RECLAMATION OF ALUMINUM FINISHING SLUDGES

by

F. Michael Saunders
School of Civil Engineering
Georgia Tech Research Corporation
Georgia Institute of Technology
Atlanta, Georgia 30332

DISCLAIMER

FOREWORD

The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water systems. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. The Clean Water Act, the Safe Drinking Water Act, and the Toxics Substances Contrl Act are three of the major congressional laws that provide the framework for restoring and maintaining the integrity of our Nation's water, for preserving and enhancing the water we drink, and for protecting the environment from toxic substances. These laws direct the EPA to perform research to define our environmental problems, measure the impacts, and search for solutions.

The Water Engineering Research Laboratory is that component of EPA's Research and Development program concerned with preventing, treating, and managing municipal and industrial wastewater discharges; establishing practices to control and remove contaminants from drinking water and to prevent its deterioration during storage and distribution; and assessing the nature and controllability of releases of toxic substances to the air, water, and land from manufacturing processes and subsequent product uses. This publication is one of the products of that research and provides a vital communication link between the researcher and the user community.

This report includes research results on the reclamation of aluminum-finishing sludges as commercial products. Results are presented for enhanced dewatering of these industrial sludges and the acidic extraction of aluminum from them. Project results indicate that sludge reclamation can be achieved, producing a commercially marketable product and eliminating a solid waste disposal problem for the industry.

Francis T. Mayo, Director
Water Engineering Research Laboratory

iii

ABSTRACT

Aluminum anodizing plants may typically produce up to 500 metric tons/ month of finished aluminum extrusions. In finishing aluminum extrusions, approximately 3 to 5 percent of the mass of the extrusions is discharged as soluble aluminum metal in plant wastewaters. This waste aluminum is typically neutralized and results in the production of a highly-gelatinous, aluminum-hydroxide sludge suspension which, when thickened and dewatered, can equal or exceed the mass of extruded aluminum products produced at a plant. This solid waste residue must then be disposed of in a landfill or by other acceptable methods. Solid waste reduction has an extremely high priority in this industry and can be addressed through alterations in aluminum finishing or waste treatment procedures or reclamation of the solid waste. This project investigated reclamation of dewatered sludges as commercial-strength, aluminum sulfate solutions, i.e., liquid alum.

Sludge dewatering with filter press systems was the focus of the initial phase of the project. Successful reclamation of dewatered sludges is dependent on obtaining a minimum dry solids content of approximately 21 percent, if a commercial-strength liquid alum is to be produced. Fixed-volume, recessed-chamber filter presses were shown to be capable of dewatering conventional-neutralization, aluminum-anodizing sludges to 25 percent and 27 percent at low (6-7 bar) and high (14-15 bar) pressures, respectively. Segregated neutralization sludges could be dewatered to solids contents of 49 percent and 51 percent at low and high pressures, respectively. Blending small volumes (5 to 30 percent) of segregated-neutralization suspensions with thickened segregated neutralization suspensions markedly improved sludge dewatering properties. A variable-volume recessed-chamber filter press produced dewatered conventional-neutralization sludges with solids contents of 25-31 percent. It was, therefore, demonstrated that fixed-volume and variable-volume filter presses were capable of producing dewatered sludge cakes with solids contents of greater than or equal to 21 percent and could be effectively used in conjunction with the reclamation of aluminum-anodizing sludge as liquid alum.

Extraction of aluminum from dewatered aluminum-anodizing sludge cakes is possible with addition of stoichiometric quantities of sulfuric acid. The exothermic, acidic extraction is extremely rapid and can be taken to completion at temperatures of 50-100°C in 30 to 60 min. Commercial-strength products of liquid alum (i.e., $\geq$ 8 percent as Al_2O_3) can be produced from conventional-neutralization, segregated-neutralizaiton, and etch-recovery solids. Trace metal concentrations are of limited concern, with the possible exception of nickel and tin concentrations in conventional-neutralization sludges. Reclamation of aluminum-anodizing sludges as liquid

alum was demonstrated to be an effective method for elimination of a major industrial solid-waste-disposal problem.

CONTENTS

FIGURES

TABLES

ACKNOWLEDGMENT

SECTION 1

INTRODUCTION

The use of extruded aluminum products for architectural and structural purposes in domestic and industrial markets in the United States is well established and continues to expand. Approximately 1 million metric tons of extruded products are annually anodized, painted or finished prior to use. Of the finishing processes employed, anodizing systems present some unique environmental problems. To anodize an extruded aluminum alloy, surface oxides, imperfections and deposits, resulting from high-temperature extrusion and manual handling, must be removed. This is commonly accomplished by chemical etching and results in the removal of a thin layer of the alloy metal from the surface of an extrusion. These alloy-metal and other finishing wastes are continuously discharged for treatment and disposal.

Conventional neutralization to neutral pH values and subsequent treat-ment of anodizing wastewaters results in formation of aluminum-hydroxide and other metal precipitates. Due to the highly hydrophilic characteristics of aluminum hydroxide, thickened sludge suspensions are usually very dilute (e.g., 1-2 percent dry solids content). In addition, these suspensions are difficult to dewater and typically contain high levels of residual moisture. The mass of dewatered sludge solids produced for disposal is extremely high relative to production rates for finished aluminum. For example, the mass of wet dewatered sludges produced at two anodizing plants, with production capacities of approximately 500 metric tons/month, was estimated to be 55 and 85 percent of the mass of finished aluminum products, assuming highly effective sludge dewatering could be accomplished (Saunders et al., 1982; Saunders et al., 1984). Without highly efficient dewatering, the final sludge mass would approach, or exceed, the mass rate of production of finished product. Therefore, regardless of issues related to environmental effects of chemicals contained in the dewatered sludges, the high relative mass, and the resulting high relative volume, of dewatered sludge solids produced constitute one of the major environmental problems with which the industry is faced. The research conducted and presented herein was focused directly on this issue, with major emphasis placed on reduction in the mass of sludge solids for disposal through use of extraction of aluminum from the sludges to produce a reclaimed, marketable product.

This report is the third in a series reporting on research focused on characterization, treatment, reclamation and disposal of aluminum anodizing wastewaters and sludges. The first report by Saunders et al. (1982) was focused on wastewater characterization and evaluation of conventional treatment technology. The focus of the second report by (Saunders et al.,

1

(1984) was on evaluation of innovative treatment technologies with potential for application in the industry. Of the technologies investigated, sludge reclamation using acidic extraction to produce a commercial-strength solution of aluminum sulfate was shown to have great potential for elimination of, or dramatic reduction in, sludge disposal requirements. This topic was therefore selected for further analysis and research and is the subject of this report.

The research project was conducted in two phases. The initial phase was focused on the high moisture content of dewatered sludges. Previous research (Saunders et al., 1984) indicated that a sludge moisture content of less than approximately 79 percent (i.e., sludge solids content greater than 21 percent) was required to be able to produce a commercial-strength solution of aluminum sulfate. Earlier research indicated that this level of dewatering was not routinely achieved at full-scale facilities and therefore presented a major impediment to implementation of reclamation of dewatered sludge as aluminum sulfate.

Pressure filtration using recessed-plate filter presses was reported to be the most effective approach to producing dewatered sludges with elevated solids contents (EPA, 1982; EPA, 1986). With this objective being central to the overall success of sludge reclamation, the initial phase of the project included a pilot-scale evaluation of the use of filter press systems to dewater thickened aluminum-finishing sludge suspensions. Two pilot-scale presses were used to investigate the performance of fixed-volume, recessed-plate filter presses at low and high pressures and variable-volume, recessed-plate filter presses, i.e., diaphragm filter press. This phase of the study was conducted at an aluminum-anodizing plant (Plant A) using thickened sludge suspensions routinely produced at the plant as feed suspensions for the filter presses. Thickened sludge suspensions were also obtained from a second aluminum-anodizing plant (Plant X) and transported to Plant A for dewatering studies with the filter presses.

The second phase of the project was focused on extraction of aluminum from dewatered sludges using sulfuric acid. This phase was conducted using laboratory-scale equipment with sludges produced by pressure filtration at the local aluminum-anodizing plant.

The overall objective of the project was to firmly establish the feasibility of the production of commercial-strength solutions of aluminum sulfate from aluminum-finishing sludges. Issues related to the use of pressure filtration for dewatering; incorporation of other innovative treatment technologies; chemical and physical requirements for aluminum extraction; and quality of aluminum-sulfate products were also investigated.

SECTION 2

CONCLUSIONS

The research was focused on filter-press dewatering and reclamation of three types of aluminum-anodizing sludges as commercial-strength liquid alum. Numerous conclusions were drawn from the study.

1. Conventional neutralization sludge suspensions obtained from Plant A had suspended solids concentrations of 0.76 to 5.12 g/L and averaged 2.5 g/L, and following gravity thickening had suspended solids concentrations which ranged from 18 to 62 g/L and averaged 36 g/L. Gravity thickened suspensions from the clarifier underflow had an average specific resistance of 3.7 Tm/kg and a specific-resistance cake solids content of 13.2 percent. Dewatered sludge cakes obtained from the vacuum filter at Plant A had solids contents which ranged from 9.9 to 15.9 percent and averaged 12.4 percent.

2. A conventional neutralization suspension following gravity thickening at Plant X had an average suspended solids concentration of 20.7 g/L, a specific resistance of 2.8 Tm/kg and a specific-resistance cake solids of 7.1 percent.

3. Segregated neutralization suspensions produced experimentally by direct neutralization of spent caustic etch with conventional anodizing acid at Plant A had suspended solids concentrations ranging from 80.1 to 180.1 g/L; an average specific resistance of 1.4 Tm/kg; and an average specific-resistance cake solids of 45 percent.

4. A dewatered etch-recovery sludge cake had a solids content of 91.6 percent.

5. Pressure filtration studies were conducted with two pilot-scale, fixed-volume, recessed-chamber filter presses at low (6-7 bar) and high (14-15 bar) pressures. Replicate runs of variable filtration time were virtually identical with respect to cumulative filtrate volume collected.

6. With data for filtration rate and mass and solids contents of dewatered cakes it was projected that the density of the aluminum-anodizing solids was 3200 kg/m^3.

7. The solids content of dewatered sludge cakes at the ultimate

3

completion of a filter press run were projected from filtration rate data. Conventional neutralization suspensions from Plant A had ultimate, dewatered-cake, solids contents of 23.4 percent and 27 percent, respectively, at low (6-7 bar) and high (14-15 bar) pressure filtration for suspensions with influent suspended solids concentrations of approximately 16-57 g/L. Plant X suspensions with an average suspended solids concentration of 20.7 g/L had ultimate dewatered-cake solids contents of 17.6 percent and 24.5 percent at low and high pressure filtration, respectively.

8. Segregated neutralization suspensions could be effectively dewatered separately and resulted in major improvement in the dewatering of conventional neutralization suspensions when blended with them. At low and high pressures, ultimate cake solids contents of 48.6 and 51.3 percent, respectively, were achieved with segregated neutralization suspensions. A batch, segregated-neutralization suspension produced at Plant A following a full-scale dump of a caustic etch tank was dewatered to 42 and 45 percent with low- and high-pressure filtration, respectively. Blends of segregated neutralization suspensions at 5 to 30 percent volumetric ratios with conventional neutralization suspensions resulted in ultimate solids contents of 33 to 39 percent with high-pressure filtration and 31 to 37 percent with low-pressure filtration.

9. A diaphragm press was used effectively to dewater all aluminum anodizing suspensions. Conventional neutralization suspensions had final cake solids contents of 25.4 to 31.2 percent, while 5 to 30 percent volumetric blends of segregated neutralization suspensions with conventional neutralization suspensions had solids contents of 31 to 43 percent.

10. Commercial-strength solutions of aluminum sulfate, i.e., liquid alum, can be effectively and rapidly produced with the addition of sulfuric acid. Addition of stoichiometric quantities of acid, based on sludge aluminum content, resulted in virtually complete extraction within 30-60 min.

11. Following the addition of acid and the initial elevation of temperature to > 95°C, maintenance of temperatures between 50-90°C had minimal impact on the extent of aluminum extraction.

12. Conventional neutralization sludge cakes with solids contents of 17.4 to 18.1 percent were extracted to produce liquid alum with concentrations of 7.4 to 8.8 percent as Al_2O_3. A total of 95 to 99 percent of the initial suspended solids were destroyed in the extraction and 93 to 97 percent of the aluminum was extracted.

13. Segregated neutralization sludge cakes with solids contents of 36.8 percent were extracted to produce liquid alum with concentrations of 8.1 to 9.0 percent as Al_2O_3 with the addition of water equal to 80 to 100 percent of the mass of wet sludge extracted.

14. An etch recovery sludge with a solids content of 91.6 percent produced a liquid alum with a concentration of 8.3 to 9.2 percent as Al_2O_3, following addition of water equal to 200 to 370 percent of the wet mass of etch recovery sludge added. Acidic extraction resulted in destruction of 54 to 85 percent of the suspended solids and extraction of 70 to 85 percent of the aluminum.

15. Addition of segregated-neutralization or etch-recovery solids to conventional neutralization solids increased the aluminum content of the blended sludge and could be effectively extracted to easily produce commercial-strength liquid alum.

16. A survey of numerous aluminum-anodizing sludges indicated that aluminum, sodium and iron were the major metals, accounting for the following respective percentages of total dry solids: 22 to 48 percent; 0.6 to 8.9 percent; and 0.17 to 1.13 percent. These three metals accounted for 27 to 57 percent of sludge dry solids.

17. Major heavy metals were nickel and tin with concentrations ranging from trace levels to 13,300 mg/kg (or 1.33 percent of dry solids). Other metals found occasionally at concentrations of 300 to 1000 mg/kg were chromium, copper, lead and zinc. Arsenic, cadmium, mercury, silver and selenium were only found at trace (<2 mg/kg) levels.

18. The cadmium, chromium and iron concentrations of liquid alum produced from conventional neutralization sludges were less than those of commercial products, while lead, silver and zinc concentrations were slightly above those for commercial product samples. The concentrations of nickel and tin were 6- to 17-fold higher than those in commercial product samples. The high nickel and tin concentrations were attributed to dragout from the two-step anodizing process and to the use of nickel in seal tanks. Segregation of these wastes from plant wastewaters may be needed to eliminate them from the sludges produced for extraction and reclamation.

SECTION 3

RECOMMENDATIONS

The experimental results presented herein confirm that virtually complete reclamation of aluminum-anodizing sludges as a commercial strength product is possible. Effective dewatering of aluminum-anodizing sludges is a necessity, as is control of heavy metal wastewaters discharged to the plant treatment systems. It is recommended that a national inventory of aluminum-finishing sludges be made to establish the quantity of aluminum available for reclamation as aluminum sulfate. The intensive survey should be focused on characterization of the sludges with respect to solids content, extractable aluminum and other extractable metals, including toxic heavy metals.

It is recommended that a treatment technology series be developed to summarize advancements in treatment and reclamation of aluminum-finishing sludges and wastewaters. The issues to be addressed should include wastewater characterization, sludge thickening and dewatering, lime recovery of etching wastes, advances in filter press systems and acidic extraction for reclamation of sludge aluminum as liquid alum.

SECTION 4

MATERIALS AND METHODS

ALUMINUM-ANODIZING-PLANT SLUDGES AND WASTEWATERS

The initial phase of the project was focused on evaluation of pressure
filtration of thickened aluminum-anodizing suspensions using pilot-scale and
laboratory-scale pressure filtration equipment. These studies were
conducted at the site of an aluminum-anodizing plant in the metropolitan
Atlanta area. This plant, referred to herein as "Plant A", is an integrated
aluminum extrusion and anodizing facility with extensive fabrication
capabilities for use in the production of prefabricated architectural
components.

The aluminum finishing line at Plant A employed a system designed for
production of anodized extrusions using conventional sulfuric-acid and
electrolytic-coloring anodizing procedures to produce a range of clear,
bronze and black finishes. The anodizing line was a single-pass system
which included the following sequential finishing steps: alkaline cleaner;
caustic etch; desmut; sulfuric-acid anodize; electrolytic coloring; and seal.
Each finishing step, except the final seal, was followed by a reclaimed-
water rinse. Finishing solutions were indicated as being standard
proprietary formulations typically employed in the industry. Further
specific elaboration was not provided. The caustic etch finishing solution
was a proprietary "no-dump" formulation which resulted in minimal direct
wastage of spent etch. However, the tank contents were annually discharged
to waste because of the accumulation of impurities. In addition, when
required for wastewater treatment, 1- to 3-m^3 (260 to 800 gallon) quantities
of caustic etch were discharged to a small holding tank for use in
wastewater neutralization. Spent concentrated acids from desmut, anodizing
and electrolytic coloring were accumulated in a large, 19-m^3 holding tank
for use with spent caustic etch in wastewater neutralization.

Wastes discharged from the anodizing line were contained in one of
three separate flows, i.e., spent finishing acids; spent caustic etch; and
rinsewaters. The rinsewater flow, in addition to rinse tank overflows,
contained all spills and dragout from finishing tanks which were discharged
to the collection pit immediately below the finishing-line tanks. Spent
finishing-acid and caustic-etch flows were collected and stored separately
for use in wastewater neutralization.

The wastewater treatment system included wastewater neutralization;
pump transfer; polymer flocculation; and gravity sedimentation. Clarified
wastewater was discharged to a storage tank for recycle and use as a

7

reclaimed rinsewater on the anodizing line. Thickened underflow suspensions from the sedimentation basin were discharged to a rotary-drum vacuum filter. Filtrate water was discharged to the neutralization system and dewatered sludge solids were accumulated in dumpster tanks for ultimate disposal in a contract landfill. A schematic diagram of the wastewater treatment system is presented in Figure 1.

The suspensions routinely used at Plant A were those collected from the underflow of the gravity sedimentation basin. No preferential operational control over the wastewater treatment system or the sedimentation basin was provided by plant personnel for the study because the treatment system was operated near the maximum available capacity throughout the experimental study. Therefore, wastewater suspensions were those available in the influent line to the vacuum filter.

On selected occasions it was necessary to examine sludge suspensions which were not conditioned with polymer. In these few instances, neutralized suspensions from the third stage of the neutralization basin were pumped into numerous 0.2-m^3 containers and allowed to gravity thicken. Clarified supernatant liquid was withdrawn by siphon and the thickened sludge samples were combined for testing purposes.

On one occasion, a thickened sludge suspension from another aluminum anodizing plant, Plant X, was examined in the pressure filtration portion of the study. This local plant was similar to Plant A with respect to both the finishing line and the wastewater treatment system. Plant X, however, did not employ electrolytic-color anodizing but used integral-color anodizing to produce bronze finishes. A 1.5-m^3 volume of a gravity thickened suspension was transported in 0.2-m^3 containers to Plant A for examination.

Finally, wastewater samples were provided by numerous plants for metal analysis and characterization with respect to alum production. In addition to Plants A and X, other plants (i.e., Plants B, C, D, E, F and I) provided conventional-neutralization, segregated-neutralization and etch-recovery suspensions and dewatered sludge cakes for analysis.

EXPERIMENTAL EQUIPMENT

Pressure Filtration Systems

Two filter presses were used to examine the dewatering properties of thickened suspensions from aluminum anodizing lines.

Netzsch Press--
A Model 470 filter press manufactured by Netzsch, Inc. of Exton, PA was used extensively throughout the initial phase of the project. The filter press system was composed of a progressive-cavity pump; an in-line accumulator tank with a separate air-compressor system; a set of 470 mm x 470 mm ungasketed polypropylene plates with filter cloths; and a manual, hydraulic-pump closure system. The fixed-volume recessed plates formed a chamber with a dewatered-cake thickness of 30 mm; had a total filter area of

Influent
Wastewater·
Combined Rinsewaters
and Spills

Spent Acid
Spent Etch and
Virgin Caustic

NEUTRALIZATION
BASIN

Polymer

Filtrate
to
Wastewater
Influent

CLARIFIER

Dewatered
Sludge

Landfill

ROTARY
VACUUM
FILTER

Clarified
Effluent

Plant
Recycle

STORAGE
TANK

Sewer
Discharge
Overflow
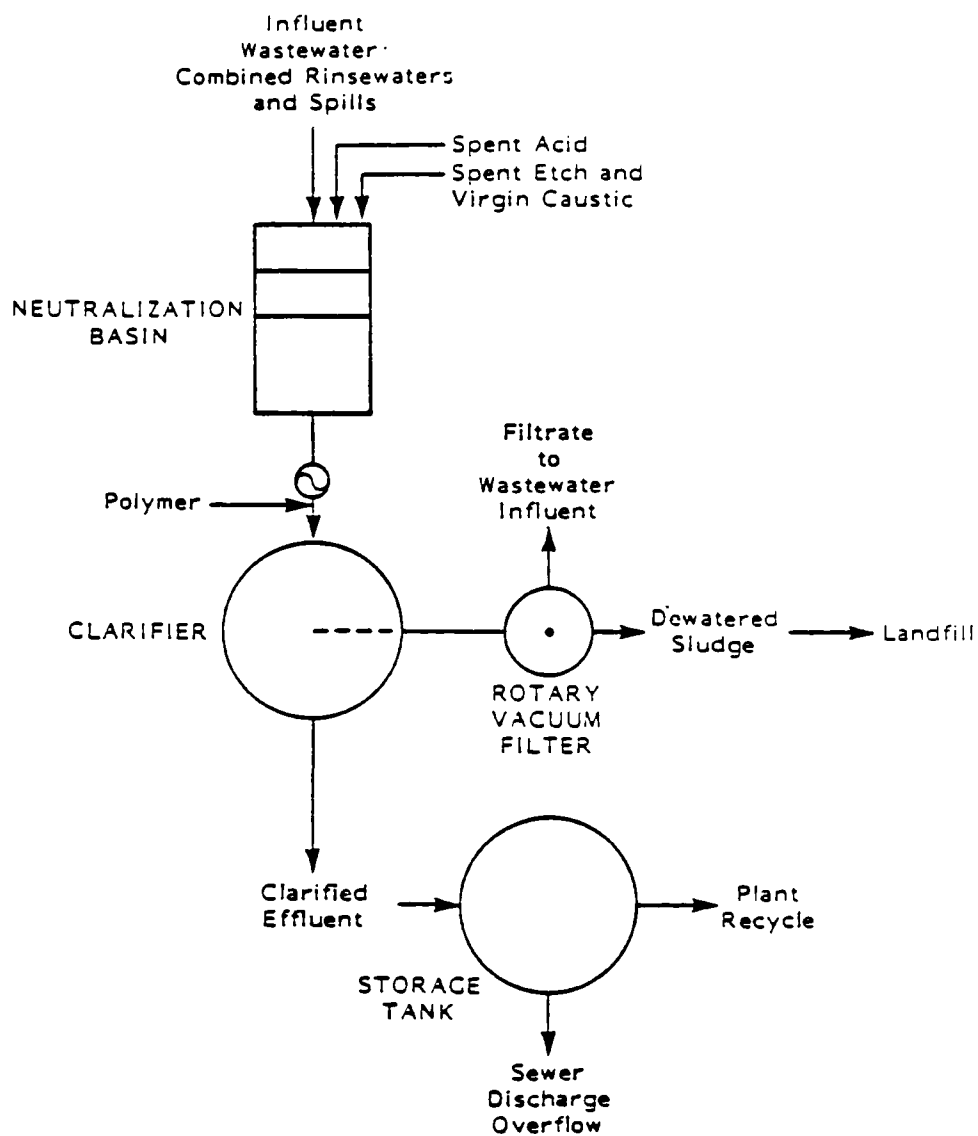
Figure 1.   Schematic diagram of wastewater treatment
system at Plant A.

9

0.346 m^2/chamber; and had a chamber volume of 4.6 x 10^{-3} m^3/chamber. The filter cloth used throughout the study was a multifilament polypropylene cloth (style number: 42614-3-HS; weight: 475 g/m^2 (14 oz/yd^2); weave: oxford; count: 75 x 21; permeability: 0.6-0.9 m^3/m^2·min (2-3 cfm/ft^2); manufacturer: C. Goodman and Company, Inc., Paterson, NJ). Initial testing by the manufacturer of the filter press indicated this to be the most appropriate media for the sludge suspensions being examined. The performance of the media was excellent throughout the study and no further evaluation of media was necessary.

The pressure regulation system for the filter press allowed for operation over a broad range of pressures and within narrow limits. Automatic adjustment of the internal pressure of the influent sludge suspension using the accumulator tank allowed for operation within pressure limits of ± 0.5 bar (± 50 kPa). When operated to simulate a filter press at low pressure, the range of pressure was manually set at 6-7 bar (600-700 kPa), while high-pressure operation was at 14-15 bar (1400-1500 kPa). These ranges are typical of those used in practice for similar systems. For example, maximum operation pressures for low- and high-pressure systems were indicated by EPA (1986) to be 6.9 bar (690 kPa) and 15.5 bar (1550 kPa). It is important to note that unlike many earlier plate-and-frame and filter press systems, the operating pressure for these systems was constant throughout a run and did not increase with time through a run. This was accomplished by use of a pressure sensor on the accumulator tank to control the feed pump. When the pressure in the accumulator tank feeding the press dropped to the lower set-point pressure (i.e., 6 bar or 14 bar), the feed pump was activated and the accumulator tank and filter chambers were gradually pressurized to the higher set-point pressure (i.e., 7 bar or 15 bar). This operating mode minimized energy requirements for the feed pump and provided for constant-pressure filtration.

In addition to operation of the Netzsch press with fixed-volume, recessed-chamber plates at low and high pressures, variable-volume, recessed-chamber filtration was examined. The variable-volume plates, or diaphragm plates, were similar in construction to the fixed-volume plates with the exception that each chamber contained an inflatable bladder used to compress or "squeeze" a sludge cake prior to discharge. The internal bladder was pressurized using compressed nitrogen to a maximum pressure of 15 bar (1500 kPa). The filter cloth employed was identical to that used on fixed-volume plates.

JWI Press--
A laboratory-scale filter press, manufactured by JWI, Inc. of Holland, MI and provided for use on the project by DMP Corporation of Fort Mill, SC, was used in parallel with the larger Netzsch filter press. The JWI press was composed of a two-stage, air-driven diaphragm pump; a set of 250 mm x 250 mm, ungasketed polypropylene plates with filter cloths; and a manual hydraulic-pump closure system. The fixed-volume recessed plates formed a chamber with a dewatered-cake thickness of 25 mm; had a total filter area of 7.63 x 10^{-2} m^2/chamber; and had a chamber volume of 9.5 x 10^{-4} m^3/chamber. The filter cloth used was identical in composition to the multifilament polypropylene cloth used on the Netzsch press. The JWI press did not have

an accumulator tank or any other pressure-regulation system. Sludge suspensions were fed into the filter press under the direct pressure provided by the diaphragm pump, which was driven using an in-plant compressor system. Filtration pressures ranged from 6 bar (600 kPa) to 7 bar (700 kPa).

Aluminum Extraction Reactor

Acidic extractions of dewatered sludge cakes were conducted in a laboratory-scale batch reactor. An insulated 2-L pyrex beaker with a watch-glass cover and placed on a laboratory hot plate was the basic reactor. A variable-speed laboratory mixer was used to continuously mix the contents of the reactor during an extraction. The hot plate was controlled by a thermostat-probe placed in the reactor and was used to maintain reaction temperatures of 50 to 90°C.

Segregated Neutralization Reactor

On several occasions, segregated neutralization of small volumes of caustic etch and spent acid was conducted at Plant A using a plastic-lined, 0.2-m^3 container. An approximate volume of 0.1 m^3 of spent caustic obtained directly from the caustic etch tank was placed in the container. Spent acid, obtained directly from a sulfuric-acid anodizing tank, was pumped with peristaltic tubing pumps into the container. A high-speed mixer was used to mix the container. Pumping was discontinued when the pH of the neutralized suspension reached a value of approximately 9.5. The neutralized suspension was then used directly in pressure filtration studies.

OPERATIONAL PROCEDURES

Pressure Filtration

The Netzsch press was used as the primary filtration system throughout the study. The operational procedures were virtually identical for low- and high-pressure operation with fixed-volume, recessed-chamber plates and operation as a diaphragm press was initially similar to the low-pressure system. The JWI press was used as a secondary system to examine low-pressure, fixed-volume, recessed-chamber filtration during the initial phase of the study. The JWI press was the only unit used for preparation of dewatered sludge cakes for the subsequent aluminum extraction studies.

Netzsch Press--
To initiate a filtration run, a sample of a thickened sludge suspension from a clarifier underflow was placed in a 0.2-m^3 plastic-lined container and mixed with two high-speed mixers placed at different depths. A sample of the mixed suspension was taken for analysis of suspended solids concentration, specific resistance and Capillary Suction Time (CST) to characterize the feed suspension. Typically, a large volume (e.g., 0.5 to 1 m^3) of a sludge suspension was collected in three to five 0.2-m^3 containers at one time to allow for a series of runs on a similar sludge suspension to be conducted over a 1- to 3-day period. The similarity of the various samples

included in such a series was confirmed by collection of an aliquot of a suspension in the feed reservoir immediately before each run and by analysis of the sample as indicated above.

To initiate filtration runs, minimum and maximum set points for filtration pressure were set at 6 and 7 bar and 14 and 15 bar for low- and high-pressure runs, respectively. The chamber plates were aligned and the press was closed using the manual hydraulic jack. The main control switch was activated and the pump was automatically started. The sludge suspension was pumped simultaneously into the accumulator tank and the filter press. In less than 30 seconds after starting the pump, filtrate began to be discharged from the press. At this time a stopwatch was activated to monitor elapsed filtration time. Within 2 to 3 min of starting the pump, the maximum set-point pressure was reached and the pump was automatically stopped. A check valve on the pump discharge prevented backflow through the pump and the pressure in the filter press was maintained with the pressurized suspension in the accumulator tank. When the pressure in the filter press dropped below the minimum set-point pressure, the pump was automatically activated and additional sludge was pumped into the press until the maximum set-point pressure was reached. This pumping cycle was continued throughout the length of a filtration run.

