

D-Glucuronic acid itself is not reduced at the d.m.e. but in aqueous solution it is converted into D-glucuro-6,3-lactone^{3,5} which is reduced in two steps. Limiting currents for both the lactone waves have been found to be linearly dependent on the concentration of D-glucuronic acid and hence can be utilized for analytical determination. *pH* of the medium has a marked effect on the magnitudes of the waves. Wave-heights decrease rapidly with increasing *pH* of the medium. Two factors may be responsible for this behaviour. Firstly, hydrogen ions are involved in the reduction process. And secondly, in alkaline medium, D-glucuronic acid largely forms D-glucuronate and not reducible D-glucuronolactone.

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Bis(acetylacetonato)nitrosyl Cr(I)

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A monomeric neutral Cr(I) complex, $[\text{Cr}(\text{acac})_2\text{NO}]$ has been prepared by the reaction of potassium pentacyanonitrosylchromate (I) monohydrate with excess of acetylacetone in the absence of air. The complex shows a magnetic moment of 1.72 B.M. and *g* value of 1.982 consistent with a low-spin d^5 configuration of Cr (I). Its IR spectrum exhibits a very strong band at 1692 cm^{-1} which is characteristic of νNO^+ . On the basis of IR and electronic spectral data, a square-pyramidal structure is proposed for the complex.

AFTER the isolation of potassium salt of nitrosyl-pentacyanochromate (I)¹, several other cationic nitrosyl complexes of hexacoordinated Cr(I)^{2,3} have been reported. However, neutral complexes of nitrosyl Cr(I) have not been reported so far. In this note we report the preparation and properties of bis(acetylacetonato)nitrosyl Cr(I), a neutral complex having comparable stoichiometry to that of $[\text{Co}(\text{acac})_2\text{NO}]$ but containing NO^+ group in contrast to NO^- group present in the cobalt complex⁴.

The complex was prepared by refluxing a mixture of $\text{K}_3[\text{Cr}(\text{CN})_5\text{NO}] \cdot \text{H}_2\text{O}$ (ref. 1) and excess acetylacetone in an air-tight compartment with an intermittent removal of the liberated HCN by passing dry CO_2 . After refluxing for 8 hr, the excess acetylacetone was distilled off and the resultant mixture was cooled *in vacuo* over solid KOH. Traces of

free acetylacetone were removed from the cooled brown mass by repeated washing with petroleum ether and the brown crust was extracted with acetone. On evaporation of acetone, the solution gave brown crystals which were washed with petroleum ether. The crystallization process was repeated thrice. The crystal thus obtained were dried and analysed [Found: Cr, 18.6; N, 5.0; C, 42.9; H, 8.32; calc. for $\text{CrNO}(\text{acac})_2$: Cr, 18.6; N, 5.0; C, 42.9; H, 8.23%]; m.p. 206° ; mol. wt 300 (Rast).

This non-electrolytic monomeric complex ($\Lambda_M = 13.2\ \Omega^{-1}\text{ cm}^2\text{ mole}^{-1}$ in acetone) is soluble in most of the common non-polar organic solvents but insoluble in water. Its stability is comparable to that of $\text{Cr}(\text{acac})_3$ as it remains unaffected by concentrated alkalis or mineral acids in cold and decomposes only on prolonged boiling. The room temperature magnetic moment (1.72 B.M.) is consistent with a low spin d^5 configuration ($g = 1.982$). Its IR spectrum shows a very strong band at 1692 cm^{-1} characteristic of $\nu(\text{NO}^+)$ (ref. 5). The low energy vibrations in the range $700\text{--}385\text{ cm}^{-1}$ consist of three envelopes. The first one contains sharp bands at 675, 654 and 640 cm^{-1} , the second at 610 and 592 cm^{-1} and the third one at 454 and 412 cm^{-1} respectively. The nature of the first and third envelopes are identical to that observed by Mikami and coworkers⁶ for several square-planar acetylacetonato complexes of bivalent metals. The bands in second envelope, then, should be due to Cr-N stretching and Cr-N-O bending vibrations respectively as reported by Miki⁷. The solid state and solution (ethanol) electronic spectra give four absorptions at identical positions, viz. 18.18 (32), 25.38 (135), 29.65 (3120), and 39.22 (1900) (peak positions in kK and molar extinction coefficients in parentheses) suggesting that solvation does not take place at the sixth position. On the basis of above data, a square-pyramidal structure may be proposed for the complex. First two of the above electronic transitions are believed to be of ligand field origin while the third and fourth peaks can be assigned to strong $\pi\text{--}\pi^*$ transitions of the complexed acetylacetone moiety^{8,9}.

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