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Microdetermination of Purine Derivatives with Bromine Monochloride Reagent in Acetic Acid Medium

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In the present work a quick and convenient method had been described for the determination of purine derivatives with the use of bromine monochloride (BrCl) in acetic acid medium at 0–5°C temperature. The accuracy of the method is within ± 1%.

INTRODUCTION

Costro and Mendoza[1] and Bontemps[2] suggested a colorimetric method for the determination of caffeine in pharmaceuticals preparation. A spectrophotometric method[3] has been developed for the determination of theophylline. Rink[4] suggested a titrimetric method for the determination of caffeine and theobromine using perchloric acid titrant in anhydrous benzene. Singh[5] has also developed new titrimetric method for the determination of xanthine, caffeine, theophylline, theobromine and uric acid with the use of N-bromosuccinimide and N-bromosaccharine reagent respectively.

In the present work a quick and convenient method has been described for the determination of purine derivatives with the use of bromine monochloride in acetic acid medium at 0°–5°C.

EXPERIMENTAL

Bromine monochloride 0.05 N solution: 0.6958 Gm potassium bromate and 0.9917 gm. potassium bromide were dissolved in 125 ml of distilled water in 500 ml volumetric flask. The solution was cooled in ice. 50 Ml of conc. HCl was added and the solution was made up to the mark with distilled water. The solution was standardised with 0.02 N hypo solution and stored in an ice and salt mixture.

Sodium thiosulphate 0.02 N solution: A stock solution of sodium thiosulphate was prepared by dissolving 4.9640 gm. of sodium thiosulphate in distilled water in 1 litre volumetric flask. The solution was standardized with 0.02 N copper sulphate solution iodometrically.

Potassium iodide: 15% (w/v) aqueous solution of potassium iodide was prepared.

Starch solution: 1% (w/v) aqueous solution of starch was prepared.

Preparation of sample solution Caffeine and theophylline were dissolved in hot water (10 ml) while xanthine theobromine and uric acid were first dissolved minimum volume of NaOH solution and make 100 ml in volumetric flask with distilled water.

General Procedure: An aliquots containing 1–5 mg of the sample was placed in a 100 ml iodine flask followed by the addition of 5 ml glacial acetic acid, 10 ml 0.05 N solution of bromine monochloride was added to it and the contents were shaken thoroughly. The flask was stoppered, placed in a ice bath containing ice-salt mixture and allowed to react for 15 minutes with occasional shaking. After the reaction was over the stopper was washed with 5 ml of distilled water and 10 ml of potassium iodide (15%) was added to it. Contents were shaken throughly and kept for a minute. The liberated iodine was titrated with standardized 0.02 N sodium thiosulphate solution using starch as indicator. A blanck experiments was also run under identical conditions using all the reagent except the sample.

Calculation

$$\text{mg of sample} = \frac{(B - A) \times M \times N}{2 \times n}$$

A = ml of hypo for sample,
B = ml of hypo for blank,
N = normality of hypo soln.,
M = molecular weight of the sample, and
n = moles of BrCl for the sample.

RESULTS AND DISCUSSION

The recommended method is quick, convenient and easy in operation for the determination of purine derivatives has successfully been achieved (Table-1). The accuracy of method is within ± 1% error. In all the cases the reaction was completed within the prescribed time limit. The stoichiometry and recovery does not change by giving more reaction time. It was observed that the two time excess of bromine monochloride reagent was always essential for quantitative and constant results.

A survey of literature reveals that the mechanism of the reaction with different reagents has not been very much studied in purine compounds. As indicated earlier the dotermination of purine compounds with the use of N-Bromo-succinimide and *n*-bromosaccharine respectively.

The effect of concentration of bromine monochloride, amount of glacial acetic acid, the reaction time and the stoiochiometry of the reaction were studied for all the compounds, Bromination of xanthine, caffeine, theophylline, theobromine and uric acid needs 15 minutes for complete reaction. Within the prescribed reaction time xanthine, caffeine, theophylline, theobromine consumes 3 equivalents of bromine monochloride while uric acid consumes only 2 equivalents.

TABLE 1
MICRODETERMINATION OF PURINE DERIVATIVES WITH BROMINE MONOCHLORIDE WITH THE RECOMMENDED METHOD

S. No.	Sample	Amount taken (mg)	Stoichiometry	Amount recovered (mg)	Reaction time	Error %
1.	Xanthine	1.0000	3	1.0076	15	+0.76
		3.0000		3.0096		+0.32
		5.0000		5.0249		+0.49
2.	Caffeine	1.0500	3	1.0577	15	+0.73
		3.1500		3.1732		+0.73
		5.2500		5.2887		+0.73
3.	Theobromine	1.0500	3	1.0396	15	–0.99
		3.1500		3.1464		–0.11
		5.2500		5.2531		+0.05
4.	Theophylline	1.0400	3	1.0350	15	–0.48
		3.1200		3.1182		–0.05
		5.2000		5.2013		+0.02
5.	Uric acid	1.0000	2	1.0004	15	+0.64
		3.0000		3.0014		+0.04
		5.0000		5.0023		+0.04

Taking into consideration of stoichiometry and the nature of reaction it is proposed that the xanthene, caffeine, theophylline, theobromine get brominated to form corresponding tribromo derivatives while uric acid only gives rise to dibromo derivatives. The above mechanism of the reaction finds support from the previous relevant references[5].

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