The volume of filtrate produced was recorded at frequent intervals initially (e.g., at 30-s to 2-min intervals) and at longer intervals (e.g., at 10- to 20-min intervals) as the filtration rate decreased through a run. The turbidity of the filtrate was monitored during the initial fifteen runs. The filtrate was virtually free of suspended solids as indicated by turbidity readings of less than 6 NTU for all runs. No further monitoring, other than visual confirmation of the lack of turbidity (i.e., <5 NTU) in the filtrate produced, was necessary or conducted for subsequent runs. The solids capture throughout all studies conducted was greater than 99.9 percent.

At the completion of a filtration run, internal pressure in the press was reduced to ambient pressure and the hydraulic closure device was released. Dewatered sludge cakes (usually two) were removed intact from the press and weighed on an industrial balance. The dewatered cakes had masses ranging from approximately 5 to 6 kg and were weighed to the nearest 30 g (i.e., ± 0.5 to 0.6 percent). After weighing a cake, portions of it were collected from each quadrant, being sure to avoid collecting samples from the tapered edges, the internal dimples and the central feed-port area. These samples were placed in a polyethylene sample bag and sealed for analysis for solids content.

For operation of the Netzsch press as a diaphragm press, the fixed-volume plates were replaced with membrane (i.e., variable-volume) plates and an auxiliary air-driven, duplex, diaphragm pump was installed to feed sludge suspensions into the diaphragm chambers. The pump discharge was located such that the accumulator tank was not used with the diaphragm system. Initial operation of the press proceeded as indicated above for low pressure filtration. After filling the press with solids by low-pressure filtration, the feed pump was stopped and the inlet valve to the chambers

was closed. The bladders internal to the membrane plates were then gradually pressurized using compressed nitrogen gas over a 5-min interval to a final bladder pressure of 15.5 bar. This latter phase using the internal bladder to compress the cake solids was referred to as the "squeeze" portion of the cycle. The time interval between termination of low-pressure filtration and initiation of the squeeze cycle was typically 2 to 3 min. Filtrate was collected and its volume was recorded throughout the run. The squeeze cycle was terminated when the filtrate production rate was zero. The dewatered solids were then collected, weighed, sampled and analyzed.

JWI Press--
    The procedures used to initiate a filtration run with this bench-scale system were similar to those for the Netzsch press. A sludge suspension was placed in a 20-L plastic bucket used as a feed reservoir and vigorously mixed. A sample was collected for analysis for suspended solids, specific resistance and CST. Since this system was only used for low-pressure filtration and the pump was driven with compressed air from the plant system, no pressure adjustment was necessary. The chambers were closed with a manual hydraulic jack and the pump was activated. Filtrate volume was monitored with time throughout the filtration cycle. At the end of a filtration run, the press pump was stopped and the dewatered cakes were removed, weighed and sampled for analysis.

Aluminum Extraction Reactor

    Aluminum extractions were conducted by addition of sulfuric acid to dewatered sludge cakes. In order to extract all available aluminum, as well as produce a product which was not excessively acidic, a procedure was developed to establish the quantity of sulfuric acid required to complete an extraction.

Sulfuric Acid Dosage--
    Earlier research by Saunders et al. (1982, 1984) indicated that aluminum-anodizing sludges could be assumed to be composed of three major components: (i) aluminum, as aluminum oxides and hydroxides; (ii) water; and (iii) trace quantities of metals, dissolved salts and other contaminants. It was also indicated that when aluminum extraction was being considered the sludge could be viewed as being composed of aluminum hydroxide ($Al(OH)_3$) and water. With this simplification, the acidic extraction of aluminum from dewatered aluminum-anodizing sludges was shown to be indicated by the following equation (Saunders and Harmon, 1984):

$$2Al(OH)_3 + x\ H_2O + 3H_2SO_4 \ \longrightarrow \ Al_2(SO_4)_3 + (6 + x)H_2O \qquad (1)$$
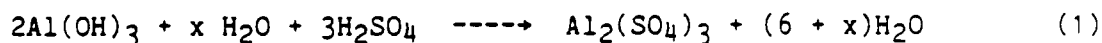
In this equation the dry solids of the dewatered sludge cake are represented by $Al(OH)_3$ and the quantity of moisture in the dewatered sludge cake is represented by $x\ H_2O$.

    From equation (1), the acid requirement for the extraction of aluminum from a sludge cake is 1.89 g H_2SO_4/g $Al(OH)_3$. From previous research it was shown that the aluminum content of the fixed solids in conventional aluminum-anodizing sludge cakes varied from 32.4 to 39.7 percent, and was in

general agreement with a theoretical value of 34.6 percent for the aluminum content of Al(OH)$_3$ (Saunders et al., 1982, 1984). Therefore, using the fixed solids content of the dewatered sludge cakes and an aluminum content of 0.346 g Al/g fixed solids, the acid requirement for extractions would be equal to 1.89 g H$_2$SO$_4$/g fixed solids. Based on measured concentrations of dry and fixed solids, as well as the added dosage of sulfuric acid, the final product alum concentration could be estimated. Commercial-grade liquid alum strength is typically presented in terms of Al$_2$O$_3$. As an example, a 1000 g (wet) aliquot of a sludge cake with a dry solids content of 21.3 percent and a fixed solids content of 16.8 percent would contain 832 g of moisture and would require 318 g of H$_2$SO$_4$ (1000 g * 0.168 g/g * 1.89 g/g). Conserving all mass components, the final product would be 28 percent as Al$_2$(SO$_4$)$_3$ or 8.3 percent as Al$_2$O$_3$. In these calculations, it was assumed that the additional loss in mass following exposure to 550°C in the analysis for fixed solids was indicative of the loss in bound moisture associated with highly gelatinous aluminum-hydroxide precipitates.

An alternative to using fixed-solids as a measure of Al(OH)$_3$ content consisted of analytically determining aluminum concentrations in dewatered sludge cakes. From equation (1), the acid requirement would be 5.44 g/g Al. Because it was based on aluminum content alone and no assumption was made regarding the chemical form of the aluminum precipitates, this acid dose is herein referred to as the "stoichiometric" acid requirement.

Aluminum Extraction--
In order to investigate the acidic extraction process with regard to reaction kinetics, stoichiometry, final product quality, and overall process feasibility, the following experimental strategy was developed. Sulfuric-acid extractions were conducted in the experimental reactor at various temperatures and levels of sulfuric acid addition. The kinetic progress of a reaction was monitored by withdrawal of samples at various intervals throughout an experimental run. A material balance on total mass, as well as aluminum, was conducted on each extraction to account for the fate of various constituents (e.g., sludge solids, residual aluminum, soluble aluminum, and acid) throughout an extraction. For example, the mass of aluminum is conserved throughout an extraction and at any point aluminum may exist in the solid phase (i.e., contained in unextracted sludge-cake solids) and the liquid phase (e.g., as soluble aluminum sulfate). Therefore, a mass balance on aluminum contributed from both phases would provide an accurate description of the conversion of solid-phase aluminum to soluble aluminum during an extraction. A material balance across the system would also provide a check on analytical procedures and techniques.

To initiate an extraction, a wet sludge cake was removed from refrigerated storage (2-5°C), weighed to the nearest 0.5 g, and placed into the reactor. Based upon a fixed solids or an elemental aluminum analysis, the quantity of required sulfuric acid was calculated according to equation (1). Due to the vigorous nature of the acid-sludge reaction, acid was slowly added to the reactor over several minutes. Because an excessive rate of acid addition resulted in the production of foam which would overflow the reactor, avoidance of excessive foam production was used to regulate the rate of acid addition. Following acid addition, the reactor was covered and

14

mixing was initiated. It is noted that initial temperatures reached approximately 90°C due to the heat of hydration from sulfuric acid addition. The contents of the reactor was allowed to, cool to and regulated at, the desired temperature (e.g., 50° to 90°C). The reactor cover was wrapped with parafilm wax to minimize evaporation and heat losses during an extraction.

Samples were withdrawn at various intervals throughout the reaction for analysis. The sampling procedure consisted of terminating mixing and disconnecting the stirring paddle shaft; removing the parafilm wax; and manually mixing the reactor contents while pouring a sample aliquot of approximately 100 mL into pre-tared sample containers. The reactor was reassembled and mixing and heat addition were continued within approximately 2-3 min. Sample preparation consisted of weighing the tared sample container with contents, followed by filtration of approximately 15 mL through a 0.45-μm membrane filter.

ANALYTICAL METHODS

In general three types of samples were analyzed during the study: (i) suspensions containing precipitated aluminum solids from wastewater treatment systems at full-scale aluminum-finishing plants; (ii) dewatered cakes produced by pressure filtration of wastewater suspensions; and (iii) highly-acidic, aluminum-rich suspensions and solutions produced by sulfuric-acid extraction of dewatered sludge cakes. Each type of sample dictated some different handling and analytical procedures, as indicated below.

Suspended Solids Concentration

Wastewater suspensions were examined for suspended solids concentrations using Gooch crucibles and glass-fiber filters as specified in Method 209 of Standard Methods (APHA, 1985). Fixed solids were determined by ignition of dried solids at 550°C in a muffled furnace for 30 min as indicated in Method 209D (APHA, 1985).

Cake Solids Content

Dewatered sludge cakes were examined for dry solids content using aluminum-foil weighing dishes. The dishes were used once and thrown away and were much easier to manage and handle than evaporating dishes for the large number of analyses required. All analyses were conducted in duplicate at 103-105°C in accordance with Method 209D (APHA, 1985). Drying periods of at least 18 h were shown to result in a constant mass for dewatered cakes; periods of 18-24 hours were used throughout the study. In addition, dessication times of greater than 3 h at ambient balance temperatures were used prior to determination of dish mass on an analytical balance. Fixed solids were determined by ignition of dried solids at 550°C.

Specific Resistance

The specific resistance of suspensions used in pressure filtration

15

studies was determined using standard procedures (O'Connor, 1984). The 9-cm Buchner funnel utilized had an effective filtration area of 6.4 x 10^{-3} m^2. A differential pressure of 0.5 bar (50 kPa) was maintained with a laboratory-scale vacuum pump and monitored with a 1-m mercury manometer. A Whatman No. 1 filter pad placed on a fine-mesh wire screen was used as a filtration medium. Filtrate was measured with time of filtration and dewatered solids were examined for total wet mass and cake solids content. A linear regression analysis was used with the linear form of the filtration equation to establish a value for specific resistance, which was expressed in units of terameters/kilogram (Tm/kg or 10^{12} m/kg).

Capillary Suction Time (CST)

Capillary Suction Time was measured with a standard CST apparatus (Type 92/1, Triton Electronics Limited; Dunmon, Essex, England) using an 18 mm reservoir. Whatman No. 40 chromatography paper was used as a standard medium throughout the studies. All measurements were taken in duplicate at the suspended solids concentration of the suspension.

Specific Gravity of Aluminum Extracts

Determination of the specific gravity of aluminum extracts was made immediately after collection of extract samples at elevated temperatures. Volume measurements for other analyses were made at these same temperatures, making subsequent temperature corrections for material balances unnecessary. The procedure followed was that in Method 213E of Standard Methods (APHA, 1985).

Aluminum Concentration

Aluminum determinations were conducted using several pretreatment and analytical procedures. Suspensions and dewatered cakes of aluminum-finishing solids were pretreated using a standard nitric-acid digestion (Method 302D in Standard Methods, APHA (1985)). Aluminum determinations were then made using atomic absorption spectrometry. A Perkin Elmer Model 303 spectrometer was used with a calibration curve, developed with primary-standard solutions, to establish aluminum concentrations.

All samples of suspensions extracted with sulfuric acid to produce high-strength aluminum-sulfate solutions were filtered through 0.45-μm membrane filters prior to analysis for aluminum. Two procedures were employed for determination of aluminum on these filtered samples. Atomic absorption spectrometry as indicated above was used. Because of the acidic properties of the filtrates, the high aluminum concentrations (e.g., Al $\cong$ 50-60 g/L), and the necessity to use extensive (e.g., 1000-fold) dilutions, experimental investigation proved that nitric-acid digestion was not necessary and was not employed prior to atomic absorption analyses.

A second analysis procedure was also used with acid extraction samples. This procedure was that used by alum manufacturers for use in determination of the aluminum content of liquid and crystalline aluminum sulfate (Allied Chemical, 1976). The procedure was based on complexation of aluminum with a

slight excess of EDTA and back titration of excess EDTA using a standard zinc sulfate solution. Comparison of the two procedures was highly favorable.

Trace Metal Analyses

Samples of suspensions, dewatered cakes and aluminum extracts were examined for trace heavy metals including silver (Ag), cadmium (Cd), chromium (Cr), nickel (Ni), lead (Pb), tin (Sn) and zinc (Zn). Analyses for these metals were preceeded by nitric acid digestion (Method 302D in Standard Methods, APHA (1985)). Atomic absorption spectrometry was used for metal analysis in accordance with Methods 303A and B (APHA, 1985).

SECTION 5

SLUDGE DEWATERING BY PRESSURE FILTRATION

Sludge suspensions from Plants A and X were examined. Two types of suspensions from Plant A were examined, as well as one produced experimentally using finishing wastes from Plant A, while only one suspension from Plant X was examined. The suspensions were examined using two filter presses (i.e., Netzsch and JWI presses). The Netzsch press was operated in three modes, i.e., as a low- and a high-pressure, fixed-volume system and a variable-volume system, while the JWI press was operated as a low-pressure, fixed-volume system.

SLUDGE SUSPENSIONS

Conventional Neutralization Suspensions

Plant A Suspensions--

At Plant A, influent wastewaters are conventionally neutralized in a 3-stage system using additions of spent acid and virgin or spent caustic-etch solutions in the two initial stages. Neutralized suspensions are collected in the variable-volume third stage, from which they are pumped to a clarifier with concurrent polymer addition. Thickened underflow suspensions from the clarifier are dewatered on a rotary vacuum filter and disposed of in a landfill.

Prior to and during pilot-scale pressure filtration studies at Plant A, the characteristics of selected wastes were monitored and are presented in Table 1. With one exception (i.e., Day 303), the suspended solids (SS) concentration of the neutralization basin effluent (designated AC1; i.e., Plant A; a Conventional-neutralization sludge; point 1 in the plant) ranged between 0.76 and 6.3 g/L and averaged 2.4 g/L. Two grab samples of neutralization basin effluent (i.e., AC1) were collected to study the dewatering of a sludge suspension without the aid of polymer conditioners. The characteristics of these two samples are presented in Table 2. The samples were concentrated to the levels indicated in Table 2 by gravity sedimentation. The relatively low suspended solids concentrations achieved, however, were consistent with previous thickening studies for unconditioned sludges (Saunders, et al., 1982) indicating poor thickening and dewatering properties for similar unconditioned suspensions.

On Day 303 at noon, a large volume (e.g., 40-60 m^3) of spent caustic etch containing elevated levels of aluminum (e.g., 100-150$^+$ g/L) was discharged to the treatment plant for neutralization and disposal. All

18

TABLE 1. CHARACTERISTICS OF NEUTRALIZATION BASIN EFFLUENT, CLARIFIER UNDERFLOW AND VACUUM-FILTER CAKE AT PLANT A DURING FILTRATION STUDIES

Date	Neutralization Basin (AC1) SS g/L	Clarifier Underflow (AC3)					Vacuum Filter C_k %
		pH	SS g/L	Specific Resistance			
				r Tm/kg	C_k %	CST s	
257(1)[a]	4.0	8.6	27	-	-	-	-
258(2)	6.3	8.0	30.7	-	-	20[b]	-
259(3)	-	-	-	-	-	-	12.6
262(5)	1.23	-	-	-	-	-	12.9
265(8)	2.49	8.6	39.5	-	-	-	10.7
269(12)	1.4	-	18.0	-	-	-	10.2
270(13)	-	-	-	-	-	-	12.2
271(14)	1.53	8.6	19.9	-	-	21[b]	-
272(15)	1.7	8.4	32.0	-	-	-	13.4
273(16)	-	-	-	-	-	-	12.3
276(19)	1.4	8.3	38.8	-	-	-	12.4
279(22)	-	-	-	-	-	-	7.4
280(23)	0.76	8.6	26.4	-	-	-	-
284(27)	-	7.7	37.4	-	-	33[b]	10.4
285(28)	4.2	-	53.2	-	-	-	9.9
290(33)	-	-	-	-	-	-	11.1
291(34)	5.12	-	-	-	-	-	-
292(35)	1.2	8.4	39.1	-	-	-	-
293(36)	-	-	-	-	-	-	15.2
294(37)	-	-	-	-	-	-	14.4
297(40)	1.41	-	-	-	-	-	-
298(41)	0.98	8.4	62.2	-	-	-	-
299(42)	-	-	-	-	-	-	14.0
300(43)	-	-	-	-	-	-	15.9
301(44)	-	8.4	56.7	6.0	13.4	237	-
302(45)	-	8.2	51.3	4.9	12.7	191	-
303(46)[c]	46.0	-	-	-	-	-	-
304(47)	-	-	91.5	1.6	19.8	182	18.8
305(48)	-	-	-	-	-	-	14.8
308(51)	-	8.2	72.8	3.7	15.4	196	-
309(52)	-	8.1	63.9	4.1	13.7	257	-
312(55)	2.1	-	-	-	-	-	-
315(58)	-	8.4	32.8	-	-	103	-
319(62)	-	8.1	20.7	4.5	12.2	63	13.6
321(64)	-	-	46.4	-	-	-	-
325(68)	-	-	17.1	2.5	11.1	50	-
326(69)	-	-	24.8	3.2	10.7	-	-
342(85)	-	8.2	39.1	3.2	12.6	110	-

(Continued)

19

TABLE 1 (Continued)

Date	Neutralization Basin (AC1) SS g/L	Clarifier Underflow (AC3)					Vacuum Filter C_k %
		pH	SS g/L	Specific Resistance			
				r Tm/kg	C_k %	CST s	
343(86)	-	8.2	38.9	3.3	12.2	139	-
350(93)	-	8.8	39.3	3.6	11.7	149	-
AVERAGE	2.4d	8.4e	40.8	3.7	13.2	f	12.7

a( ) = Elapsed time, days.
bCST values for 18-mm cylinder; others are for 10-mm cylinder.
cSample taken following batch dump of contaminated caustic-etch tank; for a concentrated sample of the suspension: SS = 104 g/L, r = 2 Tm/kg, C_k = 33.3%, and CST = 59 s.
dAverage value, exclusive of value for Day 303.
eMedian value.
fCST is a function of SS and was therefore not averaged.

TABLE 2. CHARACTERISTICS OF NEUTRALIZATION BASIN EFFLUENT
AT PLANT A PRIOR TO POLYMER CONDITIONING

Date	pH	SS g/L	Specific Resistance		CST s
			r Tm/kg	C_k %	
312	8.4	12.1	1.2	11.9	38
318	8.0	16.6	4.7	11.3	81

rinsewater flows were reduced to minimal values and the third stage of the neutralization basin was pumped to its minimum liquid volume prior to the batch dump of caustic etch. The concentrated waste was treated and neutralized on a semi-batch basis in the neutralization basin over a 12-h period. This neutralization of a concentrated waste increased the solids loading on the system and, as shown by segregated neutralization studies of spent caustic etch by Saunders et al. (1984), improved the overall thickening and dewatering properties of the resulting mixed suspension in the clarifier.

The majority of the pressure filtration studies were conducted with suspensions obtained from the clarifier underflow without alteration or adjustment of waste characteristics. These suspensions were designated as "AC3". On a limited number of occasions these suspensions were diluted to examine concentration effects; dilution water was obtained directly from the

recycle water system which was supplied by effluent from the clarifier. As indicated in Table 1, the pH of the suspensions throughout the study was between 7.7 and 8.8, with a median of 8.4. The suspended solids concentration of the underflow varied considerably throughout the study, ranging between 17.1 and 91.5 g/L. The batch etch dump on Day 303 and the resulting discharge of a large quantity of segregated-neutralization-like solids into the clarifier significantly increased the suspended solids concentration at the underflow. On the day following the dump (i.e., on Day 304), the suspended solids concentration was at its maximum value for the study, i.e., 91.5 g/L. These elevated values continued five and six days later (i.e., 72.8 g/L on Day 308 and 63.9 g/L on Day 309). After a twelve-day period the underflow concentrations returned to typical values, ranging from 17.1 to 46.4 g/L. Therefore, for evaluation of dewatering results using clarifier underflows, three evaluation periods were used: (i) prior to the etch dump on Day 303; (ii) immediately following the etch dump on Day 303 to Day 314; and (iii) the period following the etch dump on Days 315 to 350. Over the initial period, the suspended solids concentration of the underflow ranged from 18 to 62.2 g/L and averaged 38 g/L (14 values). Over the final period the suspended solids concentration ranged from 17.1 to 46.4 g/L and averaged 32.4 g/L (8 values). Within these limits, the initial and final periods appeared to be similar in terms of the performance of the clarifier. It is, however, impossible with the data available, given the highly variable operational procedures employed on the finishing line and wastewater treatment system at the plant, to establish this point conclusively. It is appropriate, however, to state that the initial and final periods were generally typical of the routine operation of the wastewater treatment system.

Pressure filtration studies were conducted over a 50-day period (i.e., Days 301-350). During this interval, grab samples of clarifier underflow were collected for filtration studies and were characterized using specific resistance and CST measurements. Specific resistance, which is theoretically independent of suspended solids concentration, varied from 1.6 to 6.0 Tm/kg (i.e., 1.6×10^{12} to 6.0×10^{12} m/kg) and averaged 3.7 Tm/kg (11 values), as indicated in Table 1. Specific resistance values did not appreciably change during the period immediately following the etch dump (e.g., Days 303-309).

As a part of the specific resistance analytical procedure, the solids content of the resulting dewatered cake, formed at a differential pressure of 0.5 bar (50 kPa) was determined. These values are also presented in Table 1. Using data prior to and following the etch dump, the specific-resistance cake solids ranged from 10.7 to 13.4 percent and averaged 12.1 percent (8 values). On the day following the etch dump (Day 304), the specific-resistance cake solids was at a high value of 19.8 percent, indicating a significant impact of the etch dump on sludge dewaterability. The values decreased to 15.4 and 13.7 percent over the following 5-day period, indicating the impact of continual removal of etch-dump solids from the clarifier and continual addition of conventional-neutralization solids to the clarifier. By the sixteenth day after the etch dump, the specific-resistance cake solids was back to a typical value of 12.2 percent. In addition to specific resistance, CST was monitored for each sludge

21

suspension examined. As indicated in Table 1, these values ranged from 50 to 257 seconds during the period of the filtration studies. However, since CST varies with suspended solids concentration and solids characteristics, it is difficult to use these data to identify changes in sludge characteristics.

Finally, a rotary vacuum filter was used at Plant A to dewater the clarifier underflow. Grab samples of dewatered sludge solids were collected from the unit and monitored for solids content. As presented in Table 1, the vacuum-filter cake solids ranged from 7.4 to 18.8 percent and averaged 12.7 percent (19 values). The average solids content of 12.7 percent is in general a low value for a dewatered sludge but was not atypical of values for aluminum-anodizing systems (Saunders et al., 1982). The vacuum-filter cake solids of 18.8 percent on Day 304 was the highest obtained throughout the study period and compared favorably with the specific-resistance cake solids of 19.8 percent on the same day.

Plant X Suspensions--
A 1.5-m^3 volume of clarifier underflow was collected on Day 313 and transported to Plant A for use in the filtration studies. The characteristics of samples of the sludge suspension used in the various pressure filtration runs are presented in Table 3. Examination of the data indicated that the parameters monitored in the various samples did not significantly deviate from average values over the 29-day period (Day 314-342). The average suspended solids concentration of 20.6 g/L was well below that for Plant A and the pH was slightly lower. The average specific resistance value was slightly lower than that for Plant A (i.e., 2.8 Tm/kg vs. 3.7 Tm/kg), while the specific-resistance cake solids value of 7.1 percent was drastically lower than the 13.2 percent average value for Plant A. Therefore, the clarifier suspension from Plant X was relatively dilute, compared to similar suspensions from Plant A, and had dewaterability characteristics similar to and slightly poorer than those from Plant A.

Segregated Neutralization Suspensions

To examine the impact of segregated-neutralization suspensions on pressure filtration of conventional-neutralization suspensions, segregated neutralization of caustic etch was conducted on a batch basis. Typically a 0.1-m^3 volume of caustic etch was obtained from the finishing line at Plant A and was placed in a 0.2-m^3 mixed reactor. Spent acid from the on-site storage container was then pumped into the reactor at the rate of approximately 1 L/min for a period of 1-2 h. The temperature of the reaction mixture ranged between 40-70°C. The neutralization reaction was typically terminated when the pH of the suspension was consistently below 10. The suspensions were then continuously mixed until they were to be used in pressure filtration runs that same day.

The characteristics of the four segregated-neutralization suspensions examined are presented in Table 4. The suspended solids concentration of each suspension was dictated by the aluminum content of each batch of spent etch and acid available, which was dictated by the operation of the finishing line. For undiluted suspensions, the suspended solids concentration

22

TABLE 3. CHARACTERISTICS OF CLARIFIER UNDERFLOW SUSPENSION (XC3)
FROM PLANT X AS USED DURING FILTRATION STUDIES

| Date[a] | pH | SS g/L | Specific Resistance | | CST s |
			r Tm/kg	c_k %	
314	7.5	19.5	2.8	7.3	60
314	7.9	20.5	2.9	6.6	75
314	7.9	20.6	3.3	7.1	70
320	7.6	21.0	2.5	-	54
320	7.6	20.2	2.3	7.0	64
321	7.7	19.7	2.4	7.2	68
321	7.7	21.5	3.1	7.5	74
341	7.2	21.1	2.7	7.1	60
342	-	21.7	-	-	-
AVERAGE	7.6[b]	20.6	2.8	7.1	66

[a]Sample collected on Day 313 and held in 0.2-m^3 containers at ambient
temperature for use over a 30-day period.
[b]Median value.

TABLE 4. CHARACTERISTICS OF SEGREGATED-NEUTRALIZATION SUSPENSIONS
PRODUCED FROM SPENT CAUSTIC ETCH AND ANODIZING ACID AT
PLANT A FOR FILTRATION STUDIES

| Date | pH | SS g/L | Specific Resistance Evaluation | | CST s |
			r Tm/kg	c_k %	
294	9.0	80.1	-	-	109[b]
297	10.1	137.3	-	-	84[b]
322[a]	8.8	173.2	1.5	49.5	581
	8.7	157.8	1.4	45.0	-
	8.8	155.1	1.9	47.0	345
	8.8	64.7	1.5	47.7	181
	8.8	58.5	1.4	41.9	-
	8.8	34.0	1.6	45.2	141
343[a]	10.6	180.1	1.3	43.8	485
	10.6	90.7	2.3	42.5	-
	10.6	41.2	1.1	41.5	-

[a]Replicates are analyses of diluted samples; clarified supernatant liquid
was used as dilution water.
[b]CST using 18-mm cylinder; others are with 10-mm cylinder.

ranged from 80.1 to 180.1 g/L, indicating the concentrated nature of these suspensions. The suspensions produced on Days 322 and 343 were used in filtration runs. As indicated by comparison with data in Tables 1-3; these suspensions had specific resistance values (1.1-2.3 Tm/kg) slightly lower than those for conventional neutralization sludges and had specific-resistance cake solids (41.5-49.5 percent) which were dramatically higher than those for conventional-neutralization sludges.

FIXED-VOLUME PRESSURE FILTRATION

Conventional Neutralization Suspensions

Plant A Clarifier Underflow (AC3)--
    High-Pressure Filtration--Numerous filtration runs were made with AC3 suspensions at Plant A. The characteristics of the suspensions are summarized by filtration run in Table 5.

    Runs 6-17 were made prior to the etch dump on Day 303. Suspended solids concentrations for underflow samples varied from 41.6 to 57.4 g/L (note: run 16, at a suspended solids concentration of 30.9 g/L, was conducted following dilution of the sample from run 15) and were near the highest experienced in the filtration studies, with the exception of those immediately following the etch-dump of Day 303. Runs 21-29 were conducted during this etch-dump period and were typically higher in suspended solids concentration than any of the conventional sludge suspensions employed. For the period following the etch dump, Runs 71 and 101 had low suspended solids concentrations (20.6 g/L and 17.3 g/L). while runs 142 and 171 had near-average concentrations of 38.4 and 37.9 g/L, respectively.

    The fixed suspended solids concentrations for all suspensions averaged 75 percent of the total suspended solids concentrations. Previous data (Saunders, et al., 1982; 1984) indicate this value is typical of these gelatinous suspensions throughout the industry. Furthermore, while the wastewater contained organic matter at measured TOC concentrations of 170-230 mg/L, the aluminum-hydroxide precipitates formed by conventional neutralization contain high levels of bound and mechanically-occluded water that is not removed by drying at 103-105°C (APHA, 1985). Therefore, the 25 percent mass loss of total suspended solids upon exposure to 550°C was attributable to bound and mechanically-occluded water, as well as volatile organic matter. Finally. the fixed suspended solids, based on previous data presented by Saunders et al. (1982, 1984), were indicative of dry aluminum hydroxides. For purposes of estimation, it can be assumed that 34.6 percent of the fixed suspended solids are equal to aluminum using the ratio $Al/[Al(OH)_3] = 0.346$ {i.e., $27/[27 + (17)(3)]$}. Therefore, estimated aluminum concentrations for the suspensions in Table 5 ranged from 5.3 to 24.2 g/L.

    During pressure filtration runs, the volume of filtrate produced witn time was monitored. These data are presented graphically in Figures 2-6. Filtrate was produced rapidly during the initial chamber-filling process.

