

## **N-Bromosaccharin as an Analytical Reagent for Micro Determination of Acridine Derivatives in Acetic Acid**

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In the present work, the method for the small scale determination of acridine derivatives with the use of N-bromosaccharin as an analytical reagent has been described. This gives reproducible results within the accuracy of  $\pm 1\%$ .

### **INTRODUCTION**

Reimers<sup>1</sup> introduced a photometric titration method based on the titration of alcoholic solution of acridine with sodium hydroxide. Hall and Powell<sup>2</sup> have commented on the method for the difficulty in determining the end point. Shaw and Wilkinson<sup>3</sup> suggested a colorimetric method for the estimation of proflavin hemisulphate with nitrous acid. A spectrophotometric method<sup>4</sup> has been reported for the determination of acridine and pyrene in the air in the presence of other analogous compounds. Singh, Gopal and Panday<sup>5</sup> have developed a method for small scale determination of acridine compound with N-bromosuccinamide as a direct titration. Chauhan<sup>6</sup> has also developed a new method for the milligram determination of acridine with bromine monochloride. In the present research paper, the small scale determination of acridine derivatives with the use of N-bromosaccharine has been described.

### **EXPERIMENTAL**

N-Bromosaccharin was synthesised in laboratory<sup>7</sup>. 1.3116 gm of N-bromosaccharin was dissolved in 100 ml glacial acetic acid in a 250 ml volumetric flask and made up to the mark with distilled water. The solution was standardised against standard solution of sodium thiosulphate<sup>8</sup>.

A stock solution of sodium thiosulphate was prepared by dissolving 2.4820 gm of sodium thiosulphate in the distilled water in a 1 litre volumetric flask. The solution was standardised with 0.02 M copper sulphate solution.

10% (w/v) aqueous solution of potassium iodide was prepared.

#### **Preparation of Sample Solution**

Stock solutions of acridine compounds were prepared by dissolving accurately weighed amount of the samples in distilled water in 100 ml

calibrated flask. Aliquots from the stock solution containing 1 to 5 mg of the sample were used for each determination.

### General Procedure

An aliquots containing 1–5 mg of the sample was taken in 100 ml iodine flask and 15 ml of 0.02 M solution of N-bromosaccharin was added. The flask was stoppered and allowed to stand for 5 minutes at room temperature, with occasional shaking. After the reaction was over the stopper was washed 5 ml of distilled water and 10 ml of potassium iodide was added to it. Contents were shaken thoroughly and kept for one minute. The liberated iodine was titrated with standardised 0.01 M sodium thiosulphate solution using starch as indicator. A blank experiment was also run under identical conditions using all the reagents except the sample.

### Calculation

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

S = ml sodium thiosulphate consumed for sample

B = ml sodium thiosulphate consumed for blank

M = Molecular weight of the sample

N = Molarity of sodium thiosulphate solution

n = Moles of N-bromosaccharin consumed for the sample.

## RESULTS AND DISCUSSION

With the recommended procedure the determination of acridine compounds has successfully been achieved on 1–5 mg of sample size within the accuracy of + 1% (Table 1) in most of the cases.

Before applying the method for acridine compounds, the reaction conditions were developed with acriflavine, effect of N-bromosaccharine concentration, reaction time, reaction temperature and concentration of acetic acid were studied. It was found that 0.02 M concentration of N-bromosaccharine reagent is sufficient for quantitative results. The bromination of all the compounds could be completed within 5 minutes. The method is applicable for the higher sample size without loss of accuracy and reproducibility.

The stoichiometry of the reaction was established for every compound by reacting 1–5 mg of the sample for different intervals of reaction time at room temperature with a calculated excess of N-bromosaccharine reagents. In this way it was found that the consumption of N-bromosaccharine for acriflavine, acridine orange, acridine yellow and acridine red was 3, 4, 4 and 2 moles respectively.

TABLE 1  
SMALL SCALE DETERMINATION OF ACRIDINE DERIVATIVES WITH  
THE RECOMMENDED PROCEDURE

Sample	Amount taken (mg)	Reaction time (min)	Amount recovered (mg)	Stoichiometry	Error
Acridiflavine	1.0160	5	1.0200	4	+0.39
	3.0480		3.0540		+0.19
	4.0640		4.0550		-0.22
Acridine orange	1.0200	5	1.0260	4	+0.58
	3.0600		3.0780		+0.58
	4.0800		4.1000		+0.49
Acridine yellow	2.0120	5	2.0180	4	+0.29
	4.0240		4.0260		+0.04
	5.0300		5.0380		+0.15
Acridine red	1.0240	5	1.0300	4	+0.58
	3.0720		3.0800		+0.26
	4.0960		4.1000		+0.8

In each case three determinations were done.

The recommended procedures was applied for the determination of acridiflavine, acridine orange, acridine orange and acridine red. Amounts ranging from 1,2 and 3 mg were taken and three experiments were performed were each sample size. The average % error reported for acridiflavine was  $\pm 0.39$  and acridine orange  $\pm 0.58$ , Acridine yellow  $\pm 0.29$  and acridine red  $\pm 0.58$ . On the whole the deviation of the results is within  $\pm 1\%$ .

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