PART I

INTRODUCTION
CHAPTER 1
IMPORTANCE OF TRANSITION METAL IONS IN INDUSTRY
Section (i) - Introduction:

Thiosemicarbazones are important organic analytical reagents. Because of the presence of thio-keto sulphur atom and azomethine nitrogen atom, they act as good chelating agents and form stable complexes with various metal ions. The metal complexes of these reagents are also found to be useful in treating many diseases. These reagents can act as unidentate, bidentate or multidentate ligands for metal ions. In view of their good chelating property, several thiosemicarbazone derivatives are used as analytical reagents for the determination of industrially important metal ions, using different physico-chemical techniques.

A short account of the importance of metal ions in industry:

One of the most rapidly developing areas of Analytical Chemistry is its application to industry. Many of the industrially important metal ions are present in trace amounts in complex materials viz. ores, alloys and minerals and their quantitative determination is frequently required in alloys and other complex materials.
Gold is an important noble metal whose quantitative determination in alloys and other noble metals is considered as an important analytical aspect. Many alloys used in dentistry contain gold, silver and copper often with small amounts of platinum and palladium. These alloys find many electrical uses as contacts, particularly where rubbing is involved.

Platinum is another important noble metal used in industry. Platinum-rhodium alloys are used in the manufacture of fibre glass and thermocouples. Platinum-iridium alloy with upto 30% iridium is used in electro-chemical anodes, electrical contacts, jewellery, surgical tools and implants. Ruthenium alloys are used as electrical contacts.

Palladium is an important industrial catalyst. Pure palladium is used in low current-electrical contacts in telephone equipment. Palladium is also used in the gold-base dental alloys to improve their strength. In the manufacture of jewellery, palladium is used alloyed with 4.5% ruthenium. Palladium is readily electroplated in thin or thick deposits for electrical contacts, printed circuits and other uses. Due to the continuously rising
Price of gold and platinum, palladium is used as an effective substitute in dental alloys and jewellery and with the increasing use of palladium for jewellery alloy and dental alloys, a need has arisen for development of simple and rapid methods for the quantitative determination of palladium in its alloys.

Osmium, like other platinum metals is catalytically active. The metal is used in incandescent lamp industry. Osmium occurs in platinum ores in the form of an alloy with iridium known as 'osmiridium' which is used for the tips of fountain pen nibs. Osmium alloyed with rhodium, ruthenium, iridium or platinum is used in fountain pen nibs, phonograph needles, electrical contacts and instrument pivots. Selective determination of osmium in trace amounts is therefore necessary and important.

Copper is one of the most important non-ferrous metal. Copper alloys are highly useful in industry. Brasses containing (5-40%) zinc constitute the largest volume of copper alloys. Nickel silvers (alloys of Cu, Ni and Zn) are used for zippers, camera parts, costume jewellery and some electrical switch gear. Copper-chromium alloys are used to make resistance welding electrodes, structural members for electrical switch gear, current
carrying members and springs. Aluminium bronzes are used for acid resisting pump parts, valve seats and guides and marine propellers. Copper alloys form a group of materials of major commercial importance. Hence analysis of copper containing compounds is an important laboratory activity.

Nickel has certain qualities suggesting that it is having great industrial importance. Nickel is an important catalyst in industry, besides its uses in the preparation of alloys, platings, ceramics, nickel-cadmium batteries, electronic circuits, cast iron etc. Hence development of methods for quantitative determination of nickel is considered to be an important analytical aspect.
Section (ii) – Objectives of the present work:

Since many of the noble metal ions are highly important in industry, analytical methods with highest possible sensitivity, which can tolerate large quantities of extraneous salts, and which will be as free as possible from error due to interfering ions, are required for their determination at micro levels in certain complex materials (alloys, ores, minerals etc.).

One of the techniques which is simple and readily in the reach of all laboratories of developing and developed countries is photometry. This is a non-destructive technique and is useful for the determination of trace amounts of constituents in presence of bulk quantities of other elements. The choice of spectrophotometric method for the determination of any constituent should generally fulfill certain conditions such as:

1. The reagent to be used should be readily available in a reasonably pure state,

2. The procedure should be specific, yet simple and sensitive,

3. The coloured species produced should be stable especially to light,
The method preferably rapid and simple without the need for heating.

If the method needs extraction stage, the partition coefficient should be high in favour of organic phase.

A study of these problems demands a reasonably rapid, simple, specific and selective analytical methods for the determination of trace amounts of metal ions.

During the course of our investigations in our laboratories on the use of thiosemicarbazones as analytical reagents, it is observed that anisaldehyde-4-phenyl-3-thiosemicarbazone (APT) and phthalimide dithiosemicarbazone (PIDT) possess many advantages over others as photometric reagents.

Hence an attempt is made for the development of new procedures for the spectrophotometric determination of metal ions viz. Au(III), Pt(IV), Pd(II), Os(VIII), and Cu(II) with anisaldehyde-4-phenyl-3-thiosemicarbazone and Ni(II) using phthalimide dithiosemicarbazone as analytical reagents. The procedures developed with APT are applied for the analysis of steel samples, alloys, minerals and ores (simulated mixtures).
The study of complexes in solid state is expected to be more interesting and informative. Therefore metal complexes of APT are synthesized and characterized on the basis of elemental analyses, electronic and infrared spectral analysis, magnetic susceptibility data, thermal analysis and conductance measurements.
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