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UNITED STATES ATOMIC ENERGY COMMISSION

DEVELOPMENT REPORT FOR ELECTROLYTIC CELL

By

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For The Atomic Energy Commission

H. F. Canale
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931-1

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EAH-135
R5OGL19
Page 1

TABLE OF CONTENTS

	<u>Page</u>
INTRODUCTION	1
APPROACH TO THE PROBLEM	2
DESIGN OF CELL	6
Tank Body	6
Cooling Jacket	6
Anode	7
Assembly of Tank	8
Tank Cover	8
Cathode Assembly	10
Pyrex Retaining Wall and Base	10
Power Supply	11
APPENDIX I	12
APPENDIX II	14

931

2

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EAH-135
R5OGL19

INTRODUCTION

✓ The objective of this project was to remove impurities in the form of lead-dioxide from a nitric acid solution by electrolysis. *PbO2* *nitric*

This electrolytic cell must process up to a 15 gal. batch in a minimum of time. It is desirable that the impurity remaining after processing be less than 20 parts-per million. *1 part / 2*

The design and construction of the cell was to be such that existing facilities at the Hanford Works would be utilized.

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931 008

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EAH-135
R50GL19
Page 2

APPROACH TO THE PROBLEM

Our analysis indicated that there are two general approaches to the solution of the problem imposed by the requirements of the subject cell. Lead impurities may be removed from the solution by:

1. Firm adherence of lead dioxide particles to the anode surfaces, or
2. Unfirm adherence of these particles, keeping fallen particles at anode potential to prevent dissolution.

Solution No. 1 requires good plating techniques as follows:

1. Large anode area.
2. Closely controlled temperature for proper plating adherence.
3. Closely controlled current density for optimum plating adherence.
4. Thorough agitation for removal of gas inclusion from anode surfaces.

There are several disadvantages involved in achieving the above listed characteristics. A large anode area is needed to keep the weight of lead impurities per unit anode area to a minimum. Thus the cost of the precious metal anode structure is high and the structural assembly of the anode is complex. It was found by test that about 6000 square inches of anode are required for good lead adherence. With plating current densities of only .1 amp/in² the total current requirements are 600 amperes. It is desirable to keep the plating current density to about .2 amp/in² which makes the current requirements

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531 004

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EAH-135
R5OGL19
Page 3

of the cell far above the capacity of available feeders at the Hanford installation.

The limitation of instrumentation devices fixed the electrode structure to a coaxial type. The minimum depth of a $7\frac{1}{2}$ gallon solution was fixed at about 6 inches and the most feasible way to obtain the necessary anode area was as shown in drawing WW-9629648.

This electrode structure is complex and makes the heat transfer problem very difficult. The most feasible cooling method is to jacket the outside of the tank. Then the liquid volume near the center of the cell would not be cooled readily even with circulation spaces in the electrodes.

Test setups (1 liter or less) were made simulating the plating condition required for operation of the described cell. The average impurity remaining in acid solution was about 75 parts/million for 8 to 12 hour runs. While this impurity is tolerable, it is not entirely satisfactory. Apparently, some of the lead impurities were going back into the solution. This dictated more anode area and more closely controlled current and temperature.

All these factors led to the conception of a different type of cell operation. If good plating adherence on the anode surface was difficult to attain, it might be possible to catch and retain the non-adhering particles at anode potential. A simple test setup proved that this type of cell was feasible. The major problems in the previously contemplated design were no longer present with this

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EAH-135
R50GL19
Page 4

second cell design. The first test run was carried out with the cell shown in Figure 1:

FIGURE I

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EAH-135
R5OGL19
Page 5

Using a batch of about 1 liter the test showed that lead dioxide particles were collected on the platinum sheet "catcher" and did not go back into solution. The impurities in the solution after 14 hours were less than 1 part per million. Several other runs were made varying current density and temperature. It was found that current and temperature were not critical and that solution purity of 5 to 15 parts per million was possible with 6 to 8 hour runs.

This type of cell design was expanded and changed in details to give the design shown in the accompanying assembly drawing W-9076925 and WW-9629649. The present design consists of a single anode surface and single cathode surface with a retaining wall and anode potential catching surface to guide and retain non-adhering lead particles.

Agitation, in the early tests, was accomplished by use of a motor driven, screw type, glass stirring rod. Since mechanical stirring presents design problems not easily overcome, several types of magnetic agitation systems were considered. The use of a single permanent magnet at the center of the cell seemed the most efficient location from the magnetic field considerations. It also presented no great design problems and was least expensive.

A test run using this type of magnetic agitation indicates that the operating time may be further reduced. This is due to the more complete stirring given the solution because of the increase in flux density (and stirring force) around the middle section of the magnet.

Results of agitation tests made on a full scale cell model are presented in Appendix 1.

