Chapter - 6

Summary
The dissertation describes the use of salicylaldehyde isonicotinoyl hydrazone for the zero, first and second order derivative spectrophotometric determination of vanadium(V). The proposed methods are simple, sensitive and selective for the determination of vanadium without the need for extraction. The dissertation is divided into five chapters.

Chapter 1 gives a brief account of the fundamental principles of spectrophotometry. A brief account of the methods employed to establish the composition of the metal complexes in solution was presented towards the end of the chapter.

Chapter 2 is divided into two sections. Section (1) incorporates a brief review of the analytical application of isonicotinoyl hydrazones derived from carbonyl compounds to focus the potentialities of these compounds as analytical reagents. Section (II) deals with the objectives of the present investigations.

Chapter 3 consists of three sections. Section (1) presents the preparation of salicylaldehyde isonicotinoyl hydrazone. Stock solutions of the reagent, the metal ion, other solutions and solvents used in the investigation are described in section (II). A brief description of the instruments employed in the studies are presented in section (III).

Chapter 4 deals with the spectrophotometric determination of vanadium(V) using salicylaldehyde isonicotinoyl hydrazone. Vanadium(V) reacts with SAINH in acidic medium to give orange-yellow colour. The maximum absorbance is observed at 425 nm at pH 2.0. Beer's law is obeyed in the range 0.127 - 5.094 ug/ml of vanadium(V). The molar absorptivity and sandell's sensitivity are $1.00 \pm 0.05 \times 10^4$ l mol$^{-1}$ cm$^{-1}$ and 0.0051 ug cm$^{-2}$ respectively.
The standard deviation for 10 determinations of 2.03 ug/ml of vanadium(V) is found to be ± 0.009. Further the method was applied for the determination of vanadium(V) in steel samples.

Chapter 5 is divided into two sections. Section 1 describes the first order derivative spectrophotometric determination of vanadium(V) using salicylaldehyde isonicotinoyl hydrazine. The first order derivative spectrum of vanadium(V) and SAINH complex has a peak at 462 nm. The amplitude of the peak varied linearly with the concentration of vanadium(V). The peak heights are measured using peak-zero method to prepare the calibration plot. The plot showed that vanadium(V) can be determined in amounts as low as 0.050 ug/ml. The interferences due to diverse ions is less in comparison to those of zero order method. The standard deviation is ± 0.0047 for 10 determinations of 2.03 ug/ml of Vanadium(V). The method was successfully applied for the determination of vanadium(V) in steel samples.

Section 2 incorporates the second order derivative spectrophotometric determination of vanadium(V) using SAINH. The second order derivative spectrum of the complex exhibits a valley and a peak respectively at 425 nm and 481 nm. The peak heights are measured using peak-peak method to prepare the calibration plot. Vanadium(V) is determined by the method in the range 0.101 to 2.020 ug/ml. The standard deviation is 0.0037 for ten determinations. The interferences are less compared to those in zero order method. The method was successfully applied for the determination of vanadium in steel samples.

Chapter 6 summarises the various studies carried out in the present investigations.