

and was identified as β -sitosterol by comparison with an authentic sample (m.m.p. and I.R.).

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1. Gamble, J. S., 'Flora of the Presidency of Madras', Botanical Survey of India, Calcutta, 1967, Vol. I, p. 79.
2. Sankara Subramanian, S. and Narayana Swamy, M., *Curr. Sci.*, 1963, 32, 308.
3. Anjaneyulu, A. S. R. and Suryanarayana Murthy, V., Communicated to *Planta Medica*.

POTENTIOMETRIC TITRATIONS OF BENZEDIAMINES IN NON-AQUEOUS MEDIUM

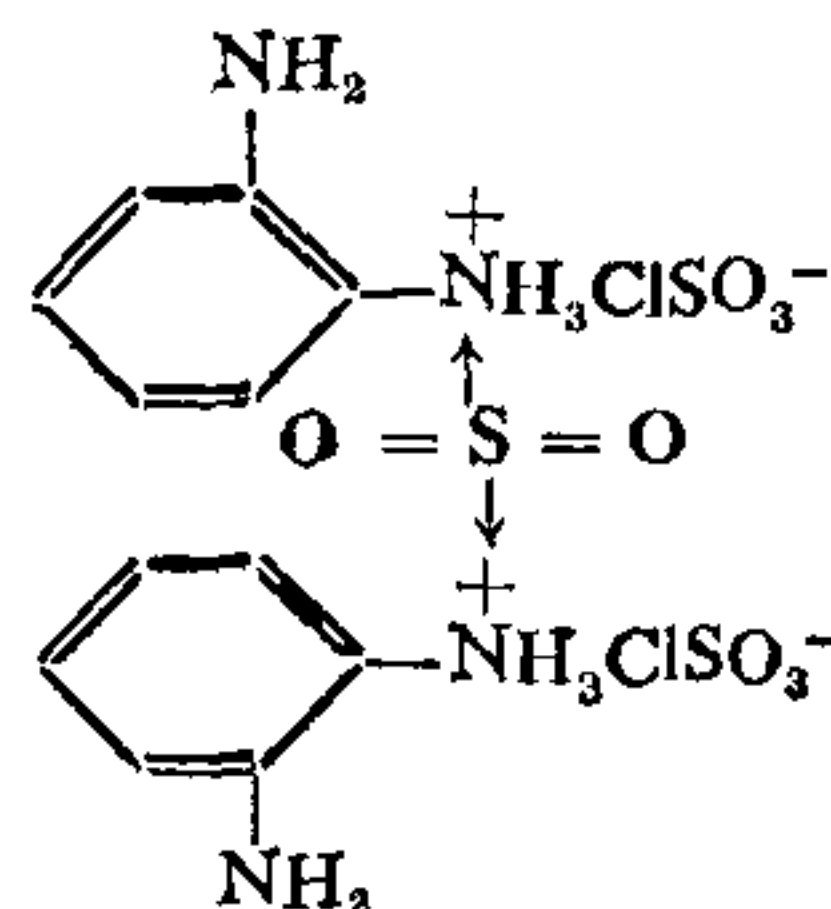
BENZEDIAMINES were determined potentiometrically in methylethyl ketone by Kreshkov and Aldarova¹. Kreshkov *et al.*² carried out differential titrations of mixtures of diamines in chloroform-methylethyl ketone medium. Both the groups employed perchloric acid as a titrant. The results reported by the former group were associated with an error of 2.6% and the error reported by the latter group was 3%. In the present investigation the isomeric benzene diamines have been determined potentiometrically in acetic acid-methylethyl ketone medium using chlorosulphonic acid as a titrant. The advantages of using chlorosulphonic acid in place of perchloric acid have been described earlier by Rao and Naidu^{3, 4}.

Chlorosulphonic acid (BDH) was used without further purification. Acetic acid (BDH) and methylethyl ketone (BDH) were purified by the method used by Rao and Naidu³. The three benzenediamines were purified by the method recommended by Vogel⁵.

Approximately 1M stock solution of chlorosulphonic acid was prepared by adding 7 ml of the acid to 250 ml of chilled acetic acid. The solution was then diluted with methylethyl ketone to obtain the solution of required concentration. The acid solution was standardised potentiometrically employing anhydrous sodium carbonate as the primary standard. Weighed samples of bases were dissolved directly in the mixed solvent.

Titration of bases dissolved in 1:1 acid-ketone medium were carried out using the Elico pH meter. The exact end point was obtained by the calculation method given in the literature⁶. Base to acid mole ratio was found to be 1:2 in respect of 1, 3 and 1, 4 benzenediamines and the ratio was 1:1.5 in the case of 1, 2 benzenediamine. These observations

suggest that both the amino groups in 1, 3 and 1, 4 isomers react with the acid to form salt. In the 1, 2 isomer the acid base reaction appears to be restricted to only one amino group due to steric effect and the residual 0.5 mole of the acid may be ascribed to a condensation reaction as indicated below:



The experimental results are given in Table I

TABLE I

Base	Amount mg	
	taken	found
1, 2 benzenediamine	43.2	42.4
	21.6	21.2
1, 3 benzenediamine	43.2	42.4
	21.6	21.2
1, 4 benzenediamine	43.2	42.6
	21.6	21.2

The data in the table show that chlorosulphonic acid can be used successfully for the determination of semimicro quantities of the three diamines with an apparently negative bias and an error of < 2%. The method is more accurate than that reported by Kreshkov and Aldarova¹.

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1. Kreshkov, A. P. and Aldarova, N. Sh., *Izv. Vysshikh Uchebn. Zavednii, Khim. Tekhnol.*, 1965, 8, 316.
2. —, Bykova, L. N. and Pevzner, Z. D. *Zh. Obshch. Khim.*, 1965, 35, 1332.
3. Rao, K. C. M. and Naidu, P. R., *J. Electroanal. Chem.*, 1972, 35, 429.
4. — and —, *Talanta*, 1972, 19, 1465.
5. Vogel, A. I., *Text Book of Practical Organic Chemistry* (Longmans), 1951, p. 612.
6. Willard, H. H. Merritt, L. L. and Dean, J. A., *Instrumental Methods of Analysis*, (Affiliated East-West Press, New Delhi), 1925, p. 552.