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STUDY TO INVESTIGATE AND IMPROVE THE ZINC ELECTRODE FOR SPACECRAFT ELECTROCHEMICAL CELLS

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ABSTRACT

Electrodeposition of zinc on slowly rotating disc electrodes was conducted. The half wave potential ($E_{\frac{1}{2}}$) on the current-potential curve was confirmed to be the critical value for change from mossy deposition to dendritic. The radius of the dendrites was measured as a function of overpotential and found to change radically around $E_{\frac{1}{2}}$.

Absorption isotherms of zincate in five separators were measured. The distribution coefficient of zincate between electrolyte and separator was measured and found to be less than one. Its value varied over a factor of five for different separators.

1. INTRODUCTION

This project is a continuation of Contract #NAS53873, Investigation and Improvement of Zinc Electrodes for Electrochemical Cells. The task of this project is to discover the foundations for control of zinc penetration through the separators.

In the previous work we established that the phenomenon of zinc penetration through the separators occurs by plating of zinc inside the separator, and not through mechanical puncture of the separator. It was found that four essentially different types of deposits can be distinguished: 1) smooth deposits, - obtained only with rotating disc electrodes at high Reynolds numbers, low overpotentials, and high concentrations of zincate; 2) moss, a soft, weak deposit of low density composed of whiskers; 3) dendrites which are hexagonal, elongated and branched crystals. The diameters of the dendrites and the density of the deposit are higher than those of whiskers; and 4) heavy sponge - a network of dendrites which is porous, rather dense and hard, with rounded edges. In cycled cells zinc deposits of types 2 and 4 are the most frequently encountered. It was found, furthermore, that on rotating (8.8 RPS) electrodes, plating of the mossy deposit takes place at overpotentials in the range of 0 to -150 mv whereas deposition of dendritic deposits takes place at more negative overpotentials. The half wave potential, therefore, is a critical potential, around which the change from one type of deposit to another type of deposit occurs.

During the previous study, absorption isotherms of zincate in separators were prepared, from which it was found that the distribution coefficient of zincate in a variety of membranes is smaller than 1. This

means that conditions for plating inside of the separator are less favorable than outside of the separator. During the present report period, an effort was made to confirm the hypothesis about two different types of deposits formed on the two sides of the half wave potential, and more analytical work has been done on absorption isotherms of zincate in separators. The separators investigated were: Du Pont PUDO-300, Silver treated PUDO-300 (Yardney Electric Corporation C-19), Mono-Sol unplasticized polyvinylalcohol (Polyfilm Corp. NYC), C-3 and 9107/5 (Borden Chemical Co.).

2. Half Wave Potential of Zincate as a Critical Parameter in Zinc Plating

It was previously established by us (1) that during plating of zinc on rotating electrodes from zincate solutions a distinct diffusion-limited current exists on voltamperometric curves. Similar diffusion-limited current can be found when plating current is taken from a series of experiments and plotted versus overpotential. It was found, further, that at potentials more positive (less than -150 mv) than the half wave potential ($E_{\frac{1}{2}}$) of current vs. potential, a mossy deposit of zinc forms, while at more negative overpotentials, the deposit is dendritic.

The above mentioned experiments suggested that mossy deposits form when deposition is activation controlled while dendritic deposits form in the region of complete diffusion control. It is worth mention here that between 5×10^{-3} molar zincate and 5×10^{-2} molar zincate, the limiting current increases linearly with concentration. $E_{\frac{1}{2}}$ stays practically constant in the entire above mentioned range of concentrations. For all concentrations tested, the mossy deposit was found at potentials more positive than $E_{\frac{1}{2}}$, and dendritic at more negative potentials.

The above experiments were done at 8.8 RPS, but when the speed was increased to 23 RPS, a virtually smooth deposit was obtained for all overpotentials up to 250 mV.

The main criterion which distinguishes mossy whiskers from dendrites is their diameter. An experiment was performed, therefore, to determine the diameter of the whiskers or dendrites as a function of zinc overpotential, and to measure plating current at the same time.

2.1 Experimental

A rotating disc cathode 1 cm. in diameter was mounted in the center of a Pyrex cell (See Figure 1) which had an external counter electrode and a zinc oxide reference electrode. The Wenking potentiostat maintained the zinc cathode at a constant potential vs. the zinc reference electrode. Zinc deposits were obtained at various overpotentials and removed from the disc, and a mean thickness of 10 whiskers was measured microscopically. The current was measured at the end of plating at approximately 190 coulombs.

2.2 Results

Most distinct differences between mossy and dendritic deposits were obtained at low rotation speeds, for example, 1 RPS. The solution used was saturated zincate in 35% KOH. Figure 1A, Curve 1, shows the dependence of current on overpotential, while Curve 2 shows the diameters of whiskers and dendrites in the same deposit. The diameters of the whiskers apparently are also dependent upon zinc overpotential; up to approximately $E_{\frac{1}{2}}$, the average diameter of the whiskers stayed practically constant, while at a potential about 50 mV more negative than $E_{\frac{1}{2}}$, a sudden change from mossy to dendritic deposit occurred. The change of diameter from 3.6 to 170 microns occurs in a very narrow range of potential. When the change to dendritic deposit occurs, there is no further increase in the diameter of the dendrites. A change of density also occurs around $E_{\frac{1}{2}}$. The density of the deposit in this experiment changed from 0.1 to 0.25 g/cm^3.

3. Absorption Isotherms of Zincate in Separators

Penetration of zinc through separators results from electrodeposition

of zinc in the separators. Electrodeposition of zinc is a function of concentration of zincate available for plating. Information about such concentrations in the separator at various concentrations of zincate in the electrolyte surrounding the separator can be presented as an absorption isotherm.

In order to discuss absorption isotherms of zincate in separators in quantitative terms, we shall utilize the following parameters:

C - Total analytical concentration of zincate in the membrane.

C_a - Part of analytical concentration of zincate bound by fibers (e.g. adsorption on fibers).

C_f - Part of analytical concentration of zincate present in interfibrillar KOH solution (concentration of free zincate).

C_o - Concentration of zincate in outer solution.

K_N - Nernst distribution coefficient.

K - Total analytical coefficient of distribution of zincate between outer solution and membrane.

K_p - Analytical coefficient of distribution of zincate between the outer solution and free electrolyte in the interfibrillar space of the membrane, $K_p = C_f/C_o$.

K_1, K_2 - Constants of Langmuir adsorption isotherm.

a_o, a_f - Activity of zincate in outer solution and in free electrolyte in the separator, respectively.

f_o, f_f - Activity coefficients of zincate in outer solution and in free electrolyte in the separator.

a, b - Constants

A', $- A = C_a/C_o$

In such a case:

11. $K = K_N (K_1 + 1)$

and

12. $C = C_o K_N (K_1 + 1)$

The absorption isotherms obtained experimentally and described below do not provide the means to separate K_1 from K_N. Therefore, we measured K only, understanding that analytically obtained partition coefficients K are equal to the Nernst partition coefficients only when adsorption is negligible.

The following relationships describe the distribution of zincate between the separators and surrounding zincate:

1. $C = C_a + C_f$

2. $K_N \overset{def}{=} \frac{a_f}{a_o} = \frac{f_f C_f}{f_o C_o} \overset{def}{=} K_p \frac{f_f}{f_o}$ (Nernst distribution law)

3. $K \overset{def}{=} \frac{C_a + C_f}{C_o} = A + K_p = A + K_N \frac{f_o}{f_f}$

4. $\log f_o = a + bC_o$; (Harned & Owen (2))

5. $\log f_f = a + bC_f$

6. $\frac{f_o}{f_f} = \exp (b(C_o - C_f))$; (from eq. 4 and eq. 5)

7. $K = A + K_N \exp (b(C_o - C_f))$

Function A is determined by the type of adsorption isotherm. For example, if adsorption is of the Langmuir type:

8. $C_a = \frac{K_1 C_f}{1 + K_2 C_f}$

and

9. $A = \frac{C_a}{C_o} = \frac{K_1}{1 + K_2 C_f} \frac{C_f}{C_o} = \frac{K_1 K_p}{1 + K_2 C_f}$

10. $K = K_p \left(\frac{K_1}{1 + K_2 C_f} + 1 \right) = K_N \left(\frac{K_1}{1 + K_2 C_f} + 1 \right) \exp (b(C_o - C_f))$

In the specific case when C depends on C_o linearly, most probably we have simultaneously, $K_2 C_f \ll 1$ and $b \ll 1$.

3.1 Experimental

The following separators were tested for absorption of zinc oxide: DuPont - 300 PUDO; YEC - Ag treated 300 PUDO (C-19); Borden Chemical Co. C-3 and 9107/5; Polyfilm Corp. - polyvinyl alcohol ("Mono-Sol", PVA).

The above separators were cut into pieces 3" x 3", weighed, soaked for three days in 45% KOH containing zincate, dried by towelling, dissolved in 10 cc of 1:1 HNO_3, evaporated and dissolved in a solution containing 1N NH_3 and 1N NH_4Cl (50 cc). The ammonia solution containing zinc was then analyzed polarographically. Concurrently, certain samples were analyzed by an alternative method which was as follows: the soaked piece was weighed and burned in a crucible, and the residue was dissolved in an ammonia buffer and analyzed polarographically with gelatin as maxima suppressor. The increase in thickness of each sample also was measured. Three samples were analyzed and measured in each experiment.

3.2 Results

Separators soaked in KOH solution swell and the quantity of KOH absorbed varies for various types of separators. The concentration of absorbed KOH does not differ much from the concentration of the surrounding KOH, but the concentration of zincate does change. The coefficient K serves as a measure of the change. To calculate K, it was necessary to measure the specific gravity of zincate solutions for the various concentrations. The results are given in Table I. The specific gravity of zincate in 45% KOH varies from 1.4520 g/cc for pure KOH, to 1.5222 g/cc for 1 M zincate.

Table II shows the absorption of zincate by cellophane 300 PUDO

soaked in zincate solution in 45% KOH. The 3" x 3" sample was analyzed by the nitric acid method. In column 1, the concentration of zincate in Moles/liter is given. Column 2 gives the thickness of the sample before and after soaking. Column 3 gives the dimensions of the sample after soaking. Column 4 gives the weight of the sample before and after soaking. Column 5 gives the difference in weight of the sample before and after soaking. Column 6 gives the absorption of zincate per sample. Column 7 gives the number of moles of zincate per kg. of dry sample before soaking. Column 8 gives the number of moles of zincate per liter of dry separator. Column 9 gives the quantity of absorbed zincate after soaking the sample Quantity is given in Moles/kg of swelled sample. Column 10 gives the quantity of absorbed zincate per liter of absorbed KOH solution after soaking. The absorption isotherm of zincate in 300 PUDO cellophane is plotted in Figures 2A and 2B. The dependence is almost linear in the entire range of concentrations; the deviation from linearity does not exceed 5%. In Figure 2A Moles of absorbed zincate per kilogram of dry membrane are plotted versus concentration of external zincate. Figure 2B shows concentration of absorbed zincate vs. concentration of external zincate in the same units. From Figure 2B the coefficient K, equal to internal over external zincate concentration, can be found. This proves to be 0.84, showing that the internal concentration is lower than the external.

