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DESIGN AND OPERATION OF A DEMONSTRATION SCALE
LIQUID-ION EXCHANGE ALUM RECOVERY PLANT

presented by

Gary C. Cline

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David A. Connell
Major professor

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DESIGN AND OPERATION OF A DEMONSTRATION SCALE LIQUID-ION EXCHANGE ALUM RECOVERY PLANT

By

Gary C. Cline

A THESIS

Submitted to
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ABSTRACT

DESIGN AND OPERATION OF A DEMONSTRATION SCALE
LIQUID-ION EXCHANGE ALUM RECOVERY PLANT

By

Gary C. Cline

Based on data developed in continuous flow, bench-scale studies of alum recovery from raw alum sludge with the liquid-ion exchange process, a 10-gpm demonstration-scale pilot plant was designed and constructed at the City of Tampa, Florida Hillsborough River Water Treatment Plant.

The pilot plant was operated a total of 10 months in 1979. Operational difficulties which were initially encountered included the emulsification of the insoluble sludge solids during aluminum extraction and the subsequent recovery of solvent from that emulsion. These problems were overcome by the replacement of the mixer-settler extractor with an RTL Contactor. The RTL Contractor did not emulsify the insoluble solids and precluded the need for solvent recovery.

The results of the pilot-plant operation confirmed bench-scale results and process scale-up methods. The liquid-ion exchange process recovered 90 percent of the aluminum in the sludge and effected a substantial reduction in the dry weight solids of the aqueous waste stream.

ACKNOWLEDGEMENTS

I would like to recognize the following people --

Mr. Elroy Spitzer and the AWWA Research Foundation for their generous support,

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And last, but not least, my wife Lise -- who makes it all worthwhile.

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CHAPTER 1

INTRODUCTION

1.1 Description of the Problem

Aluminum sulfate or alum is used as a coagulant for the removal of turbidity and color in potable water treatment. The removal of these impurities produces a sludge which is of low density and contains large amounts of water of hydration. The production of alum sludges in the United States exceeds 14 million wet weight tons each year.[1]

Traditionally, alum sludges have been disposed of by discharge to the water treatment plant raw water source. However, the enactment of PL 92-500 in 1972 has forced the water production industry to abandon traditional sludge disposal methods and replace them with environmentally sound sludge treatment and disposal techniques.

In order to comply with the terms of PL 92-500, water treatment facilities must employ the Best Practical Treatment Economically Available (BPTEA) by July 1, 1982. BPTEA is generally accepted to be a technology that has been demonstrated on an advanced laboratory or pilot-plant scale to be technically and economically feasible.

Currently, the most economical method of treatment and disposing of alum sludges is dewatering following by landfilling of the residual solids. To be amenable to landfilling with other waste material, the sludge must have no free

water, which implies a solids concentration greater than 20 percent. Furthermore, it is generally felt that the solids concentration must be increased to 40 percent in order to landfill an alum sludge alone.[1] Because of the hydrous nature of alum sludges, obtaining a solids concentration of 20-40 percent is difficult and expensive. While many sludge dewatering methods are currently available, each has problems associated with it.

Co-disposal of the sludge with wastewater sludge can be practiced if a sanitary sewer is accessible and the wastewater treatment plant will accept the sludge. The disadvantages of co-disposal are that the acceptance costs of the wastewater plant are often high and these costs are subject to inflation.

Gravity methods of sludge dewatering include drying on sand beds, freeze-thawing, and lagooning. Sand bed drying and freeze-thawing can be effective if the proper climate and necessary land coexist. Lagooning of sludge is only a temporary solution since lagoons eventually fill with solids and these solids are generally unacceptable for landfilling.

Mechanical dewatering devices such as vacuum filters, belt filters, centrifuges, and pressure filters are capable of producing a solids cake suitable for landfilling, but these devices tend to be capital and operating intensive, putting the costs of mechanical dewatering methods out of the reach of smaller plants.

Landfilling costs can also be high. The aluminum and heavy metal content of the dewatered sludge has prompted many states to classify alum sludge as a hazardous waste. This classification requires the sludge to be disposed of in secure landfills whose user costs are higher than those of conventional sanitary landfills.

Clearly, other sludge treatment alternatives must be developed if water treatment plants are to meet discharge requirements and hold down the cost of water production. The groundwork for one such alternative was laid down in 1975 by Cornwell.[2] Using liquid-ion exchange, a commerically successful operation in the field of extractive metallurgy, Cornwell proposed a system where aluminum could be recovered from alum sludges generated by wastewater treatment plants in the removal of phosphates. In conjunction with the recovery of aluminum, a substantial reduction in dry weight suspended solids was also realized. Later, this idea was expanded to include the recovery of alum from alum sludges generated by water treatment plants.

Using liquid-ion exchange to recover alum from water treatment plant sludges is a novel idea, but the recovery of alum through sulfuric acid treatment of the sludge is not. Acidification recovery systems, reviewed elsewhere,[3] could recovery 50-90 percent of the aluminum in the sludge and effect a significant reduction in the weight and volume of

solids requiring disposal. These systems, however, were plagued by the accumulation of contaminants in the recovered alum. Also, the recovered alum was often dilute and inconsistent in its aluminum content, making accurate dosing difficult.

Alum recovery through liquid-ion exchange can overcome the problems associated with acid recovery by selectively extracting aluminum from the sludge, leaving potential contaminants in the process waste stream. Liquid-ion exchange recovery can also produce alum which is consistent in nature and comparable to commercial alum in strength.

In 1976, Cornwell was awarded a research grant by the American Water Works Association Research Foundation to investigate and develop the liquid-ion exchange alum recovery process. The study was divided into three areas, each covering one year. The subject of each study area is listed below:

- 1st year - Batch Optimization and Sludge Characterization
- 2nd year - Bench-Scale Continuous Flow Studies
- 3rd year - Pilot-Scale Continuous Flow Studies

In the first year, viability of the process was demonstrated on a batch basis using synthetic aluminum solutions as feed stock. Extractants, diluents, and stripping agents were screened and selected. Extraction and stripping phase ratios were optimized and isotherms developed.

In the second year of research, the previous results were used to design a continuous flow bench-scale recovery unit. During the first months of this study area, synthetic solutions were used as feed stock. Hydraulic and chemical characteristics of the system were evaluated. When it was felt that key operating parameters had been defined, the feed solution was changed to raw alum sludge. This sludge was taken from the City of Tampa, Florida, Water Treatment Plant. Tampa sludge was chosen for the following reasons:

1. Tampa's use of 22 tons of alum per day made coagulant recovery very attractive.
2. The sludge generated at Tampa contained large amounts of organic matter and it was felt that the processing of this sludge would be a severe test for the liquid-ion exchange operation.

Key operating parameters were again evaluated with sludge as the feed solution. The final months of the second year were spent generating design data necessary for process scale-up.

1.2 Rationale for Research

After two years of laboratory study and development, the viability of the liquid-ion exchange alum recovery process had been demonstrated at both batch and continuous bench scales. Using the sludge generated at the City of Tampa, Florida, Hillsborough River Water Treatment Plant as the feed stock, operating parameters and design data were developed. Based on the proven laboratory feasibility of

the alum recovery process, the City of Tampa, Michigan State University, and the AWWA Research Foundation undertook a joint venture to have the first liquid-ion exchange alum recovery pilot plant erected at the Hillsborough River Water Treatment Plant. The subsequent design, operation, and evaluation of that pilot plant make up the third study area of the research program and are subject of this thesis.*

*The third study area was divided into two parts, the first being the subject of this thesis. The second deals with the quality and treatment of the process exit streams and is the subject of a thesis by Pryzbyla.(4)

CHAPTER 2

LIQUID-LIQUID EXTRACTION EQUIPMENT

2.1 Principles

Efficient transfer of solute from one phase to another and the subsequent separation of those phases is the task of liquid-liquid extraction equipment. The rate of solute transfer is dependent on, among other factors, the interfacial area and the deviation of the actual solute concentrations in the two phases from the equilibrium position.[5] Thus, without greatly degrading phase separation performance, an efficient design seeks to maximize interfacial area and solute concentration gradients.

In liquid-liquid extraction equipment, one phase is dispersed in the form of drops into the continuous phase. The interfacial area is a function of phase ratio and mean drop size. The drop size is dependent on the phase ratio, degree of agitation, and physical and chemical properties of the phases. Physical properties would include phase densities, viscosities, and interfacial tension. Up to a point then, a decrease in mean drop size yields an increase in interfacial area which increases mass transfer rate.

The solute transfer mechanism involves both molecular and eddy diffusion. Molecular diffusion, the random movement of molecules in the direction of the concentration gradient,

is a relatively slow process. Eddy diffusion, originating in the bulk movement of the fluid is, under turbulent conditions, much greater than molecular diffusion and for the purposes of liquid-liquid extraction to be encouraged.

Given turbulent conditions in the continuous phase, eddy diffusion will usually dominate that phase. Turbulence in the dispersed phase is a function of mean drop size, the velocity of the drops with respect to the continuous phase, and drop interaction. A high relative velocity is desirable because as a drop is moving through the continuous phase, drag at the interface sets up internal circulation in that drop. This action promotes mixing with the drop and enhances the mass transfer. Droplet interaction, the coalescence and redispersion of two drops, produces mixing within the drops. This too, promotes solute transfer in the dispersed phase. Internal circulation and droplet interaction, however, are functions of drop size. Very small drops behave as rigid spheres. So, as agitation increases, the mean drop size decreases and a point is reached at which the drops assume this rigid sphere behavior. It is here where internal circulation and droplet interaction cease and the slower molecular diffusion mechanisms becomes the primary means of mass transfer in the dispersed phase. So it can be seen that adequate mixing is necessary to effect efficient mass transfer. Excessive mixing, however, will have a negative effect on mass transfer rates.

As stated earlier, solute transfer rates are also a function of the difference in solute concentrations in the two phases with respect to the equilibrium position. This is optimized when the mixing pattern for the two phases corresponds to perfect countercurrent plug-flow. In stage-wise contactors such as mixer-settlers, the concentration profile is very nearly ideal. Differential contactors, those which provide continuous conditions for mass transfer throughout their length, usually deviate greatly from their ideal concentration profiles.

Transferring solute from one phase into another is only one part of the liquid-liquid extraction process. Once the mass transfer has taken place, the two phases must be separated. The rate of coalescence of the dispersion is primarily a function of the drop size distribution of the dispersion, the phase ratio, and the nature of the dispersion (organic or aqueous continuous).[6,7] Also, if solute transfer in the mixer is incomplete, the transfer will continue in the settler and this can have either an enhancing or inhibiting effect on the rate of coalescence.[8] Often, conditions which are desirable for mass transfer are deleterious to phase separation. Increased agitation can increase mass transfer, but the decrease in drop size of the dispersion reduces the rate of coalescence.[6] Also, mass transfer from the dispersed to continuous phase has been shown to promote coalescence.(5)

Given the interdependence of the many variables involved in the mixing and settling operations of liquid-liquid extraction, process optimization can be a difficult task.

2.2 Contactor Classification

Many types of contacting equipment are currently available ranging in sophistication from simple spray columns to centrifugal extractors. The classification and description of the various contacting devices has been carried out by several investigators.[5, 9, 10] A brief survey of liquid-liquid extraction equipment will be the subject of this section.

Industrial contacting equipment can be divided into two main categories, differential and stagewise. Differential contactors provide conditions for mass transfer throughout their length. The flow of the two phases is countercurrent. Because the concentration gradient changes along the length of the contactor, equilibrium is never reached at any point in the contactor. Stagewise contactors, on the other hand, provide a number of discrete stages in which the two phases are equilibrated and then separated before being passed countercurrent to each other.

Further divisions can be made according to the type of force which produces phase dispersion. This force may be gravity, pulsation, mechanical agitation, or centrifugal force. A list of the major types of contactors is shown in Table 2-1.

TABLE 2-1

CLASSIFICATION OF INDUSTRIAL CONTACTORS (9)

	Countercurrent Flow Produced By:			
	Gravity			Centrifugal Force
Phase dispersion → produced by:	Gravity	Pulsation	Mechanical agitation	Centrifugal force
Continuous (differential) contact	Spray column packed column	Pulsed packed column	RDC Oldshue-Rushton column	Podbielniak contractor Westfalia extractor De Laval extractor
Stagewise contact (coalescence-redispersion cycle)	Plate column	Pulsed sieve-plate column	Scheibel column Treybal contactor ARD estractor Mixer-settler	

2.3 Column Contactors

2.3.1 Unagitated Column Contactors

The simplest type of column contactor, shown on Figure 2-1, is the spray column. In this contactor, the dispersed phase is sprayed either up or down through the continuous phase. Spray columns are simple to construct and are capable of providing high throughputs per unit cross-sectional area. They are very inefficient, however, since there are no internal obstructions to check axial mixing in the continuous phase. There is so much axial mixing in these devices that the continuous phase usually has a uniform composition throughout the column. Spray columns are usually used for simple separations or washing processes requiring few stages.

The problem of axial mixing in spray columns is substantially reduced by the introduction of packing material into the column, resulting in the simple packed column, depicted on Figure 2-1. The packing material chosen must be preferentially wetted by the continuous phase to prevent premature coalescence of the dispersed phase and thereby reduce the interfacial area. The packing will enhance solute transfer by reducing axial mixing, setting up internal circulation within the dispersed phase, and promoting droplet interaction. The use of packing, however, reduces the cross-sectional area available for flow and the diameter of a column required for a given throughput will be greater for a packed column

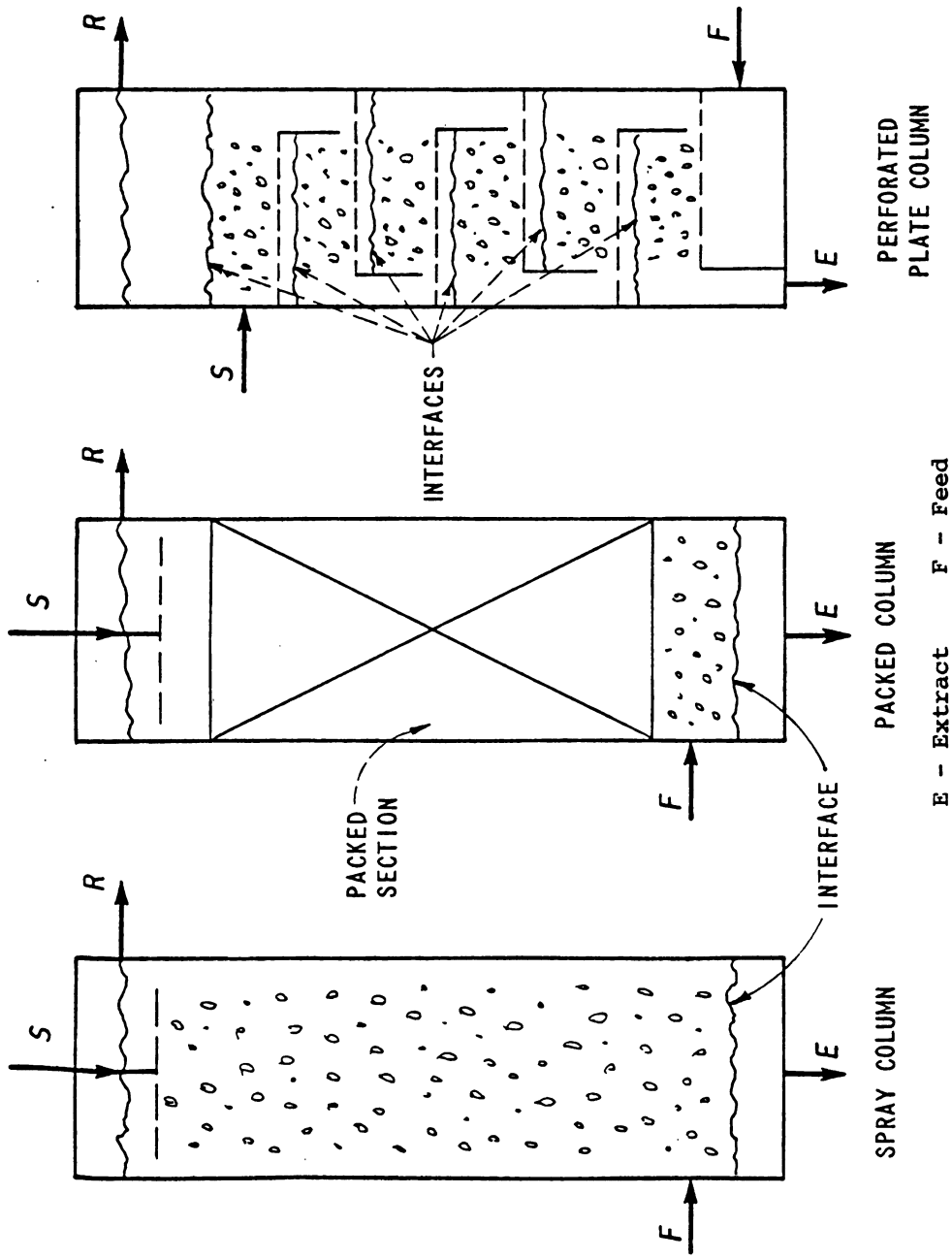


FIGURE 2-1
UNAGITATED COLUMN CONTACTORS (9)

than for a spray column. Packed column performance is hindered if there are solids present in either feed stream which would foul the packing.

The third important type of unagitated column is the perforated plate column, shown on Figure 2-1. Operating in a stagewise manner, the dispersed phase goes through a coalescence-redispersion cycle within each stage. These columns can be operated with either light or heavy phase dispersed. The dispersed phase moves either up or down the length of the column, as the case may be, collecting under or over the perforated plates until enough has coalescenced to produce an adequate head to force the liquid through the perforations and disperse it again into the continuous phase. Interstage flow of the continuous phase is handled by downcomers or upcomers, depending on which phase is continuous.

In general, gravity differential contactors are inexpensive to build and operate. They cannot handle wide ranges of flow ratios, require large amounts of headroom, and because of axial mixing are inefficient and difficult to scale-up.

2.3.2 Mechanically Agitated Columns

If mechanical agitation is provided, the efficiency of simple packed or perforated plate columns can be greatly

increased. One way of supplying this agitation is to apply an oscillating pulse to the contents of the column, resulting in the pulsed column shown on Figure 2-2. It has been suggested that a gauze packing be used in place of random packing since the pulsing tends to orient the random packing, causing channeling and lowering efficiency. The action of a pulsed column involves a coalescence-redispersion cycle. The dispersed phase will coalesce above or below the gauze or plate and will be prevented from flowing through the perforations until the upward or downward pulse pushes that phase through the perforations, dispersing it into the next stage. The continuous phase is drawn through on the other cycle of the pulse. The application of the pulse improves the solute transfer efficiency over that of an unpulsed column by increasing turbulence and interfacial area.

The mechanical problems associated with providing the pulse limits the size of pulsed columns. In order to produce larger units there has been developmental work with the use of compressed air to pulse large columns.[11]

Because of the considerable amount of energy required to pulse a liquid column, the reciprocating-plate column has been developed as an alternative. Research on this type of column and its industrial use have been reported in recent years.

While pulsed columns have had many applications in the nuclear and petrochemical industry, the most important type

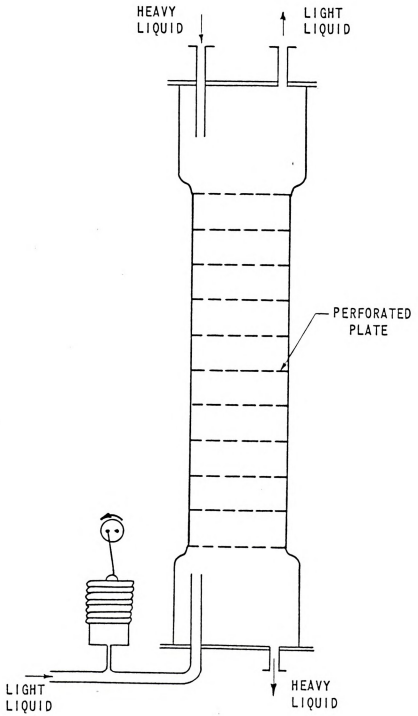


FIGURE 2-2
PULSED COLUMN CONTACTOR[11]

of mechanically-agitated columns are those which use a rotary device for mixing.

The best known and most commonly used rotary agitated columns, shown on Figure 2-3, are the Scheibel column,[12] the Rotating Disc Contactor (RDC),[13] and the Oldshue-Rushton column.[14]

Coalescence occurs only at the ends of the RDC and Oldhue-Rushton columns. In the Scheibel column, some settling takes place between stages and operation of this device is neither stagewise or differential, but a combination of the two.

In rotary agitated columns, the rotary mixing produces a more efficient dispersion of the phases. This type of agitation, however, may promote axial mixing. To minimize axial mixing, some form of baffling must be used.

The Scheibel column was the first mechanically agitated column to be introduced commercially and was quickly utilized by the petroleum industry. The original version of the device had mixing impellers located between layers of packing where coalescence occurred. Later designs used baffle arrangements to replace the packing.

In the RDC, the impellers, which are rotating discs, are located midway between static ring baffles attached to the shell of the column. These rings deflect the streams of liquid into the center of the column producing circulation

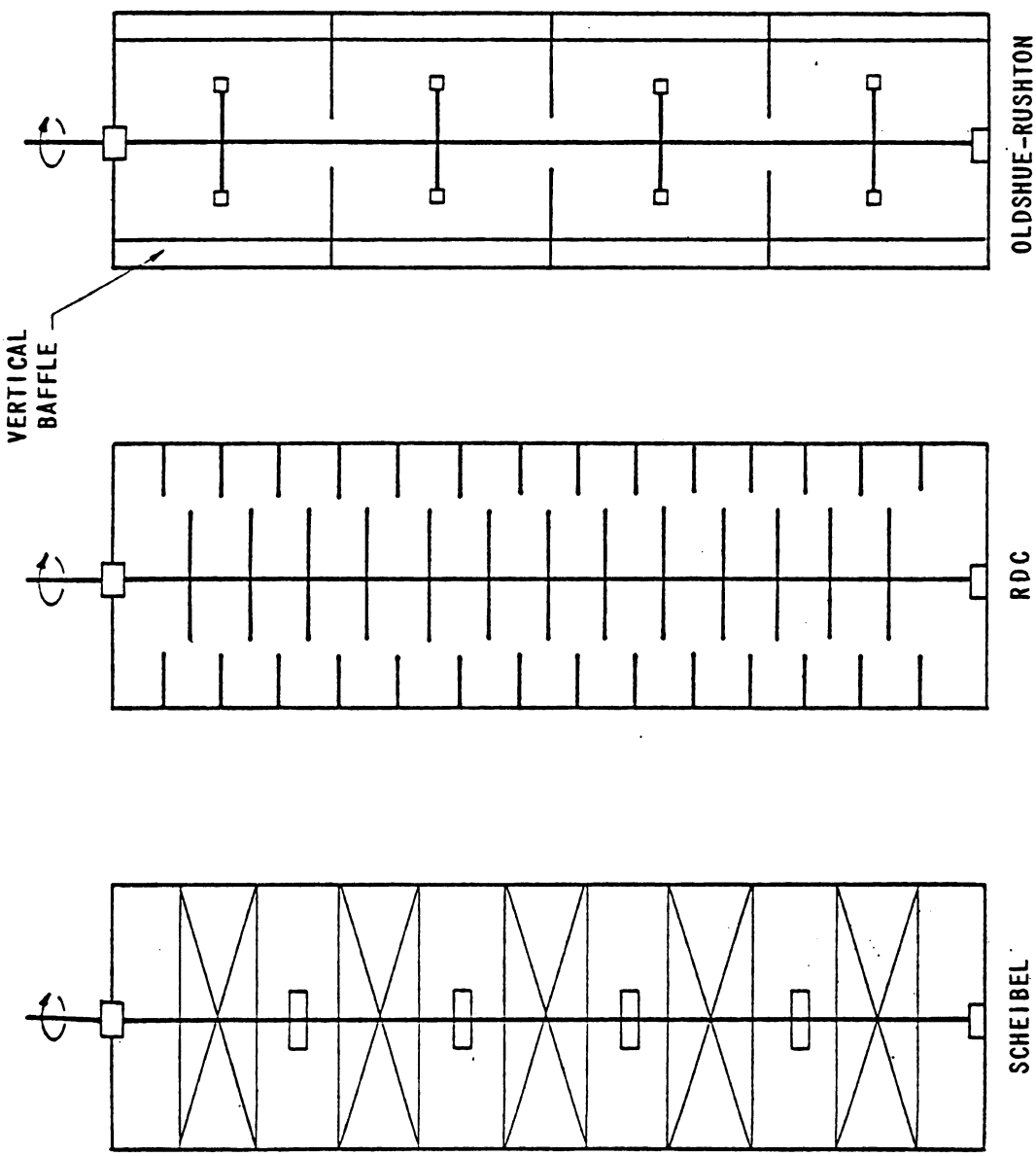


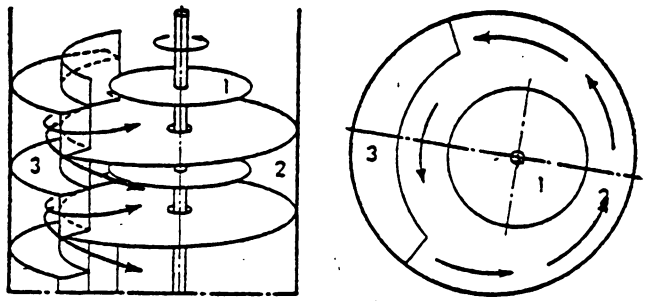
FIGURE 2-3
ROTARY AGITATED COLUMN CONTACTORS[5]

within each stage and reducing axial mixing. Phase dispersion is accomplished by the shearing action of the rapidly rotating discs. RDCs have been used throughout the world in the petrochemical industry to separate aromatics from aliphatics in the sulfolane process.

The Oldshue-Rusthon column is similar to the RDC in the use of ring baffles to inhibit axial mixing. Instead of using rotating discs, the Oldshue-Rushton device utilizes turbine-type impellers for agitation. Also, vertical baffles are placed along the shell wall within each stage to improve mixing efficiency.

In an effort to reduce axial mixing, columns have been developed which combine the efficiency of mixer-settlers with ground-space compactness of columns. Two designs were developed which have settling zones between each mixing chamber. This arrangement isolates the mixing chambers and greatly reduces axial mixing.

