Process Research Progress Report

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ABSTRACT

Treatment of Hot Liquid Waste

Three runs, D-73, D-74, and D-75, were made in the model clarifloculator during this period. In D-73 and D-74 a mixture of hydrolysis supernatant and water was used as the original solution and in D-75 a hydrolysis supernatant, bismuth free activity and water mixture was used as the starting solution. D-73 gave acceptable results, but D-74 and D-75 gave results that were not too satisfactory. It is believed that contamination in the laboratory has effected the results.

Recovery of Bismuth

Experiments were resumed on the electrolytic extraction of bismuth from a hydrochloric acid solution of bismuth chloride.

Plating at elevated temperature with the elimination of calcium chloride resulted in a lower average deposition rate of 2.07 g./amp.-hr. than was obtained at room temperature in the presence of calcium chloride with a deposition rate of 2.30 g./amp.-hr.

Another comparison was made between cathodes prepared in a wood mold and a "Herculite" mold. Unlike the results obtained in run E-15 in which the cathode prepared in the "Herculite" mold gave a greater average deposition rate, the same deposition rate of 2.00 g./amp.-hr. was obtained with both cathodes. The formation of crystals was more prevalent in the case of the cathode cast in the wood mold.

In run E-24 a cathode with a rough surface was prepared by lining the side of the mold with carborundum cloth. The deposit consisted of nodules over the entire surface and very little treasing at the edges.

Treatment of Hot Solid Burnable Waste

The final enclosure of the incinerator with asbestos and transite is in progress. Preliminary consideration is being given to the steam expansion method of cleaning air as a means of removing the activity which would be carried over in the flu gases.

DETAILED REPORT

Treatment of Hot Liquid Waste

Four runs were planned for the model clarifloculator. Two
were made using hydrolysis supernatant. The synthetic original solution was based on the possibility that one day's run of hydrolysis supernatant (estimated at 300 gallons) would run into one of the original holding tanks (approximately 27,500 gallons). Thus, an original solution of 1900 ml. (approximately one half gallon) and 49 1/2 gallons of tap water was prepared. Runs D-73 and D-74 were made with this solution. The Dorr effluent in both runs was very clear. The results of D-73 were acceptable. However, D-74 gave fluctuating results, some of which were acceptable. It is believed that outside contamination caused the fluctuation in the results. Two additional runs were planned using hydrolysis supernatant, bismuth free activity and tap water for an original solution. Run D-75 was made using this solution. These results were not good. However, it is believed that contamination from the laboratory was the cause of this. The Dorr effluent in this case was also clear.

The counts on the Dorr effluent in all three runs checked lower than the average on many of the most successful runs in the past. This indicates that the Dorr is performing satisfactorily. Prior to these runs, water was run through the system and the filter. Counts on this gave a background of 1 1/2 counts per minute per milliliter. Thus, if this was subtracted from all results of the three runs, most of them would be very satisfactory.

See Table I for the results of the runs.

Recovery of Bismuth

Experiment E-22 was run without the addition of calcium chloride or pyrogallol to a solution of 7 N hydrochloric acid containing 0.18 g. bismuth/ml. as bismuth trichloride, using a bismuth cathode, and two carbon anodes, and a current density of 17.3 amp./ft². A hot plate under the solution kept the temperature between 70⁰C. - 75⁰C. The rate of deposition of bismuth was 2.07 g./amp.-hr., as compared with 2.30 g./amp.-hr., when no heat was applied and when calcium chloride was added. It was decided not to use heat in future experiments since the escaping hydrogen chloride is objectionable. The fumes corrode the stirring motor and electrical contacts as well. Moreover, a high rate of evaporation necessitated the addition of concentrated hydrochloric acid. Some of the chlorine was drawn away by the use of an aspirator attached to funnels over the anodes, but this precaution did not prevent corrosion. The deposit in this instance was poor, flaky, and not adherent.

