C H A P T E R - VII

SUMMARY

Spectrophotometric methods are developed for the determination of trace amounts of metal ions. The new analytical reagents are used to determine the elements under specific conditions. Although several methods already exist for the analysis of each element, the search for new methods-more specific, more selective and more sensitive-continues. It is important to develop new simple methods of analysis of elements. We have investigated a new analytical reagent, cinnamaldehyde guanylhydrazone (CAG) and studied its applications.

CAG forms complexes with metal ions. The first chapter deals with synthesis and characterization of the reagent. Chapters two, three, four and five deal with the methods of determinations of copper (II), nickel (II), cobalt (II) and palladium (II) respectively. While chapter six deals with simultaneous spectrophotometric determinations of cobalt (II) and palladium (II).

Copper (II) is estimated in alkaline medium at 385 nm and a procedure for the analysis of brass has been developed. Nickel (II) forms yellow complex in alkaline medium and measured at 390 nm. This method can be used for the analysis of nickel (II) in cupronickel alloy. Cobalt (II) forms yellow complex with the reagent (CAG) and can be estimated at pH 11 at 410 nm. The method has been applied to analysis of cobalt (II)

in synthetic mixture of Co (II) and uranyl nitrate. Palladium (II) can be determined at 370 nm in alkaline medium. The method is both sensitive and selective and used for the analysis of palladium (II) in catalyst samples viz. Pd/charcoal and Pd/carbonate. The interferences and tolerance limits of foreign ions are discussed for each element separately. Applications of the reagent are given in table 7.1

Table 7.1 : Applications of the reagent (CAG)

Metal ion	Analysis of	Certified value	Experimental value
Copper (II)	Brass alloy	62.5% Cu	62.17 % Cu
Nickel (II)	Cupronickel alloy	31.2% Ni	30.5% Ni
Cobalt (II)	synthetic mixture of cobalt & uranyl nitrate.	9.0 µg Co (expected)	8.93 µg Co
		12.0 µg Co (expected)	12.05 µg Co
Palladium (II)	Pd/charcoal	10.0% Pd	9.93% Pd
	Pd/carbonate	6.0% Pd	6.08% Pd

As compared with other reagents, CAG forms complexes instantaneously and thus requires less time for estimation. CAG is stable in air. There is no action of light on the reagent. So no special care is required to protect it from light. Selectivity and sensitivity of the reagent is fairly good.

Spectral characteristics of the complexes are given in table 7.2.

Several synthetic mixtures of cobalt and palladium were prepared and analysed in triplicate by simultaneous spectrophotometric determination. Also cobalt and palladium are determined simultaneously from Co+Pd alloys.

Thus it can be concluded that CAG is fairly a good photometric reagent for the determinations of metals reported in this dissertation.

Table 7.2 : Spectral characteristics of metal complexes.

No.	Characteristics	Copper(II)	Nickel(II)	Cobalt(II)	Palladium(II)
1	Colour	Yellow	Yellow	Orange yellow	Yellow
2	λmax, nm	385	390	410	370
3	Molar extinction coefficient, ϵ (1 mole^{-1} cm^{-1})	0.2211×10^{4}	0.7647×10^{4}	0.1533×10^{4}	0.7455×10^{4}
4	pH selected	9.5	10	11	10.5
5	Composition (metal:ligand)	1:2	1:2	1:2	1:2
6	Beer's law validity, ppm	12.0	10.0	7.0	8.0
7	Sandell sensitivity, μg/cm^2	0.1988	0.05685	0.2837	0.06455
8	Degree of dissociation, α	0.1200	0.1212	0.1176	0.1273
9	Instability constant, K	1.946×10^{-10}	2.357×10^{-10}	2.123×10^{-10}	8.35×10^{-13}
10	Change in free energy, ΔF, K cal/mole	-13.229	-13.1171	-13.1776	-16.455