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MOUND LABORATORY

Operated By

MONSANTO CHEMICAL COMPANY

MIAMISBURG, OHIO

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ELECTRODEPOSITION RESEARCH PROGRESS REPORT

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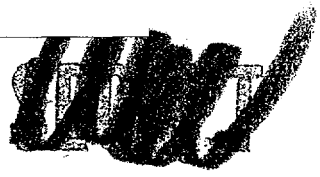


ELECTRODEPOSITION RESEARCH GROUP

W. Abel, R. Bell, J. Poppleton, E. Orban, and W. Raiff

ABSTRACT

Neutron Emission

Variation of time between rinses showed no correlation with the neutron counts on the finished gauzes.

Conversion of Production Solutions to Postum Solutions of Hydrofluoric Acid

The use of compressed teflon for filtering postum showed no improvement in the yield over uncompressed teflon. Polystyrene is not resistant to warm solutions of hydrofluoric acid which contain high concentrations of activity.

Liquid Junction Potentials

It is not possible to use a hydrogen electrode in nitric acid solutions.

Effect of Time Between Rinses on Plating Production Yields

As the time between removal of the gauze from the plating bath to immersion in the wash water is increased the amount of material remaining in the wash water is increased. The amount dissolving off the gauze greatly exceeds the solubility, and probably is removed by a mechanical process.

Effect of Current Density During Rinsing on Plating Production Yields

Contrary to general opinion the lower the current density during the rinsing operation the less the loss of postum.

Use of Detergents to Decrease the Liquid Film on Plated Gauzes

The addition of wetting agents to the plating bath is not very effective in reducing the amount of liquid which clings to the gauze when it is removed.

DETAILED REPORT

I. Study of Neutron Emission

A. Gauzes Plated out of Nitric Acid

It has appeared that neutron emission may be a surface phenomenon occurring during the preparation of postum gauzes since those gauzes exposed to air had abnormally high neutron counts and since the thicker the deposited plate the less the neutron count per unit of postum deposited.

A neutron study of gauzes which were exposed to the air for varying lengths of time between rinses was carried out along with other studies reported in Part III of this report. A series of six foils were exposed to air from eight to eighty seconds between the plating bath and the water rinse; and again between the water rinse and the acetone rinse. Table I shows the results of the neutron counts.

Table I

EFFECT OF TIME OF EXPOSURE BETWEEN RINSES ON NEUTRON COUNTS

Exposure Time (sec.)	Gauze No.	Neutrons/c./sec.
8	565	163
8	571	93
16	566	356
16	572	78
24	577	79
24	578	54
24	579	34
24	580	51
24	581	325
24	582	76
32	573	66
48	574	112
48	568	140
64	575	403
64	569	1138
80	576	65
80	570	164

It is obvious from these results that the exposure time between the plating bath and the rinses has no effect on the neutron counts the high results in the table may be traced back to imperfections in the canning process or other defects. Probably the layer of liquid over the surface protects the metal from oxygen which usually causes the high neutron count.

Future Plans

A study of surface effects on neutron counts will be made.

II. Plating of Postum from Hydrofluoric Acid Solutions

No progress to report.

A. Conversion of Production Solutions to Postum Solutions of Hydrofluoric Acid - R. Bell

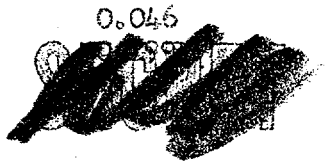
Two runs were made using the procedure described in the Electrodeposition Research Progress Report, June 1-30, 1948, MLM-138. The teflon filter used in these runs was compressed to have a pore size of two microns.

The results of these runs given in Table II show that the use of the compressed teflon showed no improvement in the efficiency of the process. The high concentration of activity has had an adverse effect on the polystyrene particularly at the elevated temperatures used. After a short exposure to activity the polystyrene began to craze. It is possible that the majority of the material lost during the process in previous runs was deposited in the cracks and should not be removed easily.

Table II

CONVERSION OF THE PRODUCTION SOLUTIONS TO POSTUM SOLUTIONS OF HYDROFLUORIC ACID

Run No.	1	2
Postum in Original Solution (c.)	26.8	27.1
Postum in First Filtrate (c.)	0.056	2.574
Postum in Product Solution (c.)	18.00	19.25
Postum in Precipitate (c.)	4.09	4.80
Postum in Rinse Solution (c.)	0.543	0.097
Postum in Vent Traps (c.)	—	0.046
Postum Lost in Process (c.)	3.93	[illegible]



Future Plans

Other plastic materials will be tried for use with "hot" hydrofluoric acid solution.

