

# THE SPECTROPHOTOMETRIC DETECTION AND ESTIMATION OF PROCAINE HYDROCHLORIDE IN AQUEOUS SOLUTION

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## INTRODUCTION

THE identification and estimation of procaine hydrochloride in aqueous solution can be accomplished by well established methods.<sup>1</sup> Nevertheless the analysis is a lengthy procedure and limited in accuracy by the amount of material available for examination, and samples, such as those from anaesthesia cases where an enquiry is instituted into the state of the anaesthetic, are often insufficient for analysis by the recognised method.

We have described<sup>2</sup> an investigation of absorption spectra in the ultra-violet as an analytical method for substances normally encountered in toxicological work. Elvidge<sup>3</sup> has studied the absorption spectra of procaine hydrochloride; we have redetermined the absorption spectra of this substance and found it to possess pronounced and characteristic absorption bands in the ultra-violet region, suitable for its identification and estimation in aqueous solution. In addition the absorption spectra of solutions of partly decomposed procaine hydrochloride have been examined and the degree of decomposition determined spectroscopically.

## EXPERIMENTAL

The absorption spectra in these experiments were determined on a Beckmann spectrophotometer as described in another paper.<sup>2</sup>

Procaine hydrochloride B.P. was dissolved in distilled water and diluted to give a concentration of 0.002 per cent. w/v. The absorption spectrum in the range 2000Å to 3500Å was determined and the results obtained are shown as curve 1 in Figure 1. Several different samples of procaine hydrochloride were examined in this way and the spectral absorption curves were found to be practically identical.

Aqueous solutions of procaine hydrochloride were examined over the concentration range 0.002 to 0.00025 per cent. w/v and Beer's Law was found to hold as the extinction coefficients given in Table I show.

TABLE I

Concentration per cent.	Extinction coefficients			
	2090 (min.)	2210 (max.)	2400 (min.)	2900 Å (max)
0.002	200	320	58	670
0.001	218	315	42	660
0.0005	220	316	60	668
0.00025	236	320	60	668

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Procaine hydrochloride was dissolved in water to give a solution of 0.1 per cent. w/v and the solution was divided into 3 parts. The first was examined immediately, the second was ampouled and kept in a refrigerator for 3 months and the third was ampouled and left in diffused sunlight for