The Asymmetric Rotating-Disk extractor (ARD), shown schematically on Figure 2-4, is a semi-compartmentalized device with phase dispersion accomplished by means of rotating disks mounted on a shaft that is off center in the column. Settling zones, located at the side of the column, provide a path for interstage flow. It is claimed that the amount of axial mixing in the ARD is much less than that in other mechanically agitated devices. Axial mixing is not entirely eliminated, as some does occur through the settling zones.



1. Rotating disc rotor
2. Mixing zone
3. Settling zone

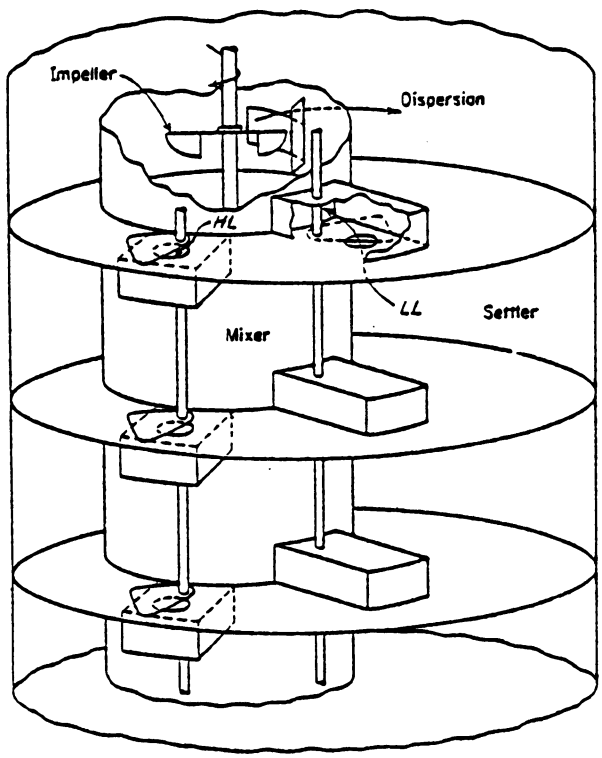
FIGURE 2-4
ASYMMETRICAL ROTATING-DISC CONTACTOR[9]

A similar type of contactor is the Treybal Liquid-Liquid Contactor,[15] shown on Figure 2-5. Originally developed as a vertical mixer-settler, it has been modified to operate in column form. This device is composed of two cylinders, the smaller set off center within the larger. Horizontal baffles divide the column into compartments. Mixing takes place in the smaller cylinder, with agitation being provided by a turbine impeller. The dispersion flows out of the mixing chamber into the outer annual chamber where coalescence takes place. The separated phases then flow countercurrently to their respective mixing chambers. The Treybal unit can produce high transfer efficiencies and flow capacity is comparable to that of the ARD extractor.

A design which combines the mixing provided in mechanically-agitated columns and the simplicity and flexibility of perforated plate columns is the Kuhni column. Featured in this unit are the use of double-shrouded turbine impellers and perforated stator plates.

2.4 Centrifugal Extractors

The contactors discussed to this point make use of gravity to effect both countercurrent flow and phase separation. The rate of countercurrent flow and phase separation can be increased by the application of a centrifugal force. This principle has led to the development of some very compact



Treybal extractor (schematic). Recycle provisions not shown. LL = light liquid, HL = heavy liquid. (U.S. Stoneware, Inc.)

FIGURE 2-5
TREYBAL LIQUID-LIQUID CONTACTOR

contacting units offering short contact times and the ability to separate dispersions which exhibit poor separation characteristics under normal conditions. Centrifugal extractors have been used extensively in the pharmaceutical industry.

The Podbielniak extractor[16] was the first centrifugal unit to be used on a wide-spread basis. It is mounted on a horizontal axis and consists of a rotating drum inside which are a series of concentric perforated cylinders. The light phase enters the drum under pressure through the horizontal shaft and flows via a channel to the periphery of the drum. The heavy phase is fed through the shaft to the center of the drum. The centrifugal force, acting on the density difference between the phases, causes the two phases to flow countercurrent to each other through the perforated cylinders. Being differential in nature, one unit can provide more than one theoretical stage.

The DeLaval extractor is a vertically mounted centrifuge bowl fitted with vertical baffles to create a complex spiral of channels from the center to the outside of the bowl. In the bowl, the heavy phase flows radially outward, while the light phase is displaced countercurrently toward the center. Orifices located alternately at the top and bottom of adjacent baffles allow the liquids to pass between concentric zones. As the phases pass through the orifices, shear forces create

a dispersion which then separates under centrifugal force before the next orifice is reached. It has been claimed that this arrangement can provide up to 20 theoretical stages in one unit.

The Westfalia Extractor consists of a vertical centrifugal bowl which can be divided into several separate chambers, one located above the next. Each chamber representing a separate stage. One advantage to this arrangement is that the light phase does not have to be fed under pressure. The maximum capacity depends upon the number of stages employed.

The Robatel Extractor utilizes a series of "mixer-settlers" stacked on their sides with mixing in each stage being carried out by a stationary disc on the shaft with the mixing chamber rotating about it. The two phases pass into the settling chamber before moving on to adjacent stages in countercurrent flow via a channel system.

The Quadronic design is a differential contactor which utilizes the concept of gradient mixing energy.

Centrifugal extractors have the advantages of being able to handle two phases with low density differences, having low hold-up, and short contact times. Also, they require little floor space and headroom. The disadvantages of these devices include high capital and operating costs and a limited number of stages in a single unit.

2.5 Mixer-Settlers

Mixer-settlers are the simplest liquid-liquid contacting equipment that are completely stagewise in operation. Some of the earliest commercial liquid-liquid extraction plants employed mixer-settlers. Early models used separate mixing and settling units with the dispersion flowing from mixer to settler by gravity. Interstage flow was carried out by pumps. A design which required only half as many pumps had the stages staggered in height so that the light phase could flow by gravity while the heavy phase was pumped from stage to stage. Both of these early models were reliable, flexible, and easy to design. The major drawbacks were the large ground-space required, the large inventory of chemicals necessary for operation, and the pumps and piping involved.

Because of the density differences between phases and the average density of the dispersion in the mixer, all interstage flow can be gravity driven. The Windscale design employed this principle and eliminated the need for interstage pumps. This design also eliminated the need for interstage piping because it was built as one box with partitions.

Although a major improvement over previous designs, the Windscale design also required a large chemical inventory. The driving force for interstage flow was a function of liquid depth and this inhibited chemical inventory reduction.

The flow limitations of the Windscale unit were overcome by interstage pumping like that provided in the Pump-Mix extractor.[17] In this design the mixing impeller also served as an interstage pump. This allowed an increase in throughputs without increasing the depth of the unit. The Pump-Mix extractor was an integral-box design, similar to the Windscale design.

The Pump-Mix design had the disadvantage that the pumping action of the impeller could apply a suction to adjacent stages and destroy the hydraulic independence of the stages. Also, use of this design reduced the flexibility of the units. Any change in phase ratio had to be coupled with an adjustment in the pumping units to avoid one phase being completely displaced from the settlers, with massive back mixing of the other phase being the result.

The hydraulic problems associated with the Pump-Mix extractor were overcome by the use of a draught tube mixer, shown on Figure 2-6. This design could handle a wide range of phase ratios and flow rates within the pumping capacity of the impeller. In the draught tube mixer, the two phases flow over their respective weirs and are mixed as they flow up the draught tube after which the dispersion flows by gravity to the settler. Changes in throughput or phase ratio are compensated for in the mixing chamber by the rising or falling of the liquid level in that vessel.

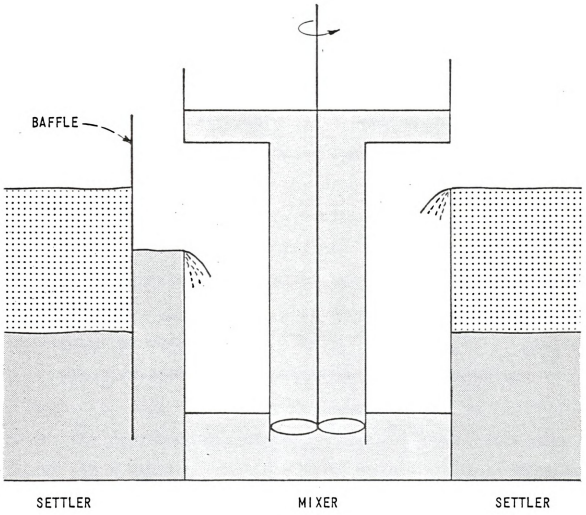


FIGURE 2-6
DRAUGHT TUBE MIXER[9]

Demand in the last decade for units capable of handling very high flowrates has results in much research and development work on mixer-settlers. Many different designs have evolved and each will be briefly discussed. A more complete discussion of mixer-settler design, performance, and operation is presented in Chapters 4 and 5.

The General Mills design,[18] shown on Figure 2-7, utilizes a round mixer fitted with vertical battles to minimize vortexing. The pumping impeller is a top-shrouded turbine. Following mixing, the dispersion flows by gravity through a trough to a settler equipped with a picket fence to reduce turbulence and distribute flow evenly across the width of the settler. A shallow settler is employed to reduce chemical inventory. This design is in commercial use for copper extraction.

The Davy-Powergas mixer-settler,[19] as shown on Figure 2-8, also uses a pumping impeller. In its basic form, the Davy-Powergas design includes a square-sectioned mixer and a shallow, rectangular settler. The feed streams enter the mixer through a draft tube where they are mixed by a double-shrouded turbine impeller. A horizontal plate with a central circular port is fitted in the top of the mixer as a vortex breaker. The mixed phases flow off the top of the mixer into the settler where a dispersion introduction

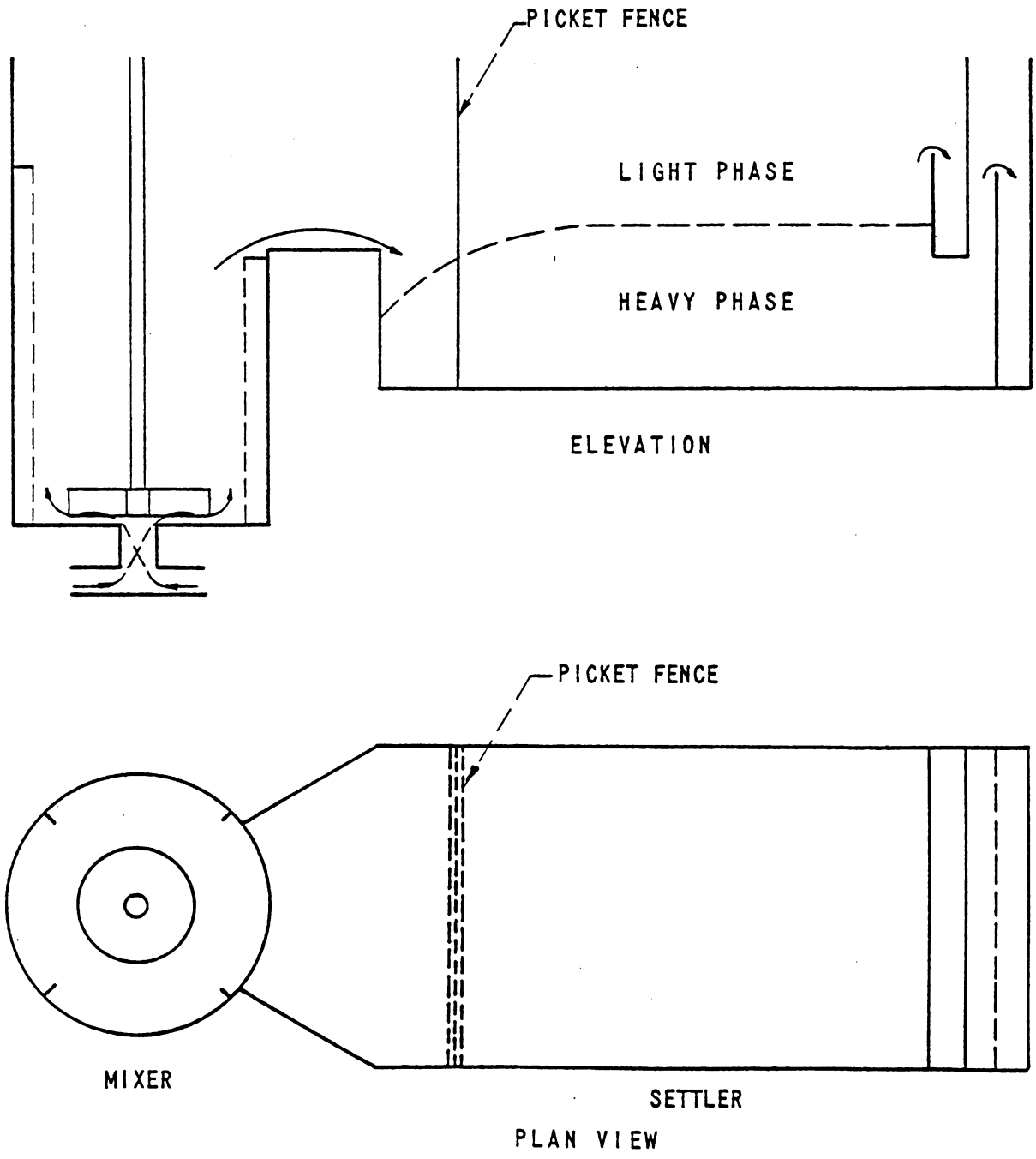


FIGURE 2-7
GENERAL MILLS MIXER-SETTLER(5)

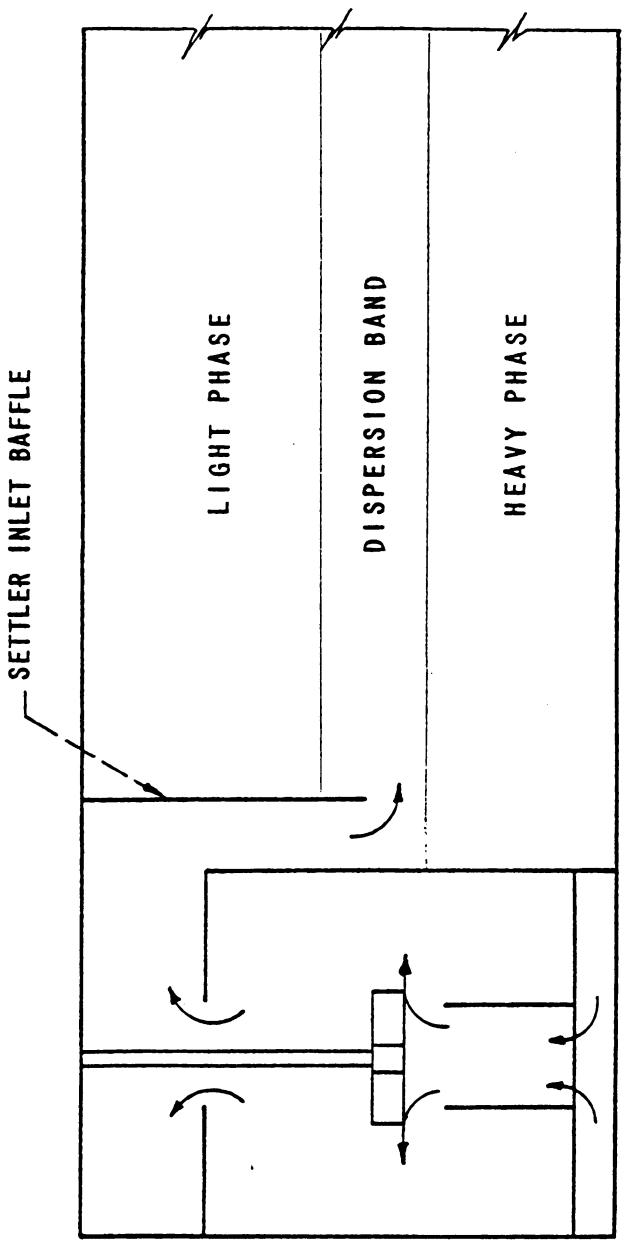


FIGURE 2-8
DAVY-POWERGAS MIXER-SETTLER[5]

baffle leads the dispersion to the proper depth in the settler. This design has been employed on a large scale for copper extraction.

The IMI mixer-settler,[20] as shown on Figure 2-9, was developed by Israeli Mining Industries, Ltd., and is currently being used for uranium extraction and phosphoric acid purification. Pumping and mixing are carried out by separate impellers mounted on the same vertical shaft in a cylindrical mixing compartment. The phases are first mixed and then drawn up a draft tube before being sent to the settler. The advantage of separating the functions of mixing and pumping is that each can be optimized individually. The dispersion enters the center of a cylindrical settler where the phases disengage radially outward. The separated phases are removed by weirs at the periphery of the settler. The settler is also fitted with turburlence reducing baffles.

The Kemira mixer-settler,[21] shown on Figure 2-10, also uses pumping and mixing devices that are separate but mounted on the same shaft. The dispersion is drawn from the mixer at a point in line with the mixing impeller. The cylindrical settler has a conical bottom from which the heavy phase is removed. The light phase is removed from the top of the settler where it flows by gravity to the next mixer. It is claimed that this design has the advantages of

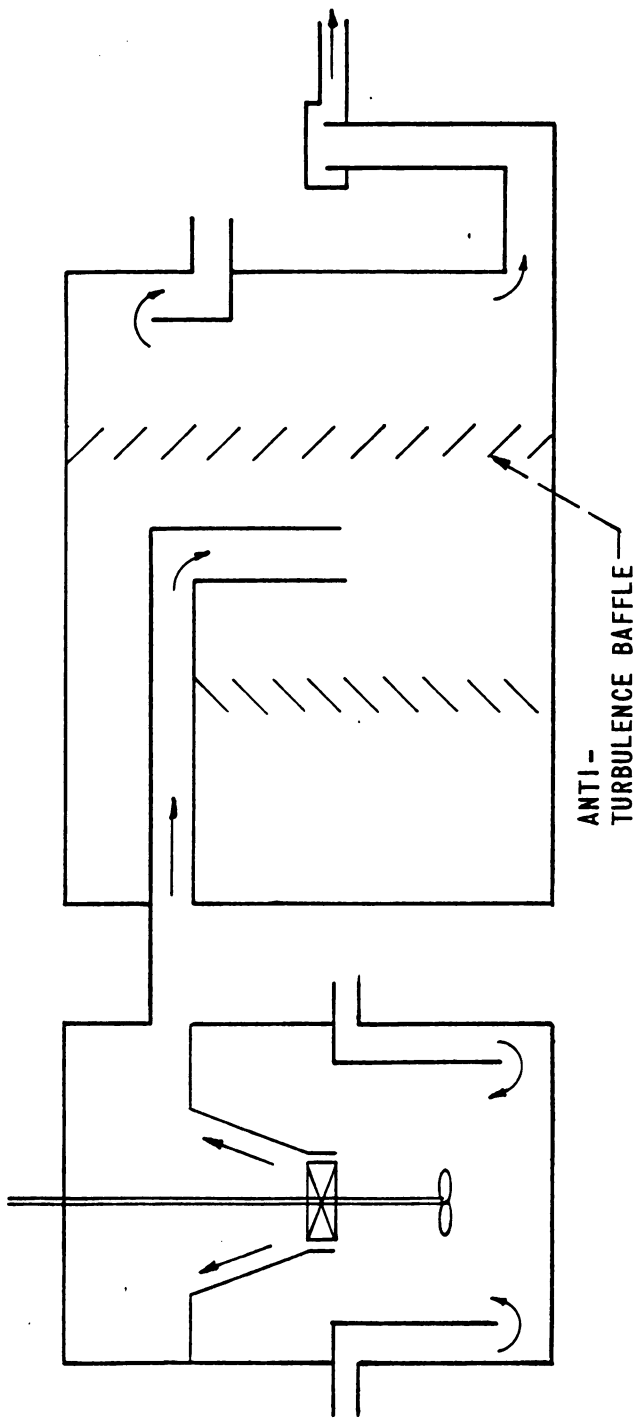


FIGURE 2-9
IMI MIXER-SETTLER[5]

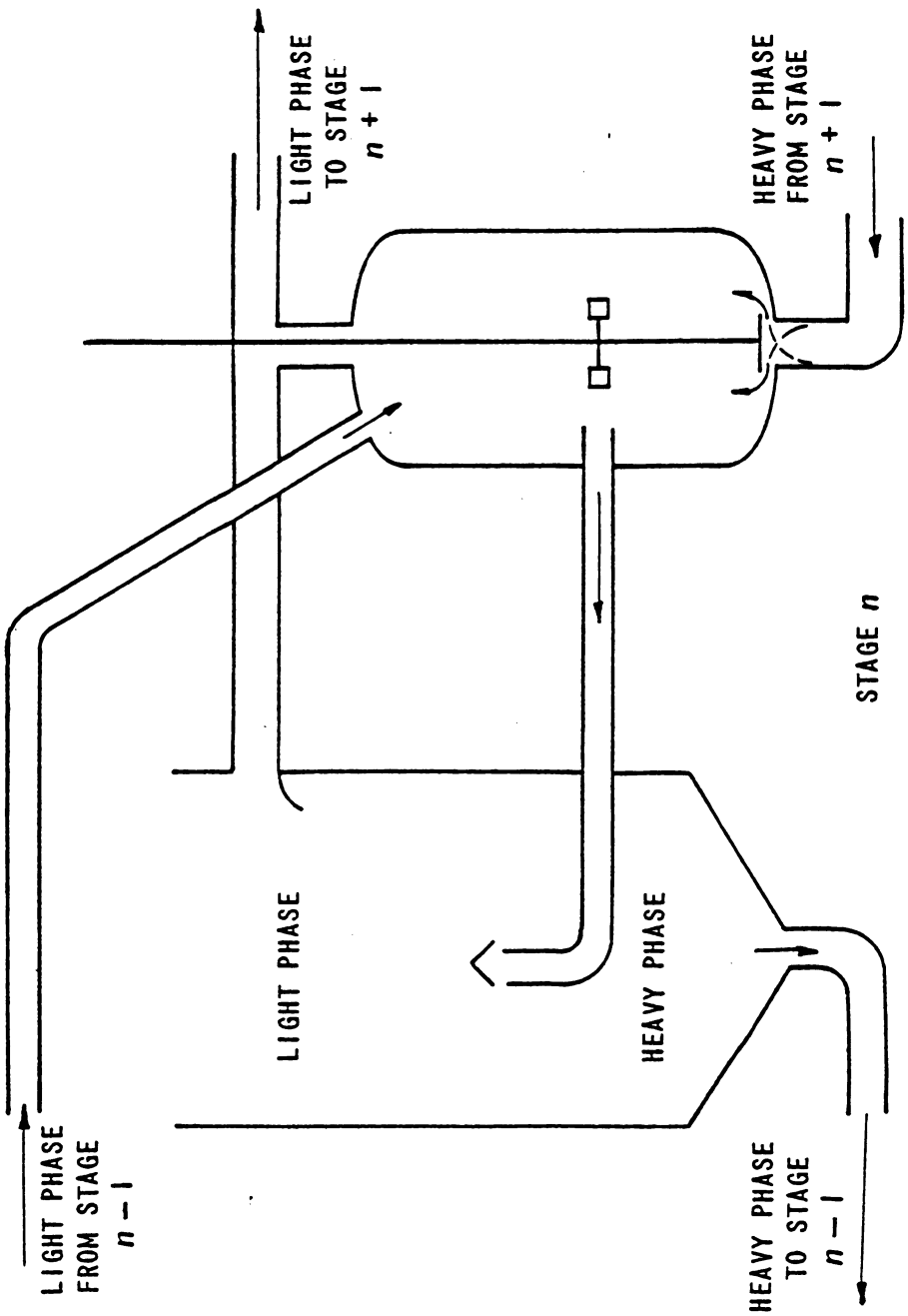


FIGURE 2-10
KEMIRA MIXER-SETTLER[21]

easy shutdown, easy start-up, and the elimination of unstable phase conditions caused by the contact of the phases prior to entering the mixing chamber.

Lurgi, of West Germany, has two designs which are currently being marketed.[22] One design is a stack of mixer-settlers forming a column. Settling compartments and interstage channels are within the column. All interstage flow and mixing is accomplished by axial-flow pumps on the outside of the column. The size of the settlers in this unit are smaller than those of other designs for a given flowrate and where applicable electrostatic forces have been used to promote phase separation. The other Lurgi design is a horizontal-type mixer-settler which employs an axial flow impeller to mix and pump. The settler is rectangular and fitted with multiple horizontal trays to allow higher settler throughputs.

Holmes and Narver, Inc., has revealed a mixer-settler design that includes multi-compartment mixers. Little information, however, is currently available on this design.

It is important to note that any mixer-settler design which has pumping impellers has the ability to recycle a phase from the settler to the mixer of a stage. This recycling ability can be a powerful tool for optimizing operating conditions within a single stage.

2.6 Other Contactors

There are several contactors which do not fall into any of the traditional categories. These are the RTL Contactor, the Morris Contactor, and the motionless mixer. Each will be briefly discussed below.

2.6.1 RTL Contactor

The RTL Contactor (formerly the Graesser Raining Bucket Contactor) shown on Figure 2-11, combines mixing and settling in the same compartment. Also, it is unique in that each phase is both continuous and dispersed. Laid out horizontally, this device has a series of plates mounted on a central shaft. Located between the plates are C-shaped buckets. This rotor assembly is fitted into a shell with a narrow gap between the shell and rotor discs. It is through this gap that lengthwise flow occurs. In operation, half the contactor is filled with heavy phase, the other half with light phase. As the rotor is slowly turned, the heavy phase is picked up and dropped through the light phase while the light phase is pulled down and bubbled up through the heavy phase. The mixing in the RTL Contactor is very gentle, making it attractive for systems with poor separation characteristics. Operation of a RTL Contactor will be discussed in Chapter 6.