Table III provides data similar to those of Table II but for silver-treated cellophane (Yardney Electric Corp., C-19). In Figure 3A the content of zincate in the separator, after soaking, is plotted versus its concentration in the surrounding zincate. Figure 4 expresses the same

experiment in terms of concentrations. The concentrations of zincate in the C-19 are lower than in the cellophane. Because it seems that K_N of cellophane should not be affected by Ag precipitated on the fibers, it would appear that the change in zincate content is due to a decrease of adsorbability on fibers, due, in turn, to a decrease in the number of adsorption centers.

Table IV gives the absorption of zincate by PVA. A sample was analyzed similarly to the previously described samples. The total Adsorption in PVA is lower than in cellophane and C-19. See Figures 4A and 4B. Table V and Figures 5A and 5B report the data obtained by a modified analytical method in which the sample was burned instead of being dissolved in HNO_3. The results obtained by both methods are practically identical.

Table VI represents absorption of zincate solution by C-3 (Borden Co.) separators (3). The content of zincate in this separator is lower than in other separators studied. Figures 6A and 6B represent changes in content and concentration of zincate. Positive deviation from linearity suggests that constant b cannot be neglected. The very low value of K suggests that the contribution of adsorption (C_a/C_o) is small so that K K_N. Data on absorption on another Borden separator, No. 9107-5, are given in Table VII and Figures 7A and 7B. The constant K is much larger for this separator (I = 0.28) but is lower than in the case of cellophane or PVA.

Table VIII summarizes the results on the distribution coefficient K for the various separators in 0.1 M zincate solution in 45% KOH. The coefficient K characterizes the content of zincate in the membrane. Because

plating in the separator is a function of this content, we assume that the value of K plays an important role in penetration. The coefficient K is largest for cellophane and smallest for C-3 (Borden) separator. The value of K, nevertheless, does not give the total picture because it is necessary to know K_N and A as well as the diffusion coefficient to predict properties of the separator with respect to zinc penetration.

4. SUMMARY AND CONCLUSIONS

Concurrent study of plating current and whisker diameter as a function of overpotential, confirmed quantitatively that the mossy deposit forms on the rotating electrode at potentials more positive than the half wave potential ($E_{\frac{1}{2}}$) of zincate and the dendritic at more negative potentials. At potentials slightly more negative than $E_{\frac{1}{2}}$ a sudden change of the whiskers' diameter occurs (about 50 times).

The second part of our work was concerned with absorption of zincate by various separators. Five separators were tested: cellophane PUDO (300), silver treated PUDO (300) - C-19, Polyvinyl Alcohol separator (PVA), and two Borden separators, C-3 and 9107/5. Separators soaked for three days in 45% KOH containing various concentrations of zincate were analyzed for content of zincate. The results of the analysis showed that the content of zincate in the solution absorbed by the separators was lower than in the solutions surrounding the separators. The distribution coefficient, based on total analytical concentrations of zincate was used as an indicator of this effect. This distribution coefficient varied for various separators but was always less than one. It was largest for cellophane PUDO (300) and smallest for Borden's C-3 separator. The absorption isotherms plotted for the five separators tested showed that dependence of inner concentration on outer concentration of zincate is almost linear. In some cases a slight positive deviation from linearity was found. The distribution coefficients are important because plating of zinc in the separator is a function of local concentration of zincate. The results indicate at least one of the reasons why conditions for electroplating inside of the separator

are less favorable than in the free electrolyte.

5. REFERENCES

1. Investigation and Improvement of Zinc Electrodes for Electrochemical Cells, Yardney Electric Corp., Contract NAS-5-3873.

2. Harned and Owen, The Physical Chemistry of Electrolytic Solutions, Reihold Publishing Co., New York, N.Y. 1950, Ch. 14.

3. Improved Separators for Silver Oxide-Zinc and Silver Oxide-Cadmium Cells for Spacecraft Application, Borden Chemical Co. Contract NAS5-9107.

TABLE I

SPECIFIC GRAVITY OF 45% KOH AS A FUNCTION OF ZINCATE CONTENT AT 74°F

Zincate Concentration Moles/liter	Specific Gravity g/cc
0.0	1.4520
0.1	1.4550
0.2	1.4616
0.3	1.4666
0.4	1.4704
0.5	1.4801
0.6	1.4897
0.7	1.4953
0.8	1.4999
0.9	1.5138
1.0	1.5222

TABLE II

ABSORPTION OF ZINCATE BY 300 PUDO CELLOPHANE SOAKED IN ZINCATE SOLUTION (45% KOH)
NITRIC ACID ANALYSIS

CONC. OF ZINCATE mol/l	THICKNESS IN MILS BEFORE SOAKING	THICKNESS IN MILS AFTER SOAKING	DIMENSIONS IN INCHES AFTER SOAKING	WEIGHT IN GRAMS BEFORE SOAKING	WEIGHT IN GRAMS AFTER SOAKING	$W_{AF.} - W_B$ GRAMS	MOL/SAMP. $x10^{-3}$	MOL/Kg (DRY SAMPLE)	MOL/Liter (DRY SAMPLE)	MOL/Kg (WET SAMPLE)	MOL/Liter (WET SAMPLE)
0.1	1.0	3.15	2.900x3.125	0.2105	0.702	0.4915	0.03305	0.1570) Mean	0.2284) Mean	0.0672) Mean	0.0978) Mean
	1.0	3.15	2.900x3.125	0.2105	0.702	0.4915	0.03305	0.1570) 0.1583	0.2284) 0.2303	0.0672) 0.0676	0.0978) 0.0984
	1.0	3.15	2.900x3.125	0.2105	0.705	0.4945	0.03385	0.1608)	0.2340)	0.0684)	0.0995)
0.2	1.0	2.8	2.900x3.325	0.2105	0.696	0.4855	0.05625	0.2682)	0.3920)	0.1162)	0.1698)
	1.0	3.1	2.900x3.080	0.2105	0.699	0.4885	0.05965	0.2834) 0.2739	0.4142) 0.4003	0.1221) 0.1185	0.1785) 0.1732
	1.0	3.0	2.900x3.125	0.2105	0.696	0.4855	0.05686	0.2701)	0.3948)	0.1171)	0.1712)
0.3	1.0	3.1	2.900x3.100	0.2100	0.714	0.504	0.08225	0.3917)	0.5745)	0.16319)	0.2393)
	1.0	3.15	2.900x3.080	0.2100	0.713	0.503	0.08185	0.3898) 0.3885	0.5717) 0.5698	0.16272) 0.1635	0.2386) 0.2398
	1.0	2.90	2.900x3.125	0.2100	0.700	0.490	0.08064	0.3840)	0.5632)	0.16457)	0.2414
0.4	1.0	3.00	2.900x3.080	0.2100	0.692	0.482	0.11855	0.5645)	0.8300)	0.24595)	0.3616)
	1.0	3.10	2.900x3.080	0.2100	0.708	0.498	0.12455	0.5931) 0.5779	0.8721) 0.8497	0.25010) 0.2486	0.3677) 0.3656
	1.0	2.90	2.900x3.125	0.2100	0.694	0.484	0.12095	0.5760)	0.8470)	0.24989)	0.3674)
0.5	1.0	2.6	2.875x3.275	0.2110	0.712	0.501	0.15322	0.7262)	1.0748)	0.30582)	0.4526)
	1.0	3.0	2.900x3.125	0.2110	0.709	0.498	0.15154	0.7182) 0.7286	1.0630) 1.0784	0.30430) 0.3050	0.4504) 0.4526
	1.0	3.0	2.900x3.100	0.2110	0.720	0.509	0.15644	0.7414)	1.0973)	0.30734)	0.4549)
0.6	1.0	3.1	2.900x3.125	0.2110	0.709	0.498	0.17419	0.8255)	1.2298)	0.34978)	0.5211)
	1.0	3.0	2.900x3.100	0.2110	0.707	0.496	0.17333	0.8214) 0.8216	1.2236) 1.2206	0.34945) 0.3490	0.5206) 0.5300
	1.0	2.9	2.900x3.150	0.2110	0.707	0.496	0.17258	0.8179)	1.2184)	0.34794)	0.5183)
0.7	1.0	3.1	2.900x3.090	0.2100	0.715	0.505	0.20317	0.9675)	1.4467)	0.40231)	0.6016)
	1.0	3.1	2.900x3.085	0.2100	0.716	0.506	0.20564	0.9795) 0.9636	1.4646) 1.4413	0.40640) 0.4010	0.6077) 0.5997
	1.0	3.0	2.900x3.100	0.2100	0.713	0.503	0.19838	0.9447)	1.4126)	0.39440)	0.5897)
0.8	1.0	3.15	2.900x3.095	0.2105	0.7335	0.523	0.22983	1.0918)	1.6376)	0.43944)	0.6591)
	1.0	3.15	2.900x3.100	0.2105	0.733	0.5225	0.22983	1.0918) 1.0842	1.6376) 1.6261	0.43986) 0.4392	0.6597) 0.6587
	1.0	2.90	2.875x3.380	0.2105	0.724	0.5135	0.22500	1.0689)	1.6032)	0.43818	0.6572
0.9	1.0	3.0	2.900x3110	0.2105	0.7175	0.507	0.2492	1.1838)	1.7920)	0.4915)	0.7440)
	1.0	3.0	2.900x3090	0.2105	0.725	0.5145	0.2540	1.2067) 1.2028	1.8267) 1.8208	0.4956) 0.4964	0.7502) 0.7548
	1.0	3.0	2.900x3080	0.2105	0.721	0.5105	0.2564	1.2180)	1.8438)	0.5022)	0.7602)
1.0	1.0	2.9	2.900x3.135	0.2115	0.725	0.5135	0.2710	1.2813)	1.9504)	0.5277)	0.8033)
	1.0	3.1	2.900x3.080	0.2115	0.7305	0.519	0.2838	1.3418) 1.3065	2.0425) 1.9888	0.5468) 0.5377	0.8323) 0.8185
	1.0	2.8	2.900x3.275	0.2115	0.721	0.5105	0.2742	1.2965)	1.9735)	0.5387)	0.8200

