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Alumina Miniplant Operations—Overall Mass Balance for Clay-HCl Acid Leaching

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

Bé	Baumé	lb/ft^3	pound per cubic foot
° C	degree Celsius	lb/hr	pound per hour
deg	degree	m^2/g	square meter per gram
ft	foot	mol pct	mole percent
ft^2	square foot	pct	percent
ft^3	cubic foot	ppm	part per million
gal	gallon	rpm	revolution per minute
g/mL	gram per milliliter	tpd	ton per day
gph	gallon per hour	vol-pct	volume-percent
hr	hour	wt-pct	weight-percent
in	inch	yr	year

ALUMINA MINIPLANT OPERATIONS–OVERALL MASS BALANCE FOR CLAY-HCl ACID LEACHING

By Dwight L. Sawyer, Jr.,[1] Theodore L. Turner,[2] and Don B. Hunter[3]

ABSTRACT

To provide technology that can help decrease the dependence of the U.S. aluminum industry on imported bauxite, the main ore of aluminum, the Bureau of Mines is investigating the extraction of aluminum from abundant domestic reserves of kaolinitic clay. Miniplant studies of the continuous leaching of calcined kaolin with boiling 25-pct HCl acid resulted in a 93- to 99-pct extraction of the acid-soluble alumina in the calcined kaolin feed.

Data are presented for an acid leaching study carried out in three or four continuously stirred tank reactors (CSTR) in series, feed rates of 100 to 250 lb/hr of calcined kaolin, and total nominal residence times of 0.7 to 2.3 hr. A material balance for a 176-lb/hr calcined kaolin feed rate in a circuit consisting of three reactors, four classifiers, and five thickeners is given. Data provide detailed compositional and physical information for calcined kaolin, process liquors, and acid-leached residue solids.

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INTRODUCTION

The United States, the world's largest producer and consumer of aluminum, imports approximately 93 pct of the raw materials necessary to support its aluminum metal industry. Aluminum is presently produced from bauxite, of which the United States has limited reserves. To provide technology that might help lessen this dependency on foreign sources, the Bureau of Mines began an alumina miniplant project in July 1973 to test and develop the most promising technologies for extracting alumina from domestic nonbauxitic resources. The aluminous materials selected as alternate sources of alumina were clay, anorthosite, alunite, dawsonite, and coal ash and coal shale. In July 1974, a cooperative Bureau of Mines-industry alumina miniplant program was initiated to promote the development of technology for the extraction of alumina from these resources.

While the miniplant project was in progress, Kaiser Engineers (1-2),[4] under contract to the Bureau of Mines, evaluated six processes on the basis of available data. The clay-HCl acid process (clay-HCl process), incorporating HCl gas-induced crystallization of $AlCl_3 \cdot 6H_2O$, was found to have the lowest capital and operating costs. Technology of the clay-HCl process had been under investigation at the Bureau's Boulder City (NV) Engineering Laboratory on a miniplant scale since 1975. The raw material used was kaolinitic clay, or kaolin, a material that has a high alumina content and occurs in large, relatively pure deposits in several eastern States.

The design, construction, and testing programs of the clay-HCl miniplant occupied a 6-yr period, 1975 to 1980 inclusive, and was the major activity in the Bureau of Mines alumina miniplant project. This report provides process information about the HCl leaching of calcined kaolin and presents detailed compositional data and a material balance for various liquors and solids in the process flowsheet.

THE BUREAU OF MINES ALUMINA MINIPLANT PROJECT

Bureau of Mines investigations on the extraction of alumina from domestic nonbauxitic resources began in the 1920's. A pilot plant for the extraction of alumina from anorthosite was operated in Wyoming after the end of World War II (24), and prior work had been done during the 1940's (4). During the period 1942 to 1945, investigation of a process to produce alumina by leaching clay with HCl acid was undertaken by the National Bureau of Standards (8). With the exception of solvent extraction, that flowsheet is similar to that of the miniplant. In the early 1960's the Anaconda Copper Co. (6) operated a 6-tpd alumina pilot plant in Montana, using a patented process similar to that of the Bureau of Mines, except that the $AlCl_3 \cdot 6H_2O$ was crystallized by evaporation.

From 1962 to 1969, 20 cost evaluations of proposed alumina extraction processes were published (7-10, 18-23), and were revised and updated in 1974; a cost estimate of the conventional Bayer process was included for comparison (17). A study in 1970 by the National Materials Advisory Board, "Processes for Extracting Alumina From Nonbauxite Ores," recommended clay as the most promising ore, with extraction by either nitric or HCl acid (12). The alternative methods of extracting alumina from domestic resources received impetus from political unrest in countries which traditionally supplied bauxite ore for refining and smelting in this country. In early 1971, Guyana nationalized Alcan's bauxite

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[4] Underlined numbers in parentheses refer to items in the list of references preceding the appendix.

mines. In 1974 the International Bauxite Association was formed and posed a threat similar to that of the Organization of Petroleum Exporting Countries. Jamaica, and subsequently other bauxite producers, drastically raised bauxite mining taxes. A continuation of these events would make an alternative domestic nonbauxitic source of alumina an attractive, secure option.

In July 1973, the Bureau of Mines began an alumina miniplant project based on past Bureau work (17) and on information in the literature. The initial plans were to construct and operate 25-lb/hr alumina miniplants using the following technologies:

1. Clay-HNO_3 leaching.
2. Clay-HCl leaching, with evaporative crystallization.
3. Clay-sulfurous acid leaching.
4. Anorthosite-lime sinter, caustic leaching.
5. Alunite-reduction roasting, caustic leaching.
6. Dawsonitic oil shale, retorting and dilute caustic leaching.

Subsequently, clay-HCl leaching, with HCl-gas induced crystallization was added as a result of a suggestion from one of the cooperating firms. Alunite investigations were sharply cut back when it was learned that a commercial pilot plant study of the process was underway. Dawsonite investigations were deferred pending completion of a Bureau of Mines experimental mine shaft, from which tonnage samples of representative material could be obtained.

Experience with the first clay-HNO_3 miniplant indicated that a calcined kaolin feed rate of at least 100 lb/hr was desirable, so that process data could be more reliably translated to industrial design. The clay-HCl miniplant was designed for a feed rate of 60 to 250 lb/hr of calcined kaolin, and built with standard commercial equipment. The acid leaching, solids-liquid separation, and solvent extraction circuits were operated to test the equipment, install improvements and modifications, and produce a purified pregnant liquor for crystallization of $AlCl_3 \cdot 6H_2O$ and decomposition to alumina.

In mid-1976 investigations were begun at several of the Bureau's research centers to provide background chemical and physical data needed for the miniplant, but not available in the literature, in order that the process engineering studies in the miniplant might proceed in a timely manner.

CYCLIC PROCESS OF THE CLAY-HCl ACID MINIPLANT

Early emphasis in the clay-HCl miniplant was placed on demonstrating process feasibility on a substantial scale of operation, but the principal aim of the project was to furnish reliable process and design data for decisionmaking on possible scale-up. Process data resulting from the project provided a substantial part of the information used in designing a 25-tpd alumina-from-kaolin pilot plant (3). Technology of the cyclic extraction process has been divided into several unit operations (fig. 1). This report summarizes information relating to four of these operations.[5]

Kaolin Feed Preparation

A typical eastern Georgia kaolin suitable for the production of alumina was

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[5] The sections describe alumina miniplant operations at the time of this work; improved feed preparation techniques and fluidized-bed calcination were developed during the project, and are the subject of separate reports.

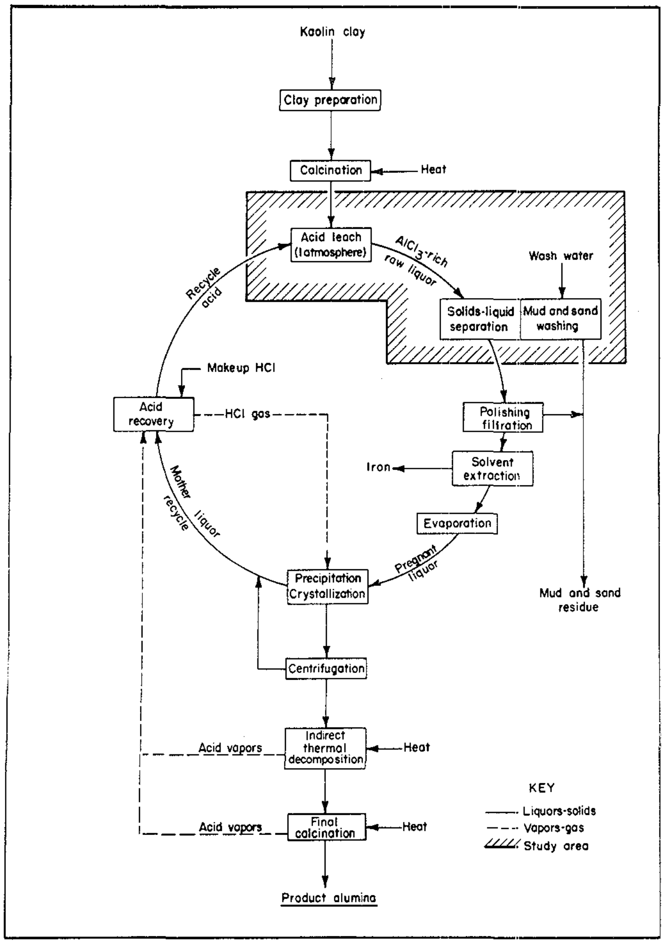

FIGURE 1. - Unit operations of the clay-HCl acid process.

obtained from the Thiele Kaolin Co., Sandersville, GA. In the miniplant, the raw kaolin was withdrawn from storage and crushed to approximately minus 0.5-in size; minus 20-mesh fines were screened out, pelletized, and partially air-dried. The crushed material was blended with pellets and stored in steel drums pending calcination.

Calcination of Kaolin

Kaolinite, $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$, the major mineral in kaolin, is attacked slowly by mineral acids, but if it is heated to about 600° to 900° C, the combined water is driven off, and the resulting material, termed "metakaolin" ($2Al_2O_3 \cdot 4SiO_2$) (13), is readily attacked by hot mineral acids. Kaolin was calcined as required in a 2.5- by 24-ft direct, gas-fired rotary kiln at 750° C. About 30,000 lb of kaolin was normally calcined during each campaign, to produce sufficient calcined kaolin for subsequent leaching operations.

HCl Acid Leaching of Calcined Kaolin

Calcined kaolin was crushed and screened to size as required, and leached at 112° C with a slight excess of boiling 25-pct HCl acid in three glass-lined, 50-gal, CSTR's arranged in cascade (fig. 2). The jacketed reactors were heated by a Therminol[6] 66 hot oil heating system with individual throttling valves for additional control of each reactor, and were stirred by a glass-coated retreating curve impeller with three arms. Limited baffling, which later was recognized as inadequate, was provided by one H-baffle. Each reactor was equipped with a 4-in-diam (7.6 ft^2) glass reflux condenser. An additional 6-in-diam reflux condenser (12.8 ft^2) was installed on the first reactor to absorb the exothermic heat of the leaching reaction, the major part of which occurred in reactor 1.