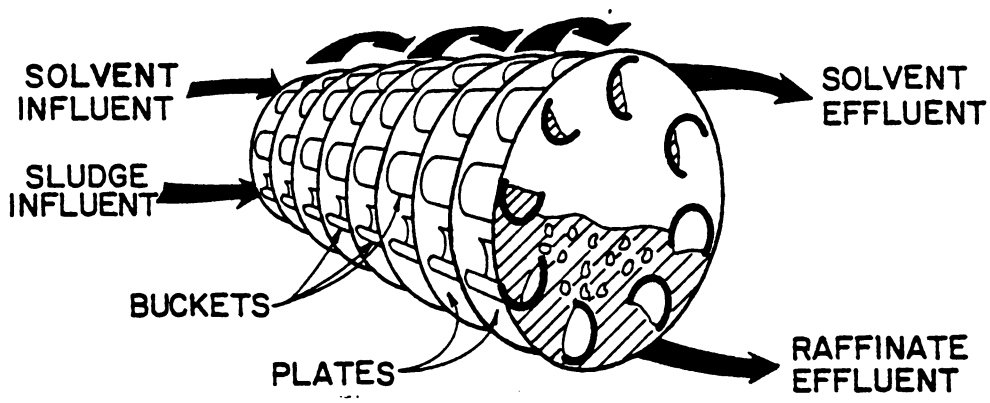


FIGURE 2-11
RTL CONTACTOR

2.6.2 Morris Contactor

The Morris Contactor,[23] shown on Figure 2-12, is a continuously agitated, horizontal unit comprised of a rectangular vessel divided into a series of compartments by alternate vertical weirs and baffles. The weirs and baffles are spaced such that narrow transfer sections are formed between two consecutive mixing sections. Each mixing section contains a vertically mounted impeller. Flow is countercurrent with each phase being introduced into the end mixing chambers at opposite ends of the contactor. If the light phase is dispersed, droplets are swept under the dividing baffle into the adjacent transfer section where they ascend. The heavy phase is swept over the weir in the opposite direction.

Under normal operating conditions, an interface exists only at the dispersed phase outlet. This helps avoid the phase separation problems which sometimes accompany mixer-settlers.

The Morris Contactor has been used in the manufacture of trinitro-toluene and the washing and lye treatment of soap curds.

Morris contactors are relatively cheap to build and are simple and flexible in operation. Throughput is said to be greater than that for comparable mixer-settlers because of

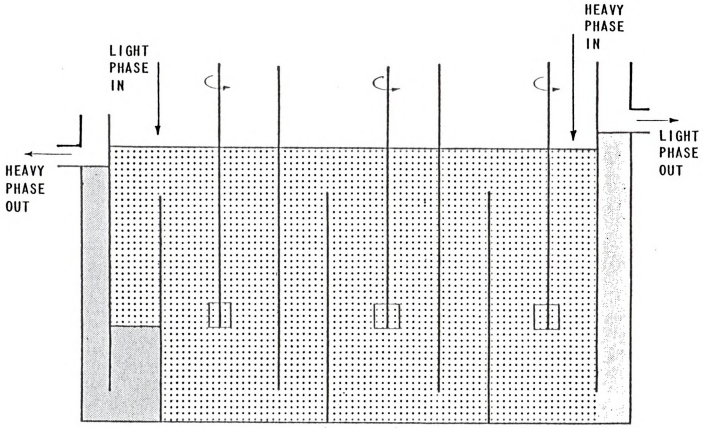


FIGURE 2-12
MORRIS CONTACTOR (9)

the absence of settler. It is the absence of these settlers, however, that causes back mixing of the continuous phase in Morris Contactors.

2.6.3 Motionless Mixer

A motionless mixer is a shell or pipe fitted with internal static elements which force multiple splittings of the feed streams as they pass through the mixer and effect intimate contacting. The geometry of the internal static elements is generally a proprietary feature of the many brands of motionless mixers on the market.

Motionless mixers usually require additional pumping head for feed streams. The additional pumping costs, however, can be offset by savings in operating and maintenance costs since the motionless mixers do not have any moving parts. Also, the static elements provide a predictable amount of mixing.

Copper is currently produced in Zaire at a liquid-ion exchange plant using motionless mixers as contactors. Other motionless mixer applications include the separation of cresylic acids and mercaptans from catalytic-cracker gasoline, the polymerizing of caprolactain in the production of nylon 6, and the hydrolysis of chlorinated organics. Units up to 5.25 feet in diameter have been reported.[24]

Motionless mixers may find applications in systems prone to the formation of emulsions because the mixing can be controlled much more closely than in mechanical mixers.

2.7 Summary

Many types of contacting equipment have been developed, each with their own advantages and disadvantages. They all, however, utilize differential or stagewise contacting principles. Variations in equipment that has evolved are generally specialized applications of one or both of the basic contacting principles. Table 2-2 lists the better known contactors, their advantages, disadvantages, and applications.

TABLE 2-2

CONTACTOR SUMMARY

Category	Type	Advantages	Disadvantages	Industrial Applications
Unagitated Column Contactor	spray column packed column perforated plate column	1. low capital costs 2. low operating costs 3. simple construction	1. limited throughput w/small density difference 2. cannot handle wide flow ratio 3. high headroom 4. low efficiency 5. difficult scale-up	hydrometallurgy saline water conversion
Mechanically Agitated Column Contactor	pulsed column reciprocating-plate column Scheibel column Rotating Disc Contactor Oldshue-Rushton column Asymmetric Rotating-Disc Extractor Treybal Liquid-Liquid Contactor Kuhni Column	1. good dispersion 2. reasonable cost 3. many stages 4. relatively easy scale-up	1. limited throughput w/small density difference 2. cannot handle emulsifying systems 3. cannot handle high flow ratio	petrochemical hydrometallurgy nuclear fuel wastewater treatment pharmaceutical fertilizer
Centrifugal Extractor	Podbielniak extractor DeLaval extractor Westfalia extractor Robatel extractor Quadronic	1. handle low gravity difference 2. low holdup volume 3. short holdup time 4. low space requirement 5. small inventory	1. high capital costs 2. high operating costs 3. high maintenance costs 4. limited number of stages in single unit	vegetable oil hydrometallurgy pharmaceutical wastewater treatment nuclear fuel

TABLE 2-2 (Cont'd)

CONTACTOR SUMMARY

Category	Type	Advantages	Disadvantages	Industrial Applications
Mixer-Settler	Windscale Pump-Mix draught tube General Mills Davy-Powergas IMI Kemira Lurgi Holmes & Narver	1. good contacting 2. handles wide flow ratio 3. low head room 4. high efficiency 5. many stages 6. reliable scale-up	1. large inventories 2. high power costs 3. high capital costs 4. large floor space	petrochemical hydrometallurgy nuclear fuel wastewater treatment
Other contactors	RTL Contactor	can handle emulsifying system	long detention times	coal chemicals, pharmaceutical, caprolactam
	Morris Contactor	low inventory	some backmixing	trinitrotoluene

CHAPTER 3

SELECTION OF CONTACTING EQUIPMENT

The batch optimization studies proved the feasibility of selectively extracting and recovering aluminum with liquid-ion exchange. It was then necessary to decide which type of contacting equipment would be best suited to carry out the recovery of aluminum.

The contactors had to be flexible, easy to operate, reliable, efficient, and relatively easy to scale-up. Flexibility was needed because of the many uncertainties which remained after the batch studies were completed. The batch tests were performed on synthetic aluminum solutions and the continuous flow studies would eventually evaluate raw alum sludges. It was suspected that the two feed stocks would have different operational characteristics and the contacting equipment had to allow a wide range of flowrates and phase ratios to be evaluated. Contactor performance is often a function not only of operating parameters but also of the size of the contactor. To ensure that full-scale performance could be reasonably predicted on the bench and pilot scale, an easily scaled-up contactor was desirable.

A review of industrial contactors revealed that mixer-settlers satisfied the requirements of the alum recovery process. Also, the successful use of mixer-settlers in

other hydrometallurgical applications, particularly those producing uranium and copper, confirmed the use of mixer-settlers for aluminum recovery.

The major drawbacks of mixer-settlers, mentioned earlier, were outweighed by the advantages. The corrections of the deficiencies of mixer-settlers were seen as refinements to equipment which had already been optimized.

The type of mixer-settlers chosen for the bench-scale study were horizontal, box-type units using pump-mix turbine impellers. The design is similar to the Davy-Powergas unit shown on Figure 2-7. The decision to use this type of mixer-settler was based on their simplicity and flexibility.

Later in the research it was decided to use an RTL Contactor (Figure 2-9) for reasons to become apparent.

CHAPTER 4

PILOT-PLANT DESIGN

4.1 Introduction

The design of the Tampa pilot plant was divided into three areas:

- Pilot-scale design data development
- Pilot program objectives and timetable
- Proposed operation and equipment design

Following the continuous flow, bench scale studies in which pilot-scale design data was generated, it was necessary to establish objectives of the Tampa pilot program. Once these objectives were defined, an operations program could be developed to obtain those objectives. When it was known how the plant was to be operated, the equipment to carry out those operations could be designed.

4.2 Pilot-Scale Design Data Development

4.2.1 Introduction

The design of the Tampa pilot plant was based on nine months of continuous flow studies on bench-scale mixer-settler units. It was felt that data on the following parameters

would be necessary to have in order to properly scale-up the process and the bench-scale program concentrated on optimizing these parameters:

1. Extractant Strength
2. Stripping Acid Strength
3. Number of Extraction Stages
4. Number of Stripping Stages
5. Extractor Dispersion Conditions
6. Stripper Dispersion Conditions
7. Extractor Phase Ratio
8. Stripper Phase Ratio
9. Extractor and Stripper Mixer Residence Times
10. Extractor and Stripper Mixer Impeller Speeds
11. Extractor and Stripper Settler Loading Rates

4.2.2 Extractant Strength

The extractant used, mono-di (2-ethylhexyl) phosphoric acid (MDEHPA), has been shown to be effective in selectively extracting aluminum from aqueous solutions.[3] That effectiveness was again demonstrated in the bench-scale testing.

The concentration of extractant needed is a function of the concentration of aluminum in the sludge to be treated. Based on an aluminum concentration of 1500 mg/l Al^{3+} in the Tampa sludge sample treated in the bench-scale studies, an

extraction concentration of 0.77 M was found to be necessary to effect the desired extraction.

4.2.3 Stripping Acid Strength

Batch experiments showed 6N H_2SO_4 to be the most effective stripping agent.[3] This solution was used successfully throughout the bench-scale program. On this basis, 6N H_2SO_4 was used as the stripping agent at the pilot scale.

4.2.4 Number of Extraction Stages

Two extraction stages were originally designed for the bench-scale unit. This was based on a McCabe-Thiele analysis of batch extraction isotherms.[3] The number of stages necessary was eventually reduced to one. There are two reasons why this reduction in stages was possible and necessary:

1. The synthetic feed stock used in the batch and bench-scale studies were fed to extraction circuit at pH 2.0. It has been shown that the efficiency of a stage can be increased by raising the pH of the feed stock.(3,25) Midway in the bench-scale program, the feed stock was switched to raw alum sludge from Tampa. The pH of the sludge was 6.9. This caused the stage efficiency to increase to the point where only one stage was necessary.

2. It was necessary to reduce the number of extraction stages to one because of a unique aspect of this application of the liquid-ion exchange process. Many extraction operations, i.e., copper extraction,(26) go to great lengths to eliminate solids from the solute feed stream. The economics of the alum recovery process are greatly enhanced if the raw sludge can be fed directly to the extraction

circuit. After extraction of the aluminum from the raw sludge, the dispersion flowing to the settler contains loaded extractant, raffinate, and solids which remain insoluble at the pH of the aqueous phase in the mixer. The solids collect at the settler interface and it would be a difficult, if not impossible task to efficiently move these solids on to the next extraction stage if more than one stage were being employed.

4.2.5 Number of Stripping Stages

A McCabe-Thiele analysis of batch stripping isotherms predicted that two stages would be necessary.[3] This was confirmed by bench-scale operations and two strip stages were designed into the pilot plant.

4.2.6 Extractor Dispersion Conditions

It was necessary to disperse the aqueous phase in the extractor regardless of any consequences which may have resulted from such a dispersion. When the aqueous phase was continuous, a stable emulsion formed which would not disengage by gravity means. This phenomena was observed in batch testing when contacting the Tampa sludge with the solvent.[3] Based on the problems associated with aqueous continuous operation of the bench-scale extractor, the pilot extractor was to be operated organic continous.

4.2.7 Stripper Dispersion Conditions

Minimizing organic contamination of the recovered alum was the overriding consideration in deciding which phase to disperse in the strippers. It has been found that organic entrainment is lowest under organic continuous conditions. It is believed that under organic continuous conditions the entrainment of organic in the aqueous phase is a function of phase disengagement and settler operation, rather than mixer operation.[28] Based on the confirmation of this in the bench-scale studies, the mode of operation in the strippers was to be organic continuous.

4.2.8 Extractor Phase Ratio

In order to minimize mixer volume and solvent pumping rates, a low solvent to aqueous ratio was sought. A phase ratio of 2:1 was chosen as the design value. While it may have been advantageous to operate at a phase ratio lower than 2:1 with respect to aqueous entrainment,[27] it was found that operation at phase ratios less than 2:1 invited phase inversions which created the stable emulsion. The 2:1 phase ratio was not necessary from an extraction efficiency standpoint. Therefore, to minimize pumping rates, the stripped organic feed rate would be equal to raw sludge feed rate. The phase ratio of the mixer would then be raised to 2:1 by recycling loading organic from the settler to the mixer.

4.2.9 Stripper Phase Ratio

Batch tests indicated that a phase ratio of 3:1 was the most efficient for stripping.[3] A 2:1 phase ratio was most effective at the bench scale and this was the phase ratio on which the design of the pilot strippers was based. Since the organic flow entering the bench-scale stripping circuit was typically 30 times higher than the acid flow, it was necessary to recycle a portion of the settler aqueous effluent to the mixer to obtain a 2:1 phase ratio.

4.2.10 Extractor and Stripper Mixer Residence Times

A mixer residence time of 15 minutes was found to be optimum for both extracting and stripping.[3] This residence time was successfully used throughout the bench program and was chosen as the design residence time on the basis of that success.

4.2.11 Extractor and Stripper Mixer Impeller Speeds

An impeller tip speed of 800 ft/minute was found to be optimal in batch studies in both the extracting and stripping processes.[3] Used throughout the bench program without adverse affects, this tip speed was used for the design of the pilot mixers.

4.2.12 Extractor and Stripper Settler Loading Rates

Developing a basis for settler scale-up was most difficult. Settlers are normally designed on the relationship between the throughput of dispersion per unit surface area of the settler and the dispersion band thickness. However, the presence of the solids emulsion at the settler interface, prevented any measurement of dispersion band thickness. Lacking traditional design parameters, it was decided to design the pilot settler using the loading rates of other settler designs. In particular, the scale-up of a successful copper extraction bench unit used a settler loading rate of 2.0 gal/min-ft^2.[6] On the basis of the success of this plant, 2.0 gal/min-ft^2 was used as the loading rate for the design of the pilot extractor settler. This loading rate was also used for the design of the stripper settlers.

4.3 Pilot Program Objectives and Timetable

The three main objectives of the Tampa facility were:

- Process optimization
- Demonstration of economic feasibility
- Generation of full-scale design data

Initially, system performance would be evaluated at design operating conditions. Once performance at these conditions was established, any modifications needed to raise the level

of performance to design specifications would be made. The number and severity of modifications needed would be a measure of the success of the scale-up. If the important parameters were identified and optimized at the bench scale and sound methods of design were used, there should have been little difference between the performance of the bench and pilot units. Thus, an important part of process optimization would include scale-up evaluation and the identification of any operating parameters important to scale-up which were overlooked at the bench scale.

Once the system had been optimized, the recovery plant would be operated to generate data related to the operating costs of the system. As operating cost data were developed, the operation of the pilot plant would be refined so that the most cost-effective method of operation could be found.

With the system economically optimized, data necessary for the design of full-scale equipment would be generated. With this data, the full-scale equipment could be sized and capital costs determined.

The pilot plant was to operate for six months; the first three months were to be devoted to making any modifications necessary to bring the process to design specifications and to optimize operating parameters. During the last three months, the system was to be operated at optimal conditions in order to develop operating and full-scale design data.

4.4 Proposed Operation and Pilot Equipment Design

4.4.1 Proposed Operation

4.4.1.1 Introduction

The Tampa alum recovery facility was designed on the assumption that the extraction and stripping circuits would operate continously, treating 10 gpm of raw alum sludge. The characteristics of the sludge as determined at the bench scale included a solids concentration of 0.8 percent and an aluminum concentration of 1500 mg/l al^{3+} at pH 2.0. While variations in sludge quality were expected, these values were chosen as representative for design purposes. How these basic assumptions apply to the liquid-ion exchange process will be detailed below.

4.4.1.2 Extraction Circuit

The single extraction stage would operate organic continuous at a phase ratio of 2:1 with a detention time of 15 minutes. The feed flow would be 1:1, the additional organic being recycled. The treatment of 10 gpm of sludge would produce approximately 2 gpm of bleed solids. These would collect at the extractor settler interface where a siphoning manifold would remove and transport the solids to the solvent recovery operation.

The raffinate would flow by gravity into the Tampa Water Treatment Plant sludge handling system.

Feed sludge would be pumped from a mixed sludge holding basin offering a representative sampling of sludge. Stripped organic was to be pumped to the extraction circuit from an organic reservoir.

Process control would be achieved by monitoring the following parameters:

- Solute and organic feed flows
- Recycle flow
- Mixer phase ratio
- Raw sludge characteristics
- Stripped and loaded solvent characteristics
- Raffinate characteristics

4.4.1.3 Stripping Circuit

The two strip stages would operate organic continuous at a phase ratio of 2:1. The detention time would be 15 minutes. The solvent to acid feed flow ratio would be 29:1, requiring recycle of the aqueous phase within each stage.

The 6N H_2SO_4 used as the stripping agent would be mixed in-line prior to being pumped into the stripping circuit.

The recovered alum would be passed through a granulated activated carbon (GAC) column to remove any color or entrained organic which may be present. The alum would then flow by

gravity to a day tank from which it would be pumped into the water treatment plant's existing alum storage tanks.

The stripped solvent would flow by gravity from the stripping circuit to the solvent reservoir.

Control of stripping would be maintained by monitoring:

- Stripping agent flow
- Stripping agent strength
- Recycle flows
- Mixer phase ratios
- Solvent characteristics before and after each stage
- Recovered alum characteristics

4.4.1.4 Solvent Recovery

Because of the large amount of solvent contained in the bleed solids siphoned out of the extractor settler, economics does not allow bleed solids disposal without recovery of the solvent. The bench-scale studies had a great deal of success recovering solvent with a solid bowl centrifuge. For this reason a centrifuge would also be used for solvent recovery at the pilot plant. To reduce maintenance and operating costs the centrifuge would operate only 8 hours per day. During the other 16 hours the bleed solids would be stored in a holding tank. The recovered solvent would be returned

to the extraction circuit via the organic recycle line, while the residual waste solids would be disposed of with the raffinate.

4.4.1.5 Personnel Requirements

It was felt that after any modifications were made and the system was brought to steady-state, operation of the system would require only one full-time person to operate the centrifuge. During the other two shifts, an hourly or bi-hourly inspection of the alum recovery system by water treatment plant personnel would be sufficient for proper operation.

In addition to the operation of the equipment, detailed operations and chemical inventory logs would need to be maintained.

4.4.2 Pilot Equipment Design

4.4.2.1 Extractor Mixer

Using a residence time of 15 minutes, a solute feed flow of 10 gpm, and an organic to aqueous ratio of 2:1 (the organic feed to be equal to the solute feed, a 2:1 phase ratio to be achieved by recycling loaded organic), the pilot mixer was designed by maintaining geometric similitude with the bench mixer. The volume of the mixer is found by:

$V = [10 + 2(10)]gpm(15\ min) = 450\ gals = 60.2\ ft^3$.

Making the mixer cubic in shape yields the dimensions, 47" x 47" x 47", plus 7" freeboard.

4.4.2.2 Extractor Impeller

The turbine impeller chosen for the extractor mixer consisted of six, flat, top-shrouded, radial vanes.[19] The length of the vanes was equal to the shroud radius. Entrainment levels are lowest for turbine impellers when $N^3D^2 \leq 20$, where,

N = rotational speed of impeller, rps

D = diameter of turbine, feet

Maintaining a tank width to impeller diameter of approximately 2:1, yields an impeller diameter equal to 28 inches. Solving for the rotational speed of the impeller,

$$N^3 = 20/D^2$$

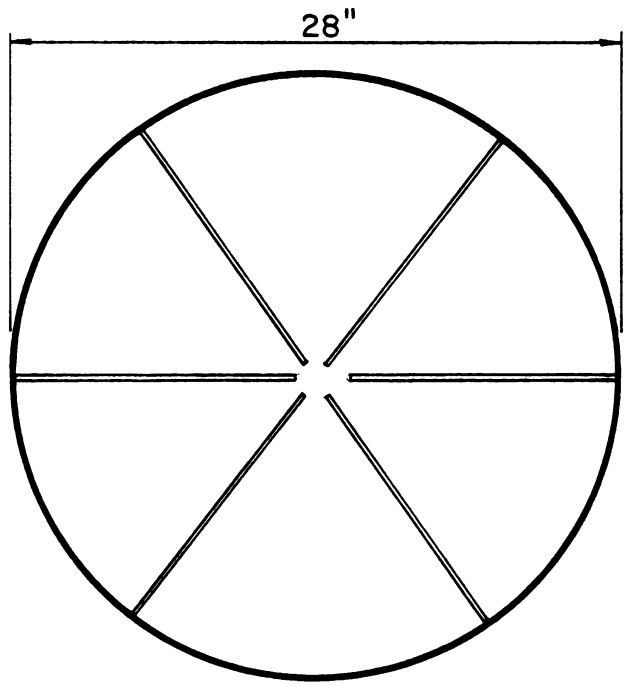
$$N = 20/(2.33)^{2\ 1/3}$$

$$N = 1.54 \text{ rps} = 93 \text{ rpm}$$

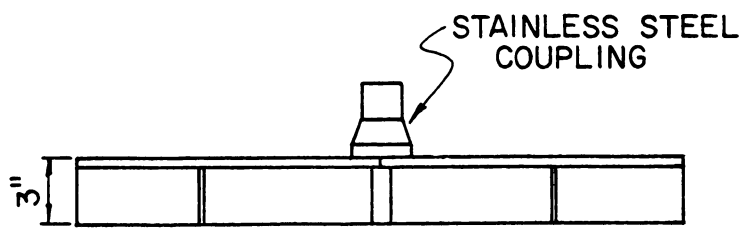
However, the tip speed for this impeller at 93 rpm is 680 ft/min. It was felt at this time that a trade-off could be made between impeller speed and entrainment, so the extractor impeller was designed at 680 ft/min instead of 800 ft/min. Shown on Figure 4-1 is the impeller used in the extractor.

4.4.2.3 Extractor Settler

The function of a settler is to efficiently separate a dispersion of two or more phases. In the alum recovery



PLAN



ELEVATION

FIGURE 4-1
EXTRACTOR MIXER IMPELLER

process the extractor settler must handle three phases: the loaded extractant, raffinate, and solids which remain insoluble during extraction. Due to the intensity of mixing developed during extraction, a large amount of solvent and raffinate become attached to the solids. This emulsion, termed bleed solids, collects at the top of the raffinate in the settler, but does not cross the interface into the solvent. The Tampa sludge sample tested in the bench-scale unit exhibited this type of behavior for the duration of the evaluation period. The presence of the bleed solids undoubtedly had a detrimental effect on phase separation but it did not hinder settler operation to the point where coalescence failed to occur. Since the solids accumulated downward into the raffinate, the volume of solvent in the settler never decreased, and hence, the mean velocity of the solvent through the settler never increased for a given solvent flow. An increase in the mean velocity of the solvent may have led to a washing out of dispersed raffinate and solids into the stripping circuit. However, no contamination of the recovered alum with raffinate or solids was observed. While the bench settler was oversized to accommodate several hours storage of bleed solids, it was not over-designed to the point where secondary dispersions of raffinate and solids would have settled out. Based on the bench-scale

findings, the bleed solids could be tolerated if they were removed periodically. The pilot settler was designed accordingly.

Given the design sludge flow of 10 gpm and a design mixer phase ratio of 2:1, the total flow of dispersion to the settler is 30 gpm. The surface area of the settler is then:

$$\frac{30 \text{ gpm}}{2 \frac{\text{gpm}}{\text{ft}^2}} = 15 \text{ ft}^2$$

Using a length to width ratio of 2:1 yields settler area dimensions of 32 3/4" x 65 3/4".[19] The depth of the settler was set at 40 inches plus 7 inches freeboard. This was based on the maintenance of about one-foot layers of raffinate, organic, and bleed solids. While much effort has been directed at minimizing the size of settlers, there were no restraints on the size of the units to be used at Tampa. Thus, priority was given to ensuring efficient phase separation, not minimizing settler size.

A dispersion introduction baffle was installed to assist in phase separation.[20] A proven aid in improving settler performance, the introduction baffle covers the full width of settler and extends downward to the level where phase disengagement is taking place. Located five inches from the mixer outlet, the baffle allows the dispersion to be introduced to the dispersion band by providing a route

down through the bulk organic phase after leaving the mixer. The dispersion introduction baffle eliminates turbulence and re-entrainment of the phases in the vicinity of the settler inlet.

Another settling aid employed was a picket fence. Effective in breaking up turbulence and distributing flow evenly across the width of the settler, picket fences have reduced entrainment by 50 percent in some installations.[19,20] The "fence" was made of a row of vertical bars stretching across the width of the settler. The space between bars being 1/2 inch. This row was followed by another row of bars located in such a way that the spaces of the first row are covered by the bars of the second row. The gap between the two rows of bars being 1/4 inch. The picket fence extended to the bottom of the settler and was located five inches downstream of the dispersion introduction baffle. Shown on Figure 4-2 is the extractor mixer-settler used at Tampa.

