
2.	[Cr(NO)(CN) <sub>2</sub> (Uril 4-CA) <sub>2</sub> H <sub>2</sub> O	Light Brown	310	54

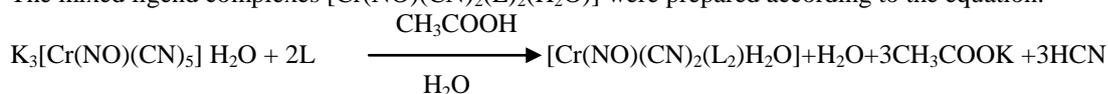
All complexes dehydration temperature and molecular weight are given in table 1.3

**Table 1.3**

S.N.	Compound	Dehydration temperature	Molecular weight	
			Found	Calculated
1.	[Cr(NO)(CN) <sub>2</sub> (Uril) <sub>2</sub> (H <sub>2</sub> O)]	112	376.02	378.20
2.	[Cr(NO)(CN) <sub>2</sub> (Uril4-CA) <sub>2</sub> (H <sub>2</sub> O)]	115	464.03	466.22

## VII. Result and Discussion

The mixed ligend complexes [Cr(NO)(CN)<sub>2</sub>(L)<sub>2</sub>(H<sub>2</sub>O)] were prepared according to the equation.



Where L = Uracil, Uracil-4-Carboxylic acid.

The partial replacement of the cyano group in the hexa-co-ordinated parent complex K<sub>3</sub>[Cr(NO)(CN)<sub>5</sub>·H<sub>2</sub>O] by two molecules of ligands arises from the trans effect of NO group. The molar conductance value measured in 10<sup>-3</sup> M dimethyl sulphoxide as well as in dimethyl formamide solution.

A comparison of the infrared spectra of the parent compound K<sub>3</sub>[Cr(NO)(CN)<sub>5</sub>·H<sub>2</sub>O] and of the synthesized complexes suggest that the appearance of the very strong bond in the region 1700-1705 Cm<sup>-1</sup> in these complexes, is of co-ordinated NO<sup>+</sup> stretching. Both the synthesized compounds reported here show a strong band in the region 2140-2160 Cm<sup>-1</sup>. The ligand uracil possesses 3 possible donor sites; two cyclic nitrogen and one Ketonic group in the ring respectively. Since all these complexes show CN stretching bond and one NO stretching bond it is reasonable to propose an octahedral structure<sup>11</sup> where CN is trans to CN and L ligands is trans to L and NO is trans to water is proposed for all the complexes.

It is observed that -

- (i) All the complexes are air stable coloured solids.
- (ii) They are soluble in DMF, DMSO, ethanol and methanol but insoluble in nitrobenzene and ethyl acetate.
- (iii) All the complexes contain {CrNO}<sup>5</sup> electronic configuration.

## References

- [1]. C.M. Lukehart and J.N. Troup, Inorg-Chem. Acta., 22,1977.
- [2]. S. Sarkar, R.C. Maurya and S.C. Chaurasia, India J. of Chem, 14.
- [3]. R.C. Maurya, R. Shukla, D.C. Gupta, N. Anandam and W.V. Malik Synthesis React Inorganic, Met-Org-Chem.
- [4]. Carmackeral. J. Amchem. Soc. (1949).
- [5]. P.T. Manoharan and H.B. Greg, Inorg. Chem., 5, 823, 1986.
- [6]. R. Silaghi-Dumitrescu, Eur. J. Inorg. Chem., 1048.
- [7]. U.P. Sing, E. Obayashi, S. Takahashi, T. Iizuka, H. Shoun, and Y. Shiro, Bio chem. BioPhys. Acta, 103, 1348 (1998).
- [8]. D.A. Scherlis, C.B. Cymeryng and D.A. Estrin, Inorg. Chem. 39, 2352 (2000).
- [9]. NORTHEY, Chem. Rev., 27,85 Eng. Chem., 35, 829 (1943).
- [10]. P.P. Desai and K.R. Desai, J. Indian Chem. Soc., 71, 153 (1994).
- [11]. Cohen Yamaski (2003) and Ker et al.
- [12]. Martins, Zila; Botta, Oliver; Fogel, Marilyn L; Sephton, Ehrenfreund, Pascale (15 June 2008).

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