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Contract Number AT-33-1-CEN-53

MOUND LABORATORY

Operated By

MONSANTO CHEMICAL COMPANY

MIAMISBURG, OHIO

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M. M. Haring
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PROCESS RESEARCH PROCESS REPORT

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PROCESS RESEARCH GROUP

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ABSTRACT

Treatment of Hot Liquid Waste

As mentioned in the previous progress report, cloth #5044 became clogged after continued use. The original filtering characteristics of this cloth were partially restored by washing with caustic soda and then a dilute solution of nitric acid.

A Lucite hopper and spray nozzle were designed for the purpose of preventing the clogging of the hopper outlet by the aluminum hydroxide cake. Muriatic acid can be sprayed into the hopper to dissolve the aluminum hydroxide which sticks to the walls.

Filtration of Hydrolysis Solution

Preliminary investigation of the "palma twill" cloth recommended by the Eimco Corporation for filtering bismuth oxychloride gave only fair results. The moisture content of the cake was 50 per cent, while the rate of filtration of the slurry was 15-20 gal./hr.

Treatment of Hot Solid Burnable Waste

Construction of the spray tower has been completed. The unit for removing the activity from the flue gases is described in the main body of this report.

Three cold runs were made to check the functioning of the equipment under operating conditions. It was found that pure oxygen had to be fed into the furnace to get good combustion. With the furnace in operation a very small amount of white smoke is discharged from the stack.

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DETAILED REPORT

Treatment of Hot Liquid Waste - Three Persons

Clogging of Cloth - Filtration (Run F-22) of the aluminum hydroxide slurry required 5-2/3 hours for completion. No particular care had been exercised in cleaning the filter cloth between previous runs. Nine runs had been made with this cloth (#5044). It was apparent that this long filtration period was due primarily to a clogged filter cloth. Various washing procedures were tried unsuccessfully in the hope of restoring the original filtering characteristics of this cloth.

The first of these and the simplest was to wash off, with a hose, all the material sticking to the cloth and ropes. But the next run F-23 still took five hours, with a filtration rate of 420 ml./min.

It was thought that perhaps a low pH might have been responsible. Run F-24 took over five hours despite a high pH of 8.9. The filtration rate was 260 ml./min.

Backwashing through the filtrate wash line was tried before F-25a without producing any considerable increase in the filtrate rate. It was 320 ml./min. Before F-25b a strong solution of NaOH was put in the drum, and after rotation, the solution was drawn through the filtrate line by vacuum. A considerable amount of insoluble material was left on the cloth. The filtrate rate was 465 ml./min.

Strong sodium hydroxide was tried again by pumping it back through the blower line and through the cloth into the filter pan. Run 26-a showed an improvement with a filtrate rate of 600 ml./min. One normal nitric acid was next placed in the filter box and the drum rotated in the acid. All visible precipitate readily dissolved. After draining the drum box backwashing with steam through the blower line was tried, followed by water. The filtrate rate of F-26b was 450 ml./min.

Work was temporarily discontinued on this phase until more of cloth #5044 could be obtained. It is planned that several runs will be made washing the cloth after each run.

Hopper Design - The elongated hopper which was built to prevent clogging proved considerably better than the original hopper. However, the wet cake occasionally built up on the side walls and caused clogging. Although this happens very seldom, its occurrence would cause considerable trouble in the permanent installation using "hot" material. To prevent

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this happening a new hopper of a even longer length was designed with a cylindrical outlet 5-1/8" O.D. to fit into the Havg inlet pipe on the Pfaudler. Provision will be made to put muriatic acid through a hole in the side of the hopper. If the hopper becomes clogged, it will be possible to dislodge the clogged aluminum hydroxide by dissolving it. An acid spray nozzle will be located inside the hopper so that the hopper walls can be kept wet. It was noticed in a trial run on the first substitute hopper that the force of a water spray alone dislodged pieces of the cake which had stuck to the side walls. The nozzle will be located at a distance sufficiently below the filter so that the acid spray will not reach any metal parts. An exhaust draft of 500 cu. ft./min. aids in pulling the acid vapors into the Pfaudler. Not only the force of the spray itself but its dissolving action will loosen any aluminum hydroxide which may stick to the walls.