TABLE 5. CHARACTERISTICS OF CLARIFIER UNDERFLOW SUSPENSIONS FOR
HIGH-PRESSURE, FIXED-VOLUME PRESSURE FILTRATION ON
NETZSCH PRESS

Run	Type	Date	Temp.	pH	Suspended Solids		Specific Resistance Evaluation		CST
					Total	Fixed	r	C_k	
			°C		g/L	g/L	Tm/kg	%	s
6	AC3	301	23	8.4	56.5	42.0	5.7	13.0	245
7	AC3	301	22	8.3	56.7	42.2	7.0	14.3	240
8	AC3	301	25	8.4	56.8	42.3	5.8	13.5	225
9	AC3	301	25	8.3	57.4	43.0	5.5	12.6	244
14	AC3	302	18	8.3	50.7	36.7	5.5	13.1	195
15[a]	AC3	302	21	8.3	44.5	34.5	4.9	11.9	138
16	AC3	302	22	8.3	30.9	22.8	4.0	12.3	84
17	AC3	302	22	8.4	41.6	31.2	3.6	12.5	141
21[b]	AC3	304	25	-	93.0	69.8	1.8	20.1	178
22	AC3	304	24	-	93.0	69.8	1.8	20.1	178
23	AC3	304	24	-	88.5	66.9	1.3	19.5	189
24	AC3	308	25	8.2	71.6	53.3	4.1	13.0	214
25	AC3	308	24	8.2	73.7	54.8	3.5	16.4	176
26	AC3	308	23	8.2	73.2	54.5	3.3	16.7	199
27[c]	AC3	308	23	-	39.1	29.1	3.1	14.8	95
28	AC3	308	21	-	36.1	28.3	2.6	14.7	90
29	AC3	308	21	-	36.8	28.7	2.2	14.6	100
71	AC3	319	16	8.1	20.6	15.3	6.0	11.8	59
101	AC3	325	17	7.8	17.3	-	2.3	11.5	53
142	AC3	342	16	8.2	38.4	-	3.2	12.6	110
171	AC3	343	13	8.2	37.9	28.2	3.6	11.7	139

[a]No filtrate-volume data were collected for run 15.
[b]Collected from clarifier underflow on day following etch dump.
[c]Runs 27-29 were conducted with a diluted sample of suspension
used with runs 24-26.

The rate of production of filtrate decreased with increasing time as the
fixed-volume chamber(s) filled with suspended solids and approached an
asymptotic value with extended time of filtration. Within common sets,
e.g., runs 6-9, 21-23, 24-26 and 27-29, the procedure was to make multiple
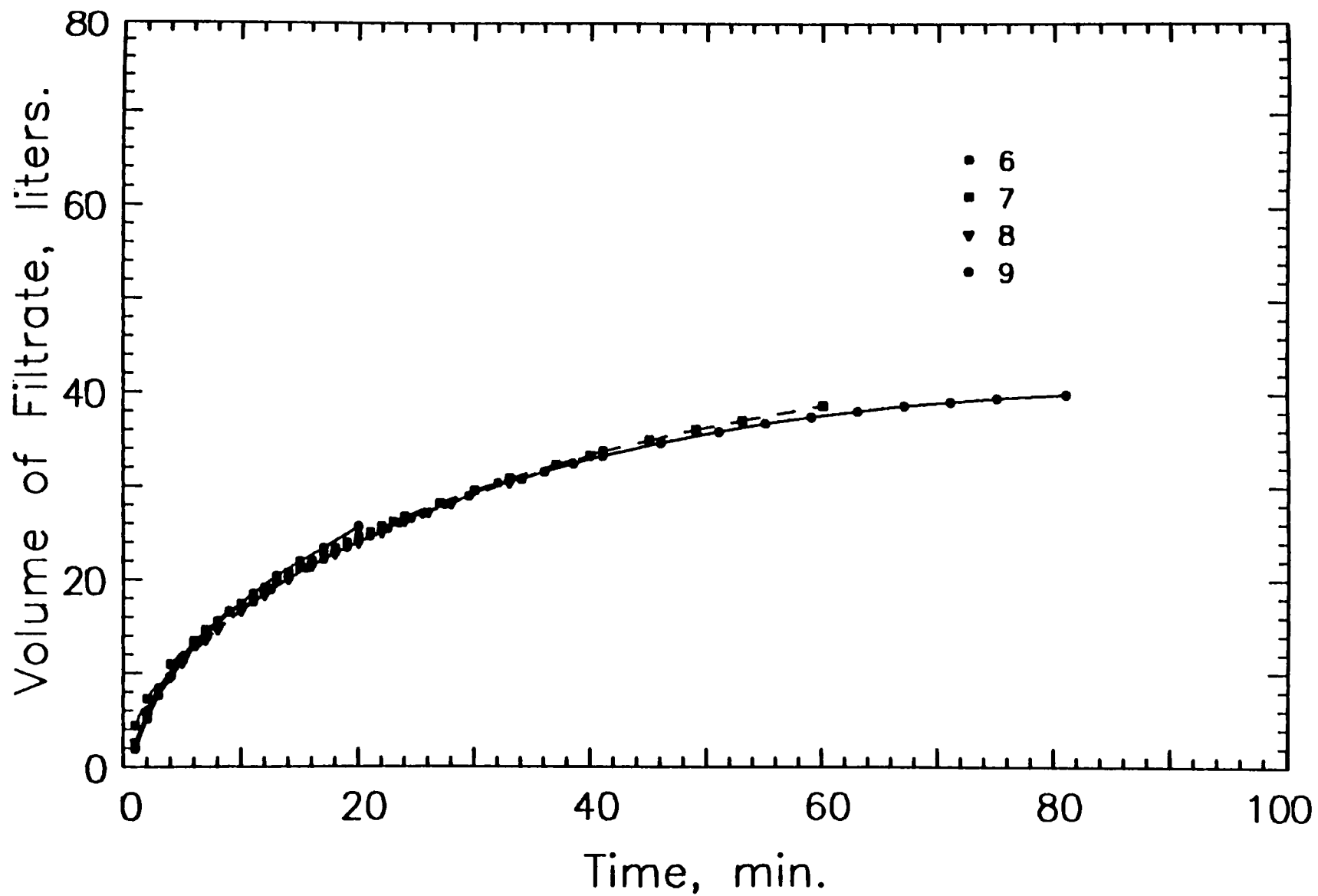
Figure 2. Cumulative filtrate volume for AC3 suspensions in runs 6-9 during high-pressure filtration.
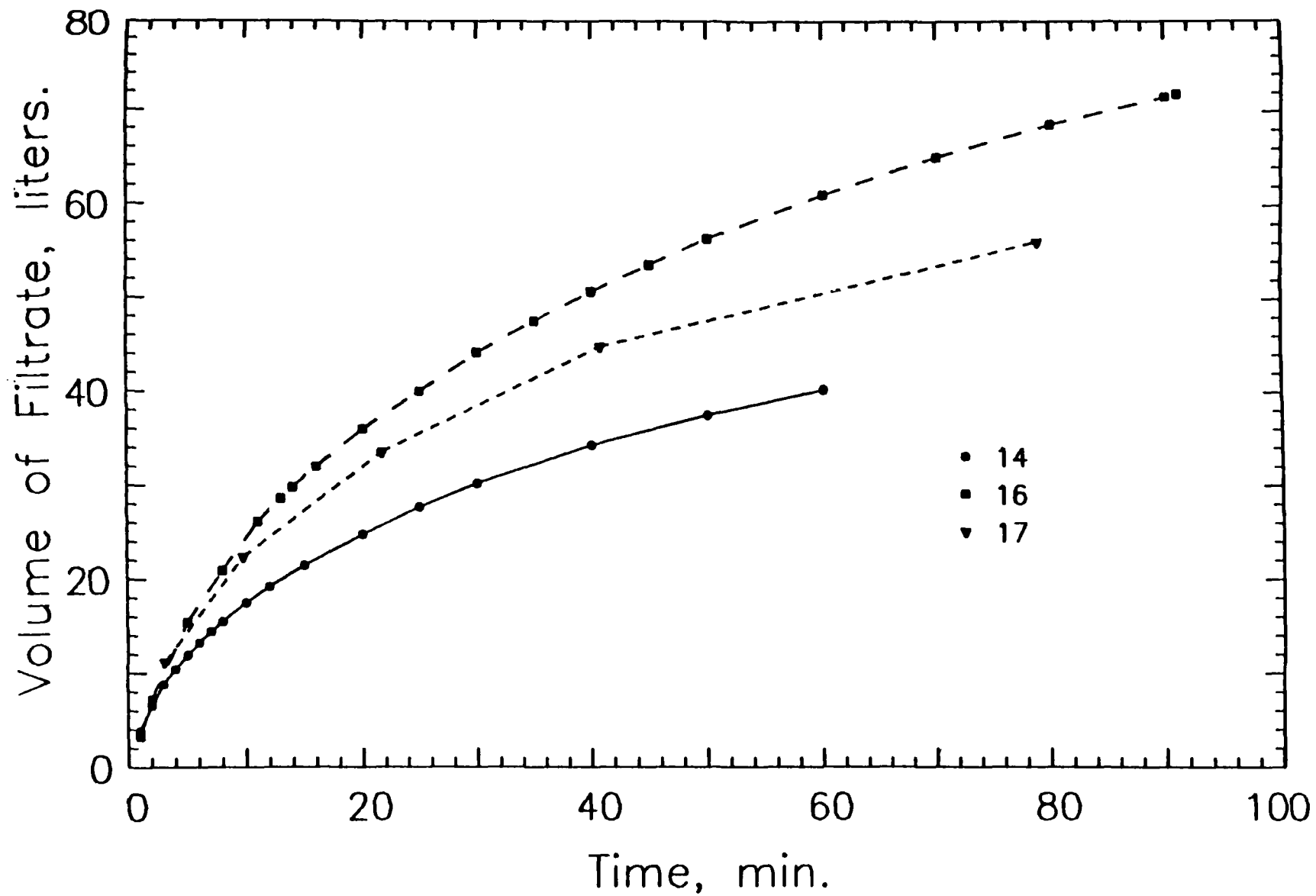
Figure 3. Cumulative filtrate volume for AC3 suspensions in runs 14, 16 and 17 during high-pressure filtration.

27
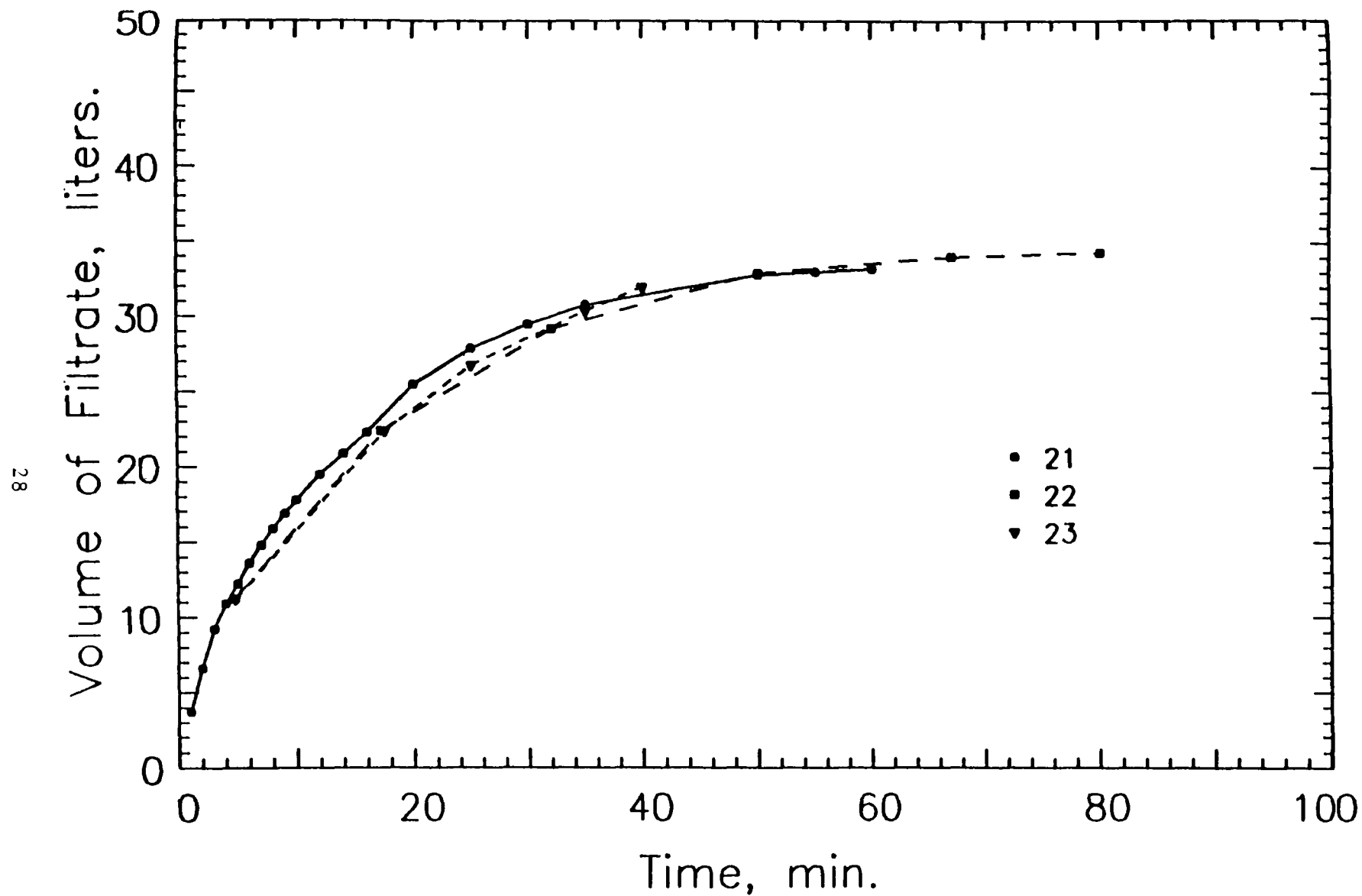
Figure 4. Cumulative filtrate volume for AC3 suspensions in runs 21-23 during high-pressure filtration.
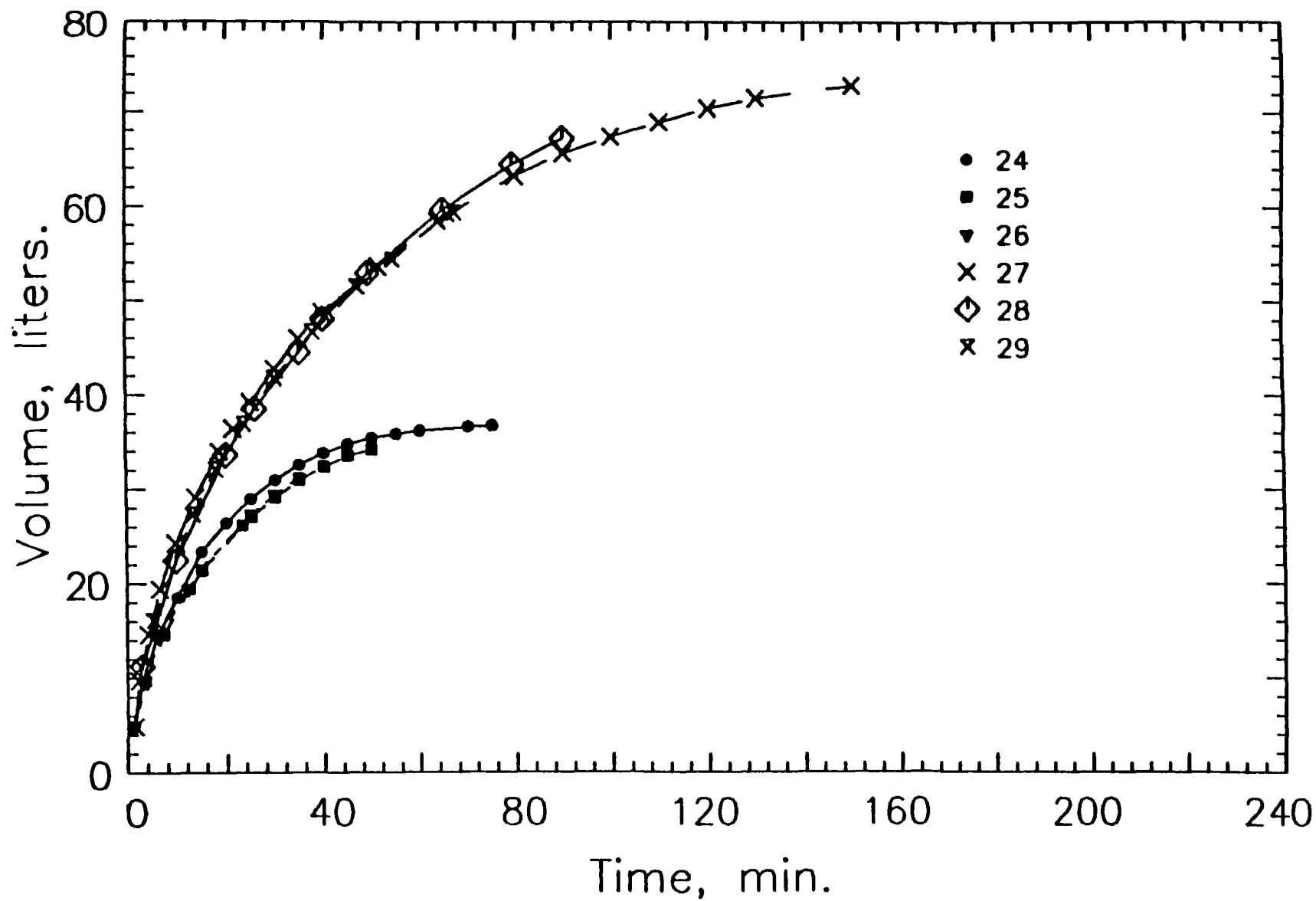
Figure 5.   Cumulative filtrate volume for undiluted (runs 24-26) and diluted (runs 27-29) AC3 suspensions during high-pressure filtration.
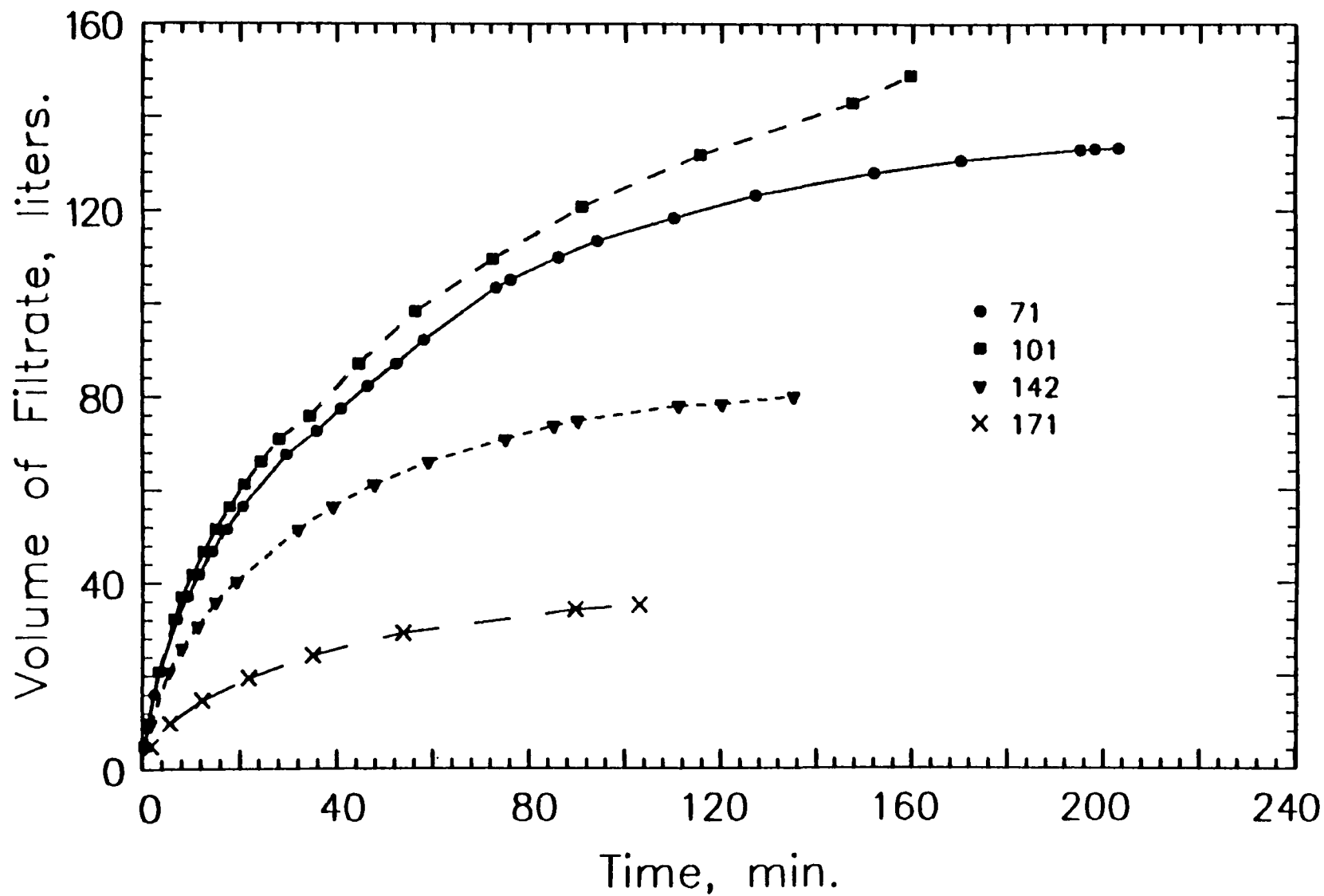
Figure 6. Cumulative filtrate volume for AC3 suspensions in runs 71, 101, 142 and 172 at high-pressure filtration.

filtration runs over various time intervals to be able to establish the effects of filtration time on filter performance. In these instances, it is shown in Figures 2, 4 and 5 that replicate runs closely coincided with previous runs. Data in Figures 3 and 7 were for numerous suspensions at various concentrations and did not overlap, as expected.

The data for each of the runs in Figures 2-6 are summarized in Table 6, including data for the dewatered cakes produced in each run. In this and all subsequent tables, the cakes produced were numbered 1 and 2, with 1 being the cake at the filter influent (i.e., face chamber) and cake 2 being the next one (i.e., the tail cake). In all runs made throughout the study, the maximum number of chambers was two, with only one chamber being used in some instances.

Inspection of the data in Table 6 indicated a slight increase in total mass of wet cakes and cake solids content with increasing time of filtration, although the correlation was very low, due, in part, to variations in initial sludge concentrations and solids characteristics. For runs prior to and well after the etch dump (i.e., runs 6-17 and 71-171), cake solids ranged from 21.6 to 28.8 percent, with fixed solids being equal to an average of 74 percent of total solids content.

For runs 21-29, clarifier underflow samples were affected by the addition of etch dump solids to the clarifier. Cake solids content ranged from 24.7 to 32.4 percent for undiluted suspensions (i.e., runs 21-26) and 23.6 to 28.9 percent for diluted suspensions (i.e., runs 27-29). It is obvious that the etch dump resulted in improved filter performance, with the improvement being attributable to increased suspended solids concentrations of the influent suspensions and improved dewaterability of the suspensions.

Low-Pressure Filtration--Low pressure filtration of AC3 suspensions was examined using the Netzsch and JWI filter presses. The characteristics of the suspensions studied are included in Table 7. The suspensions collected prior to the etch dump on Day 303 had relatively high suspended solids concentrations, while those for suspensions collected immediately following the etch dump (runs 31-34) were similarly high, although not as high as the 88.5-93.0 g/L values for runs 21-23 (see Table 5). Suspensions for runs 72, 102, J-21, J-61, J-81 and J-91 were collected after the etch dump of Day 303 and had suspended solids concentrations ranging from 16.9-39.4 g/L. These suspensions were generally typical of the clarifier underflow suspensions produced at Plant A, as indicated by comparison with data in Table 1.

The volume of filtrate collected during low-pressure filtration with the Netzsch and JWI presses are presented in Figures 7 to 10. Replicate sets in runs 10-13, 31-33 and 34-37 were virtually identical for similar sludge suspensions. Filtration data presented in these figures are summarized in Table 8. Prior to and following the etch-dump periods, the cake solids for dewatered cakes ranged from 13.6 to 24.7 percent. However, with the exception of runs conducted for short filtration times (i.e., $\leq$ 60 min), cake solids ranged from 20.1 to 24.7 percent and averaged 22.6 percent (14 values). Filtration of underflow samples collected during the period immediately following the etch dump resulted in production of cake solids of

TABLE 6. RESULTS FOR HIGH-PRESSURE, FIXED-VOLUME FILTRATION
OF CLARIFIER UNDERFLOW SUSPENSIONS (AC3)
ON NETZSCH PRESS

Run	Filtration Pressure bar	Time of Filtration min	Dewatered Cake(s)		
			Mass kg	Solids Content	
				Total %	Fixed %
6	14-15	83	1) 5.56	23	16.8
			2) 5.48	23.3	17.0
7	13-14.6	60	1) 5.39	22.6	16.5
			2) 5.45	20.9	15.3
8	13-14.6	40	1) 5.28	20.2	14.9
			2) 5.86	20.6	14.8
9	13-14.6	20	1) 5.05	15.5	11.5
			2) 5.14	16.1	11.8
14	13-14.6	60	1) 5.25	22.3	16.3
			2) 5.28	21.6	17.0
15	13-14.6	150	1) 5.48	23.4	17.6
			2) 5.53	23.7	17.5
16	13-14.6	91	1) 5.31	21.7	16.0
			2) 5.45	-	-
17	13-14.6	79	1) 5.51	22.2	16.2
			2) -	23.1	17.0
21[a]	13-14.6	60	1) 5.85	31.7	24.3
			2) 5.85	31.6	24.1
22	13-14.6	80	1) 5.9	-	-
			2) 5.9	-	-
23	13-14.6	40	1) 5.85	32.4	24.9
			2) 5.85	31.7	24.3
24	14-15	75	1) 5.71	28.6	21.9
			2) 5.71	27.6	20.6
25	14-15	50	1) 5.68	25.8	18.9
			2) 5.68	27.0	21.6
26	14-15	35	1) 5.68	24.7	18.3
			2) 5.56	25.8	20.2
27[b]	14-15	156	1) 5.73	28.8	20.9
			2) 5.68	28.9	22.5
28	14-15	90	1) 5.53	24.7	18.3
			2) 5.62	25.4	19.0
29	14-15	68	1) 5.45	23.6	17.5
			2) 5.53	23.6	17.5

(Continued)

32

Table 6 (Continued)

Run	Filtration Pressure	Time of Filtration	Dewatered Cake(s)		
			Mass	Solids Content	
				Total	Fixed
	bar	min	kg	%	%
71	14-15	203	1) 5.57	24.9	-
			2) 5.62	26.3	-
101	14-15	160	1) 5.61	24.9	-
			2) 5.66	25.5	-
142	14-15	135	1) 5.80	28.7	-
			2) 5.70	28.8	-
171[c]	14-15	103	5.62	26.3	18.8

[a]Collected from clarifier underflow on day following etch dump.
[b]Runs 27-29 were conducted with a diluted sample of suspension used for runs 24-26.
[c]Run conducted with only 1 chamber.

15.9 to 21.8 percent over very short operational periods, i.e., 18 to 75 min as opposed to 80 to 201 min for the runs with cake solids of 20.1 to 24.7 percent.

Plant A Neutralization Basin Effluent Suspensions (AC1)--
To examine the dewaterability of the neutralized suspension at the influent to the clarifier without the aid of polymer conditioning, samples were collected directly from the third stage of the neutralization basin for filtration studies.

High Pressure Filtration--As indicated in Table 9, three suspensions were examined by high-pressure filtration over a total of five runs. The suspension collected on Day 303 was that produced during the period of neutralization of the caustic etch dump. The suspension was furthermore concentrated by a factor of 2 by gravity sedimentation and was the thickest suspension examined during the study, with a suspended solids concentration of 101.1 - 109.4 g/L. The specific resistance values (i.e., 0.17 - 0.25 Tm/kg) were approximately an order of magnitude below those for all other suspensions and the specific-resistance cake solids were exceptionally high (32.9 - 33.8 percent), indicating excellent dewaterability, even without the aid of polymer conditioning.

TABLE 7. CHARACTERISTICS OF CLARIFIER UNDERFLOW SUSPENSIONS (AC3)
FOR LOW-PRESSURE, FIXED-VOLUME, PRESSURE FILTRATION ON
NETZSCH AND JWI PRESSES

Run	Type	Date	Temp. °C	pH	Suspended Solids		Specific Resistance Evaluation		CST
					Total g/L	Fixed g/L	r Tm/kg	C_k %	s
NETZSCH PRESS									
10	AC3	301	-	8.3	57.4	43.0	5.5	12.6	244
11	AC3	301	-	8.3	57.4	43.0	5.5	12.6	244
12	AC3	302	18	8.2	51.9	38.6	3.8	12.7	187
13	AC3	302	18	8.2	50.7	36.7	5.5	13.1	195
31[a]	AC3	309	24	8.1	64.5	50.1	3.7	12.6	251
32	AC3	309	24	8.1	64.5	50.1	3.7	12.6	251
33	AC3	309	22	8.1	63.2	48.8	4.5	14.7	263
34	AC3	309	22	8.1	63.2	48.8	4.5	14.7	263
35[b]	AC3	309	20	8.2	31.8	24.5	3.3	14.7	104
36	AC3	309	20	8.2	33.3	25.7	3.4	12.7	103
37	AC3	309	21	8.3	32.3	25.5	2.8	12.9	92
72	AC3	319	16	8.1	20.8	15.5	3.0	12.5	66
102	AC3	325	17	7.8	16.9	-	2.7	10.7	46
JWI PRESS									
J-21	AC3	315	-	8.4	32.8	25.1	-	-	103
J-61(141-2)[c]	AC3	342	15	8.2	39.4	29.3	3.2	12.6	103
J-81(171-2)	AC3	343	13	8.2	37.9	28.2	3.6	11.7	139
J-91(181-2)	ACE	350	16	8.8	39.3	29.2	4.2	12.8	149

[a]Suspension collected from clarifier underflow six days following
etch dump.
[b]Runs 35-37 were conducted with a diluted sample of suspension used
with runs 31-34.
[c]( ) = runs with Netzsch press which were conducted with identical
suspension.

