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DIFFUSION CONTROLLED CURRENT AT A STATIONARY FINITE DISK ELECTRODE (U)

AUG 80 K AOKI, J OSTERYOUNG

N00014-79-C-0644

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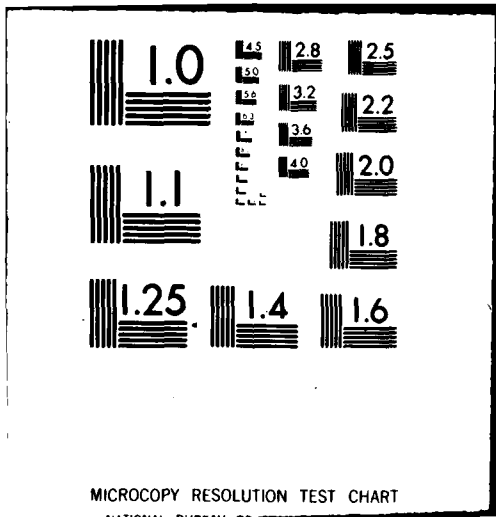
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1. REPORT NUMBER Technical Report No. 1	2. GOVT ACCESSION NO. AD-A089 063	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) Diffusion Controlled Current at a Stationary Finite Disk Electrode. Experiment		5. TYPE OF REPORT & PERIOD COVERED
7. AUTHOR(s) Koichi Aoki and Janet Osteryoung		6. PERFORMING ORG. REPORT NUMBER
9. PERFORMING ORGANIZATION NAME AND ADDRESS Department of Chemistry State University of New York at Buffalo Buffalo, NY 14214		8. CONTRACT OR GRANT NUMBER(s) N00014-79-C-0644
11. CONTROLLING OFFICE NAME AND ADDRESS Office of Naval Research/Chemistry Program Arlington, VA 22217		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
12. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE August, 1980
		13. NUMBER OF PAGES 15
		14. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

AD A089063

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17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)
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18. SUPPLEMENTARY NOTES
Prepared for Publication in Journal of Electroanalytical Chemistry

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)
Micro disk electrodes; chronoamperometric measurements

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)
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15 Contract NO0014-79-C-0644

Task No. NR 056-722

9 TECHNICAL REPORT, NO. 1

11 Aug 80

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by

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Prepared for Publication

12 24

in the

Journal of Electroanalytical Chemistry

14 TR-1

State University of New York at Buffalo
Department of Chemistry
Buffalo, New York

August, 1980

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Diffusion Controlled Current at a Stationary

Finite Disk Electrode. Experiment

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ABSTRACT

Micro disk electrodes were constructed from glassy carbon electrodes coated with photo resist by using conventional photolithography. Chronoamperometric measurements were made at these electrodes in the system ferrocyanide/ferricyanide. Dependence of currents on time and on radii of the electrodes was in good agreement with that predicted by the theory presented previously.

INTRODUCTION

Micro electrodes are of great practical interest for quantitative in vivo measurements, e.g. of oxygen tension in living tissues [1-3], because electrodes employed in vivo should be smaller than the unit size of the tissue of interest. They also have advantages in electrochemical measurements of molten salts with high temperature [4]. There is interest in their use for fundamental investigations in electrochemistry because of their low power requirements, relatively small capacity currents, and geometry which achieves steady state currents at short times. These latter points will be illustrated below. Work has been limited even concerning diffusion processes at micro disk electrodes [5-12]. Most efforts in this area have been directed to determination of edge effects as described by the first term of deviation from the Cottrell equation [5-10]. Recently we presented rigorous expressions for chronoamperometric curves under diffusion controlled conditions [12] for a finite disk electrode in an infinite insulating plane. The subject of this paper is to verify experimentally these expressions for chronoamperometric curves.

Currents at finite disk electrodes depend on both time and electrode radius. For sufficiently short times or large radii the Cottrell equation is obeyed, while at long enough times or small enough radii the current reaches a steady state. The detailed description of the dependence of the current on time and radius is formulated in terms of a reduced parameter, $\tau = 4Dt/a^2$, in which the expressions for chronoamperometric curves are expanded [12]. Here D is the diffusion coefficient (cm^2/s), t the time (s), and a the electrode radius (cm).