The use of muriatic acid in a spray nozzle and hopper necessitates that they both be made of an acid-proof material. Lucite was chosen because of its availability and the ease with which it can be worked. The top of the hopper is a rectangle 7" x 20"; the bottom a circle, 5-1/8" O.D.

Three different types of spray nozzles were tried before finding one which gave the desired kind of spray. The spray must be directed downward at about an angle of 60° with the vertical and be fine enough to wet all of the 4 walls. The first nozzle to be tried consisted of a brass disc which could be screwed into the stationary part of the nozzle. The umbrella of water produced was irregular in shape. Since the spray was flat, there was considerable splashing. Adjustment of the separation between the disc and the other section was coarse and unsatisfactory. A second spray nozzle was designed and built of Lucite. It consisted of a cone which could be screwed into the stationary part of the nozzle. The spray from this could not be adjusted satisfactorily. Either the volume of water coming from it was too large when the shape of the spray was correct or there was no water at all. A third nozzle was built with a chamber into one side of which the water entered. After swirling around the chamber the liquid sprayed out of a pin hole in the bottom. The cone of water thus produced made the desired angle; and the rate of spraying was small. This nozzle will be located in the hopper after construction.

Filtration of Hydrolysis Solution - Two Persons

A batch of "cold" bismuth trichloride was made up and precipitated with sodium hydroxide. The slurry was used to test the operation of the filter in anticipation of difficulties to be encountered in the H-H Building. The bismuth trichloride solution was

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prepared in five separate batches. A 30 liter pyrex glass battery jar was used for dissolving 4000 gm. of bismuth metal in fifteen liters of hydrochloric acid and five liters of nitric acid. Since the evolution of nitrogen dioxide and hydrogen chloride fumes were objectionable out-of-doors, the last three batches were made in the lab. hood and the fumes allowed to be drawn out the stack.

Denitration was carried out with formaldehyde. The solution was brought up to reaction temperature, about 85°C., by means of a glass steam coil placed inside the battery jar. This step was carried out in order to insure a final precipitate as much like that obtained at Unit IV as possible.

Precipitation of a mixture of the bismuth oxychloride, bismuth hydroxide, and bismuth trioxide was accomplished by adding a fifty per cent sodium hydroxide solution prepared from caustic flakes. The precipitate is a mixture, the yellow bismuth trioxide first, then white oxide and hydroxide and finally the precipitate darkens, due to the reduction of bismuth to the metal by hot formaldehyde in the presence of alkali.

The composition of the final precipitate varies with each batch at Unit IV, according to Dave Scott, apparently depending upon the conditions of precipitation. This variation is noticed because some precipitates are darker and settle faster than others. The precipitate is amorphous.

The thirty-five gallons of slurry obtained from the five batches was filtered in a preliminary run through the "palma twill" cloth which was recommended by Kimco Corporation. It was expected, according to their experiments, that the moisture content would be about twenty per cent. Instead it was found to be about fifty per cent. The filtration took between 15-20 gal. of slurry/hr. It is planned to filter about 300 gal./day, which would require 20-15 hrs. operation/day.

Treatment of Hot Solid Burnable Waste - Two Persons

The preliminary considerations of this problem and the method to be used have been described in a previous report, MLM-72. Also the construction details and description of the incinerator have been given. A sketch and explanation of the apparatus constructed for activity removal follows.

