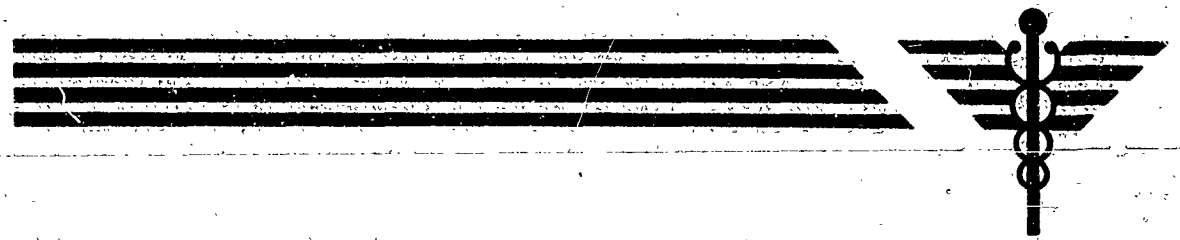
ARMY MEDICAL RESEARCH LABORATORY

FORT KNOX, KENTUCKY

REPORT NO. 87
2 July 1952

A ROTATING PLATINUM ELECTRODE FOR THE AMPEROMETRIC ARGENTOMETRIC TITRATION OF SULFHYDRYL GROUPS*

*Subtask under Effects of Irradiation, AMRL Project No. 6-59-08-013, Subtask, Enzyme, Endocrine and Metabolism Studies in Total Body Irradiation.



MEDICAL RESEARCH AND DEVELOPMENT BOARD
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DEPARTMENT OF THE ARMY

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A ROTATING PLATINUM ELECTRODE FOR THE AMPEROMETRIC ARGENTOMETRIC TITRATION OF SULFHYDRYL GROUPS*

by

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FORT KNOX, KENTUCKY
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ABSTRACT

A ROTATING PLATINUM ELECTRODE FOR THE AMPEROMETRIC ARGENTOMETRIC TITRATION OF SULFHYDRYL GROUPS

OBJECT

To develop a rotating variable speed platinum electrode which is free of any electrical interference. The electrode is used in the argentometric amperometric titration of sulfhydryl groups present in biological material.

RESULTS AND CONCLUSIONS

A rotating platinum electrode was developed for the amperometric titration of sulfhydryl groups. No electrical interference was encountered with this electrode. The electrode can be operated at speeds from 100 to 1200 rpm for long periods of time.

RECOMMENDATIONS

None.

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A ROTATING PLATINUM ELECTRODE FOR THE AMPEROMETRIC ARGENTOMETRIC TITRATION OF SULFHYDRYL GROUPS

I. INTRODUCTION

Kolthoff and Harris (1) described a specific argentometric titration for the determination of the sulfhydryl group, using a rotating platinum electrode. Benesch and Benesch (2) and Weissman et al. (3) modified this method in order to determine sulfhydryl groups in biological material.

In these methods, satisfactory electrical contact is not made with the rotating platinum electrode when operating at high speeds. If the motor is not completely insulated from the electrode, electrical interference results.

This report describes the construction of an electrode that does not have the disadvantages of the previously employed electrodes.

II. EXPERIMENTAL

The electrode, schematically shown in Figure I and illustrated in Figure II consists of two parts; the glass well and the glass tube with a bell-shaped rotating top.

The glass well, which is the stationary part of the electrode, is made from 40 mm and 16 mm diameter tubing. The side arm, made from 8 mm tubing, has a 20 mm length of platinum wire sealed through the end.

The electrode is made from pyrex tubing, the inside diameters of which are 8 mm and 28 mm. A platinum wire is sealed in the bottom of the electrode and extends to the outer edge of the bell. To insure contact the platinum wire is fastened with household cement* to the side of the bell, leaving the portion in contact with the mercury uncovered. The glass bell should not extend to a depth of more than 3 mm into the mercury in order to prevent excessive splashing of mercury at high speeds. The mercury bath is filled to a height of 6 to 10 mm.

Corks are used for bearings. The large cork which fits into the top of the well is not fastened to the glass well. The inside wall of the cork is covered completely with cement to give a smooth-finished surface. The inside walls of the two small corks, which are fastened permanently

* DUCO Household Cement, made by the DuPont Company.

to the glass well, are also covered with cement to give a smooth surface. The upper small cork should extend above the glass wall and slope outward to provide a run off for any mercury that splashes up inside the bell. The glass electrode should fit loosely into the two small corks but with no more than 1/32 inch clearance. This is to prevent the electrode from wobbling at high speed.

