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TESTS OF A VAPOR-SPACE FEED NOZZLE FOR CALCINING ALUMINUM NITRATE SOLUTIONS IN A FLUIDIZED BED

G. E. Lohse, Editor

March 15, 1962

PHILLIPS PETROLEUM COMPANY

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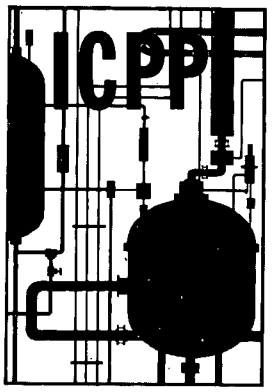

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Operation and Development Work Performed At
Battelle Memorial Institute By
G. R. Smithson, Jr.
J. E. Hanway, Jr.
F. M. Stephens, Jr.

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ABSTRACT

An investigation was conducted to determine the performance of a vapor-space feed nozzle which sprayed aluminum nitrate solution on the surface of a fluidized bed calciner. Results indicate that this type of feed system is satisfactory for calcining aqueous wastes from the processing of spent aluminum-type nuclear fuels. Process and product control were achieved by adjusting the volumetric ratio of the air to the liquid fed to the nozzle. The results obtained at various operating conditions are compared with those from a pneumatic atomizing nozzle submerged below the surface of the fluidized bed.

This report is largely based on a report submitted by Battelle Memorial Institute, summarizing work performed by BMI under a Task Agreement with Phillips Petroleum Company.

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TESTS OF A VAPOR-SPACE FEED NOZZLE FOR CALCINING ALUMINUM NITRATE SOLUTIONS IN A FLUIDIZED BED

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TESTS OF A VAPOR-SPACE FEED NOZZLE FOR CALCINING ALUMINUM NITRATE SOLUTIONS IN A FLUIDIZED BED

I. SUMMARY

An experimental program conducted at the Battelle Memorial Institute has indicated that the feed system used at Battelle for introducing slurries and solutions into a fluidized bed calciner can be adapted for reducing aqueous wastes from aluminum reactor fuels to solids. In this system the feed solution is sprayed on the surface of the bed through tubing installed axially through the top of the calciner.

Process operation and product characteristics were adequately maintained by adjusting the air rate to the feed gun. Volumetric air-to-feed ratios above 240 gave a product with a mean diameter less than 0.3 millimeter and an alpha alumina content above 30 weight per cent. Air-to-feed ratios of approximately 100 gave product with a mean size greater than 0.6 millimeter and an alpha alumina content below 20 per cent. The residual nitrate content of the solids remained below six weight per cent throughout the tests. The absolute density varied from 3.6 grams per cubic centimeter for 60 per cent alpha, to 3.0 for 10 per cent alpha. Mechanically, the feed gun operated without difficulty throughout a total operating time of 25 days, and no serious caking occurred in the bed.

The weight of solid product removed from the bed did not exceed 80 per cent of the equivalent weight of solids introduced in the feed solution. From the standpoint of product recovery, this feed system does not appear to be superior to using pneumatic atomizing nozzles spraying below the surface of the bed.

Product containing high alpha-phase alumina, i.e., greater than 50 weight per cent, was successfully contained within the calciner system. The weight of solids returned from a knockout cyclone to the bed during such a period averaged five times the theoretical product rate. Nevertheless, the solids leaving the system in the off-gas downstream of the cyclone were less than four per cent of theoretical product rate. The weight of solids returned from the cyclone to the bed during periods of low alpha generation, i.e., less than 25 per cent, was less than the theoretical product rate. However, solids in the off-gas downstream of the cyclone were as high as 30 per cent of the theoretical product rate.

The results of this work indicated that a product with high alpha alumina content could be contained in a calciner system at least to the same extent as a product with low alpha content.

This report is largely the summary report submitted by Battelle Memorial Institute under a Task Agreement with Phillips. Certain product analyses and interpretations of the data from these analyses were made at the ICPP. The major conclusions, however, are those presented by BMI.

II. INTRODUCTION

In the continental United States, radioactive liquid wastes resulting from aqueous reprocessing of spent reactor fuels are being stored presently in underground steel tanks. If these tanks were breached by any means it is obvious that radioactive contamination of a large area could result. Therefore, research efforts throughout this country have been directed toward safer and more economical waste storage. Belter (1) recently described the processes being investigated to develop the ultimate storage method.

For the past several years, the Atomic Energy Division of Phillips Petroleum Company has investigated the fluidized bed technique for the reduction of radioactive wastes to solids. This program has been concerned principally with the calcination of aluminum nitrate solutions in six-inch diameter and two-foot-square calciners. In the fluidized bed calciner simulated waste solution is introduced into a bed of hot calcined alumina through pneumatic atomizing nozzles. The aluminum content of the solution remains in the bed as spherical pellets of alumina, and water and nitrogen oxides are carried from the calciner with the fluidizing gas.

Because the restrictions regarding air pollution are far more stringent than those encountered in normal chemical processing plants, essentially complete removal of all particulate matter from the effluent gases is of particular importance in the treatment of radioactive wastes. Of the many process techniques for cleaning effluent gases, the wet scrubbing technique has received the most effort at the Idaho Chemical Processing Plant. This system includes a dry cyclone for removing the bulk of the solids leaving the calciner bed, a cooler and venturi scrubber for further reduction of solids loading, silica gel adsorbers for removing volatile fission products, and AEC-type filters for final particle removal before the gas is discharged to the atmosphere.

The solids loading in the off-gas from a fluidized bed calciner is dependent primarily upon the size and mass of the particles and the superficial gas velocity. The median particle size of the bed is significantly influenced by the amount of attrition that occurs in the bed and by the size of the liquid droplets produced by the spray nozzle. Pneumatic atomizing nozzles spraying below the surface of a fluidized bed are known to be capable of causing considerable particle attrition as well as producing liquid droplets of very small sizes. Since larger liquid droplets and reductions in energy put into the bed should, provided attrition from the fluidizing media is negligible, decrease the

(1) Recent Developments In the Processing and Ultimate Disposal of High Level Radioactive Wastes. Presented at the 16th Annual Purdue Industrial Waste Conference. May 2-4, 1961, Lafayette, Indiana.

percentage of small diameter particles in the bed as well as decrease the amount of solids entrained in the off-gas, it was believed that introduction of feed above the bed level might offer advantages over the method currently in use.

A feed system of this type had been developed at the Battelle Memorial Institute [1] for introducing solutions or slurries into a fluidized bed calciner. Feed is sprayed downward on the top of the bed. The feed gun enters the calciner axially through the top. Because of this prior work and because equipment was available for conducting pilot plant tests, Battelle was requested to carry out studies of the applicability of its technique for calcining simulated aluminum-type wastes in a fluidized bed.

This report is largely the summary report submitted by BMI covering the work done under a Task Agreement with Phillips. Contributions by the Phillips technical organization to this work included the performance of some of the product analyses, the interpretation of the data obtained from these analyses in the light of the BMI data, and the comparison of the results obtained in the BMI study with the results obtained in the ICPP method of pneumatically atomizing calciner feed below the surface of the fluidized bed.

(1) The original system was developed in cooperation with the Falconbridge Nickel Mines, Ltd., and is described in U. S. Patents 2,813,015, 2,813,016, and 2,930,587.

III. EXPERIMENTAL WORK

The testing of a special system for introducing aqueous feed into a fluidized bed calciner was accomplished at the Battelle Memorial Institute. A description of the calciner and its auxiliary equipment, an outline of the experimental procedure and methods of evaluating the products, and the tabulation of the data which were obtained are included in this section of the report.

A. Description of the Equipment

1. Fluidized Bed Calciner

The fluidized bed calcination unit which was used in the experimental program had an inside diameter of 22 inches and was 72 inches high (inside measurements). The unit was lined with 7 inches of castable refractory, and had a cast refractory orifice plate. The original orifice plate had stainless steel bubble caps, but these failed during preliminary startup operations. A new orifice plate was cast with twenty-five 3/8-inch openings shaped in the form of deformed, reclining z's (⧖), spaced on 4-inch centers. This type of orifice plate eliminated the need for bubble caps.

The calciner was mounted above a windbox combustion chamber in which natural gas was burned in an excess of air. The resulting gas, which passed through the orifice plate, fluidized the bed and provided heat for the evaporation and decomposition of the aluminum nitrate solutions.

The off-gases from the calciner were discharged through a 4-inch opening in the top of the unit into the dust collection system.

Thermocouples were placed at strategic points in the calciner to provide means of measuring the temperature of the inlet gases, the fluidized bed, the freeboard space, and the off-gas streams. The temperature of the bed was controlled at a selected value by varying either the flow of natural gas or the liquid feed rate.

A diagram of the fluidized bed calciner and the auxiliary equipment is shown in Figure 1.