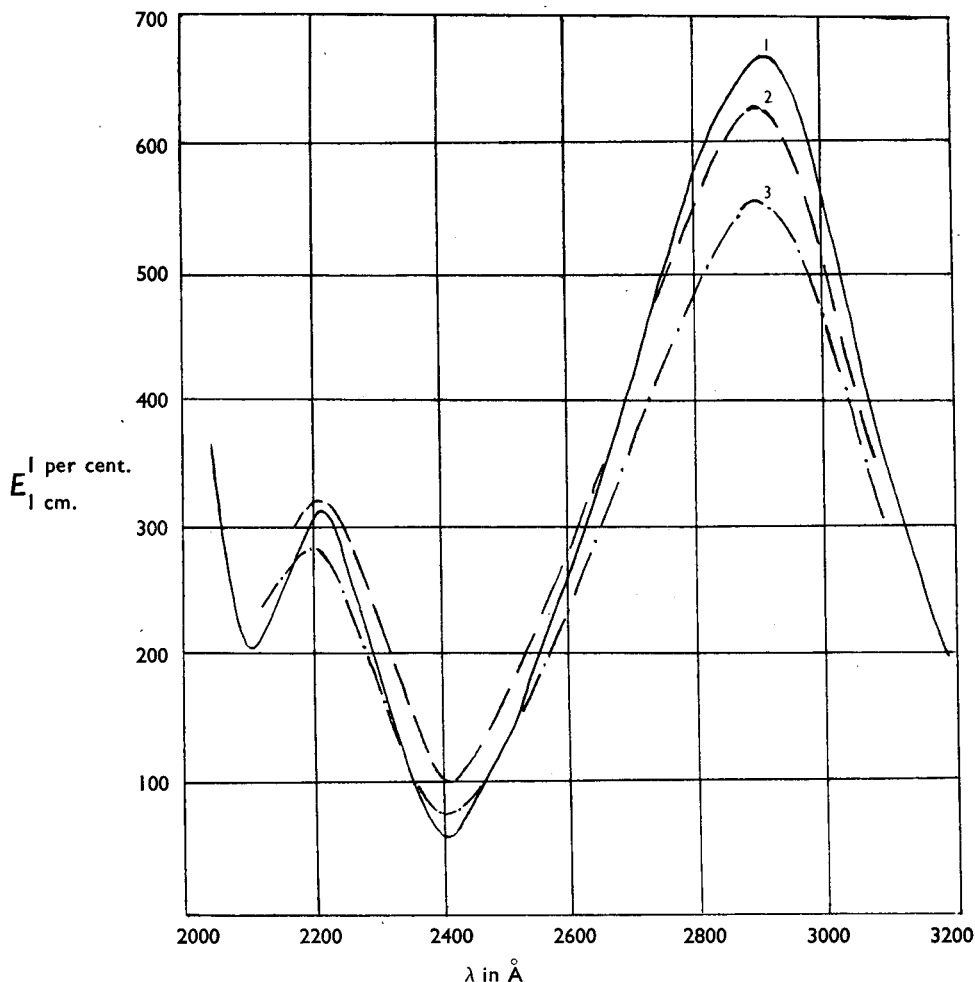


FIG. 1. 0.002 per cent. w/v of procaine hydrochloride in aqueous solution.

1. ——— solution freshly prepared.
2. - - - solution left in refrigerator for 3 months.
3. - · - · - solution left in diffused sunlight for 3 months.

the same time at room temperature (about 27° C.). After this period the second and third batches were diluted to 0.002 per cent. w/v and examined spectrophotometrically. The absorption curves are shown in Figure 1.

As an additional check on the decomposition of procaine hydrochloride a 0.1 per cent. w/v aqueous solution was ampouled and autoclaved under

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the following conditions:—(a) 40 minutes at 10 lb. steam pressure; (b) 1 hour at 10 lb.; (c) 2 hours at 10 lb.; (d) 40 minutes at 15 lb. After autoclaving, these solutions were protected from light until ready for examination. The solutions were then diluted to 0.002 per cent. w/v and examined spectrophotometrically: the results are shown in Figures 2 and 3.

