Polarographic and Spectrophotometric Determination of Zinc in Zinc Stearate

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The problem of the estimation of the metal content in heavy metal soaps has been a subject of detailed study in these laboratories for some time. Working with copper, nickel and cobalt soaps, it was found that their property of forming complexes with pyridine base[1] can be advantageously put to use for estimating the metal content by polarography[2] and spectrophotometry[3]. The present communication deals with the estimation of zinc in zinc stearate by the above two methods[2,3].

All the reagents used were either A. R. samples or purified products. Stearic acid (British Drug House) was crystallized with alcohol and finally fractionated under reduced pressure. A. R. pyridine and A. R. mercury (triple distilled) were used.

Zinc stearate was prepard by the precipitation method[4]. The solution was prepared in pyridine and metal content estimated gravimetrically.

Polarographic measurements were carried out with a Fisher Electropode in conjunction with a Multiflex galvanometer model MGF2. Experiments were performed in H-type cell[5] using dropping mercury cathode and a saturated calomel electrode. Lithium chloride and methyl hydrogen sulfate in 1 : 1 benzene-methanol mixture were used as supporting electrolytes[6]. The capillary constants, $m^{2/3}t^{1/6}$, for these supporting electrolytes (0.033 M) were 1.618 and 1.532 respectively in the open circuit. Two sets of experiments were performed: (1) fixed amount of soap (5 cc., approximately 1%) and varying amounts of supporting electrolyte (1.0, 2.0, 3.0, 5.0, 10.0 cc. of 0.165 M), making total volume 30 cc.; (2) fixed amount of supporting electrolyte (5.0 cc. of 0.165 M) and varying amounts of soap (3.0, 5.0, 8.0, 10.0, 12.0 cc. approximately 1%), making total volume 30 cc. Inert atmosphere was maintained in the cell by passing pure nitrogen and experiments were performed at 25±0.1°C in thermostatic water-bath (Townson and Mercer).

The typical polarograms of zinc stearate in lithium chloride and $MeHSO_4$ (concentration 0.005 M) are shown in Fig. 1, the values of

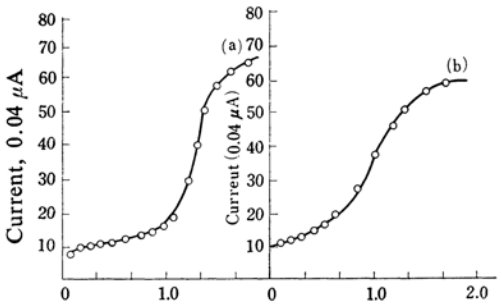

Fig. 1. Polarograms of zinc stearate pyridine complex.

a) 2.5 mM Zn^{2+} in Zn-stearate in 0.055 M LiCl

b) 2.5 mM Zn^{2+} in Zn-stearate in 0.055 M $MeHSO_4$

$E_{1/2}$ in the two supporting electrolytes being −1.4 and −0.9 V. respectively. The values of $E_{3/4}-E_{1/4}$, however, came out to be abnormally high indicating the irreversible nature of the wave. The limiting current was found to increase with increasing concentration of the soap. The values of the concentration of zinc in soap in millimoles per liter (C), the limiting current in microamperes (i_l) and i_l/C are summarized below.

Table I shows that the value of i_l/C remains fairly constant, showing thereby that the estimation of zinc can be made from these results.

TABLE I

C	0.033 M i_l	LiCl i_l/C	0.033 M i_l	$MeHSO_4$ i_l/C
1.5	2.40	1.60	1.20	0.80
2.5	4.16	1.64	2.00	0.80
3.0	4.80	1.60	2.28	0.76
4.0	6.40	1.60	3.12	0.78
5.0	7.68	1.56	3.75	0.75
6.0	9.44	1.56	4.68	0.78

1) C. J. Boner, *Ind. Eng. Chem.*, **29**, 58 (1937).
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3) W. U. Malik and R. Haque, *Z. Anal. Chem.*, in press.
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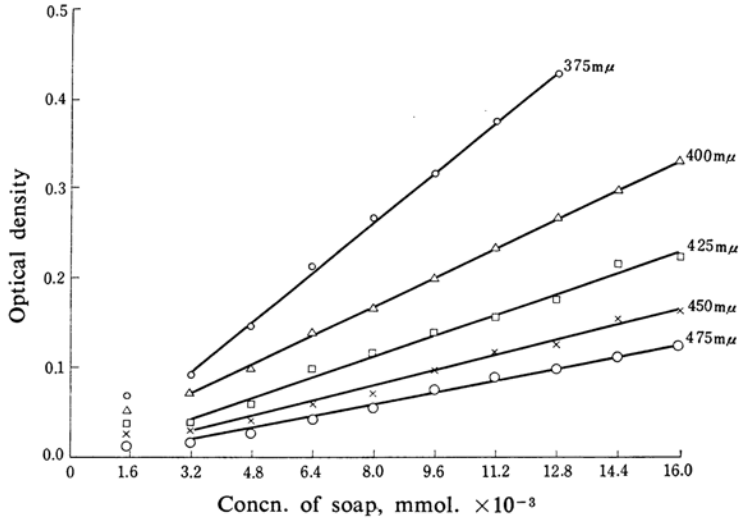

Fig. 2. Optical density as a function of concentration of soap.

Spectrophotometric measurements were carried out with a Bausch and Lomb 'Spectronic 20'. Solutions of zinc stearate of different dilutions were prepared by diluting the stock solution with pyridine. Measurements were carried out in the wavelength region 375 to 475 mμ.

The plot between the optical density and concentration of the metal in the soap gave straight lines upto a certain concentration of the soap solution (Fig. 2). Below this concentration the points did not lie on the straight line, showing thereby that the Beer's law is not obeyed for very dilute solutions and that the zinc ions can be estimated spectrophotometrically in the zinc stearate solution for concentrations higher than 3.2×10^{-3} mM.

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