In order to investigate the dependence of τ on a , it is necessary to construct electrodes with a variety of well-defined diameters in the appropriate range. We employed carbon electrodes coated with photoresist and standard techniques of photolithography to prepare micro disk electrodes.

EXPERIMENTAL

1.0 mM potassium hexacyanoferrate (II) solutions were prepared in Milli-Q water with 1.0 M KCl without any assay.

Glassy carbon rods (Atomergic Chemicals Co.) 3 mm in diameter were cut off perpendicular to the length and sealed in epoxy resin in order to insulate the sides of cylinders. They were turned with a lathe in the form of 20 mm x 5 mm cylinders and the surfaces of glassy carbon were polished with alumina powder. Further preparation was carried out by the National Research and Resource Facility for Submicron Structures at Cornell University, Ithaca, NY. The electrodes were coated with a layer of photo resist about 0.5 μm thick. An electron beam apparatus was used to generate the mask for use in the photolithographic process. (Standard photographic techniques for preparing masks would have worked equally well). For technical reasons the patterns on the masks were in the shape of octagons; slight defocusing blurs the corners and makes the shape almost circular. After developing the resist, the resist constitutes a hard resin adhering closely to the electrode surface, insulating electrically the photographically unexposed area surrounding the octagons. Thus one is left with a massive electrode insulated from solution at all but the desired circular areas. In order to increase

total currents, a number of circles were made on each electrode. The distance between adjacent circles was 0.2 mm, which makes the diffusion at each circle independent for times less than 3 seconds. Eight electrodes were constructed, four sets of identical pairs with radii 0.02, 0.04, 0.1 and 0.2 mm for the circles. Although this mode of construction was adequate for the purposes of our experiments, it is not recommended as a general procedure. The mask lifted off of the carbon surface after about twenty minutes in use.

The potentiostat, which was fast settling and immune to voltage saturation, was home made. The potential step was generated by D/A converters controlled by a Digital Equipment Corporation PDP 8/e computer equipped with 32 K words of main memory, floating point processor, CRT display, 12 bit D/A's, programmable real time clock, 12 bit A/D and 2.5 M byte hard disc for mass storage.

The electrochemical cell was kept at 25°C.

RESULTS AND DISCUSSION

First we describe the method of applying the expressions for diffusion-controlled limiting currents at the micro disk electrode to real chronoamperometric curves. The currents are described in terms of the dimensionless time $\tau = 4Dt/a^2$ and are given by [12]

$$I = 4nFC^0Da f(\tau)$$

$$f(\tau) = 1 + 0.35917 \tau^{-1/2} + 0.24648 \tau^{-3/2} + 0.20648 \tau^{-5/2} + \dots$$

$$f(\tau) = \sqrt{\pi}/2\sqrt{\tau} + \pi/4 - 3\pi\tau/2^{10} - 315\pi \tau^2/2^{21} - \dots$$

where C^0 is the bulk concentration of the electroactive species (mol dm^{-3}), F the Faraday (C equiv^{-1}) in the number of electrons transferred per mole and I the current (A).

Substituting $D = \tau a^2/4t$ into eqn. (1) and rearranging it yields

$$\tau f(\tau) = It/nFC^0a^3 \quad (2)$$

Since the term on the right hand side is an experimentally measurable value, τ can be determined from the inverse function of $\tau f(\tau)$. It is possible to evaluate the inverse function in the following way: Only two terms in the expansion for $\tau f(\tau)$ are retained and the resulting quadratic equation is solved. The value of τ thus obtained is inserted into the third and succeeding terms of the expansions as the first approximate value of τ , and the resulting quadratic equation is solved again. Iteration of this process yields accurate values of τ . The values of τ thus numerically calculated are plotted against It/nFC^0a^3 in Fig. 1.

