# ANALYTICAL APPLICATIONS OF SOME SUBSTITUTED XANTHATES

By

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# C E R T I F I C A T E

This is to certify that the thesis entitled, "Analytical Applications of Some Substituted Xanthates", being submitted by Mr. Md. Farid Hussain to the Indian Institute of Technology, New Delhi, for the award of the degree of Doctor of Philosophy, is a record of bonafide research work carried out by him. Mr. Md. Farid Hussain has worked under our guidance and supervision and has fulfilled the requirements which to our knowledge have reached the requisite standard for the submission of this thesis.

The results contained in this thesis have not been submitted in part or full to any other University or Institute for the award of any degree or diploma.

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# A C K N O W L E D G E M E N T S

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Md Farid Hussain

( Md. Farid Hussain )

# S U M M A R Y

The thesis consists of six chapters. Each chapter has been subdivided into various sections dealing with introduction, experimental, and results and discussion.

The first chapter deals with the general introduction of organic reagents with special reference to the chemistry of xanthates, their methods of preparation,purification, properties, and analytical applications. Besides these, the utilization of metal chelates in inorganic analysis, and the methods of determining the composition of metal complexes have also been discussed. A brief introduction to the new techniques of extraction, that is, 'Solid-liquid Separation After Liquid-liquid Extraction' and 'Separation of Metals by Coprecipitation of Their Chelates, on to Microcrystalline Naphthalene' has been discussed. The scope of the present work has also been included.

The second chapter deals with the spectrophotometric determination of metal trifluoroethylxanthates. Conditions have been developed for the trace determination of Co, Ni, Cu, Pd and Au in certain standard reference materials and synthetic samples. The composition of the metal complex has been established.

The coprecipitation behaviour of metal trifluoroethylxanthates on to microcrystalline naphthalene has been studied in the third chapter. Conditions have been developed for the spectrophotometric determination of Co, Ni, Cu, Mo and Te as their trifluoroethylxanthates in certain standard reference materials, and synthetic samples. Since zinc, antimony and lead form colourless trifluoroethylxanthates, therefore, these metals have been determined by atomic absorption spectrophotometric method after dissolution of the metal complex-naphthalene mixture in dimethylformamide. The method has been tested for standard reference materials. An indirect spectrophotometric method has been developed for the trace determination of vanadium and indium after coprecipitation of their trifluoroethylxanthates on to microcrystalline naphthalene by replacement with copper. The composition of each metal complex coprecipitated on to naphthalene has been established. The determination of these metals has been carried out in certain standard reference materials and synthetic samples.

In the fourth chapter, extraction spectrophotometric determination of U(VI) and Fe(III) as their trifluoroethylxanthates has been studied. Various parameters such as effect of pH, reagent concentration, digestion time, etc., have been

evaluated along with the degree of percentage of extraction . The method has been applied for the determination of these metals in certain standard reference materials and synthetic samples.

The fifth chapter comprises of the characterization and quantitative evaluation of some metal trifluoroethylxanthates by thin-layer chromatography (TLC). The order of $R_f$ values of the metal xanthates in pure solvents as well as in binary solvent mixtures have been discussed. Certain aspects of TLC behaviour of these metal xanthates on silica gel G have also been discussed. The method has been utilized for the spectrophotometric determination of cobalt and copper as their xanthates in some standard reference materials.

The last chapter consists of a brief introduction to amperometry. A simple volumetric method for the determination of trifluoroethylxanthate (the reagent) has been developed. Polarographic behaviour of the reagent has also been studied. Conditions have been developed for the amperometric determination of Co, Ni, Cu, Cd, Pb and Tl in certain complex materials and that of Cu-Cd, Cu-Pb and Hg-Pb simultaneously.

The thesis is appended with the application of potassium salts of n-butyl xanthate and 2-methoxyethyl xanthate in spectrophotometric determination of Co(III) and Co(II)

respectively, and that of tetrahydrofurfurylxanthate in the spectrophotometric determination of Co(II), Ni(II) and Ir(III) in certain standard reference materials and synthetic samples. A list of publication is also enclosed.

# A P P E N D I X

1. Solid-Liquid Separation after Liquid-Liquid Extraction: Spectrophotometric Determination of Cobalt by Extraction of its 2-Methoxyethyl Xanthate in Molten Naphthalene .

2. Spectrophotometric Determination of Cobalt using Potassium n-Butyl xanthate.

3. Spectrophotometric Determination of Cobalt, Nickel and Iridium after Coprecipitation of Their Tetrahydrofurfurylxanthates on to Microcrystalline Naphthalene .

4. Trifluoroethylxanthate as a Reagent for the Determination of Gold.

5. Spectrophotometric Determination of Tellurium after Separation by Coprecipitation of its Trifluoroethyl-xanthate with Naphthalene.

6. List of Publications.

# C O N T E N T S