In E-23 the solution contained calcium chloride as originally suggested. It was noted previously that the bismuth which plated out consisted either of shiny crystals or dull, gray nodules. Treading occurred to a varying extent on the vertical edges of all electrodes. A comparison between a bismuth cathode cast in a wood mold with one cast in a "Herculite" mold was made in E-15 A and B using 1.37 N hydrochloric acid. A repetition of this experiment (E-23 A and B) using 7 N hydrochloric acid gave the same rate of deposition, 2.00 g./amp.-hr. on both castings, but the appearance was considerably different. The small crystals originally plated on the herculite
### Table I

<table>
<thead>
<tr>
<th>RUN NO.</th>
<th>D-73</th>
<th>D-74</th>
<th>D-75</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al₂(SO₄)₃ · 18 H₂O per 50 Gal. (gm.)</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Na₂CO₃</td>
<td>20</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>Flow Rate Thru Dorr (ml./min.)</td>
<td>300</td>
<td>300</td>
<td>300</td>
</tr>
<tr>
<td>O. S. Counts/min./ml.</td>
<td>5810</td>
<td>5810</td>
<td>7350</td>
</tr>
<tr>
<td>Soln. Leaving Dorr (Aggregate) c./min./ml.</td>
<td>50</td>
<td>105</td>
<td>119</td>
</tr>
<tr>
<td>pH Soln. Leaving Dorr</td>
<td>8.2</td>
<td>8.2</td>
<td>8.4</td>
</tr>
<tr>
<td>Soln. Leaving Filter Hourly. Plating c./min./ml.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1st hour</td>
<td>4.94</td>
<td>10.31</td>
<td>11.44</td>
</tr>
<tr>
<td>2nd hour</td>
<td>8.77</td>
<td>6.82</td>
<td>12.89</td>
</tr>
<tr>
<td>3rd hour</td>
<td>4.61</td>
<td>7.91</td>
<td>7.94</td>
</tr>
<tr>
<td>4th hour</td>
<td>5.04</td>
<td>5.68</td>
<td>14.09</td>
</tr>
<tr>
<td>5th hour</td>
<td>1.09</td>
<td>6.45</td>
<td>8.22</td>
</tr>
<tr>
<td>6th hour</td>
<td>5.52</td>
<td>10.32</td>
<td>8.98</td>
</tr>
<tr>
<td>7th hour</td>
<td>5.48</td>
<td>8.16</td>
<td></td>
</tr>
<tr>
<td>Product Plated</td>
<td>4.76</td>
<td>9.92</td>
<td>7.69</td>
</tr>
</tbody>
</table>
electrode were covered up later by nodules but the wood mold electrode showed excellent crystals in the middle, nodules on the edges. The difference could be attributed either to the roughness of the surface or the original crystal formation in the cast electrode.

In run E-24 a very rough emery paper was placed in the mold so that the bismuth electrode consisted entirely of rough points. The deposit consisted entirely of nodules without the excessive treeing which had formerly appeared on the edges of all electrodes. A more tightly fitting cover was used in this experiment as well as thicker carbon cathodes.

Preliminary sketches for an electroplating apparatus on a pilot plant scale were drawn. The axis of the stirrer will be horizontal instead of vertical. The two bismuth cathodes will each be one square foot in area and held in place so that they will not have to support their own weight, as do the small scale cathodes. An arrangement for maintaining a constant bismuth trichloride concentration is being considered.

Treatment of Hot Solid Burnable Waste

The construction of the model incinerator is nearing completion. The cover of transite is being installed. The space between the fire resistant brick and transite is being packed with asbestos.

Preliminary consideration is being given to the possibility of using the steam expansion method of cleaning air as a means of removing the activity carried over in the flu gases.

FUTURE PLANS

Treatment of Hot Liquid Waste

1. Make an additional run using the same solution as used in run D-75.

2. Run water through the system in an effort to determine whether outside contamination is effecting the results.

3. Backwash the filter and then take samples of tap water run through it.

Recovery of Bismuth

Further experiments will be carried out to determine the effect of treating the surface of the cathode on the formation of the deposit. An attempt will be made to alter the cooling rate of
the bismuth cathode as it is cast in the mold.

Whenever possible, time will be spent on the design of a pilot-plant model for the electrolytic recovery of the bismuth.

Design and construction of the apparatus for the treatment of the hot solid burnable waste will continue.

Paul Hamilton

PH/roe

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