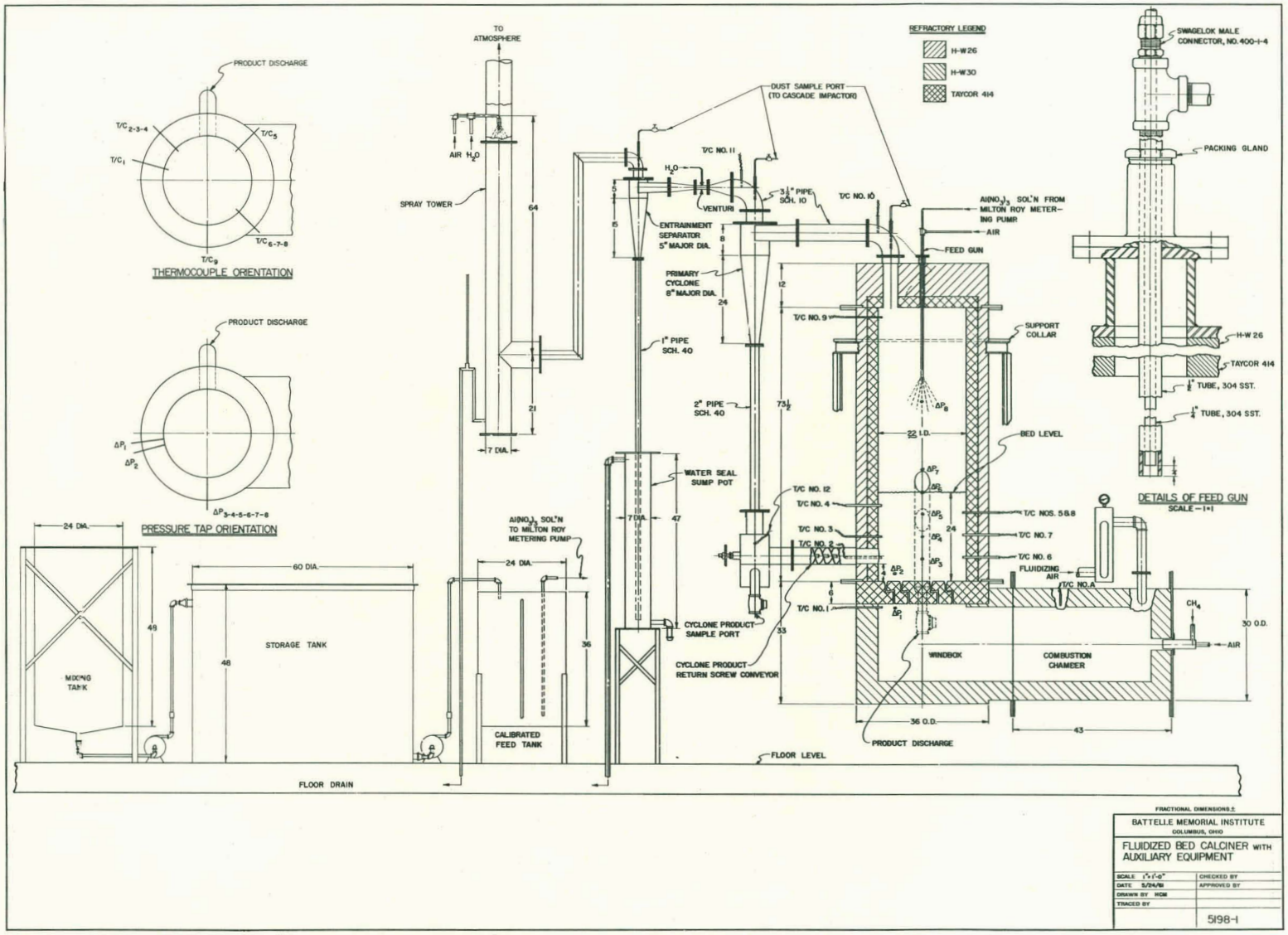

Figure 1. Fluidized Bed Calciner and Auxiliary Equipment

2. Dust Collection System

The effluent gases from the fluidized bed unit passed through a 4-inch stainless steel duct into the primary cyclone. Both the duct and the cyclone were insulated with fiber glass so that they could be maintained at a temperature above the dew point of the gaseous mixture.

The alumina particulates which were collected in the primary cyclone dropped through an insulated 2-inch pipe into a screw conveyor, which returned the material to the calciner to serve as nuclei for particle formation.

After passing through the primary cyclone, the gases passed through a venturi scrubber which had a one-inch-diameter throat, then through an entrainment separator. The mixture of solids and liquid from the entrainment separator dropped through a one-inch pipe into a sump which provided a water seal for the slurry discharge system. The slurry from this sump overflowed into the drainage system for the building. The gases from the entrainment separator were directed into a stainless steel stack, where they were contacted with a finely divided spray of water for additional removal of suspended solids and nitrogen oxides. This stream also was discharged through a water seal into the drainage system for the building. The gases passed on through the stack and were discharged to the atmosphere.

3. Feed Preparation and Introduction System

Simulated, aqueous aluminum waste solutions were prepared in a two-foot-diameter by 4-foot-high agitated tank. After mixing, the solution was transferred to a 5-foot-diameter by 4-foot-high storage tank, and from the storage tank into a calibrated feed tank, in both instances with small centrifugal pumps. The feed solution was pumped from the feed tank to the feed gun with a Milton Roy metering pump for which the length of the stroke was adjusted to provide the proper feed rate.

Two types of feed gun were used during the experimental program. The first of these consisted of a single piece of 1/2-inch-diameter stainless steel tubing which entered the calciner through a packing gland located in the center of the top. The feed solutions were pumped into this tubing through the side arm of a tee, and air was introduced into the top opening of the tee to disperse the liquid and form small droplets.

The second type of feed gun consisted of a concentric arrangement of a 1/4-inch-diameter stainless steel tube and a 1/2-inch-diameter stainless steel tube. The liquid was introduced into the inner tube of this gun and was dispersed with air which passed through the annular space between the two tubes. This feed gun was installed in the same manner as the first. Both feed guns were fitted so that the feed gun-to-bed distance could be varied, if this proved to be necessary. For the

greater part of the final run in the reactor, the feed gun extended 18 inches into the freeboard of the calciner.

B. Preparation of Simulated Waste Solutions

The simulated waste solutions which were used in the experimental program were prepared from commercially available technical grade reagents. The composition of these solutions was as follows:

1.29 M	aluminum nitrate
2.84 M	nitric acid
0.078 M	sodium nitrate
0.015 M	mercuric nitrate

The greater part of the research program was conducted with solutions which contained no mercuric nitrate. Mercuric nitrate was added to the feed only during the final 92 hours of operation.

The solution was mixed in batches of 75 gallons, and each batch was sampled and chemical analyses were made. The average compositions of the batches of solution which were prepared during each day of the operation of the calciner have been determined and are shown in Table 1.

C. Experimental Procedure

The experimental work to test the BMI feed-introduction system consisted of two runs of extended duration in the 22-inch-diameter fluidized bed system described previously.

The general experimental procedure which was used in both of these runs consisted of:

(1) Placing a starter bed of calcined alumina into the calciner.*
(2) Starting a flow of air sufficient to fluidize the charge.
(3) Igniting the gas-air mixture to the combustion chamber to provide the heat required for evaporation and decomposition.
(4) Heating the charge material to the desired operating temperature.

*The calcined alumina which was used as the starter bed for the preliminary run was produced from simulated waste solution in the Phillips' two-foot-square calciner at the Idaho Chemical Processing Plant (ICPP).

TABLE 1. CHEMICAL ANALYSES OF FEED SOLUTION PREPARED DAILY DURING RUNS 1 AND 2 IN THE 22-INCH DIAMETER FLUIDIZED BED CALCINER

Date	Volume of Solution Prepared, gal	Chemical Analyses, Molarity			
		$Al(NO_3)_3$	HNO_3	$NaNO_3$	$Hg(NO_3)_2$
4-17-61	300	1.25	2.77	0.081	--
4-18-61	150	1.20	2.80	0.076	--
4-19-61	75	1.27	2.77	0.073	--
4-20-61	150	1.29	2.81	0.070	--
4-22-61	75	1.30	2.67	0.093	--
4-25-61	225	1.39	2.79	0.081	--
4-26-61	150	1.41	2.84	0.081	--
4-27-61	150	1.79	2.84	0.076	--
4-28-61	150	1.46	2.37	0.081	--
4-29-61	150	1.44	2.62	0.078	--
4-30-61	150	1.43	2.35	0.076	--
5-1-61	150	1.43	2.69	0.076	--
5-2-61	225	1.42	2.67	0.078	--
5-3-61	225	1.43	2.64	0.076	--
5-4-61	75	1.43	2.67	0.101	--
5-5-61	225	1.45	2.57	0.101	--
5-6-61	225	1.43	2.80	0.098	--
5-7-61	150	1.48	2.77	0.016	--
5-8-61	450	1.46	2.62	0.091	--
5-9-61	300	1.45	--	0.081	--
5-10-61	300	1.44	2.72	0.079	--
5-11-61	225	1.42	--	0.076	--
5-12-61	375	1.44	2.72	0.075	--
5-15-61	300	1.44	2.47	0.082	0.017
5-16-61	375	1.43	2.72	0.082	0.014
5-17-61	375	1.45	2.62	0.080	0.014
5-18-61	225	1.43	2.72	0.080	0.014

(5) Starting flows of air and solution to the feed gun.
(6) Adjusting the natural gas flow to maintain the desired windbox temperature.
(7) Adjusting the feed rate to maintain the desired bed temperature.

After the unit was operating properly and the selected experimental conditions had been met, samples of the bed were taken through an overflow discharge port located two feet above the orifice plate. Material was discharged hourly from this port at a rate sufficient to maintain the level of the fluidized bed within a normal operating range. Every four hours a sample of the discharged product was taken for determination of its physical and chemical properties.

In addition to the regular samples of discharged product, other samples were taken at intervals as follows:

(1) Samples of the dust returned from the primary cyclone to the bed were taken at several different times so that the circulating load could be determined.

(2) The slurries discharged from the venturi-type scrubber system and the spray tower were sampled periodically to determine the amount of solids carried out of the calciner at these points.

(3) A cascade impactor was used to determine the amount and particle size distribution of the solids in the gas streams from the primary cyclone and from the entrainment separator.

The results obtained from the sampling program are given in detail in Appendix A of this report and are summarized in the section immediately following.

The first run was of 136 hours' duration, and its primary objectives were to provide information regarding the operability of the calciner and the auxiliary equipment, and to indicate whether or not the calcined material could be retained in the fluidized bed under the conditions of the experiment.

The nominal operating conditions for the first run were as follows:

Bed temperature - 450 C
Windbox temperature - 1035 C
Superficial velocity - 1.1 fps

The feed gun consisted of a single 1/4" stainless steel pipe having the same dimensions as the concentric pipe feed gun shown in Figure 1, except the inner pipe was eliminated. The air flow to the feed gun was maintained at about 8 to 9 scfm during the entire run.

A little over twenty-five per cent of the time of the first run was spent in improving the operability of the calcination equipment and the exhaust system. During the remaining 97 hours, a total of 520 gallons of simulated reactor waste solution, containing the equivalent of approximately 280 pounds of aluminum oxide (Al_2O_3), was fed into the calciner. Of the 280 lbs. of the equivalent aluminum oxide fed into the calciner as aluminum ion, approximately 200 lbs. was recovered in the material discharged from the calciner during the operation and in the material remaining in the calciner at the end of the run.

A list of the samples which were taken during this first run and the results of the physical evaluations are included in Appendix A of this report.

The second run in the twenty-two-inch-diameter calciner was carried out over a period of 24 days, during which time the simulated waste solution was fed into the reactor a total of 516 hours. Since the conditions were varied several times during this run, it will be broken down into several phases for purposes of this report. During the first two phases, the operating conditions were varied in order to study the effects of such variations on the pelletization of the material. In Phases 3 and 4, the bed temperature was decreased to increase the capacity of the unit, and the feed gun air rate was decreased to study the effects of the ratio of the nozzle air rate to feed rate on the mass median particle diameter* of the product. Mercuric nitrate was added to the feed solution at the beginning of Phase 5 in order that its effect on pelletization could be studied.

The nominal conditions used in Phase 1 of Run 2 were as follows:

Bed temperature - 450 C
Windbox temperature - 1100 C
Superficial velocity - 1.1 fps
Feed gun air - 7 to 8.5 scfm

This phase of Run 2 extended over a total of 128 hours in which aluminum nitrate solution was fed into the reactor. A total of 861 gallons of solution having an average composition of 1.48M aluminum nitrate, 2.65M nitric acid, and 0.079M sodium nitrate was fed into the reactor.

Screen analyses on samples of material from the first phase of Run 2 indicated the average particle size of the product was smaller than was considered desirable, and certain operational changes were made during the second phase of the run.

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* The mass median particle diameter is calculated by multiplying the arithmetic mean of the openings of two screens of the $\sqrt{2}$:1 series by the weight fraction of the material remaining on the screen with the smaller opening, and summing the results for the entire screen series.