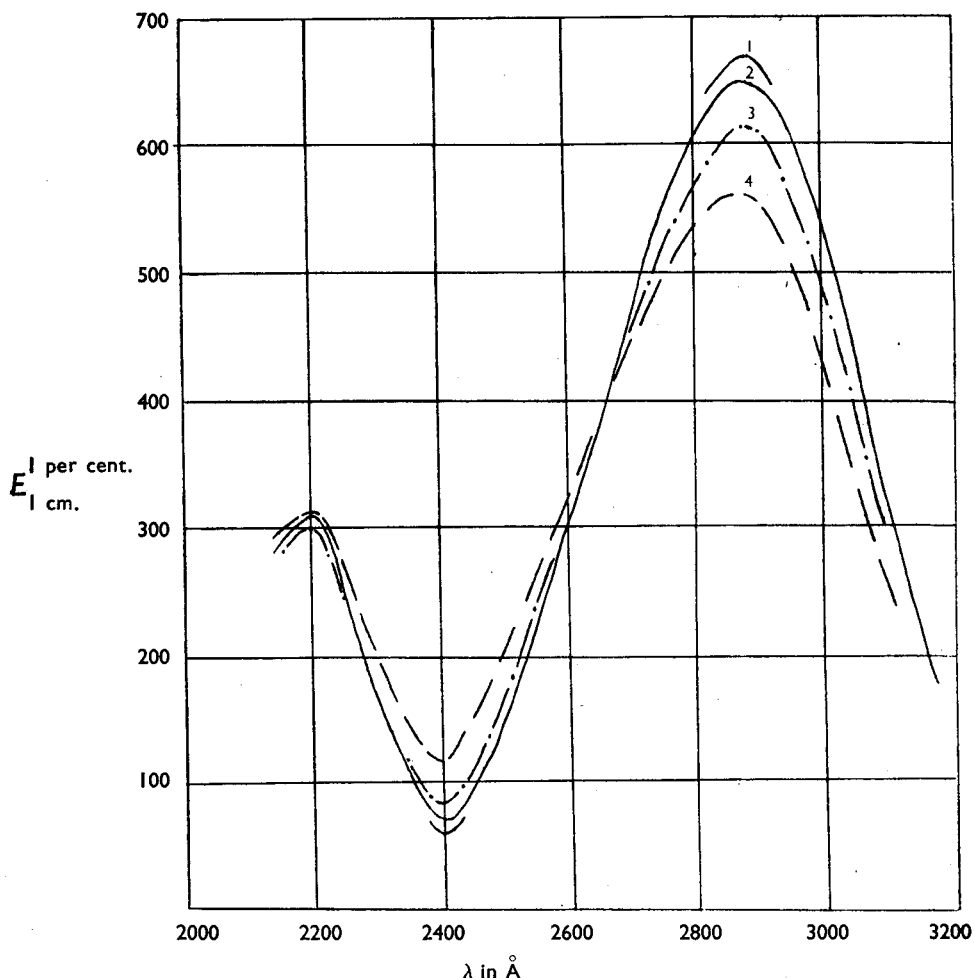


FIG. 2. 0.002 per cent. w/v of procaine hydrochloride in aqueous solution.

1. ——— solution freshly prepared (portion of curve).
2. - - - - solution autoclaved at 10 lb. steam pressure for 40 minutes.
3. - · - · - solution autoclaved at 10 lb. steam pressure for 1 hour.
4. - - - - solution autoclaved at 10 lb. steam pressure for 2 hours.

### DISCUSSION

The absorption curve for procaine hydrochloride reveals a pronounced absorption band with maxima at 2900 Å and 2210 Å and minima at 2400 Å

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and 2090Å. The  $E_{1\text{ cm.}}^{1\text{ per cent.}}$  extinction coefficients at 2900Å and 2210Å are 670 and 320 respectively. Both these values are extremely high and together with the shape of the absorption curve and its well defined maxima and minima are distinctive of procaine hydrochloride and eminently