Slurry discharged from each reactor through a 2-in-diam nozzle located in the sidewall of the reactor body. The freeboard space above the slurry surface was needed to contain foam produced during the exothermic leaching reaction. Three-way valves were installed between reactors so that complete slurry stream samples could be taken (fig. 3). The nominal working volume of each reactor was approximately 23 gal (fig. 4).

Water and 20 Bé HCl were measured by diaphragm metering pumps and mixed to produce a nominal 25-pct HCl solution. Calcined kaolin stored in drums was

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[6] Reference to specific brand names or manufacturers does not imply endorsement by the Bureau of Mines.

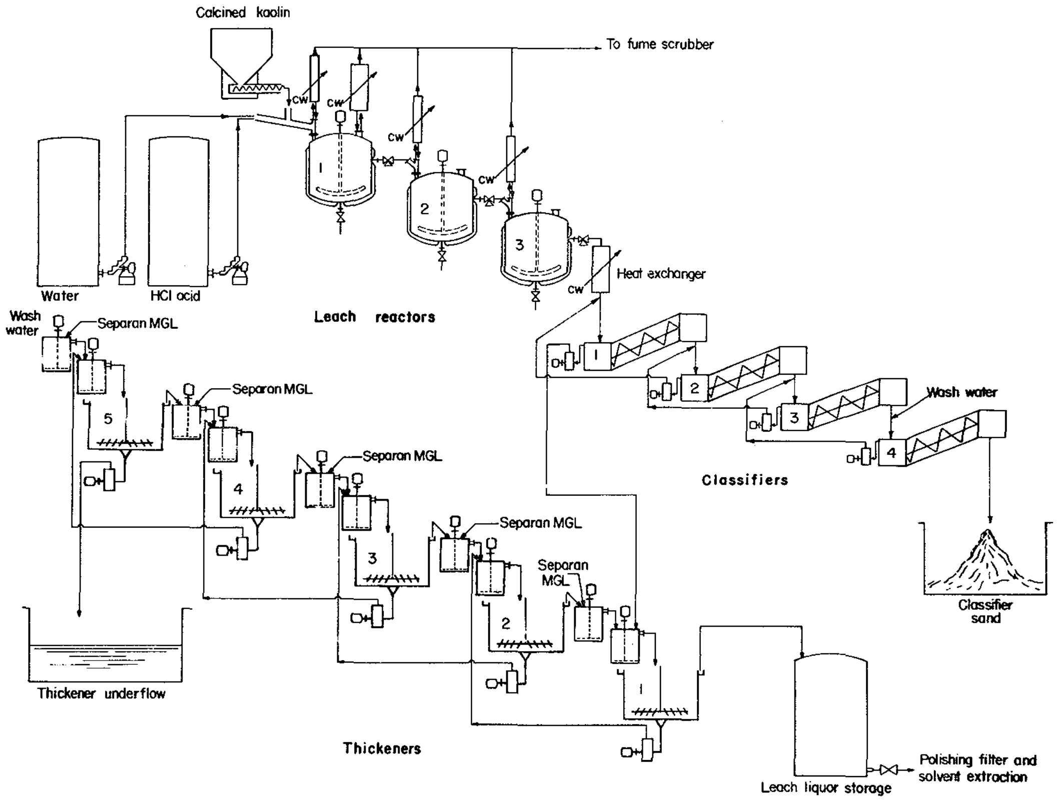

FIGURE 2. - Flowsheet and major equipment of the acid leaching and solids-liquid separation circuits.

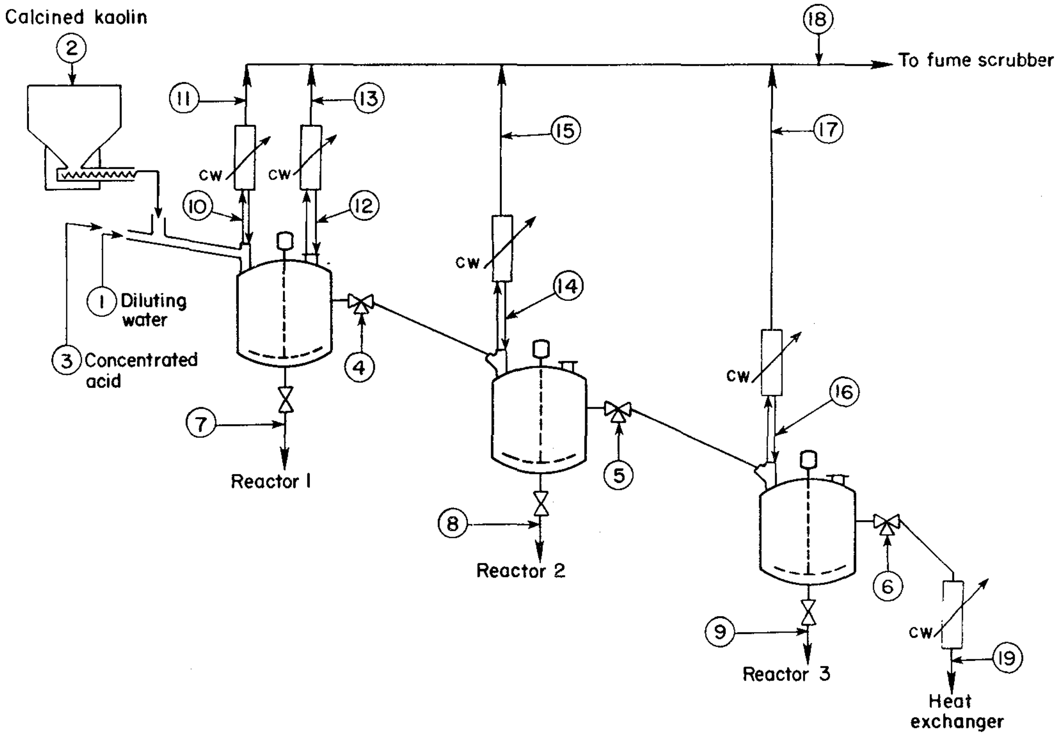

FIGURE 3. - HCl acid leaching circuit: flowsheet with sample points.

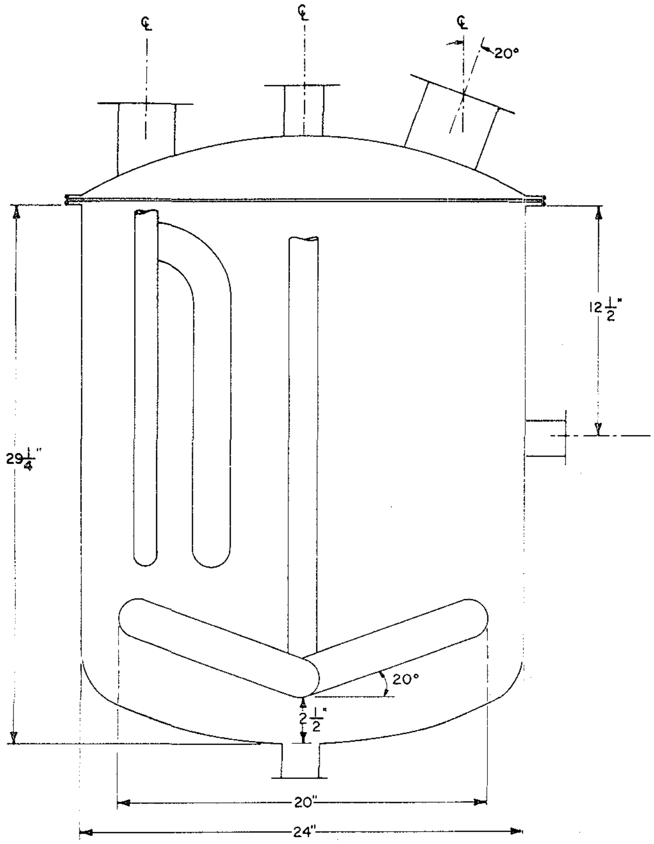

FIGURE 4. - Vertical section of 50-gal, glass-lined reactor showing side overflow nozzle, H-baffle, and retreating-curve impeller.

poured into the feed hopper of a belt conveyor (fig. 5) that automatically filled the bin of the Acrison feeder, which was equipped with a digital totalizer. Calcined kaolin from the hopper was discharged by means of a 1.9-in-diam screw feed tube and poured into reactor 1 via an inclined feed chute that was flushed with the water and acid feed streams.

HCl losses from the reactor condensers to the miniplant fume scrubber were calculated from Lear-Siegler isokinetic gas sample analyses. The isokinetic sampling technique was satisfactory because the heated sample probe ahead of the three Na_2CO_3-solution absorbers prevented condensation of moisture. Sample volumes were 10 to 20 ft^3 at standard conditions. HCl collected in the absorber solutions was determined by Volhard titration.

Leaching dissolved most of the acid-soluble alumina and most of the soluble iron and other impurities present in the calcined kaolin. The SiO_2 and the TiO_2 were present in the porous inert residue.

Solids-Liquid Separation

Any significant change in the operation of the leaching section affected the operation of the solids-liquid separation section, and prolonged periods of operation were required before a steady-state condition was established. Flow measurement and sample data, from which the overall material balance for the leaching and solids-liquid separation sections was constructed, were selected from those taken during steady-state periods. Detailed mechanical performance descriptions and material flows within the separate circuits are not included in this report.

Acid leach slurry at a temperature of 112° C from reactor 3 was cooled in a heat exchanger to about 60° C, a temperature below the maximum service-rated temperature of the elastomeric linings used in the mild steel spiral classifiers and three of the five thickeners of the solids-liquid separation section.