4.4.2.4 Extractor Settler Weir System

The weir system used to remove the separated phases is shown on Figure 4-2. The fixed organic weir was located at a level which allowed seven inches of freeboard in the settler. The aqueous weir was movable, allowing the level of the interface to be controlled. The use of full width

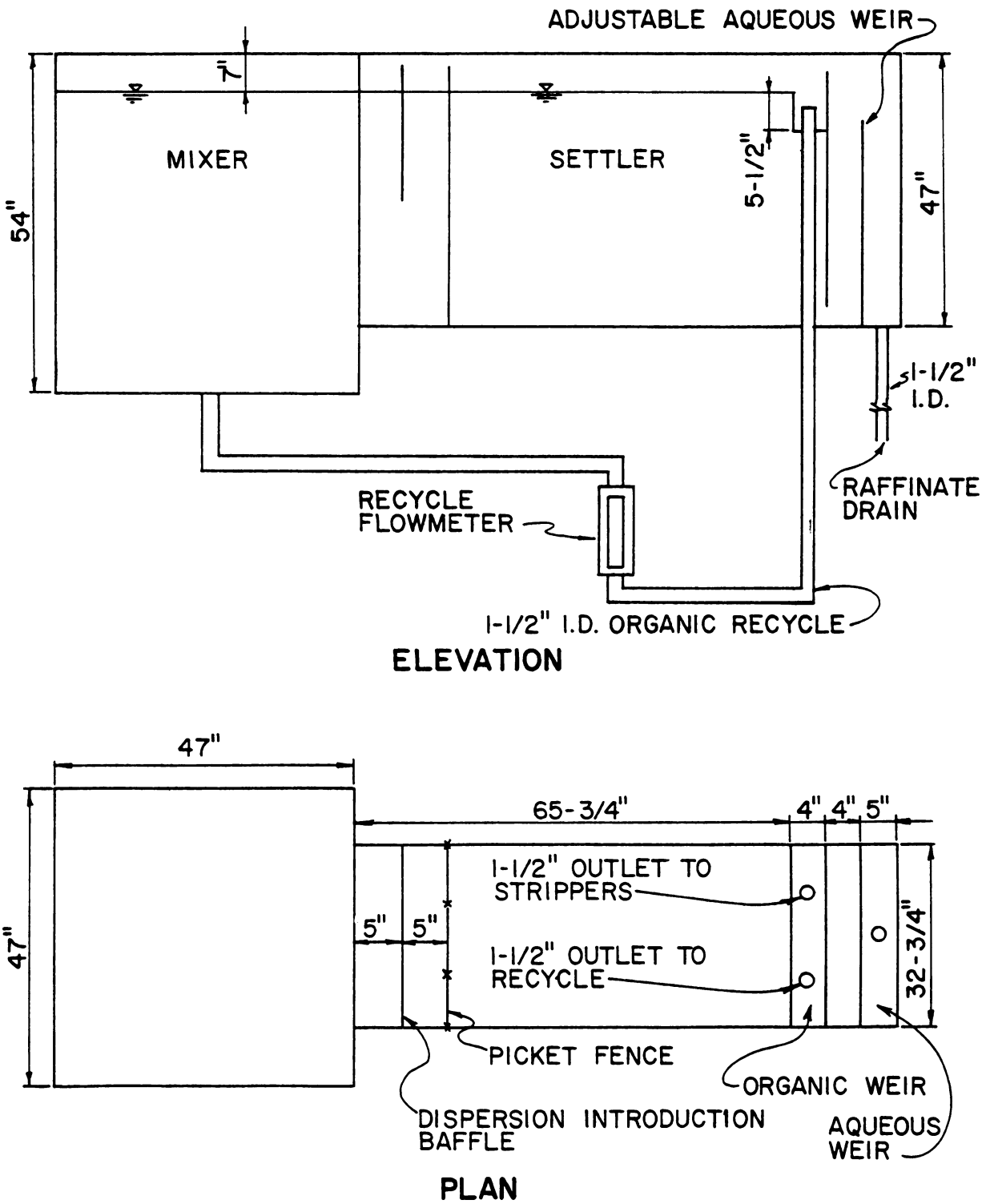


FIGURE 4-2
EXTRACTOR MIXER-SETTLER

weirs is recommended because discharge to a pipe or reduced width weir would cause the velocity of flow to that point to increase and this can reduce the effectiveness of the settler.[19,20]

To determine the size of pipe into which the weirs discharged, the following equation was used:

$$H = 1.1\ Q^2/A^2g$$

where H = depth of liquid in weir, ft

A = area of pipe, ft^2

Q = flow, cfs

Different pipe areas were inserted into the equation until a suitable depth was found. It should be noted that the use of this equation assumes that the only force acting on the liquid is gravity. In the case of this mixer-settler, the pumping action of the impeller of the mixer to which the orifice in question is connected to will also act on the liquid. Hence, the result of this equation represents a "worst case" situation and was used because it offers a conservative answer.

In the case of the organic weir, it would have two orifices, one leading to the strip circuit and the other to the organic recycle line. Each orifice must pass 10 gpm. Using a pipe with a 1-1/2-inch diameter resulted in an acceptable liquid depth in the weir of 1.1 inches.

$$H = \frac{1.1(.02)^2}{32.2(.01)^2} = 0.09 \text{ ft} = 1.1 \text{ inches}$$

The orifice of the organic recycle line was flush with the bottom of organic outlet weir, while the orifice of the pipe leading to the strip circuit was raised three inches above the bottom of the weir. This arrangement assured that the organic recycle line would always be flooded so no air could be entrained into the recycled organic. This entrained air could have a detrimental effect on coalescence.

The velocity of flow in the organic recycle pipe would be 1.6 ft/sec. The head loss through the recycle line would be approximately 0.25 feet or three inches. While this loss is equal to the head on the recycle line developed in the weir, it was considered acceptable in light of the pumping capacity of impeller.

A 1-1/2-inch pipe was used in the aqueous weir; however, its sizing is not as critical as that in the organic weir because of the large amount of head available.

4.4.2.5 Solvent Feed Pump

The head requirements of the solvent pump were negligible. Also, it was felt the pumping action of the impeller would not have a detrimental effect on the pump. A Cole-Parmer Instrument Company No. 7087-40 high capacity centrifugal pump was chosen to feed the solvent. Flow would

be controlled by means of a valved recycle around the pump and would be measured by a Fisher & Porter 10A2227A Series all metal rotameter.

4.4.2.6 Sludge Feed Pump

The sludge feed pump would have to overcome 15 feet in losses due to 300 feet of 1-1/2-inch plastic hose which would serve as the sludge feed line and a five-foot suction lift. To meet these demands, a Multi-Duti Manufacturing Company No. 1131.31 centrifugal pump was selected. This pump had a rating of 10 gpm at 80 feet of head. Sludge flow was controlled by a valved recycle and measured by a Badger Meter Recordall Model 15 water meter.

4.4.2.7 Extractor Organic Recycle

Organic recycle flow would be controlled by a gate valve and measured with a Blue-White, Inc., 0-20 gpm flowmeter.

4.4.2.7 Stripper Mixers

Two strip stages were required at Tampa, the dimensions of each being identical. Using a residence time of 15 minutes, an organic flow of 10 gpm, and a phase ratio of 1:1, the pilot mixers were designed by maintaining geometric similitude with the bench-scale mixers. Since the acid flow

to the strippers would be only 0.34 gpm, aqueous would have to be recycled to achieve a 2:1 phase ratio. While the strippers would be operated at 2:1, they were designed to accommodate a 1:1 phase ratio. The mixer volume is found by:

$$V = [10 + 1(10)]gpm(15\ min) = 300\ gals = 40.1\ ft^3$$

A cubic mixer would have the dimensions of 41" x 41" x 41", plus 6" of freeboard.

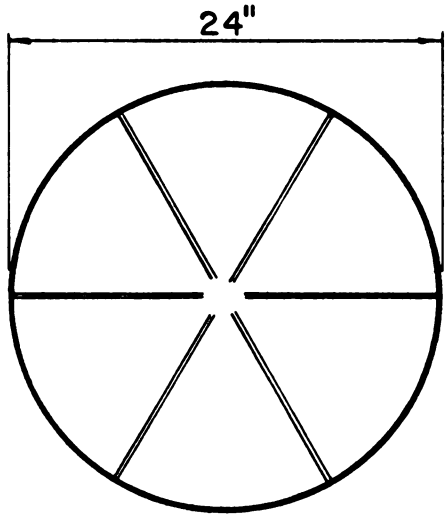
4.4.2.9 Stripper Impellers

The type of impeller chosen for the strippers was the same as for the extractor. The impeller diameter was set at approximately one-half the tank width or 24 inches. The rotational speed would found by keeping $N^3D^2 \leq 20$.

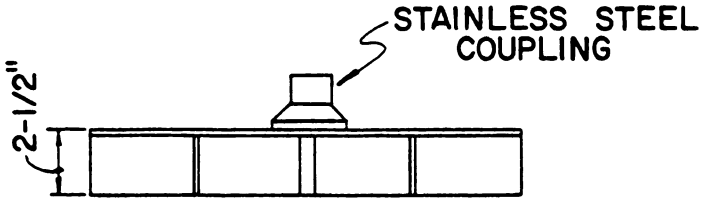
Rotational speed of impeller, $N = (20/2^2)^{1/3}$

$$N = 1.71\ rps = 103\ rpm$$

A rotational speed of 103 rpm yields a tip speed of 647 ft/min. As with the extractor, it was felt that some mixing could be sacrificed in order to lower entrainment, so the pilot mixers were designed at 647 ft/min instead of 800 ft/min. Shown on Figure 4-3 is the impeller installed in the strippers.



PLAN



ELEVATION

FIGURE 4-3
STRIPPER MIXER IMPELLER

4.4.2.10 Stripper Settlers

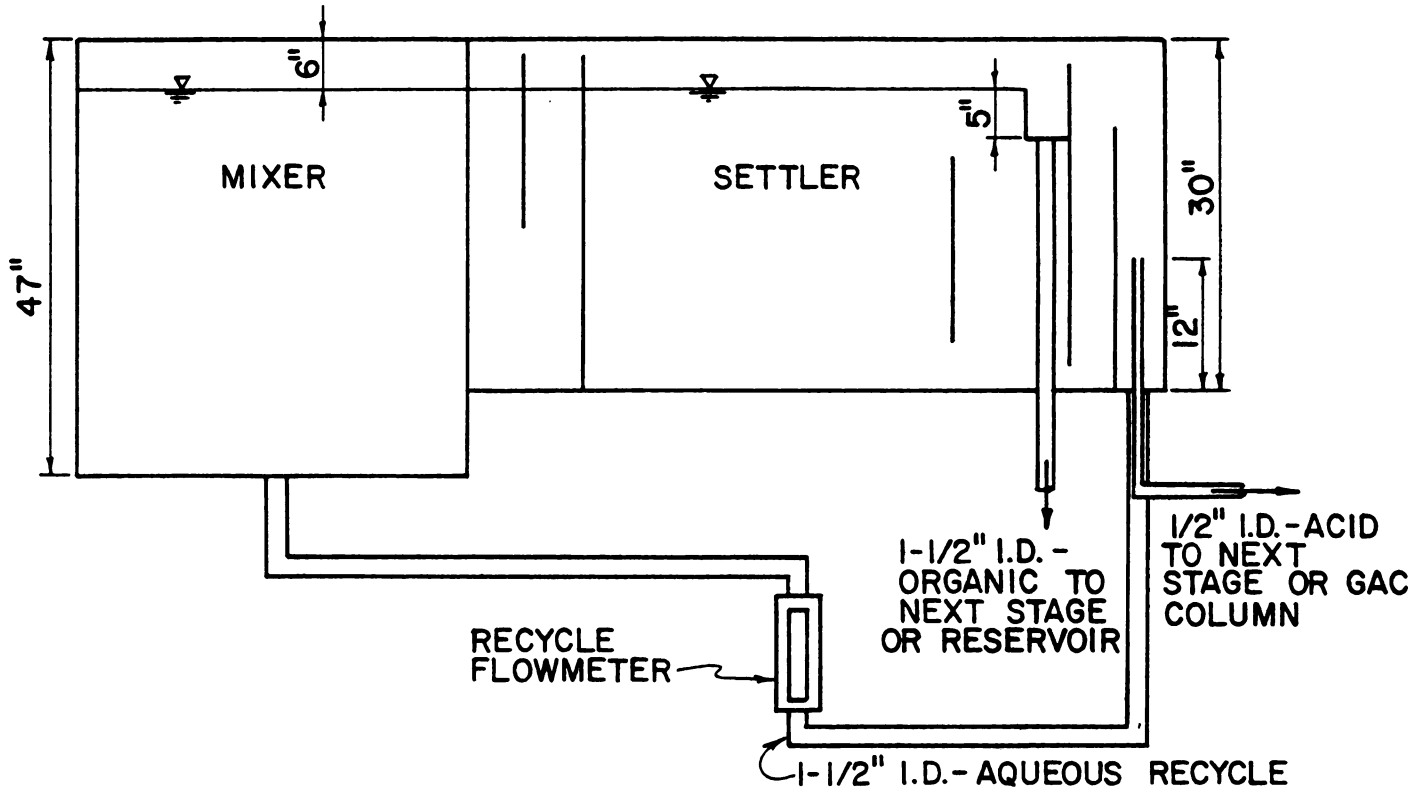
The stripper mixer-settlers used at Tampa are shown on Figure 4-4. Unlike the extractor settler, the stripper settlers had to separate only two phases, acid and solvent. Some crud did accumulate at the Strip I settler interface in the bench-scale unit. These solids consisted mainly of Ca_2SO_4 and regular cleaning of the interface kept this crud from interfering with coalescence. The interface of the Strip II settler remained crud-free throughout bench-scale evaluation of the Tampa sludge. It was not expected that crud accumulation would cause any great problems at the pilot scale and was not given any special consideration in the design of the settlers.

Given the design solvent flow of 10 gpm and a phase ratio of 1:1, the total flow of dispersion to the settler was 20 gpm. The surface area of the settler is found by:

$$\frac{20 \text{ gpm}}{2 \frac{\text{gpm}}{\text{ft}^2}} = 10\text{ft}^2$$

A length to width ratio of 2:1 yields settler area dimensions of 27" x 53". The depth of the settlers were set at 24 inches plus 6 inches of freeboard. This was based on one-foot layers of aqueous and organic phases.

The same settling aids used in the extractor settler were used in the strip settlers. The dispersion introduction was located four inches downstream of the mixer outlet. The



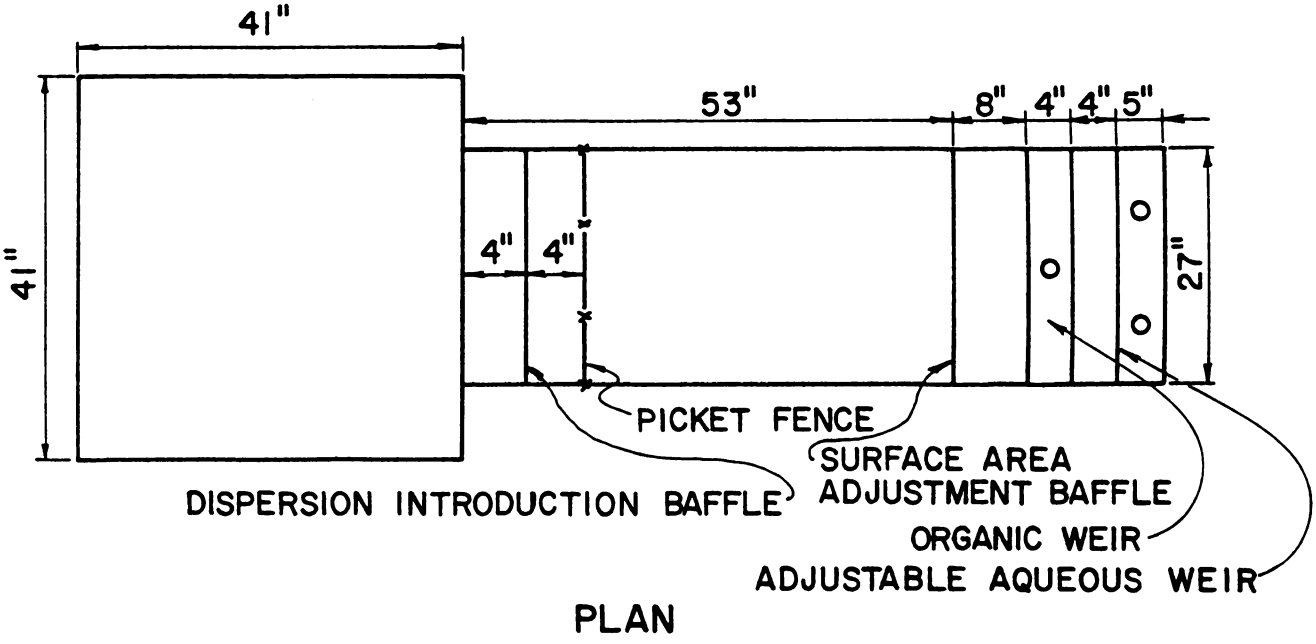


FIGURE 4-4
STRIPPER MIXER-SETTLER

picket fence was located four inches downstream of the introduction baffle. The space between vertical bars was equal to 1/2 inch and the gap between the two rows of bar was 1/4 inch.

Also added to each of the strip settlers was a baffle which intercepted the dispersion band near the effluent end of the settler. When the baffle was lowered, a 8.5 ft^2 surface area could be evaluated. When raised, the original 10 ft^2 surface area could be investigated.

4.4.2.11 Stripper Settler Weir System

The design of the stripper weirs followed that of the extractor weirs. Since no organic is recycled in the strippers, only one outlet is needed in the organic weirs and 10 gpm must flow through that outlet. The organic weirs were dimensioned as shown on Figure 4-4.

The aqueous weirs differed from those in the extractor in that a portion of the aqueous flow would be recycled to the mixer. The recycle flow is a function of feed flow ratio and mixer phase ratio. Given an organic feed flow of 10 gpm, an aqueous feed flow of 0.34 gpm, and seeking a mixer phase ratio of 2:1, it can be seen that 4.7 gpm of aqueous must be recycled. Using the same analysis as in 4.4.2.3, a 1-1/2-inch diameter orifice and pipe was found to be suitable for transmitting the recycle flow. The velocity

in the recycle line would be 0.81 ft/sec with a head loss of one inch. This loss is well within the pumping capacity of the impeller. The other outlet in the aqueous weir is that which passes the stripping agent to the next stage or out of the stripping circuit. A 1/2 inch diameter outlet is more than sufficient for this purpose. The aqueous weir arrangement, shown on Figure 4-4, serves two purposes; first, it means that at steady state, the recycle lines will always be flooded, and second, it allows a large amount of head to build up on the recycle line ensuring proper recycle performance.

4.4.2.12 Stripping Acid Pump and Accessories

To introduce the stripping agent into the stripping circuit, it would be necessary to meter, feed, and mix concentrated sulfuric acid and water. The rates of feed of the acid and water would be such that the mixture was a 6N solution. The 6N acid demand is a function of the volume of sludge entering the extraction circuit and the amount of aluminum in that sludge. At the stated design conditions, the acid demand for the production of alum containing 44,000 mg/l Al^{3+} would be 0.34 gpm or 1.29 l/min.

The acid metering pumps chosen were Fluid Metering, Inc., Models RPID-CP and RPIG400-CP. It was known that the characteristics of the sludge would vary and these pumps

provided a wide range of acid feed rates. The concentrated acid demand at design specifications would be one-sixth of 1.29 l/min or 0.22 l/min. The source of the concentrated acid would be a 10,000 gallon elevated storage tank located at the recovery plant site.

The potable water feed would originate from a faucet adjacent to the acid storage tank. The design potable water demand would be five times the acid demand or 1.10 l/min. A gate valve would be used to control water flow and a Badger Meter Recordall Model 25 water meter would measure the flow.

Figure 4-5 shows the acid-water feed system. The two liquids would be fed into a one inch diameter Pyrex standpipe connected to the Strip II inlet structure. The standpipe rose above the liquid level in the mixing tank to prevent spillage in the event of mixer failure. It was felt that enough turbulence would be created by the liquids as they hit the liquid surface in the standpipe to provide adequate mixing.

The mixing of acid and water demanded a Pyrex standpipe because it could withstand the heat generated mixing and then dissipate some of that heat before the diluted acid reached the PVC portion of the acid feed system.

4.4.2.13 Stripper Aqueous Recycle

Aqueous recycle flows would be adjusted by gate valves and measured with Blue White, Inc., 0-20 gpm flowmeters.

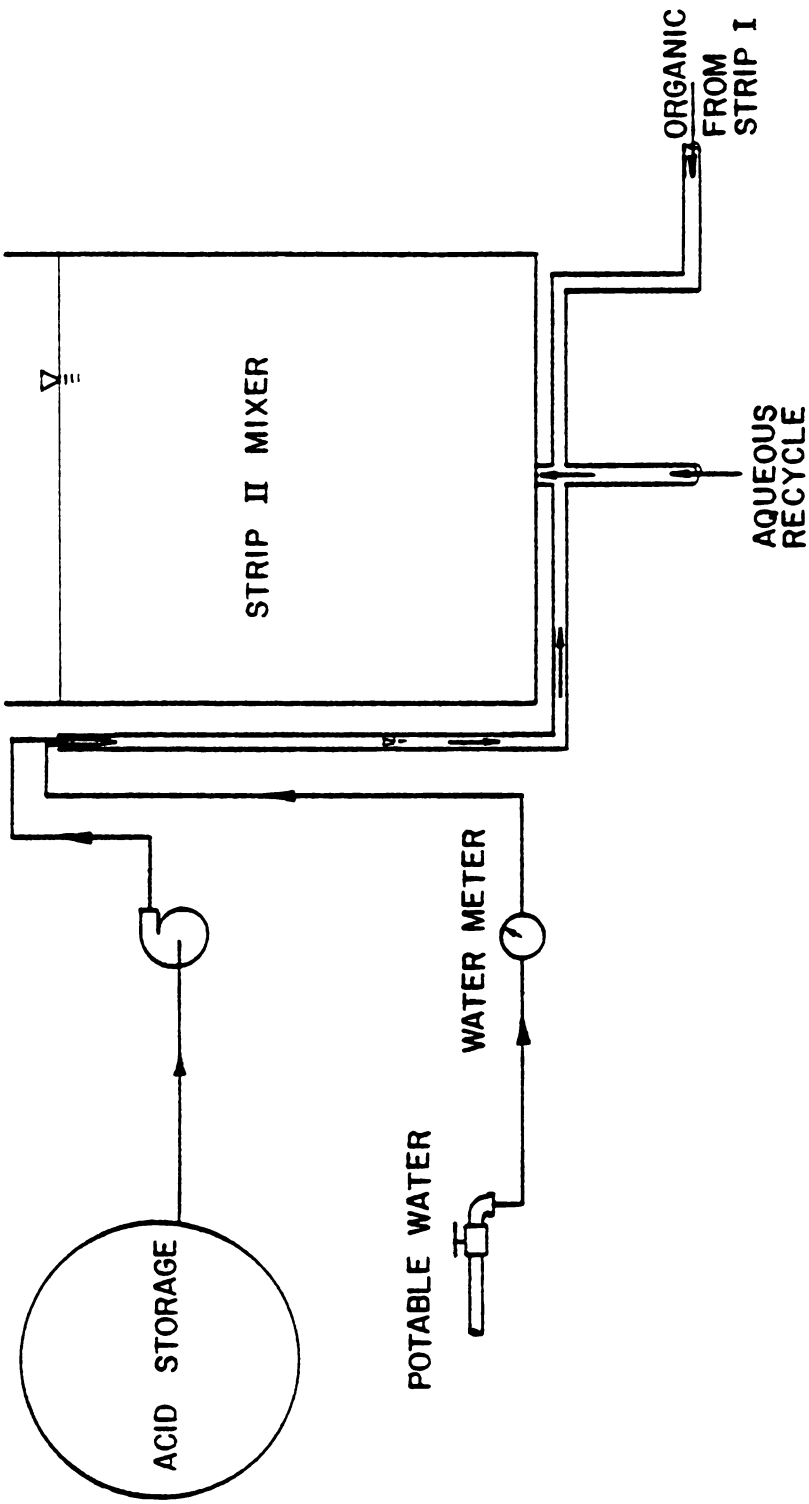


FIGURE 4-5
SCHEMATIC OF STRIPPING ACID FEED SYSTEM

4.4.2.14 Recovered Alum Treatment

Bench-scale studies indicate some of the color in the sludge is carried into the stripping circuit by the loaded organic and stripped out along with the aluminum. Also, organic is entrained in the recovered alum during stripping. To remove these contaminants, a granulated activated carbon column was placed in line between Strip I and the recovered alum day tank. Laboratory studies showed a large reduction in recovered alum TOC after contact with Filtersorb 400, a product of the Calgon Corporation.

The column was a simple design consisting of a four-foot section of 12-inch diameter PVC pipe and filled three feet of GAC. The recovered alum flowed by gravity from Strip I to the GAC column. The alum left the column by means of a screened manifold located at the bottom of the column. After GAC treatment, the alum would then flow by gravity to the day tank. Under design conditions, flow through the GAC column would be 490 gal/day.

4.4.2.15 Solvent Recovery

The solvent recovery operation was designed on the assumptions that 2 gpm of bleed solids would be produced by the extraction circuit and that the pilot centrifuge would perform as efficiently as the bench centrifuge. With the

extraction circuit operating continuously and the centrifuge operating 8 hr/day, the pilot centrifuge would have to process 6 gpm of bleed solids.

The unit chosen was a Sharples P660 Scroll centrifuge. This solid bowl device would be mounted on a platform under which a wheelbarrow could be parked into which the solids cake would fall. These solids would then be removed and studied to determine their drying characteristics. The recovered solvent and raffinate would be discharged at the liquid end of the bowl and would be returned to the extraction circuit via the organic recycle line. It was felt that the return of these liquids to the extraction circuit would not hinder extractor performance.

A siphoning manifold, situated appropriately in the extractor settler, would withdraw the bleed solids. Withdrawn continuously and discharged to a small holding tank, the bleed solids would be pumped to a larger holding tank when the centrifuge was not in operation or directly to the centrifuge when it was operating.

The pump chosen to perform the transfer of the bleed solids was a Robbins and Meyer Moyno pump. This progressing cavity pump, equipped with a variable speed motor was piped in such a way that bleed solids could be pumped from the small storage tank to the large storage tank, from the small tank to the centrifuge, or from the large tank to the centrifuge.

4.4.2.16 Tank Design

The design of the various holding tanks and reservoirs mentioned in the previous sections will now be discussed. These include an overnight solids holding tank, a short-term solids holding tank, a solvent reservoir, a recovered alum holding tank, and a spill and utility tank. Shown on Figure 4-6 is a layout of the alum recovery plant at Tampa.

The overnight solids holding tank was designed to store 24 hours worth of bleed solids or 2880 gallons. The tank chosen was a vertical cylinder with an eight-foot diameter and a 3000 gallon capacity.

The short-term solids holding tank was arbitrarily sized at 300 gallons. Its major purpose was to provide a mixing chamber where future solids neutralization studies could take place.

