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MONSANTO CHEMICAL COMPANY - UNIT III

DAYTON, OHIO

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ELECTRODEPOSITION RESEARCH PROGRESS REPORT

Date: July 1-31, 1948 UNCLASSIFIED Prepared by: E. Orban

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ELECTRODEPOSITION RESEARCH GROUP

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

ABSTRACT

Liquid Junction Potential of the Cell Used in Production Plating

Apparatus is being constructed to determine whether it is feasible to measure the liquid junction in the cell used in the production plating of postum. Literature references are skeptical about the possibility of using a hydrogen electrode in the presence of nitric acid. However, since no reference could be found which said definitely that the measurements could not be made, the decision was to investigate the use of the hydrogen electrode in nitric acid.

The Effect of Rinsing Time on Plating Production Yield

The procedure for determining the amount of loss of postum as related to the time between the removal from the plating bath and the immersion into the water rinse has been worked out. Two runs have been made, neither of which were very successful because of mechanical difficulties which occurred during the runs. The second run showed, however, that as the time increased the amount remaining in the water rinse also increased very rapidly.

Teflon as a Filter Medium

Teflon has been found to be an excellent medium for filtering aqueous hydrofluoric acid solutions.

DETAILED REPORT

J. Poppleton, who has been on temporary loan to Unit IV, has returned to the group. C. Neibel has left the employment of the company.

I. Plating of Postum from Hydrofluoric Acid Solutions

A. Plate Quality

1. Neutron Emission

Nothing to report.

2. Adherence of Plate

Nothing to report.

3. Photographic Examination of Foils

A newly designed cell for photographing postum gauzes has been received and will be tried.

4. Inert Plating Cell

Nothing new to report.

B. Solution Conversion - R. Bell

New Polystyrene equipment has been constructed to replace some that has been broken in service.

1. Teflon as a Filter Medium - R. Bell

It has been found that a very excellent method of removing suspended material from a solution of aqueous hydrogen fluoride is to filter it through a funnel machined out of teflon with a disc of a porous teflon sheet. It was found that the spongy teflon used in this experiment had a porosity of about 16.5 microns. It was possible to reduce the size by compression to about 2.0 microns. In this way any porosity of material can be obtained depending on the original size of the pores and on the pressure used to compress it. Full details of the method may be found in Information Report, MLM-146, by R. Bell.

II. Plating of Postum from Nitric Acid Solutions

A. Liquid Junction Potentials - E. Orban

The cell used in production plating has a platinum anode and a platinum cathode. The potential at which the platinum cathode rests is not independent of the current, but varies with it. In order to separate postum from other cations by electroplating out of solution, it is necessary to establish the plating potential at a value which will allow complete plating of postum, but will preclude the deposition of as many other cations as possible. This potential

has been worked out by M. M. Haring (see Information Report #22), and has been found to be about 0.00 v. to the normal calomel electrode. (-0.2801 v. to the hydrogen electrode.)

Several factors were assumed in his determination which bear experimental investigation. The first is that the bismuth is in low enough concentration that it will not deposit at the potential usually used for plating; second, that the liquid junction potential is not a serious factor in the plating of postum. In actual practice it has been found that the bismuth concentration will vary, and there is a possibility that in some runs, when the concentration is high, a small amount will plate out. Also, variations in temperature and acid concentration may effect the liquid junction to a degree which may interfere with the separation of bismuth and postum.

In view of the latter possibility, it was decided to make a measurement on the liquid junction potential by setting up a cell comprising the same liquid junctions, but in which the potentials of the two half cells were known. Hence, it would be possible to calculate the liquid junction potential by a simple subtraction of the potentials of the two half cells. The following cell was arranged:

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

In this cell the potential of the normal calomel electrode is well known; however, there is some doubt about the potential of the hydrogen half cell in nitric acid. A survey of the literature shows no data and contains no discussions about the hydrogen-nitric acid half cell. Several books say merely that the hydrogen half cell must be free of nitrate ion. Off hand, it would appear that measurements with the hydrogen-nitric acid half cell would be impossible. The oxidation-reduction potential of the half cell reaction

$$HNO_2 + H_2O \rightleftharpoons NO_3^- + 3H^+ + 2e^-$$

is -0.96 volts. Depending on the concentration of materials present this reaction may establish a potential which interferes with the potential of the hydrogen electrode. However, no other half cells which permit the use of nitric acid as a solvent are known, with the possible exception of the glass electrode; hence, the decision was made to go ahead and investigate the behavior of the hydrogen-nitric acid half cell. The investigation will determine whether the potential

falls approximately where it should, and then whether it behaves like a hydrogen half cell.

