Asian Journal of Chemistry Vol. 8, No. 2 (1996), 258–260

Analytical Studies on the Oxidation of Some Hydrazine Derivatives with Ammonium Pentavanadate(V) Reagents

R.P.S. CHAUHAN* and GEETA MISHRA
Department of Chemistry
Gaya College (Magadh University), Gaya-823 001, India

A simple quick and convenient method has been developed for the micro estimation of some hydrazine derivatives. The sample (1–5 mg) is allowed to react with 2 mL of 0.3 N ammonium pentavanadate(V) reagent and 10 mL of 10 N H_2SO_4. The unconsumed reagent can be accurately titrated with 0.025 N ferrous ammonium sulphate solution using N-phenyl anthranilic acid as indicator. Standard deviation as well as coefficient of variation was calculated for reproducible and accurate result. The accuracy of the method is within ± 1%

INTRODUCTION

Hydrazines have been quantitatively oxidized with various organic and inorganic oxidants. A number of methods *viz.*, colorimetric[1], photometric[2], and polarographic[3], have been used for the determination of hydrazine. Hydrazines have also been determined potentiometrically[4] with NBS[5], thallium(III)[6], NCSA[7], Ce(IV)[8], bromanine-T[9] and NBSA[10]. The present method is better than the existing methods and does not require a catalyst and sophisticated instrumentation.

EXPERIMENTAL

Ammonium pentavanadate (0.3 N solution) was prepared by dissolving 3.5 g of ammonium pentavanadate in 10 mL of 10% sulphuric acid in a 100 mL measuring flask and made up to the mark with distilled water. Ferrous ammonium sulphate (0.025 N solution) was prepared by dissolving 2.4508 g (AnalaR, BDH) of ferrous ammonium sulphate in distilled water in a 250 mL measuring flask and 10 mL of sulphuric acid was to suppress hydrolysis. The solution was standardized by titration with standard potassium dichromate (0.02 N) solution using diphenylamine sulphonic acid as indicator. Solid N-phenyl anthranilic acid was dissoloved in 3 mL of 5% sodium carbonate and the solution was diluted up to 150 mL with distilled water.

Sample solution : A stock solution of the hydrazine derivatives was prepared by dissolving 50–100 mg of the sample in minimum amount of glacial acetic acid in a 100 mL measuring flask and made up to the mark with distilled water. All the samples were either AnalaR, BDH grade or purified by crystallization.

Procedure: Aliquots containing 1–10 mL of the sample were placed in a 100 mL Erlenmeyer flask and 2 mL of a 0.3 N solution of ammonium pentavanadate was added, followed by the addition of 10 mL of 10 N sulphuric acid. The reaction mixture was shaken gently and heated on a boiling water bath for a prescribed reaction time. After the reaction was over, the mixture was cooled to room temperature and titrated with 0.025 N Fe(II) solution using N-phenyl anthranilic acid solution as indicator. A blank experiment was also run under identical conditions using all the reagents except the sample.

The amount of the sample was calculated by the following expression:

$$\text{mg of the sample} = \frac{M(B - S)N}{n}$$

where M = molecular weight of the sample,

N = normality of Fe(II),
B = volume of Fe(II) used titrate the blank experiment
S = volume of Fe(II) used to titrate sample experiment,
N = number of moles of the V(V) reagent consumed per mole of the sample.

RESULTS AND DISCUSSION

The effect of variables such as the volume and concentration of the reagent and sulphuric acid temperature and reaction time were studied. It was found that the recommended concentration of V(V) reagent is suitable to achieve quantitative reaction. The addition of 10 mL of 10 N H_2SO_4 before the reaction helps in detecting the end point. It was also observed that the reaction was incomplete at room temperature (27°C) and direct heating of the reaction mixture gave inconsistent results. Accurate and constant results were obtained when the reaction was carried out on a water bath. Normally, the reaction is completed within 15 minutes.

TABLE-1
DETERMINATION OF HYDRAZINES WITH V(V) REAGENT

S.No.	Sample	Amount taken (mg)	Reaction time (min)	Amount recovered (mg)	Stoichio-metry	Error	SD	CV
1.	Hydrazine sulphate	1.0045	10	0.9990	4	–0.55	0.0051	0.5087
		5.0155		5.0520		+0.72	0.0139	0.2751
		10.0680		10.1188		+0.50	0.0479	0.4700
2.	Hydrazine hydrochloride	1.0023	10	0.9998	4	–0.25	0.0041	0.4089
		5.0210		5.0492		+0.56	0.0007	0.0099
		10.0500		10.0800		+0.30	0.0006	0.0059
3.	Phenyl hydrazine hydrochloride	1.0068	15	0.9990	8	–0.78	0.0062	0.6179
		5.0220		5.0520		+0.60	0.0267	0.5302
		10.0440		10.0985		+0.54	0.0503	0.4998

The number of moles of the V(V) reagent consumed for the sample depends upon the structure of the compound (Table-1). Taking into consideration the mole of vanadium consumed for the sample and the isolation of the reaction products, a possible course of the reaction had been proposed for each compound.

While studying the oxidation of hydrazine derivatives it was noticed that hydrazine is completely oxidised to nitrogen and water. The evolution of N_2 has quantitatively been established because of phenyl hydrazine hydrochloride; the final oxidation product was found to be *p*-benzoquinone.

REFERENCES

1. P.R. Wood, *Anal. Chem.*, **25**, 1879 (1953).
2. R. Preussman, H. Hengy, D. Lvebbe and A. Von Hondenberg, *Anal. Chim., Acta,* **41**, 497 (1968).
3. D.K. Banerjee, G.C. Reichmann and C.C. Budke, *Anal. Chim.*, **36**, 220 (1964).
4. A. Berks and A.K. Busev, *Anal. Chim. Acta,* **27**, 493 (1962).
5. M.Z. Barakat, M.F.A. Wahab and M.M. Elsaov, *J. Am. Chem. Soc.*, **77**, 1670 (1955).
6. B. Singh and A. Singh, *J. Indian Chem. Soc.*, **29**, 34 (1952).
7. R.P.S. Chauhan, V.K. Dubey and U.B. Singh, *Asian J. Chem.*, **6**, 665 (1994).
8. R.P.S. Chauhan, A.L. Singh, R.K. Dwivedi and I.C. Shukla, *J. Indian Chem. Soc.*, **58**, 719 (1981).
9. I.C. Shukla and O.P. Rai, *Asian J. Chem.*, **5**, 548 (1993).
10. R.P.S. Chauhan, R. Prakash and I.C. Shukla, *University of Allabahad Studies*, **9**, 6 (1977).

(*Received*: 21 August 1995; *Accepted*: 30 October 1995) AJC1032-