Among the changes which were made in this second phase were increases in superficial velocity (0.9 to 1.2 fps) and a change in feed gun design. The second phase covered a period of 107 hours of operation, during which time 713 gallons of solution were fed into the calciner and 342 pounds of product were discharged. A new feed gun of concentric pipe design, (see Figure 1), was placed in operation at the beginning of this phase and was used continuously until the end of the entire campaign without plugging or difficulties of any other type. The particle size distribution tended to level off during this phase, but the material remained considerably smaller in size than was considered desirable.

At this point it was decided to change the operating conditions so as to obtain a higher feed rate and to vary only the operation of the feed gun and the superficial velocity, if necessary, during the remainder of the run. In order to obtain an increased feed rate, the windbox temperature was increased to 1200 C, the bed temperature was decreased to 400 C, and the superficial velocity was set at 1.2 fps. Under these conditions, the feed rate increased from the 5 to 6 gallons per hour which had been maintained during Phase 2, to 13 to 14 gallons per hour. The conditions outlined above were maintained over a period of 125 hours, during which time 1644 gallons of solution were fed into the calciner with the air flow to the feed gun set at 7.5 scfm. During this phase of the run (Phase 3), a total of 758 pounds of product was discharged from the calciner.

The mass median particle diameter appeared to have reached a reasonably constant value of about 0.25 mm during the latter part of Phase 3, and the feed gun air was lowered to 3 scfm in an attempt to increase the mass median particle diameter during the fourth phase of operation. The other experimental conditions in Phase 4 were the same as used in Phase 3. Phase 4 extended over a period of 64 hours, and the mass median particle diameter increased to about 0.4 mm and had begun to level off somewhere between this value and 0.45 mm. A total of 829 gallons of solution was fed into the calciner during Phase 4, and 399 pounds of product were discharged.

Phase 4 was discontinued in order that the remaining time would be sufficient to determine what influence, if any, mercuric nitrate would have on the formation of pellets in the calciner. This material was not available at the beginning of Run 2, and its addition to the solution was delayed until late in the run in order to avoid the introduction of an additional variable. At the beginning of Phase 5, purified mercuric nitrate was added to the feed solution in an amount sufficient to provide a concentration of 0.014 molar. The other components of the solution were kept at the same concentration which had been used throughout Run 2. Phase 5 extended over a 92-hour period, during which time 1269 gallons of solution were fed into the calciner and 558 pounds of product were discharged. The first 52 hours of this phase of the run were characterized by a rapid increase in the mass median particle diameter. At the end of 52 hours, the mass median particle diameter had risen to about 0.9 mm, and the air flow to the feed gun was increased to 7.5 scfm in an attempt to reverse the trend toward larger particle

diameters. Unfortunately, the particle size had increased to where the quality of fluidization had become rather poor at the prevailing superficial velocity, and it became necessary to increase the superficial velocity to about 1.6 fps before the effects of increasing the feed gun air could be determined. The mass median particle diameter reached a maximum of about 1.06 mm before the combination of the increased attrition rate, caused by increased superficial fluidizing velocity, and increased feed gun air appeared to have reversed the trend. The mass median particle diameter was 0.9 mm at the end of the run.

A list of the samples taken during Run 2 and the detailed results of the evaluation procedures which were carried out at Battelle appear in Appendix A. These data have been summarized in tables and graphs which appear in the following section of this report. These tables of data also include the information which was collected on the solids loading in the scrubber solutions.

IV. EXPERIMENTAL RESULTS

The primary purpose of the preliminary run (Run 1) in the 22-inch-diameter fluidized bed calciner was to provide information concerning the operational characteristics of the system. The alumina (397 pounds) used as the starting bed was produced in the ICPP two-foot-square calciner. This material had a bulk density of 60.5 pounds per cubic foot and a mass median particle diameter of 0.37 millimeter. During startup, approximately 75 pounds of material was elutriated from the calciner. (This estimate is based on the amount of discharged product which was returned to the bed at this time in order to raise the level of the static bed to the original level.) The weight of product removed during the run and the bed recovered from the calciner at the conclusion of the run exceeded the original charge by 207 pounds. The solids in the calciner at the end of Run 1 had a bulk density of 67.6 pounds per cubic foot and a mass median particle diameter of 0.35 millimeter.

A minimum of data were collected during Run 1 and, consequently, little information was obtained concerning pelletization. Several solid samples were taken, however, and their particle size distributions and bulk densities were determined. The average daily experimental conditions, and a list of samples taken together with the results of screen analyses and bulk density determinations, are included in Tables A-1 and A-2 of the Appendix. The screen analyses indicated that 70 to 75 per cent of the solids was finer than 65 (Tyler) mesh.

During the run, 520 gallons of solution containing aluminum nitrate theoretically reducible to 280 pounds of alumina were fed into the calciner. The weight of solids recovered (207 pounds) accounted for 74 per cent of the total salt content of the feed.

The second run in the Battelle 22-inch-diameter calciner was begun with a starter bed prepared from solids recovered during Run 1. This initial bed weighed 200 pounds, had a bulk density of 85.5 pounds per cubic foot, and had a mass median particle diameter of 0.22 millimeter. The average daily experimental conditions are shown in Table A-3 of the Appendix.

Samples of solid product from the calciner were removed generally at four-hour intervals, and forwarded to Phillips Petroleum Company for physical and chemical analyses. The weight of the product removed was recorded, and scrub samples were taken to determine the amount of solids collected in the scrubbing stream. These data are shown in Figures 2, 3, and 4, and discussed below.

Several efforts were made to determine the amount and particle size distribution of the alumina particulates in the off-gas stream by using a cascade impactor. The amount of solids collected was so small, however, that even after several hours of sampling, only 30 to 40 milligrams of material were collected by the impactor. These data were not consistent with the results obtained from the scrubber solution samples, which

indicated a considerably higher dust loading in the gases. The reason for this inconsistency became apparent when the gas-sampling probes were inspected at the end of the run and found to be partially plugged. The plug ahead of the cascade impactor was evidently filtering out the greater portion of the dust in the gas-sampling stream. Since these data do not appear valid, they are omitted from the report.

In addition to the sampling of the exhaust gas stream from the primary cyclone, the amount of solids returned from the cyclone to the bed via a screw conveyor was determined several times. During the early stages of Phase 3 when the mass median particle diameter of the product was low, the circulating load in the dust return system amounted to about 20 to 30 pounds per hour. Later, as the percentage of fine material in the bed decreased, this load of circulating dust decreased to a rate of two to four pounds an hour. Thereafter, sampling of this system was discontinued because the time required to obtain samples became excessive; furthermore, sampling disrupted the return of seed particles to the bed.

Differential pressure readings between several points in the calciner were affected by partial plugging of the pressure probes. Hence, no pressure data are presented.

V. DISCUSSION OF RESULTS

The results obtained for the Battelle system of introducing slurries and solution into fluidized bed calciners indicate that this system can be applied to the calcination of aluminum nitrate solutions. Obviously, the investigation of all the independent variables associated with calciner operation was not possible during this brief experimental program. However, certain trends in the physical and chemical properties of the product, and operating characteristics of the process were observed and are reported herein.

Results of Run 1 were included in previous sections of this report, and, since the run was primarily exploratory, no further discussion will be presented. Because operating conditions were varied several times during Run 2, discussion of the results of this run has been divided into the five phases representing these conditions.

Phase 1

This phase of Run 2 was operated at a bed temperature of 450°C, a superficial fluidizing velocity of 1.1 feet per second, a feed rate of five to six gallons per hour, and a feed gun air rate of 7.0 to 8.5 scfm. The original feed gun, with a single tube for both air and liquid, was used. The product characteristics, which are shown in Figures 2 and 3, were as follows:

1. The mass median particle diameter decreased from an initial value of 0.22 millimeter to 0.11 millimeter, then increased to 0.19 millimeter at the end of this phase.

FIGURE 2 PRODUCT DATA

CALCINER BMI RUN No. 2
FEED RATE Variable l/hr. BED TEMP. 450±400 °C

SUPERFICIAL FLUIDIZING VELOCITY 1.1 - 1.2 ft/sec.

FEED:
$Al(NO_3)_3$ 1.29 M $NaNO_3$ 0.078 M
HNO_3 2.84 M $Hg(NO_3)_2$ 0 except as noted M