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The flue gases from the incinerator are drawn through a three-inch black iron pipe (insulated) and mixed with live steam in the steam exhaustor (Figures 1 and 2). The steam gas mixture is discharged vertically through a nozzle into a thirty gal. drum (expansion chamber) which is contained in a 55 gal. drum. This larger drum has tap water circulating through it, which completely immerses the expansion chamber, thus cooling it. The steam exhaustor is a standard Schutte and Koerting make with a 3/8" steam connection and a 1" suction connection, constructed of brass and iron. This exhaustor serves the twofold purpose of furnishing a draft for the incinerator and mixing the flue gases with live steam. The steam starts to condense either in the nozzle or in the expansion chamber, depending on the temperature of the flue gases, on whatever particles are present. This condensation is in the form of a very fine fog, however, so that only a small amount drops out in the expansion drum, the remainder being carried up into the spray tower. That which does fall out in the drum flows back into a small collecting bottle and is then pumped from this bottle to a 55 gal. holding tank. A water-cooled condenser is necessary on the collecting bottle because of a small amount of fog which blows over from the expansion chamber through the condensate drain lines.

The fog which is carried up into the spray tower is washed out by passing through four sprays and two sections of packing. The tower is made of 4" O.D. brass pipe in four sections (excluding stack above anemometer box) with a spray nozzle in each section. The nozzles are 17" apart. There is 10" of packing (1/2" Berl saddles) directly above the third and fourth nozzles. The packing is supported on perforated brass plates held in place by the clean-out plug of the nozzles. The spray water collects at the bottom of the tower and drains by gravity to the holding tanks.

Above the fourth section of the brass tower is a laminated plywood box with an airtight door, which holds an anemometer in place in the gas stream. Because of the slow rate of flow of the gases passing through the stack it was necessary to measure the flow by this means. Two windows in this box permit reading of the anemometer at any time. A thermometer and a manometer are also connected to this box for measuring temperature and pressure of gas stream at this point. An activity assay filter paper holder is also attached to this box. A regulated flow of gas is drawn through this paper by means of a vacuum pump and a flow manometer similar to the equipment used in the laboratories for air sampling. An outlet pipe extends about 30" above this box.

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Figure 1

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Figure 2 TOWER DETAILS

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Preliminary Tests - In order to check the functioning of all the equipment under operating conditions three cold runs were made. The furnace was charged with wood scraps, cardboard, cloth, rubber gloves, Tygon tubing, and coal in various combinations and all together. The first test showed that the furnace would have to be furnished with more oxygen than the present set up allowed. When sufficient air was used to get good combustion in the furnace, the pressure inside was higher than atmospheric, thus causing smoke leakage around doors and seams of furnace. This condition was remedied by feeding only oxygen through the front face and baffle wall openings.

It was found that if the gas-oxygen jets were lighted and no other oxygen furnished, the material would not ignite. Hence, the burners are used to bring the temperature of the furnace up to about 300°F. and then turned off. Oxygen is then admitted slowly until combustion takes place. The oxygen supply to the furnace determines the rate of combustion and hence temperature but can be admitted only so fast as the steam exhauster can handle the products of combustion. However, it was found that enough oxygen could be admitted to burn the waste to a very small amount of ash and still operate the incinerator under a slight vacuum.

When the furnace is in operation there is a small amount of very white smoke being discharged from the stack.

FUTURE PLANS

Filtration of Hydrolysis Solution

An investigation will be made of the factors which govern the type of precipitate obtained when a bismuth chloride solution is neutralized with sodium hydroxide. Such factors as pH, rate of addition of sodium hydroxide, temperature, presence of aluminum chloride, and concentration of electrolyte will be considered. A Pfaudler will be mounted near the filter to carry out pilot plant experiments.

Treatment of Hot Liquid Waste

The Lucite hopper and spray will be tested. Several more runs will be made with the aluminum hydroxide slurry to see if a thorough washing of the cloth after each run will prevent a clogging of the filter cloth.

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Treatment of Hot Solid Burnable Waste

Several "hot" runs using waste containing postum will be made to determine the operation of the incinerator and gas scrubbing unit.

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