UNCLASSIFIED

THIS DOCUMENT CONSISTS OF 20 PAGES
THIS IS COPY 5 OF 13 A

DATE [redacted] INITIALS R.A.S.

MLM-213

MLM-MU-48-70-0022

Contract Number AT-33-1-GEN-53

MOUND LABORATORY

Operated By

MONSANTO CHEMICAL COMPANY

MIAMISBURG, OHIO

UNCLASSIFIED
Classification changed to by authority of LASL Report 1/25/73 10/2/73
by Carlotta L. Lewis 8/3/79
Reviewed by C.W. Huntington

M. M. Haring
Laboratory Director

SPECIAL REREVIEW
FINAL DETERMINATION
Classification: Unclassified
Category:
Signature: P.O. Dowd
Date: 2/12/80

ELECTRODEPOSITION PROGRESS REPORT

[redacted]

Caution

This [redacted] affecting the National [redacted] its transmission or the disclosure of its contents in any [redacted] person is prohibited [redacted] penalties under applicable Federal Laws.

[redacted] Restricted Data

This document contains restricted data as defined in the Atomic Energy Act of 1946.

[redacted]

October 1-31, 1948

MASTER

Prepared by: Edward Orban

Distributed: NOV 29 1948

[redacted]

This [redacted] in [redacted]

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

UNCLASSIFIED

DISTRIBUTION

1. - M. M. Haring
2. - J. J. Burbage
3. - Los Alamos
4. - Los Alamos
5. - Area Manager
6. - Area Manager
7. - Area Manager
8. - R. A. Staniforth
9. - Edward Orban
10. - Central Files
11. - Central Files
12. - Central Files
13. - Central Files

SECRET

Problem	Job Classification	Man Months
I. Plating of Postum from Hydrofluoric Acid		
A. Neutron Emission		
B. Adherence of Plate		
C. Photographic Examination		
D. Inert Plating Unit		
E. Conversion of Production Solutions to Hydrofluoric Acid Solutions		
F. High Density Run	205-C	2
G. Backing Materials	205-C	1
II. Plating Postum from Nitric Acid		
A. Rinsing of Plated Gauze		
B. Photographic Examination		
C. Plating Procedure	205-C	1/2
III. Solubility of Postum in Various Media	205-C	1/2
IV. Miscellaneous		
A. Preparation and Testing of Standard Cells		
B. Pipette Construction and Calibration		
C. Design of Unit V		
D. Design of Unit III		
Administration	202-C	

SECRET

GROUP I

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

ABSTRACT

Plating of Postum from Hydrofluoric Acid

The equipment for a high density run of postum out of hydrofluoric acid and some of the problems involved are described.

Attempts to plate large quantities of postum on copper have not been very successful.

Plating of Postum from Nitric Acid

The use of buffers as a rinsing medium has proven unsuccessful in that sizeable quantities of material still go into solution; morecver, the resulting neutron counts are extremely high.

There is no detectable amount of bismuth plating out at 0.0 v. to normal calomel electrode. This was shown by attempts to plate the bismuth out of a bismuth nitrate solution.

Solubility of Postum in Various Media

A rough check of the solubility of postum in hydrofluoric acid shows that about 2.56 units per ml. dissolve at room temperature.

DETAILED REPORT

I. Plating of Postum from Hydrofluoric Acid Solution

F. High Density Run

The general points being considered in the construction of equipment for the production of High Level Postum Gauzes were mentioned in the last progress report.[1] A more detailed discussion of the progress to date will be made here.

The development work of plating postum out of hydrofluoric acid has proceeded to the point where it is desired to make some runs plating out quantities of postum from 10 - 250 units per gauze. The following

points have been considered in a report for limited distribution.[2]

I. The amount of material requested for each run should be ten per cent greater than the amount of the final yield desired. This allows for a plating efficiency of approximately ninety per cent. To supply this amount of material it is requested that Production ship regular production gauzes, each one packed separately in a platinum can sealed in a glass ampoule. Although a separate packing for each foil would be preferable, to reduce cost it might be acceptable to place more than one gauze in a can. Tantalum would not be acceptable as a metal to be used in canning the gauzes. Hydrofluoric acid is corrosive to tantalum and could not be used to rinse the can after removal of the gauzes. The glass ampoules as received from Production will be opened by the usual method of using a scratch and a hot rod. It is not necessary to take steps to prevent the oxidation of the postum since it will be dissolved in the solution and replated anyway.

II. The present method being planned for the preparation of the solutions is to place the gauzes as they are removed from the ampoules and cans into the bottom of the beaker. Stripping action will take place for approximately twenty-four hours. After the stripping is complete, a platinum gauze will be placed over the gauzes and the solution will be stirred with a magnetic stirrer. A periodic check of the liquid level of the solution will be made since evaporation loss may be appreciable. To reduce the evaporation loss the solution will be in a cooling bath shown in Figure 1.

The amount of polonium in solution will be about 2.4 units per ml. Even larger quantities may be present because as postum plates out more ought to go into solution.

III. The plating bath will consist of a platinum beaker which will be used as an anode as well. A cooling bath will surround this beaker since the heat released by the postum will be considerable. No investigation of the plating procedure has been made except at room temperature; therefore, an effort will be made to maintain the temperature at 20°C. - 25°C. During the plating run the bath will be stirred by means of a magnetic stirrer and an iron nail sealed in fluorothene as a rotator.

