# SHORT COMMUNICATIONS

## *Polarographic Determinations of Minute Quantities of Lead in High-Purity Electrolytic Zinc Using the Rotated Dropping Mercury Electrode*

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The polarographic method with a dropping mercury electrode (DME) has been widely employed for the determination of minute quantities of lead contained in high-purity electrolytic zinc. The method, though fairly accurate, is often questioned because of its insufficient sensitivity. Recently W. Stricks and I. M. Kolthoff devised a new electrode in polarography, called rotated dropping mercury electrode (RDME), and reported an extensive fundamental work on the characteristics of the electrode and also on the polarography at the electrode[1]. Because of its high sensitivity, the RDME has been applied to the polarographic determination of minute quantities of lead contained in high-purity electrolytic zinc.

The electrode used in this study was similar to that reported by Stricks and Kolthoff[1], which was driven by means of a synchronous motor (Type A, provided with gears for speeds of 100, 150, 200, 300 r. p. m., constructed by Yanagimoto Co.), and had the characteristics of 4.957 mg./sec. for $m$ and 7.22, 5.72 and 3.54 sec. for $t$ at 100, 150 and 200 r. p. m., respectively, being measured in deaerated 0.1 M potassium chloride solution at 40.0 ± 0.1 cm. of mercury head with open circuit. A saturated calomel electrode was used for a reference electrode. Current-voltage curves were measured with Yanagimoto pen-recording polarograph Model PB-4. All measurements were carried out in a thermostat of 25° ± 0.1°C.

Current-voltage curves were obtained with various concentrations of lead in deaerated 0.1 M and 1 M sodium chloride solutions containing $5\times10^{-6}$ M polyoxyethylene lauryl ether (LEO) as a maximum suppressor. A proportionality was obtained between the concentration of lead and the limiting current. The best reproducibility was found at 100 r. p. m. as shown in Table I, although the $i_l/c$ value was the highest at 150 r. p. m.

TABLE I

LIMITING CURRENTS OF LEAD IONS IN 0.1 M KCl - $5\times10^{-6}$ M LEO

| Conc. of Pb(II), M | $i_l$, μ amp. | $i_l/c$, μ amp./$10^{-4}$ M |
|---|---|---|
| $2.94\times10^{-7}$ | $0.017_2$ | $5.8_2$ |
| $0.979\times10^{-6}$ | $0.057_5$ | $5.8_8$ |
| $2.94\times10^{-6}$ | 0.172 | $5.8_3$ |
| $0.979\times10^{-5}$ | 0.573 | 5.86 |
| $2.94\times10^{-5}$ | $1.70_4$ | 5.80 |
| $0.979\times10^{-4}$ | 5.71 | 5.83 |
| $2.94\times10^{-4}$ | 17.03 | 5.80 |

In deaerated 1 M zinc chloride solutions containing $5\times10^{-6}$ M LEO the $i_l/c$ value for lead was also found constant in the concentration range investigated, but somewhat smaller than those in 0.1 M and 1 M potassium chloride solutions. High-purity electrolytic zinc was dissolved in concentrated hydrochloric acid and the excess of the acid was removed by evaporation. The residue was dissolved into distilled water containing $5\times10^{-6}$ M LEO to make 1 M in concentration in regard to zinc chloride. The concentration of lead was determined directly from the $i_l/c$ value obtained for lead in 1 M zinc chloride and also by the standard addition method. In Table II are given some of the results obtained, which

TABLE II

LEAD CONTENT IN HIGH-PURITY ELECTROLYTIC ZINC

| Sample | Content of Lead found, % | |
|---|---|---|
| | from the $i_l/c$ value | by standard addition method |
| No. 1 | $0.0007_1$ | $0.0007_1$ |
| No. 4 | $0.0012_0$ | $0.0011_8$ |
| No. 6 | $0.0015_8$ | $0.0015_4$ |
| No. 8 | $0.0013_7$ | $0.0013_6$ |
| No. 9 | $0.0011_7$ | $0.0011_2$ |
| No. 10 | $0.0001_3$ | $0.0001_3$ |

contain the determination of lead down to 0.0001 % in content of electrolytic zinc. Further studies on the characteristics of the RDME and on other applications will be reported in subsequent publications.

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1) W. Stricks and I. M. Kolthoff, *J. Am. Chem. Soc.*, **78**, 2085 (1956); See also, Nobuyuki Tanaka, *J. Japan. Chem.*, **10**, 814 (1956).

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