An electric power-driven variable speed laboratory stirrer was used with this electrode. The use of a small torque stirrer is advisable to prevent applying too much strain on the electrode. It is important to realize that the three corks play a leading role in the operation of this electrode. The corks should be aligned and the electrode tested by hand before attaching it to the motor.

The glass well is clamped to the same stand as the stirrer. A short piece of rubber tubing connects the glass tubing of the electrode to the shaft of the motor. Minor adjustments may then be necessary for efficient operation of the electrode. As can be seen in Figure II, a short tube of paper extends from the top cork in order to stop the mercury from creeping out of the well. A glass bead near the platinum wire protects it from being broken off.

Once the electrode is in operating position it need not be removed except for repairs. Oxidized mercury will form on the inside wall of the well and on the bell but it has not been found to interfere with the function of the electrode.

III. RESULTS

Variable speeds from 100 to 1200 rpm were used with this electrode. Figure III shows an example of the titration of 1 ml of 0.82×10^{-3}N mercaptobenzoxazole with 1×10^{-3}N $AgNO_3$ at three different speeds. It can be seen from these curves that an increase in speed increases the sharpness of the end-point.

IV. DISCUSSION

The electrode described here can be operated at greater speeds than have been reported by others (1, 3). With the increase in speed it is possible to increase the sharpness of the end-point.

V. CONCLUSIONS

A rotating platinum electrode was developed for the amperometric titration of sulfhydryl groups. No electrical interference was encountered with this electrode. The electrode could be operated at speeds from 100 to 1200 rpm for long periods of time.

VI. RECOMMENDATIONS

None.

VII. BIBLIOGRAPHY

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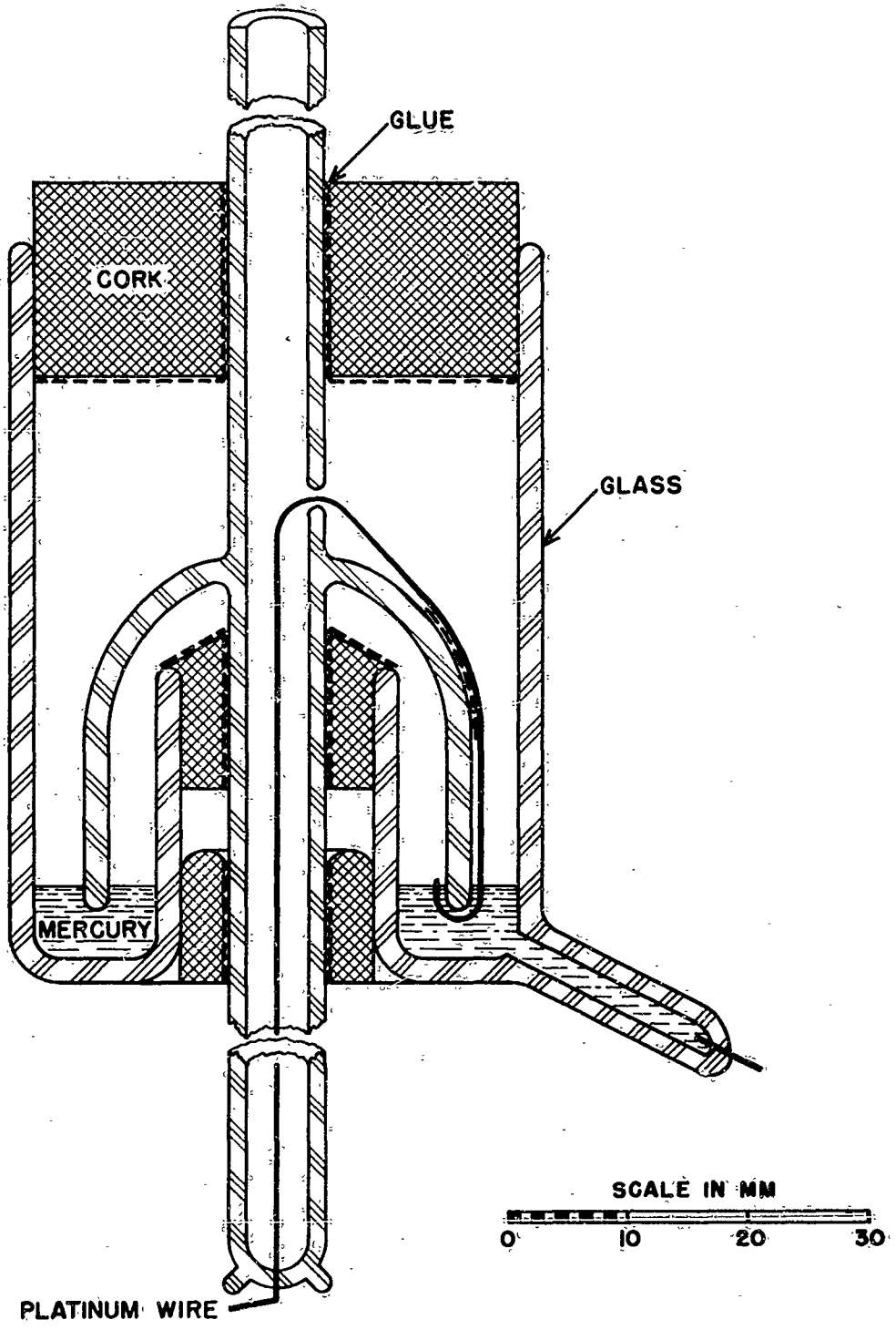