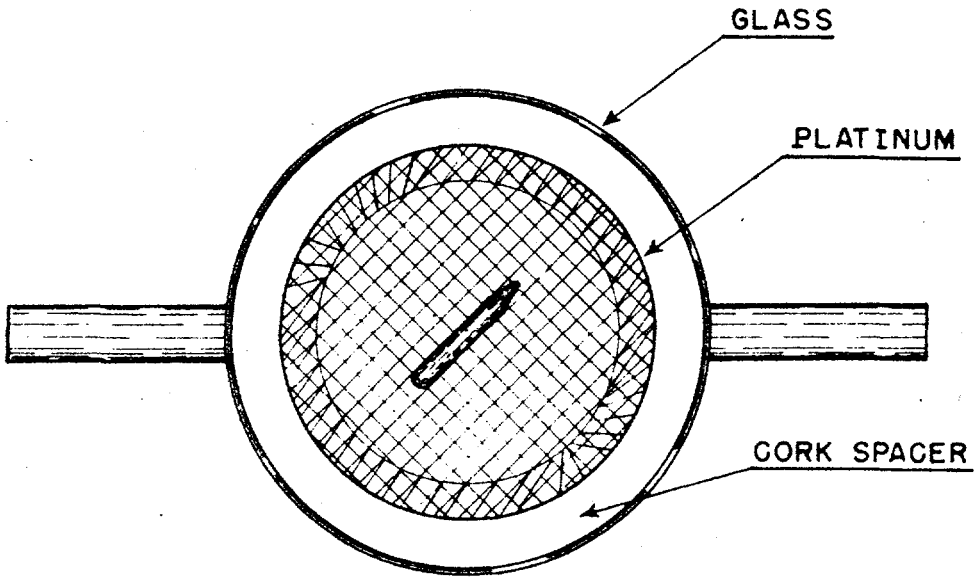
IV. It is expected that the heat liberated by large quantities of postum will heat the electrode, especially during the transfer from the plating bath to the canning process. For this reason a unique type of cathode holder has been invented. A hollow brass tube extending above the dry box

[redacted]

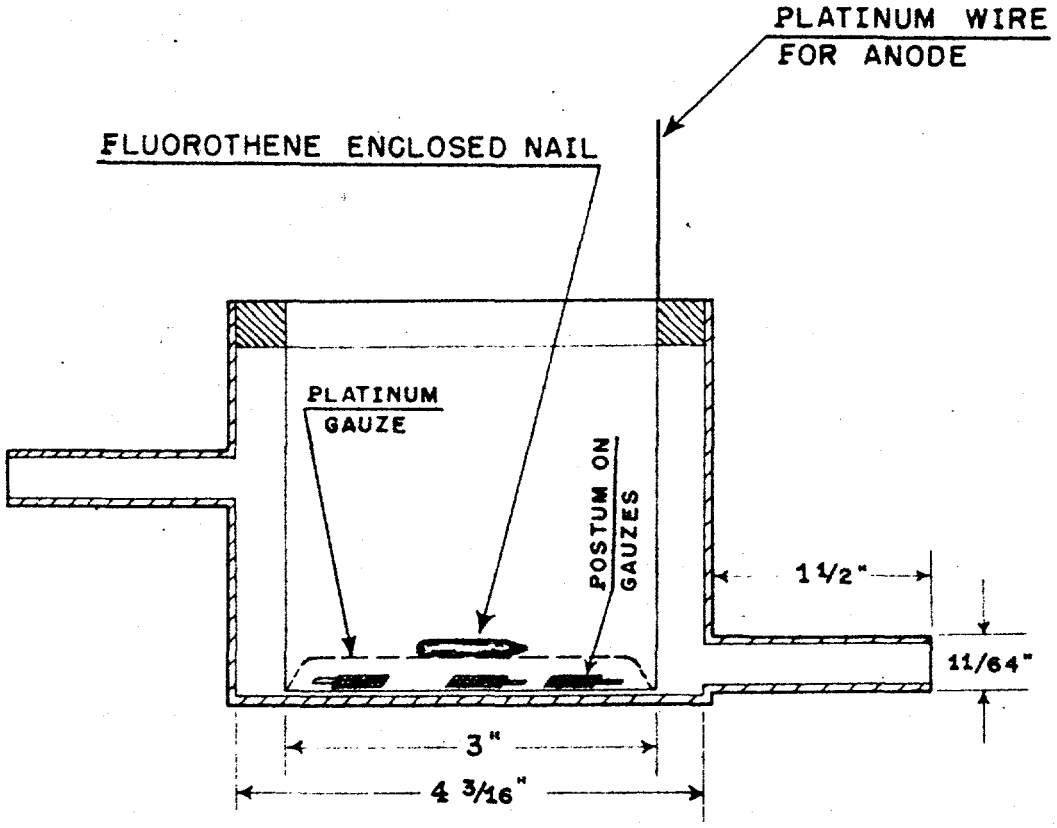
FIGURE 1

MLM-213

PLATING CELL



—TOP VIEW—



—CROSS SECTION—

[redacted]

will have the cathode holders welded on the end. (See Figure 2.) Either powdered dry ice or liquid air will be used as a coolant.

The use of postum plated out of nitric acid to prepare the hydrofluoric acid solutions might possibly make the use of a cathode potential controller unnecessary. However, unless the purity of postum to be used and the hydrofluoric acid and water used to make the solutions has been carefully checked, it might be wiser to use one. Moreover, all experimentation has been carried out with a potential controller and the potential kept at 0.0 v. to the normal calomel electrode so that deviation from this condition may give undesirable results.

V. The time required for the completion of the plating will be approximately eight hours. Some of the solution will have to be replenished since during this time some will be lost by evaporation. The solution will not be cooled to less than 25°C. because of the possibility that the plating may not proceed as has been determined at room temperature.

