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Micro-Determination of Some Hydrazine Derivatives with N-Chlorosaccharin Reagent

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A quick and convenient method has been developed for the microdetermination of few hydrazine derivatives. Milligram amount of the sample is allowed to react with excess of N-chlorosaccharin at room temperature for 10 minutes. After the reaction is over, the unconsumed reagent is back titrated iodometrically. The accuracy of the method is within ±0.5%.

INTRODUCTION

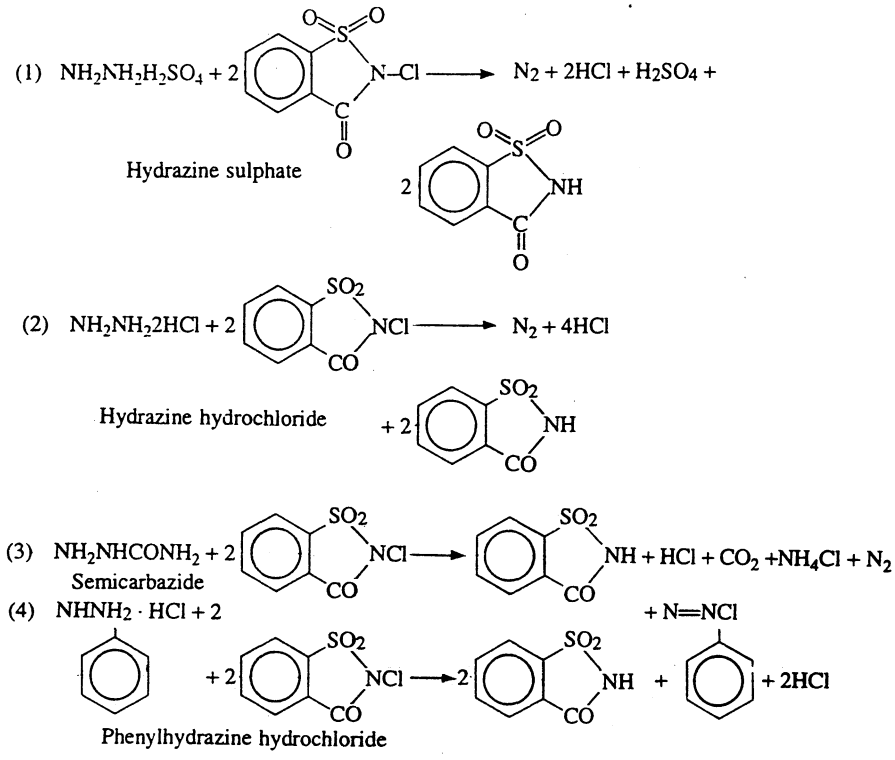
Hydrazine and its derivatives are quantitatively oxidised with organic and inorganic reagents. Depending upon this property a large number of procedures have been developed for the microdetermination of hydrazine and its derivatives.[1–5] Taking advantage of the oxidising capacity of N-chlorosaccharin[6–8] the present authors have developed a method for the small scale analysis of hydrazine derivatives. The present reagent is highly stable at room temperature and gives precise and reproducible results on micro scale within the accuracy of ±0.05% . >N–Cl bond in N-chlorosaccharin undergoes heterolytic fission in polar medium producing chloronium ion which is stronger oxidant than bromonium ion[6]. The method is superior to other existing methods[9–11].

EXPERIMENTAL

The reagent N-chlorosaccharin was synthesised in the laboratory[6, 8] and 1.0881 g of the reagent accurately weighed and desolved in 100 ml glacial acetic acid in 250 ml volumetric flask and made up to the mark with distilled water (conc. 0.02M). A stock solution was prepared by dissolving 50–100 mg of the sample in distilled water in a 100 ml volumetric flask.

Procedure

A liquid containing 1–4 mg of the sample was taken in 100 ml iodine flask and 15 ml of 0.02 solution of N-chlorosaccharin was added. The flask was stoppered and allowed to stand for 15 minutes at room temperature with occasional shaking. After the reaction was over the stoppered was washed with 5 ml distilled water and 10 ml KI was added to it. Contents were shaken thoroughly and kept for 1 minute. The liberated iodine was titrated iodometrically. A blank experiment was also run under identical condition using all the reagents except the sample and recovery was calculated[9].



RESULTS AND DISCUSSION

The proposed method has successfully been applied for the determination of hydrazine compounds with N-chlorosaccharin reagent (Table 1). It was observed that the recovery of the sample is constant for 1–4 mg of the sample size. The effect of concentration of N-chlorosaccharin reagent and reaction time was studied and the stoichiometry of the reaction was also stablished for all the compounds.

On the basis of stoichiometry and the literature[5, 9] available on the oxidation of hydrazine compounds, a possible course of reaction is also suggested.

TABLE 1
DETERMINATION OF FEW HYDRAZINE COMPOUNDS WITH N-CHLOROSACCHARIN

S. No.	Sample	Amount Present (mg)	Stoichiometry	Reaction time	Amount (mg)	% Error
1.	Hydrazine Sulphate	1.006	2	10	1.012	+0.59
		2.012			2.018	+0.29
		3.018			3.022	+0.18
2.	Hydrazine dihydrochloride	1.20	2	10	1.028	+0.78
		2.40			2.054	+0.68
		3.60			3.070	+0.32
3.	Semicarbazide	1.016	2	15	1.018	+0.19
		2.032			2.034	+0.09
		3.048			3.035	−0.42
4.	Phenyl hydrazine Hydrochloride	1.050	2	10	1.058	+0.76
		2.100			2.102	+0.09
		3.150			3.168	+0.57

In each case three determinations were done.

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