DETERMINATION OF ACRIDINE IN NON-AQUEOUS MEDIA

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ABSTRACT

Acridine dissolved in the mixed solvent, ketone-acetic acid was determined by titrating with chlorosulphonic acid dissolved in the same solvent. The end points were located by potentiometric, visual and photometric methods. The first method gave results with an error less than 1 per cent. and in the other two methods, the errors are less than 2 per cent in the estimation of semi-micro quantities of acridine.

INTRODUCTION

Very few methods are available for the determination of acridine which has valuable chemotherapeutic properties. It was estimated gravimetrically as the picrate and volumetrically by titration in aqueous ethanol with sulphuric acid using phenolphthalein as the indicator. Khmelevskii et al. devised a method of estimating acridine by using aqueous sodium bisulphite. Deal et al., reported on the potentiometric titration of acridine by perchloric acid in neutralised mixture of chlorobenzene-acetic acid (1:1) by employing glass-calomel electrode pair. Naidu and Krishnan carried out the potentiometric determination of acridine in acetic acid medium using perchloric acid as a titrant and chloranil-calomel electrode pair. In a recent paper, the present authors showed that chlorosulphonic acid could be used as a titrant in the place of perchloric acid for the accurate determination of alkali metal acetates in the mixed solvent made up of a ketone and acetic acid. In the present investigation an attempt is made to determine acridine in ketone-acetic acid medium employing chlorosulphonic acid as a titrant, the end-point being determined by potentiometric, visual and photometric methods. The amount of acridine is calculated from the following reaction which is analogous to the reaction of chlorosulphonic acid with other heterocyclic nitrogen bases:

\[ C_{13}H_{9}N + HSO_{3}Cl \rightarrow C_{13}H_{9}NSO_{3}H^+ + Cl^- \]
**ExPERIMENTAL**

**Reagents**

(a) **Ketone.**—Acetone and methyl ethyl ketone (B.D.H.) were used directly without further purification.

(b) **Acetic acid.**—Acetic acid (B.D.H.) was purified by the method described.

(c) **Chlorosulphonic acid.**—Chlorosulphonic acid (B.D.H.) was used directly.

(d) **Acridine.**—Acridine (Riedel) was used directly for the determination.

(e) **Sodium acetate.**—Sodium acetate (B.D.H.) was used as a primary standard.

(f) **Indicators.**—The indicators employed were methyl orange (B.D.H.), dimethyl yellow (B.D.H.), tropaeolin 00 (E. Merck) and methyl red (B.D.H.).

**PROCEDURE**

**Preparation of acid solution.**—A stock solution of chlorosulphonic acid in acetic acid (ca. 2 M.) was prepared by adding acetic acid (100 ml.) to cooled chlorosulphonic acid (5 ml.). The stock solution was subsequently diluted with one of the ketones to obtain a solution of required strength. The acid solution was standardised with the primary standard, sodium acetate, dissolved in the same solvent, the end-point being determined either potentiometrically, or with methyl orange indicator to a bright pink end-point.

**Preparation of acridine solution.**—0.5 molar solution of acridine in acetic acid was prepared. Aliquot portions of this solution were successively diluted with the ketone to get a series of concentrations.

**Potentiometric titrations.**—Potentiometric titrations of acridine were performed with Elico pH meter employing glass-calomel electrode pair. Titrations carried out in the mixed solvent consisting of 5, 10 and 20 volumes of methyl ethyl ketone to one volume of acetic acid gave rise to irregular potentiometric titration curves with asymmetric plateau. Hence potentiometric titrations of acridine were carried out in methyl ethyl ketone-acetic acid (1:1) medium.

Characteristic S-shaped curves as shown in Fig. 1 were obtained by plotting EMF values against millilitres of chlorosulphonic acid added. The
exact end-point was determined either by calculation method\textsuperscript{10} or by locating the maxima on first derivative curve. Amounts of acridine ranging from 45–90 mg were estimated by this method and the results are presented in Table I.