TABLE III

ABSORPTION OF ZINCATE BY C-19 SOAKED IN ZINCATE SOLUTION (45% KOH)
NITRIC ACID ANALYSIS

CONC. OF SOL.	THICKNESS IN MILS BEFORE SOAKING	THICKNESS IN MILS AFTER SOAKING	DIMENSIONS IN INCHES AFTER SOAKING	WEIGHT IN GRAMS BEFORE SOAKING	WEIGHT IN GRAMS AFTER SOAKING	$W_A - W_B$ Grams	MOL/SAMP. $x10^{-3}$	MOL/Kg (DRY SAMPLE)	MOL/Liter (DRY SAMPLE)	MOL/Kg. (WET SAMPLE)	MOL/Liter (WET SAMPLE)
0.1 Mol	1.15	3.0	3.015x3.450	0.250	0.824	0.574	0.030	0.1200) Mean	0.1746) Mean	0.0522) Mean	0.0760) Mean
	1.15	3.0	3.030x3.440	0.250	0.823	0.573	0.029	0.1160) 0.1190	0.1688) 0.1727	0.0506) 0.0520	0.0736) 0.0757
	1.15	3.0	3.030x3.425	0.250	0.813	0.563	0.030	0.1200)	0.1746)	0.0532)	0.0774)
0.2 Mol	1.15	2.9	3.015x3.540	0.250	0.833	0.583	0.0591	0.2364)	0.3455)	0.1014)	0.1482)
	1.15	3.0	3.015x3.390	0.250	0.823	0.573	0.0567	0.2268) 0.2344	0.3315) 0.3426	0.0990) 0.1008	0.1447) 0.1473
	1.15	3.05	3.020x3.390	0.250	0.838	0.588	0.0600	0.2400)	0.3508)	0.1020)	0.1490)
0.3 Mol	1.15	3.0	3.015x3.425	0.250	0.841	0.591	0.0911	0.3644)	0.5344)	0.1541)	0.2260)
	1.15	3.0	3.015x3.425	0.250	0.842	0.592	0.0922	0.3688) 0.3585	0.5409) 0.5258	0.1557) 0.1518	0.2283) 0.2227
	1.15	3.0	3.015x3.390	0.250	0.837	0.587	0.0856	0.3424)	0.5022)	0.1457)	0.2137)
0.4 Mol	1.15	2.9	3.030x3.415	0.250	0.826	0.576	0.1167	0.4668)	0.6864)	0.2025)	0.2978)
	1.15	2.9	3.020x3.415	0.250	0.827	0.577	0.1158	0.4632) 0.4648	0.6811) 0.6835	0.2007) 0.2009	0.2951) 0.2954
	1.15	2.9	3.025x3.400	0.250	0.832	0.582	0.1161	0.4644)	0.6823)	0.1994)	0.2932)
0.5 Mol	1.15	2.9	3.030x3.430	0.2495	0.8315	0.582	0.1500	0.6012)	0.8893)	0.2577)	0.3814)
	1.15	2.9	3.025x3.450	0.2495	0.824	0.5745	0.1333	0.5342) 0.5855	0.7907) 0.8666	0.2320) 0.2510	0.3434) 0.3716
	1.15	2.9	3.015x3.430	0.2495	0.838	0.5885	0.1550	0.6212)	0.9194)	0.2634)	0.3899)
0.6 Mol	1.15	2.9	3.025x3.420	0.2505	0.826	0.5755	0.1900	0.7585)	1.1299)	0.3301)	0.4917)
	1.15	2.9	3.025x3.400	0.2505	0.830	0.5795	0.2000	0.7984) 0.7880	1.1894) 1.1739	0.3451) 0.3388	0.5141) 0.5047
	1.15	3.0	3.025x3.390	0.2505	0.843	0.5925	0.2022	0.8071)	1.2023)	0.3412)	0.5083)
0.7 Mol	1.15	2.9	3.025x3.400	0.249	0.828	0.579	0.2200	0.8835)	1.3211)	0.3800)	0.5682)
	1.15	2.9	3.020x3.415	0.249	0.824	0.575	0.2133	0.8566) 0.8656	1.2809) 1.2943	0.3710) 0.3742	0.5548) 0.5596
	1.15	2.9	3.025x3.410	0.249	0.823	0.574	0.2133	0.8566)	1.2809)	0.3716)	0.5557)
0.8 Mol	1.15	3.0	3.025x3.400	0.251	0.841	0.590	0.2600	1.0359)	1.5537)	0.4401)	0.6601)
	1.15	3.0	3.025x3.400	0.251	0.837	0.586	0.2567	1.0227) 1.0182	1.5339) 1.5272	0.4380) 0.4392	0.6570) 0.6587
	1.15	2.85	3.015x3.435	0.251	0.820	0.569	0.2500	0.9960)	1.4939)	0.4394)	0.6591)
0.9 Mol	1.15	3.0	3.040x3.435	0.252	0.852	0.600	0.3033	1.2036)	1.8220)	0.5055)	0.7652)
	1.15	3.0	3.040x3.420	0.252	0.832	0.580	0.2733	1.0840) 1.1329	1.6410) 1.7150	0.4712) 0.4848	0.7133) 0.7339
	1.15	3.0	3.025x3.430	0.252	0.836	0.584	0.2800	1.1111)	1.6820)	0.4778)	0.7233)
1.0 Mol	1.15	3.1	3.030x3.370	0.253	0.856	0.603	0.3490	1.3794)	2.0997)	0.5788)	0.8810)
	1.15	3.0	3.040x3.395	0.253	0.860	0.607	0.3600	1.4229) 1.3792	2.1659) 2.0993	0.5931) 0.5792	0.9028) 0.8817
	1.15	3.1	3.040x3.375	0.253	0.850	0.597	0.3378	1.3352)	2.0324)	0.5658)	0.8613)

TABLE IV

ABSORPTION OF ZINCATE BY PVA SOAKED IN ZINCATE SOLUTION (45% KOH)
NITRIC ACID ANALYSIS

CONC. OF ZINCATE mol/l	THICKNESS IN MILS BEFORE SOAKING	THICKNESS IN MILS AFTER SOAKING	DIMENSIONS IN INCHES AFTER SOAKING	WEIGHT IN GRAMS BEFORE SOAKING	WEIGHT IN GRAMS AFTER SOAKING	$W_A - W_B$ GRAMS	MOL/SAMP. $\times 10^{-3}$	MOL/Kg (DRY SAMPLE)		MOL/Liter (DRY SAMPLE)		MOL/Kg (WET SAMPLE)		MOL/Liter (WET SAMPLE)	
0.1	1.45	2.3-2.35	3.250x3.250	0.2630	0.577	0.314	0.0132	0.0502)	Mean	0.0730)	Mean	0.0420)	Mean	0.0611)	Mean
	1.45	2.3-2.35	3.250x3.250	0.2630	0.586	0.323	0.0118	0.0449)	0.0467	0.0653)	0.0679	0.0364)	0.0379	0.0530)	0.0552
	1.45	2.3-2.35	3.250x3.250	0.2625	0.594	0.3315	0.0118	0.0450)		0.0654)		0.0353)		0.0514)	
0.2	1.45-1.5	2.25-2.3	3.250x3.250	0.265	0.613	0.348	0.0229	0.0864)		0.1263)		0.0658)		0.0962)	
	1.45-1.5	2.25-2.3	3.250x3.250	0.265	0.603	0.338	0.0200	0.0755)	0.0828	0.1104)	0.1210	0.0591)	0.0637	0.0864)	0.0931
	1.45-1.5	2.25-2.3	3.250x3.250	0.265	0.611	0.346	0.0229	0.0864)		0.1263)		0.0661)		0.0966)	
0.3	1.45-1.5	2.3-2.35	3.225x3.225	0.2695	0.5995	0.330	0.0313	0.1161)		0.1703)		0.0950)		0.1393)	
	1.45-1.5	2.3-2.35	3.225x3.225	0.2695	0.6135	0.344	0.0281	0.1043)	0.1136	0.1530)	0.1671	0.0820)	0.0921	0.1203)	0.1350
	1.45-1.5	2.3-2.35	3.225x3.225	0.2660	0.5980	0.332	0.0323	0.1214)		0.1781)		0.0992)		0.1455)	
0.4	1.35-1.4	2.25	3.335x3.335	0.260	0.591	0.331	0.0531	0.2042)		0.3003)		0.1604)		0.2359)	
	1.35-1.40	2.25	3.335x3.335	0.260	0.590	0.330	0.0531	0.2041)	0.2015	0.3001)	0.2963	0.1609)	0.1596	0.2366)	0.2346
	1.35-1.40	2.25	3.335x3.335	0.260	0.584	0.324	0.0510	0.1962)		0.2885)		0.1574)		0.2314)	
0.5	1.4	2.2	3.325x3.325	0.264	0.604	0.340	0.0609	0.2307)		0.3415)		0.1791)		0.2651)	
	1.4	2.2	3.325x3.325	0.264	0.603	0.339	0.0578	0.2190)	0.2221	0.3241)	0.3287	0.1705)	0.1736	0.2524)	0.2567
	1.35-1.4	2.2	3.325x3.325	0.260	0.589	0.329	0.0563	0.2165)		0.3204)		0.1711)		0.2532)	
0.6	1.45-1.5	2.2	3.325x3.325	0.271	0.616	0.345	0.0854	0.3151)		0.4693)		0.2475)		0.3687)	
	1.4-1.45	2.2	3.325x3.325	0.262	0.582	0.320	0.0791	0.3020)	0.3035	0.4499)	0.4525	0.2470)	0.2485	0.3680)	0.3703
	1.4-1.45	2.2	3.325x3.325	0.262	0.569	0.307	0.0771	0.2943)		0.4384)		0.2511)		0.3741)	
0.7	1.4-1.45	2.2	3.320x3.320	0.264	0.589	0.325	0.1052	0.3985)		0.5959)		0.3237)		0.4840)	
	1.5	2.2	3.320x3.320	0.272	0.609	0.337	0.0948	0.3485)	0.3741	0.5211)	0.5561	0.2813)	0.3012	0.4206)	0.4503
	1.5	2.2	3.320x3.320	0.272	0.614	0.342	0.1021	0.3754)		0.5613)		0.2985)		0.4463)	
0.8	1.5	2.3	3.265x3.265	0.278	0.610	0.332	0.1021	0.3673)		0.5509)		0.3075)		0.4612)	
	1.5	2.3	3.265x3.265	0.2755	0.610	0.3345	0.1041	0.3779)	0.3769	0.5656)	0.5649	0.3112)	0.3140	0.4668)	0.4710
	1.5	2.3	3.265x3.265	0.2755	0.604	0.3285	0.1062	0.3855)		0.5782)		0.3233)		0.4849)	
0.9	1.5	2.3	3.260x3.260	0.2725	0.596	0.3235	0.1146	0.4206)		0.6367)		0.3542)		0.5362)	
	1.5	2.3	3.260x3.260	0.2725	0.600	0.3275	0.1187	0.4356)	0.4236	0.6594)	0.6412	0.3624)	0.3537	0.5486)	0.5355
	1.5	2.3	3.260x3.260	0.2765	0.609	0.3325	0.1146	0.4145)		0.6275		0.3447)		0.5218)	
1.0	1.35-1.4	2.1	3.275x3.275	0.2575	0.573	0.3155	0.1196	0.4645)		0.7071)		0.3790)		0.5769)	
	1.35-1.4	2.1	3.275x3.275	0.2575	0.576	0.3185	0.1235	0.4796)	0.4719	0.7300)	0.7184	0.3874)	0.3867	0.5897)	0.5887
	1.35-1.4	2.1	3.275x3.275	0.2575	0.566	0.3085	0.1215	0.4718)		0.7182)		0.3938)		0.5994)	