The solution will be sampled directly as the plating proceeds in order to determine how far it has gone. It may be necessary to dilute the sample to bring the concentration of activity down into counting range.

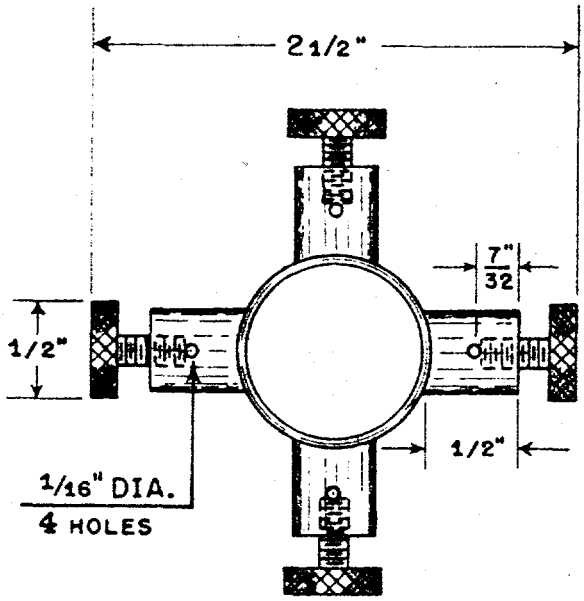
VI. The rinsing procedure must be carried out with a minimum of lost time. The cathode is arranged so that it may be raised and lowered. The plating bath will be removed and the rinsing bath (See Figure 3) put in its place. A small current is passed during the rinse to prevent the solution of any material. The procedure will be repeated for the acetone rinse but without current passing through the solution.

At the completion of the run the gauze will be removed from the cathode holder and placed in a tantalum gun. A platinum cap will be placed over the top with as tight a fit as possible. This gun will then be placed in a specially constructed aluminum container designed to dissipate the heat generated by the postum. (See Figure 4.) This aluminum can is constructed with fins to give it a larger cooling surface. It may be originally coated with copper so that the copper may be stripped after the can is sealed and in that way be decontaminated. A special vise has been constructed to hold the can during the opening and closing procedure. (See Figure 5.) A similar vise will be sent along with the aluminum can so that it may be opened easily at its destination.

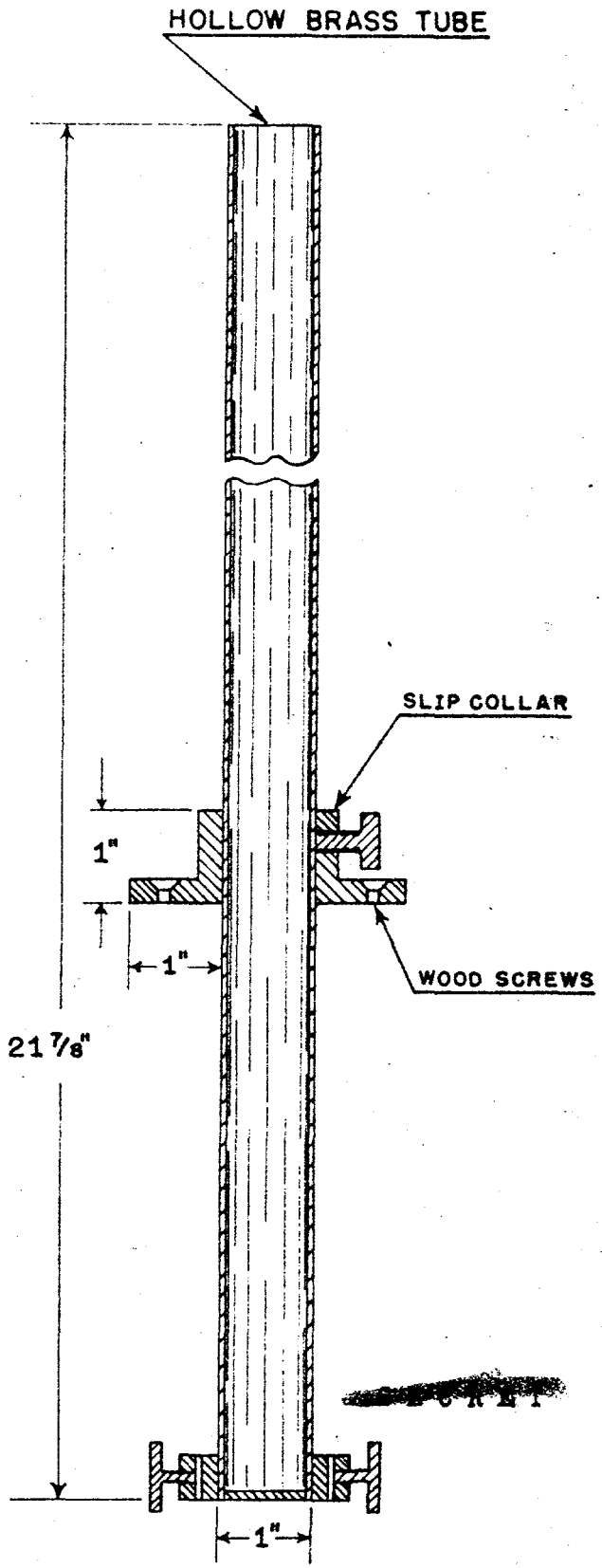
SECRET

~~DRAFT~~ UNCLASSIFIED FIGURE 2 MLM-213

CATHODE HOLDER



—TOP VIEW—
SCALE: FULL SIZE



—CROSS SECTION—
SCALE: 1/2" = 1"