The solvent reservoir was a 1000 gallon horizontal tank. This sizing is based on the assumption that the bleed solids would contain 20 percent solvent by volume. Thus, when the large solids tank was full, it would contain 600 gallons of solvent. This would leave 400 gallons of solvent in the reservoir, which was enough to compensate for any changes in phase ratios or interface levels within the extracting or stripping circuits.

The recovered alum day tank was to have a volume of 750 gallons, providing 1-1/2 days of alum storage. To transfer

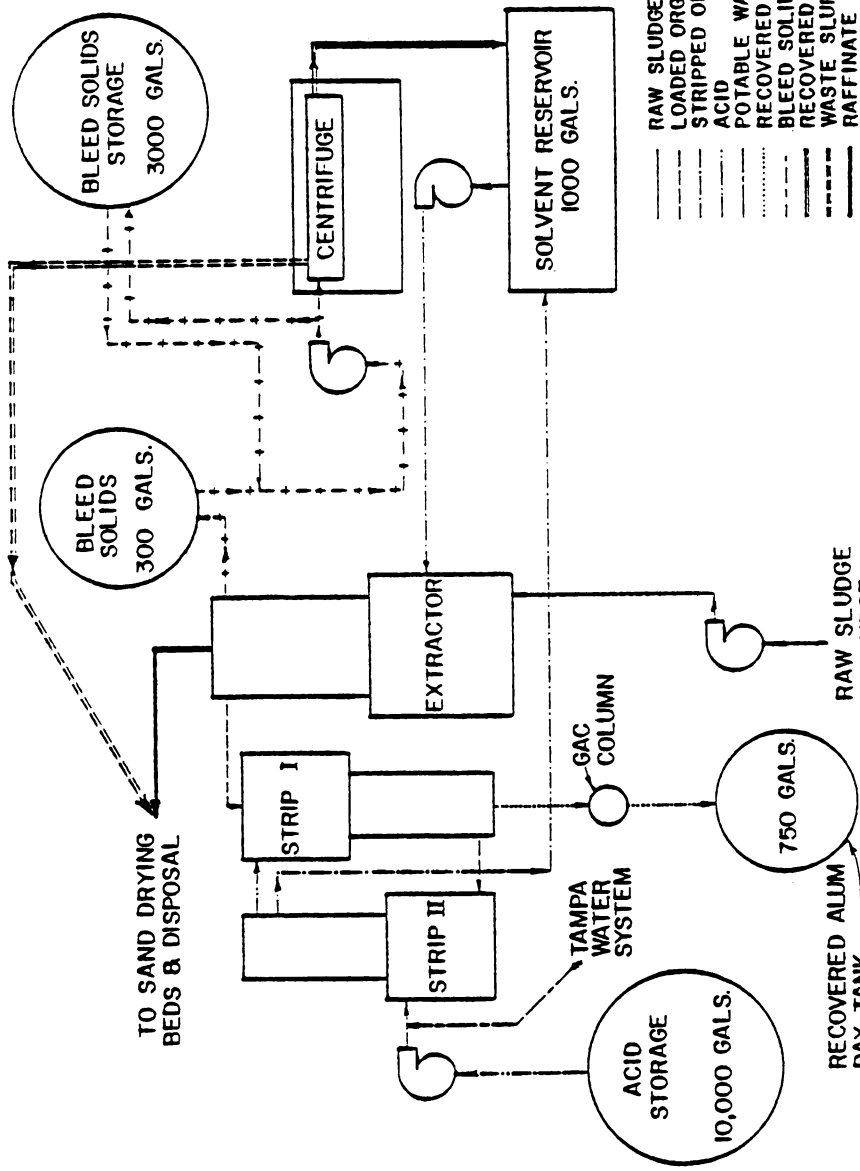


FIGURE 4-6
SCHEMATIC OF TAMPA PILOT PLANT

the alum to the water treatment plant's storage tanks, a Corcoran Model 2000E centrifugal was specified. This pump had a rating of 20 gpm at 60 feet of head. The Corcoran pump would also be used for utility purposes.

The spill and utility tank would have a capacity of 300 gallons and be used in clean-up and maintenance operations.

All of the above tanks were made of fiberglass.

CHAPTER 5

RESULTS OF MIXER-SETTLER OPERATION

5.1 Introduction

Construction of the pilot plant was completed on February 21, 1979. Operation began on February 27, 1979, and was scheduled to continue through August, 1979. However, equipment breakdowns and other delays caused operation of the mixer-settlers to continue through September, 1979. The pilot plant was in operation for a total of 479 hours and processed 141,900 gallons of sludge. Sludge flows varied from 1 to 6 gpm and sludge aluminum concentrations ranged from 80 to 1180 mg/l Al^{3+} at pH 2.0. The low capacity of the centrifuge prevented the 10 gpm design sludge flow from being evaluated. Figure 5-1 presents sludge flow, aluminum extracted, and raffinate pH as a function of time.

Upon start-up of the pilot unit, it was observed that the solids in the extractor settler were behaving differently than those in bench study. The pilot-plant solids situated themselves on the organic side of the interface, and as they accumulated their mass extended up into the organic portion of the settler. Initially, it was felt that the impeller speed might be responsible for this unexpected behavior. Various impeller speeds were evaluated, but the solids continued to accumulate in the organic phase.

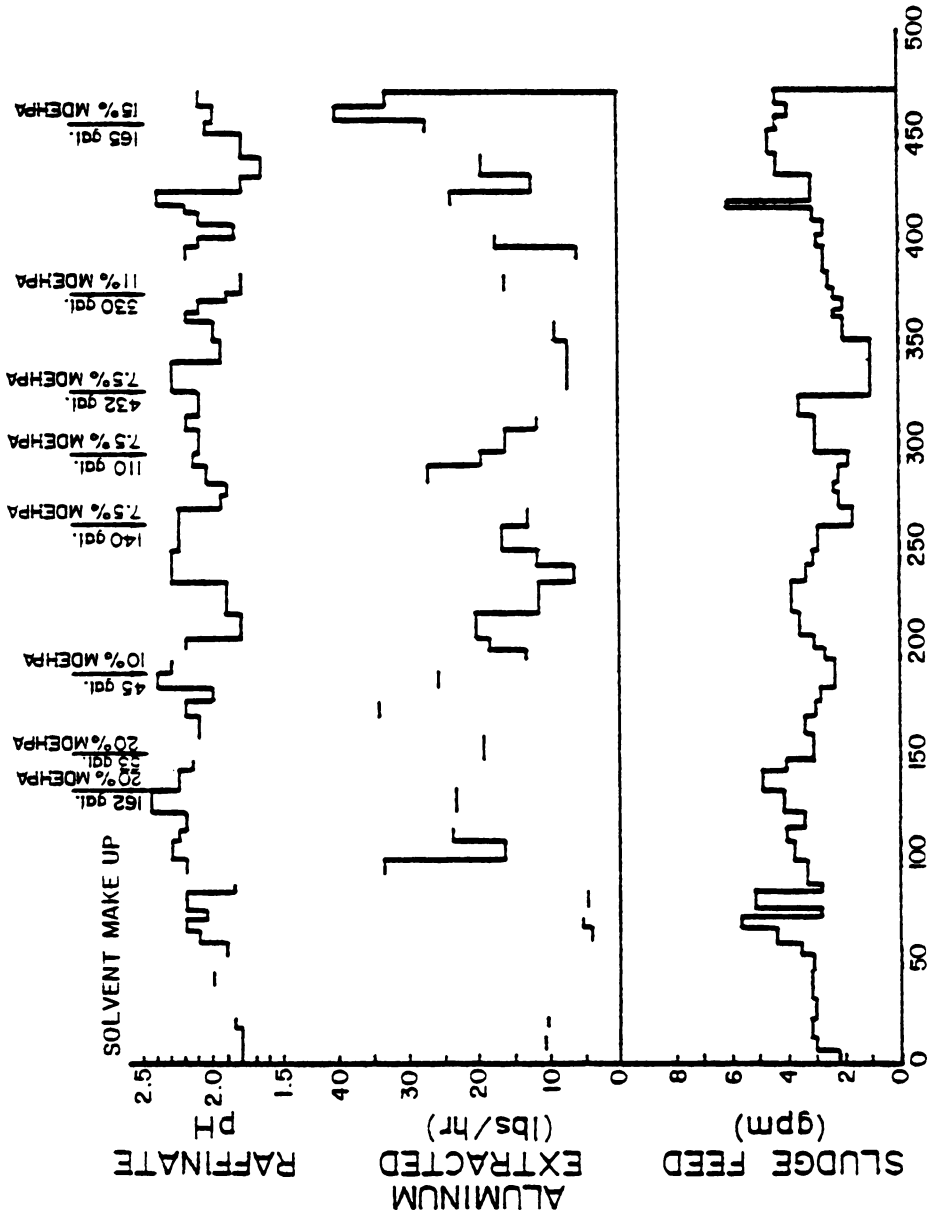


FIGURE 5-1
PILOT PLANT OPERATING DATA

The first week of operation was thus spent varying operating parameters in an effort to control the bleed solids in the extractor settler. However, it appeared that the unusual solids behavior was not related to any operating parameter. To determine if some aspect of the sludge had changed with respect to the alum recovery process, a sample was shipped to the bench-scale laboratory to be treated under the conditions determined optimum at the bench scale. The results of the bench test showed the solids to behave exactly as they had at the pilot scale. So, it appeared that some characteristic of the sludge had changed and the new behavior of the solids had to be dealt with.

Another problem which quickly arose was the performance of the centrifuge. When first operated, it could only process a fraction of the 6 gpm design value. Also, solvent recovery efficiency was poor. Since efficient solvent recovery was the key to cost-effective operation of the plant, optimization of the solvent recovery operation was given priority over other operations. Because of the low centrifuge throughput, the extraction and stripping circuits could not be operated continuously. Bleed solids production far exceeded the capacity of the centrifuge and when the solids storage capacity of the solvent recovery system was reached, the extractor and strippers had to be shut down until the stored solids were processed. The lengths of the

mixer-settler runs were very short, varying from 4 to 15 hours, depending on the raw sludge feed rate and the solids concentration of the sludge. This discontinuous type of operation meant that steady-state conditions in the mixer-settlers could not be attained for any useful length of time and without steady-state conditions, the evaluation of the mixer-settlers as proposed would be difficult if not impossible. It was under these conditions that operating data were collected. The presentation and discussion of operating results were divided into the following areas:

- Extraction circuit performance
- Entrainment and stripping circuit performance
- Solvent stability
- Personnel requirements

5.2 Extraction Circuit Performance

5.2.1 Extractor Mixer

5.2.1.1 Hydraulic Performance

The hydraulic performance of the extractor mixer was inconsistent. Phase inversions occurred at phase ratios ranging from 5:4:1 to 1:9:1. Mixer phase continuity was generally stable at these phase ratios during bench-scale tests. Also, variations in the pilot mixer phase ratio were

observed under constant mixer conditions. Both of these phenomena indicate a lack of agitation in the mixer. Variations in local phase ratios, a result of poor mixing, can lead to local phase inversions. These local inversions could then spread to the rest of the mixer. Therefore, the inversion of the whole mixer was a function of impeller rotational speed and not overall mixer phase ratio.

A phase inversion to aqueous continuous operation was a major upset to both extracting and stripping circuits. The stable emulsion which formed during aqueous continuous operation quickly filled the extractor settler and if allowed to pass into the stripping circuit would cause the stripper mixers to invert to aqueous continuous and foul the interfaces of the stripper settlers. To remedy the situation, the whole system had to be shut down to clean the extractor and stripper settlers and invert all mixers to the desired continuous phase. Because aqueous continuous operation in the extractor mixer upset the entire system, the extractor was generally run at high phase ratios to avoid phase inversions.

A simple remedy to the mixing problem would have been to increase the impeller speed. However, a mechanical flaw in the mixer drive train prevented the impeller from being rotated at the design speed. Because sufficient agitation

could not be provided in the mixer to ensure phase continuity stability at phase ratios lower than 3:1, the design phase ratio could not be evaluated.

Since the design mixer speed could not be attained, it was not possible to evaluate the method in which the mixer was scaled-up. While the successful use of $N^3D^2 \leq 20$ as a basis for mixer scale-up has been reported,[19] others suggest that the safest method of scale-up is based on maintaining geometric similitude and constant power input per unit volume.[28]

5.2.1.2 Aluminum Extraction

5.2.1.2.1 Introduction

It was originally planned to operate the extraction circuit in such a manner that flow rates and phase ratios could be systematically evaluated to determine the optimum values of these parameters. However, the poor performance of the centrifuge and the limitations imposed by poor mixing conditions did not allow the proposed evaluation to be conducted. The steady-state conditions necessary to evaluate these parameters never existed and it was in this context that the data from mixer-settler operation, presented in Appendix A, were analyzed.

5.2.1.2.2 Aluminum Extraction Efficiency

The extraction efficiency of the extractor was generally high, 85 percent or more, which is surprising in light of the poor mixing conditions which existed.

It is widely accepted that the bulk mechanism of mass transfer in liquid-liquid extraction mixers involves two zones. The first is in the immediate vicinity of the impeller. Here, dispersed drops are sheared into smaller droplets, passing through film and filament stages. The best conditions for mass transfer exist in this zone. The residence time of the droplets in this zone, however, is very short and equilibrium is usually not reached in one pass. The other mass transfer zone includes the rest of the mixer where, if it is being operated properly, mildly turbulent conditions exist. In this zone, a slower mass transfer rate is generated by droplet coalescence/ redispersion.[21]

Inadequate mixing can affect mass transfer in three ways:

1. By reducing the volume of the zone of high mass transfer rates in the vicinity of the impeller.
2. By reducing the number of times a given drop will pass through the zone with higher mass transfer rates.
3. By reducing the amount of coalescence/redispersion in the more tranquil zones of the mixer.

It is likely that all of these were taking place in the pilot mixer.

The effect of the poor mixing on mass transfer rates, however, was balanced by mixer residence times which were usually far in excess of the necessary 15 minutes and by high phase ratios. Thus, the extended mixing periods and increased concentration gradients allowed the reduced mass transfer rates sufficient time to approach equilibrium.

The effect that the recycling of organic had on extraction cannot be determined, but other research in this area suggests that the recycling of the continuous phase has both beneficial and detrimental effects. It is beneficial because the recycling of either phase will enhance extraction. It is detrimental because the addition of more of the continuous phase will dilute the dispersion and decrease the amount of coalescence/redispersion. Experimental results suggest that these effects balance each other and have no net effect on extraction.[28] If it is assumed that these findings are applicable to the alum recovery system, then it is probable that the recycled organic had little effect on extraction efficiency.

Assuming that the organic recycle has little effect on efficiency, it is possible that there exists an optimum feed phase ratio within the limits imposed by mixing conditions. If such a phase ratio does exist, it is not evident from the operating data. The apparent disorder of the data can be attributed to:

1. The lack of steady-state conditions.
2. The extending of residence times far past that required to reach equilibrium.
3. Varying residence times which did not allow isolation of the feed flow ratio.

Given a mixer that is operating with an optimum impeller speed, a constant residence time, and organic continuous conditions, an optimum feed flow ratio would be one which was as low as possible and still be stable with respect to the desired dispersion conditions.[28] As the phase ratio increases, the dispersed phase is diluted, decreasing droplet interaction and decreasing mass transfer rates.

The pH of the raffinate was used as a relative measure of extraction. This was based on bench-scale results which indicated that changes in raffinate pH closely reflected changes in extraction efficiency. This relationship can be explained by the mechanism by which aluminum is extracted. The extraction reaction involves the exchange of aluminum from the aqueous to organic for protons from the organic to aqueous. Thus, as more aluminum is extracted, extraction efficiency increases, and the raffinate pH is depressed. A plot of extraction coefficients as a function of raffinate pH is shown on Figure 5-2.

The extraction coefficient, E_a^o, is defined as the ratio of the metal concentration in the organic extract to the metal concentration in the aqueous raffinate and is a measure

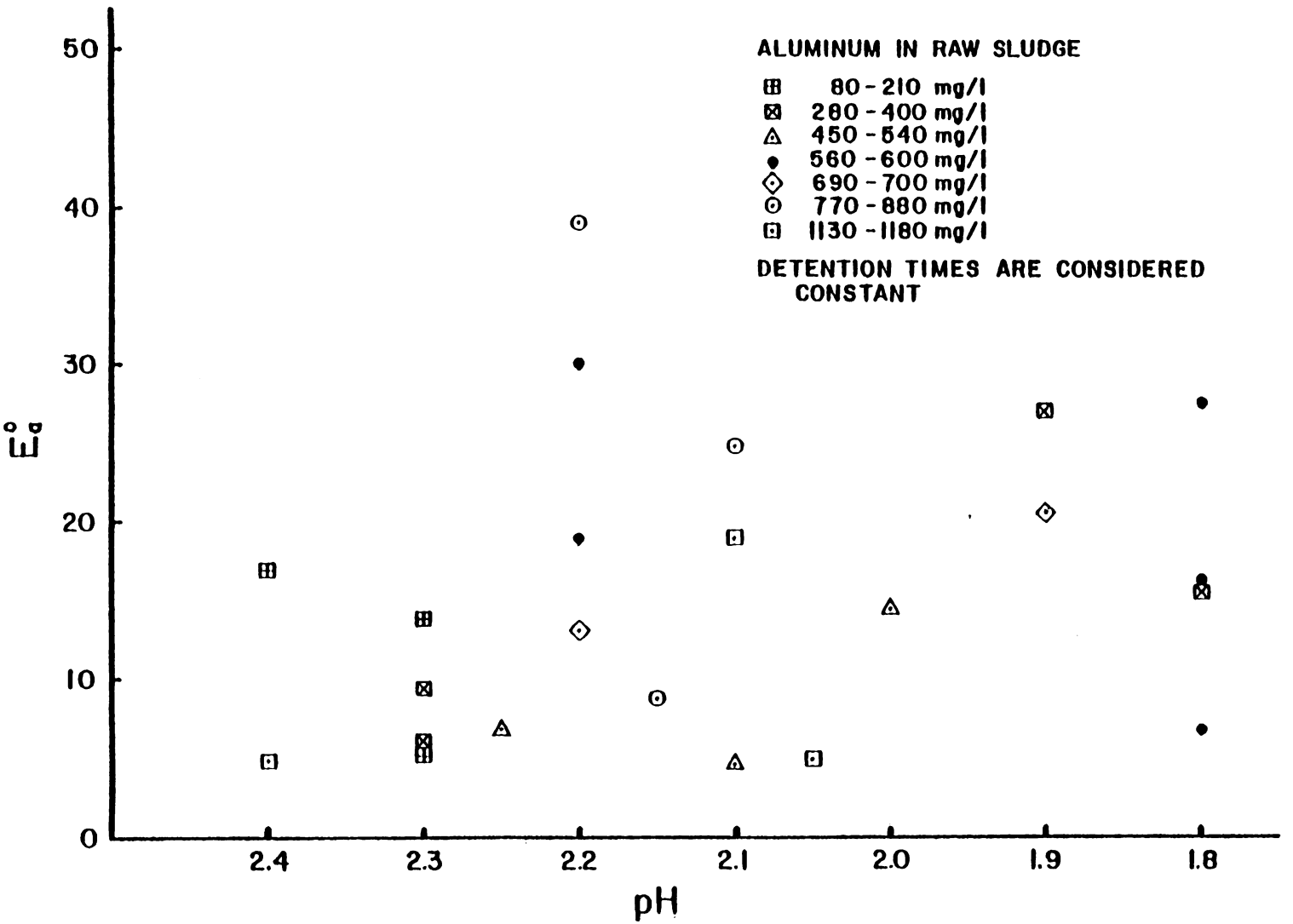


FIGURE 5-2
EXTRACTION COEFFICIENT AS A FUNCTION OF RAFFINATE pH

of the amount of metal extracted for a given solute metal concentration. As can be seen on Figure 5-2, there is no evident relationship between extraction coefficients for a given group of like solute concentrations and raffinate pH. The apparent lack of order of the data plotted on Figure 5-2 can be explained by the following:

1. The lack of steady-state conditions did not allow the isolation of the parameters in question.

2. Most of the data points on Figure 5-2 lie between pH 2.3 and 1.8. Batch studies under controlled conditions found that a drop in pH from 2.3 to 1.8 of the Tampa sludge represented a 4 percent increase in the aluminum available for extraction. Given the conditions under which the pilot plant operated, it is unlikely that such a small change could have been identified.

3. While MDEHPA is highly selective for aluminum, the presence of extractant in concentrations beyond that necessary to extract a given amount of aluminum will encourage the extraction of other cations. Since the aluminum concentration in the Tampa sludge fluctuated greatly from day to day, it was necessary to maintain a level of extractant in the solvent which could accommodate a wide range of sludge aluminum concentrations. This meant that when sludge aluminum concentrations were low, there was excess extractant present. Once equilibrium was reached with respect to aluminum exchange, another cation, calcium, which was often present in significant concentrations, would be extracted. This would cause the pH of the raffinate to be lowered without an accompanying increase in aluminum extraction and could yield misleading results.

The raffinate pH can be a useful tool in monitoring extraction efficiency, especially from an operational point of view, if the factors which alter this relationship are

understood. The most reliable measure of extraction, however, is to directly measure the aluminum in the aqueous phase before it enters the extractor and after it leaves the extractor.

Other factors which may have effected extraction were solvent stability, to be discussed later in this chapter, and alkalinity and solids in the sludge.

The presence of alkalinity in the sludge could affect extraction by buffering the aluminum exchange reaction. Extraction coefficients could be lowered by exchanged protons being consumed by alkalinity instead of depressing sludge pH.

Investigating the effect that the solids in the sludge had on extraction was beyond the scope of this study; however, it is a variable which cannot be ignored. The solids can be divided into two groups, those which are solubilized during extraction such as, the alum floc and color, and those which remain insoluble, such as silt and turbidity. Little literature is available on this subject as most operations find it cost-effective to remove all solids from the solute feed stream.

5.2.2 Extractor Settler

The performance of the extractor settler was very good when the adverse conditions under which it operated are considered and the scale-up of this unit must be considered a success.

The two settling aids employed, the dispersion introduction baffle and the picket fence were most useful. The settler was operated for a short time without them and there was a great amount of turbulence in the settler during that period. It was felt before start-up that the solids might clog the picket fence, but operation of the settler proved this fear unfounded as the solids passed easily through the slots of the fence.

It has been suggested that the use of horizontal plates, meshes, or packing can increase the efficiency of a settler. It is unlikely that any of these could be used in alum recovery because the bleed solids would quickly foul this type of settler aid. However, the use of a packing such as Knit Mesh,[6] made of a material wetted by the raffinate and solids, and placed directly in front of the organic weir might be able to reduce the amount of impurities going into the stripping circuit.

5.3 Entrainment and Stripping Circuit Performance

5.3.1 Introduction

Entrainment between stages can result in reduced efficiency, while entrainment into effluent streams can mean solvent losses and product contamination. Thus, it is imperative to minimize entrainment to ensure efficient mixer-settler operation.

Bench-scale studies did not reveal any entrainment problems in the extraction circuit and none were expected at the pilot scale. Organic entrainment losses into the raffinate were assumed to be small when compared to losses due to bleed solid processing and were given no consideration during the pilot study. Raffinate entrainment into the extract, however, turned out to be a major problem which affected stripper performance and recovered alum quality.

5.3.2 Entrainment Symptoms

Two problems which arose during mixer-settler operation were the solids which collected at the Strip I settler interface and the recovered alum aluminum concentration.

After start-up, it was quickly evident that a large amount of solids, identical to the bleed solids, were accumulating at the Strip I settler interface. Two to five gallons of these solids were scooped out of the settler daily with a strainer. Occasionally, some of the solids would be washed out with the organic into Strip II. More importantly, it is probable that the presence of the solids interfered with coalescence in the settler. Obviously, the solids were being carried from the extractor to the stripping circuit by the loaded organic.

Figure 5-3 presents a plot of stripping acid concentrations as a function of operating time. Throughout most of

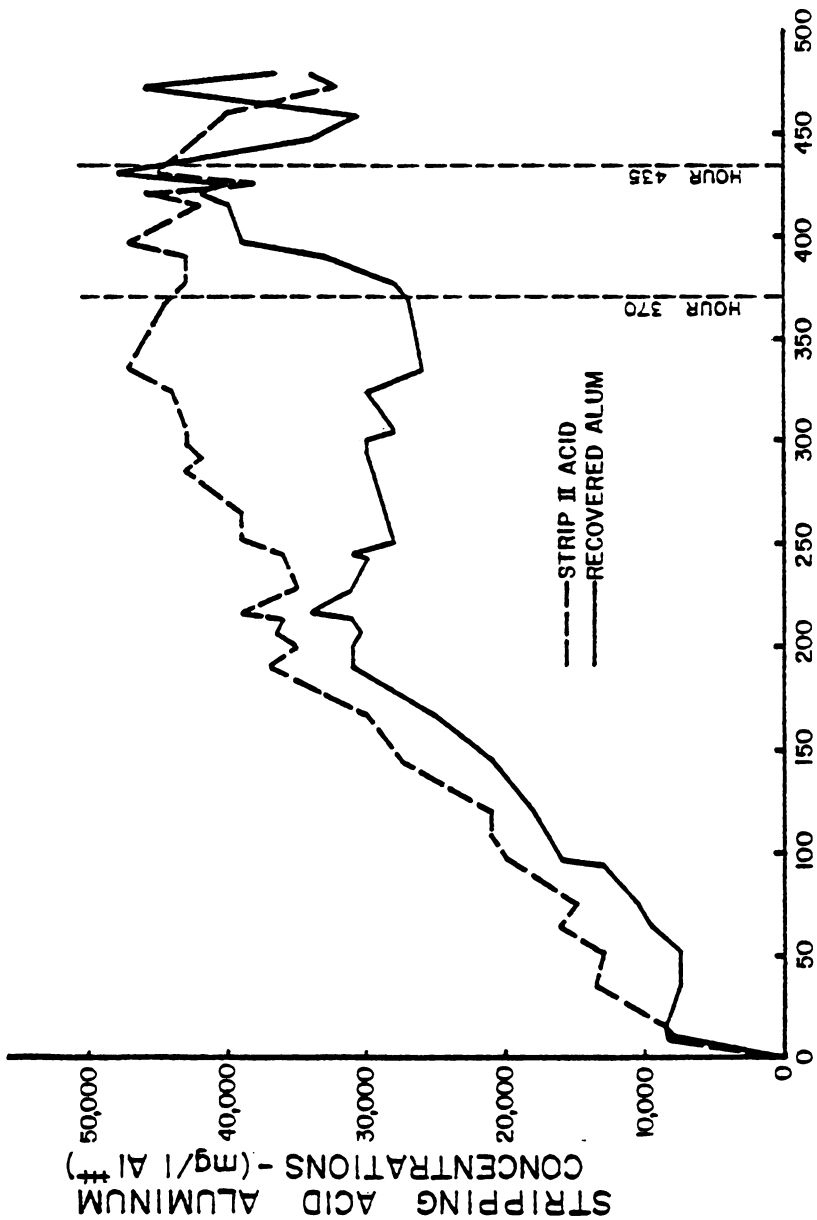


FIGURE 5-3
STRIPPING ACID ALUMINUM CONCENTRATIONS AS A
FUNCTION OF OPERATING TIME

the study, the Strip II acid contained more aluminum than the recovered alum. Normally, the alum could contain several thousand more milligrams per liter of aluminum than the Strip II acid. Even if the stripping efficiency of Strip I was zero, the alum would still have the same aluminum concentration as the Strip II acid. The alum was being diluted and the only influent stream to Strip I which could supply the aqueous solution for dilution was the loaded organic from the extractor.