FIGURE 1 CROSS SECTION OF ROTATING PLATINUM ELECTRODE.

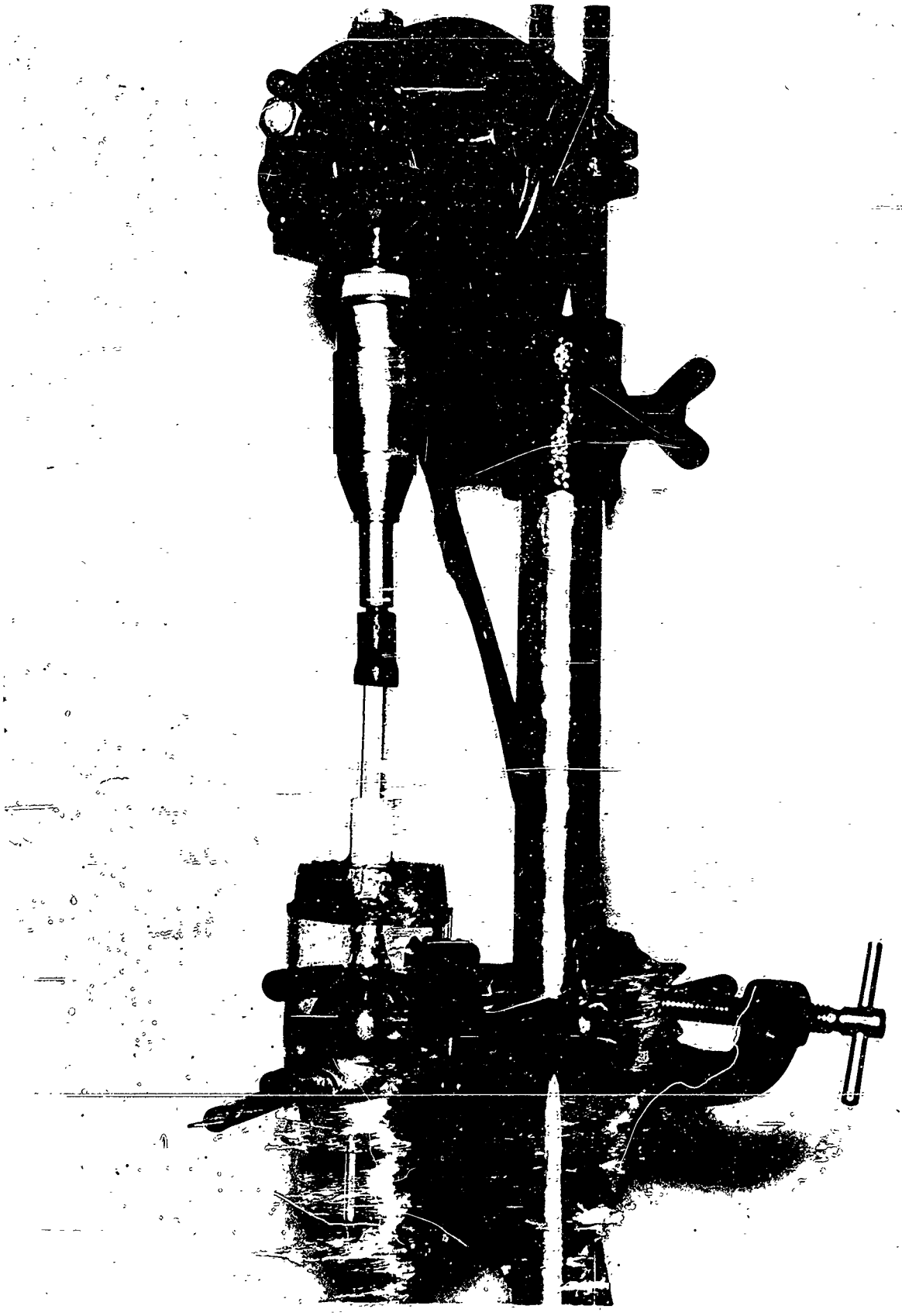

FIGURE 2 - AN ILLUSTRATION OF THE ROTATING ELECTRODE.

