

Analytical Studies on the oxidation of cysteine, cystine and Methionine with V(v) reagent

Afshan Suraiya¹, Siddharth Singh² and R. P. S. Chauhan³

¹Deptt. of Chemistry G.B.M. College, Gaya - 823001 (Bihar)

²P. G. Department of Chemistry, Gaya College (Magadh University), Gaya

ABSTRACT: A simple, quick and convenient method has been developed for the micro estimation of S-containing amino acids. The sample 1-5 mg is allowed to react with 2 ml of 0.3N ammonium metavanadate (v) reagent and 10 ml of 10N H₂SO₄ was added, before the reaction helps in detecting the end point. The unconsumed reagent can be accurately titrate 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 in within $\pm 1\%$

Keywords: Determination, Oxidation, S-containing amino acid, Vanadium (v)

I. INTRODUCTION

Sulphur containing amino acids are of great importance in the biochemical process of animals. A number of procedures have been developed for the determination of sulphur containing amino acids¹⁻⁹. In the present paper we describe a method for the determination of S-containing amino acids at the mg level using ammonium pentavanadate (v) as the good oxidizing reagent¹⁰⁻¹⁴. The present method is better than the existing methods and does not require a catalyst and sophisticated instrumentation.

II. EXPERIMENTAL

2.1 AMMONIUM PENTAVANADATE (0.03 N SOLUTION): was prepared by dissolving 3.5g of ammonium pentavanadate in 10ml of 10% sulphuric acid in a 100 ml measuring flask and made up to the mark with distilled water.

2.2 FERROUS AMMONIUM SULPHATE (0.025 N SOLUTION): was prepared by dissolving 2.4508g (AnalaR, BDH) of ferrous ammonium sulphate in distilled water in a 250 ml measuring flask and 10 ml of sulphuric acid was added to check the hydrolysis. The solution was standardized by titration with standard potassium dichromate solution (0.02N) using diphenylamine sulphonic acid as indicator.

2.3 SOLID N-PHENYL ANTHRANILIC ACID: was dissolved in a 3ml of 5% sodium carbonate and the solution was diluted upto 150 ml with distilled water.

2.4 SAMPLE SOLUTION: Stock solutions of each of the S-containing amino acids were prepared in distilled water. 100 ml of the sample was taken in a 100 ml measuring flask and the solution was raised to the mark with distilled water.

III. GENERAL PROCEDURE

Aliquots containing 1-5 mg of the sample were placed in a 100 ml Erlenmyer flask and 2 ml of 0.03N 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 0.025N 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 expressions.

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

Where

- M= Molecular weight of the sample.
- N = Normality of Fe(II) used titrate the blank experiments.
- B = Volume of Fe(II) used titrate the blank experiments.
- S = Volume of Fe(II) used titrate the sample experiments.

n = Number of moles of the V(v) reagent consumed per mol of the sample.

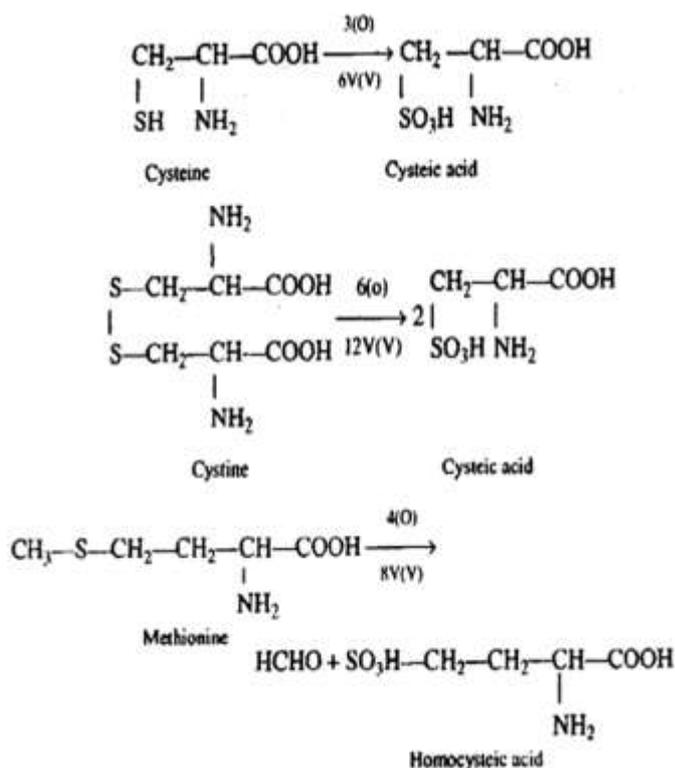
IV. RESULTS AND DISCUSSION

With recommended procedure the determination of cysteine, cystine and methionine has been successfully achieved on 1-5 mg of sample within an accuracy of $\pm 1\%$ (Table -1) in most of the cases.