Cooled slurry from the heat exchanger flowed by gravity to classifier 1. At the 175-lb/hr feed rate, the minus 100- plus 150-mesh particles (median size of 115 mesh) split equally between the classifier sands and the thickener feed. The overflow from classifier 1, consisting of diluted leach liquor and fine solids, was pumped into a small mixing tank on top of thickener 1. A 0.2-pct solution of Separan MGL, a nonionic polyacrylamide flocculant, diluted by the overlow liquor from thickener 2 was added to enhance settling and compaction of the fines, and the treated pulp flowed into the center feed well of thickener 1. Flocculant treatment of the thickener circuit was critical to the operation. The underflow pulp from thickener 1 was advanced to the

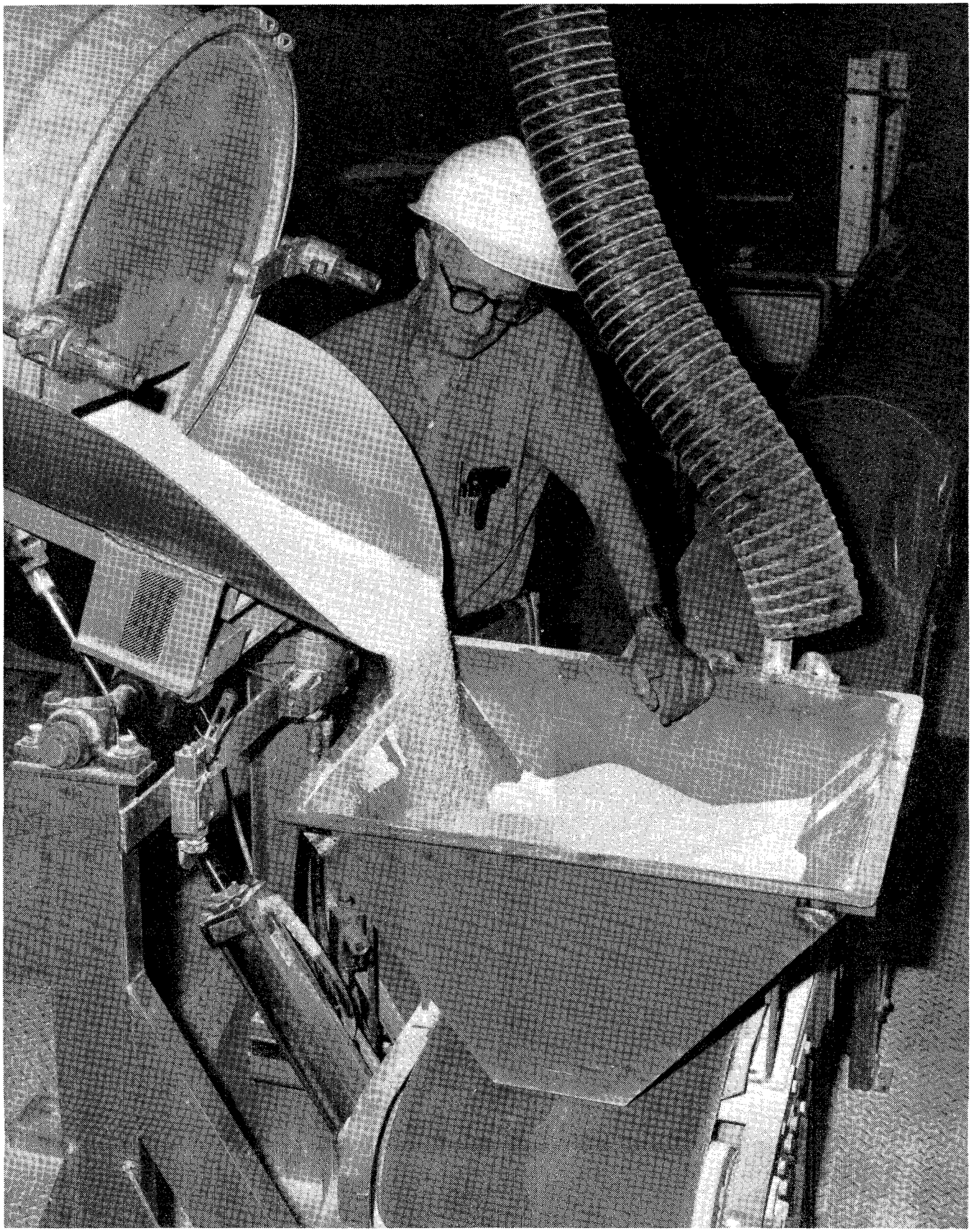

FIGURE 5. - Charging calcined kaolin to an Acrison weightometer feed conveyor.

thickener 2 feed mixing system, where it was dispersed in overflow from thickener 3 fortified with a dilute solution of Separan MGL to produce the feed pulp for thickener 2. This process was continued through the five-thickener circuit (fig. 6). At thickener 5 the underflow pulp contained about 25 pct solids, and was discharged as a slimes tailing. A timer-operated, diaphragm-suction pump was used to discharge the underflow pulp, resulting in irregular flow.

The estimated time of passage of the fine solids through the thickeners (114 hr) was based on a fine solids feed rate to thickener 1 of about 25 lb/hr. Because of the considerable time lag for the travel of fine solids through the five thickeners, it was decided to sample the slime solids contained in the thickener 1 feed stream (from classifier 1 overflow), and use these samples to construct the material balance, instead of thickener 5 underflow material. The

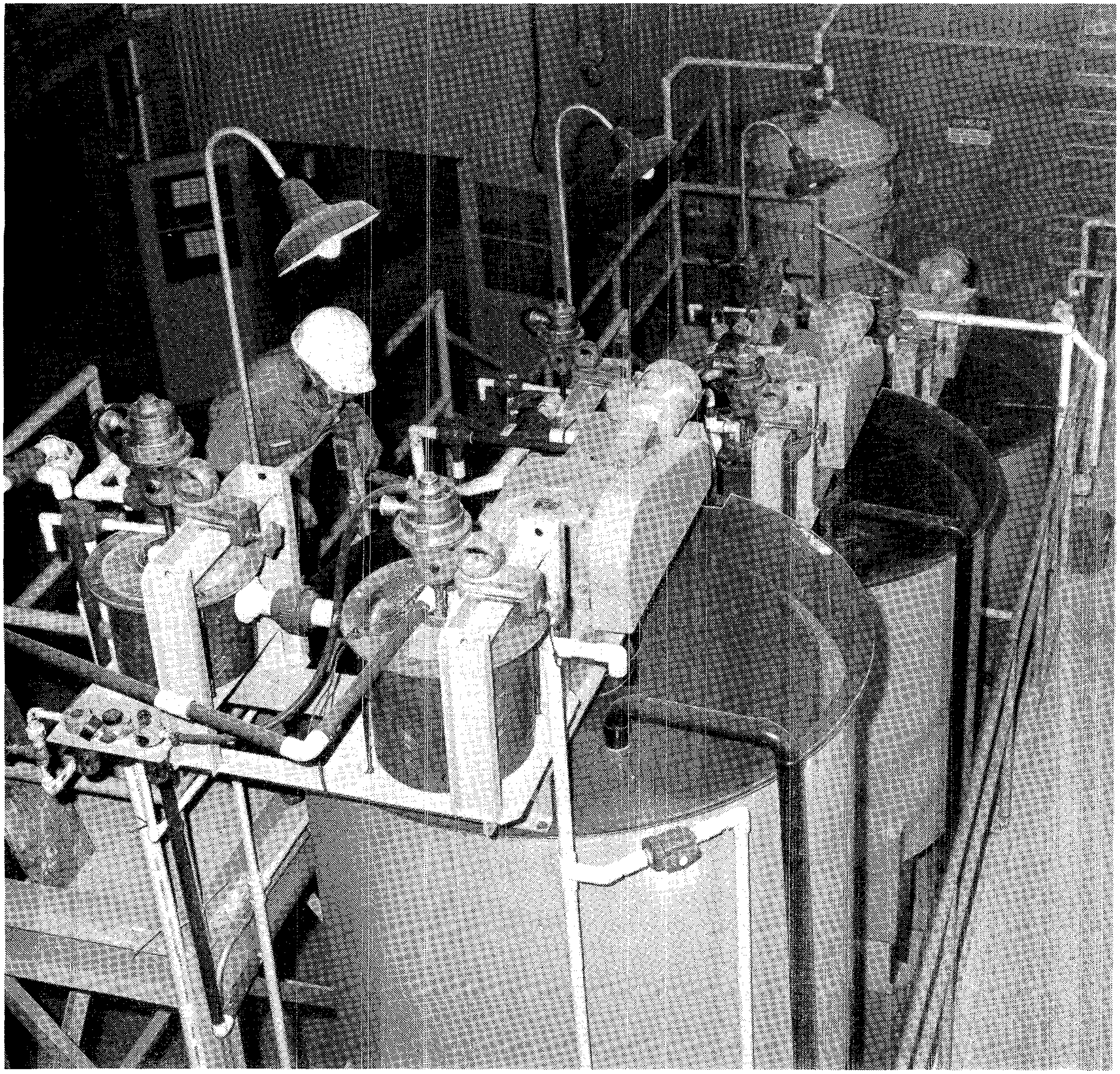

FIGURE 6. - Rubber-lined thickeners with flocculant mixing and dispersing vessels.

solids in the thickener 1 feed stream were assumed to be completely leached and at a temperature of 50° C, and were considered the equivalent of thickener 5 underflow solids for the material balance.

Thickener pulp data (table A-2) represent the averages of data obtained during steady-state conditions of runs 4 and 5. In run 5, thickener 5 underflows exceeded 28 pct solids at times and were viscous and difficult to handle with the small diaphragm-suction pumps. Therefore, the averaged pulp solids contents, though conservative, are considered satisfactory for preliminary process design.

The coarse sands from classifier 1 were transported on a conveyor belt to classifier 2, countercurrently washed, and flowed by gravity through classifiers 3 and 4. The washed sands, containing about 52 pct solids (table A-22), were discharged as waste from classifier 4.

Two sizes of washed residues, a coarse sand from the classifiers and an underflow pulp from thickener 5, were produced. Raw pregnant liquor containing $AlCl_3$ and dissolved impurities, mostly iron (table A-15), overflowed from thickener 1 to the raw pregnant liquor storage tank and was held for polish filtration before further processing.

EQUIPMENT AND OPERATION OF THE MINIPLANT

CHEMICAL REACTORS

Factors affecting the ability of the CSTR train to provide a process environment for the efficient extraction of acid-soluble alumina from calcined kaolin were (1) an adequate heat source to maintain vigorous boiling (oil-heated jackets), (2) sufficient acid to satisfy stoichiometry of the reactions, (3) effective agitation for solids suspension (solids-liquid contact), and (4) sufficient residence time (reaction rate requirement) (14-15).

Reactors were operated under conditions of vigorous boiling with full reflux of acid condensate from glass coil condensers (fig. 2). Temperatures increased slightly along the reactor train, indicating an increase in the concentration and boiling point of the aluminum chloride solution being produced. Under prevailing atmospheric pressures at the laboratory (elevation 2,500 ft), temperatures ranged from about 107° to 112° C. Temperatures were manually regulated to sustain boiling rates that produced visible condensate films on condenser coils covering about 40 pct of the total surface. Under these conditions, reserve condensing area was available and adequate freeboard above the boiling reactor contents was about 10 to 20 vol-pct. Although not serious, conditions of unstable frothy boiling were most frequently noticed in reactor 1.

The mechanical operation of the 50-gal reactor train throughout major miniplant campaigns, with few exceptions, was uniform and trouble free. However, run 5 conclusively established that the reactors did not provide adequate suspension for the larger particles of a 10-mesh feed and were unsuitable for 8-mesh feed. Ineffective baffling coupled with a 100-pct suspension requirement for slurry overflow allowed a centrifugal classification and the accumulation of heavier particles against the sidewalls and in the lower regions of the reactors. Autogeneous grinding took place within these regions and undesirable fines were produced. The problem was corrected during a later study.

HIGHLIGHTS OF PRIOR OPERATIONS

The clay-HCl miniplant acid leaching and solids-liquid separation circuits evolved as a result of experience with equipment and mode of operation during successive leaching campaigns. At the stage of development reported here, simultaneous operation of two circuits consisting of a three-reactor leaching train and a four-classifier, five-thickener solids-liquid separation system, produced a satisfactory raw pregnant liquor. Continuous vacuum filtration of the acid leaching slurry was investigated later in the miniplant project. A brief summary of early operations (table A-4) will aid in understanding the major 10-day campaign from which the majority of data and the material balance of this report have been derived.