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531 007

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EAH-135
R5OGL19
Page 6

DESIGN OF CELL

Tank Body

The tank can be made from 347 stainless steel. The bottom of the tank is a concave surface for added strength. The tank wall thickness is $3/16$ of an inch. Using 150 lbs/in^2 explosion pressure the stress setup in the cylindrical portion of the tank is $8080 \text{ \#/in}^2$, and $13,350 \text{ \#/in}^2$ in the spherically shaped bottom. The tank will be at anode potential and thus must be insulated from ground potential. This is accomplished by insulating the tank from the grounded cover as shown in the outline drawing WW-9629649.

Cooling Jacket

The cooling jacket is made up of another tank and spacers. The flow of cooling water in the jacket is directed by the spirally wound spacer. This type of cooling is used with rectifier tanks and has proved satisfactory. It will be noted that the water enters at the bottom. This lowers the cooling time before the jetting-out period and aids establishment of the jetting-out vacuum.

The cooling water pipes must also be insulated from the grounded installation piping. This can be accomplished by a length of polystyrene, polyethylene, or glass tubing. It is preferable to use the plastic materials unless steam or hot water are required for heating the cell. It is not considered that external heating will be necessary to raise the solution

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EAH-135
R5OGL19
Page 7

temperature 10° or 15° C. It has been found that the most important requirement for over-all fast plating is a good firmly adhering base coat of lead dioxide at the anode. This can be accomplished by operating with a low current density at the beginning of the process at temperatures of 35° to 60° C.

With this type of cell design the only important volume of the batch, to hold in a temperature range, is that at the anode plating surface. The total strength of the tank is increased considerably by the addition of the water flow guide spacers and jacket.

Anode

The tank anode liner will be platinum. It has been found that gold is not suitable for the plating surface. Analysis of test run solutions show that colloidal gold particles are present when gold is used as an anode.

The total effective anode plating area is about 1000 square inches. If total cell current is kept under 400 amperes, the current density will be about .4 amps/in². Attached curve CK-5146920 indicates that the total voltage drop across the cell will be about 4.5 volts for $7\frac{1}{2}$ " electrode spacing. Approximately $2\frac{1}{4}$ volts is effective for I^2R losses. The total heat power generated in the cell is then 1400 to 1800 watts including the 15 BTU/min. released by the cell solution and various losses in the gold and platinum electrodes. This

931 009

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EAH-135
R5OGL19
Page 8

checks approximately with the power released by using the theoretically calculated nitric acid resistance. (Weekly Report dated April 7, 1950.) The anode plating surface can thus be held at 50°C with less than 1 gal/min of 20°C water.

Assembly of Tank

There are three methods for fabrication of the steel tank and platinum liner.

1. Separate tanks of steel and platinum tinned and soldered in place.
2. Fabrication from 8" platinum clad steel strips.
3. Drawing the assembly from a large drawing circle of platinum clad steel.

Method No. 1 appears the most feasible. The precious metals manufacturers have indicated that a 20 or 30 mil thick platinum tank can be formed. The steel tank and platinum spinning would be tinned and sweated together by filling the assembly with hot liquid. The other two methods are alternatives and are not being actively considered at this time.

The drawing WW-9629649 shows that a flange is welded to the tank body. This flange is used to bolt the cover to the tank and also to bolt the water jacket in place. This same flange holds the copper bus bar for electrical connection.

Tank Cover

The tank cover is the structural supporting member for the entire cell. It was decided not to support from the tank

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EAH-135
R5OGL19
Page 9

because of the problems introduced by electrical insulation. The total weight of the cell including cover, tank, cooling jacket and water, cell solution, cathode structure, tubes, and magnet is about 750 lbs. The actual method of support from the cover to the installation frame must be mutually agreed upon. It is contemplated that three pads can be welded to the cover for bolting the cell in position.

Type 25-12 SCB steel can be obtained for the tank cover. This steel is slightly magnetic but can be used if final tests on type 347 steel prove it unsuitable. Type 347 steel samples have been immersed in hot acid and exposed to air. No effects were noted. A more complete test had been planned.

The maximum stress in the cover will be $20,000 \text{ #/in}^2$. This allows a factor of safety of 3.7 for the cover.

The other cell parts involved directly in the fabrication of the cover are the chemical, instrumentation, and vent piping except the three jet-out tubes.

Those tubes which must stand in the cell solution are gold. These gold tubes are flared over steel tubes and extend through the cover for strength. The gold-steel seal is made at the outer surface of the cover. The details of sealing and supporting these tubes at the cover are not complete. The outline drawings only indicate placement. It will be noted that all tubes are at cathode potential. These tubes, therefore, must be brought over near to the central cathode so that excessive current will not flow from these cathodic surfaces.

931 011

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EAH-135
R5OGL19
Page 10

Cathode Assembly

The cathode assembly consists of jet-out tubes, agitation magnet, and gold cathode "boot." The basic structure of the cathode is a stainless steel tube with one end closed. The 3 gold jet-out tubes will pass through the center of this steel cylinder. The cylinder will be covered with a gold boot. This boot will be sealed around the jet-out tubes. The drawing WW-9629649 indicates the method of connection and sealing of the cathode structure at the cover.