The apparatus being constructed is a water bath, and a hydrogen-calomel cell. Figure 1 shows a sketch of the cell.

The results of preliminary experiments to determine the behavior of the hydrogen half cell will be reported next time.

B. Effect of Rinsing Time on Plating Production Yield - W. Abel, J. Poppleton, and W. Raiff

In a discussion with the Electrodeposition Production Group on June 4, 1948 (memo to D. L. Scott by R. G. Lantz, June 14, 1948) it was learned that a serious loss of postum was occurring during the water rinse. All of the possible difficulties were discussed (see Information Report, MLM-130, by R. A. Staniforth), and the conclusion was reached that the Electrodeposition Research Group would immediately investigate the effect of rinsing time on the loss of postum in the water rinse.

The first program undertaken was the investigation of the effect of varying the length of time between the removal of the plated gauze from the plating solution until it was dipped into the water rinse; then, repeating the procedure using the same length of time between the water and the acetone rinses. The electrodes were made of 45 mesh wire gauze approximately 5 mm. x 8 mm. in size which gave a current density comparable to that of production plating on gauzes. Each of the foils was plated out of 2.5 N nitric acid. Table I shows the data obtained from this run.

The erratic results obtained immediately led us to suspect that a short had occurred during the first rinse, thereby cutting the current off the remainder of the runs and allowing a great deal of the material to redissolve.

A second effort was made following the same directions and again difficulty was encountered in the latter part of the run. However, in this case 1.5 N nitric acid was used. Table II shows the data of this run. In addition to the other data, the gauzes were calorimetered to check the amount of activity plated out. A short circuit in the last two runs caused some of the material to return into the solution.

