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NOTE

Determination of Milligram Amounts of Thiourea, Allylthiourea and Phenylthiourea Using N-Chlorosaccharin in Acetic Acid Medium

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A quick and convenient method has been developed for the micro determination of thiourea and some of its derivatives, milligram amount of the sample is allowed to react with excess of N-chlorosaccharin at room temperature for 10 min. After the reaction is over, the unconsumed reagent is back titrated iodometrically.

A number of methods[1–9] are available for the quantitative determination of thioureas. The observation that N–chlorosaccharin in acetic acid medium readily chlorinates several groups of organic compounds[10–13], led to the development of a simple, rapid, accurate procedure for the determination of milligram amounts of thiourea, allythiourea and phenylthiourea.

Reagents and solutions

N-chlorosaccharin (0.02 M): The reagent was synthesized in the laboratory[10] and 1.0880 g of the reagent accurately weighed and dissolved in 100 mL glacial acetic acid in 250 mL volumetric flask and made up to the mark with distilled water.

Sodium thiosulphate: 0.05 N sodium thiosulphate (12.415 g) was dissolved in 1 litre of distilled water and standardised against a standard soln. of $CuSO_4$.

Potassum iodide solution: 10% m/v

Starch solution: 1% m/v

Sample solution: Stock solutions of each sample were prepared by dissolving an accurately weighed amount in glacial acetic acid in a 100 mL calibrated flask. Different aliquots of the solution were used in the determination.

Procedure

An aliquot containing 1–5 mg of the samples was placed in a 100 mL iodine flask, 5 mL of glacial acetic acid followed by 10 mL of N-chlorosaccharin solution were introduced and the flask was stoppered and shaken thoroughly. About 20 min were required for completion of the reaction. The stopper was then washed with 5 mL of distilled water; 5 mL of KI solution were added and the liberated iodine was titrated with sodium thisoulphate solution using starch as indicator. A back titration was run under identical conditions except for the addition of the sample.

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The recommended procedure was applied to the determination of thiourea, allylthiourea and phenylthiourea and the results obtained are presented in Table-1. The results are moderately precise and the maximum deviation is about ± 1%. Excess of N-chlorosaccharin should be avoided as it leads to higher results. Acetic acid[10–13] has previously been used as a solvent in various reactions of N-chlorosaccharin. The recovery studies were performed at five different levels of each sample and the results reported in Table-1 (averages of three determinations). The method is superior to other methods in speed, accuracy and simplicity.

TABLE-1
DETERMINATION OF THIOUREA, ALLYTHIOUREA AND PHENYLTHIOUREA

Sample	Amount of sample/mg		Stoichiometry	Reaction time (min)	Deviation (%)
	Taken	Recovery			
Thiourea	1.0000	1.0022	6	20	+0.22
	2.0000	2.0070			+0.35
	3.0000	3.0156			+0.52
	4.0000	4.0160			+0.40
	5.0000	4.9885			–0.23
Allythiourea	1.0000	0.9974	7	20	–0.26
	2.0000	0.9968			–0.16
	3.0000	3.0105			+0.35
	4.0000	4.0080			+0.20
	5.0000	4.9795			–0.41
Phenylthiourea	1.0000	1.0032	5	20	+0.32
	2.0000	2.0020			+0.10
	3.0000	3.0066			+0.22
	4.0000	4.0040			+0.10
	5.0000	4.9790			–0.42

In each case three determinations were done.

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