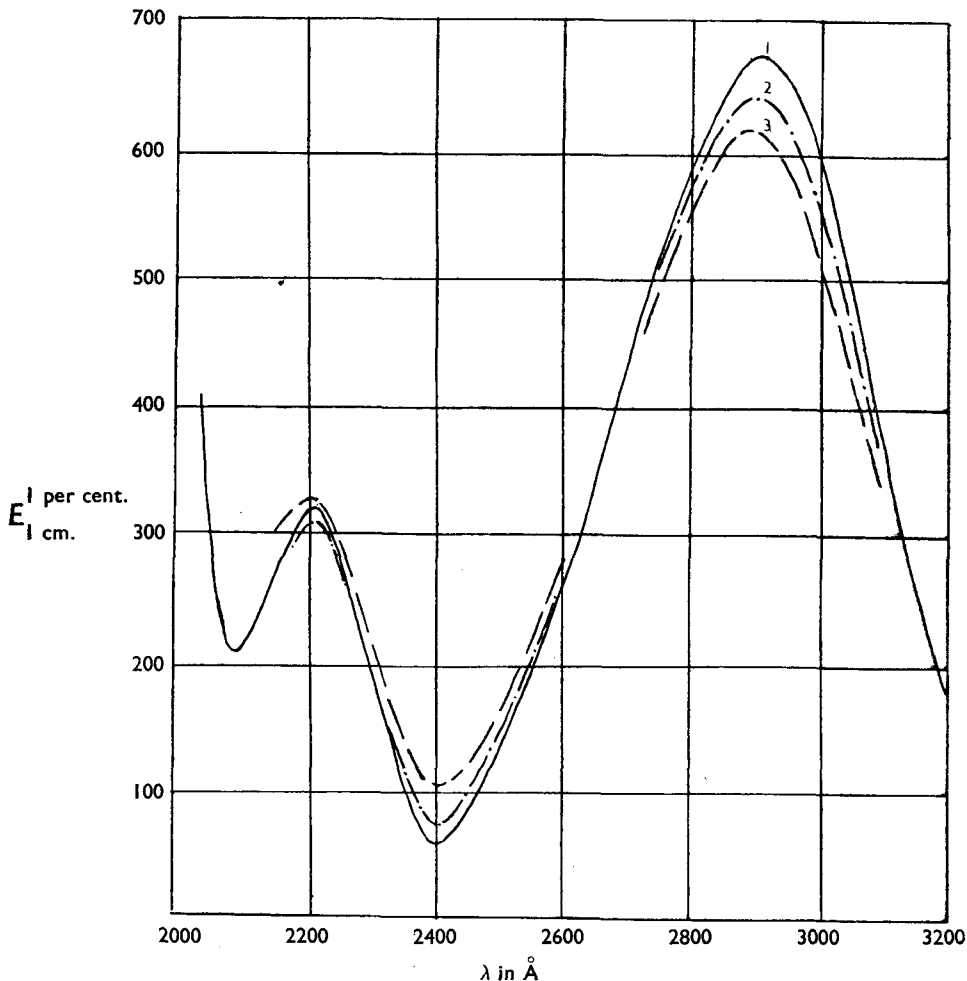


FIG. 3. 0.002 per cent. w/v of procaine hydrochloride in aqueous solution.

1. ——— solution freshly prepared.
2. - · - · - solution autoclaved at 10 lb. steam pressure for 20 minutes.
3. - - - solution autoclaved at 15 lb. steam pressure for 40 minutes.

suitable for the identification of this substance. This is particularly true of the maxima at 2900Å which shows a progressive diminution in  $E_{\text{max}}$  with increasing severity of treatment of the solution. The fact that Beer's Law is applicable at least between 0.002 and 0.00025 per cent. w/v, suggests that the method has quantitative use.

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The absorption curves in Figure 1 show the effect of time and atmospheric conditions on ampouled samples of aqueous procaine hydrochloride solutions. The solution which was kept in the dark and at a temperature of approximately 40° F. was found to be slightly decomposed and that which had been exposed to diffused sunlight at room temperature had decomposed even further. Use was made of the  $E_{\max}$  at 2900Å to calculate the degree of decomposition, and the analytical results are shown in Table II.

TABLE II  
DECOMPOSITION OF PROCAINE HYDROCHLORIDE SOLUTION

Substance	Decomposition per cent.
Left in refrigerator for 3 months .. .. .	5.2
Left in open for 3 months .. .. .	17.0

The absorption curves in Figures 2 and 3 show the results obtained when aqueous solutions of procaine hydrochloride were subjected to varying conditions of steam pressure and time of autoclaving. They indicate that excessive steam pressure and autoclaving times lead to a progressive amount of decomposition of the procaine hydrochloride. The extent of decomposition estimated from the  $E_{\max}$  values at 2900Å is recorded in Table III.

TABLE III  
EFFECT OF AUTOCLAVING CONDITIONS ON DECOMPOSITION

	Decomposition per cent.
40 minutes at 10 lb. steam pressure .. .. .	3.7
1 hour at 10 lb. .. .. .	8.1
2 hours at 10 lb. .. .. .	15.6
40 minutes at 15 lb. .. .. .	8.1

As a final experiment a sample of procaine hydrochloride which had been stored in the solid state for 6 years in the tropics was made up to an 0.002 per cent. w/v solution. The following extinction coefficients were measured and the purity of the sample estimated from the values of each maximum:— $E_{1\text{ cm.}}^{1\text{ per cent.}}$  at 2210Å, 287 = 90 per cent.  $E_{1\text{ cm.}}^{1\text{ per cent.}}$  at 2900Å, 595 = 89 per cent.

These experimental data indicate that procaine hydrochloride may be both identified and estimated spectroscopically in amounts as small as 0.06 mg. The same sample may be used for both identification and estimation, the method is quick and reliable and should be of considerable value in analysing small quantities of dilute procaine hydrochloride solutions in water.

### SUMMARY

1. Aqueous solutions of procaine hydrochloride both pure and partially decomposed, have been examined spectrophotometrically and the experimental absorption bands recorded.

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2. The use of the absorption bands occurring in the far ultra-violet region of the spectrum for identifying and estimating this substance has been discussed.

I am indebted to Professor R. A. Robinson for his interest and assistance in this work and I also wish to thank Mr. A. W. Burt, Chief Chemist, Singapore, for his help.

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