TABLE V

ABSORPTION OF ZINCATE BY PVA SOAKED IN ZINCATE SOLUTION (45% KOH)
COMBUSTION ANALYSIS

CONC. OF ZINCATE mol/l	THICKNESS IN MILS BEFORE SOAKING	THICKNESS IN MILS AFTER SOAKING	DIMENSIONS IN INCHES AFTER SOAKING	WEIGHT IN GRAMS BEFORE SOAKING	WEIGHT IN GRAMS AFTER SOAKING	$W_A - W_B$ GRAMS	MOL/SAMP $x10^{-3}$	MOL/kg (DRY SAMPLE)		MOL/Liter (DRY SAMPLE)		MOL/Kg (WET SAMPLE)		MOL/Liter (WET SAMPLE)	
0.1	1.45	2.3-2.35	3.250x3.250	0.269	0.600	0.331	0.0053	0.0197)	Mean	0.0146)	Mean	0.0160)	Mean	0.0233)	Mean
	1.45	2.3-2.35	3.250x3.250	0.269	0.609	0.340	0.0096	0.0357)	0.0295	0.0519)	0.0349	0.0282)	0.0232	0.0410)	0.0338
	1.45	2.3-2.35	3.250x3.250	0.2625	0.603	0.3405	0.0087	0.0331)		0.0482)		0.0255)		0.0371)	
0.2	1.4-1.45	2.2-2.25	3.250x3.250	0.259	0.593	0.334	0.0240	0.0927)		0.1355)		0.0718)		0.1049)	
	1.4-1.45	2.2-2.25	3.250x3.250	0.259	0.587	0.328	0.0215	0.0830)	0.0788	0.1213)	0.1152	0.0656)	0.0617	0.0959)	0.0903
	1.4-1.45	2.2-2.25	3.250x3.250	0.260	0.589	0.329	0.0158	0.0608)		0.0889)		0.0480		0.0702)	
0.3	1.45	2.35	3.250x3.250	0.270	0.591	0.321	0.0335	0.1241)		0.1820)		0.1043)		0.1530)	
	1.45	2.35	3.250x3.250	0.270	0.596	0.326	0.0343	0.1270)	0.1233	0.1863)	0.1809	0.1052)	0.1025	0.1543)	0.1503
	1.45	2.3-2.35	3.250x3.250	0.265	0.587	0.322	0.0315	0.1189)		0.1744)		0.0980)		0.1437)	
0.4	1.45	2.25	3.335x3.335	0.264	0.600	0.336	0.0390	0.1477)		0.2172)		0.1160)		0.1706)	
	1.45	2.25	3.335x3.335	0.264	0.599	0.335	0.0366	0.1386)	0.1396	0.2038)	0.2053	0.1092)	0.1105	0.1606)	0.1626
	1.45	2.25	3.335x3.335	0.264	0.593	0.329	0.0350	0.1326)		0.1950)		0.1064)		0.1565)	
0.5	1.45	2.2	3.325x3.325	0.266	0.604	0.338	0.0514	0.1932)		0.2860)		0.1522)		0.2223)	
	1.4	2.2	3.325x3.325	0.263	0.594	0.333	0.0462	0.1757)	0.1827	0.2601)	0.2704	0.1389)	0.1439	0.2056)	0.2120
	1.4	2.2	3.325x3.325	0.263	0.598	0.335	0.0471	0.1791)		0.2651)		0.1406)		0.2081)	
0.6	1.4-1.45	2.2	3.325x3.325	0.261	0.594	0.333	0.0567	0.2172)		0.3236)		0.1700)		0.2532)	
	1.4-1.45	2.2	3.325x3.325	0.261	0.585	0.324	0.0563	0.2157)	0.2022	0.3213)	0.3012	0.1690)	0.1591	0.2518)	0.2375
	1.4-1.45	2.2	3.325x3.325	0.259	0.583	0.324	0.0450	0.1737)		0.2588)		0.1400)		0.2086)	
0.7	1.4-1.45	2.2	3.320x3.320	0.2635	0.590	0.3265	0.0659	0.2501)		0.3740)		0.2016)		0.3015)	
	1.4-1.45	2.2	3.320x3.320	0.2635	0.584	0.3205	0.0695	0.2638)	0.2606	0.3945)	0.3897	0.2170)	0.2115	0.3245)	0.3163
	1.4-1.45	2.2	3.320x3.320	0.265	0.594	0.329	0.0710	0.2679)		0.4006)		0.2160)		0.3230)	
0.8	1.5	2.3	3.265x3.265	0.279	0.608	0.329	0.0746	0.2674)		0.4011)		0.2268)		0.3402)	
	1.5	2.3	3.265x3.265	0.274	0.603	0.329	0.0746	0.2723)	0.2707	0.4084)	0.4060	0.2268)	0.2279	0.3402)	0.3419
	1.5	2.3	3.265x3.265	0.274	0.598	0.324	0.0746	0.2723)		0.4084)		0.2303)		0.3454)	
0.9	1.5	2.35	3.260x3.260	0.2775	0.610	0.3325	0.0993	0.3578)		0.5416)		0.2986)		0.4520)	
	1.5	2.35	3.260x3.260	0.277	0.601	0.333	0.0978	0.3531)	0.3556	0.5345)	0.5383	0.2937)	0.2967	0.4446)	0.4489
	1.5	2.35	3.260x3.260	0.277	0.608	0.331	0.0986	0.3560)		0.5389)		0.2979)		0.4501)	
1.0	1.35-1.4	2.15	3.275x3.275	0.2585	0.572	0.3135	0.0993	0.3841)		0.5847)		0.3167)		0.4821)	
	1.35-1.4	2.15	3.275x3.275	0.2585	0.577	0.3185	0.1000	0.3868)	0.3843	0.5888)	0.5850	0.3140)	0.3141	0.4780)	0.4782
	1.35-1.4	2.15	3.275x3.275	0.260	0.577	0.3185	0.0993	0.3820)		0.5815)		0.3117)		0.4745)	

TABLE VI

ABSORPTION OF ZINCATE SOLUTION BY C-3 LOT 545-130 SOAKED IN ZINCATE SOLUTION (45% KOH) DISSOLVED IN 1:1 HNO_3

CONC. OF ZINCATE mol/l	THICKNESS IN MILS BEFORE SOAKING	THICKNESS IN MILS AFTER SOAKING	DIMENSIONS IN INCHES AFTER SOAKING	WEIGHT IN GRAMS BEFORE SOAKING	WEIGHT IN GRAMS AFTER SOAKING	$W_1 - W_2$ GRAMS	MOL/SAMP. $x10^{-3}$	MOL/Kg (WET SAMPLE)	MOL/Kg (DRY SAMPLE)	MOL/Liter (WET SAMPLE)	MOL/Liter (DRY SAMPLE)
0.1	1.55-1.65	1.9-2.0	3.100x3.100	0.247	0.427	0.180	0.00214	0.01189) Mean) 0.01211 0.01232)	0.00866) Mean) 0.00881 0.00896)	0.01730) Mean) 0.01761 0.01792)	0.01260) Mean) 0.01282 0.01304)
	1.6-1.65	1.9-2.0	3.100x3.100	0.249	0.430	0.181	0.00223				
0.2	1.5-1.55	1.85-1.95	3.100x3.100	0.2374	0.413	0.1756	0.00485	0.02762)) 0.02492 0.02222)	0.02043)) 0.01812 0.01581)	0.04037)) 0.03643 0.03248)	0.02986)) 0.02649 0.02311)
	1.5-1.6	1.9-2.0	3.100x3.100	02454	0.420	0.1746	0.00388				
0.4	1.5-1.55	1.8-1.9	3.100x3.100	0.235	0.400	0.165	0.00932	0.05649)) 0.05575 0.05500)	0.03966)) 0.03963 0.03960)	0.08306)) 0.08197 0.08087)	0.05832)) 0.05828 0.05823)
	1.55-1.65	1.9-2.0	3.100x3.100	0.250	0.430	0.180	0.00990				
0.6	1.5-1.6	1.5-1.6	3.100x3.100	0.239	0.406	0.167	0.01476	0.08838)) 0.09522 0.10206)	0.06176)) 0.06675 0.07173)	0.13166)) 0.14185 0.15204)	0.09200)) 0.09943 0.10686)
	1.6-1.65	1.9-2.0	3.100x3.100	0.249	0.424	0.175	0.01786				
0.8	1.45-1.5	1.8-1.9	3.100x3.100	0.230	0.386	0.156	0.01942	0.12449)) 0.12219 0.11989)	0.08443)) 0.08432 0.08421)	0.18672)) 0.18327 0.17982)	0.12664)) 0.12652 0.12640)
	1.5-1.6	1.9-2.0	3.100x3.100	0.242	0.412	0.170	0.02038				
1.0	1.45-1.5	1.8-1.9	3.100x3.100	0.234	0.397	0.163	0.02767	0.1698)) 0.1596 0.1494)	0.11825)) 0.11867 0.11909)	0.25847)) 0.24295 0.22742)	0.18000)) 0.18064 0.18128)
	1.5-1.6	1.9-2.0	3.100x3.100	0.2446	0.4096	0.195	0.02913				

