PROCESS RESEARCH PROGRESS REPORT

Note: Effective March 1, 1948 this report will be issued the first
of every month instead of bi-monthly.

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ABSTRACT

Treatment of Hot Liquid Waste

According to the Eimco Corporation, the filter on order is to be shipped the first week of April. Sketches have been prepared giving the general location of the Eimco filter and equipment to be used in testing the unit.

No suitable gauge is available for the tanks to be used in the W. D. Building for storing corrosive solutions. A gauge, to be fabricated from polystyrene, has been designed.

Treatment of Hot Solid Burnable Waste

Construction of the model incinerator has been completed. A general discussion of the problem and design of the incinerator is included in the Detailed Report.

Some time has been spent gathering together material for the final report covering the experimental work on the disposal of "hot" liquid wastes.

Recovery of Bismuth

Further work on the electrolytic extraction of bismuth has indicated that the formation of the bismuth deposited on the cathode is neither influenced by etching the electrode with nitric acid nor by varying the cooling rate of the cast electrode. A near theoretical yield was obtained (2.58 g./amp./hr.) by enclosing the cathode in a screen of glass cloth. The diaphragm, in all probability, reduces the amount of chlorine near the surface of the cathode with a resulting diminution of the reaction between chlorine and the bismuth metal.

DETAILED REPORT

Treatment of Hot Liquid Wastes

Since, for the time being, no further tests are planned for the model clarifloculator, attention is being given to the future work on the rotary drum filter and special equipment to be used in the W. D. Building. At present this work is in the design stage.

The housing for the Eimco filter is to be located on the south side of the boiler house. The necessary plans for the location of the
filter and equipment to be used in testing the unit have been made. Tests will also be made on the Oliver diaphragm slurry pump which is to be used to transfer the slurry from the sump to the drum filter.

Two pieces of special equipment are being designed for use in the W. D. Building. Gauges will be required on the four 50 gallon Haveg tanks to be used for storing corrosive solutions. A float gauge made of polystyrene has been constructed. No suitable means has been provided for removing the special cartridge filter from the vacuum tank. While the problem is largely one of operation, provisions are being made to construct and test a set of tongs for transferring the cartridge from the tank to a disposal drum.

Disposal of Solid Hot Burnable Wastes

The disposal of contaminated solids is being accomplished by means of burial. Due to limitations of space and the large increase of such waste to be expected at Mound Laboratory it was thought that some means of disposal, other than burial, should be investigated. Since the greater proportion of solid wastes are burnable, the problem was first attacked from this angle. Later, the problem of the un-burnable wastes, will be investigated.

The basic problem involved in the burning of waste contaminated with Q is the removal of activity from the flue gases. The volume of residual ash left after burning will be so small in comparison to the original volume of material that burial seems to be the best way of disposing of it. However, if we are successful in removing the activity from the gases, consideration could then be given to the disposal of the ash by leaching, digesting, etc., so as to eliminate the need for burial altogether.

Various methods for removing activity from gases have been investigated by Roy W. Endebrock at Unit IV. His methods consisted of; (1) scrubbing the gases in a packed column using dilute hydrochloric acid solution, dilute caustic solution and water, singly and in combination; and (2) steam expansion. In this method live steam and contaminated gases are mixed thoroughly in a nozzle and the mixture exhausted into a condensation chamber. The steam condenses on solid particles present in the gases. The cleaned gases are then exhausted to the atmosphere.

Mr. Endebrock found that the best combinations in the first method mentioned above gave a maximum efficiency of about 75 per cent. Although the results obtained in the second method mentioned above were inconclusive due to mechanical difficulties and lack of time in which to complete the tests, the results of his experiments indicated
a very high efficiency, possibly 100 per cent. Since the steam expansion method produced better results, it was selected as the method to be tried on a pilot plant scale. Because some of the equipment to be used was too heavy for the floors of the laboratory, a wood-tarpaper shanty was constructed on the southwest corner of the powerhouse (concrete floor) to house the equipment.

