

Determination of Barbituric Acid using Weisz — Ring Oven Technique.

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Summary: a method has been reported through which barbituric acid can be determined within the range from 0.50 to 10.00 μg with error of $- 4.83$ and $- 2.90\%$ in case of Segment Technique and Standard Scale methods respectively, using the Weisz Ring-Oven method. Effect of interferences by sodium barbitone, guanidine carbonate urea and ethyl malonate have also been studied. The method is quite suited to routine analysis of the substance for its purity checking.

Introduction

Barbituric acid, which does not directly possess hypnotic properties, has been used to synthesise several hundred of its derivatives, of which over 50 have been introduced as therapeutic agents. In view of its importance as a basic raw material for such important preparations there is always a good scope of a reliable method of its purity determination. Barbituric acid gives colour reaction with sodium nitrite (1). The reaction has not been studied earlier employing this technique hence it is the basis of the study.

Experimental

Reagents

Barbituric acid stock solution. Stock solution of barbituric acid was prepared by dissolving 0.5 g of the laboratory grade reagent (BDH) in 100.0 ml distilled water. Standard solutions for the Segment Technique (2-3) were prepared by the exact dilutions of the stock solution. These solutions were named I, II, IV, VI, VII and X, and contained 0.50, 1.00, 2.00, 3.00, 4.00 and 5.00 μg per μl of the substance respectively.

Sodium nitrite solution. Sodium nitrite 1.0g analytical grade (BDH) was dissolved in 100.0 ml distilled water and used as such.

Guanidine Carbonate. A 0.1 % aqueous solution was prepared and further diluted to give a solution containing 5 $\text{ng}/\mu\text{l}$.

Sodium barbitone. An aqueous solution of sodium barbitone was prepared by dissolving 0.1 g of

the substance (BDH) in 100.0 ml distilled water and diluted to give a solution containing 5.0 $\text{ng}/\mu\text{l}$.

Urea. A 10.0% aqueous solution of urea was used in the experiments.

Ethyl Malonate. An alcoholic solution of ethyl malonate was prepared by dissolving 0.3 g of the substance (Merck) in 100.0 ml of absolute alcohol and diluted to give a solution containing 0.03 $\mu\text{g}/\mu\text{l}$.

Apparatus The Weisz Ring-Oven, with 110°C working temperature, automatic filling micropipettes of 1 μl and 2 μl capacity (Karl - Kolb Scientific and Technical supplies, Buchsclag, Frankfurt) and Whatman Filter paper No: 41 were used for these studies.

All other reagents used were also of analytical grade and glassware was A-Grade.

Procedure. According to the Segment Technique, marked three points around the centre of filter paper so as to make an equilateral triangle. To one of these points, 1 μl of the unknown solution and to the other two points, two standard solutions were spotted at their respective places. Thereafter 2 μl of sodium nitrite solution was applied at the centre of this paper and while placing it at the ring-oven washing with distilled water was done as usual (2-3). Three red colour segments were developed during washing. The evaluation of these segments was done according to the already described procedure (2-3). Two more determinations were made taking various volumes of the unknown solution and final results were calculated (2-3). About 4 washings with distilled water were more than sufficient to completely wash the products to the ring-zone. The unknown samples were tested also using the usual standard scale method. The effect

Table 1. Determination of Barbituric Acid with Segment Technique and with Universal Scale Methods

Standard Scale Method			Segment Technique Method		
Barbituric acid taken μg	Barbituric acid found μg	Error %	Barbituric acid taken μg	Barbituric acid found μg	Error %
0.50	0.49	- 2.00	0.50	0.48	- 4.00
1.00	1.00	± 0.00	1.00	1.03	+ 3.00
2.00	2.00	± 0.00	2.00	2.00	± 0.00
2.50	2.43	- 2.80	2.50	2.50	+ 2.40
3.50	3.43	- 2.00	3.50	3.50	± 0.00
4.00	4.00	± 0.00	4.00	4.00	± 0.00
6.00	6.00	± 0.00	6.00	5.71	- 4.83
7.50	7.50	± 0.00	7.50	7.40	- 1.33
8.50	8.50	± 0.00	8.50	8.50	± 0.00
10.00	9.71	- 2.90	10.00	10.00	± 0.00

Table 2. Effect of Interferences on the Determination of Barbituric Acid

Barbituric acid taken μg	Interfering substance taken ηg	Barbituric acid found μg	Error %
2.00	5.00 Sodium barbitone	1.60	- 20.00
2.00	25.00	1.00	- 50.00
2.00	50.00	0.50	- 75.00
2.00	5.00	2.00	± 0.00
2.00	8.00 Guanidine carbonate	1.90	- 5.00
2.00	10.00	1.76	- 12.00
2.00	20.00	1.00	- 50.00
2.00	10.00 μg	2.00	± 0.00
2.00	20.00 μg Urea	2.00	± 0.00
2.00	100.00 μg	2.00	± 0.00
2.00	0.001 μg	2.00	± 0.00
2.00	0.005 μg Ethyl malonate	2.00	± 0.00
2.00	0.010 μg	2.00	± 0.00
2.00	0.020 μg	2.15	+ 7.50
2.00	0.030 μg	3.00	+ 50.00

of other substances on the determination of barbituric acid was made by the already reported method(3).

Results and Discussion

The reaction between barbituric acid and sodium nitrite is so fast that the red colour of the product is fully developed within one minute of the mixing of the reactants. The washing of the reaction product to the ring zone is also so easy that only 3-4 washings with water are more than sufficient. The colour of the rings or segments prepared in case of Standard Scale and Segment Technique respectively goes on increasing with the passage of time and the cause is not known. Therefore, the standard scale should be used only for the day it is prepared.

As apparent from the Table I, barbituric acid can be determined within the range from 0.50 to 10.0 μg with maximum error of -4.83% and -2.90% in case of Segment Technique and Standard Scale method respectively.

As shown in the Table II, sodium barbitones seriously interferes with the determination of barbituric acid while urea has no interference upto 250 folds and guanidine carbonate 20 folds less of barbituric acid. Ethyl malonate does not interfere with this determination upto its being 100 folds less than barbituric acid, thereafter the interference becomes so dominant that the results of no analytical importance are obtained.

The method reported here is recommended for the routine analysis of barbituric acid even in the presence of urea and guanidine carbonate within their prescribed limits.

References

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