TRACE ANALYSIS OF METALS AFTER EXTRACTION AND COPRECIPITATION OF THEIR METAL CHELATES WITH NAPHTHALENE

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C E R T I F I C A T E

This is to certify that the thesis entitled, "Trace Analysis of Metals After Extraction and Coprecipitation of Their Metal Chelates with Naphthalene", being submitted by Mr. Abdul Wasey to the Indian Institute of Technology, Delhi, for the award of the degree of 'Doctor of Philosophy' in Chemistry, is a record of bonafide research work carried out by him. Mr. Abdul Wasey has worked under our guidance and supervision and has fulfilled the requirements for the submission of this thesis which, to our knowledge, has reached the requisite standard.

The results contained in this dissertation have not been submitted in part or in 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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A B S T R A C T

The thesis consists of Seven chapters and each chapter is divided into various sections which contain introduction, experimental, results and discussion.

In the first chapter, the general introduction of the metal chelates, the methods and materials, and the scope of the present work is described.

The second chapter deals with the extraction-spectrophotometric behaviours of chromium(III), iron(II), iron(III), cobalt(II), nickel(II), copper(II), ruthenium(III), rhodium(III), palladium(II), osmium(VIII), iridium(III) and platinum(IV)-phenanthrenequinone monoxime complexes into molten naphthalene. Various parameters such as pH, reagent concentration, amount of naphthalene, volume of the aqueous phase, buffer solution, standing time, stirring time, effect of electrolytes etc have been evaluated. The nature of the metal complex extracted into molten naphthalene has been established in all the cases by Job's method of continuous variation and mole-ratio method. Procedures have been developed for the determination of these metal ions in various complex materials such as standard reference samples, waste waters, beers, wines, goat livers and human hairs. The solid complexes of the above mentioned metals with phenanthrenequinone monoxime have also been synthesized

and characterized by physico-chemical methods, viz., infrared, elemental analysis, magnetic moment, thermogravimetric analysis (TGA) and electron spin resonance spectral studies (ESR).

Coprecipitation-spectrophotometric behaviours of the above mentioned metals with phenanthronequinone monoxime have been studied on microcrystalline naphthalene in the third chapter. Various parameters similar to the above have been evaluated and conditions have been developed for the determination of these metals in certain complex materials.

The fourth chapter deals with the extraction-spectrophotometric behaviours of copper(II), palladium(II), iridium(III), and osmium(VIII) as 1-phenyl-4,4,6-trimethyl (1H, 4H)-pyrimidine-2-thiol complexes on microcrystalline naphthalene have been described in the fifth chapter. Various parameters similar to the above mentioned have been studied and conditions have also been developed for their determination in some standard reference samples and also in some synthetic mixtures.

The sixth chapter starts with a brief introduction of the exchange reactions and their applications in chemical analysis. Lead(II), zinc(II) and cadmium(II)-diethyldithiocarbamates have been extracted into molten naphthalene,

dissolved in chloroform and then replaced with copper in order to develop a yellow colour in the chloroform layer and then they are determined spectrophotometrically by measuring the absorbance at 440 nm. The accuracy of the method has been checked by the determination of these metals in some standard reference materials. The nature of the metal-diethyldithiocarbamate extracted into molten naphthalene has been established in each case.

The coprecipitation of lead(II), zinc(II), cadmium(II) lead(II), arsenic(III), indium(III), antimony(III) and thallium(I)-diethyldithiocarbamates with microcrystalline naphthelene is discussed in the last chapter. Since these complexes are colourless, the usual spectrophotometric method cannot be applied. Hence, the first three metals have been determined by atomic absorption spectrophotometric method after decomposing the complexes with nitric acid. The remaining metals have been determined spectrophotometrically after dissolution of the naphthalene containing metal complex in chloroform and then replacing with copper to develop a yellow colour in the chloroform layer. The absorbance has been measured at 440 nm in each case. Procedures have also been developed for the determination of these metals in various synthetic mixtures.

C O N T E N T S