~~DRAFT~~

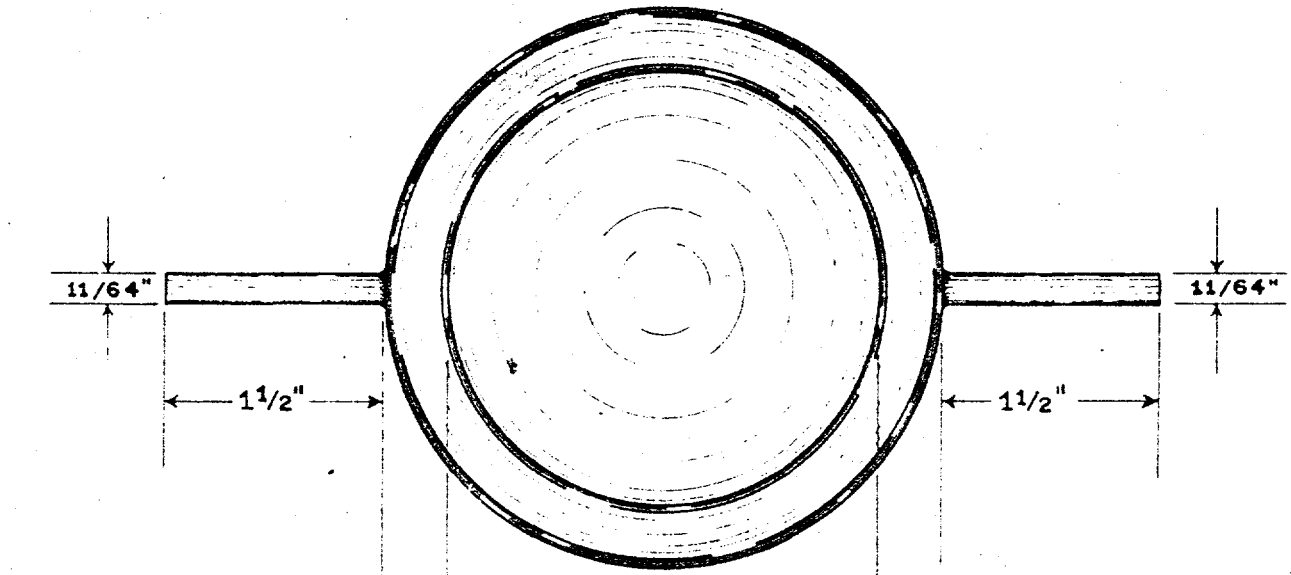
UNCLASSIFIED

~~SECRET~~

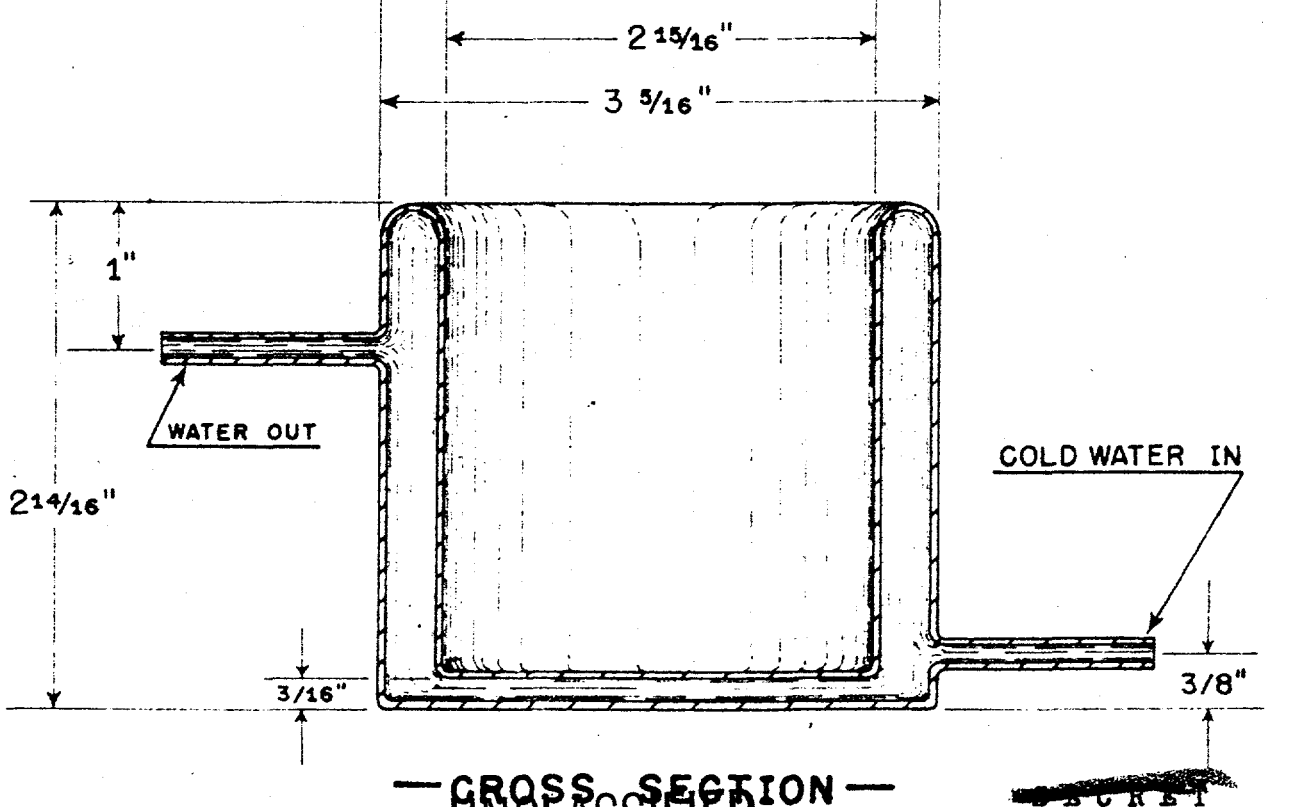
FIGURE 3

MLM-213

RINSING CELL
(ALL GLASS CONSTRUCTION)