III. Plating of Postum from Nitric Acid Solutions and Other Media

A. Liquid Junction Potentials - E. Orban

A detailed report of the tentative procedure was given in the Electrodeposition Research Progress Report, July 1-31, 1948, MLM-152. The cell prepared for the measurement was

$$Pt/H_2/HNO_3\ (xm)/KNO_3\ (1.74N)/KCl\ (1.0N)/Hg_2Cl_2/Hg.$$

There was considerable doubt that the hydrogen half-cell would give reproduceable and reliable results in nitric acid. It was found that without hydrogen bubbling through the half-cell the potential approached a value of -0.9 v. The passage of hydrogen through the half-cell caused the potential to increase slowly until a value of -0.3 v. was reached. Upon cutting off the hydrogen, the current immediately began to drift back toward its original value. It was obvious from this that equilibrium values for a hydrogen electrode in nitric acid solution could not be attained.

Future Plans

Investigation into the possibility of using a glass electrode to replace the hydrogen electrode will be made.

B. Effect of Time Between Rinses on Plating Production Yields - W. Abel, J. Poppleton, and W. Raiff

The Electrodeposition Research Progress Report, July 1-31, 1948, MLM-152, described the procedure used for studying the effect of the rinsing time on the plating production yield. The first two runs were erratic because of mechanical difficulties which occurred. The results of the third run are shown in Table III. These data are plotted in Figure 1 and show a regular increase in the amount of postum stripped with increase in the exposed time between the plating bath and the water rinse. The points fall on the curve as well as can be expected in view of the fact that the different gauzes might hold up a different amount of solution each time.

Table III

STRIPPING OF POSTUM IN RINSE SOLUTIONS

(Variable Rinsing Time)

Gauze No.	571	572	573	574	575	576
Time Between Rinses (sec.)	8	16	32	48	64	80
Postum Stripped in Water Rinse (mc.)	34	139	281	454	535	585
Postum Stripped in Acetone Rinse (mc.)	22	7	4	3	1	3
Postum Unplated (mc.)	0	0	18	503	179	349
Postum on Gauze (mc.)	1408	1091	1219	481	774	521
Total Postum (mc.)	1464	1235	1522	1441	1489	1458
Percentage of Postum Stripped in Rinse Water	2.3	11.2	18.5	31.5	35.9	40.4

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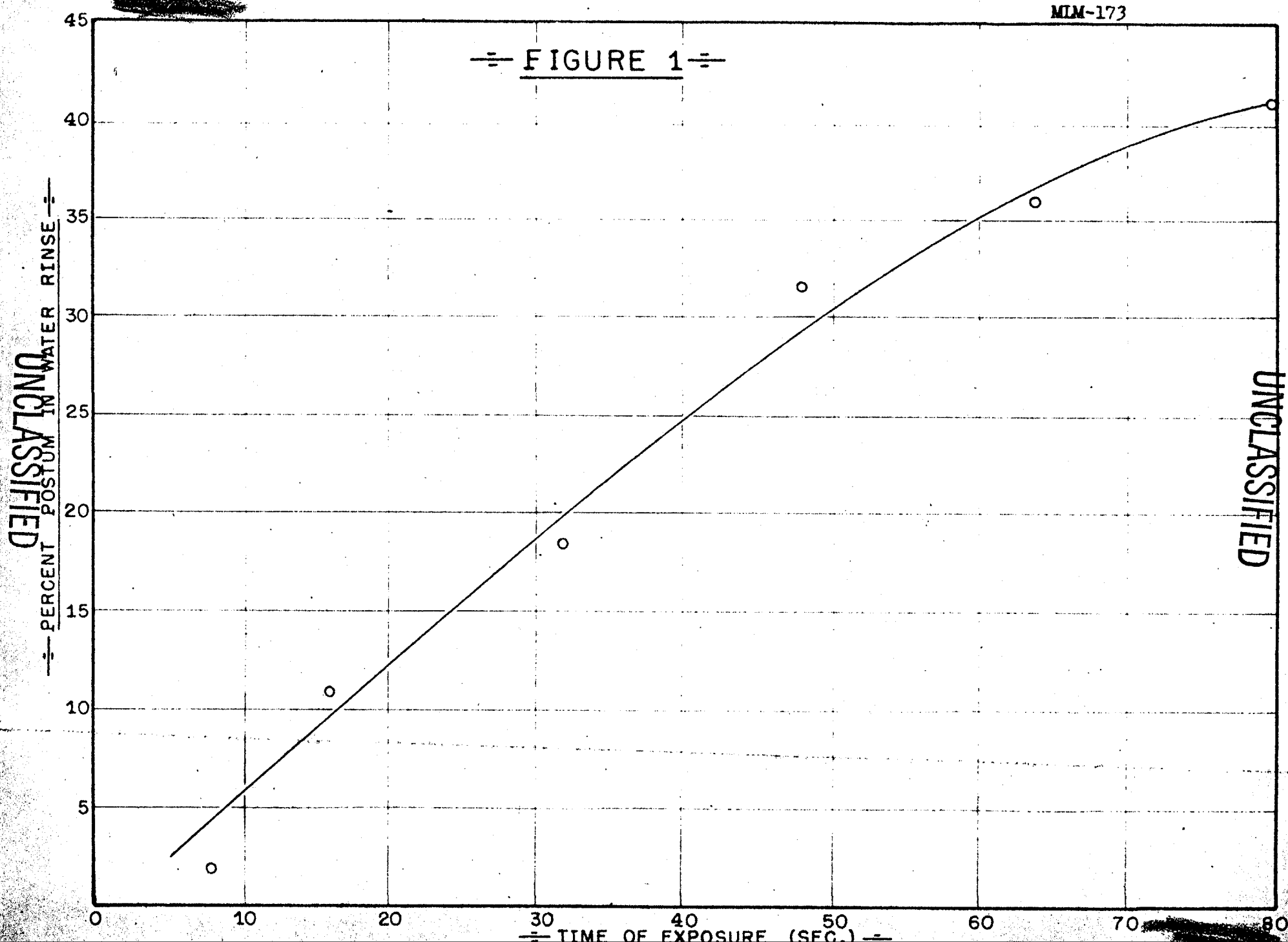
FIGURE 1