Since solid wastes are to be burned, we must have a furnace or incinerator of suitable design to accomplish this. The basic requirements then are: (1) incinerator should be air tight while in operation to prevent contamination of surrounding atmosphere, (2) should be operated under a slight vacuum as a protective measure in case of leaks, (3) should be capable of operating at temperature up to 1500°F. (pyrometer measurement) for short periods, (4) should be so designed as to obtain maximum combustion efficiency, (5) should be gas or oil fired to ignite combustibles and/or maintain high temperature, (6) should be well insulated to prevent fires and injury, and keep room temperature down, (7) should have access doors for charging and for removal of ash.

The commercial incinerators that are available do not meet all of these requirements. A specially designed incinerator to meet these requirements was found to be very expensive and would require considerable time before delivery. Therefore, the decision was made to construct our own. The details of construction are discussed below.

Figures 1, 2, and 3 show construction features of the furnace proper. Figure 4 is a front view of the finished incinerator showing charging door arrangement. Figure 5 is the rear view of the finished incinerator showing the pyrometer and draft gauge arrangement.

The base of the incinerator consists of two 4" x 4" wooden skids made rigid by two 2" x 3" cross members. A sheet of 1/2" transite was bolted to this wooden frame. A hollow square of firebrick (laid flat side down) in the outline of the furnace was mortared to the transite. The center of this hollow square was filled with asbestos board. A sheet of 3/8" boiler plate was then mortared to the top of these bricks. This plate extends 15" past the end of the bricks in front to set as a base for an oil burner if one should be desirable later on. A wooden form was then placed around this plate and a wet mix of Flibricko L-W-I castable refractory poured on the plate. This forms the refractory base of the furnace proper.

Wooden forms were used to cast the grates, which are made of the same castable refractory as the base. The side walls and ends of the incinerator were built up of firebricks. The grates were suspended with 1/4" steel straps imbedded in the side walls. An opening in the firebricks was left on one side at the base for an ash pit door and in the back wall for the flue pipe. Two gas jets (1/8" black iron pipe nipples) were installed in the front wall extending 3" into the ash pit.
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Figure 1

FRONT VIEW BEFORE ADDITION OF TOP SECTION SHOWING GRATINGS, RAFFLE AND AIR JETS
Figure 2

REAR VIEW BEFORE ADDITION OF TOP SECTION SHOWING FLUE EXHAUST, GRATES, AND CHARING DOOR
Figure 3

FRONT VIEW AFTER ADDITION OF TOP SECTION SHOWING VALVE ARRANGEMENT
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Figure 4

FRONT VIEW OF FINISHED INCINERATOR

SHOWING CHARGING DOOR ARRANGEMENT
Rear view of finished incinerator showing pyrometer and draft gauge arrangement.
The baffle wall consists of two vertical layers of firebrick laid on end so that the rear layer extends upward 2" above the front layer. An air pipe with five openings facing upward 45° from the horizontal is laid in this offset and covered with black asbestos furnace cement leaving only the air openings exposed. These jets are made of plugged tees with a 1/8" hole drilled through the plugs. A removable plug of 2 firebrick wedges was installed in the side wall (mortared around edges only) so that access to the wall behind the baffle wall could be effected in case it becomes necessary to clean this section. The flue pipe was placed in the hole left for it and secured in position with the castable refractory. The front faces of the two side walls are formed of castable refractory also. The top slab and slanting face slab were made of the castable refractory in wooden forms. A black iron 1 1/2" pipe nipple was placed in the top slab directly over the baffle plate for thermocouple installation. The front slab has an opening for the charging door and also four 1/8" black iron nipples (two on either side of the door) for air supply. These slabs were mortared to the firebrick.

The two gas jets were valved and connected into one common line into which gas and air were fed. A check valve was placed in the gas line to prevent air from backing up into the gas line when the air valve was also open. It was felt that enough air could be furnished in this manner to support combustion of the gas when the furnace was closed tightly. However, it was later found that this was not true. The fire was extinguished a few minutes after closing the doors of the furnace. To remedy this situation, two small hand torches were braised to the iron jets and fed with gas and oxygen. This worked very well. The air line was left connected to the jets in case an air supply should ever be needed below the grates.