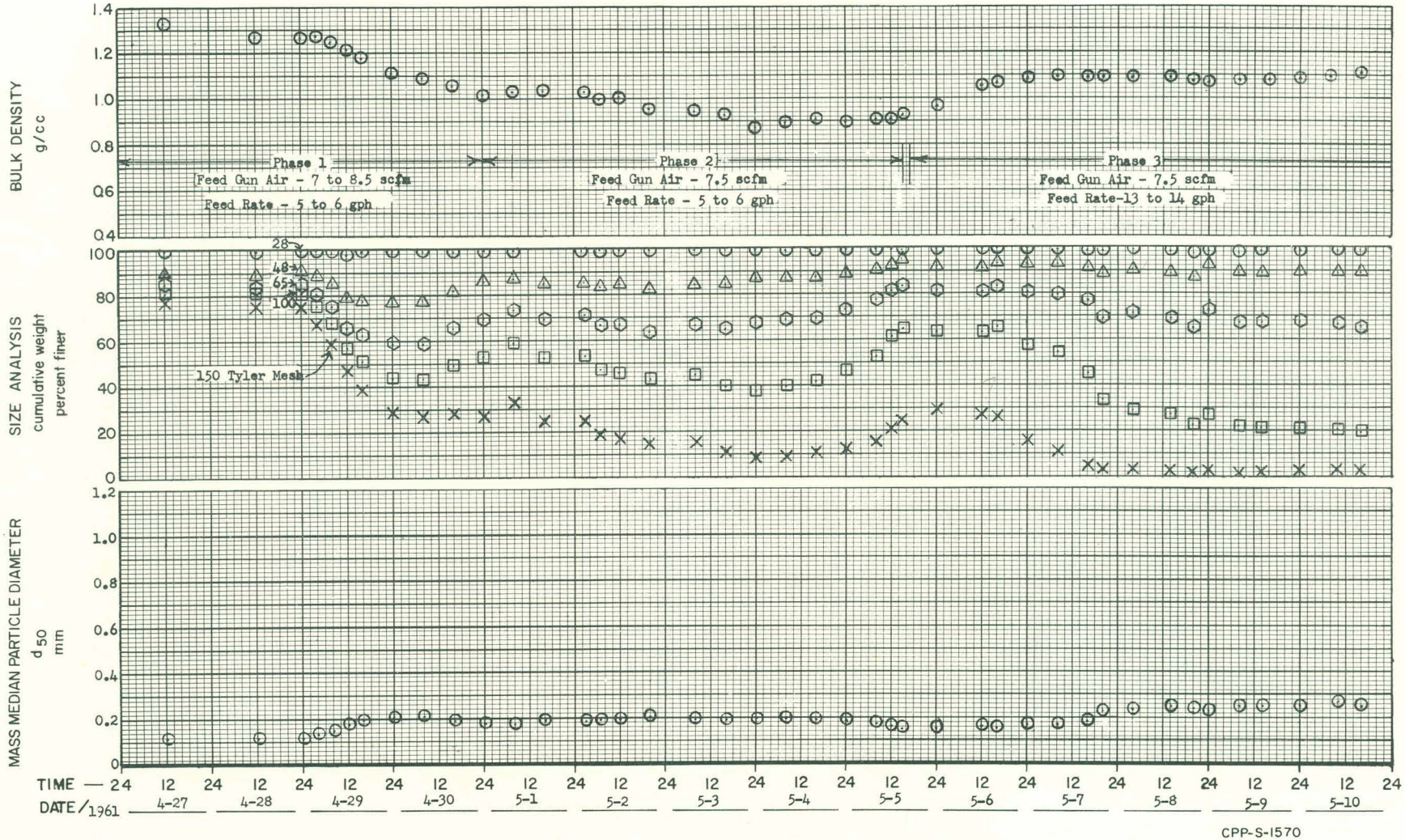

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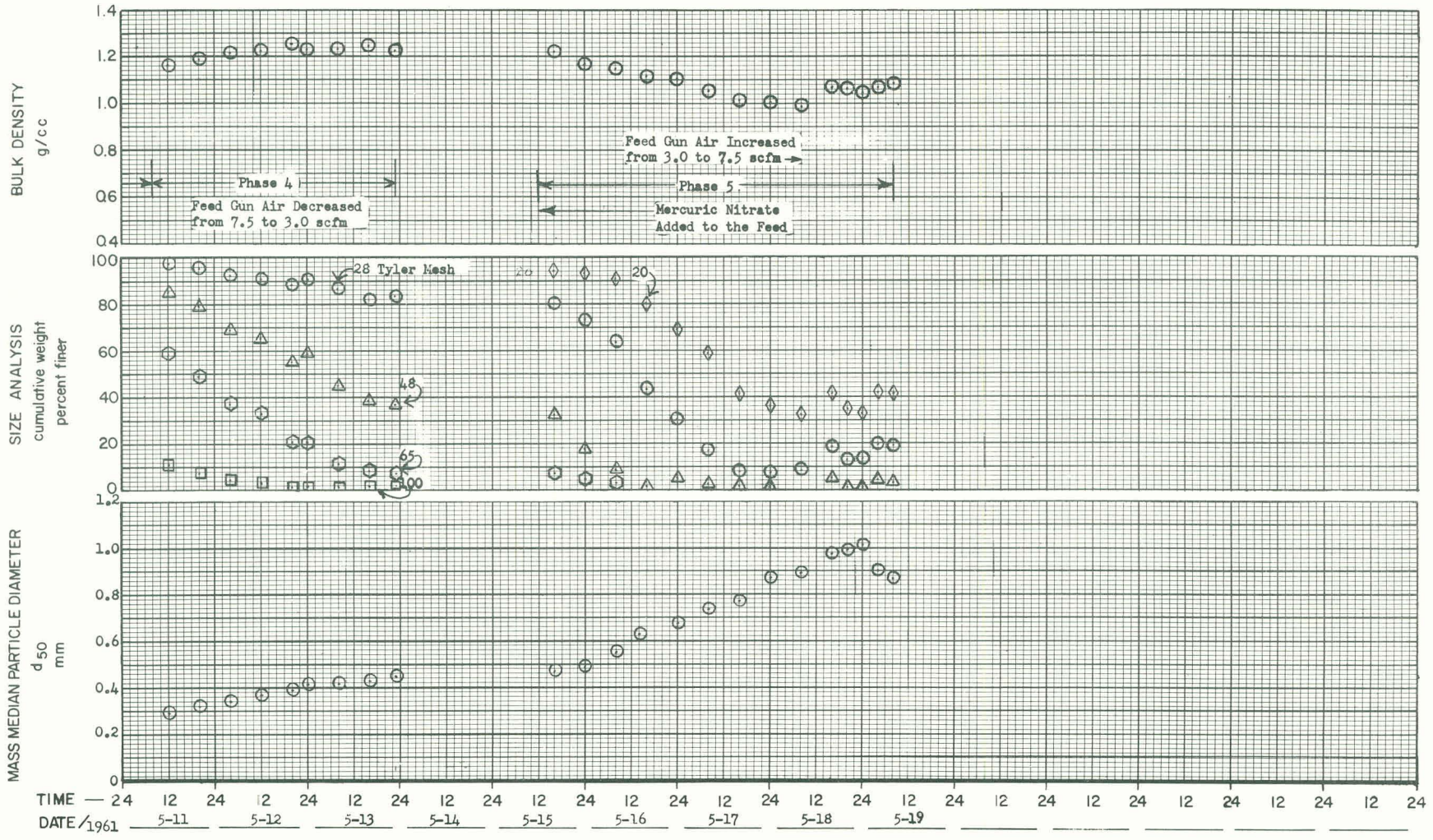
FIGURE 2 (Cont'd) PRODUCT DATA

CALCINER BMI RUN No. 2
FEED RATE Variable l/hr. BED TEMP. 450±400 °C

SUPERFICIAL FLUIDIZING VELOCITY 1.1 - 1.2 ft/sec.

FEED:
$Al(NO_3)_3$ 1.29 M
HNO_3 2.84 M
$NaNO_3$ 0.078 M
$Hg(NO_3)_2$ 0 except as noted M



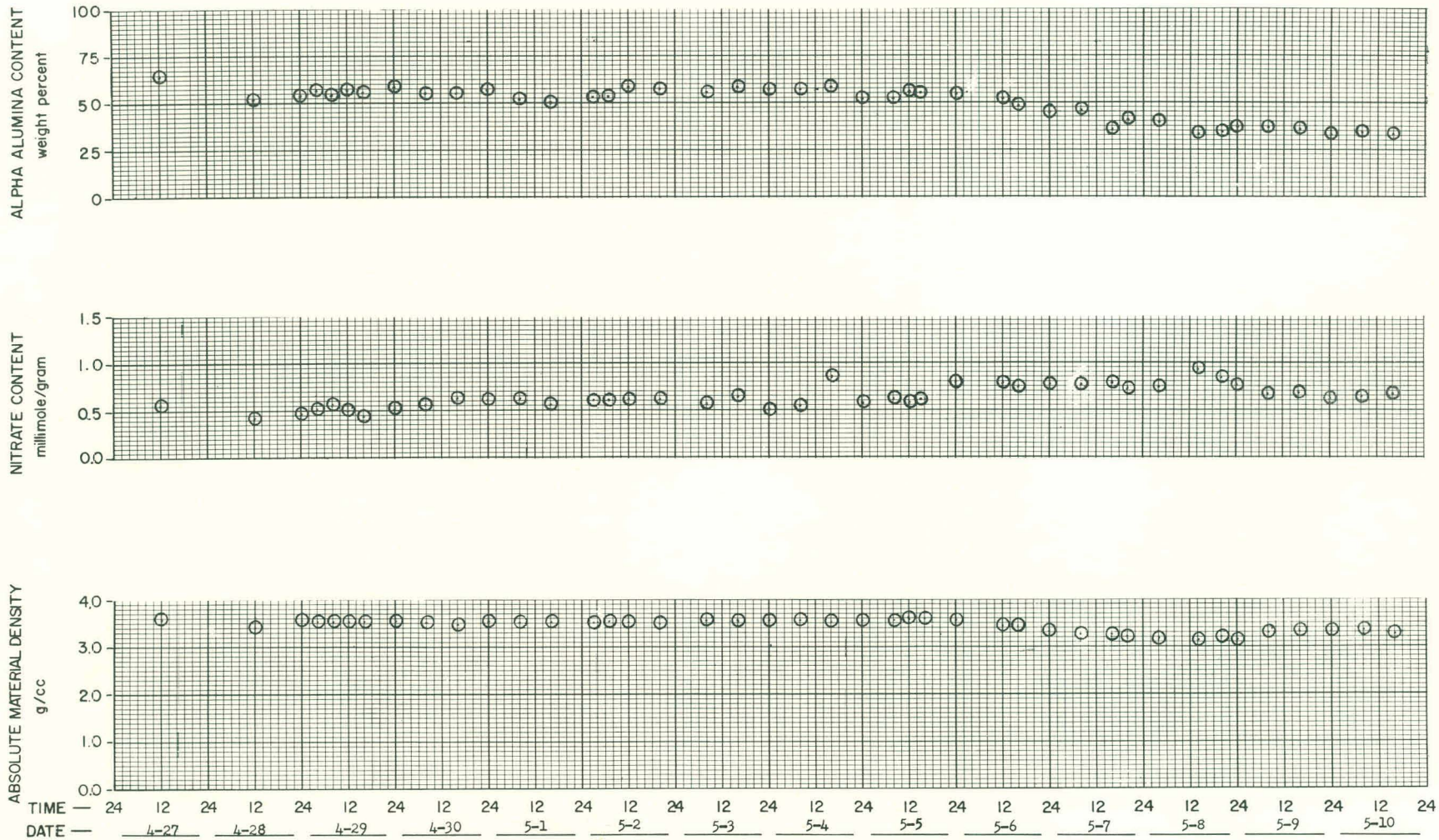
CPP-S-1570

FIGURE 3 PRODUCT DATA

CALCINER BMI RUN No. 2
FEED RATE Variable l/hr BED TEMP. 450±400 °C

SUPERFICIAL FLUIDIZING VELOCITY 1.1-1.2 ft/sec

FEED:
$Al(NO_3)_3$ 1.29 M $NaNO_3$ 0.078 M
HNO_3 2.84 M $Hg(NO_3)_2$ 0 except as noted M