**Table I**

*Potentiometric titration of acridine*

<table>
<thead>
<tr>
<th>Amount ($\times 10^{-3}$ gm)</th>
<th>Error (%)</th>
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</thead>
<tbody>
<tr>
<td>Taken</td>
<td>Found</td>
</tr>
<tr>
<td>----------------</td>
<td>-----------</td>
</tr>
<tr>
<td>8.960</td>
<td>8.978</td>
</tr>
<tr>
<td>6.720</td>
<td>6.730</td>
</tr>
<tr>
<td>4.480</td>
<td>4.514</td>
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</tbody>
</table>

![Diagram](image)

**Fig. I.** Potentiometric Titration of

(A) $2.5 \times 10^{-3}$M. Acridine with $1 \times 10^{-3}$M. Chlorosulphonic acid

(B) $5 \times 10^{-3}$M. Acridine with $1 \times 10^{-3}$M. Chlorosulphonic acid
Visual titrations.—The indicators employed in the visual titrations of acridine included methyl orange, dimethyl yellow, tropaeolin 00 and methyl red. The solvent medium used was acetone-acetic acid (20 : 1), as the colour changes of these indicators were found to be sharp as compared to those with other proportions of the components of the mixed solvent.

2–3 drops of 0.05–0.1 per cent indicator solution in the same mixed solvent, were added to about 20 millilitres of acridine solution and titrated with chlorosulphonic acid to a bright pink end-point. Amounts of acridine ranging from 14 to 28 milligrams were estimated by this method and the results obtained are presented in Table II.

**Table II**

*Visual titration of acridine*

<table>
<thead>
<tr>
<th>Indicator</th>
<th>Amount ($\times 10^{-2}$ gm)</th>
<th>Error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Taken</td>
<td>Found</td>
</tr>
<tr>
<td>Methyl orange</td>
<td>1.376</td>
<td>1.377</td>
</tr>
<tr>
<td>Dimethyl yellow</td>
<td>2.752</td>
<td>2.758</td>
</tr>
<tr>
<td>Tropaeolin 00</td>
<td>2.293</td>
<td>2.253</td>
</tr>
<tr>
<td>Methyl red</td>
<td>1.834</td>
<td>1.874</td>
</tr>
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</table>

Photometric titrations.—A systronix photoelectric colorimeter with colour filter having a peak wavelength of 5200 Å was used for the photometric determination of acridine by titrating with chlorosulphonic acid in acetone-acetic acid (20:1) medium using methyl red as indicator.

Measured absorbance values were plotted against millilitres of chlorosulphonic acid added. A typical photometric titration curve is shown in Fig. 2. Amounts of acridine ranging from 1 to 4 milligrams were estimated by this method and the results are presented in Table III.

**DISCUSSION**

The results presented in Tables I–III show that semi-micro quantities of acridine can be accurately determined in ketone-acetic acid medium by...
titrating with chlorosulphonic acid, the end-point being determined by potentiometric, visual and photometric methods.

**Table III**

*Photometric titration of acridine*

<table>
<thead>
<tr>
<th>Amount ($\times 10^{-4}$ gm.)</th>
<th>Error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Taken</td>
<td>Found</td>
</tr>
<tr>
<td>35.840</td>
<td>35.450</td>
</tr>
<tr>
<td>17.920</td>
<td>17.710</td>
</tr>
<tr>
<td>8.960</td>
<td>8.792</td>
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In the potentiometric method, 45 mg of acridine could be estimated with an error of less than 1 per cent.

![Fig. 2. Photometric Titration of $5 \times 10^{-3}$M. Acridine with $5 \times 10^{-3}$M. Chlorosulphonic acid.](image)
In the visual titrations as little as 14 mg of acridine could be determined. With methyl orange and dimethyl yellow the error was less than 0.5 per cent, whereas with methyl red and tropacolin 00 the errors were 2 per cent.

The photometric method can be employed to determine still lower amounts of acridine (as low as 1.0 mg) with an error of 2 per cent.

Acknowledgements

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References