The question arose whether the postum was being dissolved or whether it was undergoing a corrosive action and flaking off. Actual weighings determined that 0.010-0.013 ml. of acid clung to the gauze on being removed from the solution. The solubility of postum at 32.6°C. is 0.370 units/ml. as reported in Fundamental Research Progress Report, September 15-30, 1947. This permits about 5-6 mc. of postum to dissolve in the solution held up by the gauze. It is very apparent that a mechanical process is responsible for the removal of the postum from the gauze. A very short exposure time would be the recommended procedure for transferring gauzes.

C. Effect of Current Density During Rinsing on Plating Production Yields

Theoretically it would seem that high current densities would prevent any postum from going into solution during the washing procedure, and if any did it should be replated immediately. A study was instituted to investigate the effect of the current on the dissolution of postum in the plating rinses.

The runs were carried out as described in the Electrodeposition Research Progress Report, July 1-31, 1948, MLM-152, except that the current was varied from run to run. The data for six foils plated appear in Table IV. Data taken from the plating residue and acetone rinse have been omitted. Figure 2 shows a plot of the results.

Contrary to opinion increasing the current density during the rinsing procedure does not decrease the amount of material in solution, but increases it. This may be explained by the heavy gassing which occurs under high current density. The plate is loosened while it is in contact with acid during the transfer from the plating bath to water rinse. Under heavy gassing parts of the plate break off as it is dipped into the water rinse. The heavier the gassing, the more breaks off. It should be pointed out that the runs were made with a 24 second pause between rinses which is longer than is recommended for production runs. Low current densities are desirable during the rinsing procedures regardless of the length of time exposed.

Several runs will be made to investigate the effect of zero current density in order to reaffirm the low results obtained.