Consider electrodes of radius $0.15 \mu\text{m}$, area $7 \times 10^{-10} \text{ cm}^2$. Because the area is on the order of 10^{-7} - 10^{-8} times less than that of conventional electrodes, power requirements on application of a potential step are correspondingly less; therefore many of the instrumental problems involved in chronoamperometry or other similar techniques at short times are obviated. The current as a function of τ is within 5% of the steady state value for $\tau > 53$ and within 10% for $\tau > 3.5$ [12]. Thus for $a = 0.15 \times 10^{-4} \text{ cm}$, $D = 6 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$, the time required to achieve the steady state current within 5% is 0.5 ms and within 10% is 30 μs . Thus steady state currents can be used to study reaction kinetics on

short time scales. Consider finally the AC capacity current. For solution resistance of 100Ω , double layer capacity of $60 \mu\text{F cm}^{-2}$, the exponential factor $t/RC_d\pi a^2$ is $2 \times 10^{11}t$. Thus at $t = 10 \text{ ps}$ the capacity current has dropped to 14% of its initial value. When a potential step of 1V is applied, the capacity current is less than 1% of the steady state current for $\tau > 0.1 \text{ ns}$ for $C^0 = 10^{-6} \text{ mol cm}^{-3}$, $n = 2$. The same relation holds for an array of disks at the same potential but with non-interacting diffusion spheres.

Cyclic voltammograms at the smallest disk with a sweep rate of 20 mV s^{-1} in 1.0 mM potassium hexacyanoferrate (II) solution presented plateaus of diffusion-controlled currents like those obtained at a rotating disk electrode, indicating that currents are almost in the steady state in this time domain. The voltammograms had no evidence of chemical complications due to properties of the photoresist or the developing agents. In the chronoamperometric measurements, the potential was stepped from -0.05 V(SCE) to 0.7 V(SCE) , which is positive enough to oxidize the hexacyanoferrate(II) ion under diffusion control. Chronoamperometric curves between 0.05 and 3 seconds were obtained; data analyzed were the results of ensemble averaging ten runs. Average current densities are plotted against time in Fig. 2. At longer times the current is larger, the smaller the electrode radius. Thus the contribution of non-linear diffusion increases with decreasing radius, as expected.

Values of τ were obtained from values of I, t, a and C^0 by use of the inverse of Eqn. (2). Results for the four kinds of electrodes are presented in Fig. 3. The values of τ are proportional to t and the

slopes of the lines are equal to $4D/a^2$. Plots of $\ln(\tau/t)$ thus obtained against $\ln(1/a^2)$ show that the slope of the line is unity and that the intercept gives $D = 6.8 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$ (Figure 4). This is in good agreement with the value, $6.6 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$, obtained for the same solution from the Cottrell equation at a glassy carbon electrode without a mask. The literature value of D is $6.5 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$ [14]. All values of the logarithm of τ obtained in this experiment are plotted vs. $\ln(t/a^2)$ in Fig. 4. The value of D evaluated from the intercept of the line is the same as above, $6.8 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$.

Theoretical chronoamperometric curves calculated from eqn. (1) for $D = 6.8 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$ are drawn in Fig. 2. Agreement between theoretical and experimental curves is excellent.

From the predicted dependence of τ on t and a , shown in Figs. 3-5, it can be concluded that eqn. (1) with series expansion of $f(\tau)$ is verified experimentally to be valid in the ranges of $0.006 \leq \tau \leq 19.0$. Outside of this domain of τ , I - t curves obey the Cottrell equation or the steady state equation as described in the discussion previously [12]. Therefore eqn. (1) holds for any values of τ without question.

ACKNOWLEDGEMENT

This work was supported in part by the Office of Naval Research. The assistance of the National Research and Resource Facility for Submicron Structures, and in particular the assistance of Edward Wolff, Richard Tiberio, and Joseph Ballantyne in preparing the electrodes is gratefully acknowledged. The authors are especially indebted to Arthur F. Findeis for his helpful comments.

