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Microdetermination of Furfural Using Bromine Monochloride

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Synopsis. A simple micro procedure for the estimation of furfural with bromine monochloride has been presented. A 2—10 mg sample dissolved in distilled water is reacted with a known excess of bromine monochloride in acetic acid in an ice bath, and the excess reagent is back titrated iodometrically. The maximum deviation in the results is ±0.90%.

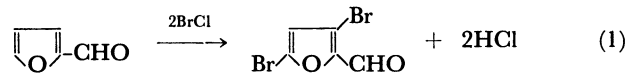
A number of methods are available for the quantitative determination of furfural depending upon the various analytical principles. Kruglikov and coworker[1] determined furfural titrimetrically with a potassium bromide–potassium bromate mixture. Spiridonova[2] reported a rapid method for the determination of furfural concentrations in non-aqueous solutions by titration with water. Similarly Nikitm and coworker[3] developed a rapid procedure for the determination of furfural by titration with water in the presence of isobutyl alcohol as a turbidity indicator. Larzlo and coworker[4] proposed a bromometric method for the determination of furfural. In the present work a new titrimetric method has been described for the microgram determination of furfural using bromine monochloride in a water–acetic acid medium.

Validity of the Reaction for the Quantitative Determination. Before applying the reaction for the determination of furfural the stoichiometry of the reaction was determined as follows: A 2—10 mg of the sample dissolved in water was reacted with a known amount of bromine monochloride solution in acetic acid. The reaction was allowed to proceed for about 15 min in an ice bath, after which the excess of the reagent was back-titrated iodometrically. Results obtained are presented in Table 1.

TABLE 1. DETERMINATION OF THE STOICHIOMETRY OF THE REACTION

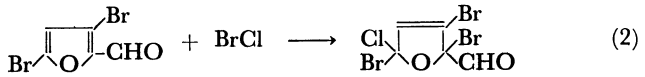
Wt. of sample (mg)	Number of moles of BrCl consumed per mole of furfural
1.999	3.002
4.996	3.000
6.994	3.004
9.992	3.006

These results show that, 3 mol of BrCl are consumed per mole of furfural. The possible explanation for this is that 2 mol of BrCl are probably used for



[A]

substitution, and then this dibromination product (A) acts as diene (just as butadiene) to add one more mole of BrCl.



[A]

This is the 1,4 addition of BrCl, and probably Br will go to the carbon 2 due to the effect of the formyl group. Furan derivatives are known to undergo easy substitution (like (1)) and also to act as diene. The effect of coexistence of impurities such as other furan derivatives has not been examined.

Experimental

Procedure. An aliquot containing 2—10 mg of the sample solution in distilled water was placed in a 100 ml iodine flask. Five ml of glacial acetic acid followed by 5 ml of bromine monochloride[5] were introduced and the flask was stoppered and shaken well. The flask was placed in an ice bath and reaction mixture allowed to cool well for about 15 min. After the reaction was over the stopper was washed with 5 ml of distilled water followed by the same volume of 15% potassium iodide solution, and the liberated iodine was titrated with 0.02 M sodium thiosulfate solution using starch as an indicator. A blank experiment was also run under the identical conditions except for the use of the sample.

Results and Discussion

The proposed method has been applied for the determination of furfural, and the results of the determination are presented in Table 2. Excess of bromine monochloride should be controlled as it leads to higher results. Acetic acid is a good solvent and reaction medium for a large variety of compounds. Cooling of the reaction mixture is necessary for obtaining good results.

TABLE 2. MICRO DETERMINATION OF FURFURAL USING BROMINE MONOCHLORIDE

Sample No.	Taken (mg)	Found (mg)	No. of Determinations	Error (%)
1.	1.999	2.016	4	+0.84
2.	4.996	5.041	4	−0.90
3.	6.994	6.936	4	−0.82
4.	9.992	10.080	4	+0.90

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