5.3.3 Causes of Entrainment

Initially, it was felt that the solids and aqueous carry over into the stripping circuit were caused by the high scour velocities developed in the organic portion of the extractor settler when bleed solids were allowed to build up in the settler. A visual inspection of the settler with approximately six inches of free organic revealed locally high velocities and the movement of fine solids particles within the bulk organic phase. Also, the loaded solvent was cloudy, indicating aqueous entrainment.

In an attempt to eliminate the solids and raffinate carry over into the stripping circuit, a deeper organic layer was maintained in the extractor settler. This reduced the high local velocities and bottom scour, but the loaded

organic remained cloudy. The deeper organic layer was maintained from hour 310 to hour 380. While the amount of solids that collected in Strip I decreased, it would appear from Figure 5-3 that it only aggrevated the alum dilution problem.

Entrainment in a system with a well designed settler is a function of phase continuity, mixer speed, and phase ratio. The phase continuity and mixer speed of the pilot-plant extractor were considered to be fixed and it was the phase ratio which was investigated with respect to the aqueous entrainment.

Other research indicates that for extractors operating organic continuous, the level of aqueous entrainment increases with increasing phase ratios.[28] The proposed mechanism for this relationship states that for a given set of mixer conditions, there is a "preferred" phase ratio at which entrainment will be minimal. In the case of organic continuous operation at phase ratios above the "preferred" phase ratio, there is an excess of organic over that required to form the "preferred" ratio. This excess organic "flashes off" when leaving the mixer and when entering the settler immediately joins the bulk continuous phase, carrying with it small dispersed aqueous droplets.

It can be seen from the stripping circuit operating data, presented in Appendix A, that from hour 220 to hour 330, the extractor was operated at generally high phase

ratios. As a result, the difference in stripping acid aluminum concentrations increased because of the increase in aqueous entrainment.

From hours 370 to 435, the extractor was operated at an average phase ratio of 3:1. The response of the aluminum concentration in the alum was both immediate and positive. Within 30 hours the aluminum concentration rose from 27,000 mg/l Al^{3+} to 39,000 mg/l Al^{3+}. By hour 426, the alum strength had surpassed that of the Strip II acid and by hour 432 had reached 48,000 mg/l Al^{3+}. Also, slightly fewer solids collected at the Strip I settler interface.

The portion of Figure 5-3 following hour 435 represents a period of operation in which priority was given to the extraction circuit and stripping circuit parameters were not monitored closely.

The effect that the extractor mixer phase had on the recovered alum aluminum concentration is shown on Figure 5-4. The higher phase ratios, producing higher levels of entrainment and dilution caused the alum aluminum concentration to decrease. At phase ratios between 6:1 and 7:1, the amount of dilution was equal to the amount of aluminum coming from Strip II. At phase ratios below 6:1, the alum generally increased in aluminum concentration.

Figure 5-5 presents a plot of aqueous entrainment as a function of extractor mixer phase ratios. Two of the data

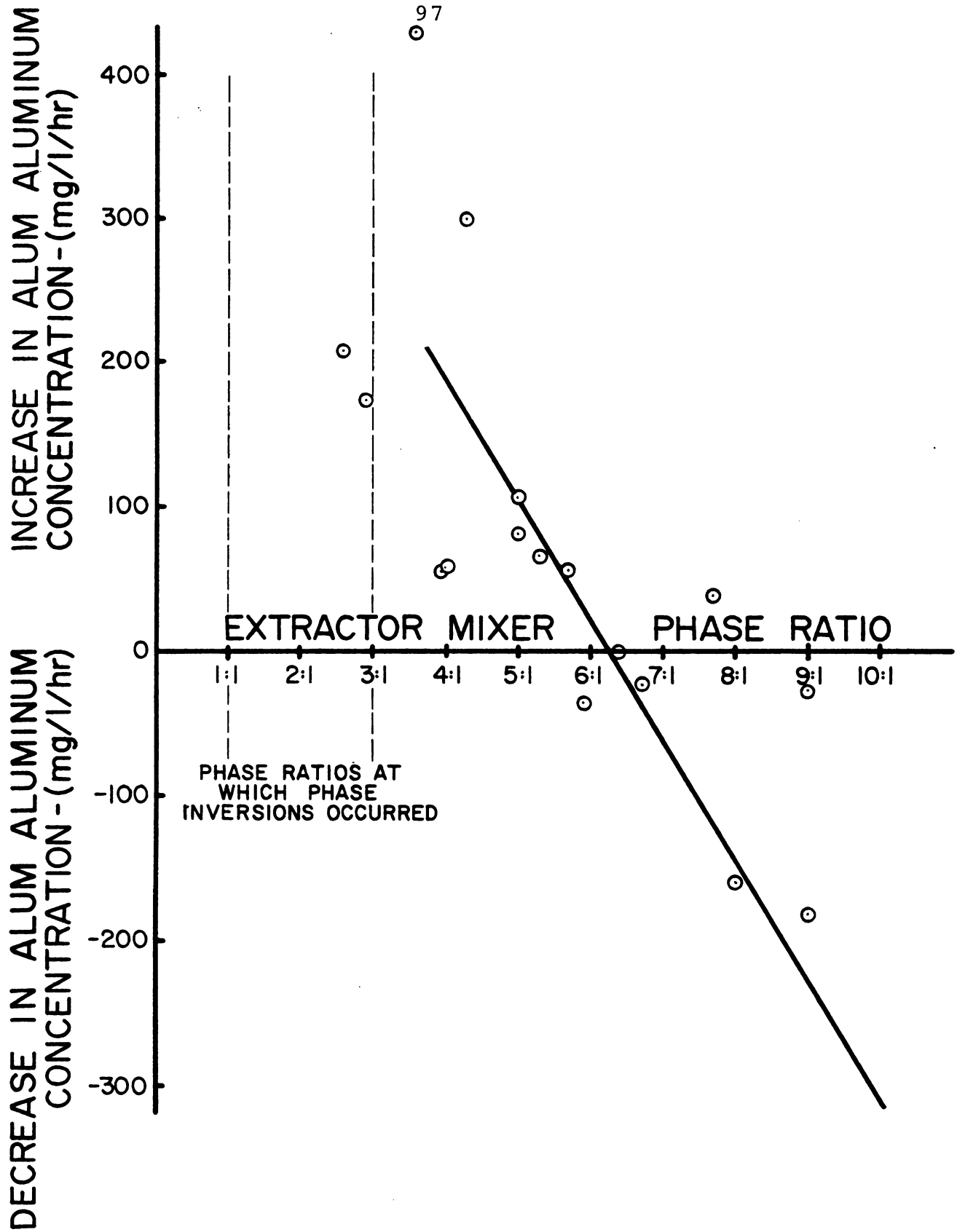


FIGURE 5-4
EFFECT OF EXTRACTOR MIXER PHASE RATIO ON
RECOVERED ALUM ALUMINUM CONCENTRATION

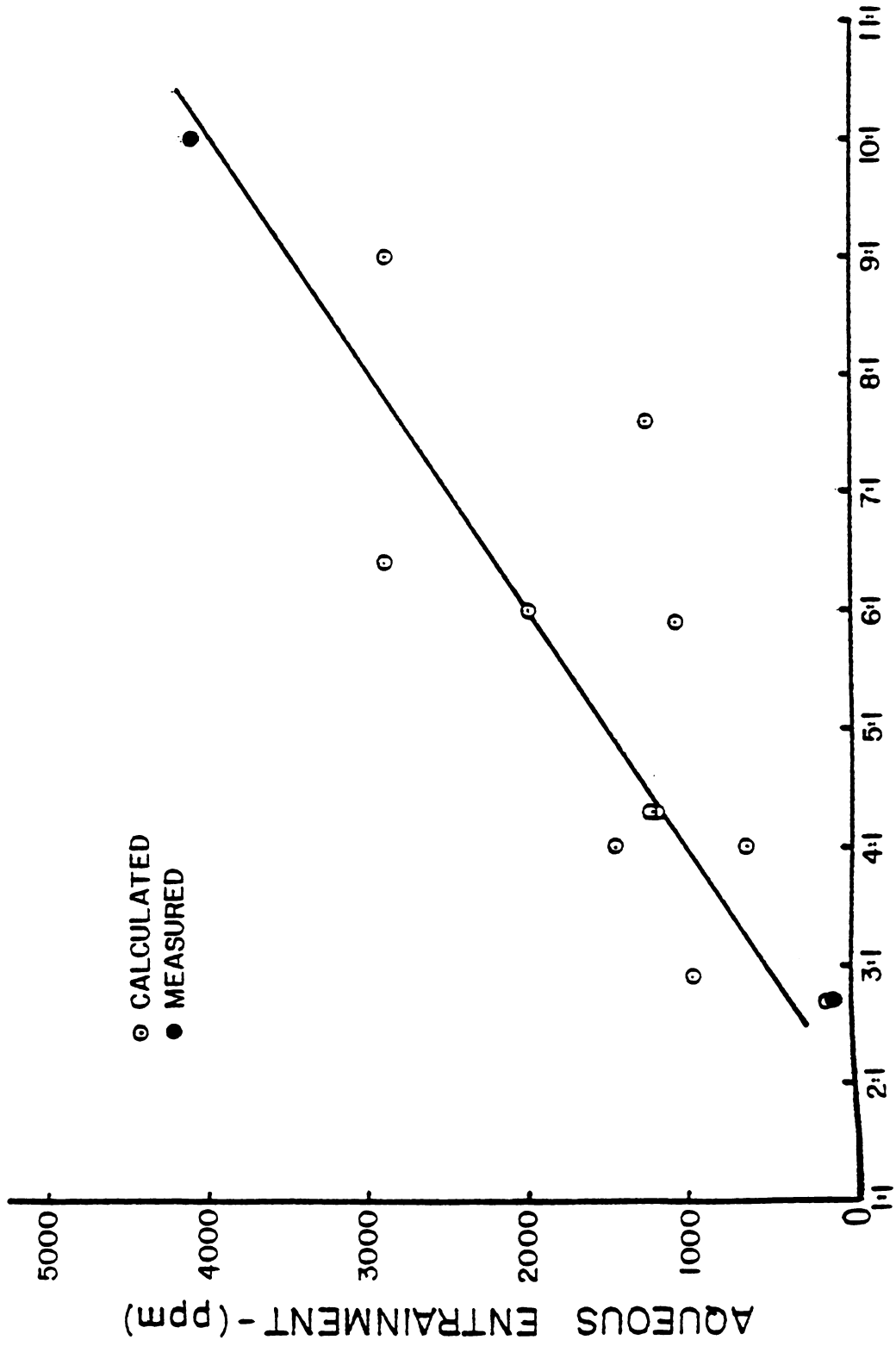


FIGURE 5-5
AQUEOUS ENTRAINMENT AS A FUNCTION OF EXTRACTOR MIXER PHASE RATIO

points on this plot were measured, while the remainder were calculated. Again, the relationship between phase ratio and aqueous entrainment is clearly evident. The method of entrainment measurement and a sample entrainment calculation are provided in Appendix C.

5.3.4 Other Aspects of Stripping Circuit Performance

5.3.4.1 Introduction

Time and personnel limitations did not allow the strippers to be operated and evaluated as originally planned, but several general comments can be made with respect to their performance.

5.3.4.2 Stripper Mixers

As with the extractor mixer, the design mixer speed did not provide enough agitation and the validity of the use of $N^3D^2 \leq 20$ as mixer design criteria, at least in this case, should be questioned. A mixer speed of 140 rpm, equivalent to a tip speed of 850 feet per minute, was necessary to provide stable phase continuity conditions at phase ratios less than 2:1.

5.3.4.3 Stripper Settlers

In contrast to the mixers, the stripper settlers were properly designed as dispersions bands varied in thickness

from one to two inches for the flow rates and phase ratios at which the settlers operated.

In addition to the bleed solids which washed out of the extractor settler, the Strip I settler also had to deal with the Ca_2SO_4 which precipitated in the Strip I mixer. The source of the calcium was the raw sludge. The calcium was extracted along with the aluminum and was also carried into the stripping circuit by the entrained raffinate. What effect the calcium had on stripping efficiency is unknown, but the presence of Ca_2SO_4 at the settler interface definitely interfered with coalescence.

The Ca_2SO_4 problem can be eliminated by the use of HCl combined with H_2SO_4 as the stripping agent. This would reduce the SO_4 concentration and thus, reduce Ca_2SO_4 production. Hydrochloric acid costs more than sulfuric, but the savings in maintenance costs may well offset this expense. Just as at the bench scale, all Ca_2SO_4 precipitated in Strip I; Strip II remained free of this contaminant.

5.3.4.4 Entrainment

No entrainment measurements were made, but a study on entrainment in strippers operating organic continuous indicates that in the range of phase ratios at which the pilot strippers operated, organic entrainment is very low

and independent of phase ratio. However, aqueous entrainment under these conditions was high.[28] Precisely how much acid was entrained cannot be determined, but its presence was confirmed by the cloudiness of the stripped organic. High levels of stripping acid entrainment can reduce aluminum extraction efficiency by lowering the pH of the feed sludge and increasing the aluminum content of the sludge.

5.4 Solvent Stability

5.4.1 Introduction

Bench-scale studies with the mixer-settlers indicated that under constant operating conditions the extracting ability of a given solvent inventory increased when fresh extractant was added to the system. One of the goals of the pilot program was to investigate and quantify the degradation of extractant quality.

5.4.2 Pilot Studies

The extractant at Tampa behaved much like it did at the bench scale. As can be seen on Figure 5-1, after an addition of fresh extractant to the system, the pH of the raffinate dropped. However, it was not possible to evaluate extractant performance over a long period of time because make-up solvent additions were made so frequently that the extractant

was never allowed to stabilize. As shown on Figure 5-1, make-up solvent had to be added approximately every 50 hours. This frequency reflects the poor performance of the solvent recovery operation.

5.4.3 Tampa Bench-Scale Studies

Since it was not possible to evaluate solvent stability with the pilot-scale mixer-settlers, a 4-inch diameter by 36-inch long RTL Contactor was set up to investigate the behavior of the extractant. Starting with a five-gallon inventory of fresh 7.5 percent (V/V) MDEHPA in Kermac 627, the solvent was contacted 23 times with Tampa sludge containing approximately 1000 mg/l Al^{3+}. Each time the entire inventory had been contacted with the sludge, it was stripped with 6N H_2SO_4 in a batch stripping operation. Throughout this evaluation operating conditions were held constant at the values listed below:

Rotor speed	=	6 rpm
Organic feed	=	20 ml/min
Sludge feed	=	20 ml/min

After the solvent was stripped, it was contacted with a synthetic solution containing 1000 mg/l Al^{3+}. The aluminum concentration of the synthetic raffinate was then measured

and the results are plotted on Figure 5-6. It is evident from this plot that there is a dramatic decrease in extracting ability after only three contacts with the sludge. By the time the solvent has been loaded 13 times, the extraction coefficient decreased from 18.7 to 0.25, a 99 percent decrease.

Recent work by Cornwell,[25] has provided some insight into the problem of solvent degradation. An analysis of extractant that had been contacted with sludge many times revealed that the "mono" portion of the extractant was gone. Titrations of the extractant with a basic solution confirm these findings and explain the observed reduction in extracting ability. Fresh extractant contains approximately equal molar quantities of mono(2-ethylhexyl) phosphoric acid and di(2-ethylhexyl) phosphoric acid. The mechanism by which the "mono" portion of the extractant is removed or neutralized is not yet known. Three possible explanations are listed below:

1. The "mono" portion of the extractant is soluble in the raffinate.
2. The "mono" portion of the extractant is neutralized by a reaction with impurities in the sludge.
3. The "mono" portion of the sludge precipitates out of solution with the calcium in the sludge.

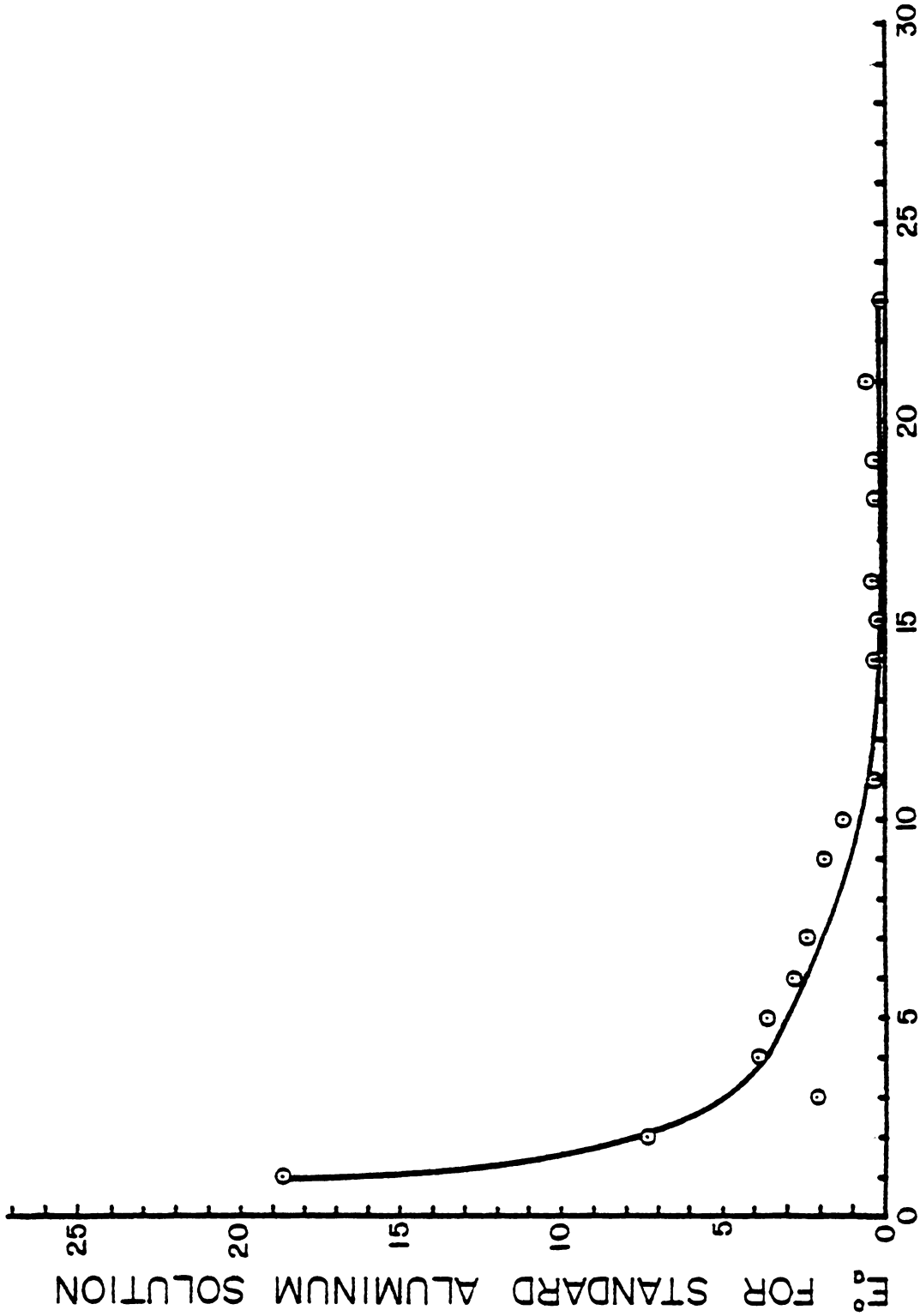


FIGURE 5-6
EXTRACTION COEFFICIENT AS A FUNCTION OF ORGANIC LOADING

5.5 Personnel Requirements

The operation of the mixer-settlers required very little attention. The operation of the centrifuge, however, required a great deal of attention to operate it at peak efficiency. For this reason it would be necessary to man a full-scale alum recovery plant with one person at all times.

CHAPTER 6

RESULTS OF RTL CONTACTOR OPERATION

6.1 Introduction

As the mixer-settler pilot program progressed, it was realized that the Sharples centrifuge was not capable of performing in a manner which would make the alum recovery process cost-effective. Centrifuge throughput was low and, more importantly, solvent recovery was extremely poor. To continue operation with the Sharples centrifuge would have been fruitless and a decision had to be made as to what course the remainder of the pilot program would take. Two alternatives were available. The first was to install and evaluate another centrifuge which was more suited to the separation problem posed by the bleed solids. The second was to replace the mixer-settler extractor with a contacting unit which did not produce a waste stream from which solvent had to be recovered.

Fortunately, it was possible to explore both alternatives. A DeLaval BRPX - 207 Solids-Ejecting Centrifuge was leased for a two-week period and the evaluation of its performance is the subject of another paper.[4] The mixer-settler extractor was replaced by a RTL Contactor, the evaluation of which is the subject of this chapter.

The problems associated with the treatment of the bleed solids as outlined in Chapter 5 were the reasons for the unsuccessful economic performance of the mixer-settler/ centrifuge system. Bench-scale studies conducted at Michigan State University with a four-inch diameter RTL Contactor showed that in cocurrent operation, aluminum could be extracted from the Tampa sludge without the production of bleed solids. The solids which remained insoluble during extraction in the RTL did not form an emulsion, but remained in the raffinate. This meant that solvent losses from the RTL system would be lower than from the mixer-settler system and these reduced solvent losses could be attained without the use of a solvent recovery operation.

The formation of bleed solids in the RTL Contactor was prevented by the relatively gentle mixing provided by the slow rotation of the buckets on the rotor shaft. While the dispersion formed in the mixer-settler was very fine, the rotating RTL buckets formed dispersed droplets many magnitudes larger than those in the mixer-settler. These large droplets coalesced into their bulk phases much more quickly and easily than the smaller droplets of the mixer-settler.

Based on the success of the bench-scale RTL studies, a one gpm RTL Contactor was constructed and installed for evaluation at the Tampa alum recovery pilot plant. The main

objectives of this evaluation were to establish the maximum throughput, solvent losses, and the operator input for proper process control.

6.2 Equipment

6.2.1 RTL Contactor

The RTL Contactor was supplied by Riotinto Til Holdings S.A. of London, England and was constructed of stainless steel throughout. A glass sight-port was installed at the discharge end of the contactor to monitor the interface level and observe solids behavior. The rotor contained 76 stages, each stage containing eight buckets. The chain driven rotor was powered by a gear reduced 3/4 HP electric motor. Sprockets were supplied which allowed the rotor speed to be varied from a maximum of ten rpm, to a minimum of two rpm. The contactor shell was supported by two saddles, these saddles were supported by a rectangular base frame.

6.2.2 Pumps and Accessories

Organic was pumped into and out of the RTL. Two Cole-Palmer Model 7314 Variable Flow pumps were used to handle the organic. These pumps had a capacity of 0-5 gpm. Sludge was fed to the RTL with a Wallace and Tiernan Diaphragm Metering Pump. Organic flow was measured with two Fisher

and Porter Rotameters. Sludge flow was monitored by manual measurements with a stopwatch and graduated cylinder. Organic and sludge transfer lines consisted of 3/8 inch ID Tygon tubing.

6.3 Plant Layout

The RTL Contactor was tied into the existing stripping circuit as shown on Figure 6-1. Stripped organic was pumped from the effluent end of the settler of Strip II to the organic inlet of the RTL. Loaded organic was pumped from the organic outlet of the RTL to the Strip I mixer. Sludge was drawn from a five gallon sludge reservoir which was fed with sludge pumped by the mixer-settler sludge feed pump. Sludge discharged into the reservoir caused enough turbulence to keep the reservoir well mixed.

While the strippers were oversized with respect to the organic flow generated by extraction with the RTL, their use was suitable for the short-term testing of the RTL.