The outer casing of the incinerator is made of 1/2" transite, fitting flush to the transite board in the base. The sides, ends, and top of this out casing are held together and to the base by 1 1/2" angle irons. The 4" space between the brick and the transite is filled with asbestos board and powdered asbestos insulation. The outer casing comes into contact with refractory material in two pieces - charging door and ash pit door. In both these places the refractory is at least 8" thick and is sealed to the transite with furnace cement. The transite is grooved around the door openings to take an asbestos gasket. The doors are made of 3/8" boiler plate with a rectangular section cut out of the center for a sight glass. The doors are hinged to the outer casing of transite and seat, when closed, on asbestos rope gaskets. A latch arrangement, secured to the transite, allows the doors to be pulled down tight, thus sealing the opening. The doors are covered with thin asbestos sheeting on the outside to prevent hand burns from the hot metal. A
two inch thickness of asbestos board is cemented to the inner faces to insulate them. The sight post is covered with a pyrex glass plate on both the inside and outside of the door (3/8" air space between). The inner glass, along with the asbestos insulation, is sealed to the door with sodium silicate and furnace cement. The ash pit door has a 1/2" iron pipe nipple with a cap, threaded into an opening in the door. The gas jets can then be lighted by inserting a gas pilot light tube through this opening without opening the door.

Air lines with regulating valves are connected to the four pipe openings in the front face and to the baffle wall pipe. These lines furnish air above the grates for secondary combustion. Air or oxygen may be furnished below the grates through the gas jet openings.

A thermocouple is inserted in the opening in top, extending 2 1/2" into the furnace. The temperature gauge is mounted on a small mantle which is secured to the charging door backstop. The thermocouple pipe has a small hole in the side into which is fitted a hose connection. An inclined draft gauge is connected to this to enable the operator to read pressure inside the furnace.

Remarks

The incinerator has not as yet been operated; i. e., actual burning of trash has yet to be tried. However, the furnace has been brought up to 400°F, and kept there for sometime by means of the gas-oxygen jets. This was done to thoroughly dry out the incinerator before actually burning at higher temperatures. The outer casing of transite is very well insulated. However, if the furnace is kept at 400°F, for more than four hours, a small area on top directly over the grates gets very hot. During pilot plant runs, we do not expect to operate the incinerator for more than an hour at a time, so that this may not happen. If it should happen, however, it can be remedied easily by removing that section of transite and replacing it with firebrick.

The equipment to be used for the steam expansion method will be described and discussed in the next Progress Report.
### INCINERATOR DIMENSIONS

<table>
<thead>
<tr>
<th>Inside Dimensions</th>
<th>Outside Dimensions</th>
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<tbody>
<tr>
<td>Height = 35&quot;</td>
<td>Height (from floor) = 60&quot;</td>
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<tr>
<td>Length = 35&quot;</td>
<td>Length (casing) = 54&quot;</td>
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<td>Width = 38 1/2&quot;</td>
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<td>Baffle Height = 21 1/2&quot;</td>
<td>Length (slant face) = 22 1/2&quot;</td>
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<td>Baffle Width = 6&quot;</td>
<td>Length (skids) = 68&quot;</td>
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<td>Baffle Well Width = 6&quot;</td>
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<tr>
<td>Grate Thickness = 1 1/2&quot;</td>
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<td>Grate Width (top) = 9&quot;</td>
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<td>Grate Width (middle) = 7&quot;</td>
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<tr>
<td>Grate Width (bottom) = 7&quot;</td>
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<tr>
<td>Flue Pipe Diameter = 5&quot;</td>
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<tr>
<td>Flue Pipe Height = 3 1/2&quot; (to bottom)</td>
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### GENERAL

- Wall Thickness (firebrick) = 4 1/2"
- Base Thickness (refractory) = 2"
- Base Thickness (steel) = 3/8"
- Base Thickness (firebrick) = 2 1/2"
- Top and Slant Face Thickness -
- Charging Door Opening = 12" x 14"
- Ash Pit Door Opening = 9" x 8"
- Asbestos Insulation Thickness = 4"
Recovery of Bismuth

Treatment of the Cathodes

The cathode prepared for Run E-25 was cooled rapidly while the ore prepared for Run E-26 was cooled slowly. There was no visual difference between the deposits on the two electrodes. The average deposition rate of Run E-25 was 1.74 g./amp.-hr. and in Run E-26 it was 1.92 g./amp.-hr.