FIGURE 1

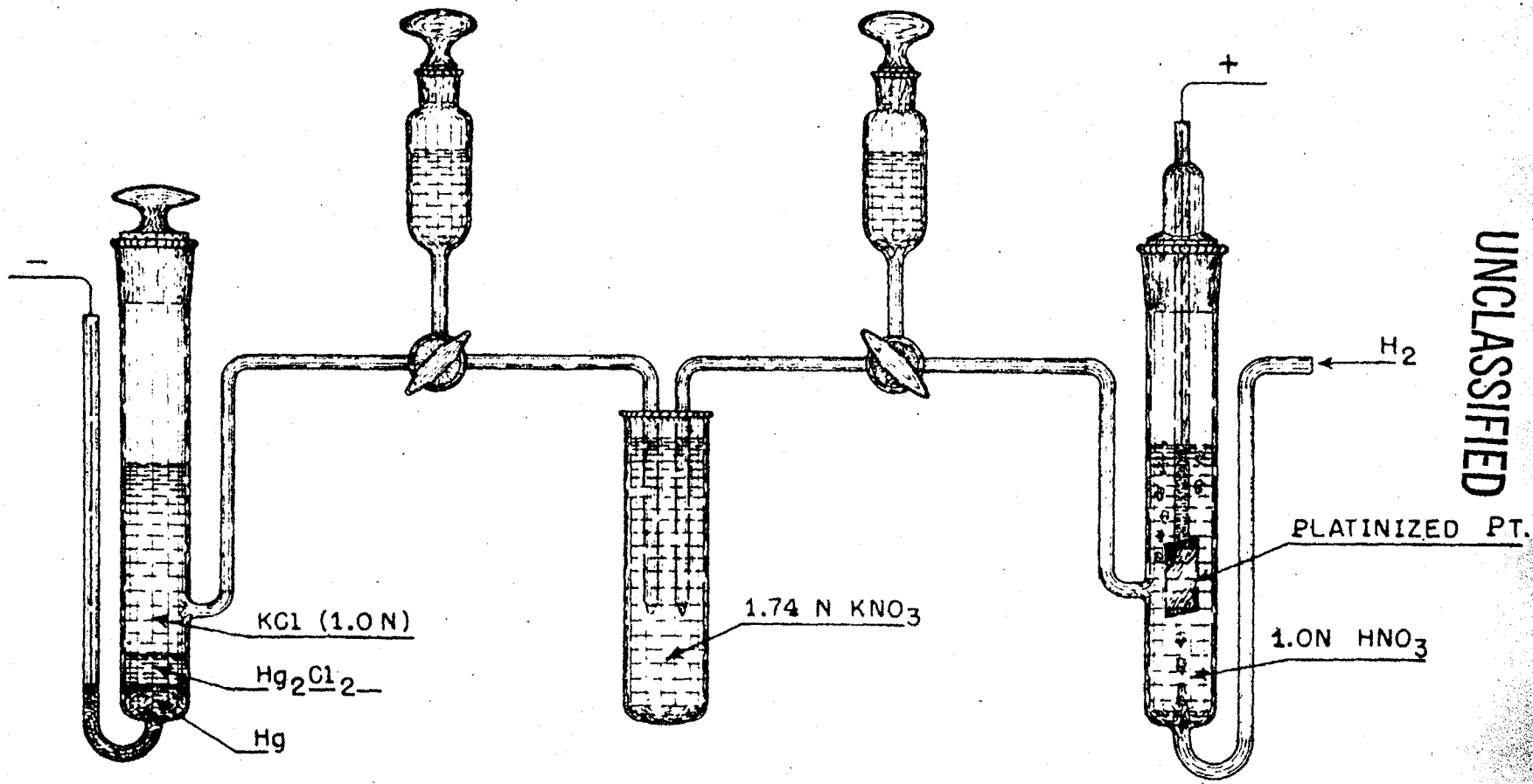

Table I

EFFECT OF VARYING RINSING TIME

Gauze No.	559	560	561	562	563	564
Total Activity Taken (mc.)	2770	2770	2770	2770	2770	2770
Time Between Rinses	8 sec.	16 sec.	32 sec.	48 sec.	64 sec.	80 sec.
Postum Dissolved in Water Rinse (mc.)	280	230	364	501	91	354
Postum Dissolved in Acetone Rinse (mc.)	8	9	5	7	3	2
Postum Remaining in Plating Residue	19	1236.0	1826.0	1479.0	2325.0	2075.0

Table II

Gauze No.	565	566	567	568	569	570
Total Activity Taken (mc.)	2721	2721	2721	2721	2721	2721
Time Between Rinses (sec.)	8	16	32	48	64	80
Postum Dissolved in Water Rinse (mc.)	34	370	930	824	1070	1169
Postum Dissolved in Acetone Rinse (mc.)	8	4	1	2	2	3
Postum Remaining in Plating Residue (mc.)	1	21	2	46	1093	870
Postum Plated out on Gauze (mc.)	2441	2534	-	2407	2702	2464
Neutrons/sec./c.	163	356	-	140	1138	164
Percentage of Total Activity in Water Rinse	1.2	13.6	34.2	30.3	[illegible]	[illegible].0

Figure 2 gives a plot of the percentage of the total activity which went into the water rinse against the time between the removal of the gauze from the plating bath and the immersion into the water rinse. The points as plotted appear erratic and the dashed line is a rough estimate of the average line.

It will be noted that the amount of material dissolving in the acetone in both experiments is small enough to be ignored in the overall consideration of the problem.

FUTURE PLANS

A third experiment is underway in which the mechanical errors involved in the previous two experiments will be eliminated.

III. Solubility of Postum in Various Media

No further developments on this problem.

IV. Miscellaneous

a. Preparation and Testing of Standard Reference Cells - W. Abel

A new series of reference normal calomel electrodes is being prepared.

b. Pipet Construction and Calibration - W. Raiff and W. Abel

A number of polystyrene pipets have been constructed and calibrated for use at Unit III, and for future use at Unit V.

c. Unit V Design - J. Poppleton

A plating unit in which the entire mechanism can be turned so that each cell will be at the front of the dry box while being put into operation has been designed.

Photographic equipment is being gathered and is being put into working order for use at Unit V.

EO/mw

FIGURE 2