Run 1

Operation of a four-reactor leaching system began with a minus 20-mesh calcined kaolin feed and 20-pct HCl and continued for 51 hr. Steady-state conditions were attained shortly after startup and operation at the 60-lb/hr (minimum)

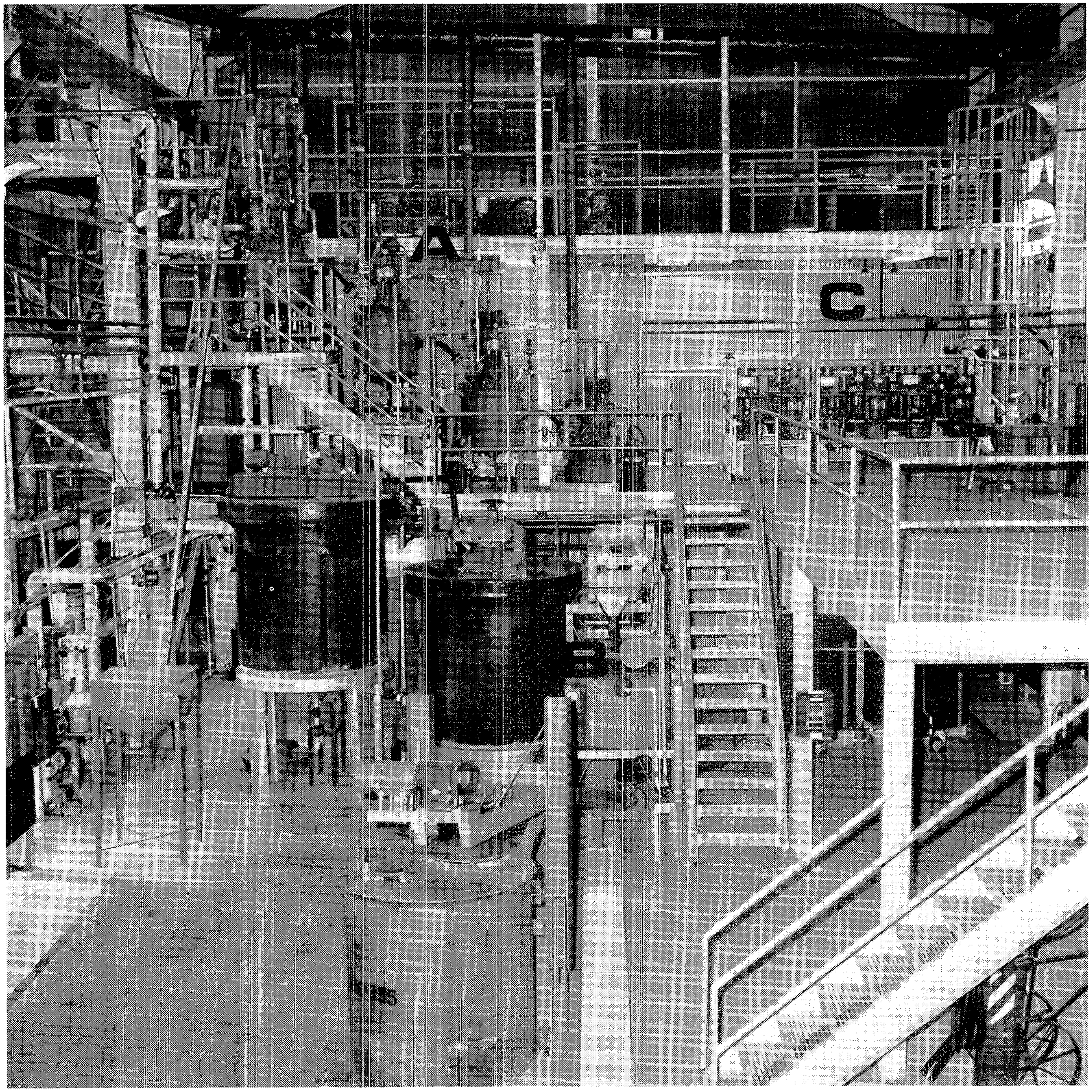

FIGURE 7. - Early miniplant equipment arrangement. The acid leaching reactors (A) are shown upper left; the two-classifier, two-thickener solids-liquid separation circuit (B) mid-center; and solvent extraction (C) mid-right.

design rate was continued for a period of 38 hr. The feed rate was doubled to 120 lb/hr and steady-state conditions were maintained until the calcined kaolin feed was exhausted 13 hr later. Limited heat and material balance data were obtained.

Run 2

The objective of integrating (fig. 7) acid leaching, solids-liquid separation (two screw classifiers and two thickeners), liquor filtration, and solvent extraction was accomplished. Feed rates of 100 and 150 lb/hr of minus 20-mesh calcined kaolin were attained and 67 hr of stable operation of the miniplant resulted. In both runs 1 and 2 excessive fines in the feed hindered classification of a sand product in classifier 1 (table A-3).

Run 3

Data and experience from run 2 were employed to improve operations. The high-precision Acrison weight-loss feeder was "locked-in" at a 100-lb/hr calcined kaolin feed rate, and the leaching section operated uniformly for 99 hr. During the operation, 44 hr of steady-state conditions in the solids-liquid separation system were achieved. Data and sample acquisition were extensive, and the distribution of important minor impurities in the process was investigated. Feed to the reactors was changed from minus 20- to minus 10-mesh size (table A-3) to improve the operation of the spiral classifiers and to facilitate crushing and screening of calcined kaolin. The production of minus 10-mesh feed entailed crushing and sizing a mixture of calcined minus 4- plus 20-mesh crushed kaolin and pellets. This feed material was structurally weak and consisted largely of irregularly shaped particles with sharp, jagged edges and rough surfaces, which were easily abraded during all phases of handling. Particles suffered severe degradation on passing through processing circuits and large quantities of undesirable fines were produced (fig. 8). The true significance of reactor design relative to this characteristic became apparent as extensive data from run 4 were being analyzed and plans for run 5 were being made.

Run 4

A 10-day campaign was undertaken to operate and evaluate the leaching and solids-liquid separation circuit, which had been expanded from two classifiers and two thickeners to four classifiers and five thickeners. The minus 10-mesh calcined kaolin feed rate was 100 lb/hr. The thickener underflow pulps ranged from approximately 11 to 18 pct solids. Simultaneous bench-scale settling tests showed that the underflow pulp solids

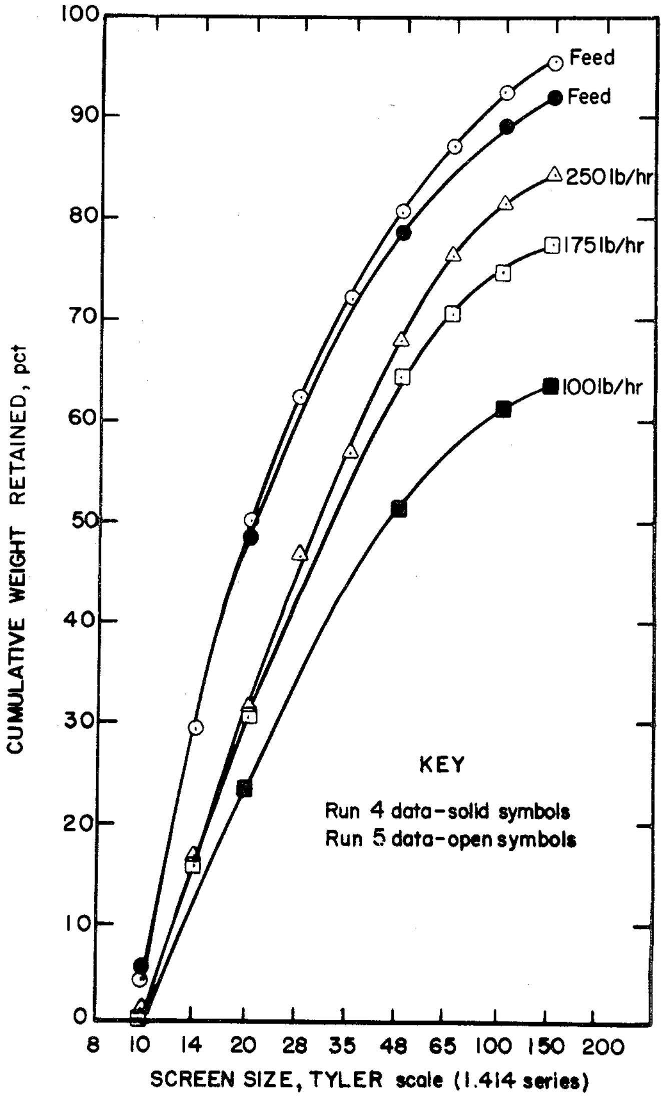

FIGURE 8. - Particle size distribution of minus 10-mesh calcined kaolin feeds compared with respective acid-leached residues as a function of reactor residence time (feed rate).

could be increased to 14 to 26 pct if adequate residence time was provided. One reactor failed because of corrosion and loss of the agitator and was removed from service. Test coupons for evaluating the corrosion resistance of different materials of construction were in place during this operation (5).

PLANNING AND CONDUCT OF RUN 5

Run 5 of the clay-HCl leaching and solids-liquid separation sections entailed running minus 10-mesh crushed calcined kaolin feed to the reactors at an initial rate of 175 lb/hr. Previous runs had not exceeded a 150-lb/hr rate. If no problems were encountered at the 175-lb/hr rate, minus 8-mesh feed would be used for the rest of the run to determine if coarser feed would decrease the fines load on the thickening circuit. Performance of the mechanical circuits as a function of calcined kaolin feed particle size was extensively studied, but details are not reported. It was planned to increase the feed rate to 250 lb/hr--the maximum rate of the Acrison weigh feeder--for the last 24 hr of the run in order to evaluate the three-reactor train and other equipment under conditions of high loading.

Reactor 1 was first filled with 25-pct HCl and heated to incipient boiling. Then calcined kaolin feed was started and sluiced into the reactor with cold 25-pct HCl. The exothermic reaction began rapidly, the boiling slurry overflowed into heated reactor 2 and, shortly thereafter, into reactor 3. This procedure was developed to avoid boilovers, or runaway reactions at startup.