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FIGURE CAPTIONS

- Fig. 1 Variation of τ with It/nFC^0a^3 ; (a): calculated from the descending series, (b): calculated from the asymptotic expansion.
- Fig. 2 Real chronoamperometric curves at electrodes with radii of (Δ) 0.02, (\diamond) 0.04, (\odot) 0.1, (\ominus) 0.2 and (\circ) 1.5 mm and theoretical curves calculated from eqn. (1) by use of the value, $D = 6.8 \times 10^{-6} \text{ cm}^2\text{s}^{-1}$ at electrodes of (a) 0.02, (b) 0.04, (c) 0.1, (d) 0.2 and (e) 1.5 mm in radius.
- Fig. 3 Variations of values of τ with t at electrodes of (Δ) 0.02, (\diamond) 0.04, (\odot) 0.1 and (\circ) 0.2 mm in radius.
- Fig. 4 Dependence of τ/t on a^2 . Points are slopes from Fig. 3.
- Fig. 5 Plots of $\ln(I)$ against $\ln(t/a^2)$ at electrodes of (Δ) 0.02, (\diamond) 0.04, (\odot) 0.1 and (\circ) 0.2 mm in radius.

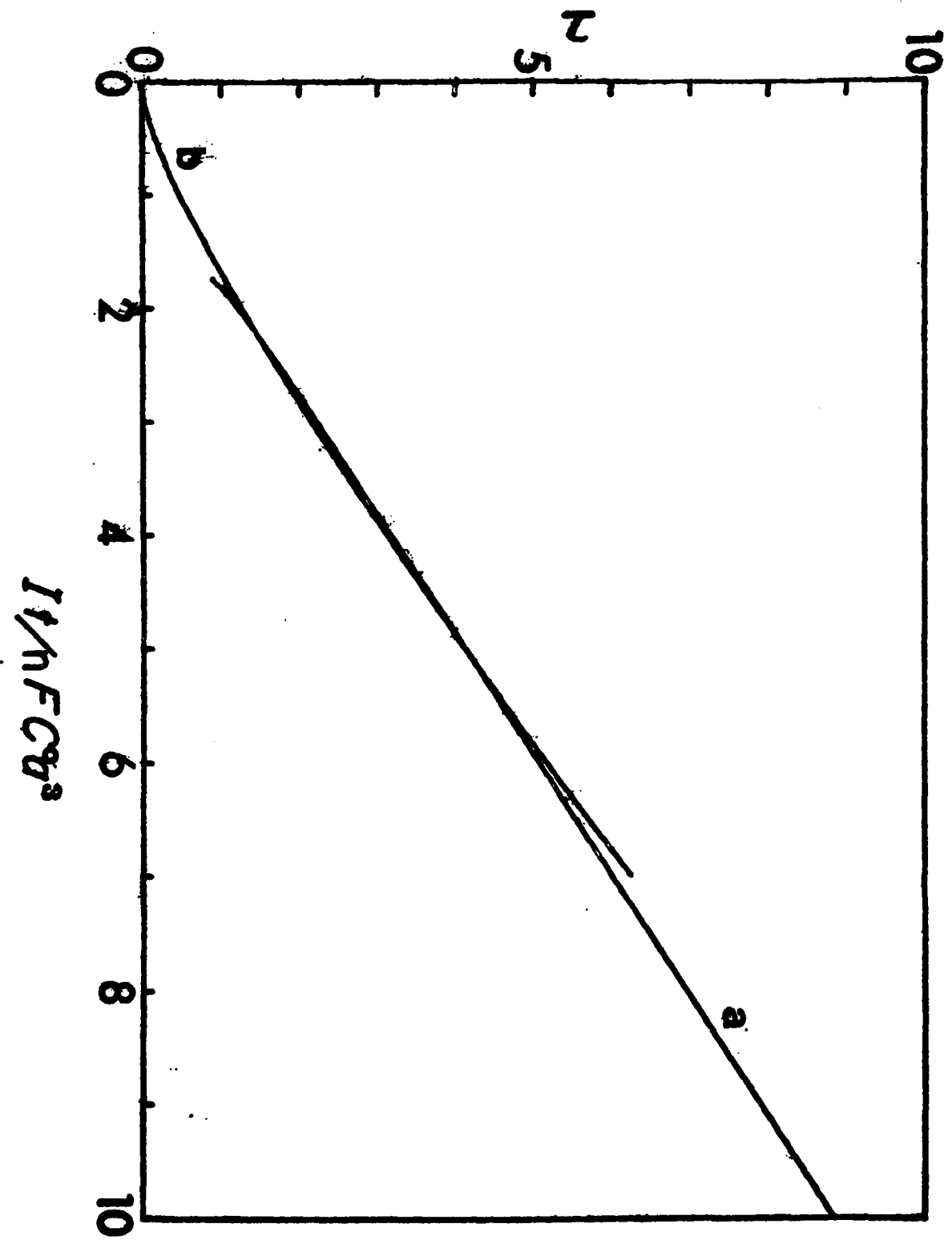


Figure 1