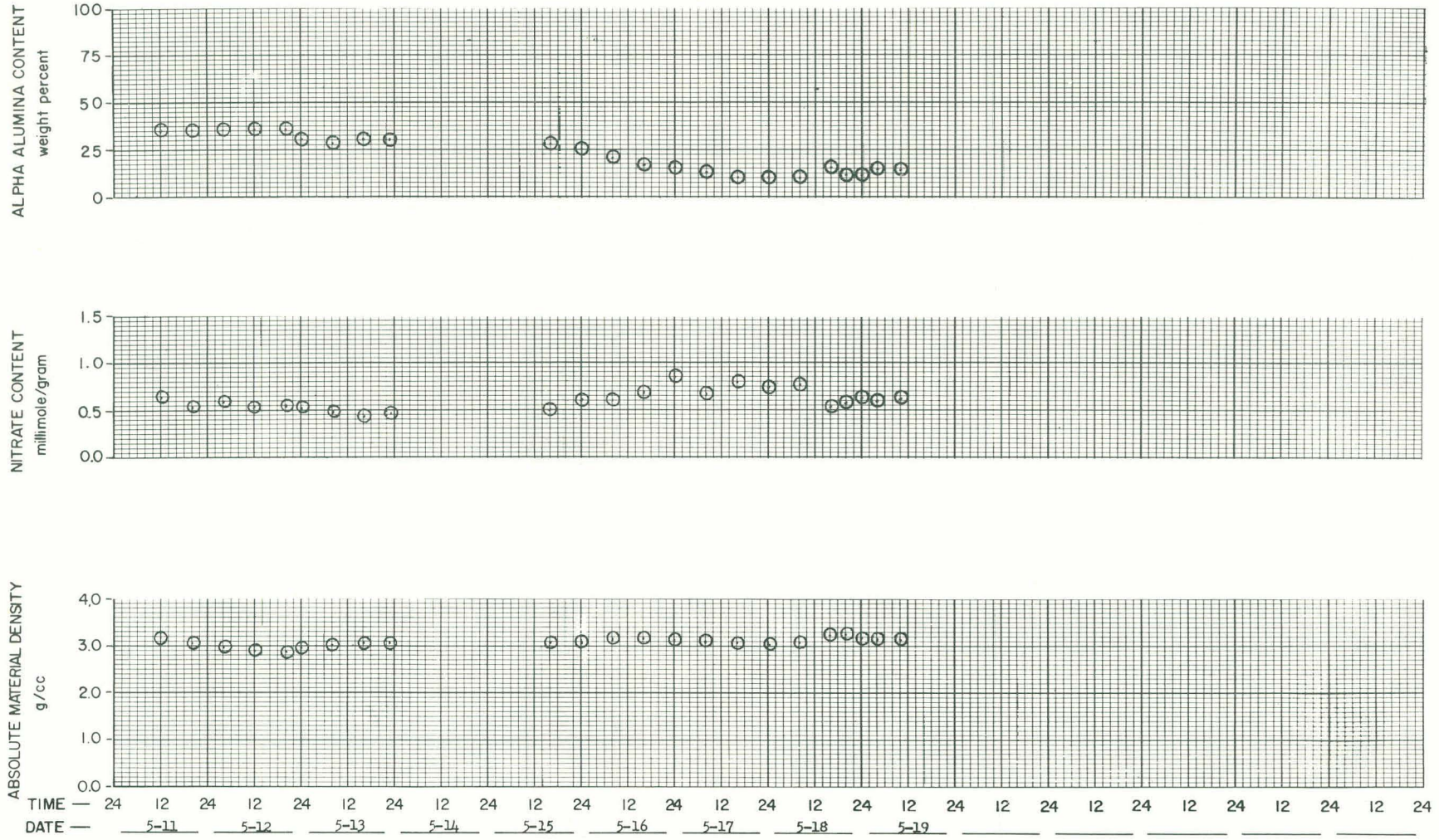
CPP-S-1572

FIGURE 3 (Cont'd) PRODUCT DATA

CALCINER BMI RUN No. 2
FEED RATE Variable l/hr BED TEMP. 450±400 °C

SUPERFICIAL FLUIDIZING VELOCITY 1.1 - 1.2 ft/sec

FEED:
$Al(NO_3)_3$ 1.29 M
HNO_3 2.84 M
$NaNO_3$ 0.078 M
$Hg(NO_3)_2$ 0 except as noted M

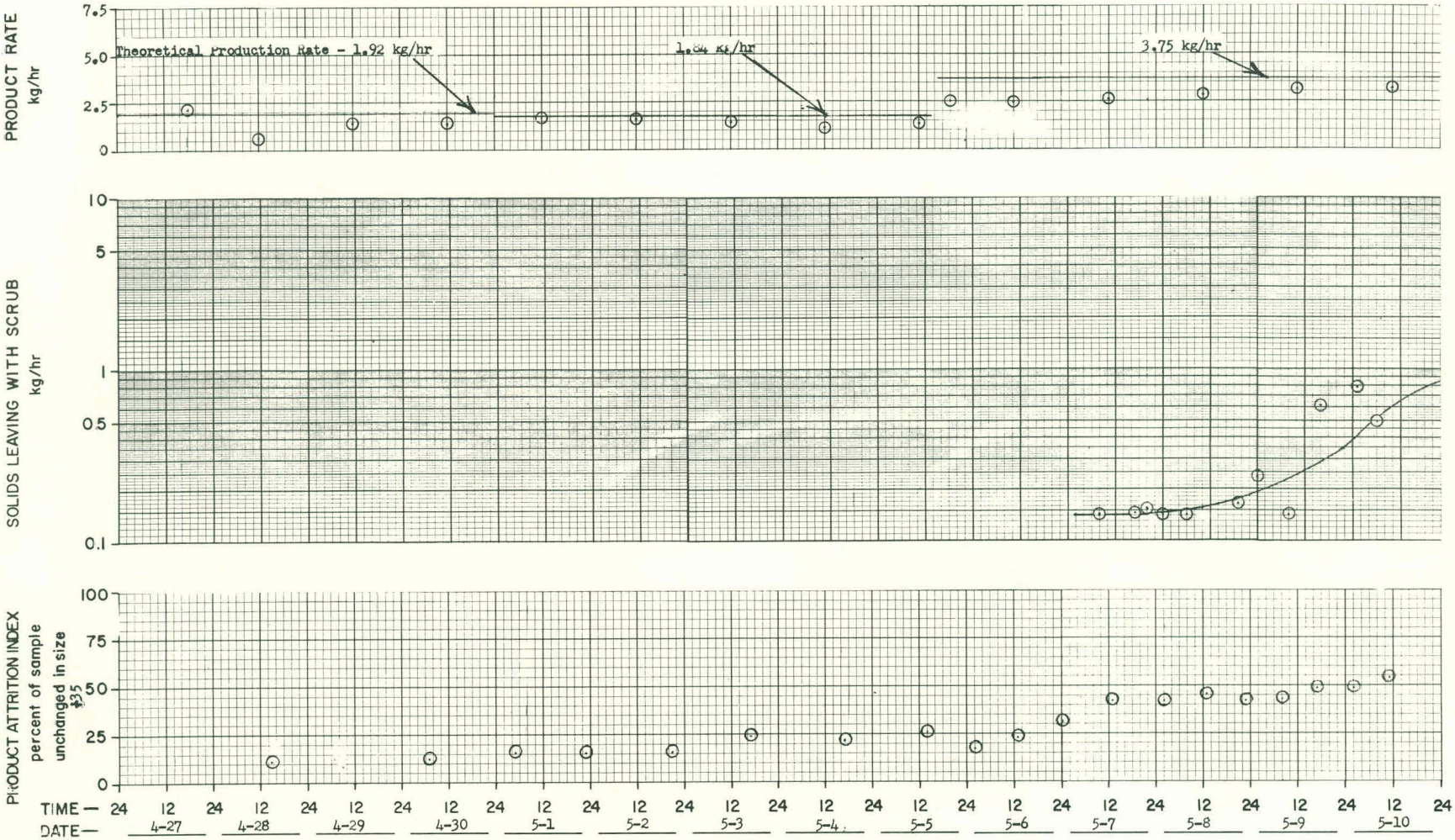


CPP-S-1572

FIGURE 4 GENERAL DATA

CALCINER BMI RUN No. 2
FEED RATE Variable l/hr BED TEMP. 450±400 °C
SUPERFICIAL FLUIDIZING VELOCITY 1.1 - 1.2 ft/sec

FEED:
$Al(NO_3)_3$ 1.29 M $NaNO_3$ 0.078 M
HNO_3 2.84 M $Hg(NO_3)_2$ 0 except as noted M



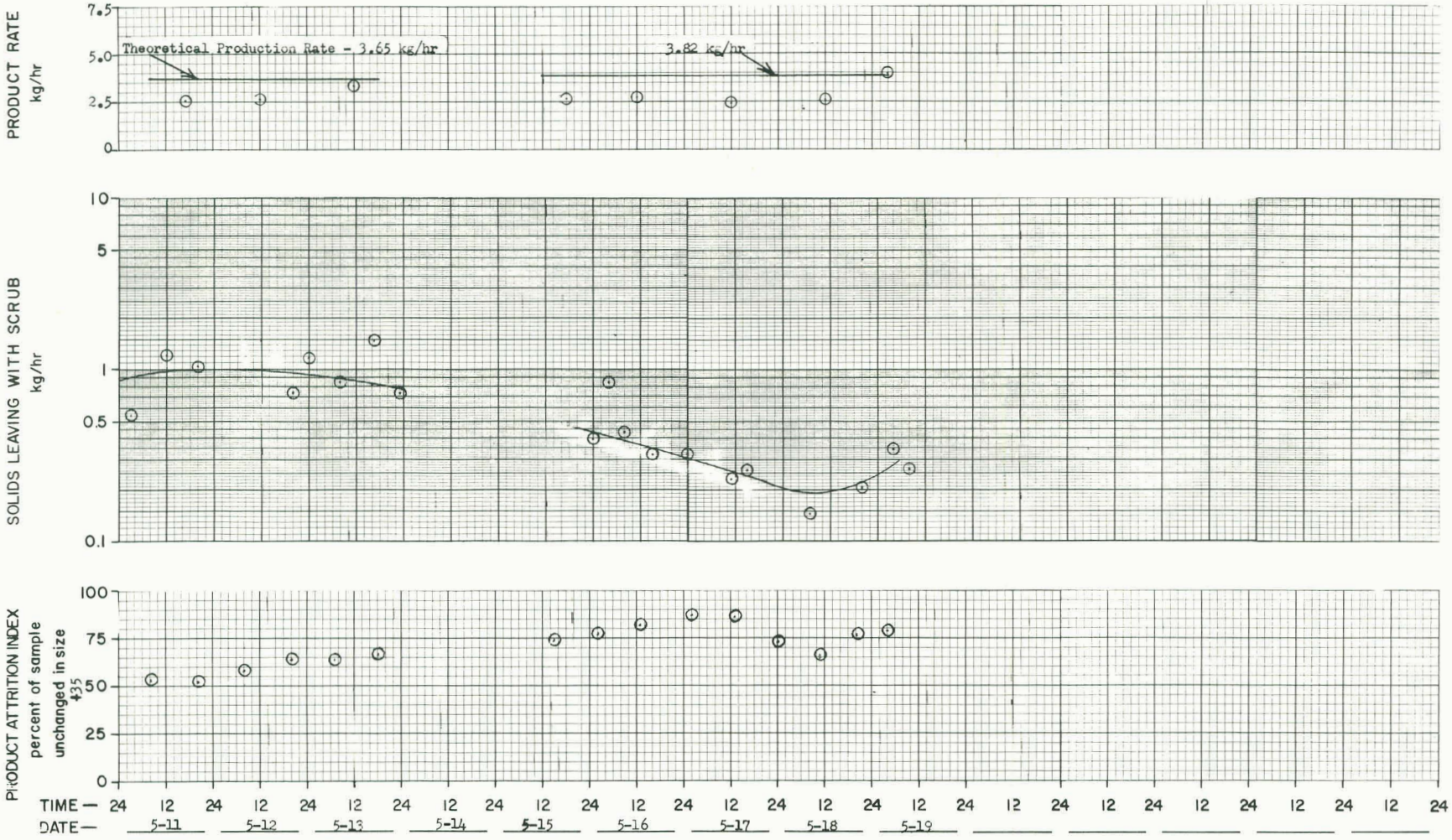
CPP-S-1573

FIGURE 4 (Cont) GENERAL DATA

CALCINER BMI RUN No. 2
FEED RATE Variable l/hr BED TEMP. 450/400 °C

SUPERFICIAL FLUIDIZING VELOCITY 1.1 - 1.2 ft/sec

FEED:
$Al(NO_3)_3$ 1.29 M
HNO_3 2.84 M
$NaNO_3$ 0.078 M
$Hg(NO_3)_2$ 0 except as noted M



CPP-S-1573

2. The absolute material density was essentially constant throughout at 3.52 g/cc.

3. The nitrate content decreased from an initial value of 0.75 millimole per gram to 0.45, then increased to 0.61 at the end of this phase.