Because the leaching and solids-liquid separation circuits operated smoothly, when the minus 10-mesh calcined kaolin supply was exhausted the minus 8-mesh feed was started. During the next 8 hr, reactor operation was plagued by a stable foam that rose into the reflux condensers. Foam was suppressed by the addition of an antifoaming agent and the use of minus 8-mesh feed was continued. As the run progressed, serious problems developed in the solids-liquid separation circuit. With the minus 10-mesh feed, three-quarters of the leached solids reported to the classifiers, and only one-quarter to the thickeners. However, with minus 8-mesh feed, an increasing quantity of unclassified coarse solids were suspended in a thick viscous pulp that overflowed from the first classifier, until more than two-thirds of the leached solids were going to the thickeners. The autogenous grinding of settled coarse particles, mostly in reactor 1, generated fines, which stabilized foam in the reactors. The presence of a large quantity of fines in the reactor discharge slurry increased the effective viscosity and suspending property of the slurry, and prevented classification of the sands. For these reasons, and because previous operations using finer sized feed had been without foaming and classification problems, the remaining 8-mesh feed was ground to minus 10 mesh (fig. 9). Operations were resumed using the minus

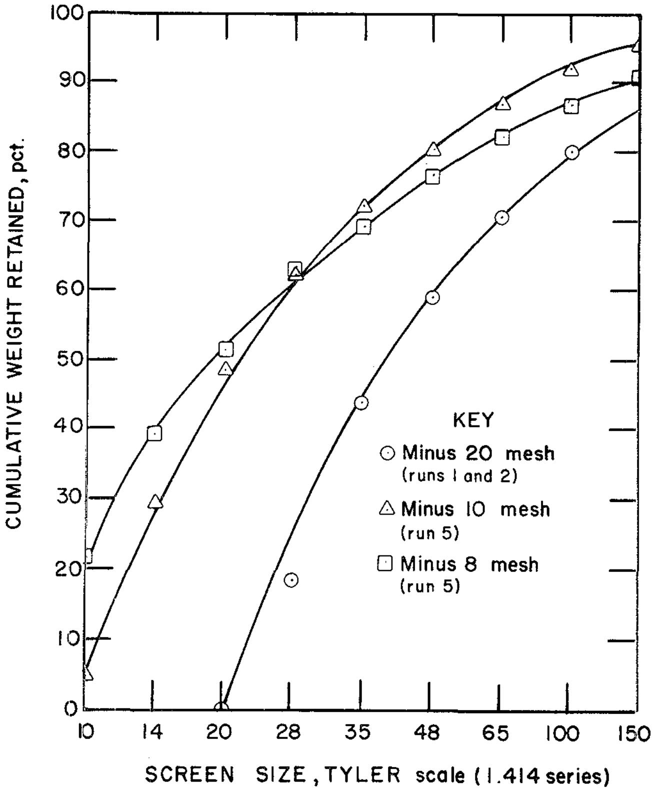

FIGURE 9. - Particle size distribution of three calcined kaolin feeds processed in acid leaching reactors of the alumina miniplant.

10-mesh calcined kaolin feed and addition of the antifoam agent was discontinued. Except for normal froth and bubbles above the surface of the boiling slurry in the reactors, foaming ceased and classifier operation returned to normal.

No further problems were encountered during the remainder of the run at the 175-lb/hr rate, and the leaching and solids-liquid separation sections were operated under steady-state conditions for 60 hr. On completion of the run, a 59-hr data collection period was selected to form the base for the material balance study.

The feed rate was increased to 225 lb/hr for slightly more than 5 hr. When no operating difficulties were experienced, feed was increased to 250 lb/hr for the last 20 hr of the run (feed rate data are shown in table A-1). The objective of the higher feed rate was to determine how an increased processing rate would affect the leaching capabilities of the three 50-gal reactor circuit and operation of the four-classifier, five-thickener, solids-liquid separation circuit. At higher feed rates, particle residence time in the reactors was decreased, short circuiting was increased, and the larger feed particles were not fully leached. Incomplete leaching was detected as a decline in the dissolved alumina content of the reactor 3 discharge slurry. Leach liquor Al_2O_3 analyses at the 250-lb/hr rate averaged 9.55 pct, compared to 9.98 pct Al_2O_3 for the 175-lb/hr rate (table A-5). The Al_2O_3 content of the leached classifier sand solids increased from 5.2 to 6.99 pct.

MATERIAL BALANCE

The boundary for the material balance was arbitrarily constructed around the three acid leaching reactors, the four spiral classifiers, and the five thickeners because the process trains were operated, monitored, and sampled simultaneously as an interconnected unit. The combined operation produced a product, the raw pregnant liquor, that was feed for all subsequent process studies[7] in the clay-HCl miniplant.

In order to construct the material balance, it was necessary to normalize the chemical analyses of major solids streams. This could have been done by factoring, but since silica was the major component of the solids and some problems had been associated with the reported analyses, the total corrections were made on the silica analyses. Principal material balance controls were the weight and composition of the calcined kaolin feed, analyses of the classifier 1 overflow solids (thickener feed solids) and classifier 4 discharge solids, and the composition of the thickener 1 overflow liquor (raw pregnant liquor, table A-15).

The solids in the classifier 1 overflow stream (thickener 1 feed) were assumed to be compositionally equivalent to thickener 5 underflow solids. Since accurate measurement of the discharge of thickener 5 underflow pulp was difficult, the combined total TiO_2 and SiO_2 (adjusted value) content of the classifier 1 overflow feed solids was designated "insolubles" and served as a tracer to develop the weight of the thickener 5 underflow. The average weight rate of the classifier 4 sands and complete chemical analyses provided enough information to permit calculation of the weight of the thickener 5 underflow. Coupled with the chemical analyses and weight of the liquid fractions of these streams, an alumina accountability was obtained. In turn, the weight of the pregnant liquor was

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[7] Subsequent operations conducted in the miniplant to test individual circuits, or to operate and evaluate unit operations, such as solvent extraction, evaporation, and crystallization of $AlCl_3 \cdot 6H_2O$, have followed major operating campaigns of the combined acid leaching and solids-liquid sections and are reported elsewhere (11, 25).

calculated. Balances of Al_2O_3 and (TiO_2 + SiO_2) also were readily obtained.

Although the soluble Al_2O_3-Fe_2O_3 ratios in the reactor 3 discharge slurry liquor fraction and in the other process liquors differed slightly, these data were used in the calculations. A process iron (Fe_2O_3) balance accounted for less iron than that contained in the calcined kaolin feed. Therefore Fe_2O_3 equal to the discrepancy was proportioned ("best fitted") to the solids fractions of the classifier 4 sands and the thickener 5 underflow pulp.

"Other solubles" contained in the process water, commercial Bé HCl, calcined kaolin feed[8] and process liquor streams were, for simplication, considered to be CaO, MgO, K_2O, Na_2O, and $SO_4^{=}$. Other minor impurities were ignored as relatively unimportant, or assigned as trace elements to the acid-leached solid residues.

Detailed compositional and physical data for calcined kaolin, process liquors, and acid-leached residues are reported in tables A-8 to A-24. These data were used in the material balance calculations.

HCl requirements were determined from the acid-soluble Al_2O_3 and Fe_2O_3 analyses and weight of the calcined kaolin feed. To insure effective leaching, compensate for reactor condenser losses, and maintain a 0.5 to 1.0 pct free HCl concentration in the pregnant liquor product, the stoichiometry was set at 105 pct. However, the material balance indicated that the attained stoichiometry was 98 pct and reflected a minor problem with the acid metering pumps, because measured HCl losses through the condensers were small (fig. 10). Acid requirements for other dissolved solubles were determined by calculation. No corrections were needed for $SO_4^{=}$ levels, and it was assumed that P_2O_5 levels were derived from the apatite [$Ca_5(PO_4)_3F$] (16) presumed to be present in the original kaolin. HCl acid is reported either as HCl (free) or HCl (combined), meaning that quantity associated with dissolved oxides.

The material balance (fig. 10) shows that for a calcined kaolin feed rate of 176 lb/hr, 93 pct of the acid-soluble alumina fraction was extracted. Recovery of the soluble alumina contained in the acid leaching slurry fed to the four-classifier, five-thickener solids-liquid separation circuit was 95 pct. The overall recovery of acid-soluble alumina in the consolidated circuit was (93) (0.95) = 88 pct.

SUMMARY AND CONCLUSIONS

The integrated operation and testing of the acid leaching and solids-liquid separation sections in the clay-HCl miniplant resulted in the following information:

1. A 25-pct solution of HCl acid is effective in extracting 93 to 99 pct of the acid-soluble alumina[9] contained in calcined kaolin in a conventional reactor train over a wide range of operating conditions. Residence time, feed particle size, and reactor design were critical factors affecting leaching, operation of the solids-liquid separation circuit, and recovery of aluminum chloride in the raw pregnant liquor product.

2. A mean particle retention time of 1.5 hr in a three-stage CSTR acid-leaching circuit was required to obtain a 98-pct extraction of the acid-soluble alumina contained in minus 10-mesh calcined kaolin. A decrease of residence time (equivalent to an increased feed rate) will decrease the extraction of

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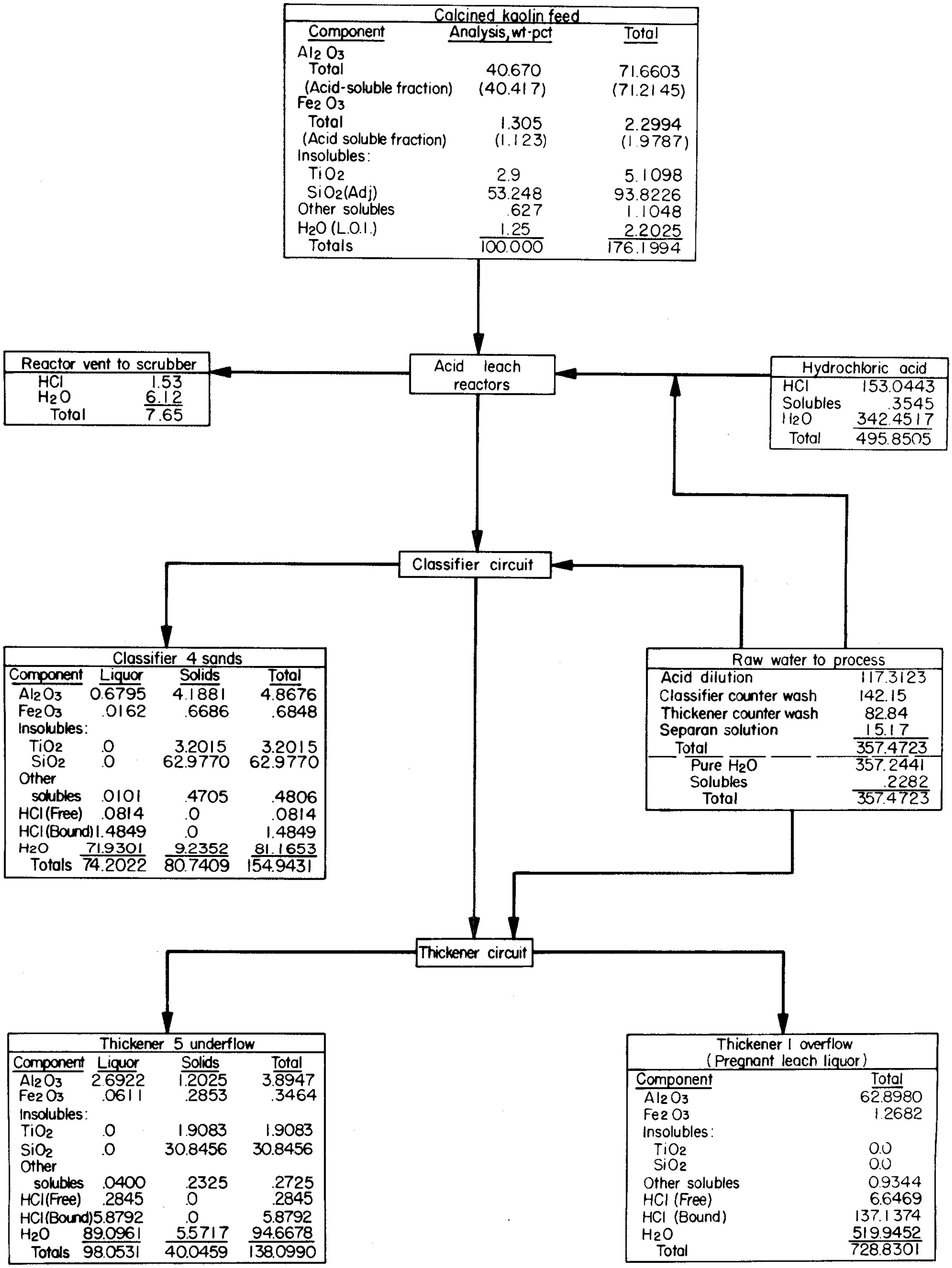
[8] Compositional variation of different lots of calcined kaolin processed in the miniplant was relatively small except for the presence of a fine-grained silica sand noted in run 5 (table A-6). Physical properties of minus 10-mesh calcined kaolin were uniform (table A-7).

