FOREWORD

Since the earliest days of analytical chemistry, organic reagents have been employed in various ways to facilitate the detection and determination of chemical substances. During the past few decades these compounds have been extensively used in analytical procedures. At present these are rapidly gaining in popularity because of their greater sensitivity and specificity as compared to inorganic reagents, and also because of greater convenience.

In this connection solvent extraction has come to the forefront as a unique technique because of its elegance, simplicity, speed and applicability to both tracer and macro amounts of metal ions. The objective of these analytical liquid-liquid extraction procedures developed in the laboratory lies in the extrapolation to full-scale commercial hydrometallurgical processes from the technical point of view. The methods are particularly applicable to the

solvent extraction processes of numerous systems in metallurgical separation from mine waste and dump leach liquors.

This thesis is the outcome of a series of investigations carried out to study the analytical potentialities of cetyltrimethyl ammonium bromide (CTAB), 4-nitrosoresorcinol, diphenyl thiocarbazide and ninhydrinoxime as reagents for the spectrophotometric determination of iron, cobalt, palladium, platinum, gold, molybdenum, vanadium and copper. Auxiliary ligands namely iodide, thiocyanate, pyridine and some of its methyl substituted derivatives and solvents like chloroform, benzene, ethylacetate, 1,2-dichloroethane were made use of during the investigations.

The primary aim of this thesis is to develop analytical methods for the spectrophotometric determination of some heavy metals using some organic reagents. Some methods are promising from the view point of separation of the aforesaid metals from each other.

Attempts have been made to collect information relating to the analytical uses of a given compound and to present a discussion of the methods employing these reagents. References to the literature have been made so as to indicate, at least to some extent, the subject matter of each.

This thesis is divided into five chapters. A brief chapter-wise summary of the work done is as follows.

CHAPTER I

Part I of the chapter opens with a general introduction of liquid-liquid extraction. Here the theoretical aspects of solvent-extraction are described with special reference to the method of extraction, classification into ion association and chelate extraction systems and the equilibria involved.

Cetyl trimethyl ammonium bromide, 4-nitrosoresorcinol, diphenylthiocarbazide and nihhydrinoxime produce characteristic colour reactions with the metal ions. This permits extraction of the metals under some optimum conditions followed by spectrophotometric determination in the organic phase.

Part 2 deals with absorption spectrophotometry in ultraviolet and visible region as applied to analytical chemistry. A description of the instruments used in the present investigations along with the application of spectrophotometry in determination of inorganic ions is given.

CHAPTER II

In this chapter use of cetyltrimethylammonium bromide (CTAB) as a simple and sensitive reagent for the spectrophotometric determination of iron, cobalt, palladium, platinum gold, molybdenum and vanadium has been described.

In the beginning a literature survey on the use of CTAB in analytical chemistry is given. Use of other auxiliary ligands namely, thiocyanate and iodide has also been reviewed.

CTAB along with these auxiliary ligands produce characteristic colour reactions with the above metals, which is the basis of the extraction-spectrophotometric determination of the metals. The methods developed have been applied to the analysis of some synthetic mixtures with satisfactory results.

CHAPTER III

This chapter describes the use of 4-nitrosoresorcinol as an analytical reagent. The reagent forms coloured complexes with palladium, cobalt and copper. In some cases pyridine and some of its methyl substituted derivatives have been used as auxiliary ligands. The methods developed find applications to the analysis of some synthetic mixtures.

CHAPTER IV

Ninhydrinoxime has been used as an analytical reagent for the extraction-spectrophotometric determination of cobalt under some optimum conditions. Cobalt has been

determined in synthetic mixtures.

CHAPTER V

A spectrophotometric method for the determination of copper has been presented. The reagent used is diphenylthiocarbazide. The method developed is very simple and rapid and extended to the analysis of synthetic and standard samples.

Many obviously inferior reagents and methods are included since the treatment of the subject is intended to be complete. Their inclusion is justified because of two considerations; in the future so time need be lost in attempting analytical methods that in the past have been demonstrated as unsatisfactory; and further, an inferior method may, with suitable modifications, be made useful for certain purposes.