4. The alpha alumina was initially 70 weight per cent, then decreased rapidly to 55 per cent in 24 hours and remained at this value until the end of this phase.

5. The bulk density decreased slowly from an initial value of 1.35 g/cc to 1.02 at the end of this phase.

6. The size analyses indicated that the bed increased in size. The percentage of material finer than 150 mesh decreased from 67 to 27 per cent during the operation.

7. The product was too small for determination of particle density. The change in bulk density may have resulted from changes in particle density and/or porosity.

8. The attrition-susceptibility tests, using a technique of Forsythe and Hertwig (1), indicated that 90 to 95 per cent of a -28 to +35 mesh material was reduced in size. This high attrition rate is attributed to the high alpha alumina content.

Operating characteristics observed during Phase 1, which are shown in Figure 4, were as follows:

1. The measured ratio of feed-gun-air rate to feed rate varied between 525 and 760. This range of nozzle air ratio was much higher than that normally used for nozzles in the ICPP calciners. However, the velocity of the air through the throat of the ICPP nozzle was 1100 to 1200 feet per second, i.e., sonic; whereas the superficial velocity through the BMI feed gun was 120 to 140 feet per second. Atomizing characteristics were obviously different.

2. The theoretical production rate for this phase should have been 1.92 kilograms per hour. The amount of solids removed from the calciner averaged only 66 per cent of the theoretical.

3. Samples of the solutions from the spray tower and venturi scrubber were not taken during this period. Hence, no estimate of the solids content of the scrub solution is possible.

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(1) Ind. Eng. Chem., Vol. 41, pp 1200 - 06.

Phase 2

The nominal operating conditions of this phase of Run 2 were a bed temperature of 450°C, a feed rate of five to six gallons per hour, and a feed-gun-air rate of 7.5 scfm. The superficial fluidizing velocity was varied between 0.9 and 1.2 feet per second. The new feed gun of concentric pipe design was used. The physical and chemical properties of the product were as follows:

1. The mass median particle diameter was essentially constant at 0.19 millimeter for most of the phase, then decreased rapidly to 0.15 in the final twelve hours.

2. The absolute material density remained between the values of 3.50 and 3.60 g/cc throughout Phase 2.

3. The nitrate content varied between 0.5 and 0.6 millimole per gram of solids.

4. The alpha alumina content averaged 55 weight per cent. Variations shown on Figure 3 were within the limits of analytical error.

5. The bulk density of the product decreased from 1.02 g/cc to 0.92.

6. The size distribution of the product indicated that the material finer than 65 Tyler mesh increased initially, then decreased slightly, and finally increased gradually toward the end of Phase 2. The net result was an increase from 70 to 82 weight per cent.

7. The product was too small for accurate determination of particle density.

8. The attrition tests indicated that the product became somewhat less susceptible to attrition. The percentage of -28 to +35 mesh material which was reduced in size dropped from 85 to 75 per cent.

Operating characteristics observed during Phase 2 were as follows:

1. The volume ratio of feed-gun-air rate to feed rate was 670 for five gallons per hour and 560 for six gallons per hour. The velocity of the air for this feed gun was 190 feet per second.

2. The theoretical production rate for this phase was 1.84 kilograms of solids per hour. The actual measured rate of production was 79 per cent of the theoretical, the highest attained in the BMI tests.

3. Scrub samples were not taken during this period.

Of prime significance during this phase was the high percentage of the solids that was removed from the calciner in the product stream, even though the alpha alumina content was high. In the ICPP 24-inch calciner[1], high alpha has invariably caused high solids loading in the off-gas. The attrition index for product from this run was very high, i.e., 75 to 85 per cent reduction, but a high product removal rate was still attained. Thus, product with high alpha alumina content can be tolerated under certain conditions while calcining aluminum-type wastes.

Phase 3

The nominal operating conditions for Phase 3 of Run 2 included a bed temperature of 400°C, a superficial gas velocity of 1.2 feet per second, a feed rate of 13 to 14 gallons per hour, and a feed-gun-air rate of 7.5 scfm. The product properties of Phase 3 were as follows:

1. The mass median particle diameter increased from an initial value of 0.15 millimeter to 0.25 at the end of this period.

2. The absolute material density decreased from 3.60 g/cc to 3.15, then increased to 3.25 g/cc.

3. The nitrate content decreased from 0.75 to 0.65 millimole per gram of solids.

4. The alpha alumina percentage decreased steadily from an initial value of 55 weight per cent to 35 per cent at the end of this phase.

5. The bulk density of the product increased from 0.92 g/cc to 1.11 g/cc during this period.

6. The weight per cent of solids finer than 65 mesh decreased from 82 to 65 per cent. The largest change in size distribution occurred on that material finer than 100 mesh. This percentage decreased from 65 to 19 per cent during Phase 3.

7. The product again was too small for accurate particle density measurements.

8. Attrition susceptibility decreased during this period. The percentage of -28 to +35 mesh material which was reduced in size dropped from 75 to 45 per cent. This change is related to the decrease in the alpha alumina content.

(1) Bower, J. R., Chemical Processing Technology Quarterly Progress Report, July - September, 1960, IDO-14540.

Operating characteristics observed during Phase 3 were as follows:

1. The ratio of feed-gun-air rate to feed rate ranged from 258 for 13 gallons per hour to 240 for 14 gallons per hour. The velocity of air through the feed gun was essentially unchanged from Phase 2, i.e., 190 feet per second.

2. The theoretical production rate for Phase 3 was 3.75 kilograms per hour. The actual measured product rate was 2.75, approximately 73 per cent of the theoretical. The percentage recovery increased gradually as the operation progressed. Near the end of the period, the amount of solids removed as product approached 87 per cent of the theoretical, although initially the value was 67 per cent.

3. Samples of the scrub solution indicated that the rate of solids removal by the off-gas increased from an initial rate of 0.14 kilogram per hour to approximately 0.95 at the end of Phase 3.

Phase 4

The nominal operating conditions for Phase 4 of Run 2 were the same as those for Phase 3, except that the feed-gun-air rate was decreased from 7.5 to 3.0 scfm. Trends in the product properties were as follows:

1. The mass median particle diameter increased from 0.28 millimeter to 0.45 millimeter and was rising slowly when the operation was halted.

2. The absolute material density decreased from 3.2 g/cc to 2.85, then increased to 3.05 g/cc.

3. The nitrate content decreased from 0.65 to 0.45 millimole per gram.

4. The alpha alumina content decreased from 35 to 30 per cent throughout the period.

5. The bulk density increased from 1.1 g/cc to 1.22 g/cc.

6. The weight per cent of solids finer than 65 mesh decreased from 60 per cent to 7.0 per cent. Solids finer than 100 mesh made up less than 1.5 per cent of the total.

7. The amount of material changed in size during attrition tests dropped from 45 to 33 per cent.

Operating characteristics during Phase 4 were as follows:

1. The ratio of feed-gun-air rate to feed rate ranged from 104 for 13 gallons per hour to 96 for 14 gallons per hour. The superficial velocity of air through the one-half-inch tube at the tip of the feed gun was 76 feet per second.

2. The theoretical production rate during Phase 4 was 3.65 kilograms per hour. The measured rate based on the solids recovered was 2.82 kilograms per hour, 77.5 per cent of the theoretical.

3. The concentration of solids in the effluent scrubbing solutions was such that between 0.75 and 1.5 kilograms of solids per hour were removed from the off-gas.

Presently, there is no explanation for the high solids loading in the scrubbing solution which occurred toward the end of Phase 3 and during Phase 4. The weight of solids being returned from the primary cyclone to the bed was estimated to be between one and two kilograms per hour, as compared to 10 to 15 kilograms per hour during the initial stages of Phase 3. The solids removed by the scrub had been 0.18 kilogram per hour during a period of Phase 3 at comparable alpha alumina content of the product.

Similarly, the simultaneous decrease in both alpha alumina and nitrate contents of the product, and an increase in the bulk density with a decrease in absolute material density, cannot be precisely explained.

Phase 5

The nominal operating conditions for Phase 5 were the same as those for Phase 4. Mercuric nitrate was added to the feed to determine any possible effect on the operation. After 52 hours, the feed-gun-air rate was increased from 3.0 to 7.5 scfm. Trends in the product properties were as follows:

1. The mass median particle diameter rose rapidly from 0.46 millimeter to 1.09 millimeters in 80 hours, then decreased to 0.86 during the remaining 40 hours of the run.

2. The absolute material density averaged 3.1 g/cc and was nearly constant throughout the phase.

3. The nitrate content increased from 0.5 millimole per gram to 0.8 millimole per gram in 70 hours. It averaged 0.6 millimole per gram for the remainder of the run.

4. The alpha alumina content of the solids dropped from 30 weight per cent to 10 per cent, also in 70 hours. There was a slight increase to 15 per cent at the end of the run.

5. The bulk density decreased from 1.22 g/cc to 0.99 g/cc in 70 hours, then increased to 1.08 at the end of the run.

6. Small particles disappeared from the product during Phase 5. Those solids less than 20 mesh dropped from 95 per cent to 25 per cent in 70 hours, then increased to 30 per cent at the end of the run.

7. The product showed increased resistance to attrition. The per cent unchanged in size increased from 72 per cent to 87 per cent in 50 hours, then the percentage dropped to 75 at the end of the run.

8. Particle densities were determined for two samples; one taken eighteen hours after the start of Phase 5, the other 66 hours after the startup. Mercury contents were also determined. Comparisons are shown in Table 2.