TABLE VII

ABSORPTION OF ZINCATE BY 9107-5 LOT 545-135 SOAKED IN ZINCATE SOLUTION (45% KOH) DISSOLVED IN 1:1 HNO_3

CONC. OF ZINCATE mol/l	THICKNESS IN MILS BEFORE SOAKING	THICKNESS IN MILS AFTER SOAKING	DIMENSIONS IN INCHES AFTER SOAKING	WEIGHT IN GRAMS BEFORE SOAKING	WEIGHT IN GRAMS AFTER SOAKING	$W_1 - W_2$ GRAMS	MOL/SAMP. $x10^{-3}$	MOL/Kg (WET SAMPLE)		MOL/Kg (DRY SAMPLE)		MOL/Liter (WET SAMPLE)		MOL/Liter (DRY SAMPLE)	
0.1	1.25-1.35	1.4-1.6	3.125x3.125	0.123	0.256	0.133	0.002935	0.02206)	Mean 0.02103	0.02385)	Mean 0.02329	0.03209)	Mean 0.03103	0.03471)	Mean 0.03351
	1.5-2.9	1.9-3.1	3.125x3.125	0.1585	0.3325	0.1740	0.003585	0.02060)		0.02262)		0.2997)		0.03291)	
0.2	1.25-1.35	1.4-1.6	3.125x3.125	0.1243	0.2683	0.1440	0.00848	0.05889)	0.06153	0.06822)	0.07110	0.05889)	0.08993	0.09971)	0.10393
	1.9-2.2	2.4-2.7	3.125x3.125	0.1542	0.3320	0.1778	0.01141	0.06417)		0.07397)		0.06417)		0.10814)	
0.4	1.35-1.5	1.6-1.8	3.125x3.125	0.1368	0.2808	0.1440	0.01782	0.12378)	0.11434	0.13030)	0.11967	0.18200)	0.16811	0.19159)	0.17596
	1.7-2.0	2.2-2.5	3.125x3.125	0.1515	0.3190	0.1575	0.01652	0.10489)		0.10904)		0.15423)		0.16033)	
0.6	1.3-1.5	1.5-1.7	3.125x3.125	0.1253	0.2507	0.1254	0.02228	0.17767)	0.16945	0.17781)	0.16635	0.26467)	0.25242	0.26488)	0.24781
	1.6-2.0	1.9-2.3	3.125x3.125	0.153	0.3000	0.1470	0.02370	0.16122)		0.15490)		0.24017)		0.23075)	
0.8	1.3-1.5	1.6-1.9	3.125x3.125	0.1375	0.2715	0.1340	0.02870	0.21418)	0.21348	0.20873)	0.21103	0.32125)	0.32019	0.31307)	0.31652
	1.6-2.0	2.0-2.3	3.125x3.125	0.150	0.3004	0.1504	0.03200	0.21277)		0.21333)		0.31913)		0.31997)	
1.0	1.3-1.6	1.6-1.9	3.125x3.125	0.1394	0.2896	0.1502	0.0500	0.33289)	0.33223	0.35868)	0.3595	0.50672)	0.50571	0.54598)	0.5492
	1.3-1.6	1.6-1.9	3.125x3.125	0.1392	0.2906	0.1514	0.0502	0.33157)		0.36063)		0.50471)		0.54895)	

TABLE VIII

ZINCATE DISTRIBUTION COEFFICIENT, K, FOR SEPARATORS SOAKED IN 0.1M ZINCATE SOLUTION IN 45% KOH

SEPARATOR	K
Cellophane PUDO	0.84
C-19	0.73
PVA	0.45
C-3 (Lot 545-130)	0.18
9107 (Lot 545-135)	0.28

