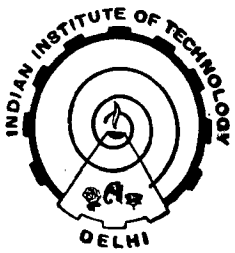
ANALYTICAL APPLICATIONS OF MORPHOLINE - 4 - CARBODITHIOATE

By
CHAMAN LAL SETHI
DEPARTMENT OF CHEMISTRY

Submitted
in fulfilment for the requirements
of the degree of
DOCTOR OF PHILOSOPHY



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This is to certify that the thesis entitled, "Analytical Applications of Potassium Morpholine-4-Carbodithioate", being submitted by Mr. Chaman Lal Sethi to the Indian Institute of Technology, Delhi for the award of the degree of Doctor of Philosophy, is a record of bonafide research work carried out by him. Mr. Chaman Lal Sethi has worked under my guidance and supervision and has fulfilled the requirements for the submission of this thesis, which to my knowledge, has reached the requisite standard.

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

(Dr. B.K. PURI)
Thesis Supervisor

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

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(C.L. SETHI)

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

The first chapter starts with the general introduction to metal chelates and organic complexing reagents. A brief introduction to the new method of extraction, "Analysis of Metals by Solid-Liquid Separation after Liquid-Liquid Extraction" has been discussed. This chapter also includes the scope of the present work.

The second chapter describes the method of preparation and standardization of potassium morpholine-4-carbodithioate. An indirect spectrophotometric method has been developed for the determination of Zn, Cd and Pb after extraction of their morpholine-4-carbodithioate complex into molten naphthalene. The nature of the metal complex extracted into molten naphthalene has been established in all the cases. Conditions have been developed for the determination of some of the metals in complex materials and synthetic samples.

In the third chapter, a systematic study has been undertaken to study the adsorption behaviour of various metals, using this reagent, over microcrystalline naphthalene. Conditions have been developed for the trace

determination of In, Sb, Tl, Co, Ni, Te, Cu, Bi, Fe and Pd spectrophotometrically. The formula of the metal complex adsorbed over naphthalene was established in each case. The methods so developed have been applied for the determination of some of the metals in complex materials like alloys, minerals, ores and biological samples.

Zinc, cadmium and lead form colourless complexes with this reagent. Therefore, an atomic absorption spectrophotometric method has also been developed for their determination after coprecipitation of their morpholine-4-carbodithioate complexes with micro-ccystalline naphthalene.

The last chapter starts with a brief introduction to the amperometric determination of metals. The analytical applications of this reagent in the amperometric determination of Ag, Hg, Tl, In, As, Sb and Pd have been explored. Various parameters like pH, base electrolyte concentration and formula of the metal complex have been established in each case. Selective, sensitive and rapid methods have been developed for some of these metals in synthetic samples. A method has also been suggested for the simultaneous determination of Ag-Pb or Hg-Pb from their synthetic sample present together in different ratios.

The thesis is appended with the list of publications.

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