TABLE 2

PROPERTIES OF TWO PRODUCT SAMPLES FROM PHASE 5

Time	Densities (g/cc)			Alpha		Calculated	Bulk Density
	Absolute	Particle	Bulk	Alumina	Mercury	Porosity	Particle Density
18 hrs.	3.15	1.95	1.145	21%	0.2 wt.%	0.38	0.589
66 hrs.	3.08	1.67	0.985	10%	1.0 wt.%	0.46	0.590

These data show that (a), the particle and bulk densities decreased simultaneously at a constant ratio; (b), the products became more porous; and (c), the mercury content of the product increased to the point where approximately 40 per cent of the mercury introduced during the 48-hour period between samples was retained in the solids.

Operating characteristics observed during Phase 5 were:

1. During the first 52 hours, the ratios of the feed-gun-air to feed rate were the same as those for Phase 4. Thereafter, the ratios were those in Phase 3.

2. The theoretical production rate during Phase 5 was 3.82 kg/hr. The actual rate averaged 2.75 kg/hr, 72 per cent of the theoretical.

3. The weight of solids leaving with the scrubbing solution decreased from 0.5 kg/hr, initially, to 0.2 kg/hr after 70 hours, then increased to 0.3 kg/hr at the end of the run.

The rapid increase in the mass median particle diameter may have been caused by the presence of mercury in the feed. However, the size analysis at the end of Phase 4 shows that the fines in the bed were being depleted rapidly, even without mercury in the feed. Consequently, the changes observed during Phase 5 may have been continuations of changes which began in Phase 4. When the feed-gun-air rate was increased from 3.0 to 7.5 scfm, the increase in the mean particle size was halted, and a decrease was observed at termination of the run.

VI. CONCLUSIONS

The objectives of the experimental program conducted at the Battelle Memorial Institute were to investigate the feed-introduction system used at Battelle when calcining aluminum nitrate solutions in a fluidized bed, and to compare this system with the pneumatic-atomized feed system used at the ICPP.

The results pertaining to the first objective were:

1. The BMI feed gun was satisfactory for introducing solutions into fluidized bed calciners.

2. The feed gun was operated without any mechanical difficulties for a total of 25 days.

3. Operation of the process and characteristics of the product were controlled principally by adjusting the feed-gun-air rate. Very fine product (mean size below 0.2 millimeter) with high alpha alumina was produced when the ratio of feed-gun-air rate to feed rate was greater than 560. Intermediate-size product (0.3 millimeter) was produced when the ratio was approximately 250. Large particles (above 0.6 millimeter) with low alpha alumina content were formed when the ratio was 100.

4. No serious caking occurred in the bed.

5. The weight of product removed from the bed was from 65 to 80 per cent of the equivalent weight of solids in the feed solution.

6. From 10 to 15 per cent of the theoretical solids equivalent of the feed solution was accounted for by the solids suspended in the scrubber liquid.

7. A few analyses for dissolved solids in the scrubbing streams indicated that a considerable portion of the "unaccounted for" solids may have been dissolved in the scrubber solution.

8. The weight of solids recovered from the bed as product averaged 79 per cent of the theoretical production rate during the period when the alpha alumina content of the product averaged 55 weight per cent. During a period of operation with low alpha alumina content, i.e., less than 15 per cent alpha, the

highest average recovery was 72 per cent of the theoretical production rate.

When the alpha alumina content of the product was high, the weight of undissolved solids leaving with the scrub was as low as 3.8 per cent of the theoretical production rate. When the alpha content was low, the weight of undissolved solids leaving with the scrub never averaged less than 5.2 per cent of the theoretical production rate.

The above results indicate that losses of solids to the off-gas stream due to high alpha alumina can be as low as, or lower than, losses occurring when the alpha content is low.

The comparison of the BMI and ICPP feed-introduction systems does not show conclusively a superiority of one over the other. The properties of the product from the limited Battelle tests compare favorably with the product properties from similar tests in the ICPP two-foot-square calciners. For runs in both calciners at 400°C with 1.3 M aluminum nitrate feed, the ranges of product properties were generally as follows:

Absolute density	3.5 to 3.6 g/cc
Nitrate content	0.5 to 0.6 millimole/gram
Bulk density	0.9 to 1.1 g/cc
Intra particle porosity . . .	0.4 to 0.45 void fraction

From the process viewpoint, there does not appear to be any advantage for the Battelle system over the ICPP system. Mechanically, the ICPP nozzles should be better for remote operation, since cleanout plungers for clearing the tips, should plugging occur, are easily installed. On the other hand, the velocity of the air through the ICPP nozzles is approximately 10-fold greater than that through the BMI feed gun, and erosion of the nozzle tips has caused considerable concern. If erosion cannot be eliminated by changes either in design or in materials of construction, the BMI system may prove the more desirable.

VII. ACKNOWLEDGEMENT

The editor gratefully acknowledges the efforts of G. R. Smithson, Jr., J. E. Hanway, Jr., and F. M. Stephens, Jr. of Battelle Memorial Institute, who directed the tests and whose report of their work under a research agreement with Phillips Petroleum Company constitutes the major part of this document.

APPENDIX A

DETAILED EXPERIMENTAL DATA

DETAILS OF RUN 1

The material used as a starter bed for Run 1 was produced by calcining simulated waste solution in the Phillips' two-foot-square calciner at the ICPP. At the beginning of Run 1, 397 pounds of this material, which had the following screen size distribution:

Cumulative Weight Per Cent

Mesh (Tyler)					
+14	+20	+28	+35	+65	+100
0.0	0.2	5.6	52.8	99.0	99.9

was used as a bed for starting up the Battelle calciner. This material had a bulk density of 60.5 pounds per cubic foot.

During the early stages of the run, approximately 75 pounds of material was elutriated from the calciner during startup. (This estimate of material lost from the unit is based on the amount of discharged product which was returned to the bed at this time in order to raise the level of the static bed to the same height that it was before the loss occurred.) The solid material which was recovered from the system during and at the conclusion of the run was as follows:

Discharge product (less 75 lbs returned to the bed)	417 lbs
Lost from bed during a startup	75
Remaining in the calciner at the end of Run 1	112
Total material recovered	604 lbs
Initial bed weight	397
Net gain	207 lbs

During this run, 520 gallons of solution containing 280 pounds of alumina was fed into the calciner. The retention of this material in the bed amounted to about 74 per cent of the total available solids fed into the calciner.

The material remaining in the calciner at the end of Run 1 had a bulk density of 67.6 pounds per cubic foot, and the following screen-size distribution:

Cumulative Weight Per Cent

Mesh (Tyler)					
+14	+20	+28	+35	+65	+100
0.2	0.2	1.4	27.6	82.8	88.2

The final bed material and some of the discharge products from Run 1 were mixed and used as a starter bed for Run 2.

The average daily experimental conditions for Run 1 and a table of samples which were taken together with the results of screen analyses and bulk density determinations are included in Tables A-1 and A-2 of this Appendix.

DETAILS OF RUN 2

Run 2 was begun with a starter bed prepared from the material recovered from the calciner during Run 1. This initial bed weighed 200 pounds, had a bulk density of 85.5 pounds per cubic foot and had a screen size distribution as follows:

Cumulative Weight Per Cent

Mesh (Tyler)					
+14	+20	+28	+35	+65	+100
0.0	0.0	0.7	14.1	47.6	51.9

During the third day of operation a brief shutdown became necessary, and while starting the unit up approximately 115 pounds of material was elutriated from the bed. (This estimate of material lost is based on the static bed level before and after the loss and on the bulk density of the material at the time of the loss.) In order to replace the material which was lost, 96 pounds of discharged product was returned to the calciner. The unit was operated for the remainder of the run without further losses except the normal carry over of dust in the exhaust gas stream.

During Run 2, material was discharged from the calciner as follows:

Phase 1	366 pounds
Phase 2	342 "
Phase 3	758 "
Phase 4	399 "
Phase 5	558 "
Total	2423 pounds

TABLE A-1. AVERAGE DAILY EXPERIMENTAL CONDITIONS FOR RUN 1

Date	Hours of Operation	Average Experimental Conditions: Temperature, °C Windbox	Bed	Superficial Velocity, fps (1)	Volume of Solution Fed, gals	Product Discharged, lbs
4-17	6	1020	450	1.07	30	19
4-18	22-1/2	1020	455	1.07	116	104
4-19	0	--	--	--	0	0
4-20	17-1/2	1065	455	1.07	103	57 (2)
4-21	18-1/2	1060	455	1.07	122	139 (2)
4-22	19-1/2	1050	455	1.07	149	173

(1) Calculation of superficial gas velocity does not take into consideration either water vapor or the products of nitrate decomposition.

(2) 75 lbs of discharge product returned to calciner after elutriating about this amount from the calciner during a startup.

TABLE A-2. DISCHARGE SAMPLE LOG AND RESULTS OF PHYSICAL-PROPERTY EVALUATION PROCEDURES FOR RUN 1

Date	Time Period	Sample No.	Weight, lbs	Total Elapsed Operating Time, hours (1)	Screen-Size Distribution, Weight Per Cent of Mesh			Bulk Density lbs/ft^3
					+20	-20 +65	-65	
4-22	1000 to 1300	18122-51-2	91	72	0.1	25.0	74.9	86.0
4-22	1400 to 1800	18122-51-3	67	76	0.0	21.4	78.6	77.0
4-22	1900 to 2200	18122-51-4	15	81	0.0	30.7	69.3	--

(1) Elapsed operating time to beginning of sampling period.

Taking into consideration the 115 pounds of material lost from the calciner during the previously described startup, the return of 96 pounds of product to the bed, and the 341 pounds [1] of material remaining in the calciner at the end of Run 2, a net gain of 2564 pounds was effected during the run. A total of 5316 gallons of simulated waste solution containing 3,296 pounds of alumina was fed into the calciner during this run; thus, the overall recovery of the alumina in the calciner was about 78 per cent of the total fed into the system.

The average daily experimental conditions, a list of the samples taken for evaluation, and the results of screen analyses and bulk density determinations for Run 2 are included in Tables A-3 and A-4 of this Appendix.

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(1) Includes 37 pounds of material adhering to the walls of the calciner at the end of the run.

TABLE A-3. AVERAGE DAILY EXPERIMENTAL CONDITIONS FOR RUN 2

Date	Hours of Operation	Average Experimental Conditions: Temperature, C Windbox	Bed	Superficial Velocity, fps(1)	Volume of Solution Fed, gal	Product Discharged lbs
			Phase 1			
4-25	23	945	440	1.1	125	--
4-26	18	1000	445	1.2	131	--
4-27	15	1035	445	1.2	95	70
4-28	24	1075	450	1.2	150	34
4-29	24	1100	450	1.2	179	75
4-30	24	1100	450	1.2	181	72
			Phase 2			
5-1	22	1100	440	1.2	167	85
5-2	23	1100	450	1.1	147	77
5-3	23	1100	445	0.9	148	74
5-4	24	1090	450	1.0	156	62
5-5	15	1090	450	1.0	95	44
			Phase 3			
5-5	6	1130	400	1.2	72	33
5-6	24	1210	395	1.2	344	129
5-7	24	1190	400	1.2	297	134
5-8	23	1200	395	1.2	309	142
5-9	24	1200	400	1.2	323	168
5-10	16	1200	395	1.2	209	115
5-11	8	1200	400	1.2	90	37
			Phase 4			
5-11	16	1200	395	1.2	210	91
5-12	24	1200	400	1.2	314	137
5-13	21	1200	400	1.2	270	156
5-15	3	1140	395	1.2	35	15
			Phase 5			
5-15	12	1190	400	1.2	151	70
5-16	24	1185	400	1.2	310	143
5-17	24	1165	395	1.2	325	131
5-18	24	1110	390	1.5	365	145
5-19	8	1090	395	1.6	118	69

(1) Calculation of superficial gas velocity does not take into consideration either water vapor or the products of nitrate decomposition.