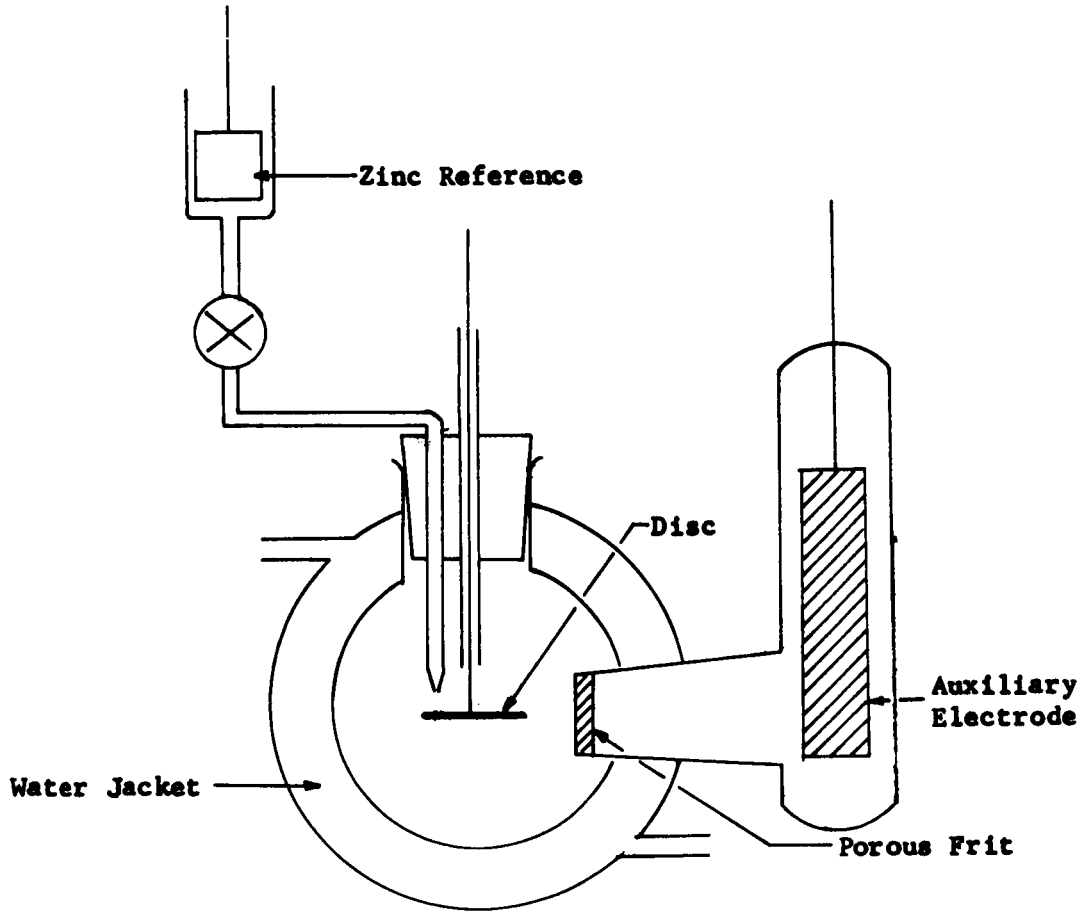

Figure 1A Rotating Disc Apparatus

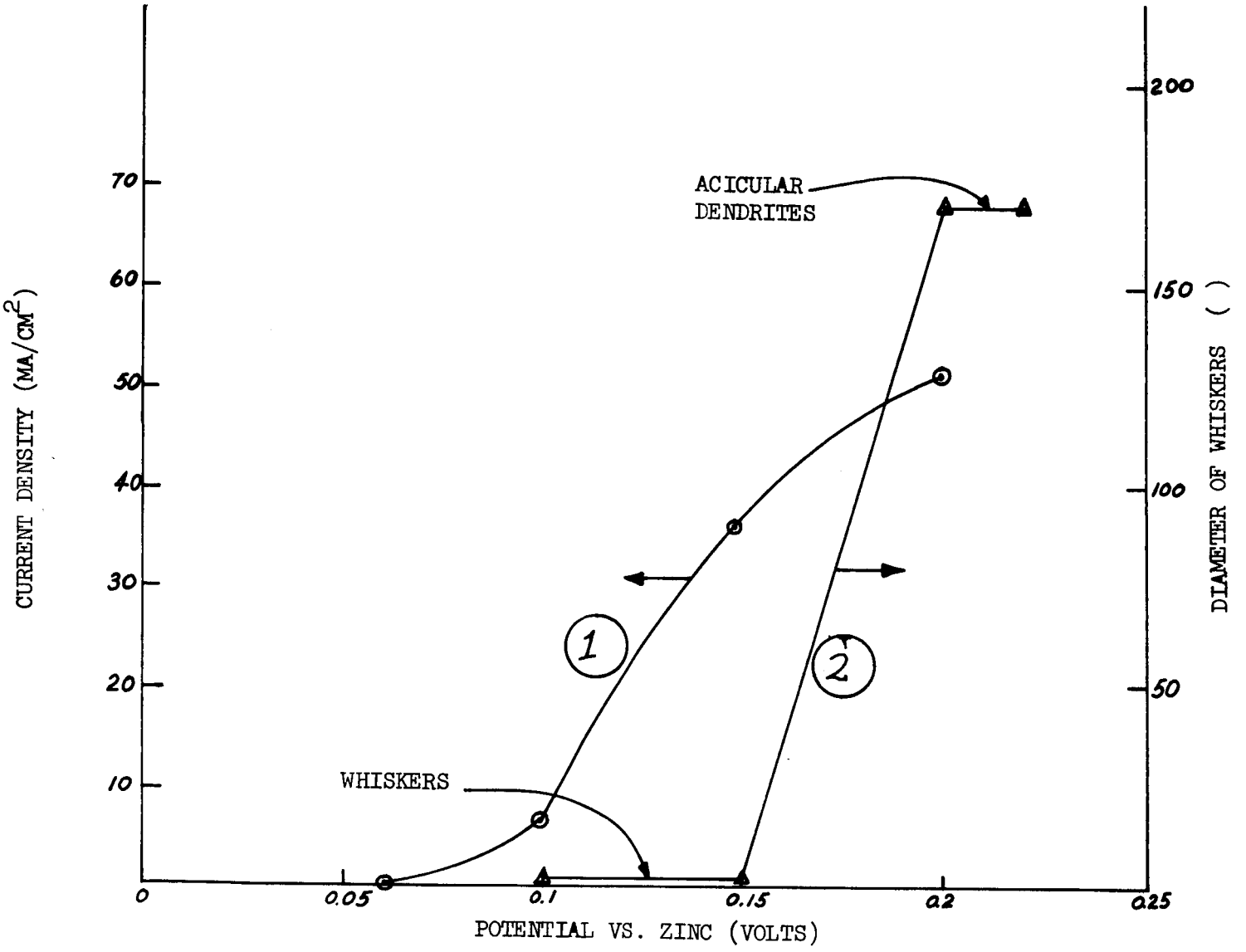

FIG. 1B CURRENT AND DIAMETERS OF WHISKERS
AS A FUNCTION OF POTENTIAL

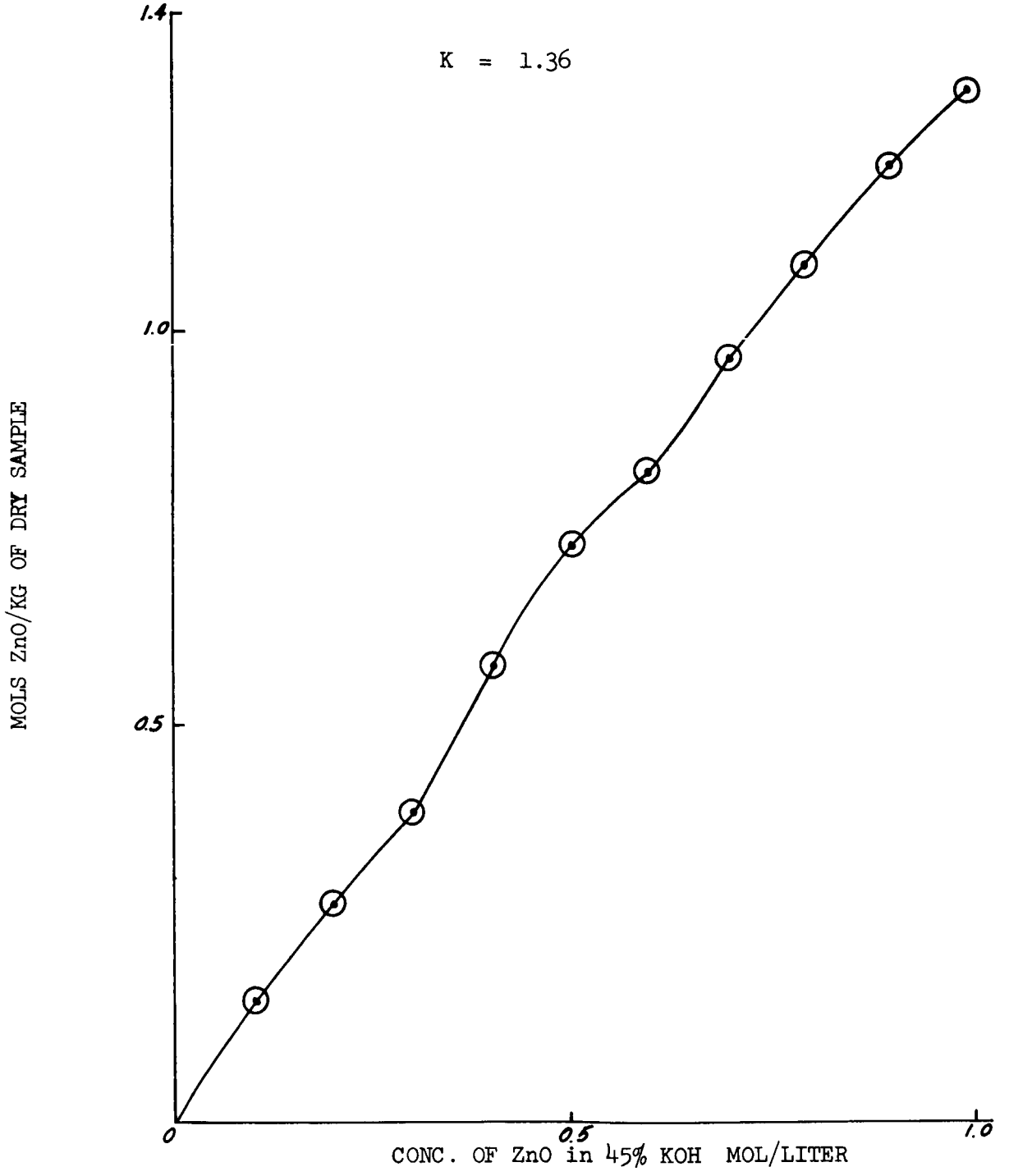

FIG. 2A.300 PUDO CELLOPHANE; HNO_3 METHOD

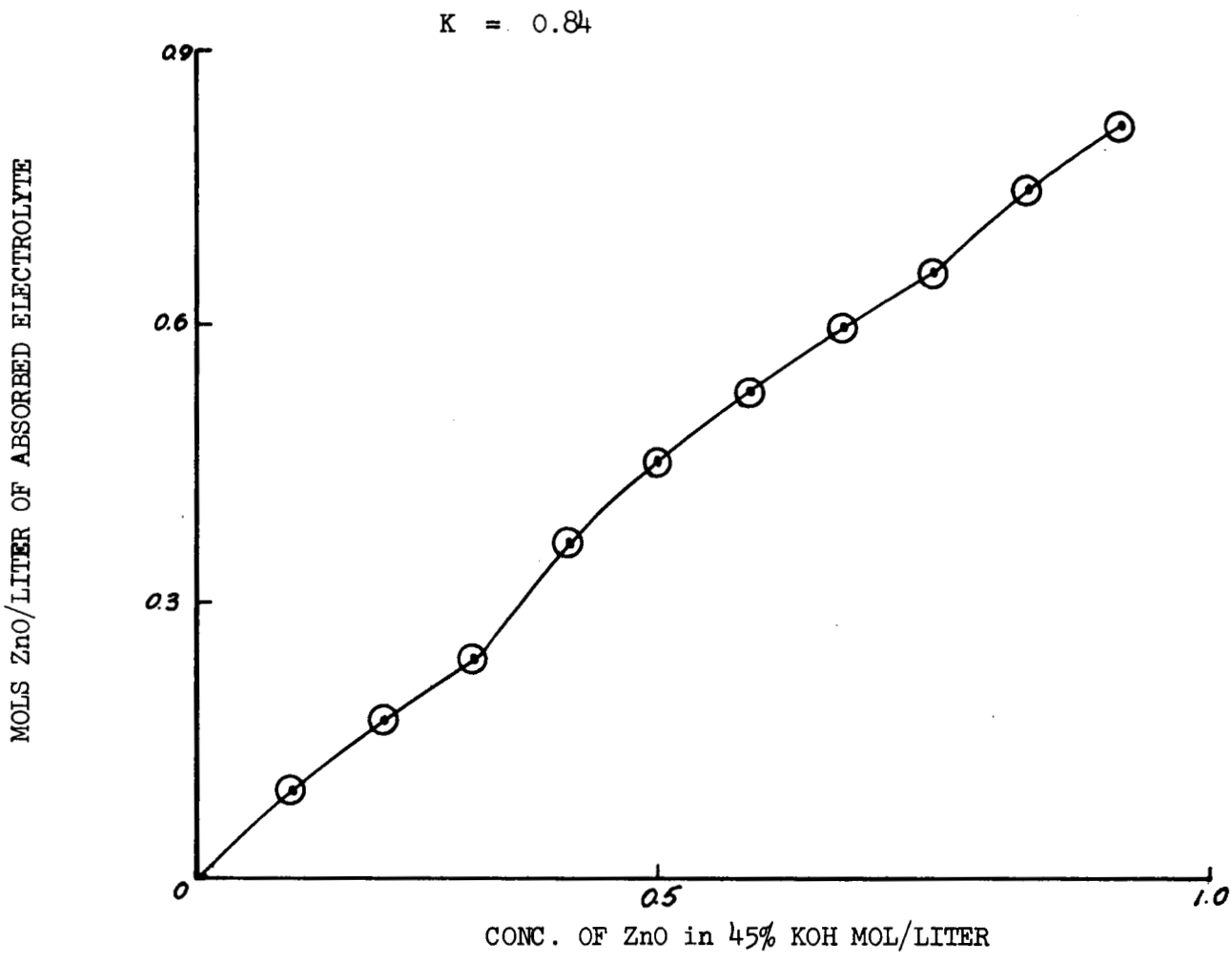

FIG. 2B 300 PUDO CELLOPHANE; HNO_3 METHOD

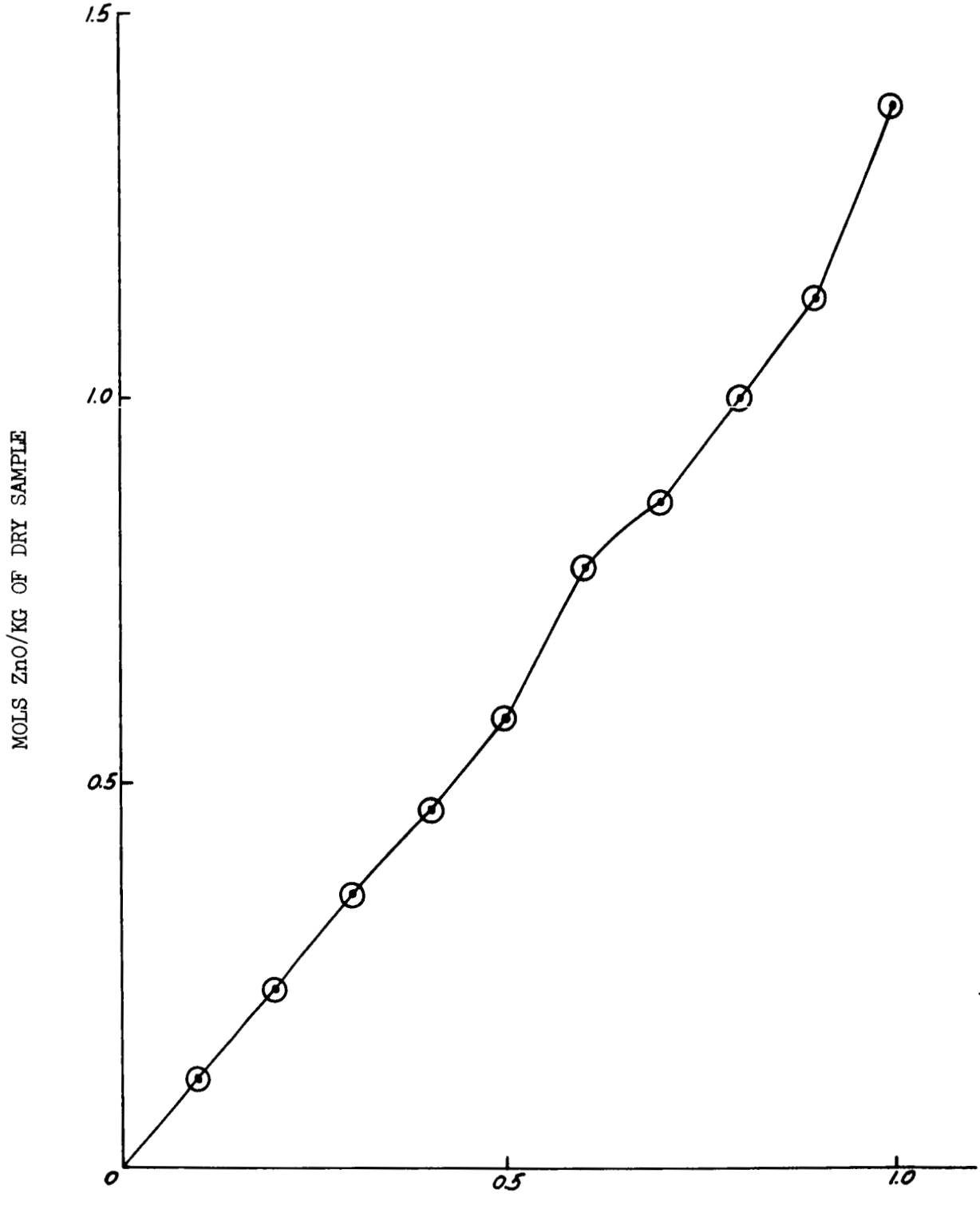