Raffinate was not pumped out of the RTL. The raffinate discharge line contained an air break which allowed the interface level to be controlled by the relative flow rates of the stripped and loaded organic. If the influent organic flow rate was greater than the effluent, the interface would drop and the raffinate flow would be greater than the sludge

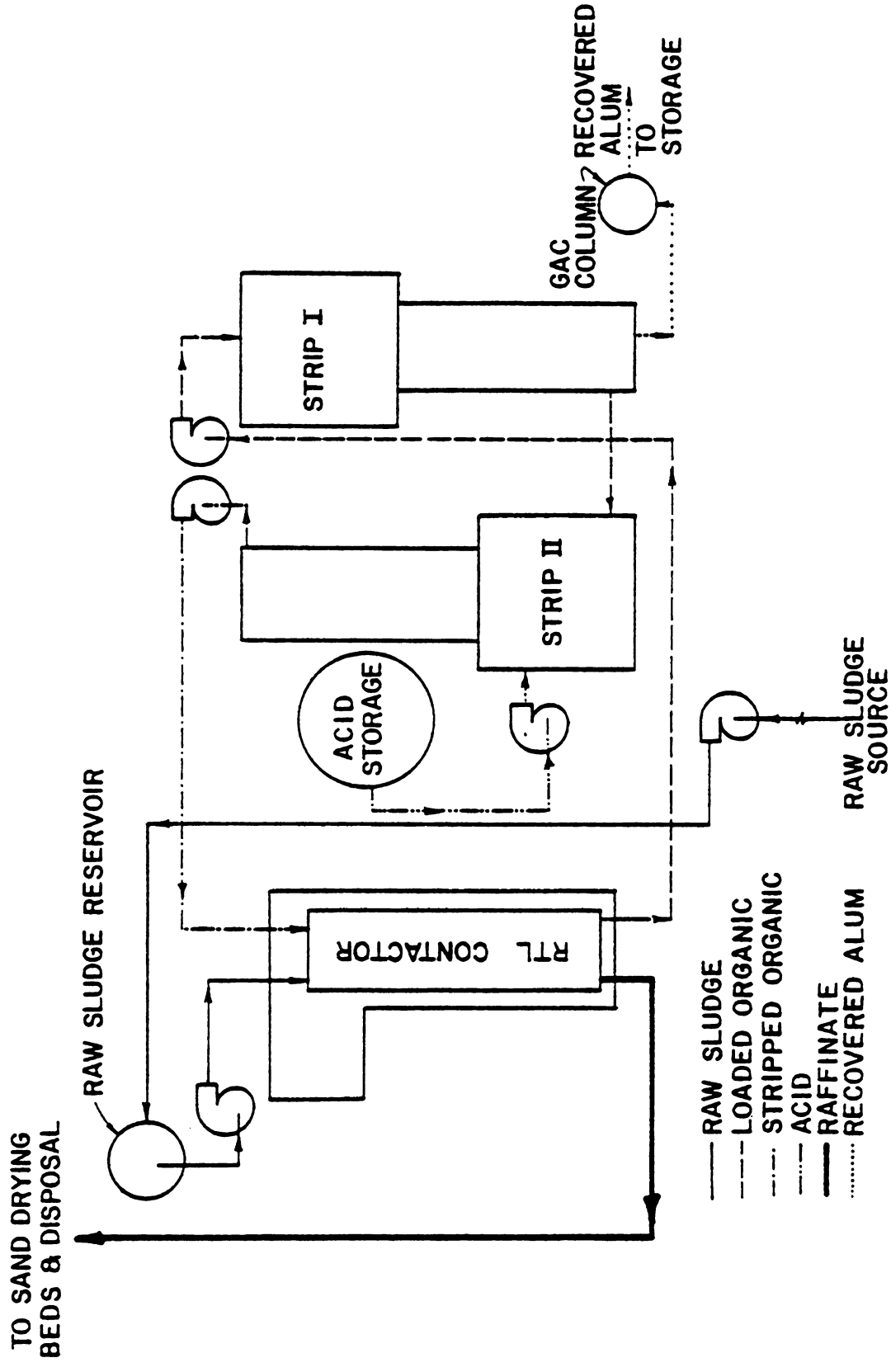


FIGURE 6-1
SCHEMATIC OF TAMPA PILOT PLANT WITH RTL CONTACTOR

feed flow. If the influent organic flow rate was less than the effluent, the interface would rise and the raffinate flow would be less than the sludge feed flow.

6.4 Operations

Between September 18, 1979, and September 27, 1979, the RTL Contactor was operated for 208 hours. The data from that run are presented in Appendix B.

The extractant used for the RTL run was that which remained in the mixer-settler system. The extractant strength was approximately 10 percent (V/V) MDEHPA.

Operational parameters which were monitored are listed below:

- Organic flow
- Sludge flow
- Raw sludge characteristics
- Raffinate characteristics

The RTL was operated by plant personnel and readings were taken hourly. This frequent attention was for data collection purposes only and under actual operating conditions the RTL would require much less attention.

Equipment performance was generally good. The RTL Contactor was operated at two rpm for the duration of the run and operated flawlessly. Low operating and maintenance costs are a major advantage of the RTL Contactor. Sludge flows held steady at the desired levels, as would be expected with a metering pump. Maintaining the organic flow rates at equal values, however, proved at times to be a problem. Several times the effluent pump speed drifted such that it was slightly greatly than the influent pump. This caused an air gap to form at the top of the RTL. In turn, this caused the effluent pump to draw air. With no liquid to pump, the effluent pump overheated and shut down. With the effluent pump out-of-service and only the influent pump running, the contactor interface quickly dropped. However, no major upsets occurred due to the effluent pump shutdown because the problem was noticed and remedied in the course of the hourly system monitoring.

6.5 Extraction

Sludge flow rates from 0.40 gpm to 0.88 gpm were evaluated at various phase ratios. While the RTL run was short and the amount of data collected limited, it was possible to discern several relationships. As shown on Figure 6-2, for a given sludge aluminum concentration, phase ratio, and rotor speed, the amount of aluminum extracted is proportional

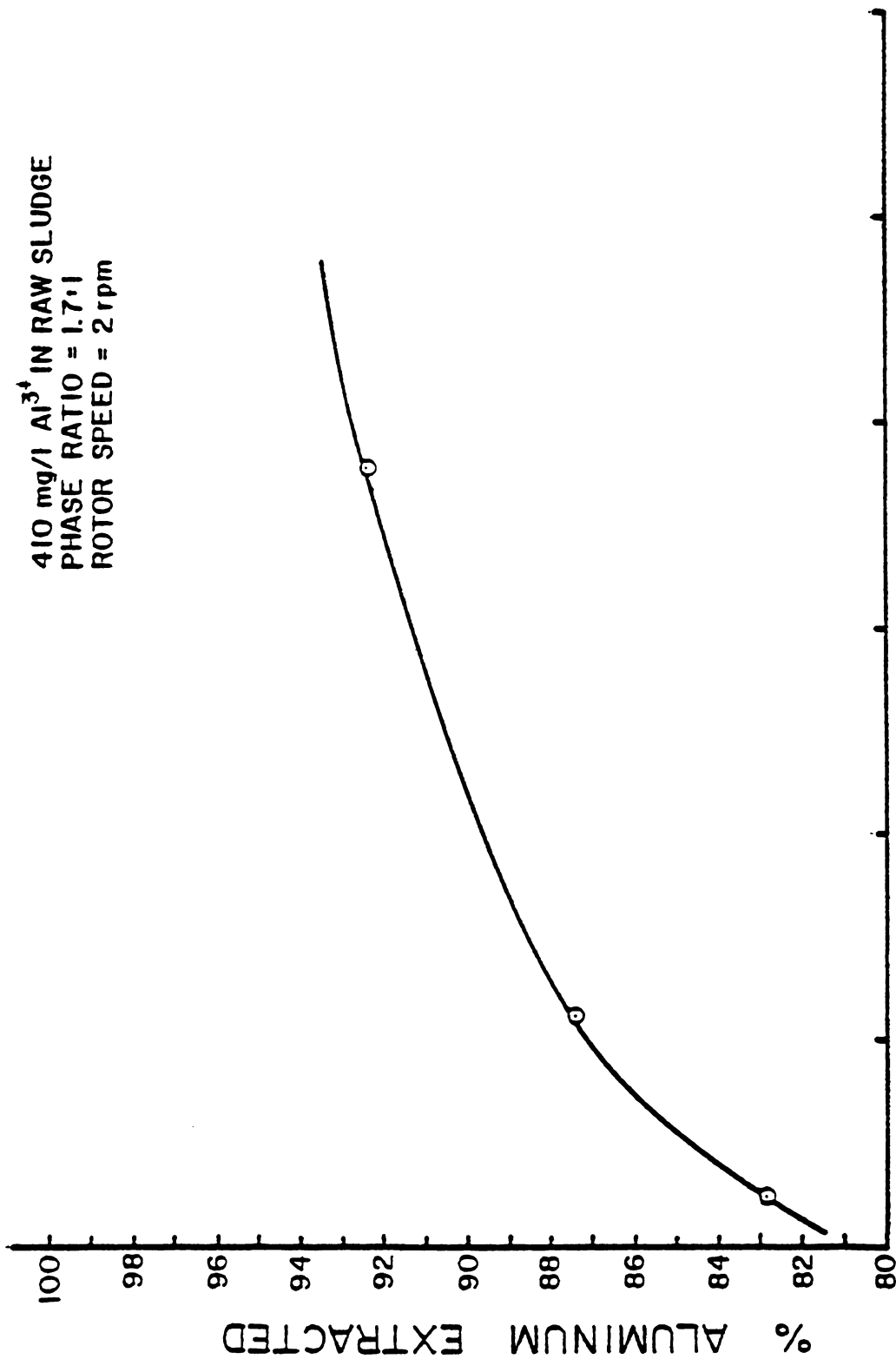


FIGURE 6-2
PERCENT ALUMINUM EXTRACTED AS A FUNCTION OF SLUDGE DETENTION TIME

to detention time. The RTL approached equilibrium at a sludge detention time of 88 minutes or a sludge flow rate of 0.40 gpm. Extraction of 90 percent of the aluminum in the sludge under the conditions listed on Figure 6-2 required a sludge detention time of 65 minutes or a sludge flow rate no greater than 0.54 gpm.

The phase ratio at which the RTL was operated also had an effect on the amount of aluminum extracted. As shown on Figure 6-3, for a given sludge aluminum concentration, sludge flow rate, and rotor speed, the percent of aluminum extracted is proportional to the phase ratio. Higher phase ratios would increase the amount of aluminum extracted by increasing the aluminum concentration gradient throughout the RTL.

Raffinate pH was used as an indicator of aluminum extraction throughout the RTL run. Figure 6-4 shows raffinate pH plotted as a function of detention time. Longer detention times resulting in lower raffinate pH's. When this relationship is considered in conjunction with Figure 6-2, the validity of the use of raffinate pH as an indicator of extractor is proven. The fact that steady-state conditions existed in the RTL and did not exist in the mixer-settler can explain why this relationship developed during RTL operation.

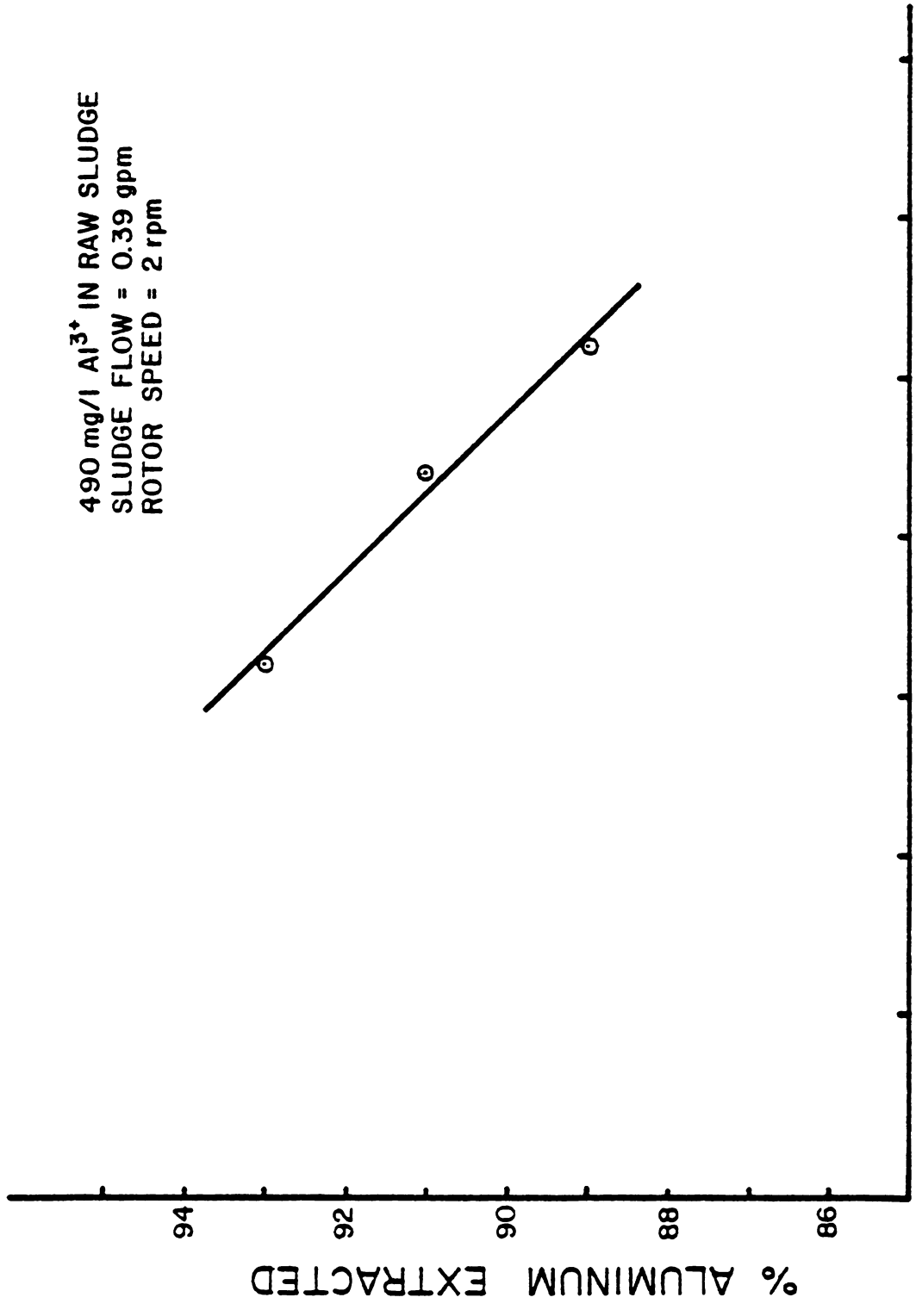


FIGURE 6-3
PERCENT ALUMINUM EXTRACTED AS A FUNCTION OF PHASE RATIO

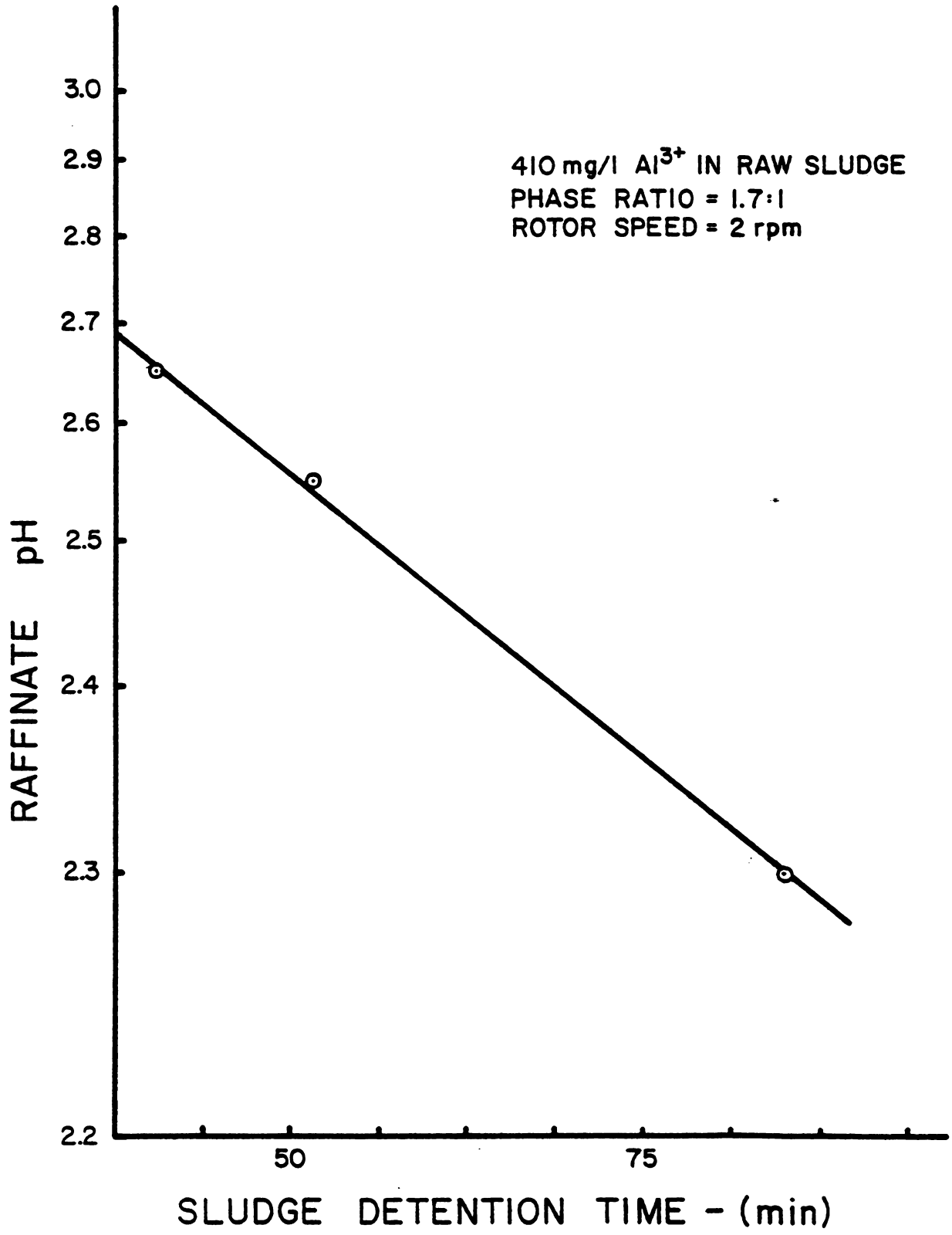


FIGURE 6-4
RAFFINATE pH AS A FUNCTION OF SLUDGE DETENTION TIME

Due to time limitations, only one extractant strength could be evaluated. Because of the long detention times involved with the RTL, very large contactors would be needed to carry out efficient aluminum extraction. To reduce capital costs, it would be desirable to reduce the necessary detention time. One way to do this would be to increase the extractant concentration. While bench or pilot work would be necessary to evaluate the effect of extractant concentration on detention time, it is not unreasonable to assume that, for a given sludge aluminum concentration, higher extractant concentrations could be used to reduce detention times and still maintain high extraction efficiencies.

CHAPTER 7

COST ANALYSIS OF ALUM RECOVERY

7.1 Introduction

This analysis is based on the results of pilot-plant operation and compared mixer-settler and RTL Contactor options. Three alternatives were analyzed:

1. Mixer-settler with centrifuge for solvent recovery
2. RTL Contactor with centrifuge for solvent recovery
3. RTL Contactor

In order to conduct this analysis, it is necessary to make assumptions with respect to the sludge flow rate and average amount of alum used. The following values were used in this analysis:

1. The average sludge flow rate is 300,000 gal/day.
2. The average alum usage is 22 tons/day.

These values are for the purposes of this analysis only. Pilot work has shown that the character and volume of sludge produced at Tampa varies greatly and a final design would have to take this into consideration.

7.2 Tampa Facility Design

7.2.1 Sludge Pretreatment

7.2.1.1 Sludge Collection

The sludge can be collected via the existing mechanical removal system and pumped into a thickener with existing sludge handling pumps.

7.2.1.2 Thickener

An elevated thickener is currently in use at the sludge dewatering facility. This unit could be moved and installed adjacent to the thickened sludge holding tank.

7.2.1.3 Thickened Sludge Holding Tank

The existing mixed holding tank will be used to collect the thickener underflow and to keep the sludge mixed for uniform feed to the recovery circuit.

7.2.1.4 Sludge Pumping to Recovery Site

The sludge will be pumped to the recovery site by an existing submerged pump in the thickened sludge holding tank via an existing six-inch PVC line which runs directly to the recovery site.

7.2.2 Acid Storage

The concentrated sulfuric acid demand of the stripping circuits will be 1750 gallons per day. An existing elevated acid storage tank with a capacity of 10,000 gallons will be moved to the recovery site. Another elevated tank of equal size will be purchased. Together, these tanks will provide about 11 days of acid storage. The acid will be pumped to the stripping circuits via metering pumps.

7.2.3 Mixer-Settler Extractor

Two stainless steel mixer-settler units are required for Alternative 1. The 4815 gallon mixers would measure 9' x 9' x 9' and would contain two equally sized mixing chambers separated by a horizontal baffle. The settlers would have a surface area of 162 ft^2 and be 5 feet deep.

7.2.4 RTL Contactor Extractor

If Alternative 2 or 3 is chosen, four stainless steel RTL units measuring 7.5 feet in diameter and 26.2 feet long are required.

7.2.5 Centrifuge - Alternative 1

Alternative 1 requires two 36 gpm centrifuges (DeLaval Model PX) to recover solvent from 75,000 gallons of bleed solids produced each day in the mixer-settlers. The bleed solids will be siphoned from the settler by three independently operated, perforated manifolds. The siphons will transport the bleed solids to a 500 gallon holding tank from which two positive displacement pumps with 35 gpm capacities would feed the centrifuges. The operation of the centrifuges will require one operator on all shifts. The cost-effective operation of the mixer-settler option is dependent on the

efficient operation of the centrifuges. Pilot results show the solvent losses of two gallons per 1000 gallons of sludge can be expected.

7.2.6 Centrifuge - Alternative 2

The raffinate leaving the RTL Contactor contains 0.5 to 1 percent by volume of floating material which contains recoverable solvent. The loss of solvent in the floating material corresponds to 1.5 to 3.0 gallons of solvent lost per 1000 gallons of sludge treated. The use of a 10 gpm DeLaval Model PX centrifuge to process the floating solids can reduce solvent losses to 1 gallon per 1000 gallons of sludge. The centrifuge would operate 40 hours per week. The floating solids would be collected in a 3000 gallon float cell fitted with a surface skimmer. The floating solids would then flow by gravity to a 5000 gallon holding tank.

7.2.7 Strippers

In all alternatives, four stainless steel mixer-settler strippers will be used. The 2570 gallon mixers will measure 7' x 7' x 7'. The settlers will have a surface area of 85 ft^2 and be 2 feet deep.

7.2.8 Solvent Reservoir

The stripped solvent reservoir will have a 5000 gallon capacity and be located such that it is fed by gravity from the strippers.

7.2.9 Recovered Alum Storage

A 3500 gallon wet well will be provided for storage of recovered alum. A 100 gpm transfer pump will transfer the recovered alum to one of the two existing alum storage tanks. The other tank will be used to store commercial alum for make-up and emergency purposes.

7.2.10 Granulated Activated Carbon Columns

The granulated activated carbon column required for each circuit will be two feet in diameter and six feet high. It can be operated as a gravity feed column (if head requirements exist) or a pressurized column. Backwash provisions will be required. The carbon volume of the column is 18 ft^3 for each unit.

7.2.11 Waste Stream Neutralization

It will be necessary to neutralize the raffinate and the centrifuged waste solids prior to disposal. A 1000 gallon (five-minute detention) mixed tank will be provided

for lime addition. Slaked lime will be pumped from the existing slakers adjacent to the recovery plant site to the neutralization tank.

7.2.12 Building

For Alternative 1, a 20' x 40' building will be required to house the centrifuges, laboratory, and control room. Alternatives 2 and 3 would require a 20' x 30' building.

7.2.13 Sitework

The sitework includes preparing the ground for equipment installation, setting foundations for the mixer-settlers and/or RTL Contactors, moving the thickener and acid tank, preparing the site for the solvent reservoir, alum wet well, float cell, float solids storage tank, and pump house.

7.3 Capital Cost

The capital costs of the alum recovery alternatives are shown in Table 7-1.

7.4 Operating and Maintenance Cost

7.4.1 Acid

The demand is 11 tons of concentrated sulfuric acid per day. The bid price of acid in Tampa is about $38 per ton.

TABLE 7-1

ESTIMATED CAPITAL COSTS OF TAMPA FACILITY

	Alternative 1 Mixer-settler with Centrifuge	Alternative 2 RTL Contactor with Centrifuge	Alternative 3 RTL Contactor
Mixer-settler extractors	$125,000	-	-
RTL Contactors	-	$800,000	$800,000
Strippers	188,000	188,000	188,000
Additional acid tank	18,000	18,000	18,000
Solvent reservoir	8,000	8,000	8,000
Neutralization tank	5,000	5,000	5,000
GAC Filters	13,000	13,000	13,000
Float cell	-	19,000	-
Float storage	-	25,000	-
Bleed solids storage	2,000	-	-
Centrifuge	650,000	113,000	-
Centrifuge pump system	20,000	10,000	-
Alum holding tank	6,000	6,000	6,000
Alum transfer pump	6,000	6,000	6,000
Building	96,000	72,000	72,000
Sitework	100,000	100,000	100,000
Piping, valves	120,000	120,000	120,000
Electrical/Instrumentation	95,000	95,000	95,000
Organic inventory	37,000	40,000	40,000
SUBTOTAL	$1,565,000	$1,638,000	$1,471,000
Technical Fees and Contingencies (35 percent)	548,000	574,000	515,000
TOTAL CAPITAL COST	$2,212,000	$1,986,000	$1,986,000

7.4.2 Solvent

The cost of mixed solvent is $2.30 per gallon, based on $1.00 per gallon for kerosene and $.90 per pound of MDEHPA. Solvent losses for the three alternatives are listed below:

Alternative	Solvent Loss, gal per 1000 gal sludge
1	2.0
2	1.0
3	3.0

7.4.3 Alum Credit

Alum will be recovered at a rate of 19.8 tons per day. The cost of alum is assumed to be $100 per ton.

7.4.4 Lime Demand

Waste stream neutralization will require 1.5 tons of quicklime per day at $50 per ton.

7.4.5 Carbon Demand

The carbon demand is assumed to be 30 pounds per day at $1.00 per pound.

7.4.6 Labor

Alternative 1 will require one operator at all times or a total of four. The City of Tampa has one man available and only three additional operators are necessary.

Alternative 2 will require one operator and this person is already available.

Alternative 3 requires no additional personnel.

7.4.7 Sludge Conditioning and Hauling Savings

Tampa personnel indicate that savings in truck rental for dewatered sludge hauling and polymer used for sludge conditioning will amount to $102,000 per year.

7.4.8 Downtime

In Alternative 1, it is assumed that the system will be down 20 percent of the time due to dependence on the centrifuges. This is reflected in reductions in sludge conditioning and hauling savings and alum credit.

7.4.9 Power

Power costs are based on $0.04 per KWH.