[9] See tables A-4 and A-5.



Calcined kaolin feed		
Component	Analysis, wt-pct	Total
Al_2O_3		
Total	40.670	71.6603
(Acid-soluble fraction)	(40.417)	(71.2145)
Fe_2O_3		
Total	1.305	2.2994
(Acid soluble fraction)	(1.123)	(1.9787)
Insolubles:		
TiO_2	2.9	5.1098
SiO_2(Adj)	53.248	93.8226
Other solubles	.627	1.1048
H_2O (L.O.I.)	1.25	2.2025
Totals	100.000	176.1994

Reactor vent to scrubber	
HCl	1.53
H_2O	6.12
Total	7.65

Hydrochloric acid	
HCl	153.0443
Solubles	.3545
H_2O	342.4517
Total	495.8505

Classifier 4 sands			
Component	Liquor	Solids	Total
Al_2O_3	0.6795	4.1881	4.8676
Fe_2O_3	.0162	.6686	.6848
Insolubles:			
TiO_2	.0	3.2015	3.2015
SiO_2	.0	62.9770	62.9770
Other solubles	.0101	.4705	.4806
HCl (Free)	.0814	.0	.0814
HCl (Bound)	1.4849	.0	1.4849
H_2O	71.9301	9.2352	81.1653
Totals	74.2022	80.7409	154.9431

Raw water to process	
Acid dilution	117.3123
Classifier counter wash	142.15
Thickener counter wash	82.84
Separan solution	15.17
Total	357.4723
Pure H_2O	357.2441
Solubles	.2282
Total	357.4723

Thickener 5 underflow			
Component	Liquor	Solids	Total
Al_2O_3	2.6922	1.2025	3.8947
Fe_2O_3	.0611	.2853	.3464
Insolubles:			
TiO_2	.0	1.9083	1.9083
SiO_2	.0	30.8456	30.8456
Other solubles	.0400	.2325	.2725
HCl (Free)	.2845	.0	.2845
HCl (Bound)	5.8792	.0	5.8792
H_2O	89.0961	5.5717	94.6678
Totals	98.0531	40.0459	138.0990

Thickener 1 overflow (Pregnant leach liquor)	
Component	Total
Al_2O_3	62.8980
Fe_2O_3	1.2682
Insolubles:	
TiO_2	0.0
SiO_2	0.0
Other solubles	0.9344
HCl (Free)	6.6469
HCl (Bound)	137.1374
H_2O	519.9452
Total	728.8301

FIGURE 10. - Material balance—acid leaching and solids-liquid separation circuits, run 5. All values, except those in weight-percent, are in pounds per hour.

soluble alumina; e.g., at the 175-lb/hr feed rate, nominal particle residence time was 0.9 hr, and extraction was 93 pct.

3. A 95-pct recovery of acid-solubilized aluminum chloride can be attained in a countercurrent washing solids-liquid separation circuit consisting of four spiral classifiers and five thickeners, arranged in parallel. Increased recovery may be realized by increasing the number of stages in the individual circuits.

4. The process design engineer and investigator is provided with extensive compositional information on solid and liquid products and an overall process material balance for the combined acid leaching and solids-liquid separation steps of the clay-HCl process.

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APPENDIX

TABLE A-1. - Run 5 calcined kaolin feed rates

Calcined kaolin, nominal particle size, mesh	Feed time, hr	Nominal feed rate, lb/hr	Total feed, lb	
			Nominal	Actual
10	63.3	175	11,070	10,659
8	49.2	175	8,620	8,191
10	71.9	175	12,590	12,494
10	5.2	225	1,170	1,162
10	19.8	250	4,940	4,902
Total	209.4	NAp	38,390	37,408

NAp Not applicable.

TABLE A-2. - Thickener circuit pulp retention data

Pulp property	Thickening stage				
	1	2	3	4	5
Solids content ... pct[1]	13.40	16.05	18.78	19.98	23.88
Height ... in	27.47	32.82	14.57	23.22	31.16
Volume ... ft^3	42.02	50.28	23.56	37.77	50.62
Solids ... lb/ft^3	10.88	12.59	14.39	14.84	17.67
Total weight ... lb	457.2	633.0	339.0	560.5	894.5
Retention time[2] ... hr	18.09	25.05	13.42	22.19	35.40

[1] Values derived by averaging data from run 4 and run 5.

[2] Total retention time, 114.15 hr (4.76 days).

TABLE A-3. - Dry screen analyses[1] of calcined kaolin feed materials to acid leaching reactors

Screen size, mesh (Tyler standard sieve)	Cumulative weight retained, pct		
	Minus 20 mesh (runs 1 and 2)	Minus 10 mesh (run 5)	Minus 8 mesh (run 5)
Plus 10	ND	4.52	21.94
Plus 14	ND	29.22	39.12
Plus 20	0.0	48.95	51.71
Plus 28	18.6	62.31	62.42
Plus 35	44.0	72.23	69.73
Plus 48	59.6	80.66	76.86
Plus 65	70.9	87.34	82.36
Plus 100	80.3	92.51	87.35
Plus 150	ND	95.49	91.08
Plus 200	90.3	ND	ND
Pan	100.0	100.00	100.00

ND Not determined.

[1] "Market-grade" screens, having a mesh opening slightly larger than Tyler standard sieves, were used in the sizing equipment.

TABLE A-4. - Summary comparison of operating details of major miniplant runs

Detail	1	2	3	4	5
Calcined kaolin feed:					
Nominal size........................mesh..	-20	-20	-10	-10	-10
Total feed............................lb..	3,500	10,100	9,900	21,900	37,400
Nominal feed rate, lb/hr:					
Low..................................	60	100	100	100	175
High.................................	120	150	[1]100	[1]100	250
Analysis, pct:					
Total Al_2O_3..........................	NA	43.1	43.0	43.2	40.7
Acid-soluble Al_2O_3....................	40.3	40.8	41.8	42.7	40.4
HCl, 20 Bé..............................lb..	10,600	30,300	29,600	69,300	106,500
Diluted concentration...............pct..	20	25.9	26.3	24.4	25.0
Stoichiometry.................mol pct[2]..	101	105	105	104	98
Operations:					
Running time..........................hr..	51	69	98.5	219.5	209.4
Reactors.................................	4	4	4	4 or 3	3
Agitator speed......................rpm..	120	120	120	120	120
Retention time, hr:					
Low..................................	1.9	1.53	ND	ND	0.68
High.................................	3.8	2.3	2.3	1.62	0.97
Classifiers..............................	0	2	2	4	4
Thickeners...............................	0	2	2	5	5
HCl lost as vapor.................lb/hr..	NA	NA	2.26	0.014	1.53
Wash water used...................lb/hr..	0	195	137	198	240
Pregnant liquor, pct:[3]					
Al_2O_3..................................	(7.64)	(10.47)	8.55	8.31	8.63
Fe_2O_3..................................	NA	(0.27)	0.19	0.22	0.21
HCl (free)...............................	NA	(2.1)	0.53	1.48	0.78
Acid-soluble Al_2O_3 extracted.........pct..	NA	99.1	99	98.1	93.1
Dissolved Al_2O_3 recovered............pct..	NA	ND	66.8	88.5	88.3

NA Not available. ND Not determined.

[1] These feed rates were a constant 100 lb/hr.

[2] Stoichiometry in mole percent for the HCl requirement is based on the molar ratio of $HCl/(Al_2O_3 + Fe_2O_3)$.

[3] Values in parentheses are for undiluted reactor discharge liquor.

TABLE A-5. - Extraction of acid-soluble alumina from minus 10-mesh calcined kaolin at different feed rates

Run	Feed rate, lb/hr	Total reactor train residence time, hr	Reactors in service	Acid-soluble alumina extraction, pct
3......	100	2.1	4	99
4......	100	1.45	3	98
5......	175	.92	3	93
5......	250	.64	3	90

TABLE A-6. - Chemical analyses of minus 10-mesh calcined kaolin used in miniplant runs 3, 4 and 5, percent

Component	Run 3	Run 4	Run 5	Component	Run 3	Run 4	Run 5
Al_2O_3:				CoO............	<0.0013	<0.0017	0.010
Total........	43.0	43.21	40.670	CuO............	<.0025	<.005	.003
Acid-soluble				ZnO............	.014	.012	.013
fraction....	(41.8)	(42.7)	(40.417)	NiO............	.005	.0079	.013
Fe_2O_3:				Ga_2O_3..........	ND	.0015	.008
Total........	1.32	1.26	1.305	HgO............	ND	<.0002	<.00005
Acid-soluble				SrO............	<.0025	<.002	.005
fraction....	(1.16)	(1.18)	(1.123)	BaO............	ND	ND	.009
TiO_2:				MoO_3...........	<.0015	<.02	<.0045
Total........	3.06	3.27	2.9	PbO............	.011	ND	.012
Acid-soluble				$SO_4^{=}$...........	.117	.18	.095
fraction....	(.11)	(.127)	(.077)	SnO_2...........	<.0025	<.005	ND
P_2O_5..........	.08	.087	.032	As_2O_3..........	ND	.0002	ND
CaO............	.048	.034	.103	B_2O_3...........	<.032	.005	.045
MgO............	.095	.087	.066	V_2O_5...........	<.011	ND	ND
K_2O............	.110	.063	.118	ZrO_2...........	.014	ND	ND
Na_2O...........	.053	.032	.034	LOD at 125° C..	.12	.051	ND
SiO_2...........	51.4	51.8	55.9	LOI:			
F^-.............	.025	.025	.007	At 750° C....	.71	.645	ND
Cl^-............	ND	ND	.038	At 1,000° C..	ND	ND	1.25
Cr_2O_3..........	.009	.014	.010	Total[1]....	100.25	100.83	100.66
MnO............	.006	.0037	.006				

LOD Loss on drying. LOI Loss on ignition. ND Not determined.

[1] Values in parentheses not included in total.