34

Figure 7. Cumulative filtrate volume for AC3 suspension in runs 10-13 during low-pressure filtration.
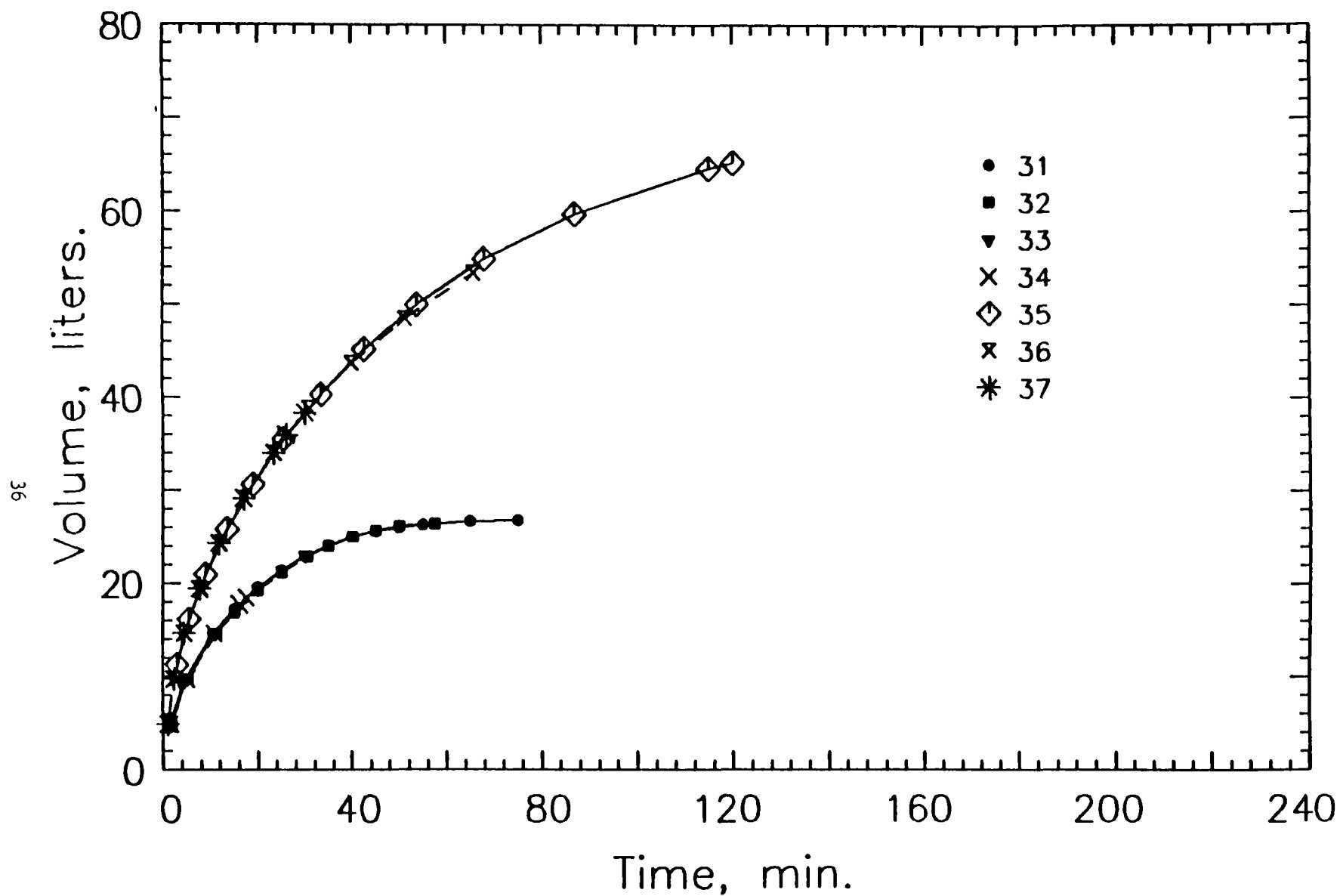
Figure 8.   Cumulative filtrate volume for undiluted (runs 31-33) and diluted (runs 34-37)
AC3 suspensions during low-pressure filtration.

Figure 9. Cumulative filtrate volume for AC3 suspensions in runs 72 and 102 at low-pressure filtration.
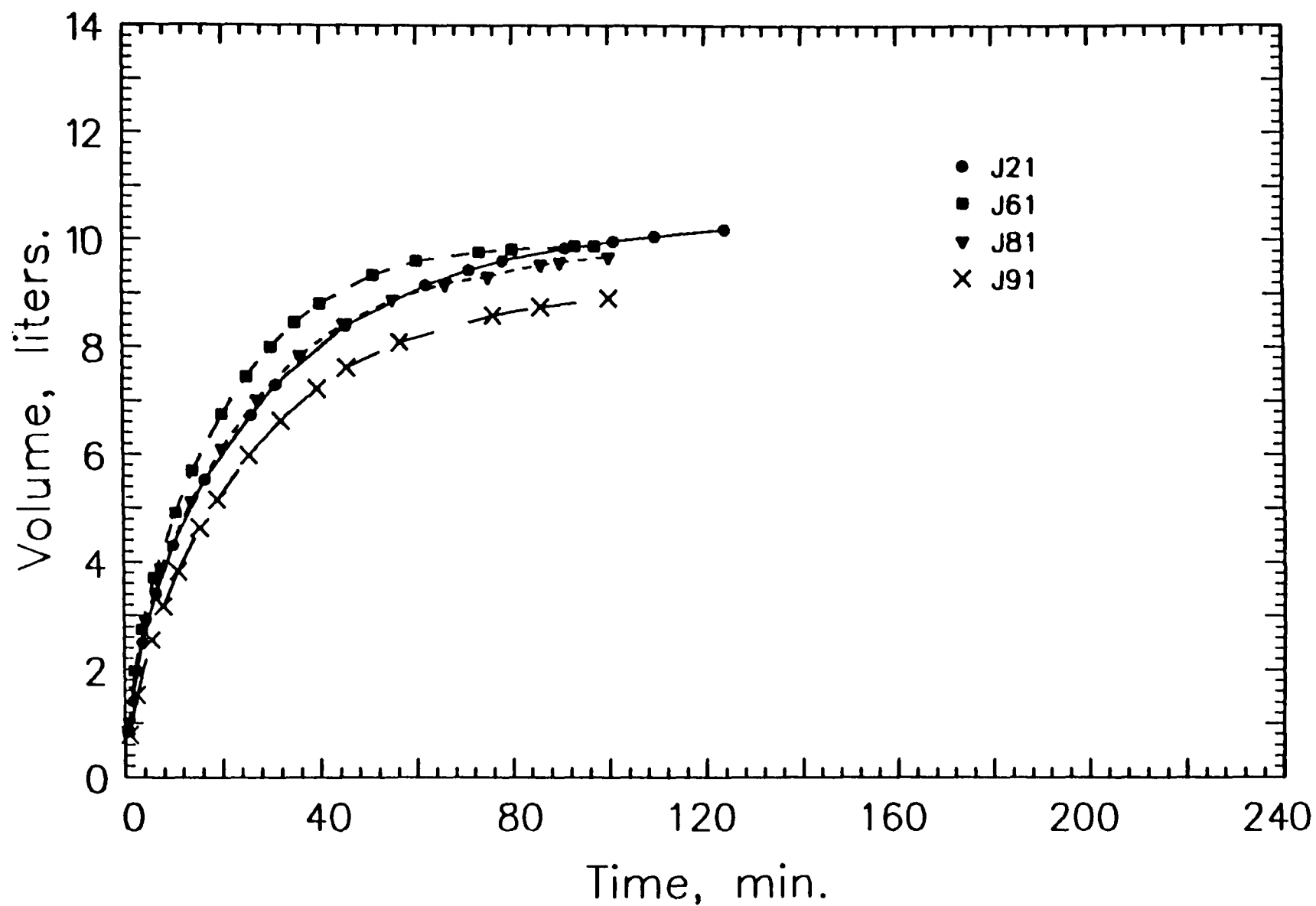
Figure 10. Cumulative filtrate volume for AC3 suspensions in runs J-21, J-61, J-81 and J-91 during low-pressure filtration.

TABLE 8. RESULTS FOR LOW-PRESSURE, FIXED-VOLUME FILTRATION
OF CLARIFIER UNDERFLOW SUSPENSIONS (AC3)
ON NETZSCH AND JWI PRESSES

Run	Filtration Pressure	Time of Filtration	Dewatered Cake(s)		
			Mass	Solids Content	
				Total	Fixed
	bar	min	kg	%	%

NETZSCH PRESS

Run	Filtration Pressure (bar)	Time of Filtration (min)	Mass (kg)	Total (%)	Fixed (%)
10	6-7	80	1) 5.31	20.1	14.6
			2) 5.34	20.1	14.7
11	6-7	40	1) -	17.3	13.0
			2) 5.28	18.7	13.0
12	6-7	60	1) 5.25	19.3	14.1
			2) 5.34	19.5	14.1
13	6-7	20	1) 4.88	-	-
			2) 4.82	13.6	10.6
31[a]	6.5-7.5	75	1) 5.34	21.5	15.4
			2) 5.34	21.8	15.6
32	6-7	58	1) 5.34	21.1	15.9
			2) 5.39	21.1	14.9
33	6-7	30	1) 5.28	19.8	15.1
			2) 5.39	19.7	14.6
34	6-7	18	1) -	16.1	12.4
			2) 5.22	15.9	12.2
35[b]	6-7	120	1) 5.34	22.4	16.0
			2) 5.42	21.3	16.4
36	6-7	66	1) 5.31	18.0	12.9
			2) 5.22	19.2	14.5
37	6-7	30	1) -	15.3	11.0
			2) 5.02	14.8	10.7
72	6-7	201	1) 5.42	22.4	-
			2) 5.42	22.8	-
102	6-7	160	1) 5.44	24.7	-
			2) 5.43	22.3	-

JWI PRESS

Run	Filtration Pressure (bar)	Time of Filtration (min)	Mass (kg)	Total (%)	Fixed (%)
J-21(71-2)	6-7	124	1) 0.915	21.3	-
			2) 0.945	21.7	-
J-61(141-2)	6-7	97	1) 0.955	23.4	-
			2) 0.960	23.1	-

(Continued)

39

TABLE 8 (Continued)

Run	Filtration Pressure	Time of Filtration	Dewatered Cake(s)		
			Mass	Solids Content	
				Total	Fixed
	bar	min	kg	%	%
J-81(171-2)	6-7	100	1) 0.965	23.8	16.7
			2) 0.970	23.6	16.7
J-91(181-2)	6-7	97	1) 0.985	24.1	-
			2) 0.985	23.1	-

aCollected from clarifier underflow six days following etch dump.
bRuns 35-37 were conducted with a diluted sample of suspension used
for runs 31-34.

TABLE 9. CHARACTERISTICS OF NEUTRALIZATION BASIN EFFLUENT SUSPENSIONS
(AC1) FOR HIGH-PRESSURE FIXED-VOLUME FILTRATION ON NETZSCH
PRESS

Run	Type	Date	Temp.	pH	Suspended Solids		Specific Resistance Evaluation		CST
			°C		Total	Fixed	r	C_k	
					g/L	g/L	Tm/kg	%	s
18*	AC1	303	21	-	101.4	-	0.19	33.2	64
19*	AC1	303	21	-	101.2	-	0.25	32.9	63
20*	AC1	303	20	-	109.4	-	0.17	33.8	51
41	AC1	312	19	8.4	12.1	8.9	1.2	11.9	38
62	AC1	318	14	8.0	16.7	-	2.1	11.1	56

*Suspension for runs 18-20 was collected immediately after etch dump and
concentrated by a factor of approximately 2; no polymer conditioner was
added.

The two remaining suspensions examined were conventional sludges and,
even with extensive gravity thickening on a batch basis, were low in
suspended solids concentration. The specific resistance values were not
significantly different from those for AC3 suspensions, although
specific-resistance cake solids were relatively low at 11.1 - 11.9 percent.

The volumes of filtrate collected during high-pressure filtration are

presented in Figures 11-13. Replicate runs 18, 19 and 20 were virtually identical. In addition, the rate of production of filtrate after the initial 10-min period decreased drastically. This was significantly different from the responses demonstrated during other runs (e.g., see Figures 2-6) of even longer duration.

As indicated by data in Figure 12, run 41 was terminated very early in its filtration cycle due to the lack of sufficient volume of influent suspension. Data for run 62 are presented in Figure 13 in conjunction with that for run 61, a low-pressure run.

Data in Table 10 for runs 18-20 confirmed the excellent dewaterability

TABLE 10. RESULTS FOR HIGH-PRESSURE, FIXED-VOLUME FILTRATION OF NEUTRALIZATION BASIN EFFLUENT SUSPENSIONS (AC1) ON NETZSCH PRESS

Run	Filtration Pressure	Time of Filtration	Dewatered Cake(s)		
			Mass	Solids Content	
				Total	Fixed
	bar	min	kg	%	%
18	13-14.6	40	1) 6.58	42.8	34.0
			2) 6.30	41.6	32.3
19	13-14.6	15	1) 6.44	41.6	32.4
			2) 6.38	44.7	32.1
20	13-14.6	27	1) 6.58	42.5	32.9
			2) 6.23	41.8	32.1
41	14-15	54	1) 5.13	18.1	13.6
			2) 5.32	17.5	13.2
62*	14-15	180	5.34	19.8	14.5

*Only 1 chamber was used for run 62.

of the suspensions produced during the batch etch dump. In time periods of 15-40 min, dewatered cake solids of 41.6 - 44.7 percent were achieved. The dewatered cake solids for runs 41 and 62 were low (17.5 to 19.8 percent), indicating the dramatic difference between conventional- and segregated-neutralization suspensions.

Low-Pressure Filtration -- Three AC1 suspensions were examined during four filtration runs. The characteristics of the suspensions are summarized in Table 11 and were similar to those for the suspensions collected at times other than during the etch dump period. The suspension for run J-32, which was the same one used for run 61 on the Netzsch press, was polymer conditioned on a batch basis prior to the filtration run to examine the impact of conditioning on dewatering. While the suspended solids concentration
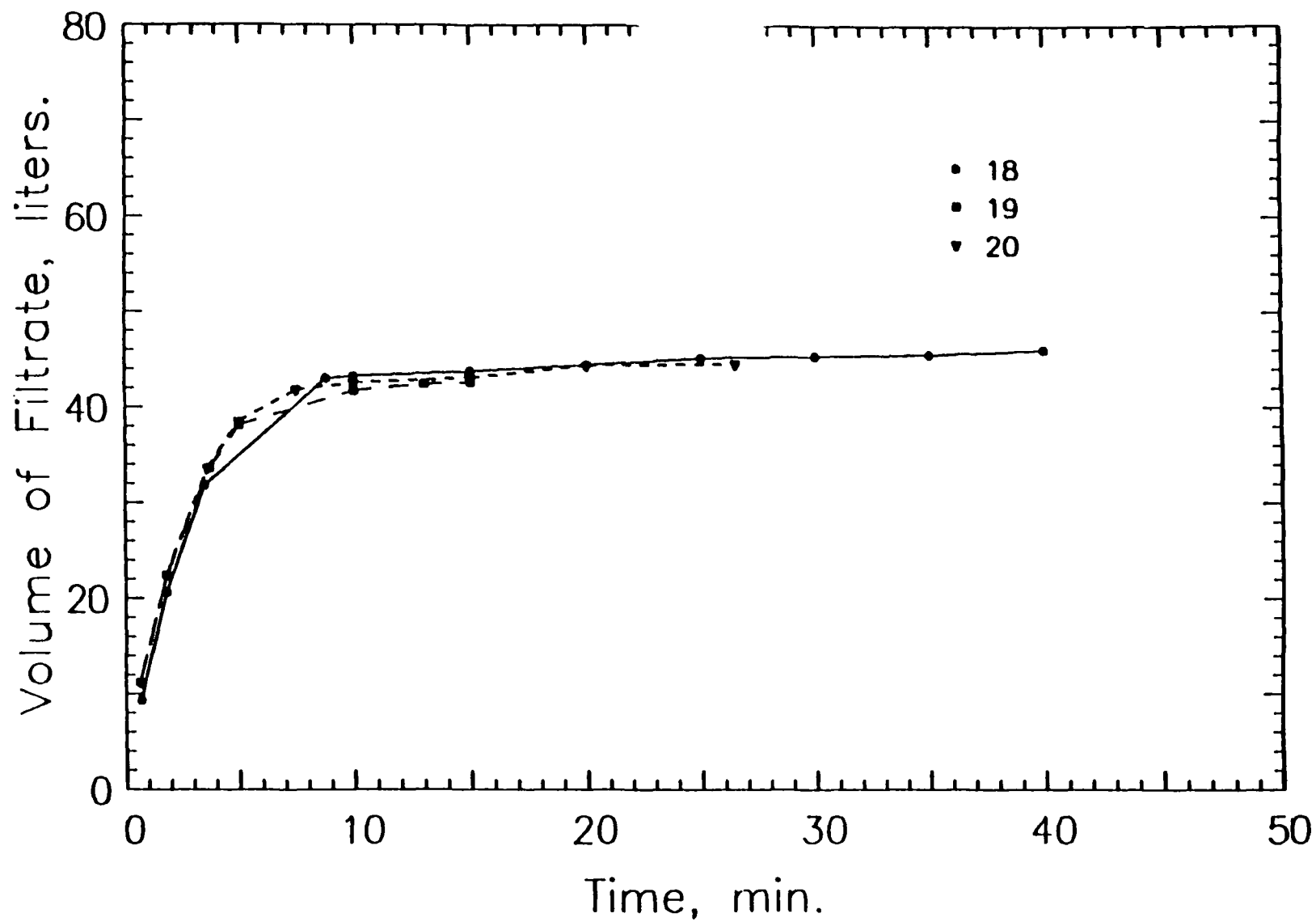
Figure 11. Cumulative filtrate volume for ACl suspensions in runs 18-20 during high-pressure filtration.

42

Figure 12.   Cumulative filtrate volume for an ACl suspension in run 41 during high-pressure filtration.
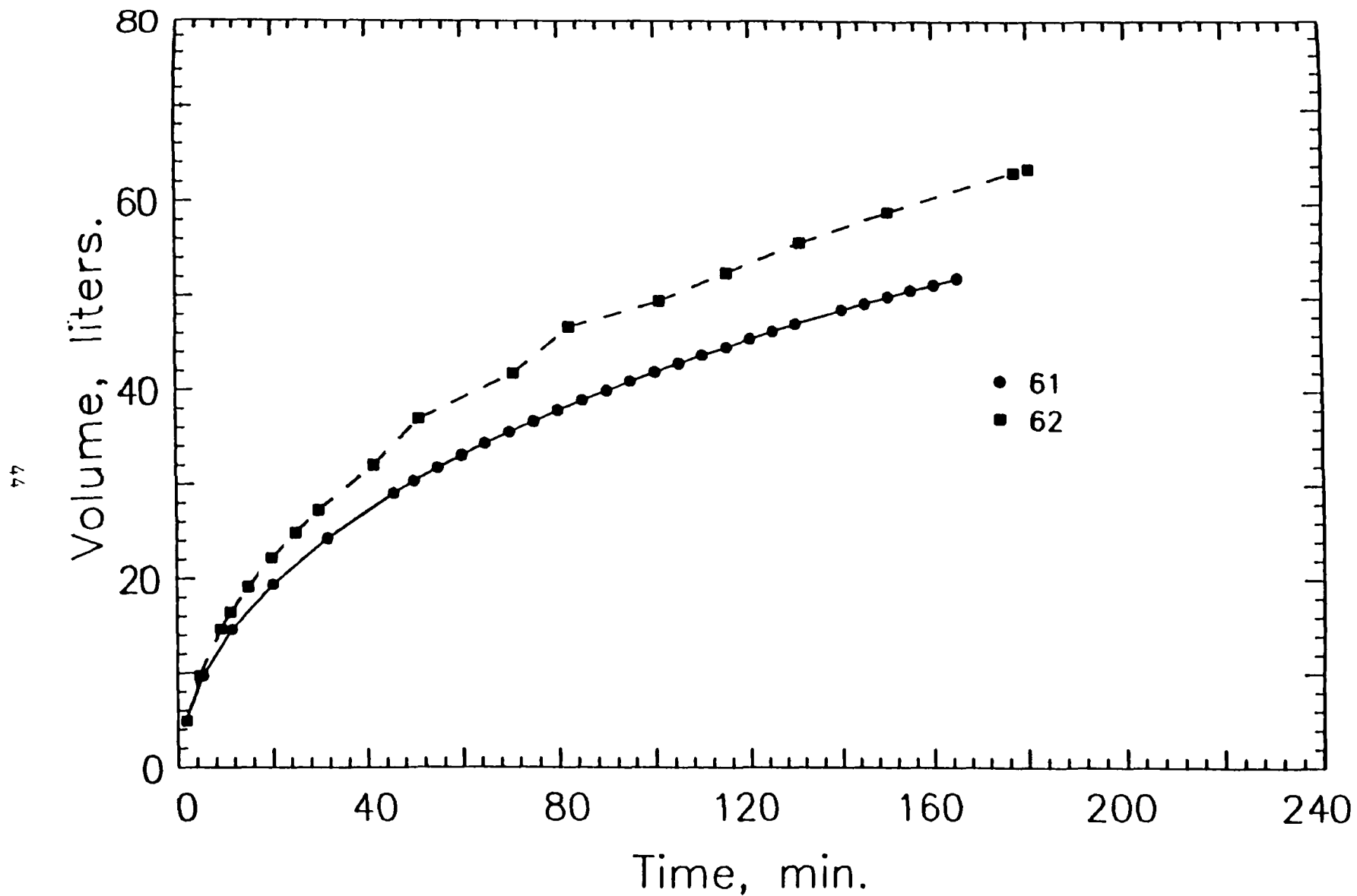
Figure 13. Cumulative filtrate volume for ACl suspensions during high-pressure (run 62) and low-pressure (run 61) filtration.

TABLE 11. CHARACTERISTICS OF NEUTRALIZATION BASIN EFFLUENT SUSPENSIONS (AC1) FOR LOW-PRESSURE FIXED-VOLUME FILTRATION ON NETZSCH AND JWI PRESSES

Run	Type	Date	Temp. °C	pH	Suspended Solids		Specific Resistance Evaluation		CST
					Total g/L	Fixed g/L	r Tm/kg	C_k %	s
NETZSCH PRESS									
61	AC1	318	13	8.0	16.5	-	4.8	-	70
JWI PRESS									
J-31(61)	AC1	318	11	8.2	17.1	12.7	4.7	11.9	66
J-32(61)[a]	AC1	328	11	8.1	40.1	-	4.6	10.9	108
J-71(61)	AC1	303/(342)[b]	18	9.1	82.3	61.7	-	-	-

[a]Suspension was polymer conditioned with 2 percent (by volume) of polymer solution used at plant during a 30-s rapid mix, followed by 150-s slow mix, gravity settling and decantation of clarified water.

[b]Notation (303/(342)) indicates sample was collected on Day 303 and was dewatered on Day 342.

was dramatically increased as a result of polymer conditioning and gravity settling, the specific resistance of the suspension was not altered significantly but the specific-resistance cake solids decreased from 11.9 percent to 10.9 percent. This in general was an expected and predictable response for polymer conditioning.

The AC1 suspension for run J-71 was similar to that for run 161 and was collected during the etch dump on Day 303, but was not examined by low-pressure filtration until Day 342. The suspended solids concentration was exceptionally high at 82.3 g/L and well above the average value of 2.4 g/L for AC1 suspensions, as presented in Table 1.

Filtrate volume data for low pressure runs are presented in Figures 13-15. The rate of filtrate production for run 61 was lower than that for high-pressure run 62, as indicated in Figure 13. The combined effects of (i) polymer conditioning and (ii) concentration by gravity settling are apparent in Figure 14. Although run J-32 was terminated prematurely, it is obvious that the rate of filtration for the thicker, polymer-conditioned suspension was much higher, indicating a dramatic improvement in sludge dewaterability. The filtration response for run J-71 (Figure 15) was excellent initially, as indicated by a rapid filtration rate, followed very quickly by an abrupt decrease in rate as the press was filled with solids.
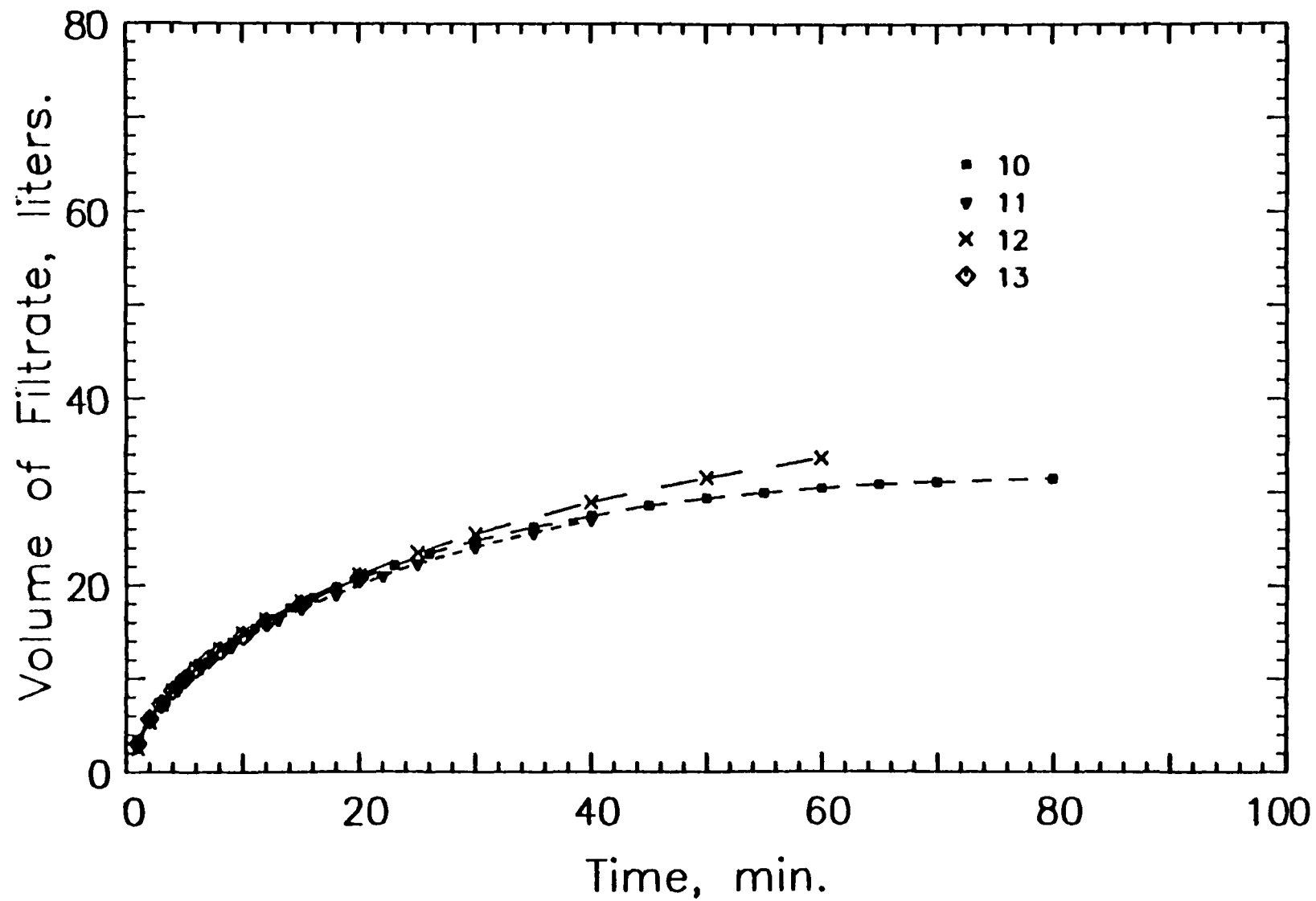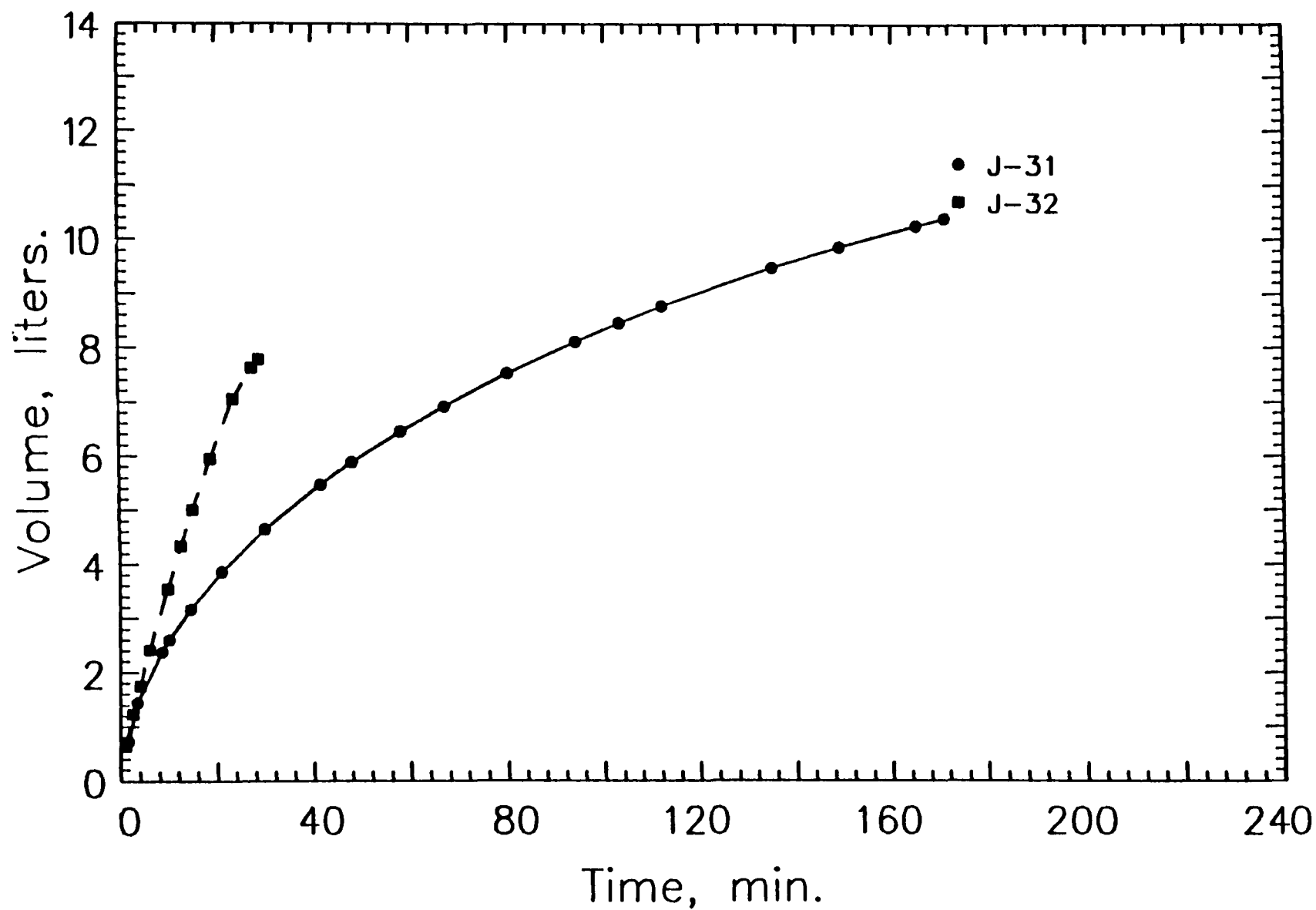
Figure 14. Cumulative filtrate volume for ACl suspensions In runs J-31 and J-32 during low-pressure filtration.
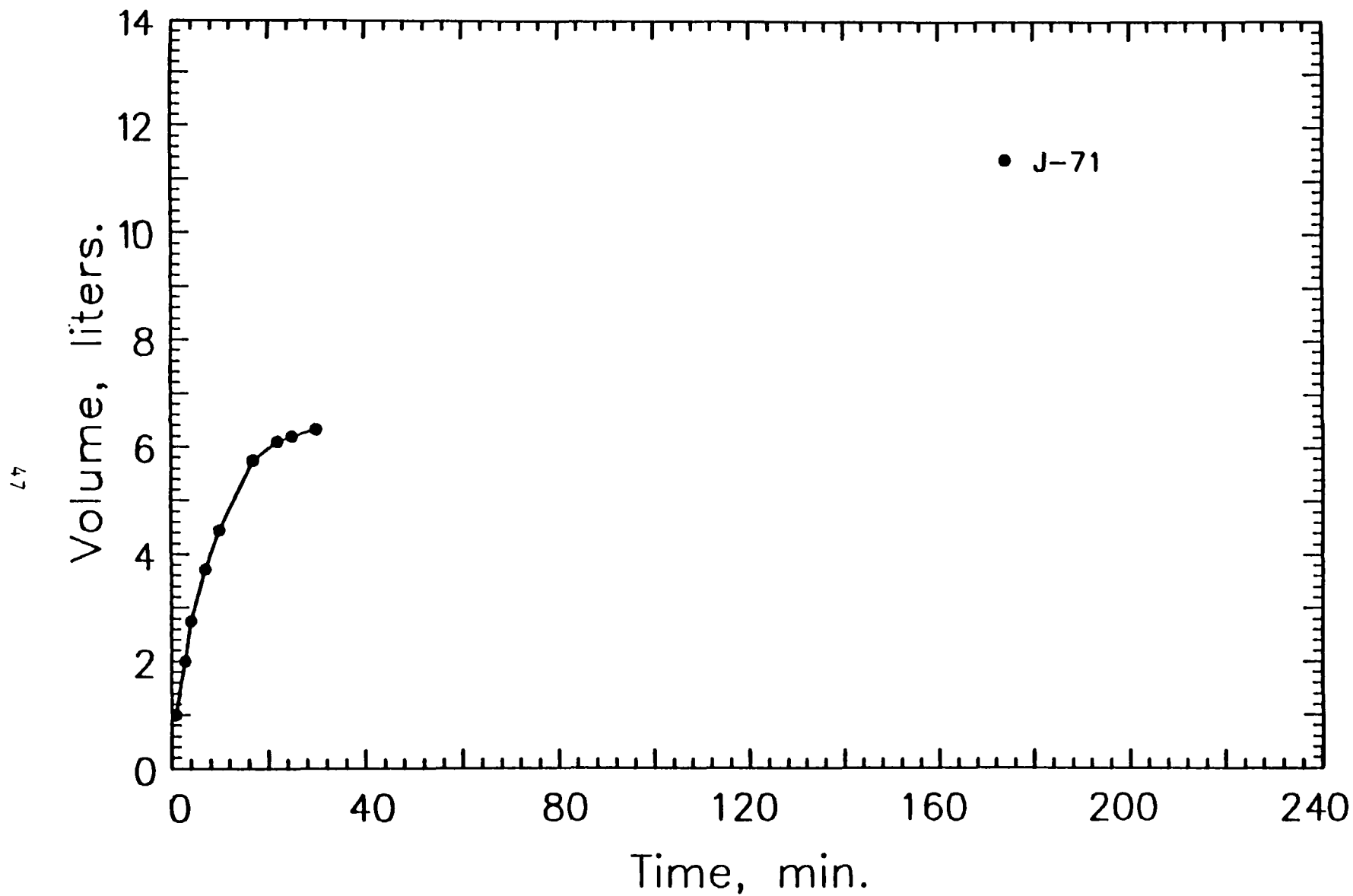
Figure 15. Cumulative filtrate volume for ACl suspension in run J-71 during low-pressure filtration.