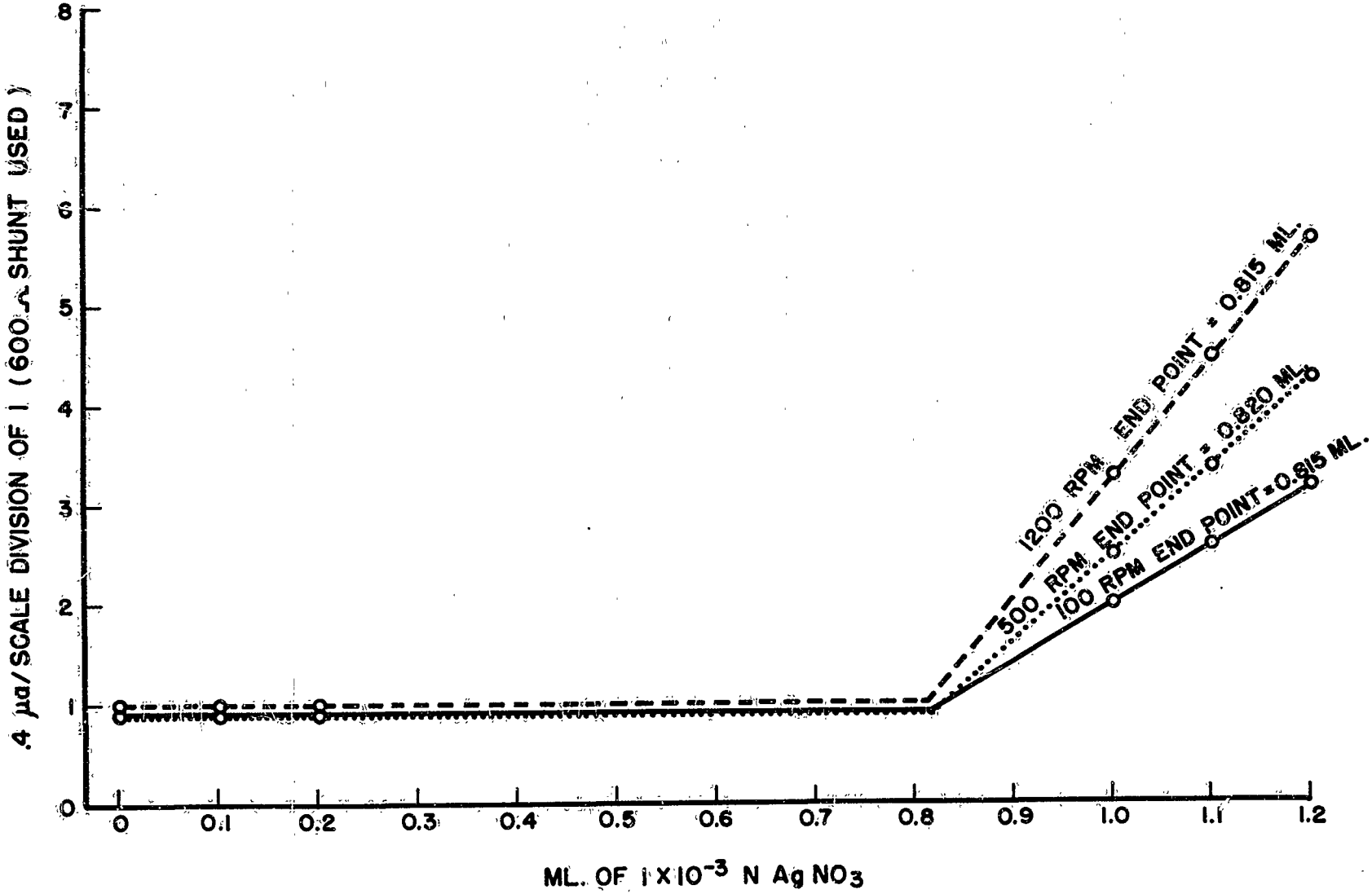

FIGURE 3 THE EFFECT OF CHANGE IN ROTATIONAL SPEED ON THE SLOPE OF THE TITRATION CURVE.

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Compounds, Organic - Analysis
Sulfhydryl
Electrodes

Chemistry (52)
Analytical Chemistry (3)

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23 Electrochemistry
Electrodes
Platinum
25 Platinum Electrodes, Sulfhydryl
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DIVISION: Chemistry (52)
SECTION: Analytical Chemistry (3)
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1. Compounds, Organic - Analysis
2. Sulfhydryl
3. Electrodes
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