TABLE A-7. - Physical properties of minus 10-mesh calcined kaolin

Property	Typical value
Density, lb/ft^3:	
Absolute......................	163.4
Pour..........................	57.2
Tap...........................	63.8
Angle of repose............deg..	37
Surface area (BET)........m^2/g..	21

TABLE A-8. - Detailed calcined kaolin feed rate data, run 5

Elapsed time, hr	Cumulative total feed, lb
175-lb/hr FEED RATE PERIOD (AV. 176.1994 lb/hr)	
0.00	0
7.45	1,320
24.30	4,104
31.12	5,480
46.67	8,401
55.00	9,698
58.99	10,394
250-lb/hr FEED RATE PERIOD (AV. 252.5937 lb/hr)	
0.00	0
2.26	581
9.43	2,273
19.47	4,918

TABLE A-9. - Chemical analyses of minus 10-mesh calcined kaolin used in run 5, percent[1]

Sample	1	2	3	4	5	6	Average
Al_2O_3:							
Total	40.6	39.6	40.5	40.9	41.2	41.2	40.670
Acid-soluble fraction	(40.2)	(39.7)	(40.3)	(40.4)	(40.9)	(41.0)	(40.417)
Fe_2O_3:							
Total	1.30	1.30	1.29	1.32	1.32	1.30	1.305
Acid-soluble fraction	(1.14)	(1.10)	(1.12)	(1.12)	(1.13)	(1.13)	(1.123)
LOI at 1,000° C	1.0	.9	1.1	1.4	1.6	1.5	1.250
Total[2]	42.90	41.80	42.89	43.62	44.12	44.00	43.225
Insolubles:[3]							
SiO_2	ND	57.0	56.9	54.9	55.3	55.6	55.9
TiO_2:							
Total	2.9	2.8	2.9	2.9	2.9	3.0	2.9
Acid-soluble fraction	(.09)	(.11)	(.08)	(.07)	(.07)	(.04)	(.077)
Total[2]	NAp	59.8	59.8	57.8	58.2	58.6	58.8
Other solubles:[3]							
P_2O_5	.034	.032	.031	.030	.032	.030	.032
CaO	.057	.057	.060	.063	.11	.27	.103
MgO	.061	.061	.068	.070	.068	.068	.066
K_2O	.13	.13	.11	.12	.11	.11	.118
Na_2O	.027	.038	.036	.031	.032	.038	.034
F^-	.004	.013	.003	.008	.008	.008	.007
Cl^-	.08	ND	.04	.04	.01	.02	.038
Cr_2O_3	.01	.01	.01	.01	.01	.01	.010
MnO	.0052	.0067	.0057	.0062	.0046	.0049	.006
CoO	.011	.0099	.010	.0095	.010	.011	.010
CuO	.0029	.0029	.0034	.0034	.0035	.0035	.003
ZnO	.011	.010	.015	.014	.013	.013	.013
NiO	.013	.012	.013	.013	.012	.013	.013
Ga_2O_3	.009	.005	.009	.008	.007	.008	.008
SrO	.0048	.0050	.0052	.0051	.0053	.0060	.005
BaO	.009	.008	.009	.010	.008	.008	.009
B_2O_3	.06	.03	.03	.06	.03	.06	.045
PbO	.012	.012	.013	.012	.012	.013	.012
$SO_4^=$	.08	.02	.05	.13	.17	.12	.095
Total	.6209	.4625	.5213	.6432	.6554	.8144	.627
Grand total	NAp	102.06	103.21	102.06	102.9	103.41	102.65

LOI Loss on ignition. NAp Not applicable. ND Not determined.

[1] HgO <0.5 ppm, MoO_3 <45 ppm; As_2O_3, BeO, V_2O_5, SnO_2 less than detection limits.

[2] Values in parentheses are not included in totals.

[3] "Insolubles" and "other solubles" are arbitrary groupings of components used to simplify the material balance calculations.

TABLE A-10. - Chemical analyses of 20 Bé HCl acid, percent

Sample	1	2	3	4	Average
HCl	30.93	30.98	30.79	30.76	30.865
Minor elements:					
CaO	.0073	.0073	.0073	.0073	.0073
MgO	.0032	.0032	.0032	.0032	.0032
K_2O	.0092	.0092	.0092	.0092	.0092
Na_2O	.0094	.0094	.0094	.0094	.0094
$SO_4^=$	.0414	.0425	.0435	.0420	.0424
Total	.0705	.0716	.0726	.0711	.0715

TABLE A-11. - Trace element analyses[1] of 20 Bé HCl acid, parts per million

Sample	5	6	7	Average
Al_2O_3	0.12	0.2	0.4	0.2
Fe_2O_3	.2	.2	.7	.4
TiO_2	<.004	<.004	<.009	<.006
SiO_2	1.4	2	2	2
Cr_2O_3	.01	.01	ND	.01
MnO	.003	.003	.008	.005
CuO	<.003	.003	.13	.04
NiO	.001	.001	.001	.001
SrO	<.2	<.2	.6	.2
MoO_3	<.003	<.003	<.008	<.005
B_2O_3	.07	.07	<.16	.07
PbO	<.02	<.02	ND	<.02
Ag_2O	.005	<.003	.01	.006
Suspended solids	217	224	522	321

ND Not determined.

[1] Elements not detected: Co, Sb, Ba, Be, Sn, W, Zn, Au, Hf, Nb, Pt, Ta, V, As, Cd, Ga, In, Re, Te, Tb.

TABLE A-12. - Chemical analyses of process water, parts per million

Sample	1	2	3	Average
Major solubles:				
Al_2O_3	<0.1	<0.1	<0.1	<0.1
Fe_2O_3	<.05	<.05	<.05	<.05
Insolubles:[1]				
SiO_2	16.4	21.1	14.3	17.3
TiO_2	<.08	<.08	<.08	<.08
Total	16.48	21.18	14.38	17.38
Other solubles:[1]				
P_2O_5	<1	ND	ND	<1
CaO	139.9	139.9	132.9	137.6
MgO	63.74	62.19	54.42	60.12
K_2O	6.99	6.99	6.02	6.67
Na_2O	167	162	142	157
$SO_4^=$	278	ND	ND	278
F^-	<1	1	<1	<1
Cr_2O_3	<.01	<.01	<.01	<.01
ZnO	<.01	<.01	<.01	<.01
NiO	<.02	<.02	<.02	<.02
B_2O_3	1.2	1.2	.5	.97
Cl^-	73	69	94	79
NO_3^-	2	2	1	2
Total[2]	<733.87	NAp	NAP	<723.4
Density, at 23° C, g/mL	0.9997	0.9978	0.9966	0.9980
pH	7.9	8.3	8.3	8.2

NAp Not applicable. ND Not determined.

[1] "Insolubles" and "other solubles" are arbitrary groupings of components used to simplify the material balance calculations.

[2] "Total other solubles," when compared to that in other tables and the material balance, should be calculated without including HCl (free), Cl^-, and NO_3^- for a total of 639 ppm.

TABLE A-13. - Chemical analyses of reactor 3 discharge slurry--solids fraction, percent

Sample	1	2	3	4	5	6	7	8	Average
Al_2O_3	4.7	4.6	4.2	3.0	2.8	3.7	3.6	6.0	4.08
Fe_2O_3	.4	.5	.5	.4	.4	.4	.4	.6	.45
TiO_2	4.0	4.1	3.9	4.0	3.9	4.1	4.2	4.1	4.04
LOI[1]	13.12	12.53	12.14	12.60	12.32	12.60	13.17	12.18	12.58

[1] Loss on ignition at 1,000° C.

TABLE A-14. - Chemical analyses and physical data for reactor 3 discharge slurry--liquid fraction

Sample	Slurry solids, pct	Density at 24° C, g/mL	Analysis, pct		
			Al_2O_3	Fe_2O_3	Free HCl
1	14.60	1.2909	10.8	0.23	0.67
2	14.66	1.2909	10.7	.23	.91
3	12.92	ND	8.8	.19	.42
4	16.95	1.2773	10.2	.22	1.36
5	14.33	1.2800	10.4	.22	1.05
6	12.70	ND	8.7	.20	.68
7	13.90	1.2805	10.4	.22	.99
8	12.40	1.2835	10.5	.23	.90
9	13.66	ND	9.3	.14	.08
Average	14.01	1.2839	9.98	.209	.784

ND Not determined.

TABLE A-15. - Chemical analyses of thickener 1 overflow liquor (raw pregnant liquor), percent

Sample	1	2	3	4	5	Average
Major solubles:						
Al_2O_3	8.42	8.40	8.53	8.88	8.92	8.63
Fe_2O_3	.17	.17	.17	.18	.18	.17
(FeO)	(.016)	(.015)	(.013)	(.011)	(.012)	(.013)
HCl (free)	.77	.90	.96	.99	.94	.912
Insolubles:[1]						
SiO_2	<.0035	<.0035	<.0035	<.0035	<.0035	NAP
TiO_2	<.005	<.005	<.005	<.005	<.005	NAp
Suspended solids	.011	.015	.015	.012	.011	.0128
Other solubles:[1]						
P_2O_5	.019	.019	.015	.017	.017	.0174
CaO	.017	.013	.012	.012	.012	.0132
MgO	.017	.017	.016	.017	.017	.0168
K_2O	.013	.013	.014	.013	.014	.0134
Na_2O	.016	.015	.015	.015	.015	.0152
$SO_4^=$	.053	.051	.050	.053	.054	.0522
F^-	<.001	<.001	<.001	<.001	<.001	NAp
Cr_2O_3	.0024	.0021	.0024	.0025	.0024	.0024
SnO	<.004	<.004	<.004	<.004	<.004	NAp
MnO	.0013	.0013	.0014	.0014	.0014	.0014
CoO	.0023	.0023	.0023	.0024	.0024	.0023
CuO	.00071	.00076	.00068	.00071	.00073	.0007
ZnO	.0026	.0023	.0023	.0025	.0026	.0025
NiO	.0029	.0028	.0028	.0029	.0030	.0029
Ga_2O_3	.0020	.0021	.0020	.0021	.0022	.0021
HgO	<.010	<.010	<.010	<.010	<.010	NAp
SrO	.0018	.0017	.0017	.0018	.0018	.0018
BaO	<.0010	<.0010	<.0010	<.0010	<.0010	NAp
As_2O_3	<.0008	<.0008	<.0008	<.0008	<.0008	NAp
BeO	<.0003	<.0003	<.0003	<.0003	<.0003	NAp
MoO_3	<.0011	<.0011	<.0011	<.0011	<.0011	NAp
PbO	.0018	.0017	.0018	.0018	.0018	.0018
V_2O_5	.0011	.0014	.0014	.0014	.0018	.0014
B_2O_3	ND	ND	ND	ND	ND	ND
Total	<.17211	<.16466	<.15898	<.16471	<.16733	.1475
Density, at 24° C...g/mL	1.2215	1.2214	1.2250	1.2348	1.2351	1.22756

NAp Not applicable. ND Not determined.

[1] "Insolubles" and "other solubles" are arbitrary groupings of components used to simplify the material balance calculations.