Data in Table 12 for runs 61 and J-31 indicate that the two presses produced similar dewatered cake solids contents when operated at similar intervals and pressures. Comparison of data for runs J-31 and J-32 confirm, in part, previous conclusions from Figure 14. The polymer conditioned sludge was dewatered to 14.5 percent solids in 29 min while the unconditioned suspension required 171 min to achieve 18.1 percent solids. Run J-71 was conducted with an etch-dump suspension which was dewatered to an average cake-solids content of 46.2 percent in a 30 min period, indicating the dramatic improvements of an etch-dump on suspension dewatering characteristics.

TABLE 12. RESULTS FOR LOW-PRESSURE, FIXED VOLUME FILTRATION OF NEUTRALIZATION BASIN EFFLUENT SUSPENSIONS (AC1) ON NETZSCH AND JWI PRESSES

Run	Filtration Pressure	Time of Filtration	Dewatered Cake(s)		
			Mass	Solids Content	
				Total	Fixed
	bar	min	kg	%	%
NETZSCH PRESS					
61	6-7	165	5.15	17.3	12.9
JWI PRESS					
J-31(61)	6-7	171	0.95	18.1	13.2
J-32(61)	6-7	29*	0.895	14.5	10.2
J-71(161)	6-7	30	1) 1.16	47.1	-
			2) 1.15	45.3	-

*Run terminated prematurely due to lack of sufficient volume of the influent sludge suspension.

Plant X Clarifier Underflow (XC3)--
     A single sample of a clarifier underflow suspension from Plant X was examined over a 29-day period (Day 314-342; Table 3). The characteristics of suspension aliquots and results of pressure filtration studies are presented below.

     High-Pressure Filtration -- The characteristics of the suspensions examined by high-pressure filtration are presented in Table 13. No major differences in suspension characteristics are apparent, with the small exception of a decrease in suspension pH with increased storage time. This variation was, however, not indicative of a well-defined trend, as indicated by data in Table 3.

48

TABLE 13. CHARACTERISTICS OF CLARIFIER UNDERFLOW SUSPENSIONS (XC3) FOR HIGH-PRESSURE, FIXED-VOLUME FILTRATION ON NETZSCH PRESS

| Run | Type | Date | Temp. °C | pH | Suspended Solids | | Specific Resistance Evaluation | | CST |
					Total g/L	Fixed g/L	r Tm/kg	C_k %	s
51	XC3	314	16.5	7.9	20.5	16.4	2.9	6.6	75
81	XC3	314/321	11	7.7	19.7	15.6	2.4	7.16	68
82	XC3	314/321	12	7.7	21.5	17.1	3.1	7.5	74
121	XC3	314/341	14	7.2	21.1	16.8	2.7	7.1	60

The volumes of filtrate collected during high-pressure filtration are presented in Figures 16-18. Data for replicate runs 81 and 82 were virtually identical and all four runs were carried well into the declining filtration phase. Performance data for the four runs are presented in Table 14. Cake solids contents of 17.7 to 20.3 percent were achieved for runs with elapsed filtration times of 170 to 225 min.

TABLE 14. RESULTS FOR HIGH-PRESSURE, FIXED-VOLUME FILTRATION OF CLARIFIER UNDERFLOW SUSPENSIONS (XC3) ON NETZSCH PRESS

Run	Filtration Pressure	Time of Filtration	Dewatered Cake(s)		
			Mass	Solids Content	
				Total	Fixed
	bar	min	kg	%	%
51	14-15	225	1) 5.36	20.3	15.4
			2) 5.44	19.6	14.9
81	14-15	170	1) 5.28	17.7	-
			2) 5.33	-	-
82	14-15	90	1) 5.24	16.2	-
			2) 5.25	15.8	-
121	14-15	170	1) 5.20	18.8	-
			2) 5.30	18.5	-

Low-Pressure Filtration -- Characteristics of the XC3 suspensions used during low-pressure runs are presented in Table 15 and are consistent with those presented in Tables 3 and 13. Cumulative filtrate volumes for the
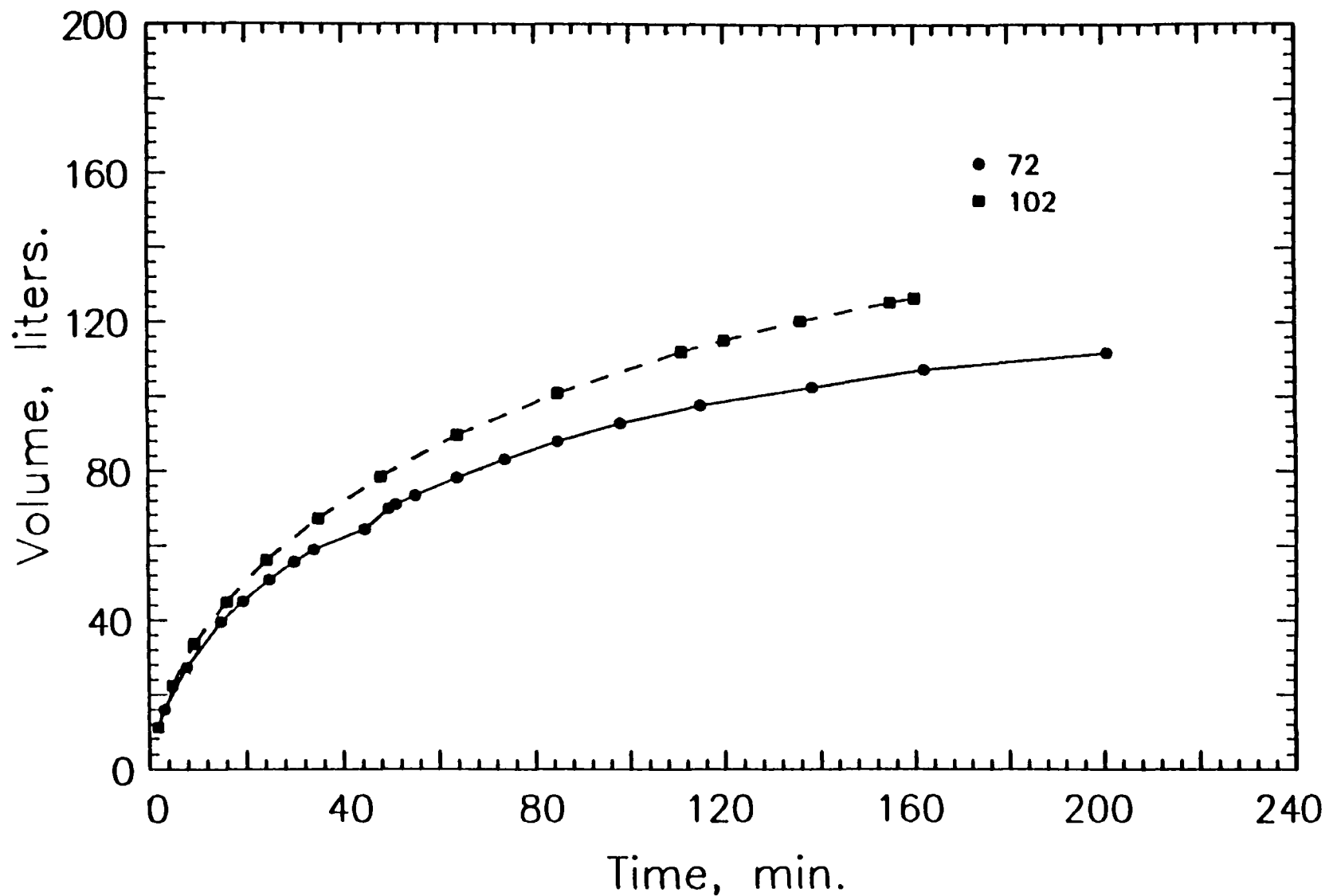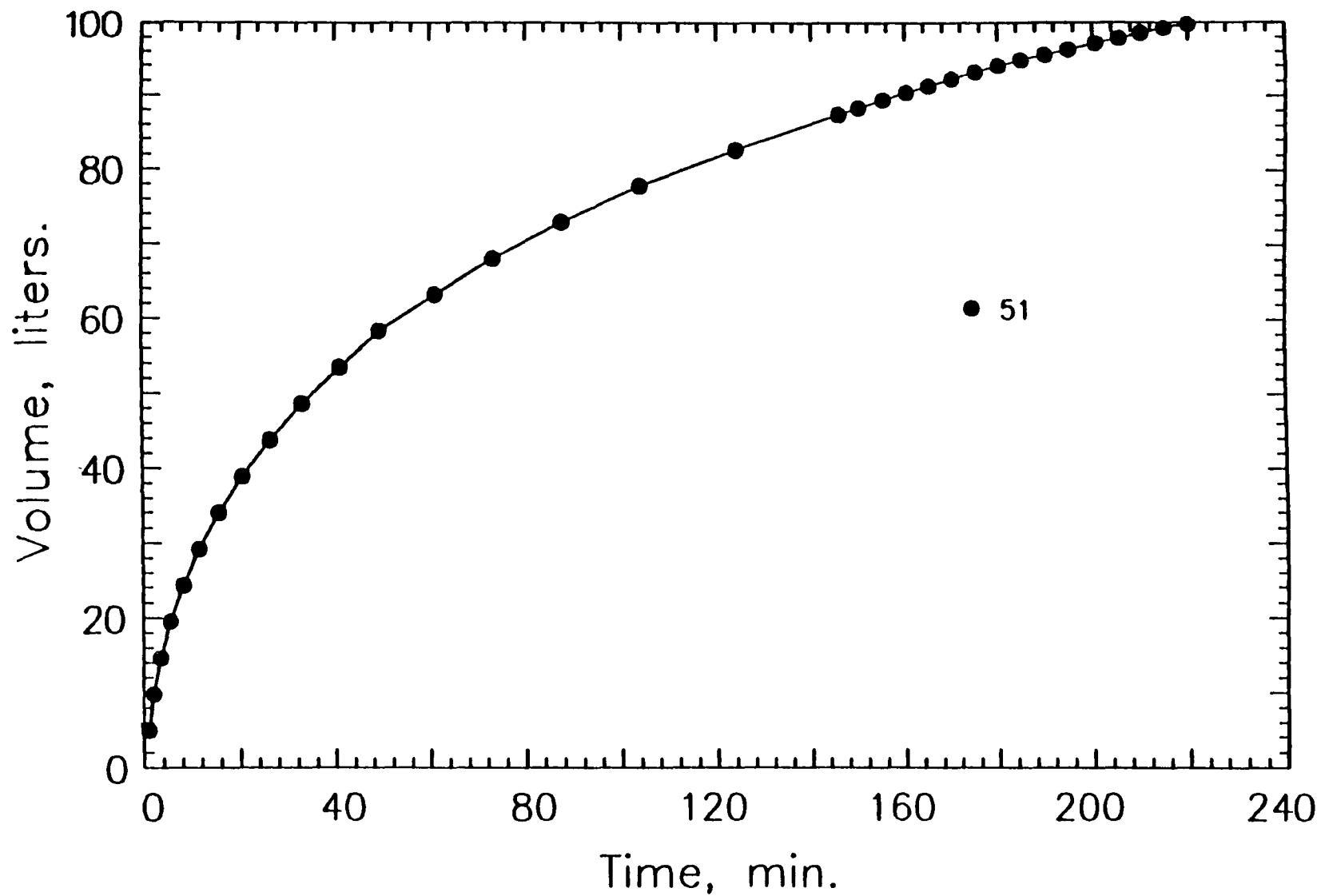
Figure 16. Cumulative filtrate volume for XC3 suspension in run 51 during high-pressure filtration.
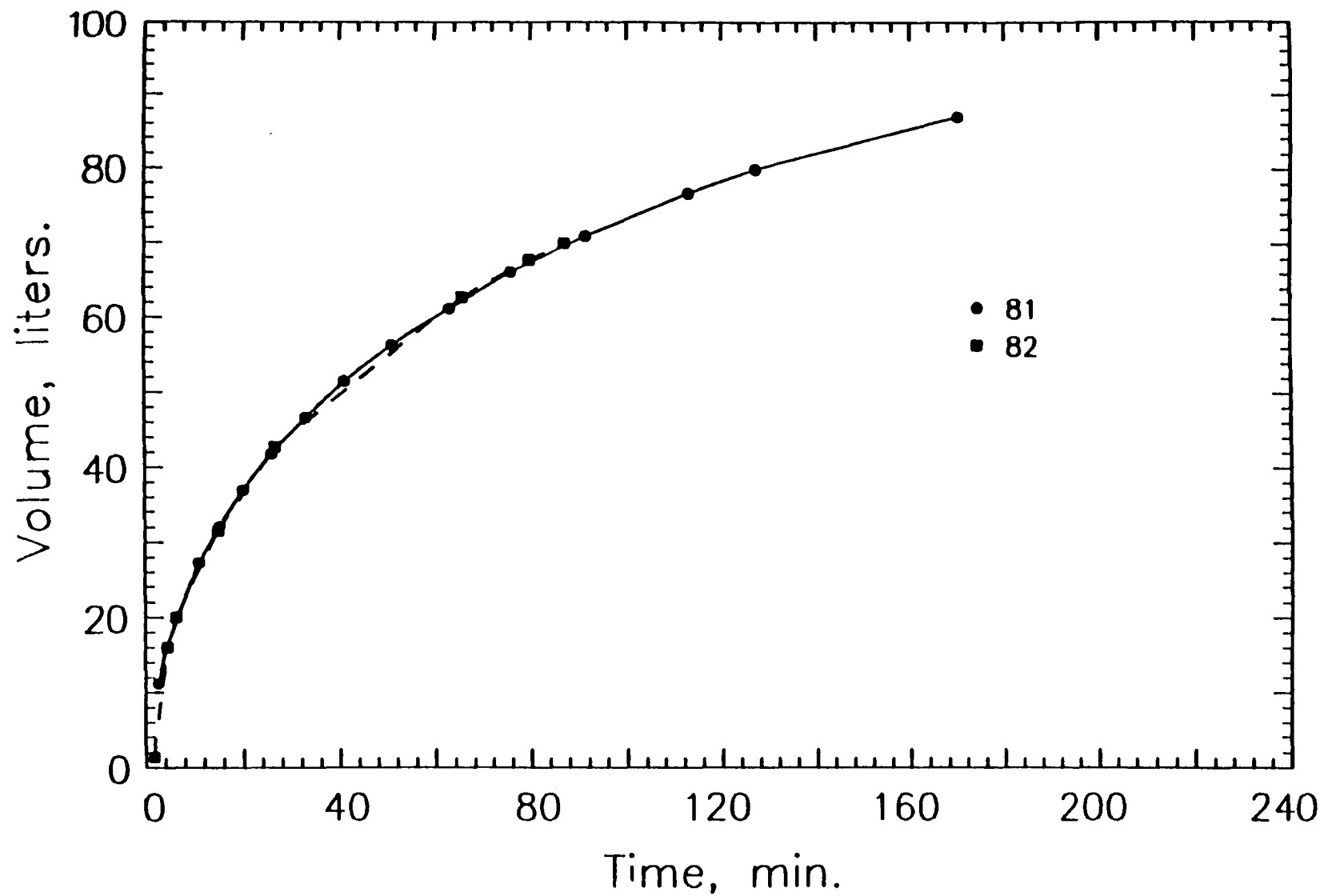
Figure 17.   Cumulative filtrate volume for XC3 suspensions in runs 81 and 82 during high-pressure filtration.

52
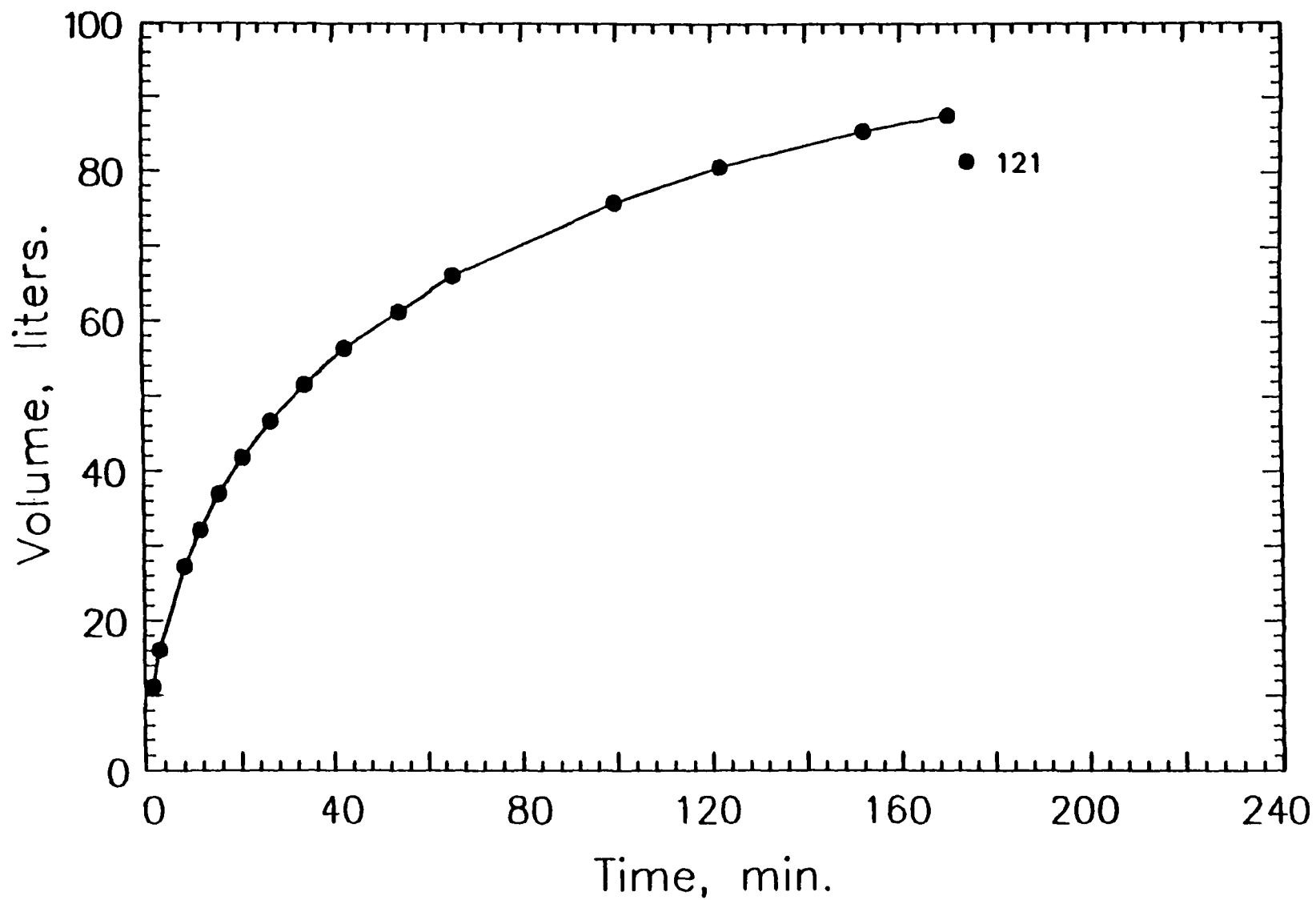
Figure 18. Cumulative filtrate volume for XC3 suspension in run 121 duirng high-pressure filtration.

low-pressure runs are presented in Figures 19-23. Cumulative filtrate data were, however, not taken for run J-42, an extremely short (14 min) run. Data for replicate runs 73 and 74, and J-11 and J-12 were virtually identical, as experienced in previous replicate runs. It should be recognized that runs 52, J-11 and J-12; and runs 73, 74, J-41 and J-42 were, respectively, performed on the same days with virtually identical aliquots of sludge suspensions. It is, however, difficult here to confirm the similarities and differences between the various runs; this will be examined later.

TABLE 15. CHARACTERISTICS OF CLARIFIER UNDERFLOW SUSPENSIONS (XC3) FOR LOW-PRESSURE, FIXED-VOLUME PRESSURE FILTRATION ON NETZSCH AND JWI PRESSES

Run	Type	Date	Temp. °C	pH	Suspended Solids Total g/L	Fixed g/L	Specific Resistance Evaluation r Tm/kg	C_k %	CST s
NETZSCH PRESS									
52	XC3	314	16	7.9	20.6	16.7	3.3	7.1	70
73	XC3	314/ (320)*	14	7.6	21.0	16.7	2.5	-	54
74	XC3	314/ (320)*	13	7.6	20.2	16.0	2.3	7.03	64
JWI PRESS									
J-11	XC3(51/2)	314/ (320)*	17	7.8	20.5	16.5	2.9	7.5	67
J-12	XC3(51/2)	314/ (320)*	17	8.0	20.3	16.2	3.2	7.5	70
J-41	XC3(73/4)	314	15	7.5	19.5	16.4	2.8	7.3	60
J-42	XC3(73/4)	314	15	7.5	19.5	16.4	2.8	7.3	70
J-121	XC3(121/2)	314/ (341)*	18	7.2	21.1	16.8	2.7	7.1	60

*Notation indicates suspension was collected on first day indicated and filtration run was conducted on second day indicated.

