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AN APPLICATION OF POTASSIUM PERMANGANATE AS AN OXIDANT FOR THE DETERMINATION OF NITROGEN IN AGRICULTURAL AND ANIMAL PRODUCTS

M. Akhtar Siddiqui, Shafiq A. Khan and M.K. Bhatty

PCSIR Laboratories Lahore, Pakistan

Muhammad Ashraf

Faculty of Pharmacy, University of Punjab, Lahore, Pakistan

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A method has been described for the determination of nitrogen in agricultural and animal materials in the Kjeldahl digests by the use of potassium permanganate. The material is digested with sulphuric acid using mercury and potassium sulphate as catalyst. The resultant ammonium sulphate is reacted with potassium permanganate in the presence of potassium bromide and sodium hydroxide in a special flask. The unreacted potassium permanganate is then determined iodometrically.

INTRODUCTION

In the past, several successful attempts have been made for the determination of ammoniacal nitrogen in the Kjeldahl digests without distillation. The distillation step has been eliminated by using formaldehyde [1] and hypochlorite [2]. Nitrogen has successfully been determined particularly by the later method without the distillation on micro, semi-micro nand macro [3] scales and also in agricultrual and animal products. [4].

A bromometric method [5], has been reported in which brominating mixture and potassium tetraborate have been employed. Similarly in an other method potassium bromate and potassium bromide have been used [6]. It has also been reported that potassium permanganate and potassium dichromate act like bromate for the determination of nitrogen in organic compounds [7]. Potassium permanganate in acidic medium generates bromine in the presence of bromide, which on the addition of an alkali, is converted to hypobromite *in situ.*

During the present investigations, potassium permanganate has been applied to the determination of nitrogen in agricultural and animal products for the first time; the results are within the experimental errors, when compared with the results using potassium bromate. Its standard solution is stable for about two months. Hypohalite solutions have extensively been used for the determination of nitrogen in soil [8], biological materials [4] and organic compound [9]. Difficulties are normally encountered in their preparation. Such difficulties have now been removed by the use of potassium permangnate as an oxidant for the determination of nitrogen in Kjeldahl digests.

EXPERIMENTAL

Apparatus. A 500 ml round bottom flask, with B 24 neck fitted with a 15 cm tube attached to B-24 cone, is used for digestion. After the digestion the cone is replaced with an N–type tube. [7].

Reagents. Sulphuric acid (d,1.84), G.R.; potassium sulphate, G.R; Mercuric sulphate G.R; Potassium permanganate solution, 0.5 N; Potassium bromide, G.R; sodium hydroxide solution, 60% W/V; Potassium iodide, G.R; Sulphuric acid solution 10 N; Sodium thiosulphate solution, 0.25 N; Sod.bicarbonate, G.R; starch solution (aquous), 2%.

Procedure

Weigh accurately 1 to 2 g of the fresh vegetables, 0.1 to 0.2 g dried blood, or 1g meant, cereals, tea leaves into the flask. Add 7 g of potassium sulphate 0.7 g of mercuric sulphate and 14 ml concentrated sulphuric acid. Digest the material for 1– hr. Cool and dilute the digest by rinsing the cone with 50 ml of distilled water. Put the flask in cold water and run 20 ml of potassium permanganate

Table 1. Recovery of nitrogen

Commercial Materials	Method followed: Bromate %		Permanganate %	
1. Rice	1.21	1.28	1.20	1.20
2. Grams (Black)	3.30	3.19	3.10	3.2
3. Wheat	1.90	1.86	1.80	1.92
4. Peas (Green)	1.30	1.18	1.22	1.30
5. Tea leaves (Lipton)	3.50	3.45	3.50	3.50
6. Milk (Cow)	0.50	0.53	0.58	0.54
7. Meat (Sheep)	3.18	3.30	3.24	3.26
8. Dried blood (Commercial)	12.10	12.20	12.30	12.20
9. Cotton seed cake	3.81	3.72	3.69	3.78
10 Potato	0.34	0.38	0.35	0.38
11. Tabacco (Dried) leaves	2.00	1.95	2.10	2.05
12. Egg (Hen)	1.98	2.06	2.04	2.00
13. Mustard-seed cake	2.85	2.90	3.00	2.90

solution and add 5 g of potassium bromide. Immediately fit in the stoppered N-shped tube containing 40 ml of sodium hydroxide solution. Transfer the alkali portion wise from the tube to the cold contents of the flask by tilting the system slightly. Shake the flask and allow it to stand for 10–15 min. Add 8 g of potassium iodide followed by 25 ml of 10 N sulphuric acid. Allow to stand further for 5 min. Remove the tube by rinsing it with distilled water and titrate the liberated iodine with sodium thiosulphate solution using a small amount of sodium bicarbonate near the end point. Use strach as an indicator. Carry out a blank determination under similar conditions.

Calculations

1 ml 0.1N $KMNO_4$ or 1 ml 0.1N $Na_2S_2O_3$ = 0.467 mg N_2

DISCUSSION AND CONCLUSION

Potassium permanganate is commonly used as an analytical reagent for the oxidimetric titrations. Its solutions when prepared and stored according to the established procedure, is far more stable than hypohalite solutions. Difficulties as encountered in the hypohalite solutions, do not arise in the preparation of potassium permanganate solutions and considerable time is thus saved.

Some difficulties are also faced when hypochlorite solution is used for the determination of nitrogen in agricultural and animal products, for example in the case of tea leaves, meat and blood, the digestion mixture is not clear and presence of iron interferes in the titration. However, these difficulties have been minimised by decreasing the amounts of the sample to be digested [4]. Such diffi-

culties do not appear, when potassium permanganate is used in oxidimetric titrations. The results are usually higher if the materials are not completely digested, as some amount of potassium permanganate is also consumed to oxidise the organic matter left out in the digests. Therefore, in order to obtain accurate results, the digestions must be completed.

Table 1 shows the results are within the experimental errors when compared with that of potassium bromate method.

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