TABLE A-16. - Chemical analyses and physical data for washed and dried solids from classifier 1 overflow

Sample	Chemical analyses, pct				Physical data[1]			
	Al_2O_3	Fe_2O_3	TiO_2	LOI at 1,000° C[2]	Slurry solids, pct	Density (absolute), g/mL	Surface area (BET), m^2/g	Plus 100-mesh particles, pct
1................	2.5	0.4	4.3	14.2	4.33	2.24	346	ND
2................	2.8	.4	4.3	13.8	4.43			4.89
3................	3.3	.4	4.4	14.2	4.46	2.21	ND	5.13
4................	3.2	ND	4.6	15.2	4.40			4.98
5................	3.6	.4	4.0	11.5	3.16	2.34	351	3.34
6................	2.7	.3	4.2	15.4	3.47			11.26
7................	3.0	.4	4.3	13.2	4.56	ND	347	24.31
8................	2.6	.5	4.7	12.9	3.51			11.92
9................	3.0	.3	4.1	13.5	3.35	2.28	351	16.91
10................	3.3	.3	4.4	15.2	3.91			4.96
Average.......	3.0	.38	4.33	13.91	3.958	2.27	349	9.74

LOI Loss on ignition. ND Not determined.

[1] Density and surface area measurements were made on 5 composite samples of solids on which chemical analyses were made.

[2] Air-dried at room temperature before analysis. (Weight loss at 125° C averages 8.4 pct. The residues dried at 125° C then lose about an additional 4.5 pct on ignition at 1,000° C for 1 hr.)

TABLE A-17. - Detailed chemical analyses of washed and dried classifier 1 overflow--solids fraction,[1] percent

Sample................	1	2	3	4	5	Average
Al_2O_3..................	2.65	3.25	3.15	2.80	3.15	3.0
Fe_2O_3..................	.40	.40	.35	.45	.30	.38
Cl^-..................	.33	ND	.18	.20	.20	.228
LOI at 1,000° C........	14.00	14.90	13.45	13.05	14.40	13.96
Total.............	17.38	18.55	17.13	16.50	18.05	17.568
Insolubles:[2]						
TiO_2................	4.3	4.5	4.1	4.5	4.25	4.33
SiO_2................	74.3	84.7	68.8	ND	91.50	79.82
Total.............	78.6	89.2	72.9	4.5	95.75	84.155
Other solubles:[2]						
P_2O_5................	.005	ND	.006	.005	.003	.0048
CaO................	.088	.083	.078	.070	.070	.0778
MgO................	.051	.045	.045	.043	.045	.0458
K_2O................	.095	.10	.11	.098	.098	.1002
Na_2O................	.093	.092	.15	.069	.073	.0954
$SO_4^=$................	.28	.34	.25	.25	.12	.2480
F^-................	.012	.015	.014	.012	.014	.0134
SnO................	.007	.01	.01	.02	.008	.0110
MnO................	.00090	.00065	.00065	.00037	.00090	.0007
CoO................	.0067	.0064	.0064	.0067	.0067	.0066
CuO................	.0025	.0014	.0023	.0014	.0056	.0026
ZnO................	.0027	.0024	.0022	.0010	.0025	.0022
NiO................	.0087	.0080	.0090	.0087	.0090	.0087
BaO................	.004	.007	.006	.004	.005	.0052
B_2O_3................	.03	.03	.03	.03	.03	.0300
PbO................	.013	.013	.012	.012	.013	.0126
V_2O_5................	.04	.05	.05	.05	.05	.0480
Total.............	.73950	.80385	.78155	.68117	.55370	.7130
Grand total.......	96.71950	108.55385	90.81155	21.68117	114.35370	102.4355

LOI Loss on ignition. ND Not determined.

[1] Classifier 1 overflow solids are ultimately equivalent to thickener 5 overflow solids.

[2] "Insolubles" and "other solubles" are arbitrary groupings of components used to simplify the material balance calculations. Cr_2O_3, Ga_2O_3, and SrO were not detected.

TABLE A-18. - Chemical analyses and physical data for thickener 5 underflow--liquid fraction

Sample	Slurry solids, pct	Liquor density at 20° C, g/mL	Liquor analysis, pct		
			Al_2O_3	Fe_2O_3	HCl (free)
1........................	29.02	1.0644	2.70	0.065	0.14
2........................	28.40	1.0635	2.67	.062	.15
3........................	28.54	1.0633	2.67	.060	.13
4........................	ND	1.0614	2.59	.058	.13
5........................	28.89	1.0614	2.60	.059	.12
6........................	29.80	1.0665	2.84	.066	.16
7........................	ND	1.0668	2.84	.066	.16
8........................	ND	1.0670	2.86	.063	.13
9........................	ND	1.0647	2.74	.060	.095
10.......................	ND	1.0661	2.83	.062	.12
Average..............	28.93	1.0645	2.73	.062	.134

ND Not determined.

TABLE A-19. - Chemical analyses and physical data for classifier 4 overflow--liquid fraction[1]

Sample	Suspended solids, pct	Liquor density at 20° C, g/mL	Liquor analysis, pct		
			Al_2O_3	Fe_2O_3	HCl (free)
1........................	0.70	1.0207	0.79	0.019	0.10
2........................	.63	1.0214	.79	.018	.099
3........................	.48	1.0224	.85	.020	.095
4........................	.43	1.0216	.85	.020	.094
5........................	.46	1.0186	.76	.018	.074
6........................	.45	1.0187	.76	.018	.074
7........................	.24	1.0207	.84	.020	.11
8........................	.37	1.0211	.84	.019	.11
9........................	.63	1.0323	1.33	.033	.17
10.......................	.58	1.0324	1.34	.033	.17
Average..............	.597	1.0230	.915	.0218	.1096

[1] Also used as analysis for liquor associated with the sand discharged to tailings.

TABLE A-20. - Chemical analyses of washed and dried classifier 4 sands---solids fraction, percent

Sample	1	2	3	4	Average
Al_2O_3	6.9	4.4	4.7	4.8	[1]5.2000
Fe_2O_3	.5	.5	.6	.4	.5000
Cl^-	.01	.04	.17	.05	.0675
LOI at 1,000° C	11.5	11.8	ND	11.1	11.4667
Total	18.91	16.74	NAp	16.35	17.2342
Insolubles:[2]					
TiO_2	3.9	4.1	3.9	4.0	3.9750
SiO_2	81.3	86.7	77.8	80.4	81.5500
Total	85.2	90.8	81.7	84.4	85.5250
Other solubles:[2]					
P_2O_5	.003	.004	.002	.003	.0030
CaO	.066	.098	.060	.060	.0710
MgO	.036	.036	.032	.030	.0335
K_2O	.098	.100	.093	.095	.0965
Na_2O	.059	.070	.059	.075	.0658
$SO_4^=$	.16	.15	.09	.28	.1700
F^-	.010	.008	.009	.006	.0082
SnO	.002	.003	ND	ND	.0025
MnO	.00065	.00090	.00065	.0011	.0008
CoO	.0064	.0064	.0067	.0067	.0066
CuO	.0014	.0014	.0013	.0013	.0014
ZnO	.0020	.0017	.0016	.0017	.0018
NiO	.0094	.0080	.0087	.0090	.0088
BaO	.006	.002	.004	.004	.0040
B_2O_3	ND	.06	ND	.03	.0450
PbO	.013	.013	.012	.013	.0128
V_2O_5	.09	.05	.05	.07	.0650
Total	.56285	.61240	.42995	.6858	.5967
Grand total	104.67285	108.15240	87.59995	101.4358	103.3559

LOI Loss on ignition. NAp Not applicable. ND Not determined.

[1] Classifier 4 sands produced during the 250-lb/hr feed rate contained 6.99 pct total and 5.73 pct acid-soluble alumina, respectively.

[2] "Insolubles" and "other solubles" are arbitrary groupings of components used to simplify the material balance calculations. Cr_2O_3, Ga_2O_3, and SrO were not detected.

TABLE A-21. - Chemical analyses of washed and dried classifier 4 sand screen fractions, percent

Screen size, mesh (Tyler standard sieve)	Sample 1	Sample 2	Sample 3	Sample 4	Average
Al_2O_3 (total):					
Plus 14	7.0	5.6	3.7	4.5	5.20
14 by 28	7.3	6.5	4.4	4.5	5.68
28 by 48	7.2	6.5	5.1	6.1	6.23
Minus 48	14.8	11.7	9.2	9.9	11.40
All fractions	6.9	4.4	4.7	4.8	5.20
Fe_2O_3 (total):					
Plus 14	.4	.4	.4	.4	.4
14 by 28	.5	.3	.2	.3	.3
28 by 48	.3	.3	.4	.4	.4
Minus 48	.3	.3	.4	.4	.4
All fractions	.5	.5	.6	.4	.5
TiO_2 (total):					
Plus 14	4.0	4.2	3.7	4.3	4.0
14 by 28	4.1	4.2	4.2	4.3	4.2
28 by 48	4.1	4.0	4.0	3.9	4.0
Minus 48	3.2	3.5	3.6	3.5	3.4
All fractions	3.9	4.1	3.9	4.0	4.0
Screen fractions, total sample:					
Plus 14	22.26	21.90	17.67	17.96	19.95
14 by 28	39.24	37.68	34.66	37.12	37.18
28 by 48	26.61	26.31	26.81	25.37	26.28
Minus 48	11.89	14.11	20.86	19.55	16.60
Total	100.00	100.00	100.00	100.00	100.00

TABLE A-22. - Classifier 4 wet coarse sand discharge data

Sample	Flow rate, lb/hr	Composition, pct Liquid	Composition, pct Solids
1	141	41.56	58.44
2	146	45.77	54.23
3	159	46.05	53.95
4	160	44.62	55.38
5	158	45.72	54.28
6	128	45.85	54.15
7	143	48.13	51.87
8	142	48.10	51.90
9	138	51.89	48.11
10	177	52.48	47.52
11	193	52.35	47.65
12	173	53.15	46.85
Average	154.8	47.97	52.03

TABLE A-23. - Washing water flows to solids-liquid separation circuit

Measurement	Flow rate, lb/hr	
	Classifier 4	Thickener 5
1............	139.64	83.65
2............	144.64	66.26
3............	145.56	75.61
4............	143.06	ND
5............	143.85	85.63
6............	145.00	ND
7............	151.49	84.31
8............	144.64	84.31
9............	140.95	85.63
10............	141.48	85.63
11............	123.04	84.31
12............	143.59	83.65
13............	140.95	86.68
14............	142.27	84.31
15............	ND	86.94
Average..	142.15	82.84

ND Not determined.

TABLE A-24. - Separan MGL solution flow to thickener circuit

(0.2 pct Separan MGL solution to thickeners)

Measurement	Flow, gph
1............................	1.998
2............................	1.645
3............................	1.814
Average................	[1]1.819

[1] Average flow 15.17 lb/hr.

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To provide technology that can help decrease the dependence of the U.S. aluminum industry on imported bauxite, the main ore of aluminum, the Bureau of Mines is investigating the extraction of aluminum from abundant domestic reserves of kaolinitic clay. Miniplant studies of the continuous leaching of calcined kaolin with boiling 25-pct HCl acid resulted in a 93- to 99-pct extraction of the acid-soluble alumina in the calcined kaolin feed. Data are presented for an acid leaching study carried out in three or four continuously stirred tank reactors (CSTR) in series, feed rates of 100 to 250 lb/hr of calcined kaolin, and total nominal residence times of 0.7 to 2.3 hr. A material balance for a 176-lb/hr calcined kaolin feed rate in a circuit consisting of three reactors, four classifiers, and five thickeners is given. Data provide detailed compositional and physical information for calcined kaolin, process liquors, and acid-leached residue solids.

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