The results for the cakes produced during the low-pressure runs are presented in Table 16. Cake solids data for runs 52, J-11 and J-12 indicated minor improvements in performance with increased filtration time. Run times of 100, 176 and 224 min resulted in production of average cake
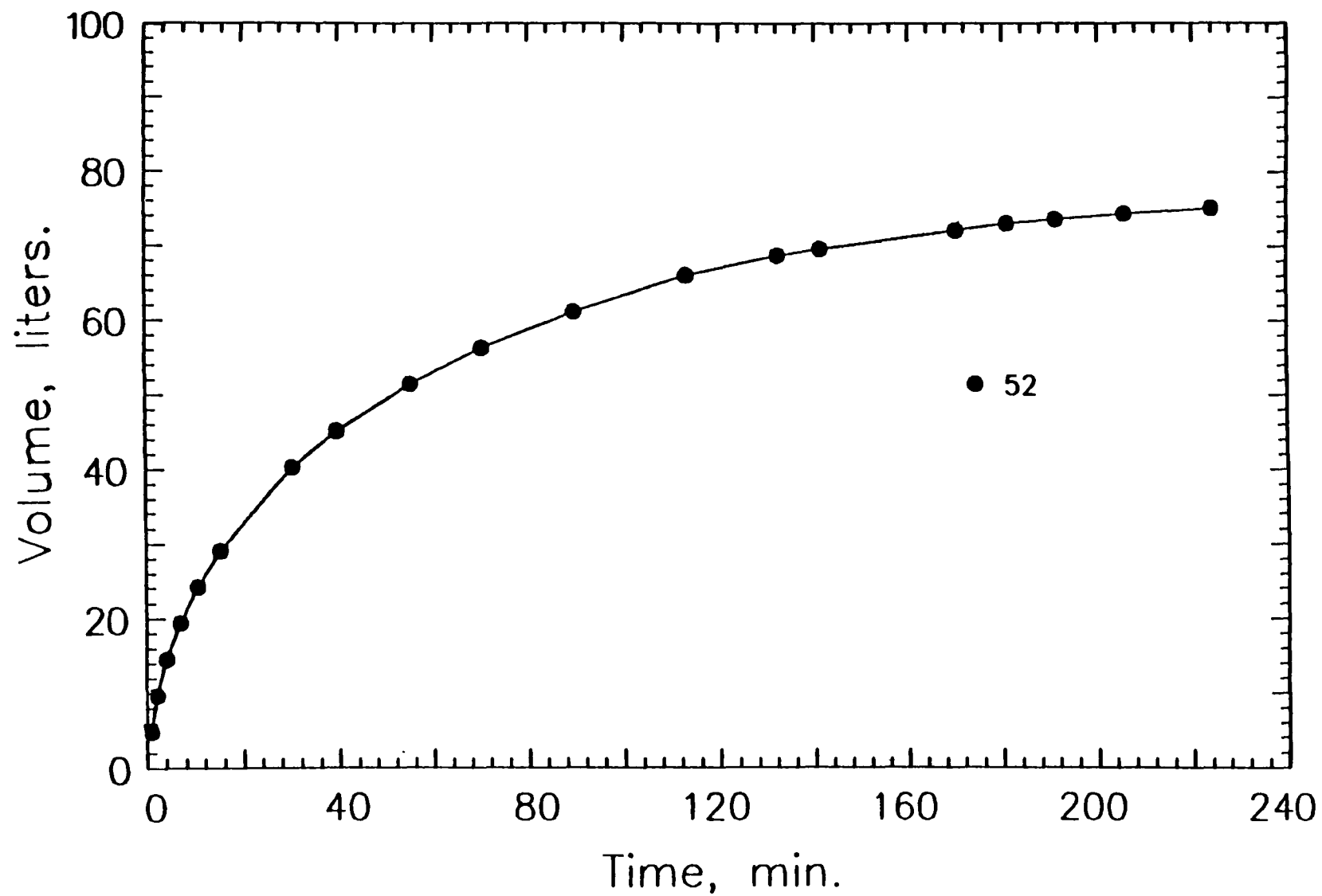
Figure 19.   Cumulative filtrate volume for XC3 suspension in run 52 during low-pressure
filtration on Netzsch press.
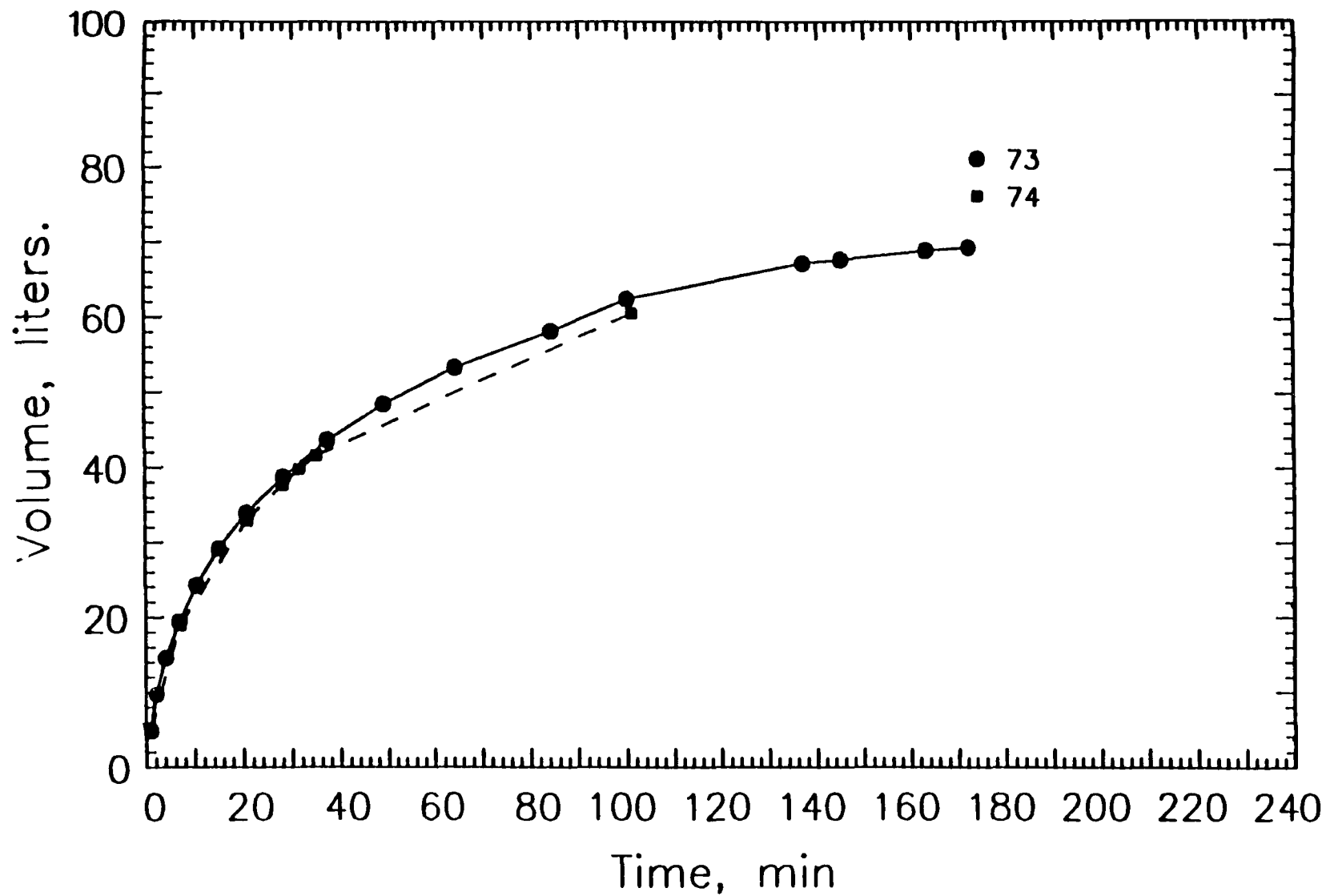
Figure 20. Cumulative filtrate volume for XC3 suspensions in runs 73 and 74 during low-pressure filtration on Netzsch press.
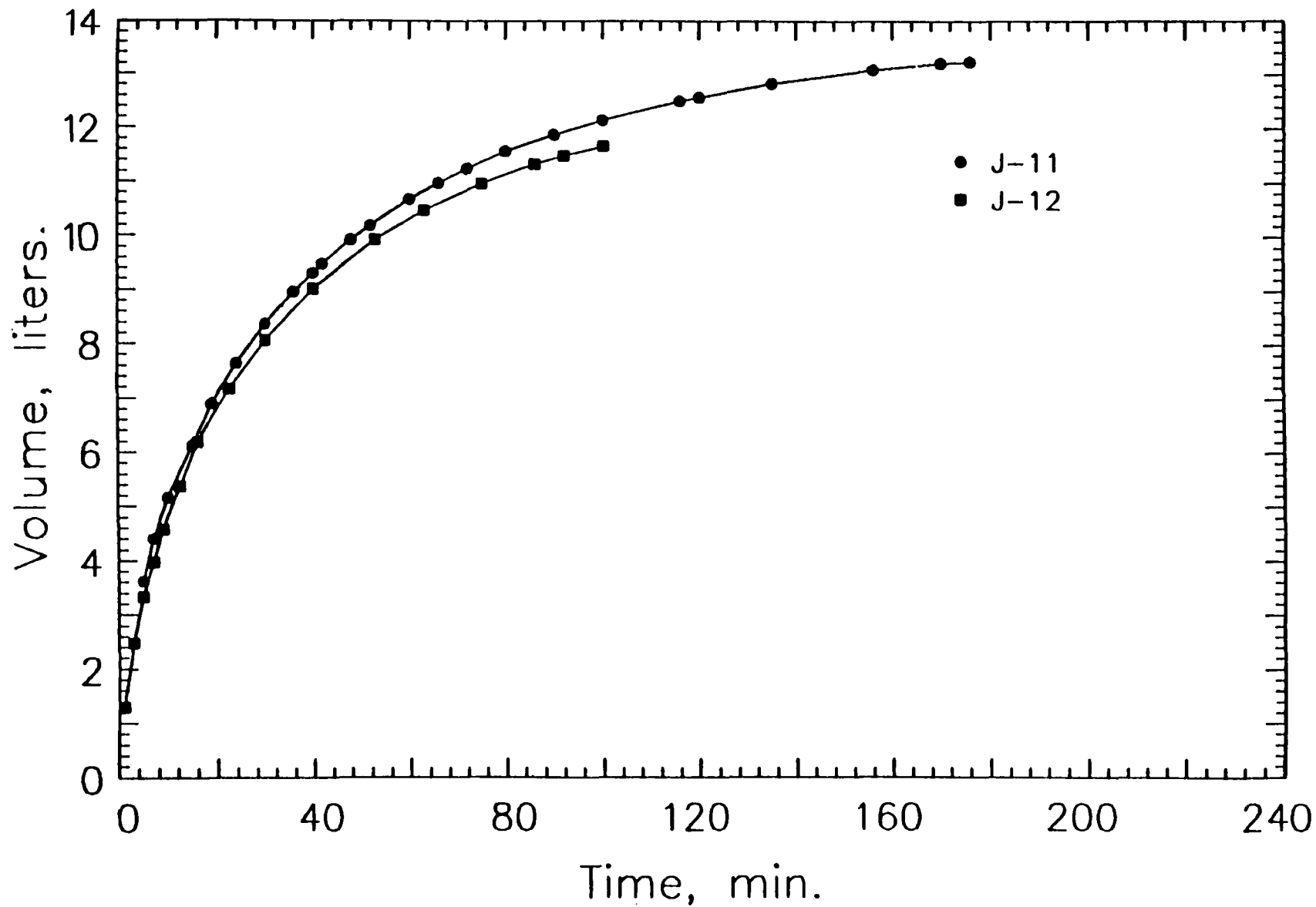
Figure 21. Cumulative filtrate volume for XC3 suspensions in runs J-11 and J-12 during low-pressure filtration on JWI press.
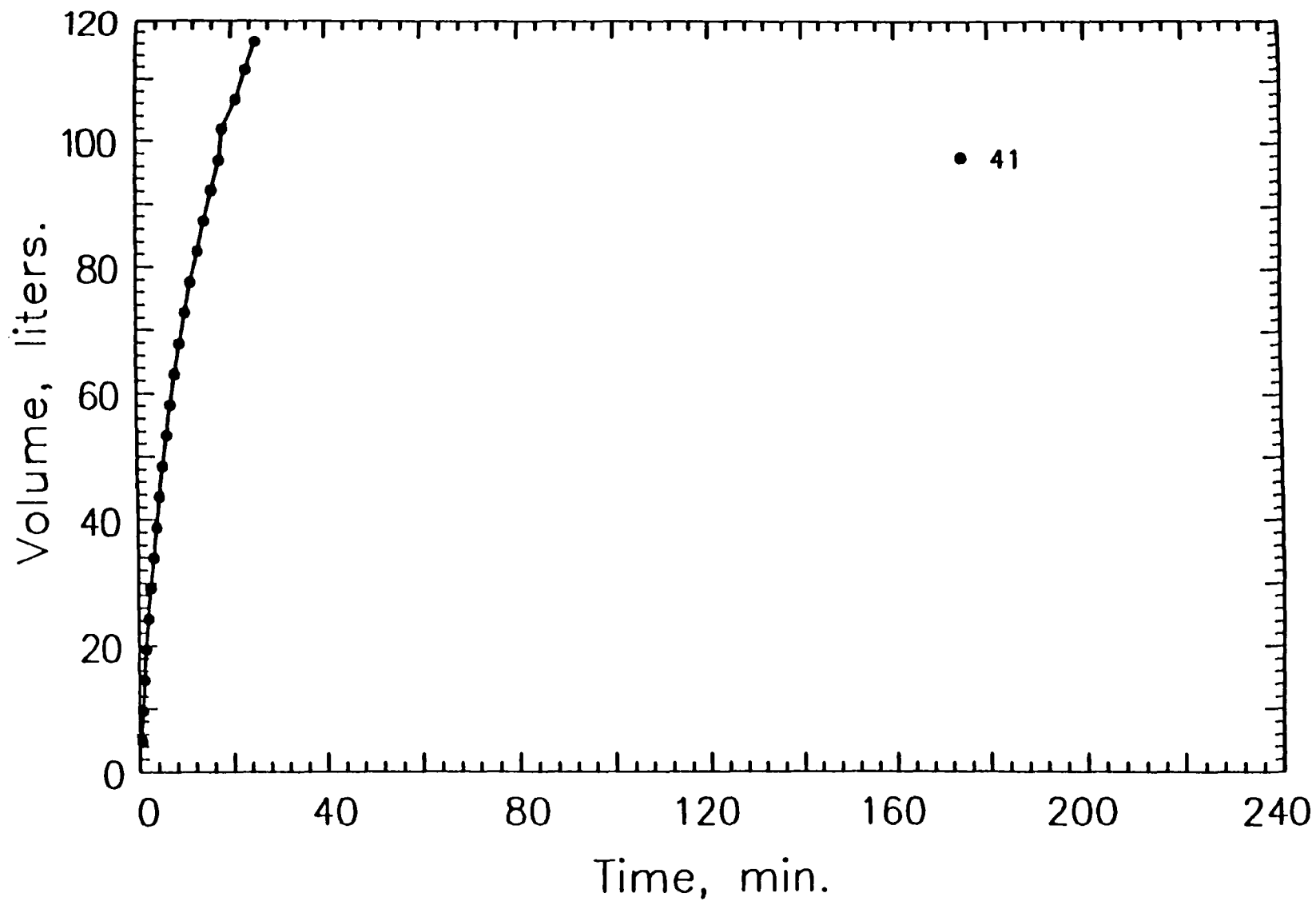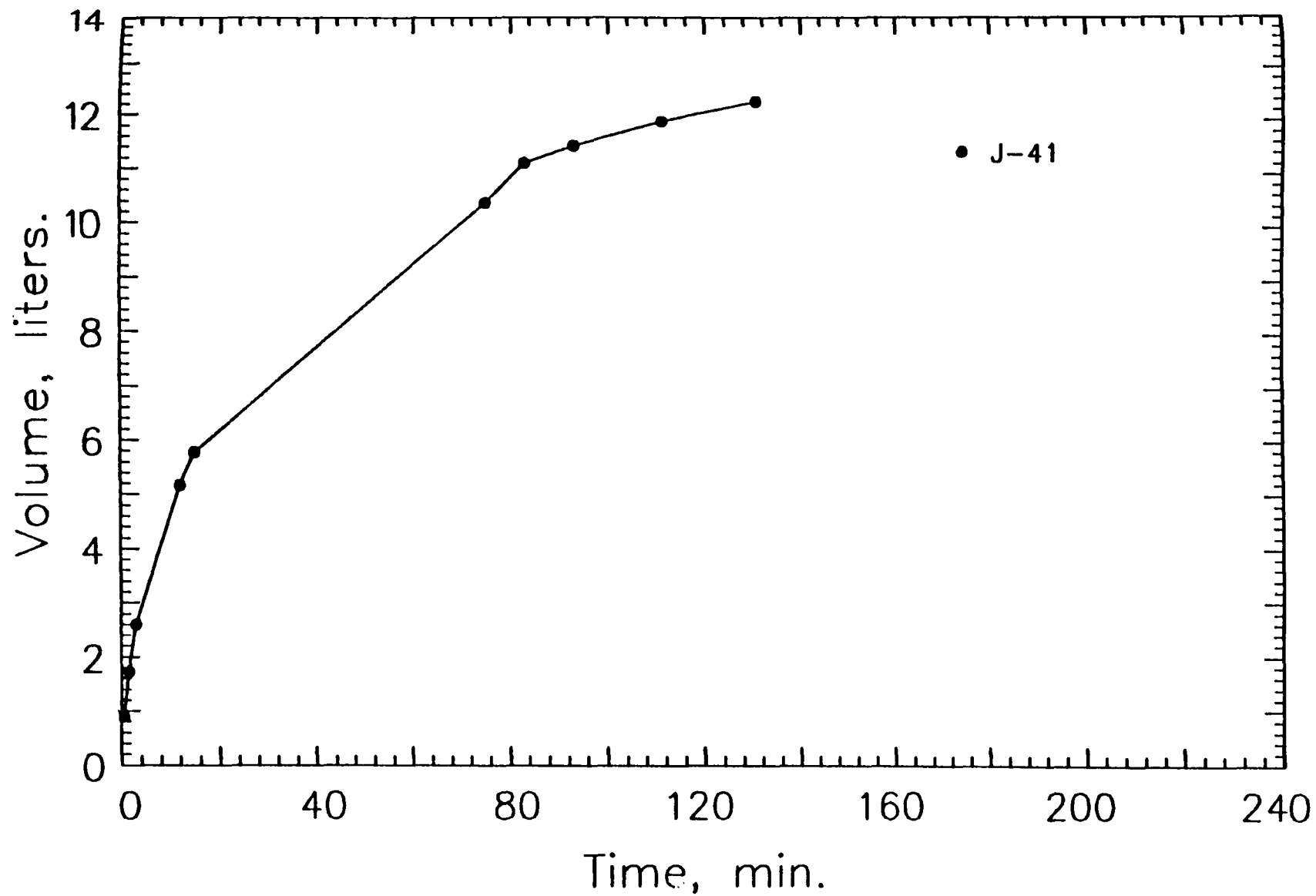
Figure 22. Cumulative filtrate volume for XC3 suspension in run J-41 during low-pressure filtration on JWI press.
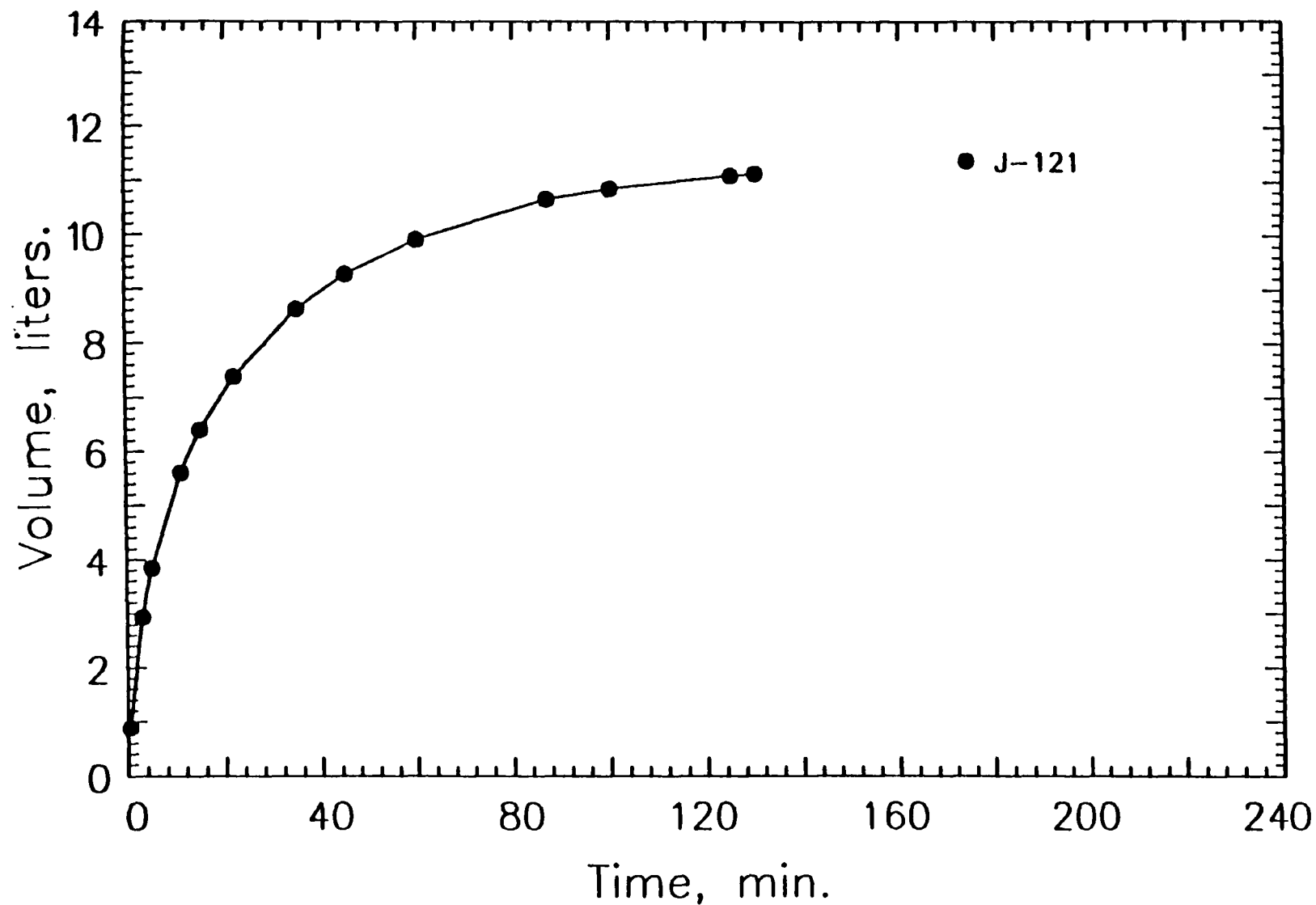
Figure 23. Cumulative filtrate volume for XC3 suspension in run J-121 during low-pressure filtration on the JWI press.

58

solids of 15.4, 16.3 and 16.2 percent, respectively. Cake solids data for runs 73 and 74 indicate improved performance with an increase in filtration time from 101 min to 172 min as did runs J-41 and J-42 for an increase from 17 min to 135 min. Since these four runs were conducted on the same day, it

TABLE 16. RESULTS FOR LOW-PRESSURE, FIXED VOLUME FILTRATION OF CLARIFIER UNDERFLOW SUSPENSION (XC3) ON NETZSCH AND JWI PRESSES

Run	Filtration Pressure	Time of Filtration	Dewatered Cake(s)		
			Mass	Solids Content	
				Total	Fixed
	bar	min	kg	%	%
NETZSCH PRESS					
52	6-7	224	1) 5.18	16.5	12.6
			2) 5.23	15.9	12.1
73	6-7	172	1) 5.2	15.6	-
			2) 5.0	15.1	-
74	6-7	101	1) 5.14	14.4	-
			2) 5.14	14.0	-
JWI PRESS					
J-11	6-7	176	1) 0.905	16.2	12.1
			2) 0.905	16.4	12.4
J-12	6-7	100	1) 0.905	15.5	11.8
			2) 0.91	15.3	11.8
J-41	6-7	135	1) 0.905	16.4	-
			2) 0.905	16.1	-
J-42	6-7	17	1) 0.805	9.4	-
			2) 0.855	7.8	-
J-121	6-7	130	1) 0.875	15.4	-
			2) 0.880	15.6	-

is furthermore apparent that the data, as expected, indicate that increased filtration time, over a broad range, produced drier cakes, although run J-41 appeared to indicate higher performance at 135 min than did run 73 at 172 min. Subtle differences between the two presses, as well as analytical variations, could have produced this difference.

Segregated Neutralization Suspensions

Filtration studies with segregated neutralization suspensions were generally conducted with suspensions formed by batch neutralization of spent

caustic-etch and anodizing-acid suspensions collected from the finishing line at Plant A. These segregated-neutralization suspensions, designated AS7, were then generally dewatered on a filter press in a series of experiments in which the suspensions were blended with conventional-neutralization suspensions from Plants A and X (i.e., suspensions AC3 and XC3, respectively). Each series typically included one run in which a segregated-neutralization suspension was dewatered without blending with another suspension. The results of these filtration studies are presented below with respect to suspensions with which the segregated-neutralization suspensions were blended.

Blends with Plant A Suspensions (AC3)--
    High-Pressure Filtration--The characteristics of a segregated-neutralization suspension and its blends with AC3 suspensions examined with high pressure filtration are presented in Table 17. Data for runs 171 and 173 indicate, respectively, the properties of the two suspensions. Suspended solids concentrations and pH values indicate two major differences for the suspensions. The segregated neutralization suspension (see run 173 in Table 17) was not thickened prior to analysis, while the conventional neutralization suspension (see run 171 in Table 17) was collected from the underflow

TABLE 17. CHARACTERISTICS OF CLARIFIER UNDERFLOW (AC3) AND SEGREGATED NEUTRALIZATION (AS7) SUSPENSIONS AND BLENDS OF THESE SUSPENSIONS USED IN HIGH-PRESSURE FILTRATION ON NETZSCH PRESS

| Run | Blend % by Volume | | Date | Temp. | pH | Suspended Solids | | Specific Resistance Evaluation | | CST |
	AS7	AC3		°C		Total g/L	Fixed g/L	r Tm/kg	c_k %	s
171	0	100	343	13	8.2	37.9	28.2	3.6	11.7	139
173	100	0	346	13	10.6	180.1	134.0	1.3	43.8	485
174	5	95	346	12	8.7	47.0	35.0	3.9	10.8	172
177	15	85	346	12	9.0	59.9	44.6	2.5	21.9	186
178	30	70	346	9	-	78.0	58.0	2.5	28.9	254

of the gravity thickener at Plant A. The suspended solids concentrations of 37.9 g/L (run 171) and 180.1 g/L (run 173) indicate a wide disparity in potential solids loadings for a filter press. The specific resistance data indicated that the resistances to filtration were similar (i.e., 3.6 and 1.3 Tm/kg) but that the solids in the segregated-neutralization suspensions were less hydrophilic or gelatinous, as indicated by a specific-resistance solids content of 43.8 percent (run 173) vs a value of 11.7 percent (run 171) at a differential pressure of 0.5 bar (50 kPa).

The disparity in suspended solids concentrations for the two suspensions created a problem in evaluating blends of the suspensions. Blended suspensions with higher portions of segregated-neutralization solids would be more concentrated and would require less filtration time to achieve a specified degree of dewatered solids. Notwithstanding this problem, however, it was decided that the most appropriate method of analysis would be through directly blending the two suspensions. From a plant viewpoint, a logical operational procedure would be to blend segregated-neutralization suspensions with influent wastewaters discharged to the neutralization basin and form one combined suspension that would be collected for dewatering from the clarifier underflow. Due to the limited volume of the experimentally-produced segregated-neutralization suspension, this option was not available at Plant A or Plant X and direct formation of blended suspensions on an experimental level was required. With regard to the origin of suspended solids discharged to an aluminum-finishing plant, Saunders et al. (1984) indicated that 65.6 percent of the aluminum mass flow into the wastewater treatment system at one plant was contained in the concentrated spent etch and acid suspensions. These two spent solutions constituted only 6 percent of the total wastewater flow at the plant. On this basis a blend of approximately 6 percent (i.e., 6 volumes of segregated-neutralization suspension to 94 volumes of conventional-neutralization suspension) would appear to be appropriate at this plant. However, the operational procedures of the caustic etch system will significantly affect the mass flow of aluminum discharged in the spent etch. For example, 29 percent of the overall aluminum mass flow at the above plant (Saunders et al., 1984) was contained in caustic-etch rinsewaters, indicating a high dragout rate from the etch tanks and an apparent high-level of aluminum in the etching solution. Conversion of this etch tank to a system employing limited chemical additives, or extenders, could be more economical, but would result in increased mass flow of aluminum in spent etch. Use of segregated neutralization could, however, result in the production of less wet mass of dewatered sludge solids, which would be attributable to a higher solids content of the final dewatered sludge. It was therefore deemed appropriate to investigate blends of from 5 to 15 percent by volume to represent typical values for those systems using segregated neutralization on a routine, steady-state basis. In some instances where batch treatment of spent etch is practiced, blends of higher volumetric ratios could be utilized and for this reason blends of 30 percent by volume were examined in some experiments. Therefore, a series of experimental runs typically included runs with each of the two suspensions and three blends of the two suspensions, as indicated for example by runs 171, 173, 174, 177 and 178 in Table 17.

The data indicating percent by volume for each blend were indicative of the actual volumes employed, while the suspended solids data were based on actual measurements of each blended or neat suspension, not calculations using concentrations of initial suspensions. The suspended solids concentrations for blended and neat suspensions ranged from 37.9 to 180.1 g/L, indicating considerable variation in the solids loading for each run. The specific resistance values did not vary considerably but followed a trend consistent with blend variations. Specific-resistance cake solids increased dramatically as the percent of AS7 solids increased, ranging from 10.8 percent (run 174) to 28.9 percent (run 178). CST values generally increased

as suspended solids concentration increased, as expected.

Cumulative filtrate volume data for the high-pressure runs are presented in Figure 24. As the percentage of AS7 suspension increased, the volume of filtrate collected decreased considerably. As shown in Table 18, the solids content of the dewatered cake increased (i.e., from 26.6 percent (run 174) to 34.8 percent (run 178)) with increasing percentage of AS7 suspension in a blend. The addition of 5 percent AS7 suspension to the AC3 suspension resulted in improved performance in that similar dewatered cake solids concentration was achieved in 70 min, as opposed to 103 min for the conventional-neutralization suspension alone.

TABLE 18. RESULTS FOR HIGH-PRESSURE, FIXED-VOLUME FILTRATION OF CLARIFIER UNDERFLOW (AC3) AND SEGREGATED-NEUTRALIZATION (AS7) SUSPENSIONS AND BLENDS OF THESE SUSPENSIONS

Run	Blend % by Volume		Filtration Pressure	Time of Filtration	Mass	Solids Content	
	AS7	AC3	bar	min	kg	Total %	Fixed %
171	0	100	14-15	103	5.62	26.3	18.8
173	100	0	14-15	35	6.97	53	-
174	5	95	14-15	70	5.65	26.6	20.1
177	15	85	14-15	87	5.94	33.1	-
178	30	70	14-15	70	6.10	34.8	-

Low-Pressure Filtration--Characteristics of the various blends of AS7 and AC3 suspensions are presented in Table 19. The five runs (J-81 through J-85) were conducted in parallel with the high-pressure runs in Table 17 with aliquots of the same suspensions and suspension blends. Cumulative filtrate volumes for the low-pressure runs are presented in Figure 25. It is apparent that filtration rates decreased with increasing levels of AS7 in blended suspensions, as did the total volume of filtrate produced. These responses are reflective of the increased solids loadings and of the improved dewatering properties of the AS7 suspensions.