FIG. 3A C-19 IN HNO_3 METHOD

K = 0.73 at 0.1M

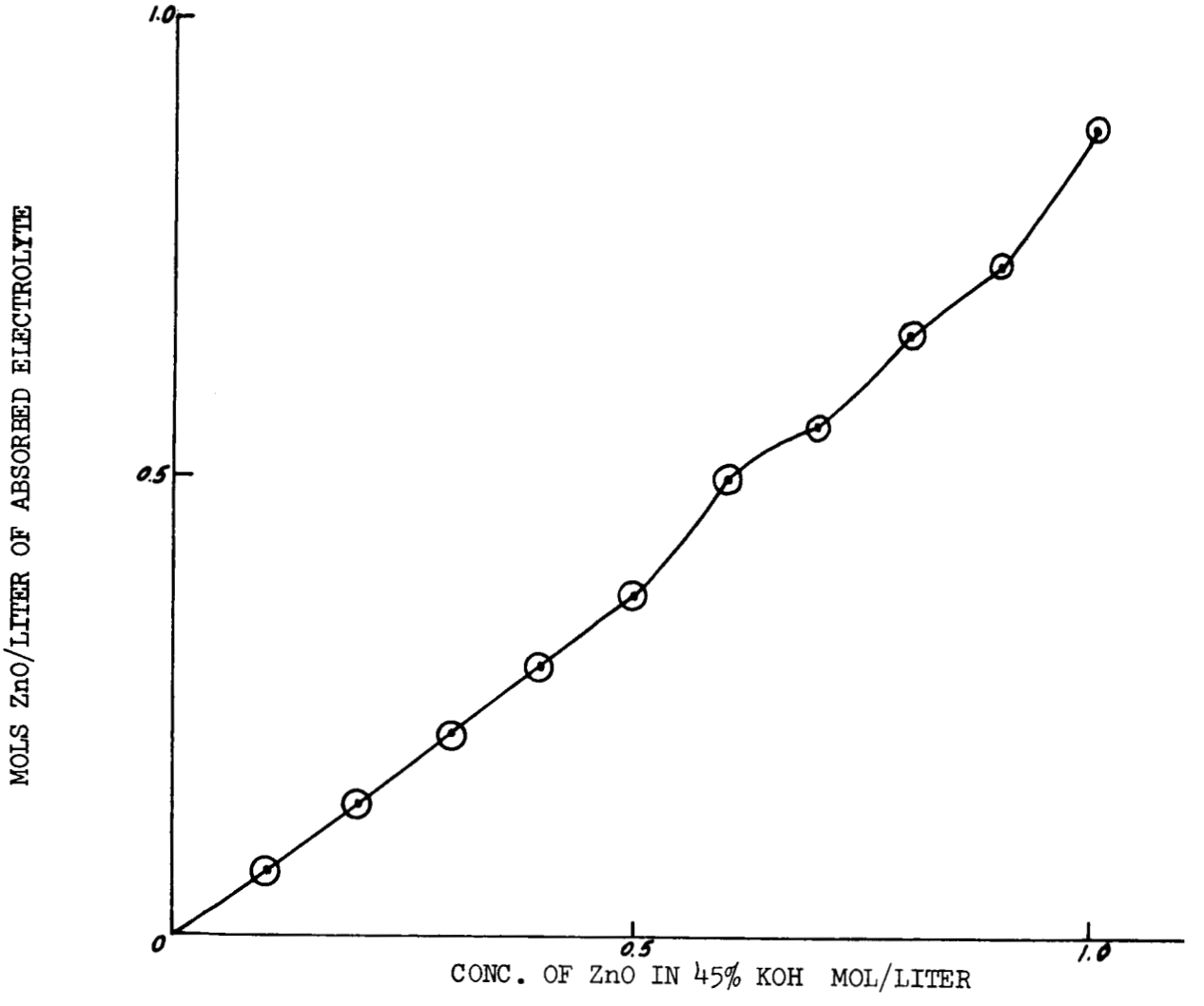

FIG. 3B C-19 HNO_3 METHOD

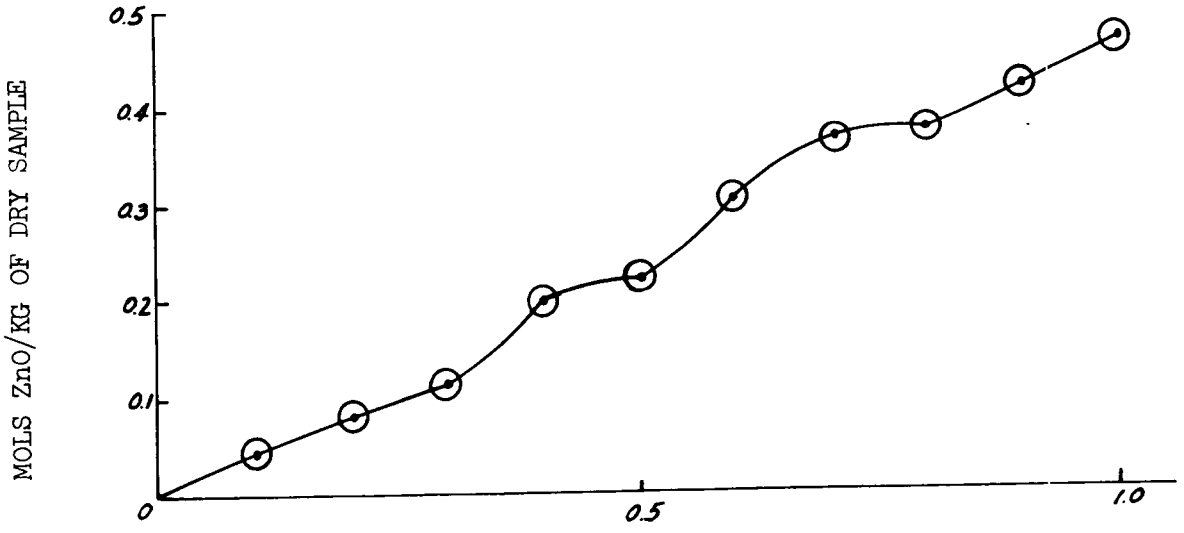

FIG. 4A PVA; HNO_3 METHOD

K = 0.45 at 0.1M
K = 0.62 at 1.0M

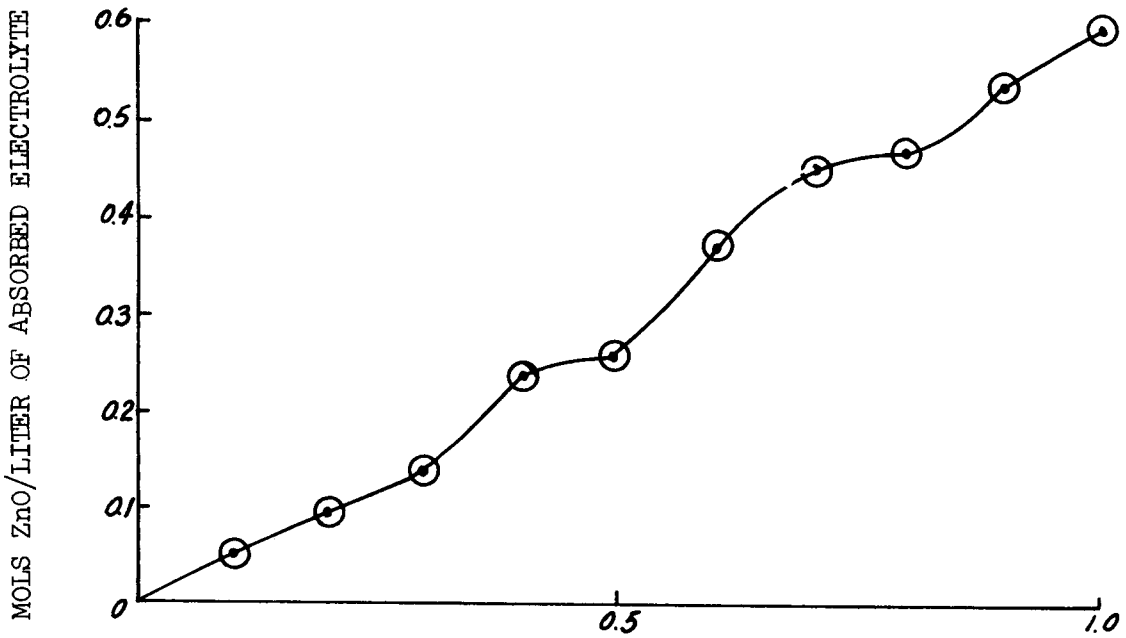

FIG. 4B PVA; HNO_3 METHOD

K = 0.40 at 0.1M

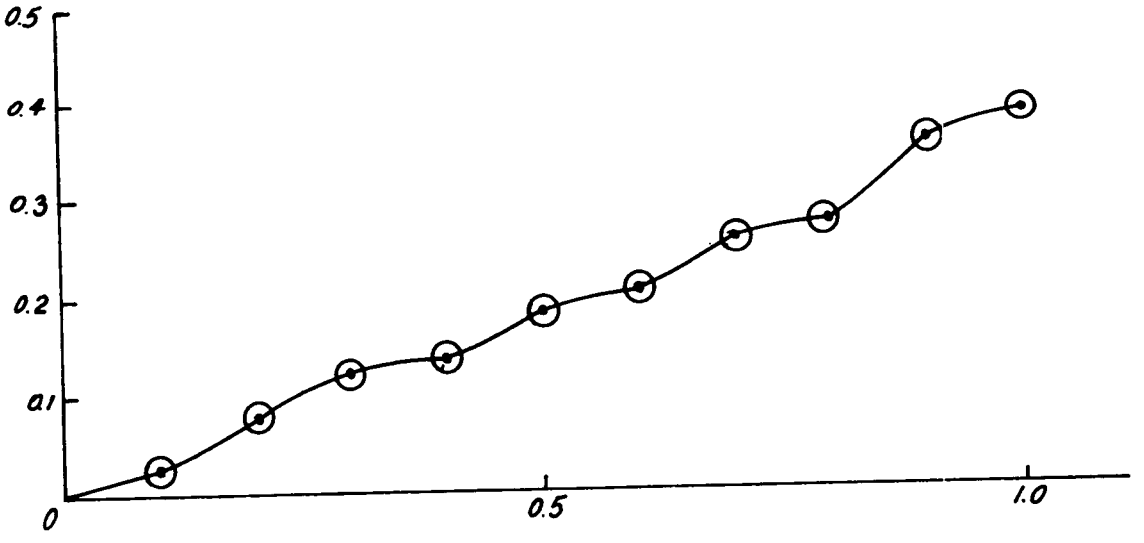

FIG. 5A PVA; BURNED IN CRUCIBLE

K = 0.46 at 0.1M

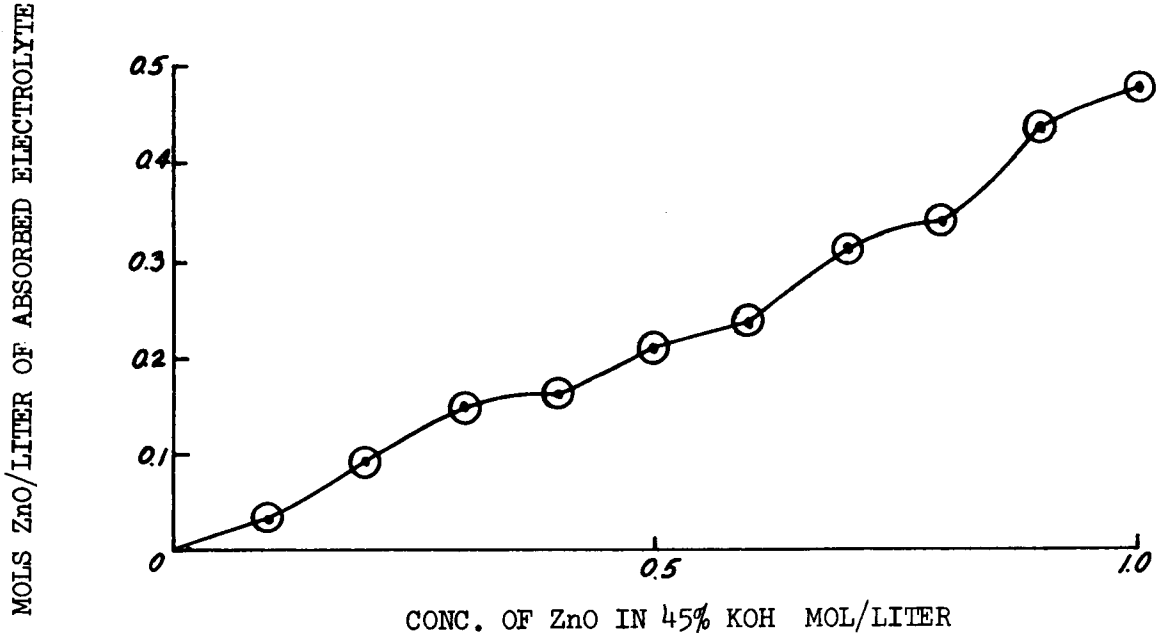

