PROCESS RESEARCH PROGRESS REPORT

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P. M. Hamilton, F. M. Huddleston, F. C. Mead, Jr., R. L. Norton, P. J. Schauer, and S. H. Styles were engaged in work on special problems.

ABSTRACT

Treatment of Hot Liquid Waste

Word was received that the Eimco filter has been shipped from the Salt Lake City plant. It is expected within two weeks or about May 10, 1948.

In place of a float gauge referred to in the Process Research Progress Report, March 1-31, 1948, a Uehling Tank-O-Meter will be used on the chemical solution tanks for the W. D. Building.

Treatment of Hot Solid Burnable Waste

Word was also received that the steam ejector has been shipped. It can be expected within the next few days. This item will complete the equipment to be used for the removal of activity from the flue gases produced in the incinerator.

Recovery of Bismuth

Laboratory experiments using synthetic bismuth chloride solutions are considered as completed for the time being. The experiments have shown that good yields, and adherent deposits can be obtained from solutions containing 0.18 g. bismuth/ml. with a 7 N hydrochloric acid concentration, providing a diaphragm is inserted between the anode and cathode. Neither calcium chloride nor pyrogallol are necessary under the conditions mentioned above.

Bismuth was plated from a production solution in Run EP-1 in which the activity has been reduced from $4.2 \times 10^7$ c./min./ml. to $4.2 \times 10^4$ c./min./ml. A yield of 2.48 g./amp.-hr. was obtained. The deposit contained several large nodules which were easily rubbed off.

A greater portion of the past month was taken up with the preparation of an exhibit for the Miamisburg Atomic Energy Show.
Treatment of Hot Liquid Waste - One Person

Word has been received that the Eimco filter has been shipped and can be expected within the next two weeks. A support for the filter has been designed and constructed.

Uhling Tank-O-Meter gauges are to be used on the Haveg tanks in the W. D. Building in place of float type gauge as discussed in the Process Research Progress Report, March 1-31, 1948. The advantages of the Tank-O-Meter gauge are that there are no moving parts and that the meter can be located at a distance from the tank.

Treatment of Hot Solid Burnable Waste - One Person

The unit for removing activity from the flue gases produced in the incinerator will be tested as soon as the steam injector is installed. We expect it within the next few days. Tests will also be made on the other equipment described in the Process Research Progress Report, March 1-31, 1948.

Recovery of Bismuth - One Person

In Run E-33 the spent solution of E-31 was renewed with bismuth trichloride to give the original bismuth concentration. It contained calcium chloride but no pyrogallol. The yield was 2.33 g./amp.-hr.

Run E-34 was made using a solution containing neither calcium chloride nor pyrogallol. The air stirrer stopped during the night, resulting in a low yield of 2.02 g./amp.-hr.

In Run E-35 the spent solution of E-34 was renewed with bismuth trichloride. During the regular 24 hour period the average rate of deposition was 2.59 g./amp.-hr. However, the deposit was not entirely adherent. Large nodules could be loosened. A heavier and stronger glass cloth diaphragm was substituted in the middle of the experiment. The voltage rose to 3.0 but finally leveled off at 2.0.

This series of experiments has led to conditions of plating which give:

1. A theoretical yield of 2.59 g. bismuth/amp-hr.
2. A deposit of satisfactory adherency.
The third requirement, high purity, has not been determined.

The conditions are as follows:

1. Ca. 7 N hydrochloric acid.
2. Ca. .18 g. Bi⁺⁺⁺/ml.
3. Glass cloth diaphragm separating the anode and cathode.
4. Moderate circulation of the electrolyte.

Neither calcium chloride nor pyrogallol are necessary under these conditions.

**Plating from a Production Solution**

Six liters of a depleted scrub solution were obtained from Unit XIV. By scrubbing twice with bismuth powder the α count was reduced from 4.2 x 10⁷ c./min./ml. to 4.2 x 10⁵ c./min./ml.

A Run EP-1 was made on the resulting solution in the laboratory scale apparatus previously used. The yield was 2.48 g./amp.-hr.

The composition of the solution was:

a. Bismuth concentration — .19 g./ml. as determined by plating a 2 ml. sample on a platinum gauze. The method used has been discussed in Process Research Progress Report, August 15-31, 1947.

b. Hydrochloric acid concentration unknown.

c. Activity: α 4.2 x 10⁴ c./min./ml.

Γ 0.0962 units/ml.

The deposit contained several large nodules which fell off.

The plate was radioactive. When held seven inches away from a Zeuto, the needle read full scale x 10. However, three weeks later the needle registered full scale x 1 when the plate was held four inches away. Ra Χ has a half-life of 5.0 days and is a beta emitter. Moreover, a measurement of the plate by the Health Department gave a tolerance of four hours at one foot. Thus, the radiation was primarily a beta radiation. The aluminum shield on the Geiger-Muller tube or a beaker around the plate cut down the reading almost to zero.
An $\alpha$ monitor count on the plate was $1.1 \times 10^7$ c./min.

A $\gamma$ count was made on the plating solution before and after plating. 5.0 $\gamma$ units were lost from the solution. This activity was probably due to silver rather than to postum. From the original $\alpha$ count per 2000 ml. it was calculated that the total number of $\gamma$ units from postum was not more than 0.05.

Recovery on a Pilot Scale

Figures 1, 2, and 3 show the plating tank arrangement (Process Research Progress Report, March 1-31, 1948) which is to be used for recovering the bismuth on a pilot plant scale.

An air lift to pump the depleted electrolyte from the overflow tank to the reservoir was constructed of 1 1/4" (O. D.) glass tubing. Seven small holes forming a ring admit air into the central tube. The three rings of air holes are 2 1/2" apart.

A jacket 1 3/4" (O. D.) surrounds the central portion of the tube. A side arm brings air into the jacket and through the holes to the liquid which it carries up into the reservoir.

The ceramic monkey pump procured from Unit IV has a pipe diameter too large for operations on the present scale.

Casting of a Large Bismuth Electrode

Two pieces of plate glass with a 1/8" lucite spacer formed the first bismuth mold. The molten metal would not flow into the mold but solidified in the neck because there was no exit for the displaced air.

A second attempt was made to pour a cathode. One air hole was cut in the lucite spacer and asbestos was wrapped around the glass. Resistance wire was wrapped around the asbestos and connected in series with a rheostat. The wire was heated gradually until all the resistance of the rheostat was cut out. This pouring proceeded satisfactorily but only half the mold was filled since the other half had no air hole. As the bismuth cooled it expanded and broke the plate glass.

The second mold was made with two wooden sides instead of glass. The same lucite spacer was used even though it became more deformed with each pour. This time the clamps were not tight enough and the bismuth flowed through to the bottom.
The next pour formed a complete cathode. However, it fitted so closely to the spacer, due to its peculiarity of expansion on cooling, that it was difficult to free the plate from the spacer. This plate broke.

Steel straps were put across the mold to prevent the wood from expanding, as it was noticed that the central part of the plate was thicker than that part near the edges. This plate came out whole but its surface was rough. It weighed 8 7/8 pounds.

A second cathode was poured satisfactorily.

FUTURE PLANS

The Kimo filter and accessory equipment will be installed for the purpose of gathering filtration data to be used in the operation of the W. D. Building.

"Cold" runs will be made with the incinerator and with the unit for removing activity from the flue gases, for the purpose of testing the operation of the various pieces of equipment.

A circulating system and reservoirs are being set up to test the operation of the bismuth plating unit.

Work will be started to determine a suitable means of removing the remaining amounts of postum from large amounts of the production solution by scrubbing with bismuth.

RM/ree