The results of the filtration studies are included in Table 20. The results indicated the positive impact of increased addition of segregated-neutralization solids to the blended suspension. At similar filtration times, the average dewatered-cake solids content increased from 23.7 percent for the conventional-neutralization suspension to 26.4, 30.8, and 36.5 percent for 5, 15 and 30 percent blends, respectively. These values were similar to those achieved with high-pressure filtration at shorter filtration times (see Table 18).
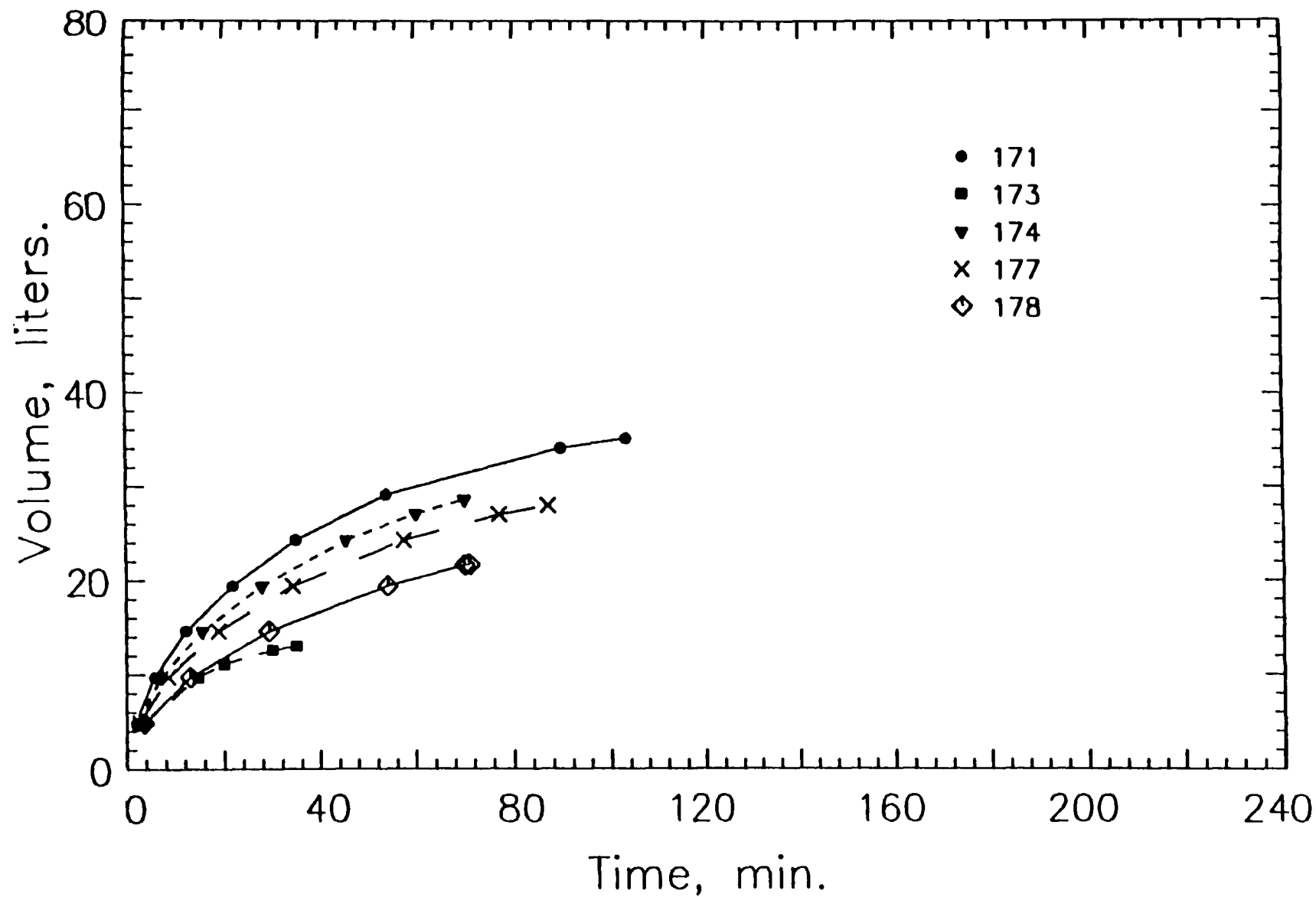
Figure 24. Cumulative filtrate volume for AC3 and AS7 suspensions and blended suspensions in runs 171, 173, 174, 177 and 178 during high-pressure filtration.

TABLE 19. CHARACTERISTICS OF CLARIFIER UNDERFLOW (AC3) AND SEGREGATED NEUTRALIZATION (AS7) SUSPENSIONS AND BLENDS OF THESE SUSPENSIONS USED IN LOW-PRESSURE FILTRATION ON JWI PRESS

Run	Blend % by Volume		Date	Temp. °C	pH	Suspended Solids		Specific Resistance Evaluation		CST
	AS7	AC3				Total g/L	Fixed g/L	r Tm/kg	C_k %	s
J-81(171)*	0	100	346	13	8.2	37.9	28.2	3.6	11.7	139
J-82(173)	100	0	346	22	10.6	180.1	134	1.3	43.8	485
J-83(174)	5	95	346	12	8.7	47.0	35.0	3.9	10.8	172
J-84(177)	15	85	346	22	9.0	59.9	44.6	2.5	21.9	186
J-85(178)	30	70	346	10	-	78.0	58.0	2.5	28.9	254

*Companion runs performed at high pressure on Netzsch press.

TABLE 20. RESULTS FOR LOW-PRESSURE, FIXED-VOLUME FILTRATION OF CLARIFIER UNDERFLOW (AC3) AND SEGREGATED-NEUTRALIZATION (AS7) SUSPENSIONS AND BLENDS OF THESE SUSPENSIONS

Run	Blend % by Volume		Filtration Pressure bar	Time of Filtration min	Mass kg		Solids Content	
	AS7	AC3					Total %	Fixed %
J-81(171)*	0	100	6-7	100	1)	0.965	23.8	16.7
					2)	0.970	23.6	16.7
J-82(173)	100	0	6-7	45	1)	1.195	38.7	-
					2)	1.195	39.7	-
J-83(174)	5	95	6-7	110	1)	0.995	26.7	19.5
					2)	0.985	26.1	19.2
J-84(177)	15	85	6-7	100	1)	1.03	30.7	23.6
					2)	1.03	30.9	-
J-85(178)	30	70	6-7	107	1)	1.10	36.5	-
					2)	1.085	36.5	-

*Companion runs performed at high pressure on Netzsch press.

Blends with Plant X Suspensions (XC3)--
    High-Pressure Filtration--Data for characteristics of the blends examined by high-pressure filtration on the Netzsch press are presented in
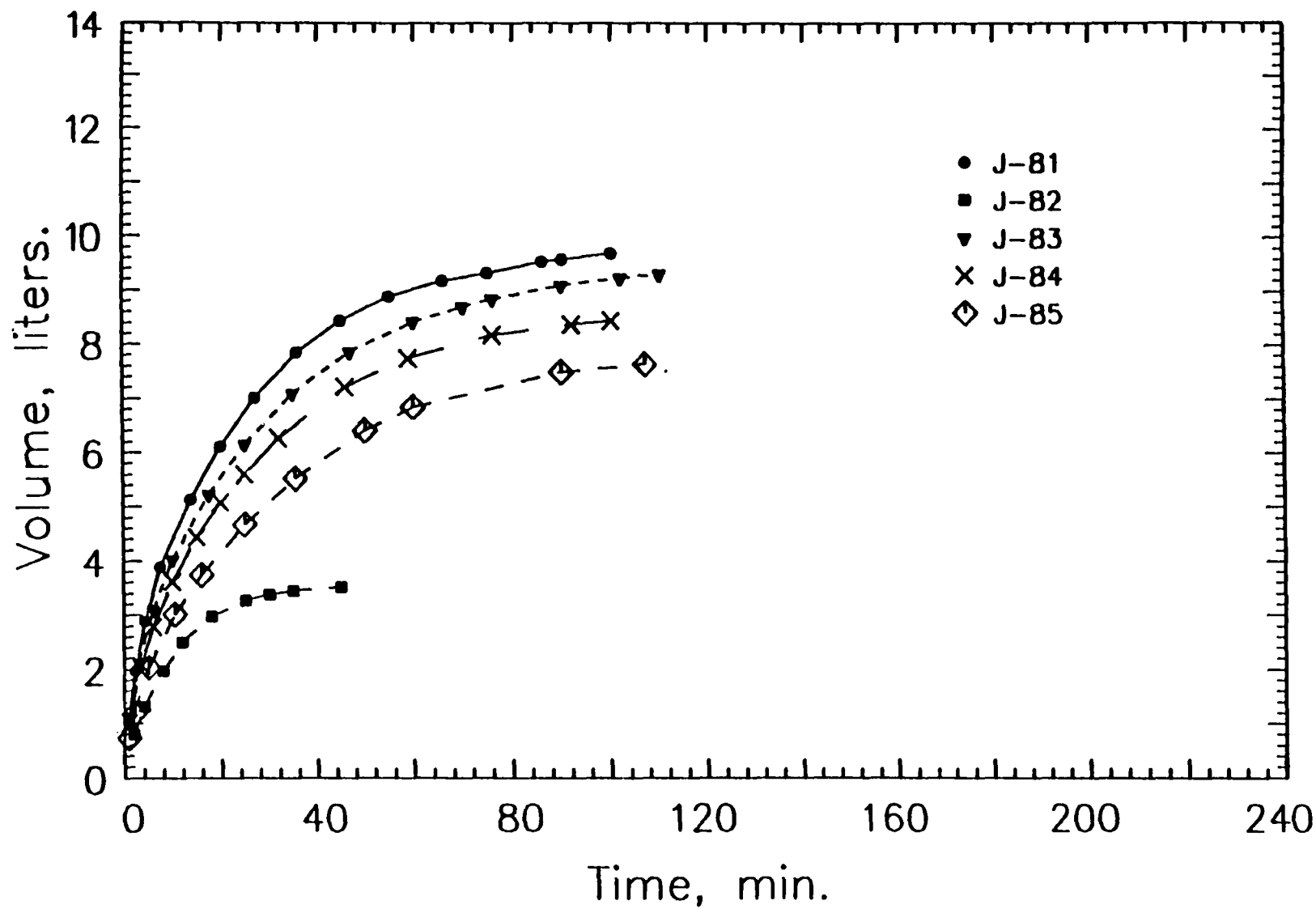
Figure 25. Cumulative filtrate volume for AC3 and AS7 suspensions and blended suspensions in runs J-81 through J-85 during low-pressure filtration.

Table 21. A sample of the conventional-neutralization suspension alone was not included in the 90-series since this suspension had been examined extensively in previous studies (see Tables 13 and 14 and Figures 16-18).

TABLE 21. CHARACTERISTICS OF A SEGREGATED NEUTRALIZATION (AS7) SUSPENSION AND BLENDS WITH A CLARIFIER UNDERFLOW SUSPENSION USED IN HIGH-PRESSURE FILTRATION ON NETZSCH PRESS

Run	Blend % by Volume		Date	Temp.	pH	Suspended Solids		Specific Resistance Evaluation		CST
	AS7	XC3		°C		Total g/L	Fixed g/L	r Tm/kg	C_k %	s
91	15	85	322	18	8.3	42.2	33.5	1.5	16.9	90
92	35	65	322	–	8.6	71..7	56.9	1.4	19.3	134
93	100	0	322	36	8.8	173.2	137.5	1.5	49.5	581

Data in Tables 13 and 14 for runs 81 and 82 were collected on the day prior to the 90-series and will be used for comparative purposes. As with AC3 suspensions, the suspended solids concentration increased dramatically for the blended suspensions. The specific resistance of the blends decreased to that of the segregated-neutralization suspension but specific-resistance cake solids increased with increasing percentage of segregated-neutralization solids.

The filtrate-volume data for runs 91-93 are presented in Figure 26 and other results are presented in Table 22. For a 15 percent blend, the dewatered cake solids increased from an average value of 16 percent (run 82 in Table 14) to approximately 28 percent, while a 35-percent blend produced a cake with an approximate solids content of 37 percent.

Low-Pressure Filtration--Characteristics of the blended suspensions examined by low-pressure filtration are presented in Table 23 and data in Table 15 are representative of the XC3 suspension used to make the blended suspensions.

Data in Figure 27 and Table 24 indicate the filtration results for the three low-pressure runs. The dewatered solids for the 15-percent blend was 26.4 percent, while that for the XC3 suspension at a comparable time of filtration (i.e., run 75 in Table 16) was 14.2 percent. The 35-percent blend produced a dewatered-cake solids of 35.7 percent, while that for the segregated-neutralization suspension was 50.8 percent.

A comparison of low- and high-pressure filtration for the AC3 and XC3 blends is presented in Table 25. In the majority of the instances, high-pressure filtration resulted in higher cake solids contents or comparable solids contents in less filtration time. However, the
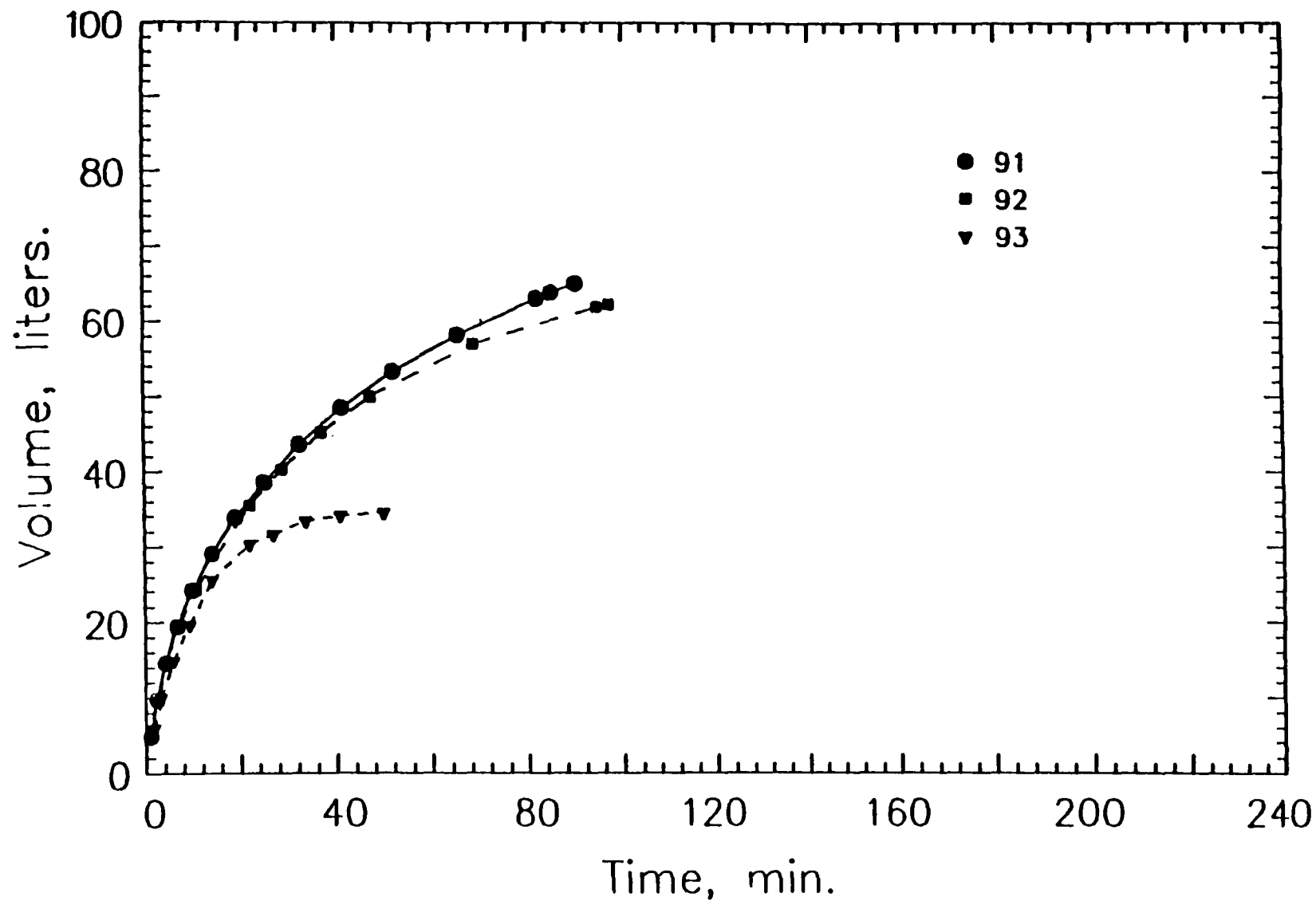
Figure 26. Cumulative filtrate volume for an AS7 suspension (run 93) and blends of XC3 and AS7 suspensions in runs 91 and 92 during high-pressure filtration.

TABLE 22. RESULTS FOR HIGH-PRESSURE, FIXED-VOLUME FILTRATION
OF A SEGREGATED NEUTRALIZATION (AS7) SUSPENSION
AND BLENDS WITH A CLARIFIER UNDERFLOW (XC3)
SUSPENSION

Run	Blend % by Volume		Filtration Pressure	Time of Filtration	Mass	Solids Content	
	AS7	AC3	bar	min	kg	Total %	Fixed %
91	15	85	14-15	90	1) 5.69	26.9	-
					2) 5.75	28.9	-
92	35	65	14-15	97	1) 6.15	37.4	-
					2) 6.31	36.8	-
93	100	0	14-15	50	1) 7.36	53.8	-
					2) 7.12	54.2	-

TABLE 23. CHARACTERISTICS OF A SEGREGATED NEUTRALIZATION (AS7)
SUSPENSION AND BLENDS WITH A CLARIFIER UNDERFLOW
(XC3) SUSPENSION USED IN LOW-PRESSURE FILTRATION
ON JWI PRESS

Run	Blend % by Volume		Date	Temp.	pH	Suspended Solids		Specific Resistance Evaluation		CST
	AS7	XC3		°C		Total g/L	Fixed g/L	r Tm/kg	C_k %	s
J-51	15	85	322	20	8.3	46.4	36.8	1.3	18.3	88
J-52	35	65	322	20	-	65.0	51.6	0.9	26.5	100
J-53	100	0	322	20	8.7	155.1	123.1	1.9	47.0	345

TABLE 24. RESULTS FOR HIGH-PRESSURE, FIXED-VOLUME FILTRATION
OF A SEGREGATED NEUTRALIZATION (AS7) SUSPENSION
AND BLENDS WITH A CLARIFIER UNDERFLOW (XC3)
SUSPENSION

Run	Blend % by Volume		Filtration Pressure	Time of Filtration	Mass	Solids Content	
	AS7	XC3	bar	min	kg	Total %	Fixed %
J-51	15	85	6-7	112	1) 0.985	26.0	-
					2) 0.945	26.8	-
J-52	35	65	6-7	105	1) 1.06	35.8	-
					2) 1.05	35.6	-
J-53	100	0	6-7	47	1) 1.22	51.6	-
					2) 1.22	50.0	-
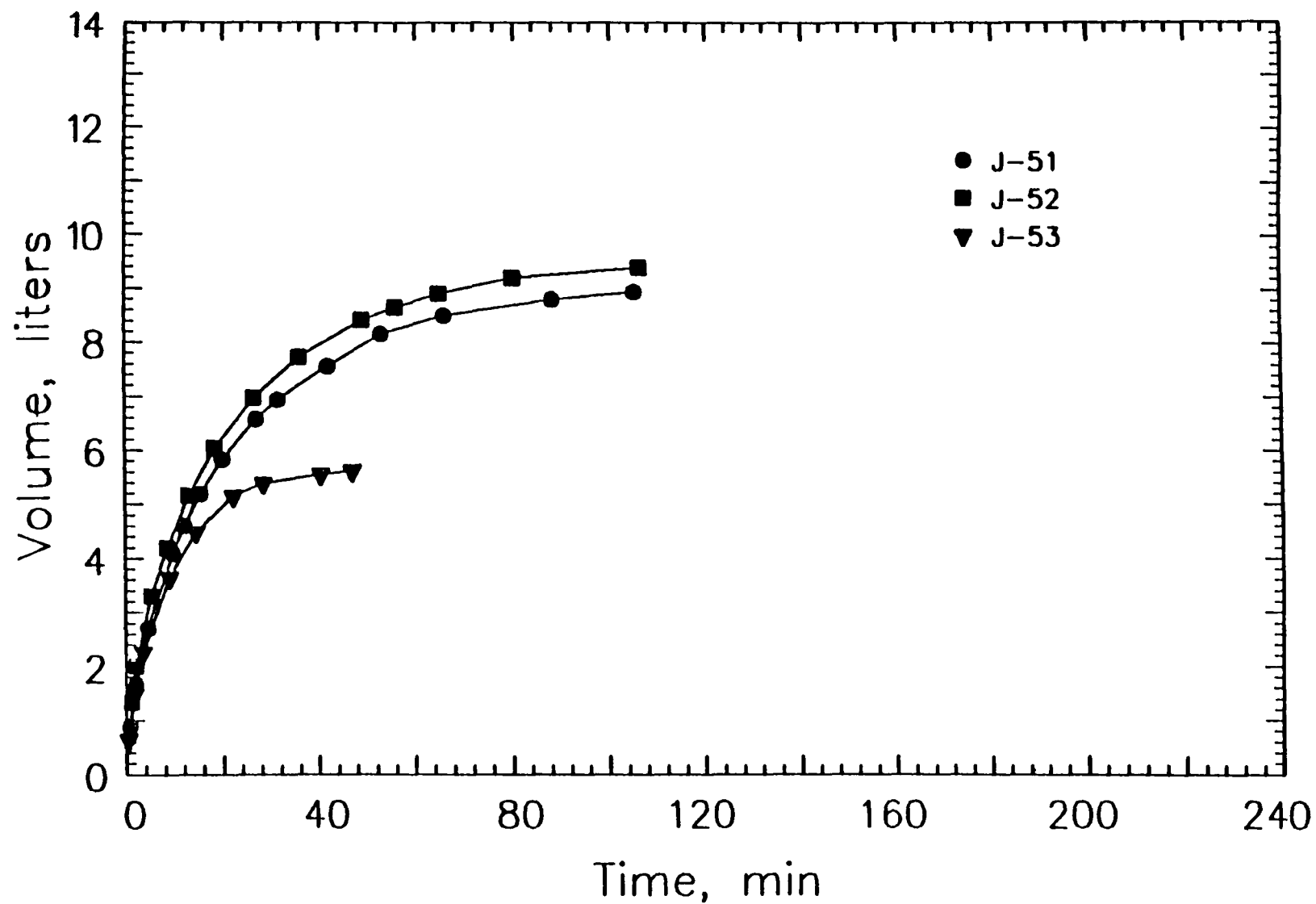
Figure 27. Cumulative filtrate volume for an AS7 suspension and blends of XC3 and AS7 suspensions in runs J-51 through J-53 during low-pressure filtration.

TABLE 25. COMPARISON OF LOW- AND HIGH-PRESSURE FILTRATION FOR BLENDS
OF CLARIFIER UNDERFLOW (AC3; XC3) AND SEGREGATED
NEUTRALIZATION (AS7) SUSPENSIONS

Blend % by Volume		Filtration Pressure bar	Time of Filtration min	Average Dewatered Cake Solids %
AS7	AC3 or XC3			
AC3 Suspensions				
0	100	6-7	103	23.7
0	100	14-15	100	26.3
5	95	6-7	110	26.4
5	95	14-15	70	26.6
15	85	6-7	100	30.8
15	85	14-15	87	33.1
30	70	6-7	107	36.5
30	70	14-15	70	34.8
XC3 Suspensions				
0	100	6-7	100	15.4
0	100	14-15	90	26.0
15	85	6-7	112	26.4
15	85	14-15	90	27.9
35	65	6-7	105	35.7
35	65	14-15	97	37.1
100	0	6-7	47	50.8
100	0	14-15	50	54.0

differences between low- and high-pressure filtration only became
significant with the higher blends with segregated-neutralization
suspensions.

VARIABLE-VOLUME PRESSURE FILTRATION

Selected suspensions were examined using variable-volume filtration
plates (i.e., diaphragm plates) on the Netzsch press. The chambers were
filled with sludge solids at low pressure (i.e., 6-7 bar) and then
pressurized to 15.5 bar. Filtrate volume was collected and monitored
throughout a run, which was terminated when filtrate production ceased. All
of the data collected were for suspensions from Plant A.

Characteristics of clarifier underflow (AC3) suspensions examined using a variable-volume filter press (i.e., diaphragm press) are presented in Table 26. No discernible variations in characteristics from previous suspensions are apparent. The data for run 161 were for a suspension (AC1) collected from the neutralization basin following a massive dump of caustic-etch suspension at Plant A. The sample had an exceptionally high suspended solids concentration and an elevated pH, both as expected for the etch dump.

Cumulative filtrate volumes are presented in Figures 28-33. No filtrate-volume data were collected for initial runs 111-113. Data in Figure 30 for runs 175, 176 and 179 are for blends of AC3 and AS7 suspensions and will be discussed later. The data for run 172 were, however, for an unaltered AC3 suspension. Filtrate data for runs conducted with the diaphragm press were divided into "filter" and "squeeze" cycles.

TABLE 26. CHARACTERISTICS OF CLARIFIER UNDERFLOW (AC3) AND NEUTRA-
LIZATION BASIN (AC1) SUSPENSIONS FOR DEWATERING BY
VARIABLE-VOLUME PRESSURE FILTRATION

Run	Type	Date	Temp.	pH	Suspended Solids		Specific Resistance Evaluation		CST
					Total	Fixed	r	C_k	
			°C		g/L	g/L	Tm/kg	%	s
111	AC3	326	17	-	24.8	-	3.2	10.7	-
112	AC3	326	17	-	24.8	-	3.2	10.7	-
113	AC3	326	17	-	24.8	-	3.2	10.7	-
141	AC3	342	16	8.2	39.7	-	3.2	12.6	110
151	AC3	342	13	8.0	37.1	26.7	3.3	12.2	120
152	AC3	342	13	8.0	36.9	27.5	3.3	12.2	120
153	AC3	342	13	8.0	37.2	27.1	3.3	12.2	120
172	AC3	343	13	8.2	39.9	28.8	3.6	11.7	139
181	AC3	350	16	8.8	39.3	29.2	4.2	12.8	149
182	AC3	350	16	8.8	39.3	29.2	4.2	12.8	149
161	AC1*	303/341	11	9.1	82.3	-	-	-	-

*AC1 suspension collected on Day 303 immediately following caustic-etch dump to neutralization basin (see discussion of Table 1)

The squeeze cycle was initiated with the application of pressure to the internal diaphragms in the recessed chambers. As indicated by data for the squeeze cycle, each filter run was conducted until the filtration rate was

