C H A P T E R - V I I

SUMMARY

The new analytical reagents are used to determine the elements under specific conditions. The science and technology demands a corresponding development in methodology and in techniques of analysis. Therefore, although several methods already exist for the analysis of each element, the search for new methods continues. It is important to develop new simple methods of analysis of elements which are more specific, selective and sensitive. We have investigated two reagents and studied their applications.

(1) Thiophene-2-aldehyde guanylhydrazone (TAG) :

The first part of the dissertation deals with the reagent TAG which forms complexes with metal ions. The first chapter deals with synthesis and characterisation of the reagent. Chapters two and three deal with the methods of determinations of palladium (II) and iron (II) respectively.

Palladium is estimated in alkaline medium at 365 nm and a procedure for the determination of palladium chloride in palladium (II) carbon catalyst has been developed. Iron (II) forms yellow complex in alkaline medium at pH 10 and measured at 360 nm. The method is both sensitive and selective and can be used for the analysis of iron (II) in Tonoferon syrup. Applications of the reagent are given in table 7.1.

Table 7.1 : Applications of the reagent (TAG)

Metal ion	Analysis of	Certified value	Experimental value
Palladium(II)	$PdCl_2$ in Pd(II)	6.00 %	5.88 %
	Carbon catalyst	4.00 %	3.93 %
Iron (II)	Tonoferon Syrup	5.00 %	4.90 %

TAG forms complexes instantaneously and thus requires less time for estimation. TAG 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.

The λ_{maxs}, molar extinction coefficients, pH values and composition of the complexes are given in table 7.2.

Table 7.2 : Spectral characteristics of complexes :

Metals	λ_{max} nm	Molar extinction coefficients, ϵ	pH values	Molar Ratio M:L
Palladium(II)	365	1.5441×10^4 l $mole^{-1} cm^{-1}$	10.5	1:2
Iron(II)	360	0.7262×10^4 l $mole^{-1} cm^{-1}$	10.0	1:2

The validity of Beer's law, Sandell sensitivity, the degree of dissociation, instability constant and change in free energy of the complexes are given in table 7.3.

Table 7.3 : Properties of metal complexes

Metals	Validity of Beer's Law, in ppm	Sandell sensitivity in $\mu g/cm^2$	Degree of dissociation α	Instability constant K	Change in free anergy ΔF K cal/mole
Palladium(II)	15.0	0.01148	0.1059	4.690×10^{-13}	-16.920
Iron(II)	8.0	0.05397	0.06897	4.518×10^{-13}	-16.944

The interference and tolerance limits of foreign ions are discussed for each element separately.

(2) <u>o-Hydroxy acetophenone guanylhydrazone (HAG)</u> :

The second part of the dissertation deals with the reagent o-hydroxy acetophenone guanylhydrazone (HAG). HAG forms complexes with metal ions. The fourth chapter deals with synthesis and characterisation of the reagent. Chapters five and six deals with the methods of determinations of nickel (II) and cobalt (II) respectively.

Nickel forms yellow complex and can be estimated at pH 10.5 at 380 nm. The method has been applied to analysis of nickel (II) in nickel steel. Cobalt (II) can be determined at 375 nm in alkaline medium. The method is both sensitive and selective and used for the analysis of cobalt in synthetic mixture of Co(II) and uranyl nitrate. Applications of the reagent are given in table 7.4.

Table 7.4 : Applications of the reagent (HAG)

Metal ion	Analysis of	Certified value		Experimental value
Nickel(II)	Nickel steel	3.0 %		2.88 %
Cobalt(II)	Synthetic mixture of Co(II) and uranyl nitrate	Expected	9.0 μg	8.98 μg
			12.0 μg	12.03 Pg

As compared with other reagents, HAG forms complexes with metals instantaneously. There is no action of light on the reagent. It is stable in air. Sensitivity and selectivity of the reagent is fairly good.

The λ_{maxs}, molar extinction coefficients, pH values and composition of the complexes are given in table 7.5

Table 7.5 : Spectral characteristics of complexes

Metals	λ_{max} nm	Molar extinction coefficients, ϵ	pH Values	Molar Ratio M:L
Nickel(II)	380	$0.882 \times 10^4 l mole^{-1} cm^{-1}$	10.5	1:2
Cobalt(II)	375	$0.854 \times 10^4 l mole^{-1} cm^{-1}$	11.0	1:2

The validity of Beer's law, Sandell sensitivity, the degree of dissociation, instability constant and change in free energy of the complexes are given in table 7.6

Table 7.6 : Properties of metal complexes

Metals	Validity of Beer's law, in ppm	Sandell sensitivity in $\mu g/cm^2$	Degree of dissociation α	Instability constant K	Change in free energy ΔF K cal/mole
Nickel(II)	22.0	0.02694	0.0750	5.288×10^{-13}	-16.850
Cobalt(II)	6.0	0.03289	0.1053	1.503×10^{-12}	-16.283

The interference and tolerance limits of foreign ions are discussed for each element separately.

It can be concluded that both TAG and HAG are fairly good photometric reagents for the determination of metals reported in the dissertation.