ALTERNATIVE 1

4 strippers @ 10 HP each	=	40 HP @ $0.04/KWH	$11,000
2 extractors @ 15 HP each	=	30 HP @ $0.04/KWH	8,000
2 centrifuges @ 100 HP each	=	200 HP @ $0.04/KWH	52,000
		TOTAL	$71,000

ALTERNATIVE 2

4 strippers @ 10 HP each	=	40 HP @ $0.04/KWH	$11,000
4 contactors @ 5 HP each	=	20 HP @ $0.04/KWH	5,000
1 centrifuge @ 30 HP each	=	30 HP @ $0.04/KWH	3,000
		TOTAL	$19,000

ALTERNATIVE 3

4 strippers @ 10 HP each	=	40 HP @ $0.04/each	$11,000
4 contactors @ 5 HP each	=	20 HP @ $0.04/each	5,000
		TOTAL	$16,000

7.4.10 Maintenance

The annual maintenance cost will be assumed to be three percent of the initial equipment costs. A comparison of the operating and maintenance costs of the three alternatives is presented in Table 7-2.

7.5 Cost Analysis

It can be seen from Table 7-2 that Alternative 2 is the least expensive alternative and can be operated with a net savings in operating and maintenance costs. The costs of the three alternatives will be examined by two methods: capital amortization and capital pay back.

TABLE 7-2

ESTIMATED OPERATING AND MAINTENANCE COSTS FOR TAMPA FACILITY

	Alternative 1 Mixer-settler with Centrifuge	Alternative 2 RTL Contactor With Centrifuge	Alternative 3 RTL Contactor
Acid	$153,000	$153,000	$153,000
Solvent	504,000	252,000	756,000
Alum credit	-578,000	-723,000	-723,000
Lime demand	28,000	28,000	28,000
Carbon demand	11,000	11,000	11,000
Labor	60,000	-	-
Sludge savings	-82,000	-102,000	-102,000
Power	71,000	19,000	16,000
Maintenance	39,000	23,000	18,000
Patent royalty	50,000	50,000	50,000
TOTAL ANNUAL OPERATING COST	$256,000	-$289,000	$207,000

7.5.1 Capital Amortization

Listed below are the equivalent annual costs of the alum recovery alternatives:

Alternative	Equivalent Annual Cost[1]
1	$455,000
2	-$ 80,000
3	$394,000

1. Assumes seven percent interest over 20-year period (P/A = 0.09439)

Analyzing the cost data in this manner shows that the sludge treatment savings and alum credit in Alternative 2 not only offset operating costs, but also offset the amortized capital cost.

7.5.2 Capital Pay Back - Alternative 2

The City of Tampa has expressed an interest in determining how many years would be required to recover their capital investment. To conduct this analysis, an inflation rate of 10 percent was assumed. Only Alternative 2 can be analyzed in this manner. The results are presented below:

Year	Savings	Capital Outstanding
0	$ 0	$2,212,000
1	289,000	1,923,000
2	318,000	1,605,000
3	350,000	1,255,000
4	385,000	870,000
5	424,000	446,000
5.96	446,000	0

The capital pay back period with 10 percent inflation is 5.96 years if Alternative 2 is implemented.

CHAPTER 8

CONCLUSIONS AND RECOMMENDATIONS

8.1 Conclusions

In spite of the handicaps under which the mixer-settler pilot plant was operated, the feasibility of the alum recovery process was demonstrated at this scale. The cause of the discontinuous operation which prevented the desired evaluation of the alum recovery plant was not the fault of the basic recovery processes, extraction and stripping. Rather, it was the fault of the poor performance of the original centrifuge; a machine whose performance has no bearing whatsoever on the extracting and stripping of aluminum. The data which were collected showed that 90 percent of the aluminum in the feed sludge can be recovered as contaminant-free, functional alum.

The scale-up of the mixers, both extractor and stripper, was poor, this was caused by the method in which they were designed. Mixing was inadequate at design mixer speeds and this greatly reduced the flexibility of the entire system. Future mixers should be designed, not on the basis of $N^3D^2 \leq 20$, but on the maintenance of geometric similitude and constant power input per unit mixer volume.

The optimum extractor mixer operating phase ratio was 2:1. This is based on the results of the aqueous entrainment

analysis. At operating phase ratios greater than 2:1, excessive raffinate was entrained into the loaded organic. This entrainment diluted and contaminated the recovered alum. Operating phase ratios less than 2:1 invited phase inversions.

The design of the settlers, in both extraction and stripping circuits, was proven sound by results of the pilot study. When properly operated, the settlers efficiently separated the dispersions fed to them. To prevent solids carry-over from the extraction to stripping circuit, the bleed solids layer had to be kept as low as possible in the settler to prevent high scour velocities in the organic portion of the settler.

The picket fences and dispersion introduction baffles were effective as settling aids and should be included in the design of future settlers.

The solvent was found to be unstable and lost extracting ability with use. Bench-scale tests with an RTL Contactor revealed that after being loaded with aluminum 13 times, the extractant could extract only 20 percent of the aluminum that it could when it was fresh. The mechanism by which the extracting ability of extractant is lost is not yet known.

A pilot-scale RTL Contactor was also evaluated at Tampa and the data produced indicate that aluminum can be extracted from the Tampa sludge at an efficiency of 90 percent or

greater. The major advantage of the RTL Contactor is that it does not produce bleed solids. Because there are no bleed solids, the RTL Contactor can make the liquid-ion exchange process cost-effective without a solvent recovery operation. To achieve 90 percent aluminum recovery efficiency, a detention time of 65 minutes was required. It is believed that the detention time can be reduced by using higher extractant concentrations and operating at phase ratios greater than unity.

A cost analysis comparing construction and operation of full-scale mixer-settler extractors and RTL Contactors at Tampa favors the use of RTL Contactors in conjunction with a centrifuge to recover solvent from the raffinate. If the cost of the recovered alum is credited against the amortized capital and operating costs, the City of Tampa could save $80,000 per year and recover their capital outlay in six years.

8.2 Recommendations

Based on the findings of this study, the City of Tampa should proceed with the design and construction of a full-scale liquid-ion exchange alum recovery facility for sludge treatment.

Alum recovery with liquid-ion exchange should be given serious consideration as a sludge processing alternative at

other facilities where the economic feasibility of coagulant recovery exists. Basic researching of the interaction between the extractant, diluent, and constituents of coagulant sludges is also warranted. In particular, the mechanism by which extracting ability of the extractant is lost should be investigated in detail.

APPENDICES

APPENDIX A

APPENDIX A

MIXER-SETTLER
OPERATING DATA
1979

Date / Parameter	3-23	3-26	3-29	3-30	3-31
Time of Operations, hrs.	6	12	16	21	30
Average sludge Flow, gpm	2.6	3.0	3.2	3.0	3.0
Average Solvent Flow, gpm	13.0	13.0	13.0	13.0	13.0
Feed Flow Ratio, O/A	5:1	4.3:1	4.1:1	4.3:1	4.1:1
Mixer Phase Ratio, O/A	-	5.5:1	-	-	6.6:1
Mixer Detention Time, min	-	23	-	-	19
Al^{3+} in Sludge @ pH 2.0, mg/l	-	320	-	320	-
Al^{3+} in Raffinate, mg/l	-	19	21	-	-
Raffinate pH	1.8	1.8	1.8	1.85	-
Strip I Phase Ratio, O/A	-	6:1	-	-	9:1
Al^{3+} in Recovered Alum, mg/l	-	7900	8600	-	-
Strip II Phase Ratio, O/A	-	6:1	-	-	5.5:1
Al^{3+} in Strip II Acid, mg/l	-	8200	8600	-	-

MIXER-SETTLER
OPERATING DATA

Date / Parameter	4-1	4-6	4-7	4-10	4-11
Time of Operations, hrs.	37	45	52	58	65
Average sludge Flow, gpm	3.2	3.2	3.1	3.6	4.5
Average Solvent Flow, gpm	13.0	13.0	13.0	13.0	13.0
Feed Flow Ratio, O/A	4.1:1	4.1:1	4.1:1	3.6:1	3.0:1
Mixer Phase Ratio, O/A	6.7:1	-	-	5.0:1	4.0:1
Mixer Detention Time, min	18	-	-	20	20
Al^{3+} in Sludge @ pH 2.0, mg/l	-	-	-	-	80
Al^{3+} in Raffinate, mg/l	6	-	10	8	4
Raffinate pH	-	2.0	-	1.9	2.1
Strip I Phase Ratio, O/A	-	-	-	4.0:1	-
Al^{3+} in Recovered Alum, mg/l	7850	-	7800	8700	9600
Strip II Phase Ratio, O/A	-	-	-	4.0:1	-
Al^{3+} in Strip II Acid, mg/l	13500	-	13000	15000	16000

MIXER-SETTLER
OPERATING DATA

Date	4-12	4-16	4-17	4-20	4-21
Parameter					
Time of Operations, hrs.	70	75	83	86	92
Average sludge Flow, gpm	5.7	2.8	5.2	2.8	3.3
Average Solvent Flow, gpm	12.0	12.5	12.5	12.0	12.0
Feed Flow Ratio, O/A	2.0:1	4.5:1	2.4:1	4.3:1	3.6:1
Mixer Phase Ratio, O/A	3.0:1	-	3.0:1	5.3:1	4.4:1
Mixer Detention Time, min	19	-	21	25	25
Al^{3+} in Sludge @ pH 2.0, mg/l	80	-	80	-	-
Al^{3+} in Raffinate, mg/l	-	5	-	-	82
Raffinate pH	2.2	2.05	2.2	1.85	-
Strip I Phase Ratio, O/A	-	-	3.0:1	-	-
Al^{3+} in Recovered Alum, mg/l	-	10600	-	-	13000
Strip II Phase Ratio, O/A	-	-	4.0:1	-	-
Al^{3+} in Strip II Acid, mg/l	-	15000	-	-	19000

MIXER-SETTLER
OPERATING DATA

Date	4-23	4-24	4-25	4-26	4-27
Parameter					
Time of Operations, hrs.	98	107	113	122	133
Average sludge Flow, gpm	3.3	3.8	4.1	3.4	4.2
Average Solvent Flow, gpm	12.0	12.5	12.5	12.0	12.5
Feed Flow Ratio, O/A	3.6:1	3.3:1	3.1:1	3.5:1	3.0:1
Mixer Phase Ratio, O/A	-	5.7:1	-	5.7:1	4.0:1
Mixer Detention Time, min	-	17	-	19	21
Al^{3+} in Sludge @ pH 2.0, mg/l	880	400	540	-	510
Al^{3+} in Raffinate, mg/l	22	38	-	60	-
Raffinate pH	2.2	2.3	2.25	2.2	2.45
Strip I Phase Ratio, O/A	-	3.3:1	-	3.4:1	3.7:1
Al^{3+} in Recovered Alum, mg/l	16000	17000	-	18000	-
Strip II Phase Ratio, O/A	-	4.0:1	-	4.0:1	4.0:1
Al^{3+} in Strip II Acid, mg/l	2000	21000	-	21000	-

MIXER-SETTLER
OPERATING DATA

Date / Parameter	4-28	4-29	5-1	5-2	5-3
Time of Operations, hrs.	142	147	158	167	176
Average sludge Flow, gpm	4.9	4.1	3.1	3.4	3.0
Average Solvent Flow, gpm	13.0	13.0	12.0	11.5	12.0
Feed Flow Ratio, O/A	2.7:1	3.2:1	3.9:1	3.4:1	4.0:1
Mixer Phase Ratio, O/A	-	4.0:1	5.3:1	4.9:1	4.7:1
Mixer Detention Time, min	-	21	23	22	25
Al^{3+} in Sludge @ pH 2.0, mg/l	-	-	580	-	1120
Al^{3+} in Raffinate, mg/l	-	34	-	107	-
Raffinate pH	2.25	2.15	-	2.1	2.0
Strip I Phase Ratio, O/A	2.9:1	3.1:1	4.0:1	7.6:1	11:1
Al^{3+} in Recovered Alum, mg/l	-	21000	-	25000	-
Strip II Phase Ratio, O/A	4.0:1	3.8:1	4.0:1	3.8:1	4.0:1
Al^{3+} in Strip II Acid, mg/l	-	27000	-	30000	-

MIXER-SETTLER
OPERATING DATA

Date	5-11	5-12	5-15	5-16	5-17
Parameter					
Time of Operations, hrs.	181	189	199	205	210
Average sludge Flow, gpm	2.8	2.3	2.3	2.3	2.7
Average Solvent Flow, gpm	-	13.5	11.0	11.5	11.5
Feed Flow Ratio, O/A	-	5.9:1	4.8:1	5.0:1	4.3:1
Mixer Phase Ratio, O/A	9.0:1	5.9:1	9.0:1	7.6:1	4.3:1
Mixer Detention Time, min	16	30	19	22	21
Al^{3+} in Sludge @ pH 2.0, mg/l	-	1130	-	510	600
Al^{3+} in Raffinate, mg/l	-	195	38	30	30
Raffinate pH	2.0	2.4	2.3	-	2.2
Strip I Phase Ratio, O/A	5.7:1	4.0:1	-	3.3:1	3.0:1
Al^{3+} in Recovered Alum, mg/l	-	31000	31000	30500	31000
Strip II Phase Ratio, O/A	4.0:1	4.0:1	-	3.2:1	2.9:1
Al^{3+} in Strip II Acid, mg/l	-	37000	35000	36500	36000

MIXER-SETTLER
OPERATING DATA

Date / Parameter	5-18	5-21	5-22	5-23	5-24
Time of Operations, hrs.	215	227	242	250	257
Average sludge Flow, gpm	3.1	3.6	3.9	3.3	3.1
Average Solvent Flow, gpm	12.0	12.0	12.0	12.5	13.0
Feed Flow Ratio, O/A	3.9:1	3.3:1	3.1:1	3.8:1	4.2:1
Mixer Phase Ratio, O/A	4.3:1	5.9:1	4.0:1	4.0:1	5.0:1
Mixer Detention Time, min	27	18	23	27	24
Al^{3+} in Sludge @ pH 2.0, mg/l	570	280	150	350	520
Al^{3+} in Raffinate, mg/l	20	10	10	52	67
Raffinate pH	1.8	1.9	2.3	2.3	2.25
Strip I Phase Ratio, O/A	-	4.4:1	3.9:1	4.0:1	4.6:1
Al^{3+} in Recovered Alum, mg/l	34000	31000	30000	31000	28000
Strip II Phase Ratio, O/A	-	4.8:1	4.0:1	4.0:1	4.2:1
Al^{3+} in Strip II Acid, mg/l	39000	35000	36000	35000	39000

MIXER-SETTLER
OPERATING DATA

Date / Parameter	5-25	5-27	5-28	5-29	5-30
Time of Operations, hrs.	269	279	285	290	298
Average sludge Flow, gpm	2.9	1.7	2.2	2.3	2.2
Average Solvent Flow, gpm	13.0	13.0	13.0	12.5	13.0
Feed Flow Ratio, O/A	4.5:1	7.7:1	5.9:1	5.4:1	5.9:1
Mixer Phase Ratio, O/A	6.1:1	7.7:1	5.9:1	6.4:1	9.0:1
Mixer Detention Time, min	21	30	31	26	20
Al^{3+} in Sludge @ pH 2.0, mg/l	430	-	-	1180	830
Al^{3+} in Raffinate, mg/l	-	-	160	200	85
Raffinate pH	2.25	1.95	1.9	2.05	2.15
Strip I Phase Ratio, O/A	4.0:1	4.0:1	4.2:1	4.0:1	5.1:1
Al^{3+} in Recovered Alum, mg/l	-	-	30000	30000	30000
Strip II Phase Ratio, O/A	4.0:1	4.2:1	4.7:1	4.7:1	3.6:1
Al^{3+} in Strip II Acid, mg/l	-	-	43000	42000	43000

MIXER-SETTLER
OPERATING DATA

Date	5-31	6-4	6-5	6-6	6-8
Parameter					
Time of Operations, hrs.	304	316	323	335	348
Average sludge Flow, gpm	1.8	3.0	3.0	3.5	1.0
Average Solvent Flow, gpm	12.5	12.0	12.5	12.5	8.5
Feed Flow Ratio, O/A	6.9:1	4.0:1	4.2:1	3.6:1	8.5:1
Mixer Phase Ratio, O/A	9.0:1	5.7:1	4.6:1	-	9.0:1
Mixer Detention Time, min	24	19	24	-	44
Al^{3+} in Sludge @ pH 2.0, mg/l	770	360	-	210	950
Al^{3+} in Raffinate, mg/l	30	-	17	32	-
Raffinate pH	2.1	2.2	2.1	2.3	1.95
Strip I Phase Ratio, O/A	5.3:1	-	3.9:1	4.0:1	9.0:1
Al^{3+} in Recovered Alum, mg/l	28000	-	30000	26000	-
Strip II Phase Ratio, O/A	4.0:1	-	3.8:1	3.9:1	3.0:1
Al^{3+} in Strip II Acid, mg/l	43000	-	44000	47000	-

MIXER-SETTLER
OPERATING DATA

Date / Parameter	6-9	6-13	6-16	6-20	6-21
Time of Operations, hrs.	359	369	372	378	383
Average sludge Flow, gpm	1.0	2.0	2.3	2.0	2.3
Average Solvent Flow, gpm	6.0	6.0	7.0	6.0	6.0
Feed Flow Ratio, O/A	6.0:1	3.0:1	3.0:1	3.0:1	2.6:1
Mixer Phase Ratio, O/A	11:1	3.0:1	3.0:1	3.0:1	2.6:1
Mixer Detention Time, min	37	68	64	58	56
Al^{3+} in Sludge @ pH 2.0, mg/l	-	-	-	700	-
Al^{3+} in Raffinate, mg/l	-	57	-	31	-
Raffinate pH	2.0	2.2	2.1	1.9	1.8
Strip I Phase Ratio, O/A	7.3:1	8.1:1	-	7.3:1	6.4:1
Al^{3+} in Recovered Alum, mg/l	-	27000	-	28000	-
Strip II Phase Ratio, O/A	2.7:1	3.0:1	-	3.4:1	3.3:1
Al^{3+} in Strip II Acid, mg/l	-	44500	-	43000	-

MIXER-SETTLER
OPERATING DATA

Date / Parameter	7-6	7-11	7-12	7-13	7-14
Time of Operations, hrs.	391	399	405	410	417
Average sludge Flow, gpm	2.5	2.7	2.7	2.9	2.7
Average Solvent Flow, gpm	9.0	9.0	7.0	9.0	9.0
Feed Flow Ratio, O/A	3.6:1	3.3:1	2.6:1	3.1:1	3.3:1
Mixer Phase Ratio, O/A	3.6:1	5.8:1	3.3:1	3.1:1	3.3:1
Mixer Detention Time, min	46	24	38	38	45
Al^{3+} in Sludge @ pH 2.0, mg/l	210	560	-	-	-
Al^{3+} in Raffinate, mg/l	15	18	-	-	41
Raffinate pH	-	2.2	2.1	1.85	2.1
Strip I Phase Ratio, O/A	13:1	9.0:1	9.0:1	4.6:1	3.6:1
Al^{3+} in Recovered Alum, mg/l	33000	39000	-	-	40000
Strip II Phase Ratio, O/A	-	3.7:1	3.5:1	4.7:1	3.6:1
Al^{3+} in Strip II Acid, mg/l	43000	47000	-	-	42000

MIXER-SETTLER
OPERATING DATA

Date / Parameter	7-20	7-23	9-6	9-11	9-12
Time of Operations, hrs.	423	426	431	439	450
Average sludge Flow, gpm	3.1	6.1	3.1	3.1	4.3
Average Solvent Flow, gpm	9.0	14.5	-	17	-
Feed Flow Ratio, O/A	3.9:1	2.4:1	-	5.4:1	-
Mixer Phase Ratio, O/A	2.9:1	2.7:1	3.2:1	5.9:1	3.8:1
Mixer Detention Time, min	37	19	34	21	22
Al^{3+} in Sludge @ pH 2.0, mg/l	690	180	600	-	570
Al^{3+} in Raffinate, mg/l	49	10	77	-	33
Raffinate pH	2.2	2.4	1.8	1.65	1.8
Strip I Phase Ratio, O/A	5.7:1	4.9:1	9.0:1	6.9:1	3.8:1
Al^{3+} in Recovered Alum, mg/l	42000	40000	48000	-	34000
Strip II Phase Ratio, O/A	2.1:1	1.9:1	4.8:1	3.3:1	5.7:1
Al^{3+} in Strip II Acid, mg/l	46000	37500	45000	-	42000

MIXER-SETTLER
OPERATING DATA

Date	9-13	9-14	9-18	9-19
Parameter				
Time of Operations, hrs.	461	467	474	480
Average sludge Flow, gpm	4.7	4.4	3.9	4.9
Average Solvent Flow, gpm	-	14	14	14.5
Feed Flow Ratio, O/A	-	3.2:1	3.6:1	3.3:1
Mixer Phase Ratio, O/A	3.9:1	4.0:1	4.3:1	-
Mixer Detention Time, min	20	21	21	-
Al^{3+} in Sludge @ pH 2.0, mg/l	770	680	450	540
Al^{3+} in Raffinate, mg/l	52	-	29	99
Raffinate pH	2.05	2.0	2.0	2.1
Strip I Phase Ratio, O/A	2.3:1	3.6:1	3.2:1	-
Al^{3+} in Recovered Alum, mg/l	30500	-	46000	36000
Strip II Phase Ratio, O/A	3.3:1	3.8:1	4.7:1	-
Al^{3+} in Strip II Acid, mg/l	40000	-	32000	34000

APPENDIX B

APPENDIX B

RTL CONTACTOR OPERATING DATA

Date	Time	Raw Sludge Flow, gpm	Phase Ratio	Raffinate pH	Al^{3+} @ pH 2.0, Raw Sludge mg/l	Al^{3+} Raffinate mg/l	Al^{3+} @ pH 2.0, Raffinate mg/l
9/18/79	1530	0.40	0.5:1	2.2	450	19	22
9/18/79	2230	0.38	0.9:1	2.4	500	19	43
9/19/79	0430	0.40	0.6:1	2.5	540	26	58
9/19/79	2230	0.38	2.0:1	2.2	480	22	32
9/20/79	2230	0.40	1.3:1	2.3	490	24	46
9/22/79	2230	0.40	1.9:1	2.4	420	17	30
9/23/79	0700	0.40	1.9:1	2.3	450	42	36
9/24/79	1700	0.42	1.9:1	2.3	420	19	32
9/25/79	2230	0.63	1.6:1	2.6	390	27	49
9/26/79	0430	0.60	2.5:1	2.4	370	32	38
9/26/79	2230	0.88	1.7:1	2.7	420	51	72
9/27/79	0430	0.45	4.5:1	2.6	480	47	58
9/27/79	0730	0.47	4.3:1	2.6	460	69	80

APPENDIX C

APPENDIX C

CALCULATION OF AQUEOUS ENTRAINMENT IN LOADED ORGANIC

I. INTRODUCTION

Measurements of aqueous entrainment into the loaded organic were made by allowing one liter of organic taken from the extractor settler organic weir to settle for 24 hours. Two measurements were taken. The amount of entrained aqueous and the phase ratio at which the mixer was operating at the time of sampling are listed below:

Mixer Phase Ratio	Aqueous Entrainment, ppm
10:1	4100
2.4:1	100

II. SAMPLE CALCULATION

The measured entrainment values were confirmed by calculated values of entrainment. The calculated values were based on operating conditions existing at the time of entrainment measurements. Presented below is the calculation of an entrainment value based on the following operation conditions:

Aluminum in sludge at pH 2.0 = 950 mg/l Al^{3+}

Sludge flow = 1 gpm

Aluminum in raffinate = 32 mg/l Al^{3+}

Organic flow to stippers = 8 gpm

Aluminum in alum = 26,000 mg/l Al^{3+}

Aluminum in Strip II acid = 47,000 mg/l Al^{3+}

These are the conditions that existed when the measured entrainment equalled 4100 ppm.

The aluminum transferred from Strip II to Strip I was equal to:

$$\frac{77 \frac{ml}{min}}{1000 \frac{ml}{l}} \text{ X } 47{,}000 \text{ mg/l } Al^{3+} = 3600 \frac{\text{mg } Al^{3+}}{min}$$

The aluminum transferred to Strip I from the extractor can be founded by subtracting the amount of aluminum leaving the extractor in the raffinate from the amount of aluminum entering the extractor in the raw sludge. This assumes that the amount of aluminum leaving the extractor with bleed solids was negligible.

$$(950 \text{ mg/l } Al^{3+} \text{ X } 3.785 \text{ l/gal X } 1 \text{ gpm}) -$$
$$(32 \text{ mg/l } Al^{3+} \text{ X } 3.785 \text{ l/gal X } 1 \text{ gpm}) = 3500 \frac{\text{mg } Al^{3+}}{min}$$

Therefore, every liter of solvent entering the stripping circuit contained 120 mg/l Al^{3+}.

$$\frac{3500 \text{ mg } Al^{3+}}{3.785 \frac{l}{gal} \text{ X } 8 \text{ gpm}} = 120 \text{ mg/l } Al^{3+}$$

If it is assumed that 40 mg/l Al^{3+} are removed from the solvent by Strip I, the total amount of aluminum leaving the stripping circuit in the recovered alum was:

$$3600 \frac{\text{mg } Al^{3+}}{\text{min}} + (40 \text{ mg/l } Al^{3+} \text{ X } 3.78 \text{ l/gal X } 8 \text{ gpm}) =$$

$$4800 \frac{\text{mg } Al^{3+}}{\text{min}}$$

If the recovered alum flow was 77 ml/min, the recovered alum aluminum concentration in the absence of any dilution should have been:

$$\frac{4800 \frac{\text{mg } Al^{3+}}{\text{min}}}{77 \frac{\text{ml}}{\text{min}}} = \frac{62 \text{ mg } Al^{3+}}{\text{ml}}$$

$$62 \frac{\text{mg } Al^{3+}}{\text{ml}} \text{ X } 1000 \frac{\text{ml}}{\text{l}} = 62{,}000 \text{ mg/l } Al^{3+}$$

Having calculated what the recovered alum aluminum concentration should have been and having measured what it actually was, the amount of raffinate being carried into the stripping circuit as entrainment can be calculated. The variable, E, being the amount of entrainment having the units of ml/min.

$$(62{,}000 \text{ mg/l } Al^{3+} \times 77 \text{ ml/min}) + (32 \text{ mg/l } Al^{3+} \times E) = (26{,}000 \text{ mg/l } Al^{3+})\ (77 \text{ ml/min} + E)$$

$$E = \frac{110 \text{ ml}}{\text{min}} = 3600 \text{ ppm}$$

This calculated value of entrainment is very close to the measured value of 4100 ppm. The two measured entrainment values are plotted on Figure 5-5. The other data points on Figure 5-5 are calculated entrainment values derived with the above calculations.

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