TOP VIEW



CROSS SECTION

~~SECRET~~

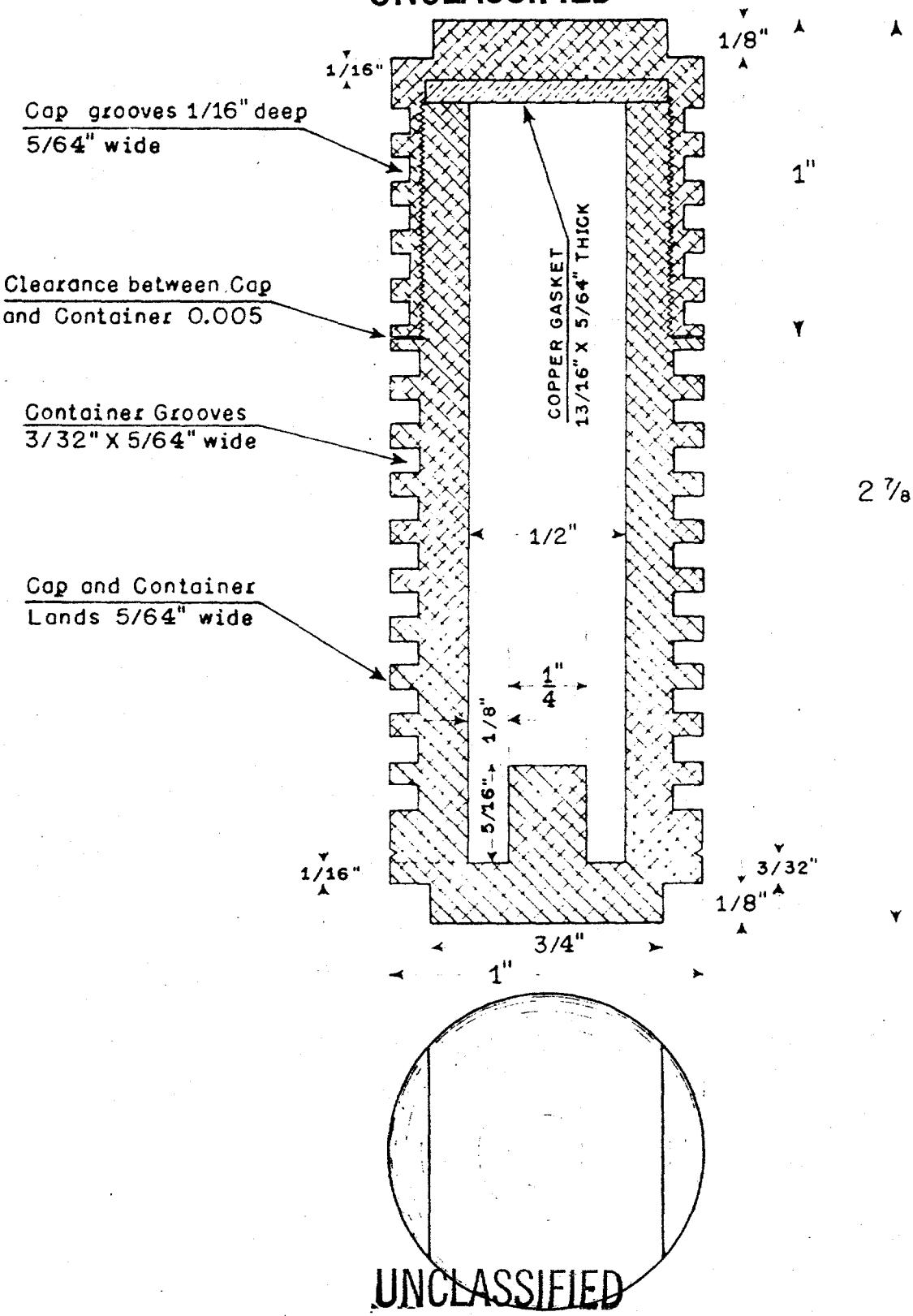
ALUMINUM ACTIVITY CONTAINER

~~SECRET~~

SCALE: DOUBLE SIZE

MIM-213

UNCLASSIFIED

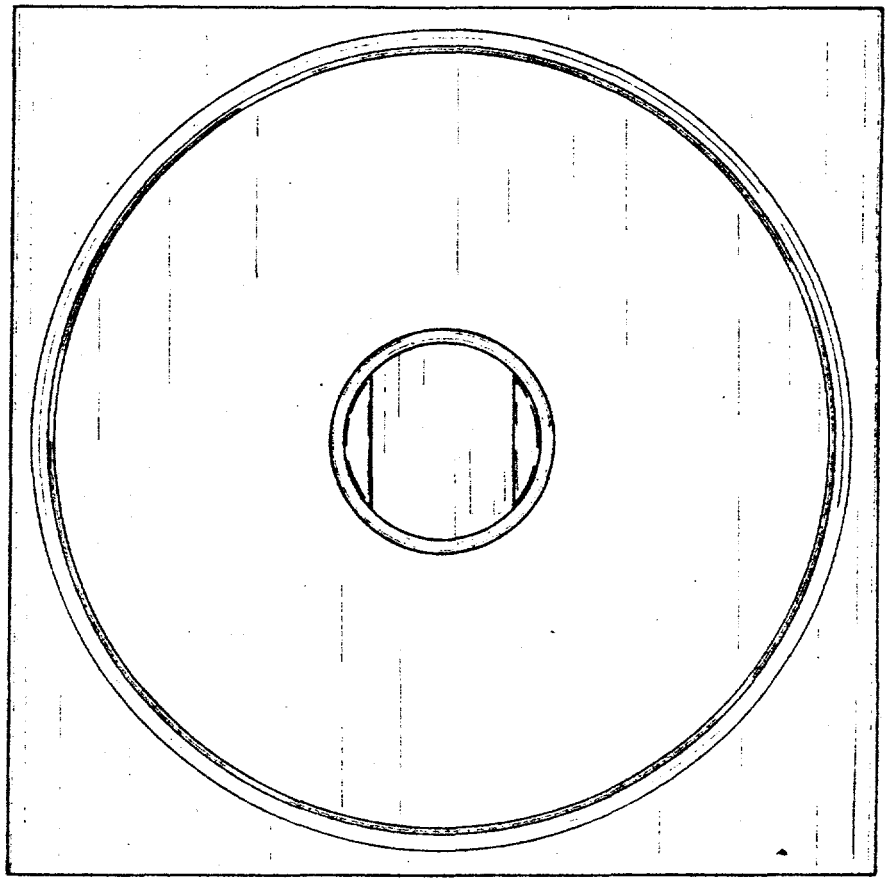


UNCLASSIFIED

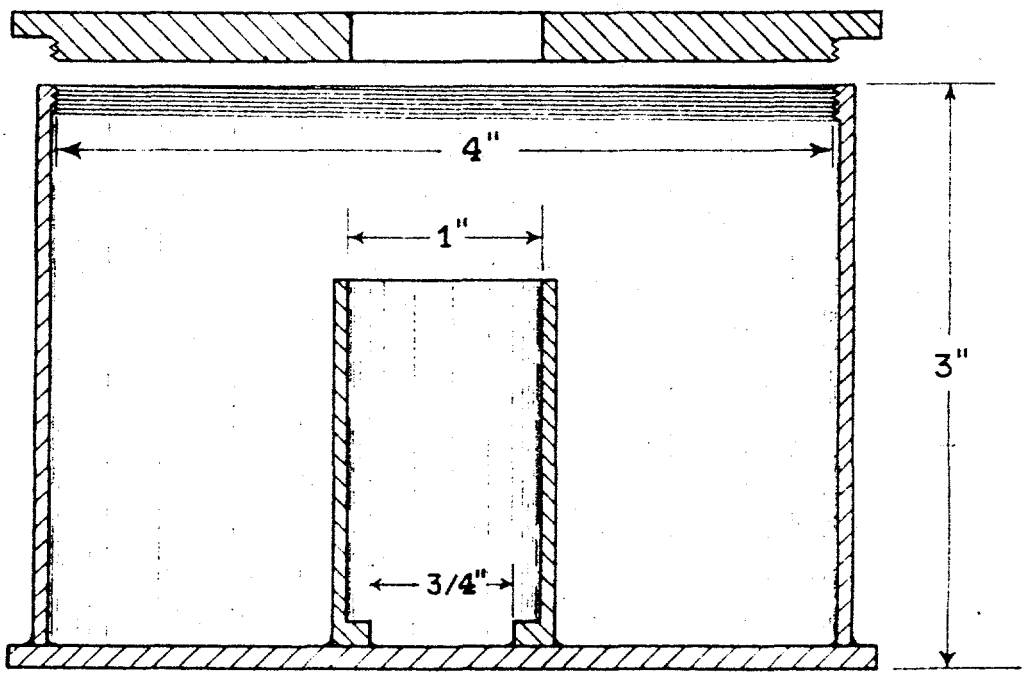
~~SECRET~~

FIGURE 4

VISE TO HOLD ALUMINUM CAN



— TOP VIEW LID OFF —



— CROSS SECTION —

SECRET

After the tantalum gun has been placed in the can, a special cap with a needle valve is placed on the can. This container is attached to the vacuum system where it can be evacuated. This dries the gauze. It is then filled with dry helium. The helium filled can will be returned to the helium filled dry box where a quick change of caps may be made with a stream of dry helium directed at the top of the can during the change. The closed aluminum can will be placed in a copper block with pieces of dry ice above and below it to cool the gauzes during the trip to its destination.

A calculation of the increase of pressure due to the helium released by alpha particles shows that it will be approximately one atmosphere after twenty days.

In the event that the cap of the aluminum can sticks and cannot be opened easily, a groove has been cut around the bottom of the can to mark the point where the can may be cut without cutting into the tantalum gun. (See Figure 4.) The bottom of the can being cut off allows the gun to slide out easily.

If the cap is removed the tantalum gun may be lifted out with a small loop of platinum wire that catches under the bottom and extends over the top. This sort of an arrangement will be necessary because the can may not be tipped to allow the gun to slide out.

VII. The neutron count of the completed gauze should be less than 3×10^4 n./sec. Indications are that this figure should be easy to meet. In the past it has been observed that the less surface area per curie, the lower the neutron count.

VIII. The amount of postum plated out can be determined by a calorimetric measurement. Care should be taken not to have the size of the can larger than will fit into a large calorimeter.

A serious problem with very large deposits is the high equilibrium temperature in the calorimeter. The vapor pressure of postum at 500°C. is about 1.9 mm. of mercury. Approximately a half curie of postum will volatilize if the can has a volume of 6.6 cc. The calorimetric run should be as short as possible to reduce to a minimum the amount of postum distilling out of the can.