TABLE-1
DETERMINATION OF CYSTEINE, CYSTINE AND METHIONINE WITH 0.3N, V (v)

Sl. No	Sample	Amount taken (mg)	Reaction time (min)	Amount recovered (mg)	Stoichiometry	Error*	SD	CV
1	Cysteine	1.0040	10	0.9980	6	-0.60	0.0042	0.4194
		5.0190		5.0425		+0.47	0.0012	0.0238
		10.0330		9.9721		-0.58	0.0420	0.4167
2	Cystine	1.0050	10	0.9996	12	-0.54	0.0043	0.4289
		5.0105		5.0387		+0.54	0.0239	0.4759
		10.0210		10.0685		+0.47	0.0018	0.0179
3	Methionine	1.0040	10	1.0098	8	+0.58	0.0051	0.6071
		5.0250		5.0528		+0.55	0.0003	0.0059
		10.0400		10.0982		+0.58	0.0559	0.5357

Considering the oxidation reaction of S-containing amino acids and the number of equivalents of V(V) consumed for a particular sample the following course of reaction may be suggested for the oxidation of cysteine, cystine and methionine.



It was found that easily oxidisable organic compounds like alcohols, phenols, aromatic hydrocarbons and thioureas interfere in the determination, while the other amino acids like glycine, alanine are unaffected by the V(v) reagent.

REFERENCES

- [1] F.G. Hopkins, Amino acid and human behaviour, *J. Biol. Chem.* 84, 1929, 269
- [2] G. J. Mulder, Determination of ascorbic acid, *Anal. Chem.* 28, 1838, 73.
- [3] H.B. Vickery and A.White, *Amino acids used for the titration of Trivalent Thallium*, *J. Biol. Chem. (B)* 99, 1932, 701.
- [4] R. J. Thibert and M. Sarwer, Determination of hydrazine and hydrazide, *Mikrochim Acta Wien*, 1942, 259.
- [5] M. M. Beg, Q. S. Usmani and I. C. Shukla, Milligram Determination of thiamino acid with ammonium hexanitratocerate (iv) reagent, *Mikrochim. Acta Wien*, II, 1977, 221.
- [6] I. C. Shukla, R. P. S. Chauhan and Ravi Prakash., A New Titrimetric method for the determination of some thioureas and thioamino acids *Proc. Nat. Acad. Sci. Ind.* 51(A), 1981, 149.
- [7] C. P. Ivanov, B. A. Alexien and M. Krustava, The titration of amino acids Amperometrically *Anal Chem.* 30, 1964, 549.
- [8] M. Krustava, B. A. Alexien, C. P. Ivanov and B. Yordanov, Evaluation of amino acid content of certain foods stuffs, *Anal Chem.* 32, 1965, 465.
- [9] R. P. S. Chauhan, V. K. Dubey, U. B. Singh and R. Mishra, Analytical studies on the oxidation of cysteine, cystine and methionine using potassium dipertelluratocuparate (III) as oxidant at milligram scale *Oxidation. Comm.* 16, 1993, 240.
- [10] Jamaluddin, S. Ahmad and I. C. Shukla, Micro determination of hydrazine and its derivatives with V(v) reagent. *Analyst*, 109, 1984, 1102.
- [11] S. Ahmad, Jamaluddin and I. C. Shukla, Micro determination of aromatic amines & phenol with ammonium vanadate reagent *Acta Chimica Hungarica*, 119, 1985, 339.
- [12] A. Suraiya, I. Haque and S. Singh, Determination of Vitamin – C in pure and pharmaceutical preparations at microscale, *Oxid. Comm.* 34(4) 2011, 877.
- [13] R.P.S. Chauhan and A. Suraiya, Bromine monochloride as an analytical reagent for the microgram determination of hydrazine derivatives in acetic acid medium. *Oxid. Comm.* 22(4), 1999, 581.
- [14] R.P.S. Chauhan, Siddharth Singh and A. Suraiya, Determination of some antimalarial aminoquinolines by ammonium hexanitratocerate (iv) reagent as oxidant at micro scale *Oxid. Comm.* 31(1), 2008, 219.