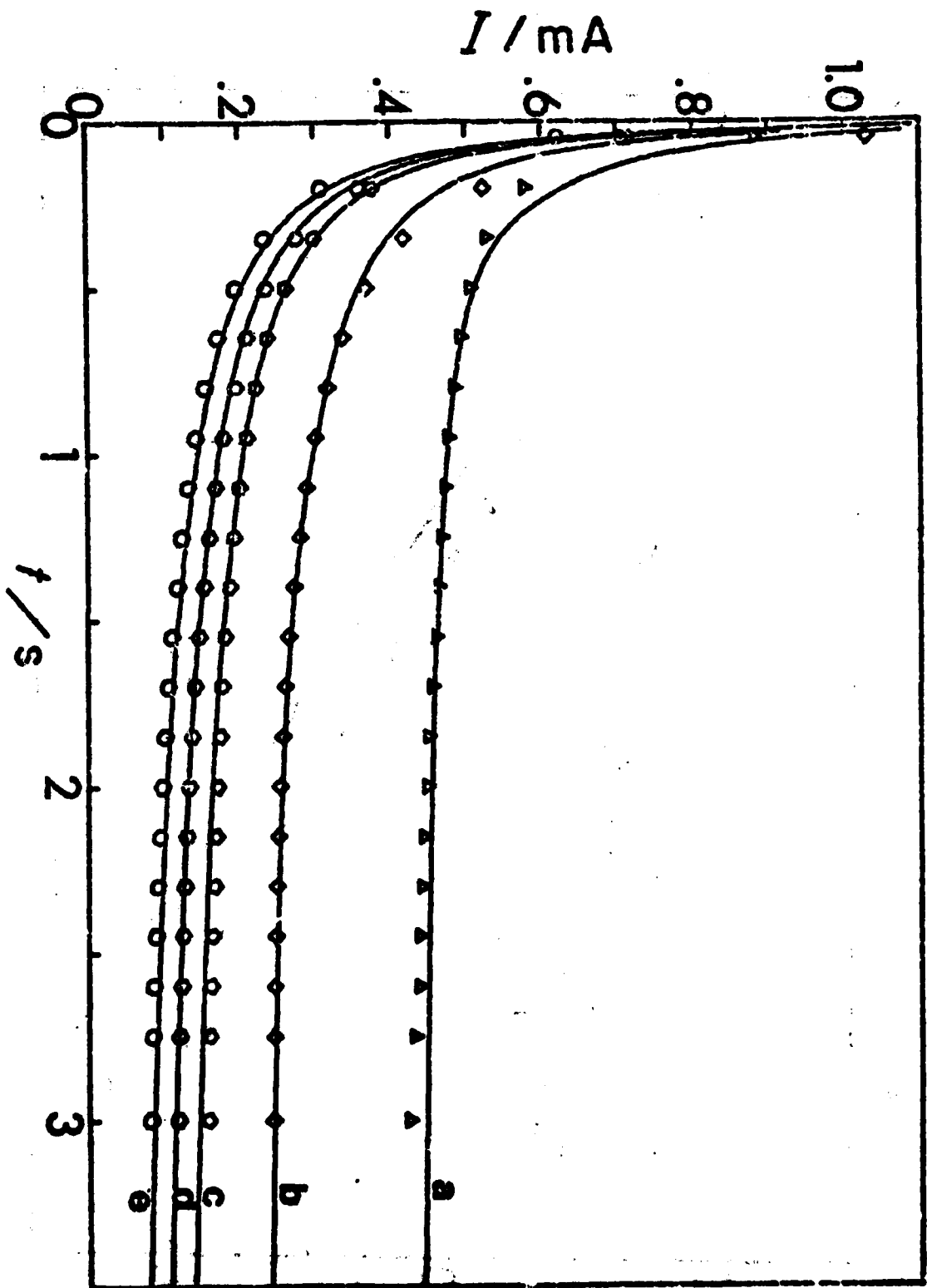


Figure 2

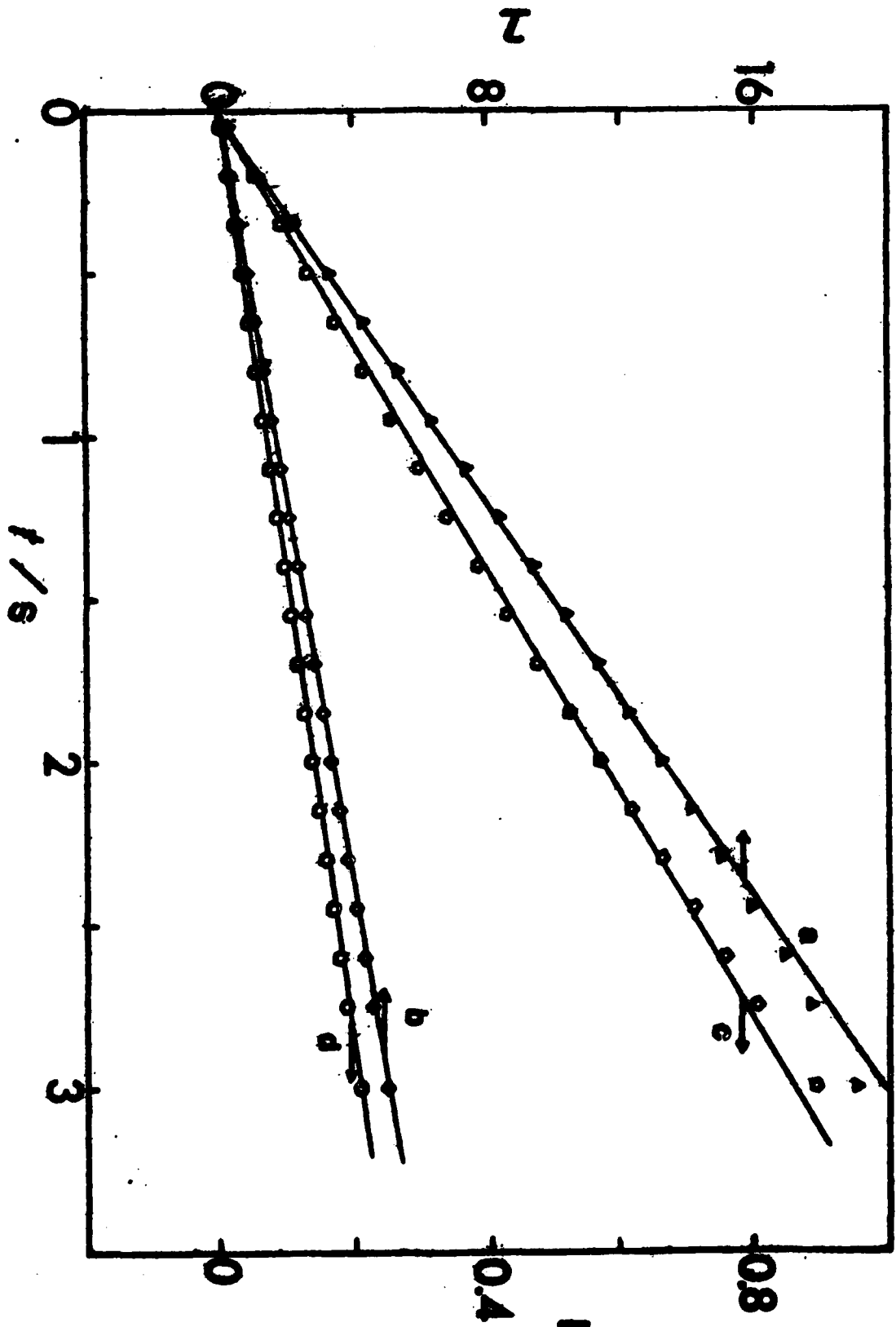


Figure 3

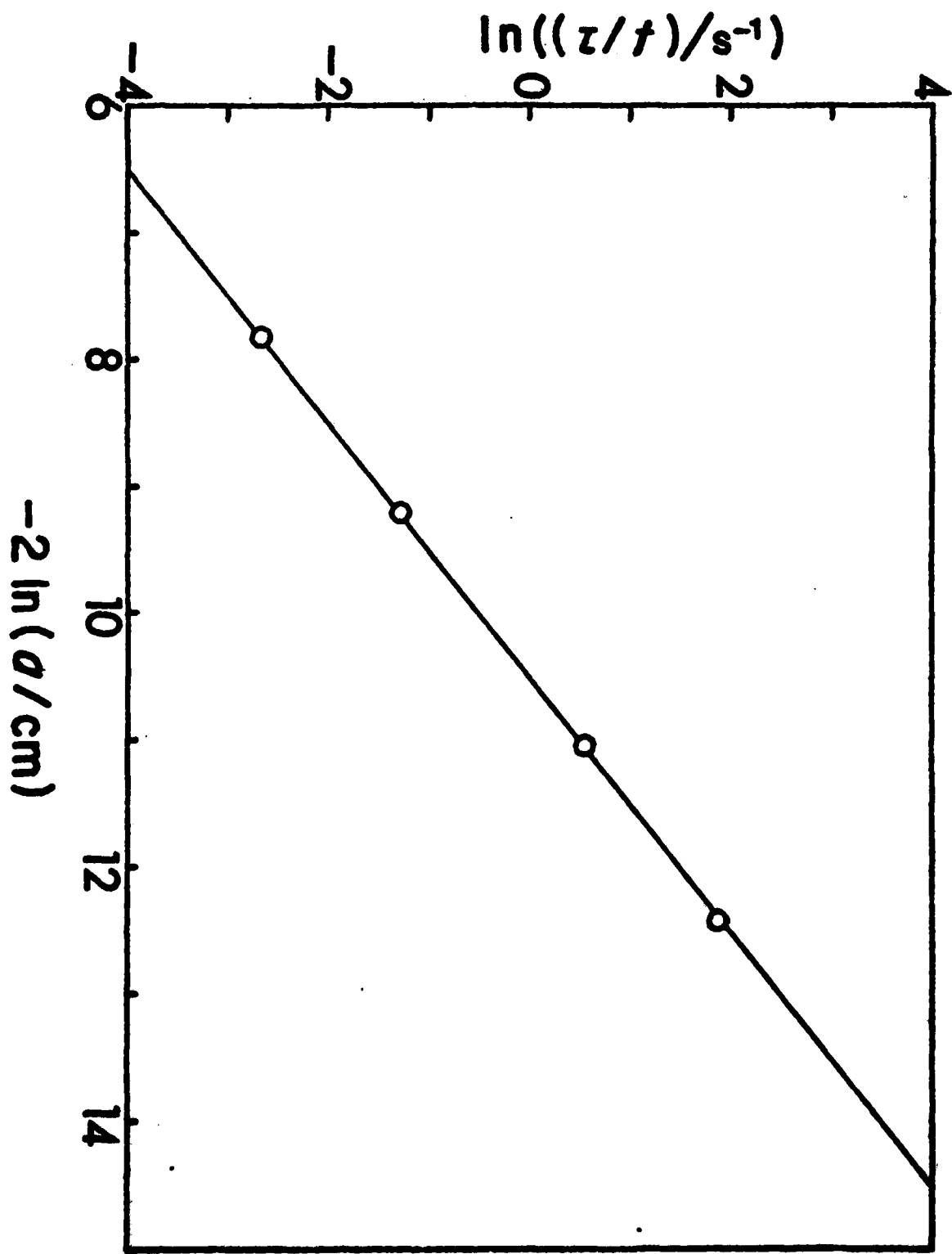


Figure 4

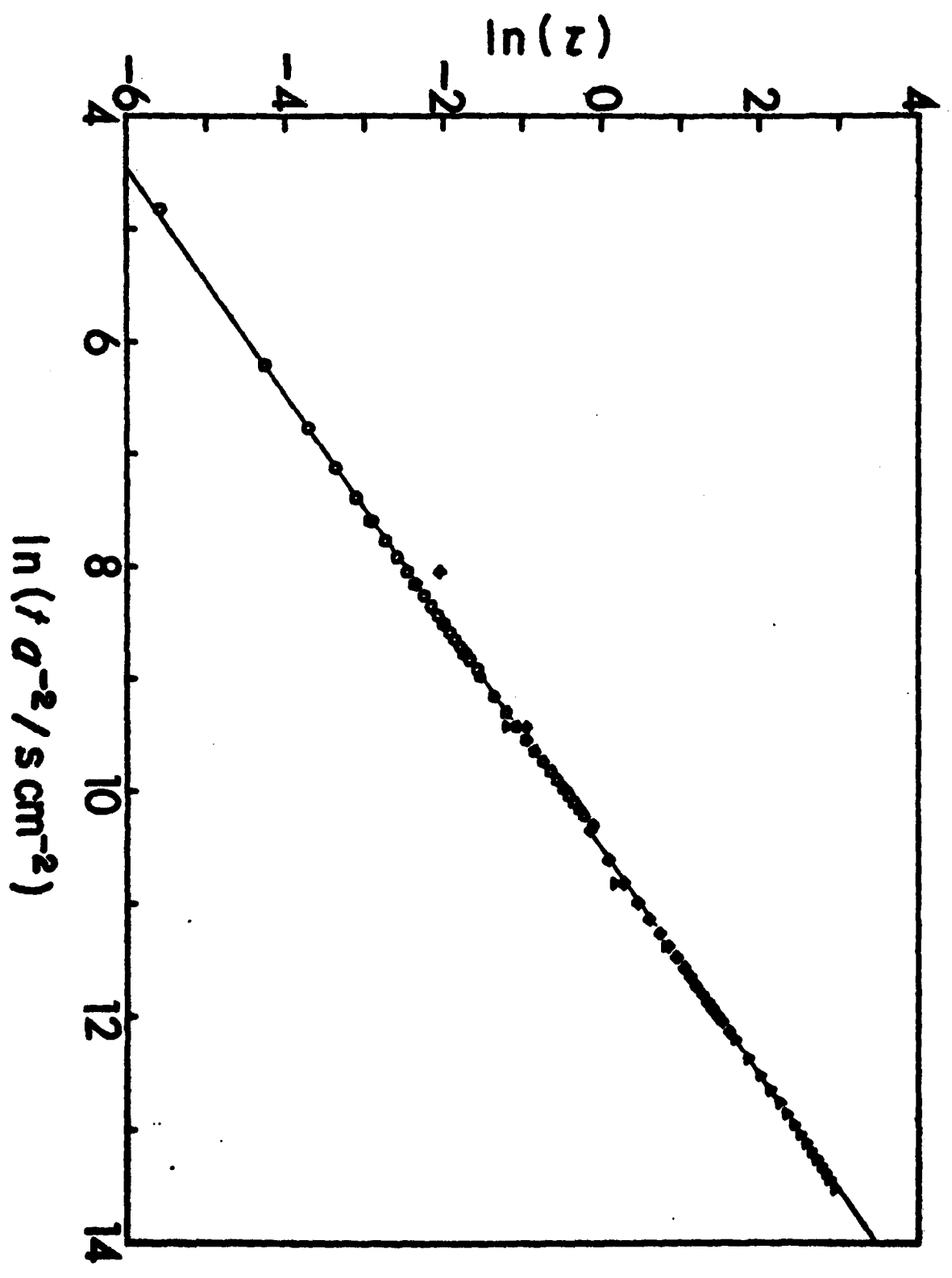


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