ANALYSIS OF METALS BY SOLID-LIQUID SEPARATION AFTER LIQUID-LIQUID EXTRACTION

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CERTIFICATE

This is to certify that the thesis entitled, Analysis of Metals by Solid-Liquid Separation after Liquid-Liquid Extraction being submitted by Miss Mamta Gautam 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 her. Miss Mamta Gautam 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.

14/2/80

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Thesis Supervisor

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(MAMTA GAUTAM)

ABSTRACT

The thesis consists of six 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 past work on this technique and the scope of the present work is described.

The determination of ethylthioxanthate and its application in the extraction-spectrophotometric determination of cobalt and palladium has been described in the second chapter. The nature of the metal complex extracted into molten naphthalene is established in both the cases. Based upon the thermal instability of nickel thioxanthate, a method is developed for the simultaneous determination of cobalt and nickel in various alloys.

The third chapter deals with the determination of ethylxanthate and its application in the extraction-spectrophotometric determination of nickel, cobalt, palladium and bismuth. In each case the formula of the metal complex extracted into molten naphthalene has been established. Since the rate of formation of cobalt xanthate is very slow compared to that of nickel xanthate, a method is developed for the simultaneous determination of nickel and cobalt in various alloys.

Morpholine-4-carbodithioate as complexing agent in the extraction-spectrophotometric determination of bismuth, copper, cobalt, nickel and tellurium has been discussed in the fourth chapter. A method is developed for the determination of this

reagent. Conditions have also been established for the determination of copper, cobalt and nickel in various alloys. Based upon the thermal instability of the selenium complex of this reagent, a method is suggested for the simultaneous determination of tellurium and selenium when present together. The formula of the metal complex extracted into molten naphthalene has been established in all the cases.

In the fifth chapter, a systematic study has been undertaken to study the extraction behaviour of metals using 8-hydroxyquinoline as the complexing agent. Successful extraction-spectrophotometric methods have been developed for chromium(III), gallium(III), lanthanum(III), niobium(V), osmium(VIII), rhodium(III), ruthenium(III) and vanadium(V). In all the cases the nature of the metal complex extracted into naphthalene has been determined. Conditions have been established for the determination of chromium, vanadium and niobium in various alloys.

The literature for the direct polarographic determination of metals after extraction into organic solvents using various complexing agents has been reviewed in the last chapter. The polarographic behaviour of metal oxinates has been studied in dimethylformamide-naphthalene using suitable supporting electrolyte and conditions established for the determination of copper, bismuth, palladium, iron(III) and uranium(VI) after extraction into molten naphthalene. Vatious alloys containing copper and iron has also been analysed.

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APPENDIX