SECRET

IX. Special dry boxes have been constructed for use with very large quantities of postum. (See Figure 6.) One will be used for the preparation of solutions. The second will be used for plating and canning. This one will be filled with oxygen-free dry helium. A vacuum system will be built into the dry box to dry the plated gauze and to introduce very pure helium into the final aluminum can. The third dry box will be used to clean the aluminum can and prepare it for shipment.

The interior of the dry boxes will be covered with removable paper in order to facilitate the cleaning of the dry box after the run is completed.

X. A constant check of the laboratories will be made in the usual way for alpha contamination. For gamma radiation a G. M. tube will be used. The tolerance level for gamma radiation is about 0.1 γ/day or 3300 photons/cm.^{2}/sec. On the basis that one gamma is emitted for every 10^5 alphas, approximately 1×10^7 gammas would be emitted every second by the total quantity of material. Using these values the tolerance limits shown in Table I would have to be observed.

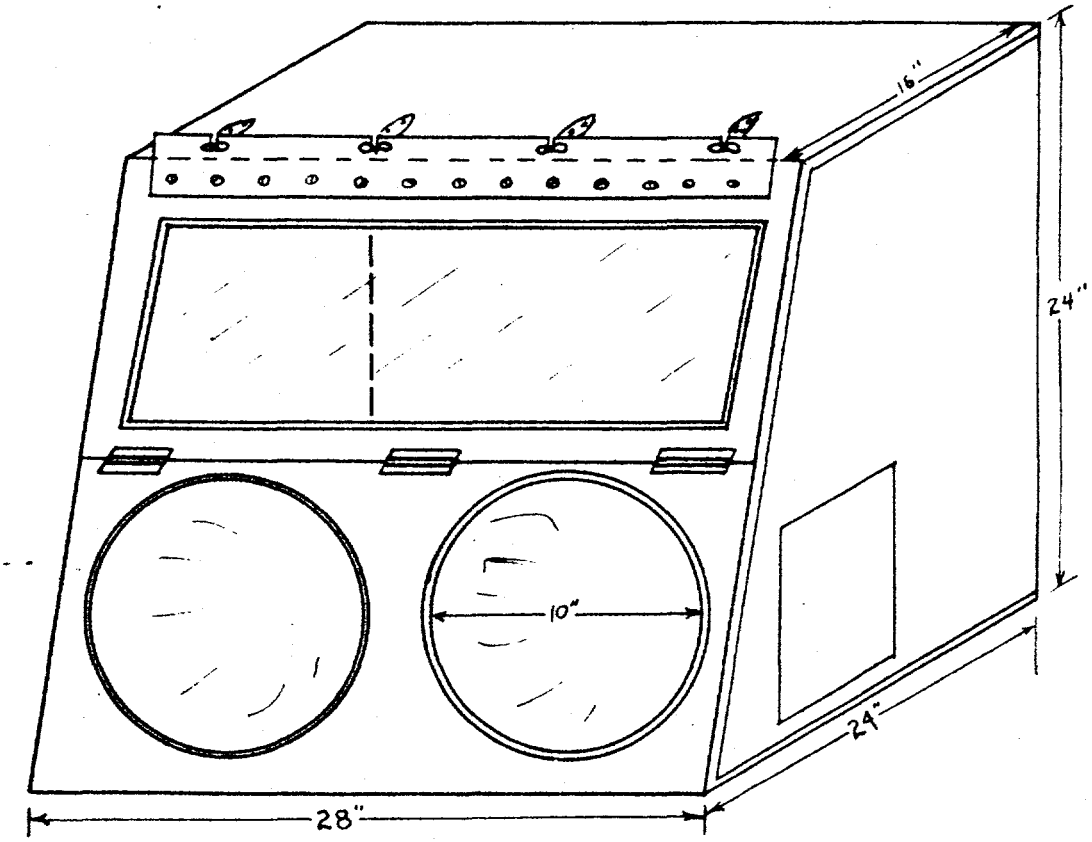
A B-wall tube will be used to monitor for neutrons. From a test run it has been estimated that approximately 1.7×10^7 neutrons per sec. will be released. The tolerance level for fast neutrons as reported by the Health Division is 150 M./cm.^{2}/sec. If the neutrons from the γ-F source are considered to be fast neutrons, the tolerance limits shown in Table II should be taken.

Two important points are made evident by these tables. First some protection should be provided for personnel who will be working near the source. Lead and paraffin shields will be installed. Second, after the solution has been prepared, all handling should be done by remote control. Construction of remote control equipment is being carried out now and will be reported in the next progress report.

[redacted]

HIGH DENSITY DRY BOX

MLM-213



[redacted]

FIGURE 6

Table I

GAMMA RADIATION TOLERANCE LIMITS WITH 350 UNITS OF POSTUM

Distance from Source	Photons/cm.^{2}/sec.	Tolerance Limit
2 feet	200	Below
1 foot	1000	24 hours
6 inches	4400	6.2 hours
1 inch	1.5×10^5	12 minutes
1 cm.	1.05×10^6	1.56 minutes

Table II

NEUTRON PARTICLE TOLERANCE LIMITS WITH 350 UNITS OF POSTUM IN 1.0 N HYDROFLUORIC ACID

Distance from Source	Neutrons/cm.^{2}/sec.	Tolerance Limit
4 feet	91.5	13 hours
3 feet	167	7.2 hours
2 feet	368	3.3 hours
1 foot	1470	49 minutes
6 inches	5.94×10^3	12 minutes
1 inch	2.14×10^5	20.2 seconds
1 cm.	1.38×10^6	3.3 seconds

I-G. BACKING MATERIALS

The desire to place ten units of material on copper stemmed from a request by Oak Ridge for that amount of material on a copper target in an area 1/4" x 1/8".