FIGURE 2

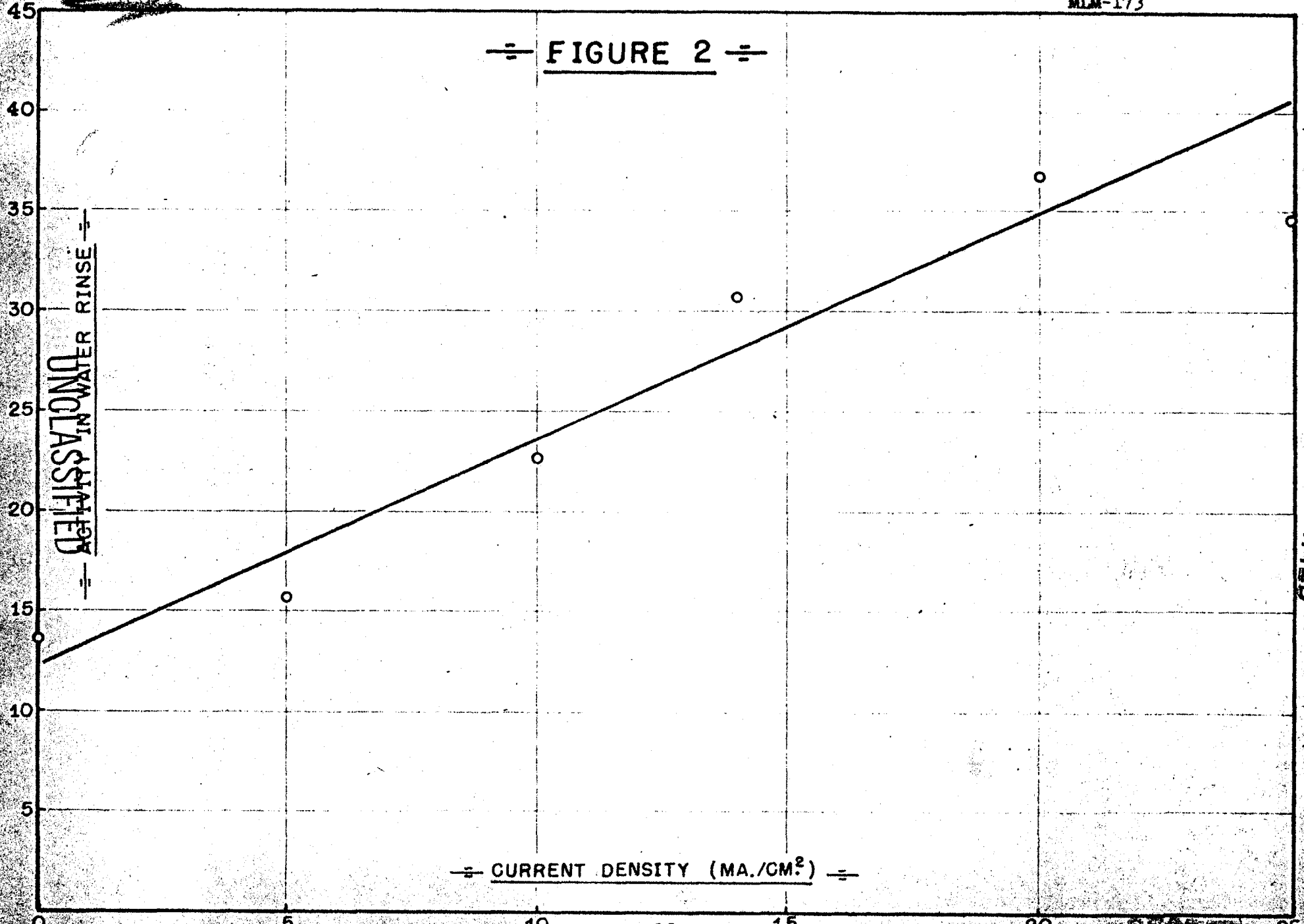


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Table IV

EFFECT OF CURRENT ON LOSS OF POLONIUM DURING RINSING

Gauze No.	577	578	579	580	581	582
Time Between Rinses (sec.)	24	24	24	24	24	24
Current Density ma./cm.^{2}	25	20	15	10	5	0
Postum Dissolved in H_2O Rinse (mc.)	655	694	579	428	293	258
Postum of Gauze Calorimetric Value (mc.)	1190	1127	1246	1404	1554	1574
Total Postum Used (mc.)	1884	1885	1880	1878	1864	1880
Per cent Postum in Water Rinse	34.7	36.8	30.8	22.8	15.7	13.7

D. Use of Detergents to Decrease the Liquid Film on Plated Gauzes

The use of detergents for decreasing the amount of acid retained by a gauze when removed from the plating bath was suggested by R. A. Staniforth (See Information Report, MLM-130). The reduction of the surface tension of the acid solution by the addition of a detergent should decrease the holdup of the gauze.

The investigation was carried out on the assumption that 1.5 N nitric acid solutions would behave the same as the production solutions at Unit IV.

Two simple weight measurements made on a dry gauze in a weighing bottle and on the same gauze after it had been dipped in the acid and placed back in the same weighing bottle. This gave the amount of acid solution which the gauze carried with it. The procedure was repeated except that five per cent detergent was added to the acid solution. The following

wetting agents were tried. Dupont Petrowet R., Triton 720, 1 amino - 2 - napthol - 4 - sulfuric acid and Neomerpin.

The volume of liquid on the gauze with and without detergent in the solution showed that it was reduced ten per cent by Triton 720 and only five per cent by the other three. A visual examination showed that a small droplet which had remained upon removal from the acid solution was much smaller after the addition of the wetting agent. No change should be observed in the amount of solution filling the wire mesh. Since the reduction of material was such a small amount, it is evident that the addition of a wetting agent would do little to decrease the amount of postum being lost, but might do much to increase the amount of contamination in the final gauze.

Future Plans

After the completion of more urgent work this research will be extended to an investigation of other wetting agents and to the use of hot solutions.

IV. The Solubility of Q in Various Media

No progress to report.

V. Miscellaneous

A. A circular type plating unit has been designed for Unit V.

B. Photographic equipment has been revised and put into working order.

C. A new set of calomel cells have been prepared.

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