C H A P T E R - V I

SUMMARY

The applications of analytical reagents are more important in research. Therefore, under specific environmental conditions, new methods for the determination of elements are continuously being developed. Science and technology demand a development in methodology and new techniques of analysis. Therefore, although several methods already exist for the analysis of each element, the search for new methods continues which are more specific, selective and sensitive. It is therefore, necessary to develop new Simple methods of analysis of elements. We have investigated new analytical reagent, salicylaldehyde guanylhydrazone (SAG) and studied its applications.

SAG forms complexes with metal ions. The first chapter deals with synthesis and characterisation of the reagent. Chapters two, three, four and five deal with the methods of determinations of cobalt(II), manganese(II), nickel(II) and iron(II) respectively.

Cobalt is estimated in alkaline medium at 380 nm and a procedure for the analysis of cobalt in synthetic mixture of Co(II) and uranyl nitrate has been developed. Manganese(II) forms yellow complex in alkaline medium and measured at 400 nm. This method can be used for the analysis of manganese(II) in mild steel. Nickel forms yellow complex and can be estimated at pH 9.52, at 370 nm. The method has been applied to analysis of nickel(II) in alloy steel. Iron(II) can be determined at 425 nm in alkaline medium. The method is both sensitive and selective and used for the analysis of iron(II) in Vitcofol syrup. Applications of the reagent are given in table 6.1.

Table 6.1 : Applications of the reagent (SAG)

Metal ion	Analysis of	Certified value	Experimental value
Cobalt(II)	Synthetic mixture of cobalt and uranyl nitrate	Expected 7.0 µg 10.0 µg	6.97 µg 10.02 µg
Manganese(II)	Mild steel	0.6 % Mn	0.585 % Mn
Nickel(II)	Alloy steel	3.58 % Ni	3.55 % Ni
Iron(II)	Vitcofol syrup	0.65 % Fe	0.642 % Fe

As compared with other reagents, SAG forms complexes instantaneously and thus requires less time for estimation. SAG 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 λmaxs, molar extinction coefficients, pH values and composition of the complexes are given in table 6.2.

Table 6.2 : Spectral characteristics of complexes

Metals	λmax nm	Molar extinction coefficients, ϵ	pH values	Molar Ratio (M:L)
Cobalt(II)	380	1.2375×10^4 l mole^{-1}cm^{-1}	8.80	1:2
Manganese(II)	400	0.5214×10^4 l mole^{-1}cm^{-1}	8.20	1:2
Nickel(II)	370	1.0563×10^4 l mole^{-1}cm^{-1}	9.52	1:2
Iron(II)	425	0.4240×10^4 l mole^{-1}cm^{-1}	8.85	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 6.3.

Table 6.3 : 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 energy ΔF in Kcals/mole
Cobalt(II)	10	0.03337	0.1670	6.445×10^{-12}	-15.260
Manganese(II)	14	0.07842	0.1304	3.358×10^{-12}	-15.647
Nickel(II)	5	0.03907	0.0877	8.595×10^{-13}	-16.453
Iron(II)	16	0.09665	0.1273	1.708×10^{-10}	-13.320

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

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