Asian Journal of Chemistry Vol. 5, No. 3 (1993), 637–638

Photometric Determination of Benzoyl and Salicyloyl Hydrazines with Vanadium(V)

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The results of the photometric determination of benzoyl hydrazine (BH) and salicyloyl hydrazine (SH) with vanadium(V) in phosphoric acid medium are reported. Osmium tetroxide is found to catalyse the reaction. All the titrations are carried out in presence of 2–6 ml of orthophophoric acid and 0.5 ml of OsO_4 in a total volume of 25 ml, by following the increase in intensity of blue colour of vanadium(IV) at its maximum wavelength.

INTRODUCTION

Benzoyl hydrazine (BH) and salicyloyl hydrazine (SH) find extensive use in chemotherapy and chemical analysis. The $-NH{\cdot}NH_2$ group in these compounds is susceptible for oxidation. The analytical aspects of these compounds have been very little studied[1-3]. In this communication, we report the photometric determination of benzoyl and salicyloyl hydrazines with vanadium(V) in phosphoric acid medium using osmium tetroxide as catalyst. The advantage of the photometric procedure is that it affords accurate results even when the overall reaction is slower near the equivalence point while it is not the case in the potentiometric titration or titration to visual end point.

EXPERIMENT

0.025 M solutions of BH and SH were prepared by dissolving Fluka AG's pure sample. The soulutions were standardised against a standard solution of potassium bromate. An approximately 0.1 M solution of sodium vanadate was prepared from E. Merck sample and standardised with a standard solution of iron(II) solution. E. Merk's pro-analysis grade (85%) orthophosphoric acid was used. A 0.1% solution of osmium tetroxide was prepared and stored in an amber-coloured glass bottle. All other chemicals were of AnalaR grade. A Klett-Summerson photoelectric colorimeter with filter No. 66 was employed for photometric titrations.

RESULTS AND DISCUSSION

Vanadium(IV) has considerable absorption in the region 650–750 nm. Under similar experimental condition, the hydrazides or their oxidation products have little effect on the absorption of vanadium(IV) solution. Hence, a photometric

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titration of BH or SH with vanadium(V) can be carried out by following the increase in the intensity of blue colour of vanadium(IV).

The optimum concentrations of acid and catalyst were ascertained by varying the concentration of either phosphoric acid or osmium tetroxide. The experiments indicated that 2–6 ml of orthophosphonic acid and 0.2 to 0.6 ml of 1% osmium tetroxide in a total volume of 25.0 ml are sufficient for a rapid photometric titration.

Recommended Procedure

An aliquot of (0.0630 to 0.3150 mmole)BH or SH taken in an optical cell (2 × 4 × 8 cm) and treated with 2–6 ml of orthophosphoric acid and 0.5 ml of osmium tetroxide. The mixture is diluted to 25.0 ml and the contents of the cell are allowed to attain the room temperature. The initial duel reading of the instrument is adjusted to read zero and the solution is stirred by passing carbon dioxide. The solution is titrated with standard solution of vanadium(V) and the absorbance of the vanadium(IV) formed is measured 30 seconds after stopping the passing of carbon dioxide using filter No. 66. In order to account for the dilution effect on the absorbance readings, they are multiplied by a factor $\frac{V+v}{V}$ where V is the volume of reaction mixture taken and v is the volume of titrant added. Some representative results are recorded in Table 1. Our stoichiometric studies of the reaction show that four moles of oxidant were consumed for one mole of hydrazide.

TABLE 1
PHOTOMETRIC DETERMINATION OF BH AND SH WITH VANADIUM(V)

Sl. No.	Benzoyl hydrazine (mmoles)			Salicyloyl hydrazine (mmoles)		
	Taken	Found†	% relative error	Taken	Found†	% relative error
1.	0.0630	0.0630	0.00	0.0631	0.0631	+0.16
2.	0.1260	0.1262	–0.16	0.1262	0.1200	+0.16
3.	0.1890	0.1881	+0.47	0.1893	0.1883	0.21
4.	0.2520	0.2512	+0.32	0.2525	0.2512	0.51
5.	0.3150	0.3143	+0.22	0.3030	0.3017	0.42

† Average of four experiments.

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(*Received*: 3 January 1992; *Accepted*: 15 October 1992) AJC–499