The effect of etching the electrodes was determined by subjecting one side of the cathode used in Run E-25 to a nitric acid solution while the other surface remained untreated. No apparent difference in the nature of the deposit was observed.

The low rate of deposition obtained in Runs E-25 and E-26 was attributed to the fact that the plating tank was covered with a lucite plate. As a result, there was an increase in the chlorine gas content of the solution.

Diaphragm

The cause of the low results in the two runs discussed above was substantiated in a series of runs in which the cathode was enclosed in a diaphragm. The diaphragm was constructed of glass cloth. Near theoretical yields of 2.59 g./amp.-hr. were obtained.

In Run E-28, besides separating the cathode from the anodes by means of a diaphragm, the solution was agitated by bubbling nitrogen through the solution. A 1/2" lucite plate was made with nine holes (0.035" dia.) half-way through the plate. The holes were arranged in three rows, the rows 1" apart. Channels were bored in the plate and a glass rod inserted to lead in the nitrogen. This assembly was cemented to the bottom of the plating dish and the electrodes placed in position. The nitrogen rose in small bubbles through the electrolyte and very close to the surface of the electrodes.

A frame work of lucite rod was made and glass cloth stretched to form vertical walls. The top and bottom were left open. The frame work was cemented to the electrode supporting cover. The cathode was placed inside.

The calculated average rate of deposition turned out to be 2.69 g./amp.-hr. which is .10 g. more than the theoretical rate. However, the adjustment of the 1.5 amp. current is made with a wire-wound resistor. Only course adjustments can be made. If 1.55 amp. were used in the calculation the theoretical rate would be obtained.
E-29 was run to determine which of these innovations had the most effect on the rate of deposition. Bubbling nitrogen was used without the glass diaphragm. The experiment failed because the cathode slipped along its supporting rod until it was stopped by the anode.

E-30 was run using ordinary mechanical stirring and the glass cloth diaphragm. The yield was 2.87 g./amp.-hr. This experiment was considered to give satisfactory results both in appearance and adherence of deposit and in the rate of deposition. In this case the amount of circulation of the electrolyte inside the diaphragm was not as great as when the diaphragm had not been used.

But it was possible in this case to obtain a high rate of deposition without rapid agitation, which had previously been considered necessary.

Elimination of Additives

Run E-31 was similar to the previous run except that pyrogalol was eliminated. The rate, 2.63 g./amp.-hr., again turned out to be higher than the theoretical value.

In Run E-32 the spent solution from Run E-30 was used. Sufficient bismuth chloride was added to attain the original bismuth concentration. With mechanical agitation and the cathode enclosed in a diaphragm an average deposition rate of 2.56 g./amp.-hr. was obtained.

To compare the same situation in the absence of pyrogalol the solution used in Run E-21 was treated in a similar manner. The resulting average rate of deposition was 2.33 g./amp.-hr.

If it is found that it is desirable to maintain a constant bismuth concentration, then the above results would indicate that pyrogalol should be added.

Pilot Plant Model

A lucite vessel containing three anodes and two cathodes each 1 ft² has been designed and is being built. The accompanying sketch shows the method by which:

1. The electrolyte is circulated.

2. Bismuth oxychloride is dissolved.

Since a glass pump could not be obtained, a monkey pump was procured from Unit IV to lift the overflow liquid to the bismuth oxychloride dissolution tank.
PILOT PLANT MODEL FOR ELECTROLYTIC EXTRACTION OF BISMUTH
FUTURE PLANS

Experiments will be carried out to determine suitable operating conditions for the Rimco filter and Oliver diaphragm slurry pump.

The acid resistant gauge for the Haveg tanks and the tongs for removing the special filter cartridge will be tested.

Small scale runs will be continued to determine whether or not calcium chloride and pyrogallol are necessary in the bismuth plating solution. Some runs will also be made on depleted production solutions and the deposit analyzed for purity.

Experiments will be carried out using the small pilot model of a plating tank as soon as it is completed. A method for casting the larger size cathodes to be used in the model will be investigated.

As soon as the steam ejector is received, experiments will be carried out to determine the practicability of the steam expansion method for removing activity from the flue gases.