

Gallacetophenone isonicotinolyhydrazone-a new sensitive reagent for rapid spectrophotometric determination of chromium (VI)

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ABSTRACT

Gallacetophenone isonicotinoyl hydrazone (GAPINH) forms a brownish yellow coloured soluble complex with chromium (VI) instantaneously at P^H 2.70. The complex exhibits an absorption maximum at 420nm with a molar absorptivity of 1.57×10^4 litre mole⁻¹ cm⁻¹ and photometric sensitivity of 0.0033 μ g cm⁻² ml⁻¹. Beer's law is valid over the concentration range 0.10 – 2.75 ppm of Chromium (VI) with an optimum concentration range of 0.50 – 2.60 ppm. A 5-fold molar excess of the reagent is necessary for the full development of the colour intensity. The stoichiometry between chromium and GAPINH is established as 1:1. The stability constant of the complex calculated from Job's method was 1.56×10^5 . The method is simple, sensitive and rapid.

Key words: GAPIMH, spectrophotometry, chromium (VI).

INTRODUCTION

Apparatus

A Beckman DK-2A UV-Visible recording spectrophotometer, an Elico Spectrophotometer Model CI-24 and an Elico P^H meter Model LI-10T for optical and P^H measurements respectively.

Reagents

Gallacetophenone isonicotinoyl hydrazone was prepared from Gallacetophenone and isonicotinoyl hydride in the laboratory and purified by recrystallisation from a 50% solution of aqueous dimethyl formamide. Chromium (VI) solution was prepared by dissolving the requisite amount of potassium dichromate (ARBDH) in double distilled water. Buffer solutions in the P^H range 0.65 – 5.2 were prepared from 1M Hydrochloric acid and 1M sodium acetate. All the other chemicals used were of analytical reagent grade or equivalent. Solutions of lower concentrations were prepared by appropriate dilutions.

General procedure

An aliquot of the sample containing of 0.05 – 3.12 μ g of chromium (VI), 2.0ml of buffer solution

of P^H 2.7 and 1ml of GAPINH (0.001M) solution were taken in 10ml volumetric flask and the solution was diluted to the mark with doubly distilled water. The absorbance of the resulting solution was measured at 420nm against the reagent blank.

RESULTS AND DISCUSSION

Absorption spectra

The absorption spectrum of the reaction mixture containing chromium (VI) and the hydrazone in the buffer solution against blank solution revealed that the mixture has absorbance maxima at 420nm.

Effect of the hydrazone concentration

The effect of the hydrazone concentration is carried out keeping the concentration of the hydrazone to be added for the maximum colour formation. The results indicate that five-fold excess is sufficient to give reproducible results.

Effect of time on the colour reaction

The absorbance is recorded at different intervals of time to establish the effect of time on the stability of the colour. The absorbance is stable for more than 24 hours.

Table 1: Tolerance limit of diverse ions in the determination of 2.08 mg of Cr(VI)

Ion added	Tolerance Limit(mg)
Fluoride	2500
Chloride	5000
Bromide	5000
Iodide	2000
Oxalate	1000
Bromate	5000
Nitrate	5000
Sulphate	2000
Citrate	60
Tartrate	600
Ascorbic acid	5000
Iodate	0.5
Mg(II)	5000
Pb(II)	5000
Ni(II)	800
Bi(II)	2
V(V)	15
Zn(II)	80
Ba(II)	1000
Zn(IV)	2000
Mn(II)	160
U(II)	90
Co(II)	100
Mo(VI)	4
Cu(II)	0.1
Fe(III)	<0.1
Al(III)	<0.1

Effect of P^H

The absorbance of Cr(VI) was studied over the P^H range from 1.0 to 8.0. The optimum P^H range for the formation of brownish yellow complex was found to be 2.0-3.5. the absorbance of the complex decreased below P^H 2.0 and above 3.5. a buffer medium of P^H 2.7 was conveniently chosen for all further studies.

Beer,s law range, sensitivity

The cr(VI)-GAPINH complex obeys Beer,s law over the concentration range 0.10-2.60 ppm at 420nm. The optimum working range as evaluated by Ringborn;s methoad was 0.50-2.60 ppm.

The molar absorptivity was 1.57×10^4 lit. mole⁻¹ cm⁻¹. According to Sandell,s expression, the sensitivity of the reaction was 0.0033 $\mu\text{g.cm}^2.\text{ml}^{-1}$.

Composition of the complex

The composition of Cr(VI) – GAPINH complex was studies by Job's continuous variation¹, mole ratio² and slope ratio^{3*} methods. Job's method showed that the stoichiometric ratio of Cr(VI) – GAPINH to be 1:1 and this was confirmed by the other two methods. The stability constant of the complex as evaluated by Job's continuous variation method was 1.56×10^5 .

Nature of the complex

The nature of the complex was studied by passing an aliquot of the solution of the complex through the cataion exchange resin, Dowex 50W – X8 and Amberlite IR – 120 (H). The complete absorbance of the coloured complex by the ionic exchangers has indicated that the complex is cationic.

Effect of diverse ions

In order to asses the possible analytical applications of Cr(VI) – GAPINH complex, the effects of some ions which often accompany chromium were studied by adding different amounts of ionic species to 2.08 mg of chromium (VI) in solution in 10ml volumetric flasks and the colour was developed as described in the procedurern the tolerance limits are shown in Table -1. An error of $\pm 2\%$ in the absorbance was considered tolerable.

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