

Short Communication

Spectrophotometric determination of micro amounts of vanadium (V) with resacetophenone—indirect method

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Abstract

A method has been developed for the indirect spectrophotometric determination of vanadium(V) using resacetophenone as reagent. It involves the oxidation of iron(II) to (III) by vanadium(V) and the resulting ferric iron gives wine-red coloured complex with resacetophenone. The complex was stable for 20 hr. The absorbance of the complex measured at 365 nm obeys Beer's law over the concentration range 2-8 $\mu\text{g/ml}$ of vanadium. The molar absorptivity and Sandell sensitivity are $(2.25 \pm 0.05) \times 10^3 \text{ lit. mol}^{-1} \text{ cm}^{-1}$ and $0.0226 \mu\text{g/cm}^2$ respectively. The effect of various ions was studied.

Key words : Spectrophotometry, vanadium (V), resacetophenone.

1. Introduction

Resacetophenone (2,4-dihydroxy-acetophenone) was first introduced by Cooper¹ as analytical reagent for the detection of iron in slightly acid medium. The reagent was applied for the fluorimetric detection of boron in concentrated sulphuric acid medium². The ketone was employed for the estimation of copper by many authors³⁻⁷. The reactions of the reagent with various metal ions was studied both in acid and alkaline media⁸. In acid medium the reagent gives no colour reactions with vanadium (IV) and iron (II). This fact was utilized for the spectrophotometric determination of microgram amounts of vanadium(V) with the reagent.

2. Experimental

Apparatus

ELICO Spectrophotometer Model GS 865A and ELICO Digital pH Meter Model L1-10 were used for these investigations.

Reagents

Resacetophenone was prepared from resorcinol⁹. The reagent solution was prepared in 50% methanol.

Stock solutions of ferrous ammonium sulphate and ammonium metavanadate were prepared by dissolving AnalaR grade samples in water. The concentrations were checked by standard methods.

The buffer solution of pH 3.0 was prepared by mixing sodium acetate (1 M)-hydrochloric acid (1 M).

3. Results and discussion

Oxidation of iron (II) to (III) by vanadium is well known¹⁰. The ferric iron formed gives wine-red coloured complex with resacetophenone⁹. The complex has absorption maximum at 365 nm and was stable for about 20 hr. Under the experimental conditions neither the vanadium(IV) formed nor iron(II) present in excess do not form any coloured complexes with the reagent. The system obeyed Beer's law over the concentration range 2-8 $\mu\text{g/ml}$ of vanadium. The molar absorptivity and Sandell sensitivity are $(2.25 \pm 0.05) \times 10^3 \text{ lit. mol}^{-1} \text{ cm}^{-1}$ and $0.0226 \mu\text{g/cm}^2$ respectively.

Procedure

A 15 ml aliquots of sodium acetate-hydrochloric acid buffer (pH 3.0) were taken into different 25 ml standard flasks. 2 ml of 0.01 M ferrous iron and 2 ml of resacetophenone (0.01 M) solutions were also added to the same standard flasks. A known volume of the standard vanadium(V) solution containing 50-200 μg of vanadium were then added and the solutions were made up to the mark with water. The contents in the flasks were shaken well and their absorbance was measured at 365 nm against the blank containing ferrous iron and the reagent. The data obtained showed suitability of the method for the determination of micro amounts (2-8 $\mu\text{g/ml}$) of vanadium.

Effect of various ions

K^+ , Na^+ , Cl^- , NO_3^- , SO_4^- did not interfere. Oxalate, citrate, tartrate, phosphate, ascorbate, Mo (VI), W(VI) and Al(III) interfered. Cr (III), Zn(II) and acetate interfered when

present in 100 fold excess. 80 fold excess of Co(II), Ni(II), Mn(II), Mg(II), Pb(II), 25 fold excess of Cd(II), Cu(II), 10 fold excess of V(IV), Br⁻, I⁻, SCN⁻ and 2 fold excess of U(VI) did not interfere.

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