TABLE A-4. DISCHARGE SAMPLE LOG AND RESULTS OF PHYSICAL-PROPERTY EVALUATION PROCEDURES FOR RUN 2

Date	Time	Sample Number	Total Elapsed Operating Time, hours	Screen-Size Distribution, Wt Per Cent of Mesh +20	-20 +65	-65	Bulk Density, lbs/ft^3
4/27	0400	18122-52-2	45	0.0	13.7	86.3	--
4/27	0800	18122-52-3	49	0.0	12.9	87.1	80.5
4/27	1200	18122-52-4	53	0.0	16.1	83.9	80.5
4/28	1200	18122-52-5	68	0.0	16.5	83.5	76.8
4/28	1300	18122-52-6	69	0.0	13.4	86.6	78.3
4/28	1500	18122-52-7	71	0.0	13.9	86.1	77.8
4/28	1700	18122-52-8	73	0.0	12.9	87.1	78.7
4/28	2000	18122-52-9	76	0.0	13.9	86.1	78.0
4/28	2400	18122-52-10	80	0.1	15.5	84.4	78.3
4/29	0400	18122-52-11	84	0.0	19.9	80.1	79.2
4/29	0800	18122-52-12	88	0.1	24.9	75.0	76.5
4/29	1200	18122-52-13	92	0.1	36.9	63.0	77.1
4/29	1600	18122-52-14	96	0.1	37.2	62.7	73.6
4/29	2000	18122-52-15	100	0.1	43.1	56.8	70.6
4/29	2400	18122-52-16	104	0.1	46.9	53.0	68.0
4/30	0400	18122-52-17	108	0.1	46.3	53.6	68.0
4/30	0800	18122-52-18	112	0.1	41.4	58.5	66.9
4/30	1200	18122-52-19	116	0.1	39.9	60.0	70.3
4/30	1600	18122-52-20	120				
4/30	2000	18122-52-21	124	0.1	38.3	61.6	67.7
4/30	2400	18122-52-22	128	0.1	33.0	66.9	63.0
5/1	0400	18122-52-23	132	0.1	32.0	67.9	63.2
5/1	0800	18122-52-24	136	0.1	30.2	69.7	63.8
5/1	1200	18122-52-25	140	0.1	40.9	59.0	63.6
5/1	1600	18122-52-26	144	0.1	33.9	66.0	63.7
5/1	2300	18122-52-27	149	0.1	25.5	74.4	63.9
5/2	0300	18122-53-1	153	0.1	31.5	68.4	64.8
5/2	0700	18122-53-2	157	0.1	39.9	60.0	62.0
5/2	1200	18122-53-3	162	0.1	39.7	60.2	60.2
5/2	1600	18122-53-4	166	0.2	41.7	58.2	59.1
5/2	2000	18122-53-5	170	0.1	37.4	62.5	59.6
5/3	0400	18122-53-6	176	0.2	40.0	59.8	58.3
5/3	0800	18122-53-7	180	0.2	39.9	59.9	58.0
5/3	1200	18122-53-8	184	0.2	38.4	61.4	57.8
5/3	1600	18122-53-9	188	0.1	40.2	59.7	58.5
5/3	2000	18122-53-10	192	0.1	44.7	55.2	56.4
5/3	2400	18122-53-11	196	0.2	39.5	60.3	54.3
5/4	0400	18122-53-12	200	0.3	34.6	65.1	53.6
5/4	0800	18122-53-13	204	0.3	36.2	63.5	54.6
5/4	1200	18122-53-14	208	0.3	34.9	64.8	56.0
5/4	1600	18122-53-15	212	0.6	35.3	64.1	56.3
5/4	2000	18122-53-16	216	0.4	32.0	67.6	56.0
5/4	2400	18122-53-17	220	0.4	32.8	66.8	55.4
5/5	0400	18122-53-18	224	0.4	28.5	71.1	54.8
5/5	0800	18122-53-19	228	0.3	24.9	74.8	55.5
5/5	1200	18122-53-20	232	0.2	25.2	74.6	55.5

TABLE A-4 (Continued)

Date	Time	Sample Number	Total Elapsed Operating Time, hours	Screen-Size Distribution, Wt Per Cent of Mesh +20	-20 +65	-65	Bulk Density, lbs/ft^3
5/5	1500	18122-53-21	235	0.3	19.4	80.3	56.3
5/5	2000	18122-53-22	238	0.1	20.9	79.0	56.7
5/5	2400	18122-53-23	242	0.1	21.4	78.5	58.7
5/6	0400	18122-53-24	246	0.1	20.1	79.8	62.4
5/6	0800	18122-53-25	250	0.1	19.7	80.2	63.7
5/6	1200	18122-53-26	254	0.3	21.7	78.0	63.8
5/6	1600	18122-53-27	258	0.1	25.3	74.6	65.4
5/6	2000	18122-53-28	262	0.3	25.6	74.1	66.0
5/6	2400	18122-53-29	266	0.2	25.0	74.8	66.5
5/7	0400	18122-53-30	270	0.3	30.1	69.6	67.1
5/7	0800	18122-53-31	274	0.1	25.4	74.5	67.4
5/7	1200	18122-54-1	278	0.2	30.9	68.9	67.0
5/7	1600	18122-54-2	282	0.1	32.3	67.6	66.6
5/7	2000	18122-54-3	286	0.4	52.7	46.9	66.3
5/7	2400	18122-54-4	290	0.7	46.7	52.6	66.1
5/8	0400	18122-54-5	294	0.7	45.6	53.7	67.3
5/8	0800	18122-54-6	298	0.9	44.3	54.8	67.5
5/8	1400	18122-54-7	303	1.0	41.2	57.8	67.1
5/8	1700	18122-54-8	306	1.0	48.4	50.6	67.3
5/8	2000	18122-54-9	309	1.1	51.8	47.1	67.2
5/8	2400	18122-54-10	313	0.7	49.4	49.9	65.3
5/9	0400	18122-54-11	317	0.7	50.0	49.3	65.5
5/9	0800	18122-54-12	321	0.6	44.2	55.2	66.7
5/9	1200	18122-54-13	325	0.9	52.8	46.3	66.7
5/9	1600	18122-54-14	329	0.6	60.3	39.1	66.5
5/9	2000	18122-54-15	333	0.7	53.3	46.0	66.1
5/9	2400	18122-54-16	337	0.5	54.2	45.3	66.8
5/10	0400	18122-54-17	341	1.1	58.8	40.1	66.9
5/10	0800	18122-54-18	345	0.8	55.8	43.4	67.4
5/10	1200	18122-54-19	349	0.6	56.7	42.7	67.8
5/10	1600	18122-54-20	353	0.6	55.0	44.4	68.0
5/11	0400	18122-54-21	356	0.9	56.5	42.6	69.0
5/11	0800	18122-54-22	360	0.9	58.4	40.7	66.1
5/11	1200	18122-54-23	364	0.8	62.5	36.7	71.6
5/11	1600	18122-54-24	368	1.6	64.1	34.3	72.7
5/11	2000	18122-54-25	372	1.9	71.0	27.1	73.6
5/11	2400	18122-54-26	376	2.0	73.5	24.5	74.5
5/12	0400	18122-54-27	380	2.7	76.4	20.9	75.2
5/12	0800	18122-54-28	384	3.2	80.5	16.3	75.0
5/12	1200	18122-54-29	388	3.1	78.5	18.4	75.6
5/12	1600	18122-54-30	392	3.4	82.9	13.7	76.0
5/12	2000	18122-54-31	396	3.9	85.6	10.5	76.2
5/12	2400	18122-55-1	400	2.5	87.2	10.3	76.2
5/13	0400	18122-55-2	404	4.6	86.8	8.6	75.9
5/13	0800	18122-55-3	408	3.4	89.8	6.8	76.4
5/13	1200	18122-55-4	412	3.8	90.1	6.1	76.3
5/13	1600	18122-55-5	416	3.7	90.2	6.1	76.7
5/13	2000	18122-55-6	420	4.6	89.3	6.1	75.2

TABLE A-4 (Continued)

Date	Time	Sample Number	Total Elapsed Operating Time, hours	Screen-Size Distribution, Wt Per Cent of Mesh +20	-20 +65	-65	Bulk Density, lbs/ft^3
5/13	2300	18122-55-7	421	5.6	88.8	5.6	74.4
5/15	1200	18122-55-8	424	4.7	88.7	6.6	75.5
5/15	1600	18122-55-9	428	4.9	88.9	6.2	74.1
5/15	2000	18122-55-10	432	5.5	89.7	4.8	73.3
5/15	2400	18122-55-11	436	5.6	89.6	4.8	71.5
5/16	0400	18122-55-12	440	11.8	86.4	1.8	71.2
5/16	0800	18122-55-13	444	8.4	88.3	3.3	70.5
5/16	1200	18122-55-14	448	14.2	83.0	2.8	69.6
5/16	1600	18122-55-15	452	19.2	79.2	1.6	68.5
5/16	2000	18122-55-16	456	20.1	75.5	4.4	68.3
5/16	2400	18122-55-17	460	29.1	67.3	3.6	--
5/17	0400	18122-55-18	464	27.5	70.8	1.7	66.5
5/17	0800	18122-55-19	468	31.1	66.3	2.6	66.7
5/17	1200	18122-55-20	472	47.8	50.7	1.5	64.0
5/17	1600	18122-55-21	476	56.7	42.2	1.1	63.9
5/17	2000	18122-55-22	480	55.9	40.0	4.1	63.6
5/17	2400	18122-55-23	484	63.5	35.5	1.0	62.5
5/18	0400	18122-55-24	488	65.9	34.0	0.1	62.4
5/18	0800	18122-55-25	492	75.2	24.4	0.4	61.8
5/18	1200	18122-55-26	496	63.4	35.6	1.0	64.0
5/18	1600	18122-55-27	500	56.5	40.9	2.6	67.8
5/18	2000	18122-55-28	504	69.2	30.5	0.3	65.9
5/18	2400	18122-55-29	508	60.1	38.3	1.6	65.8
5/19	0400	18122-55-30	512	55.0	44.3	0.7	68.4
5/19	0800	18122-55-31	516	51.9	45.8	2.3	67.0

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