The three gold jet-out tubes will be made up with steel pipes at the top. The assembly of the magnet is done before the three tubes are flared apart at the top of the cathode.

A three section clamp plate is used to hold the steel section of the jet-out tubes in place. The drawing W-9076925 another clamping plate set above the cover. These two plates will hold the jet-out tubes rigidly in place. The magnet can also be removed by bending the jet-out tubes together.

The jet-out tubes may be made of gold since they will be at cathode potential and will not go into solution.

Pyrex Retaining Wall and Base

The pyrex base at the bottom of the tank serves two functions:

1. Impedes the plating process at the bottom of the tank.
2. Affords a supporting member for the pyrex "retaining wall" rings.

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EAH-135
R5OGL19
Page 11

Obviously some cell solution will leak in between the pyrex base and tank bottom and some plating may occur. Any trapped fluid, however, will be in negligible quantities.

Power Supply

It has been planned that copper-oxide rectifiers would be used for the power supply. This is a change from the motor generator type of power supply contemplated earlier. The motor generator sets do not operate efficiently over the desired current and voltage range.

The rectifiers have a 7% ripple but this will not impair the plating operation.

Type "500" copper oxide rectifiers, having 440 v., 3 phase, 60 cycle, input and 1 to 6 or 2 to 12 volt direct current output, are standard items. The output voltage is controlled by a 60 step (.166 volts/step) transformer. This gives adequate control of the process in order to obtain the desired cell operating conditions.

931 013

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EAH-135
R5OGL19
Page 12

APPENDIX I

MAGNETIC AGITATION OF A CURRENT CARRYING SOLUTION

Purpose

To demonstrate the use of a permanent magnet to cause agitation in a circular plating tank with concentric electrodes. (See Diagrams 1 and 2.)

Results

When current flowed through the solution, forces were set up that caused it to circulate as shown in diagram 1. There was also a secondary flow pattern which caused the solution to circulate as shown in diagram 2. This was due to the greater angular velocity at "A" than at "B" because of the greater flux density at "A". The magnitude of the secondary flow could be increased by properly placed vanes.

The average speed with which the solution circulated around the center electrode as a function of current is tabulated below:

<u>Average RPM</u>	<u>Total Current</u>	<u>Current Density at Anode</u>
12	21.4	.0395
18	51	.094
25	75	.138
30	110	.203
38	127	.224
48	148	.273

The average RPM was obtained by counting the revolutions made by a small piece of wood floating on the solution about 1" from the cathode.

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EAH-135
R5OGL19
Page 13

A "water logged" paper match was seen to sink below the surface, move out radially, then return to the surface. This random motion was superimposed on the circular motion.

Test Setup

A container, made from $\frac{1}{4}$ " lucite, 18" in diameter and 18" deep was used to hold the solution. Two concentric copper strips, each $\frac{1}{32}$ " thick and 10" deep, with a $\frac{1}{4}$ " round copper bar soldered around the upper edge, were used as electrodes.

The outer electrode had an inside diameter of $17\frac{1}{4}$ " and the center was $4\frac{1}{4}$ ".

Since the full depth of the container was not used in this test, a false bottom (lucite) was inserted 8" above the bottom. This supported the electrodes which were now flush with the top of the container. There were 4 finger holes at the outer edge of the false bottom so that the liquid could pass freely from one side to the other.

A tubular magnet $4\frac{1}{4}$ " outside diameter x $2\frac{1}{4}$ " inside diameter x 10" long was inserted into the center electrode. This magnet, which was made from Alnico 2, furnished a field between the electrodes of about 260,000 lines.

Procedure

The cell was filled with warm water. Two thousand cubic centimeters of nitric acid was added. Six and one half volts was applied to the electrodes. This resulted in a current of 148 amperes when the solution temperature was 37° C. Lower current values were obtained by reducing the voltage.

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EAH-135
R50GL19
Page 14

Test Cell
Diagram 1

Test Cell
Diagram 2

Notes: Only the top 10" of the Test Cell was used because the available magnet was 10" long.

331 016

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EAH-135
R50GL19
Page 15

APPENDIX II
LOCATION OF DATA

1. The original tests on the plating rates and purity obtained were filed by John A. Palm, General Engineering and Consulting Laboratory, General Electric Company, Building #37, Schenectady Works. This data is recorded on data sheets GL 63887.
2. The calculations used in applying the laboratory test data to the design of the 15 gallon cell with heat transfer and power supply calculations are filed by Gene R. Peterson, General Engineering and Consulting Laboratory, General Electric Company, Campbell Avenue Plant, Schenectady Works.
3. Copies of correspondence with Hanford, telegrams, and correspondence with manufacturers are filed by Gene R. Peterson, General Engineering and Consulting Laboratory, General Electric Company, Campbell Avenue Plant, Schenectady Works.

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