FIG. 5B PVA; BURNED IN CRUCIBLE

K = 0.09 at 0.1M

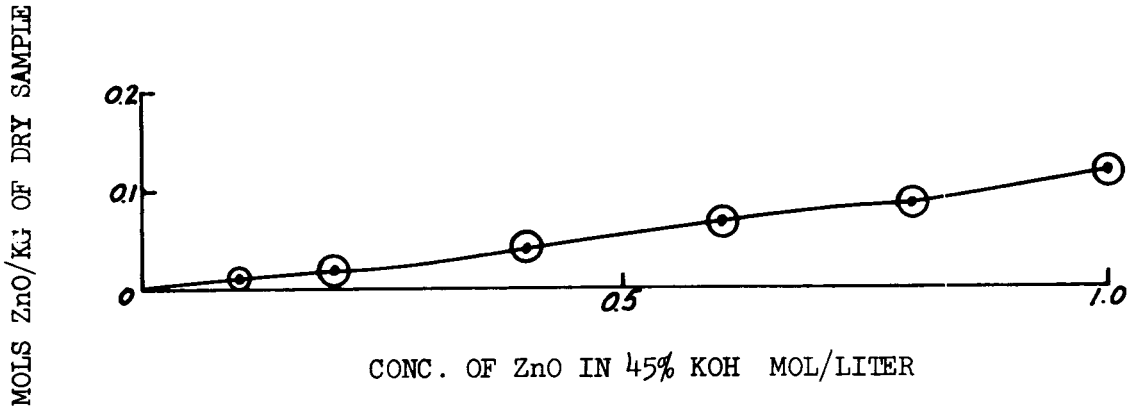

FIG. 6A C-3 LOT 545-130; HNO_3 METHOD

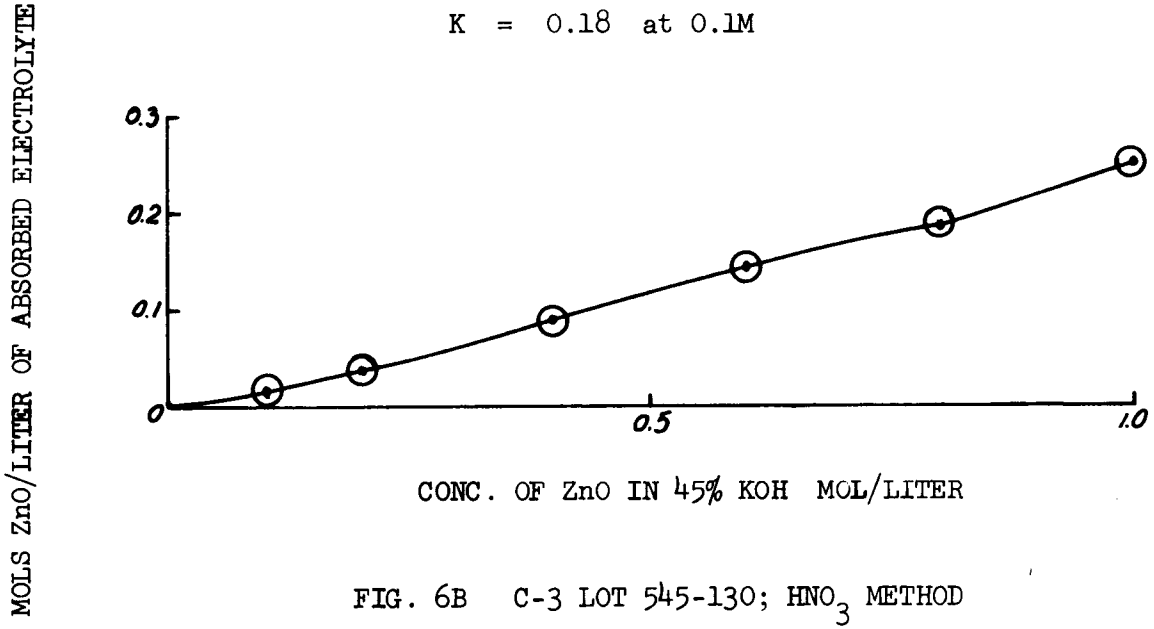

FIG. 6B C-3 LOT 545-130; HNO_3 METHOD

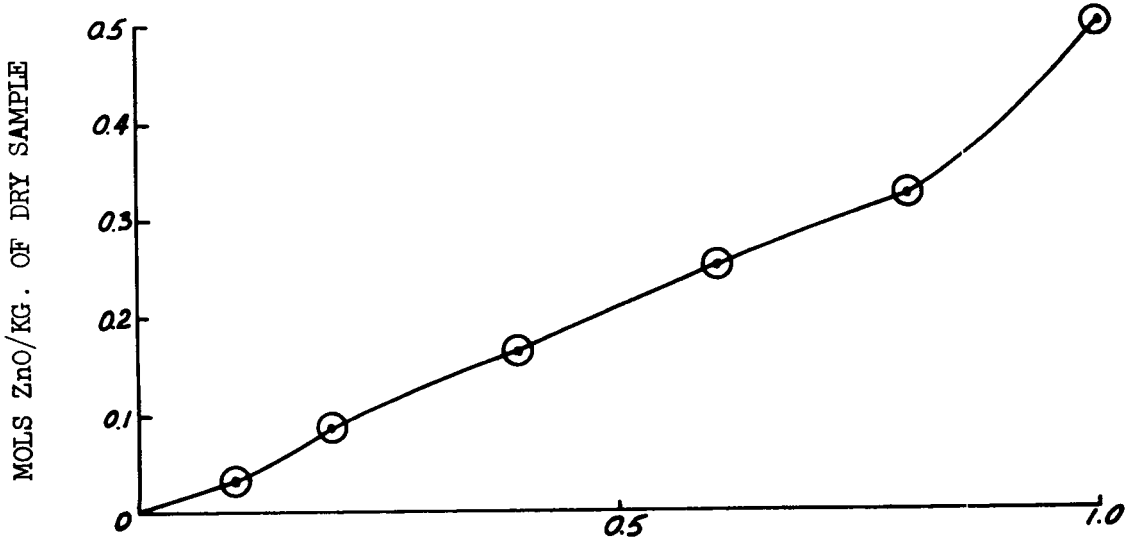

FIG. 7A 9107-5 LOT 545-135; HNO_3 METHOD

K = 0.44 at 0.1M

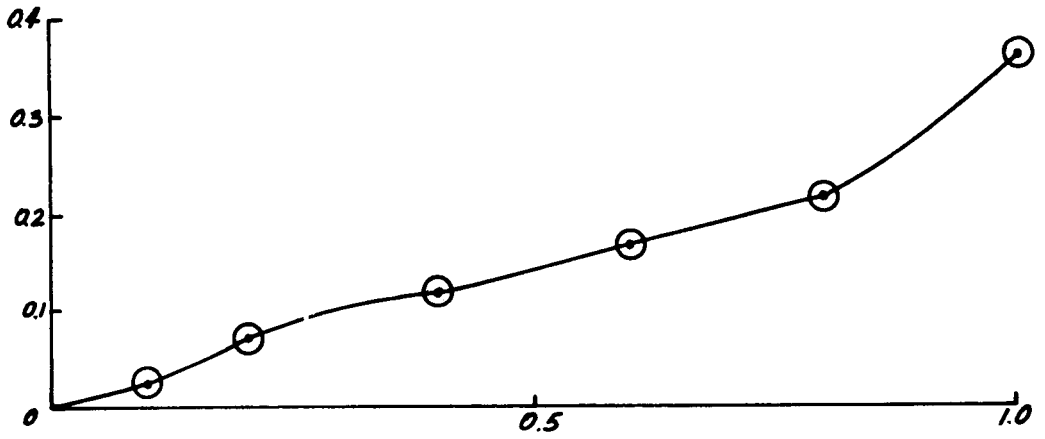

FIG. 7B 9107-5 LOT 545-135; HNO_3 METHOD