It was found over a year ago that copper could be plated out of hydrofluoric acid since the acid was a non-oxidizing medium.[3] The deposit was good and appeared to go on easily. However, only low density deposits were plated.

An attempt to plate ten units of postum on a flat surface 1/4" x 1/8" proved unsuccessful. The postum plated slowly until three units were plated and then ceased. The plating potential was held at -0.5 v. to the normal calomel electrode.

It will be necessary to make some preliminary runs to determine the optimum conditions for plating on copper before any attempt is made to plate on the X-ray absorption spectrum target.

II. Plating of Postum from Nitric Acid

A. Rinsing of Plated Gauze

Past experiments have shown that a good deal of postum is lost in the rinse solution.[4] This seems to be the direct cause of the acid which clings to the electrode as it is removed from the solution.

A method of rinsing was used for two series of experiments in which the gauze was placed in a buffer solution. The buffer was a sodium acid phosphate buffer which had a pH of 8.9, and held this value after the addition of an amount of 1.5 N nitric acid which would be carried by the gauze. Table III shows the results of these runs. The gauze was allowed to be in the air 24 seconds before being rinsed. It remained in the rinse solution two minutes before being removed. The results indicate that the rinse should be a pure solution without contaminants. There was no improvement in the amount of postum lost in the rinse and the neutron count on the gauzes were excessive. The recommended procedure here is not to use a buffer to neutralize the acid clinging to the gauze. In this experiment the fact that zero current produced the least loss of postum was again illustrated.

Table III

RINSING OF PLATED GAUZES IN SODIUM ACID PHOSPHATE BUFFER SOLUTION

Gauze No.	Current Density Rinse	Postum Dissolved in Rinse (mc.)	Postum in Residue (mc.)	Case Density mc./cm.^{2}	Neutrons per sec. per Unit
589	20	398	40	1391	6843.0
590	15	146	268	1505	5104
591	10	284	1366	90	316
592	5	335	1198	172	570
593	0	113	1153	592	2424
594	0	73	1424	340	1204
595	20	222	0	1826	7274
596	15	1582	0	120	4847
597	10	1344	1	76	256
598	5	1457	1	199	747
599	0	134	307.0	1637	4537
600	0	140	7.0	1884	78,890

C. Plating Procedure

An investigation of whether any amount of bismuth plates out at 0.0 v. to N.C.E. has been started. This is being carried out by weighting some platinum gauzes, plating them at 0.0 v. to N.C.E. for six hours and reweighing them.

The solutions used were 1.0 N, 1.25 N, 1.50 N, 1.75 N, and 2.0 N nitric acid. Each solution contained exactly 0.998 gm. Bi^{+++} as $Bi(NO_3)_3$ per 100 ml.

After plating, the cathodes were rinsed, without a flow of current, first in water and then alcohol. They were dried over a hot plate and allowed to cool before reweighing. Table IV shows the weight change of the platinum cathodes.

Apparently no bismuth plates out at this potential. The change in weight is probably due to errors in weighing. The plating potential will be changed and the experiment repeated.

III. Solubility of Postum in Various Media

The solubility of postum in hydrofluoric acid was checked roughly by placing a quantity of postum in a "Teflon" vial and adding 1.0 N hydrofluoric acid. A goodly excess of postum was added to 4.5 ml. of postum in each case. The results in Table V were obtained for three runs. The ambient temperature was room temperature with no effort made to control it. It was found that the solution temperature was considerably higher than ambient temperature because "Teflon" is a good insulator and did not allow the heat to dissipate very well. The average solubility may be considered to be 2.56 units per ml. at room temperature. The cross section for the α, n reaction with fluorine is large, hence, neutron counts were made of the solutions. These indicate that for large volumes of concentrated solution the neutron radiation becomes a serious health hazard.

Table IV

PLATING OF BISMUTH AT 0.0 v. TO N.C.E.

Run No.	Acid Concentration	Weight Change
1	0.98 N HNO_3	+ 12 gamma
2	" "	- 1 "
3	" "	- 7 "
4	" "	+ 8 "
5	1.22 "	- 58 "
6	" "	- 10 "
7	" "	- 7 "
8	" "	- 18 "
9	1.48 "	- 26 "
10	" "	- 29 "
11	" "	- 20 "
12	" "	- 14 "
13	1.75 "	+ 8 "
14	" "	+ 16 "
15	" "	+ 6 "
16	" "	+ 30 "
17	2.0 "	- 34 "
18	" "	- 16 "
19	" "	- 19 "
20	" "	- 25 "

Table V

Run	HF Normality	Solubility of Postum	Neutrons/Sec.	Volume
1	4.0	2.39	2.5×10^5	4.5 ml.
2	1.02	2.89	9.8×10^4	4.5 ml.
3	1.02	2.41	7.4×10^4	4.5 ml.

REFERENCES

1. Electrodeposition Research Group Progress Report for September 1-30, 1948, MLM-193.

2. Ad Interim Report by Edward Orban, October 15, 1948, MLM-209.

3. Ad Interim Report #10 by Ralph Wehrmann, November 30, 1947.

4. Production Electrolysis Studies - I by R. A. Staniforth, MLM-130.

5. Production Electrolysis Studies III by W. Raiff and E. Orban, MLM-171.

EO/ts.