71
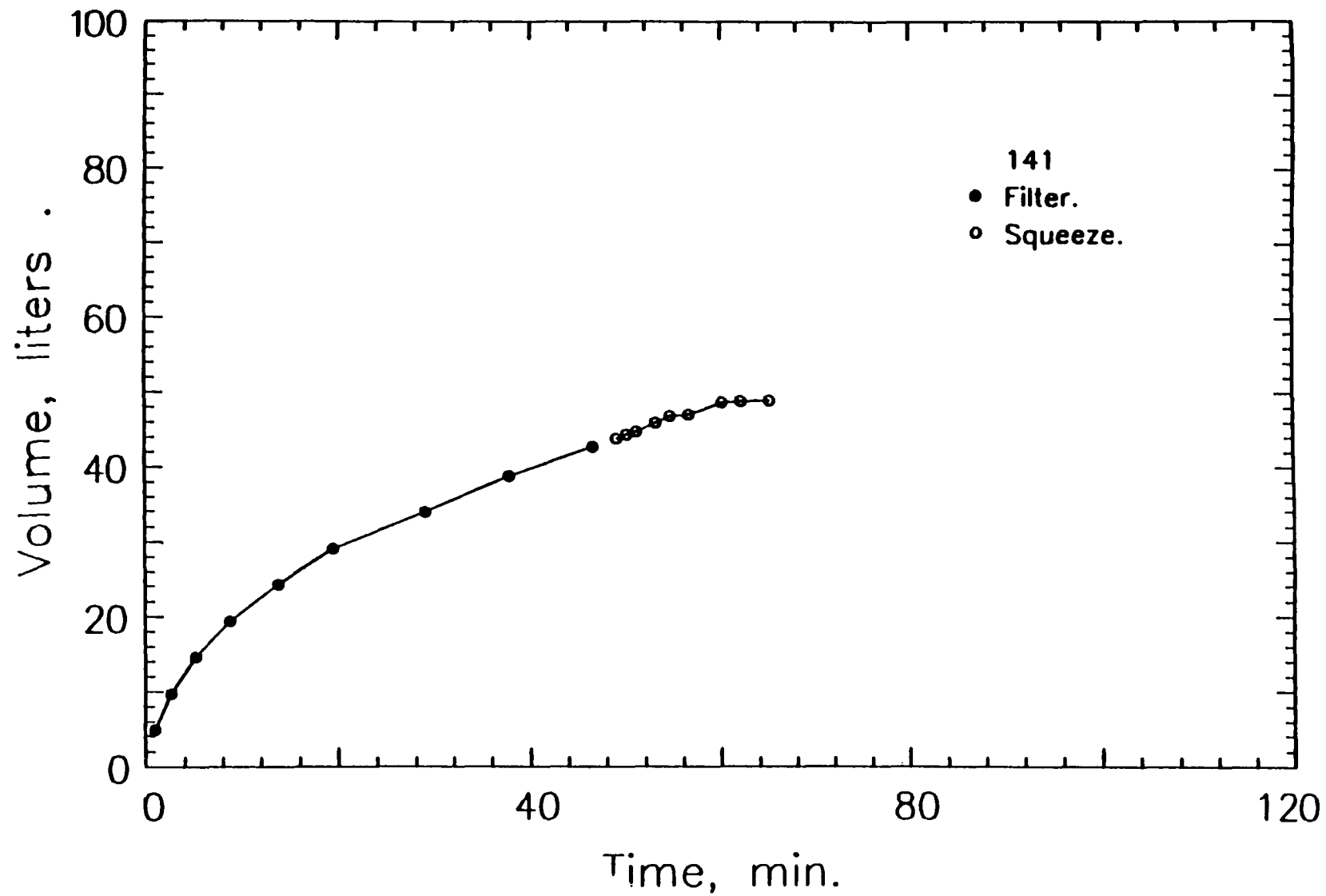
Figure 28. Cumulative filtrate volume for AC3 suspension during run 141 using Netzsch diaphragm press.
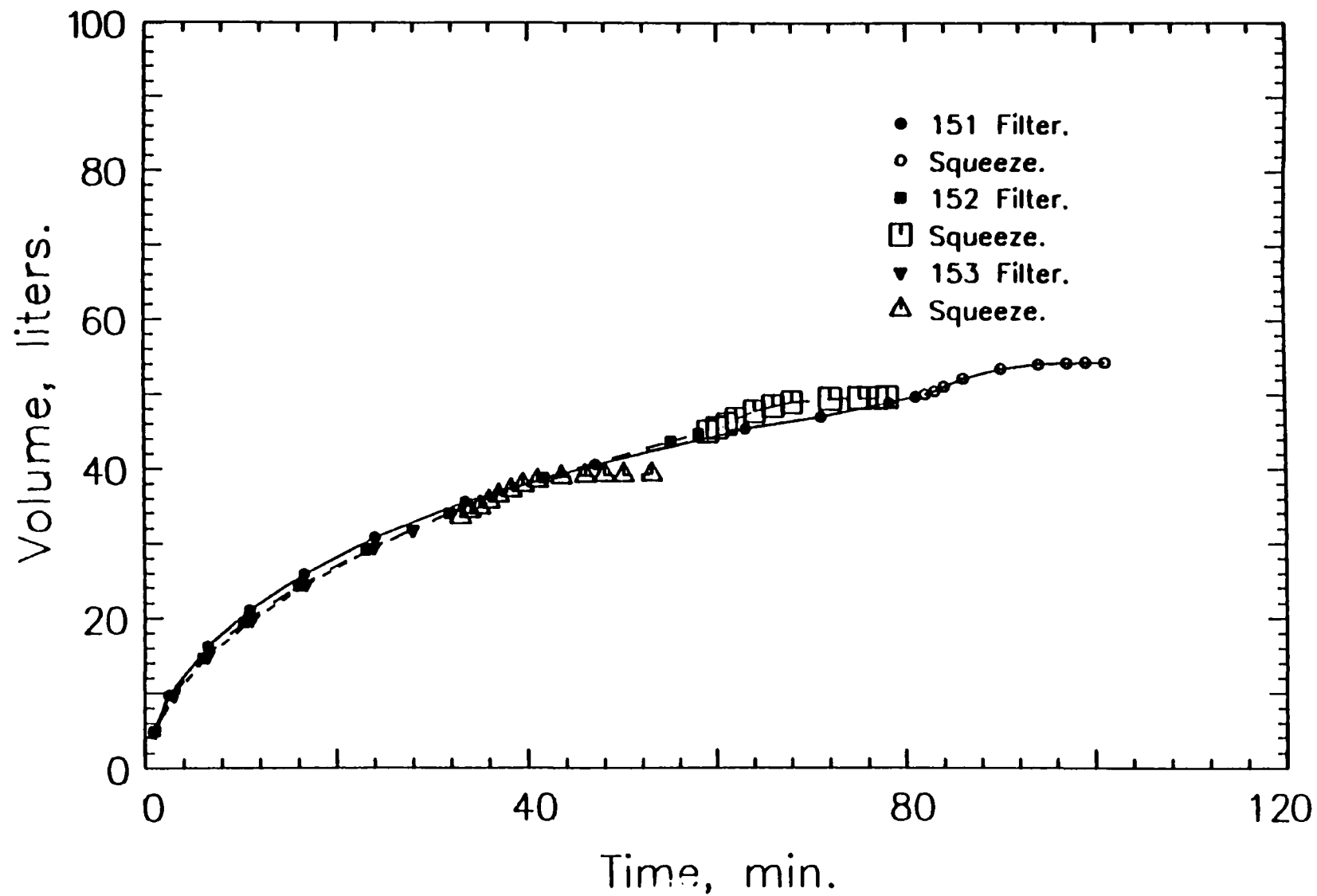
Figure 29.   Cumulative filtrate volume for AC3 suspension during runs 151-153 using Netzsch
diaphragm press.
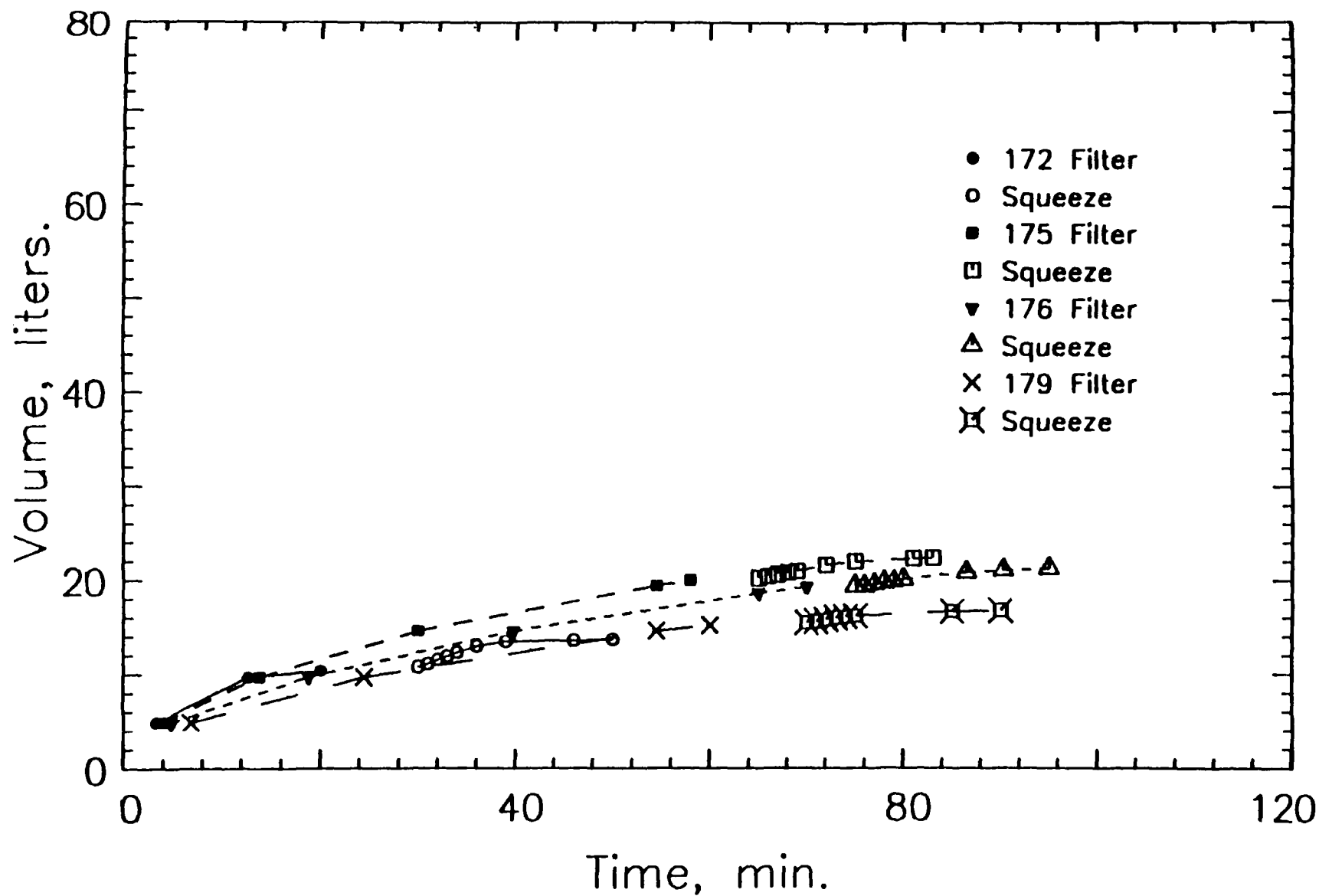
Figure 30. Cumulative filtrate volume for AC3 and blends of AC3 and AS7 suspensions during runs 172, 175, 176 and 179 using Netzsch diaphragm press.
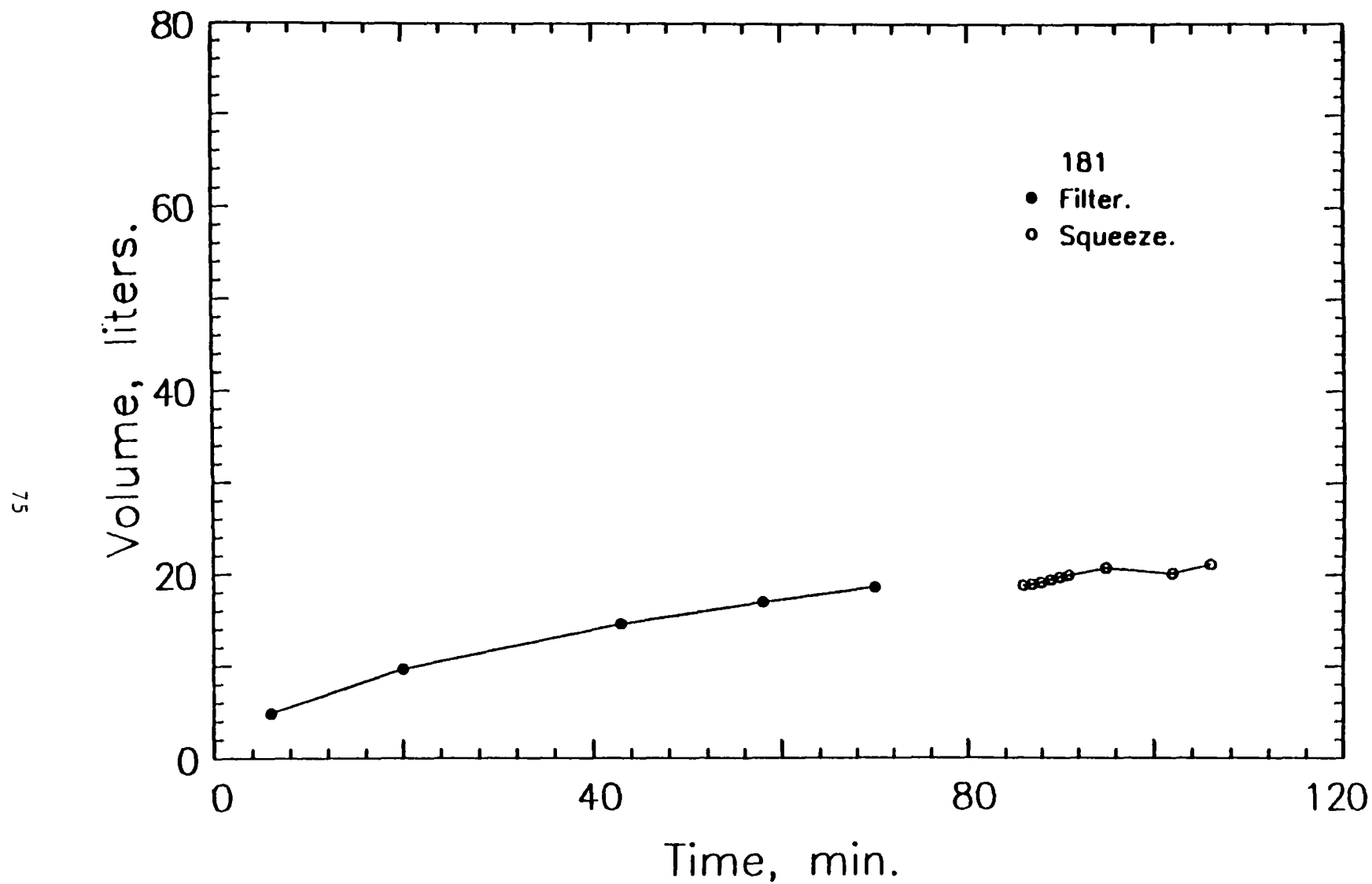
Figure 31. Cumulative filtrate volume for AC3 suspension during run 181 using Netzsch diaphragm press.
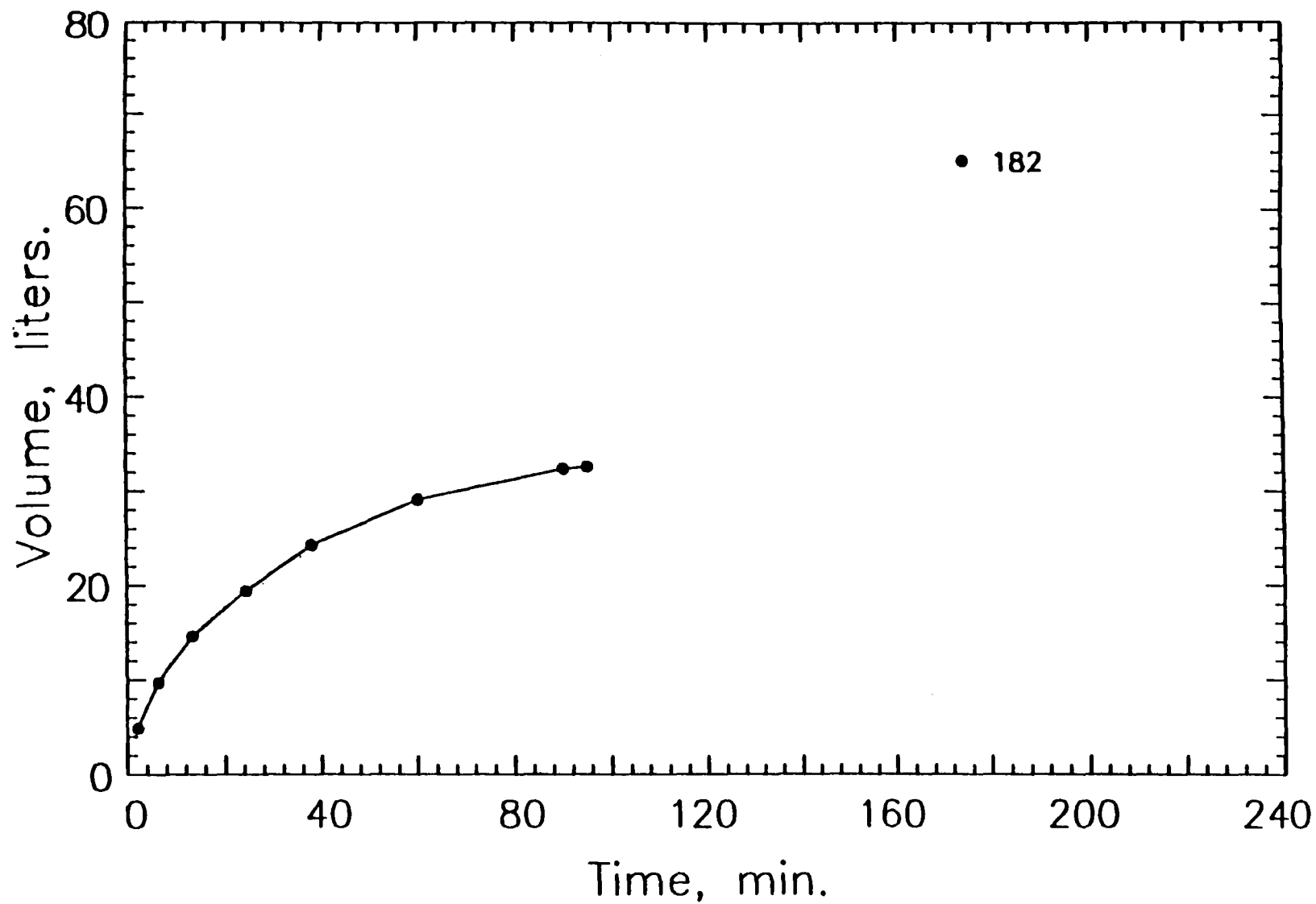
Figure 32. Cumulative filtrate volume for AC3 suspension during run 182 (filter portion only) using Netzsch diaphragm press.
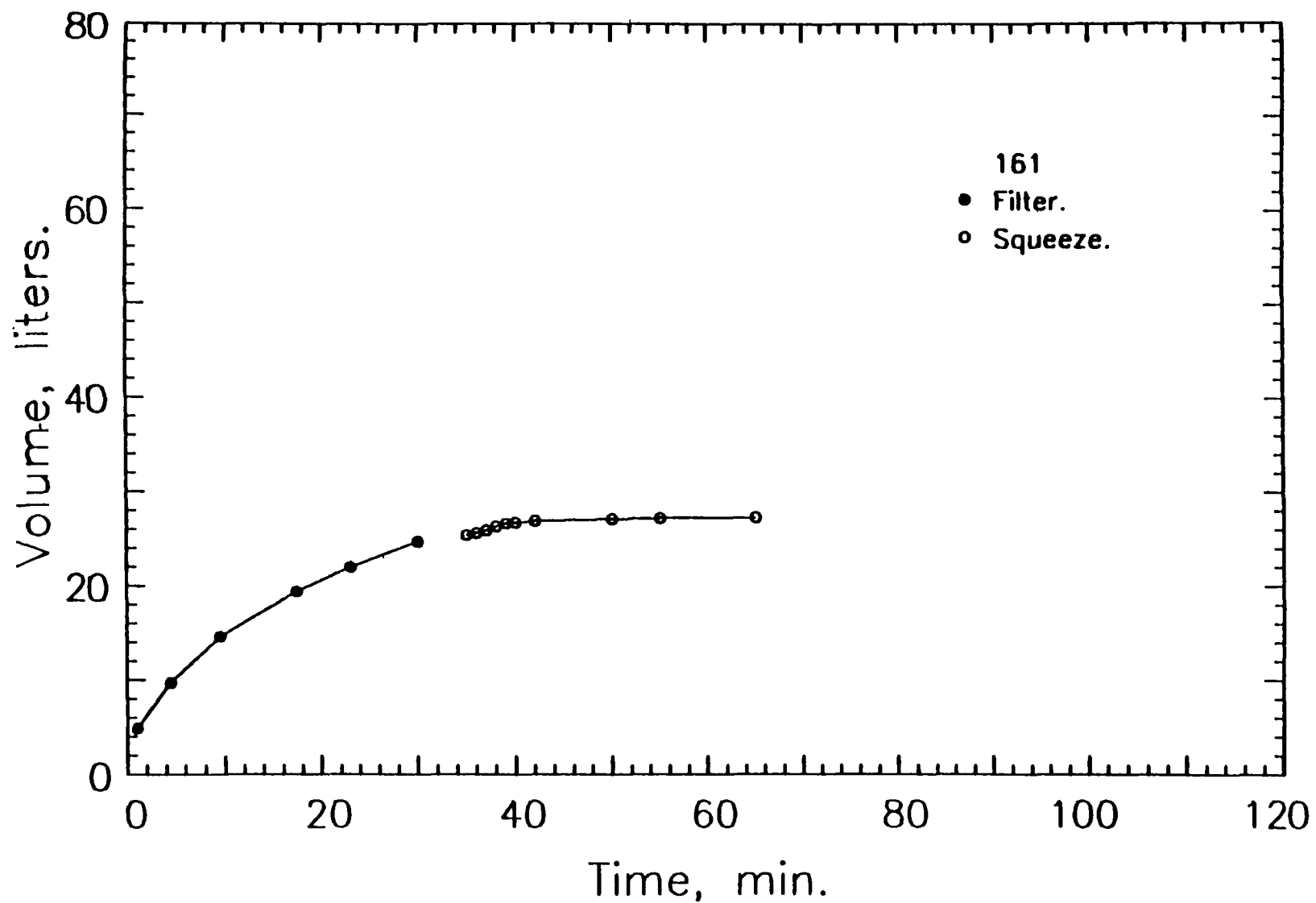
Figure 33. Cumulative filtrate volume for ACI suspension during run 161 using Netzsch diaphragm press.

zero, i.e., no additional water could be squeezed from the sludge cake at a pressure of 15.5 bar.

Results of each run on the diaphragm press are presented in Table 27. For AC3 suspensions, the solids content of dewatered cakes ranged from 25.4 to 31.2 percent and averaged 28.7 percent, for a broad range of operational conditions. While no attempt was made to systematically evaluate the variables involved in diaphragm filtration, the results indicated the relative insensitivity of the final cake solids content to these variables.

TABLE 27. RESULTS FOR VARIABLE-VOLUME, PRESSURE FILTRATION
OF AC3 and AC1 SUSPENSIONS ON NETZSCH PRESS

Run	Time of Filtration, min		Dewatered Cake(s)			
	Filter	Squeeze	Mass kg	Thickness mm	Solids Content	
					Total	Fixed
111	11	8	1) 1.93	8	27.4	-
			2) 1.81	8	27.1	-
112	32	-	1) 2.30	-	25.4	-
			2) 2.21	-	25.4	-
113	50	17	1) 3.13	-	25.4	-
			2) 2.94	-	25.4	-
141	49	17	1) 3.2	17	30.9	-
			2) 3.3	17	31.2	-
151	81	20	1) 3.5	20	30.1	-
			2) 3.46	20	30.3	-
152	58	20	1) 3.5	-	29.8	-
			2) 3.33	-	30.3	-
153	33	20	1) 2.16	15	30.0	-
			2) 2.72	15	30.6	-
161	30	30	4.98	-	49.6	-
172	20	50	2.10	-	31.1	-
181	70	20	3.46	-	30.5	-
182	90	5	5.58	29	26.1	-

The results for run 161 for an AC1 suspension produced following a massive batch dump of spent caustic etch indicated the excellent dewatering properties of this segregated-neutralization-like suspension.

Data in Tables 28 and 29 are for a series of runs conducted on blends of AC3 and AS7 suspensions to determine the impact of segregated neutralization solids on dewatering of conventional suspensions. Despite broad variations in solids loadings and time of filtration, it was apparent that

78

TABLE 28. CHARACTERISTICS OF CLARIFIER UNDERFLOW (AC3) AND BLENDS
WITH A SEGREGATED NEUTRALIZATION (AS7) SUSPENSION FOR
VARIABLE-VOLUME, PRESSURE FILTRATION

Run	Blend % by Volume		Date	Temp.	pH	Suspended Solids		Specific Resistance Evaluation		CST
	AS7	AC3		°C		Total g/L	Fixed g/L	r Tm/kg	C_k %	s
172	0	100	343	13	8.2	39.9	28.8	3.6	11.7	139
175	5	95	343/346	12	8.7	47.0	-	3.9	10.8	172
176	15	85	343/346	13	9.0	59.9	44.6	2.5	21.9	186
179	30	70	343/346	10	-	78.0	58.0	2.5	28.9	254

TABLE 29. RESULTS FOR VARIABLE-VOLUME, PRESSURE FILTRATION
OF AC3 AND BLENDS OF AC3 AND AS7 SUSPENSIONS

Run	Blend % by Volume		Time of Filtration, min		Dewatered Cake(s)			
	AS7	AC3	Filter	Squeeze	Mass kg	Thickness mm	Solids Content Total	Fixed
172	0	100	20	50	2.10	16	31.1	-
175	5	95	58	18	3.56	-	32.1	-
176	15	85	70	20	3.96	20	37.6	-
179	30	70	60	20	3.20	18	42.9	-

segregated neutralization enhanced the extent to which a suspension could be
dewatered. Comparison with data in Table 18 for high-pressure fixed-volume
filtration of other aliquots of the blended suspensions indicated that
diaphragm filtration produced cakes with significantly higher solids
contents (e.g., for blends of 5, 15 and 30 percent, high-pressure filtration
gave solids contents of 26.6, 33.1 and 34.8 percent, while diaphragm
filtration gave values of 32.1, 37.6 and 42.9 percent, respectively).
Diaphragm filtration therefore produced superior dewatering performance when
compared to fixed-volume pressure filtration.

SUMMARY ANALYSIS OF PRESSURE FILTRATION

The primary objective for investigation of pressure filtration systems
was to establish the extent to which aluminum-finishing sludges could be
dewatered in preparation for acidic aluminum extraction. To produce a
commercial-strength product of aluminum sulfate $[Al_2(SO_4)_3]$ in the liquid

form, i.e., "liquid alum", previous studies indicated that a dewatered-cake solids content of >21 percent was required (Saunders et al., 1984).

Fixed-Volume Pressure Filtration

The pressure-filtration data collected during fixed-volume filtration generally indicated that it was possible, in many instances, to achieve a cake solids content of >21 percent. However, the filtration studies were infrequently run to completion and, in many series, replicate runs were terminated prematurely to determine the extent of dewatering during a run. It was therefore necessary to examine the data and predict, if possible, performance of the filtration runs as they would be conducted in practice.

Numerous investigators have indicated that pressure filtration for dewatering proceeds through three phases. In a companion study, Johns (1987) indicated that during the final consolidation phase of a pressure filter run, the rate of filtration became linear with the time of filtration. Johns (1987) furthermore indicated that the ultimate volume of filtrate to be produced on a given run could be predicted in accordance with such a plot. To illustrate this point, filtration rate data for several runs on the Netzsch press are presented in Figures 34-37. As indicated in Figures 34 and 35, replicate runs (e.g., runs 24-26 and 27-29 in Figure 34) were highly reproducible. Using the terminal linear portion of each curve, or sets of replicate curves, the ultimate volume of filtrate was estimated. For example, the ultimate filtrate volume for runs 24-26 was equal to 0.052 m^3/m^2 of filtration area or a total filtrate volume of 36 L for the 2-chamber run. Using procedures developed by Johns (1987) in a companion study, the density of the dry aluminum-finishing solids was estimated to be 3200 kg/m^3. Using these data and the projected volumes of filtrate, the maximum values for the solids content, C_k, of the dewatered cakes were estimated using the following equation (Johns, 1987).

$$C_k = \frac{V_o C_o}{V_c \rho + (C_o V_o (\rho_s - \rho)/\rho_s)} \qquad (2)$$

where V_o = Volume of influent suspension processed
C_o = Suspended solids concentration of influent suspension
V_c = Total volume of press chambers
ρ = Density of filtrate
ρ_s = Density of dry sludge solids.

In addition, since filter presses are not typically run to produce the projected ultimate volume of filtrate, the solids content at the point of production of 90 percent of the ultimate filtrate volume was estimated.

Data for Plant A suspensions are presented in Table 30. It was projected that high-pressure filtration of AC3 suspensions could produce dewatered cakes with solids contents of from 26.5 to 34.9 percent. The data indicated that solids content generally increased with influent suspended solids concentration. However, three of the data sets (i.e., 36.3 g/L, 72.8
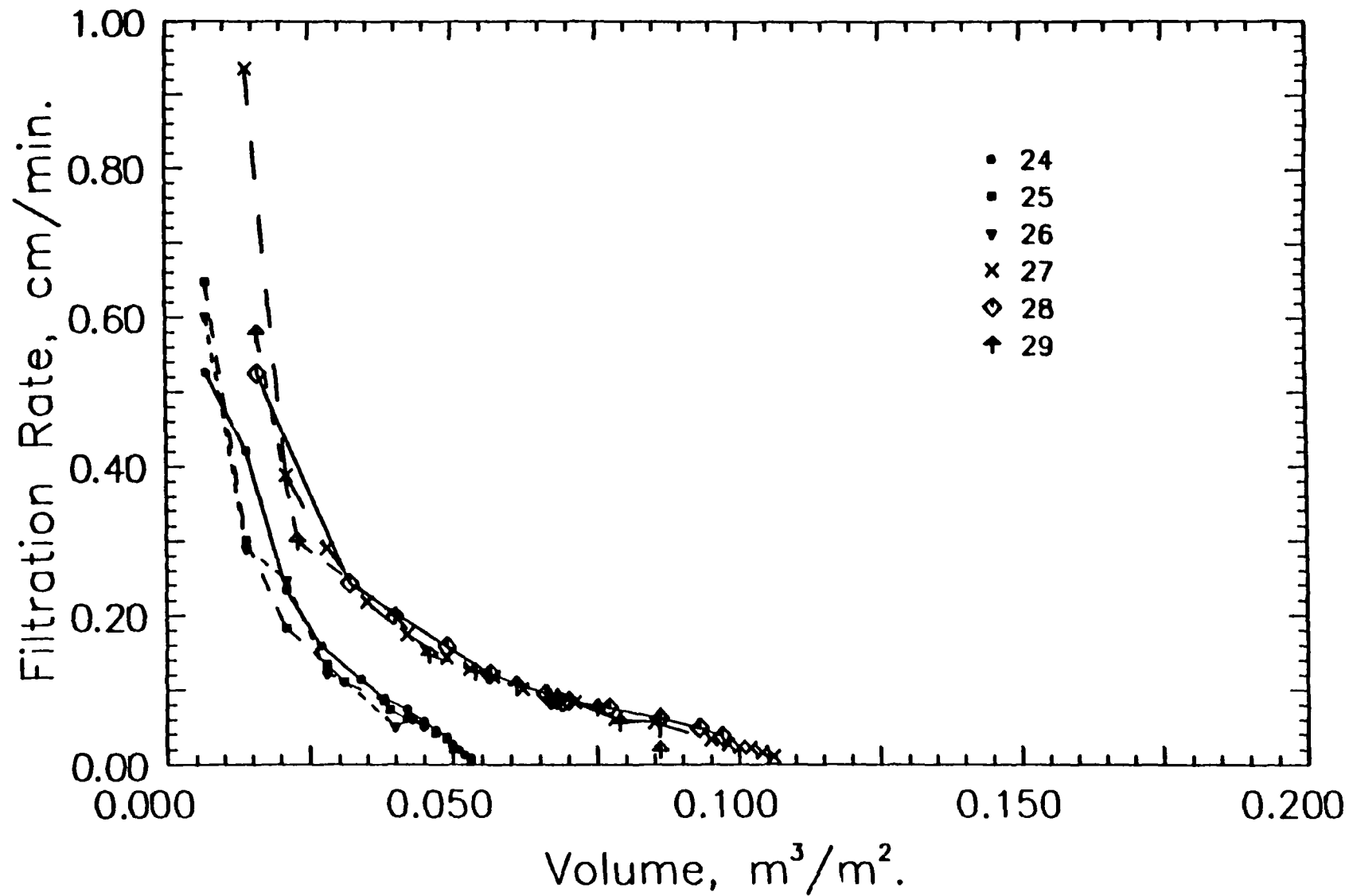
Figure 34. Filtration rate data for series-20 runs with AC3 suspensions for projection of ultimate filtrate volume.

81
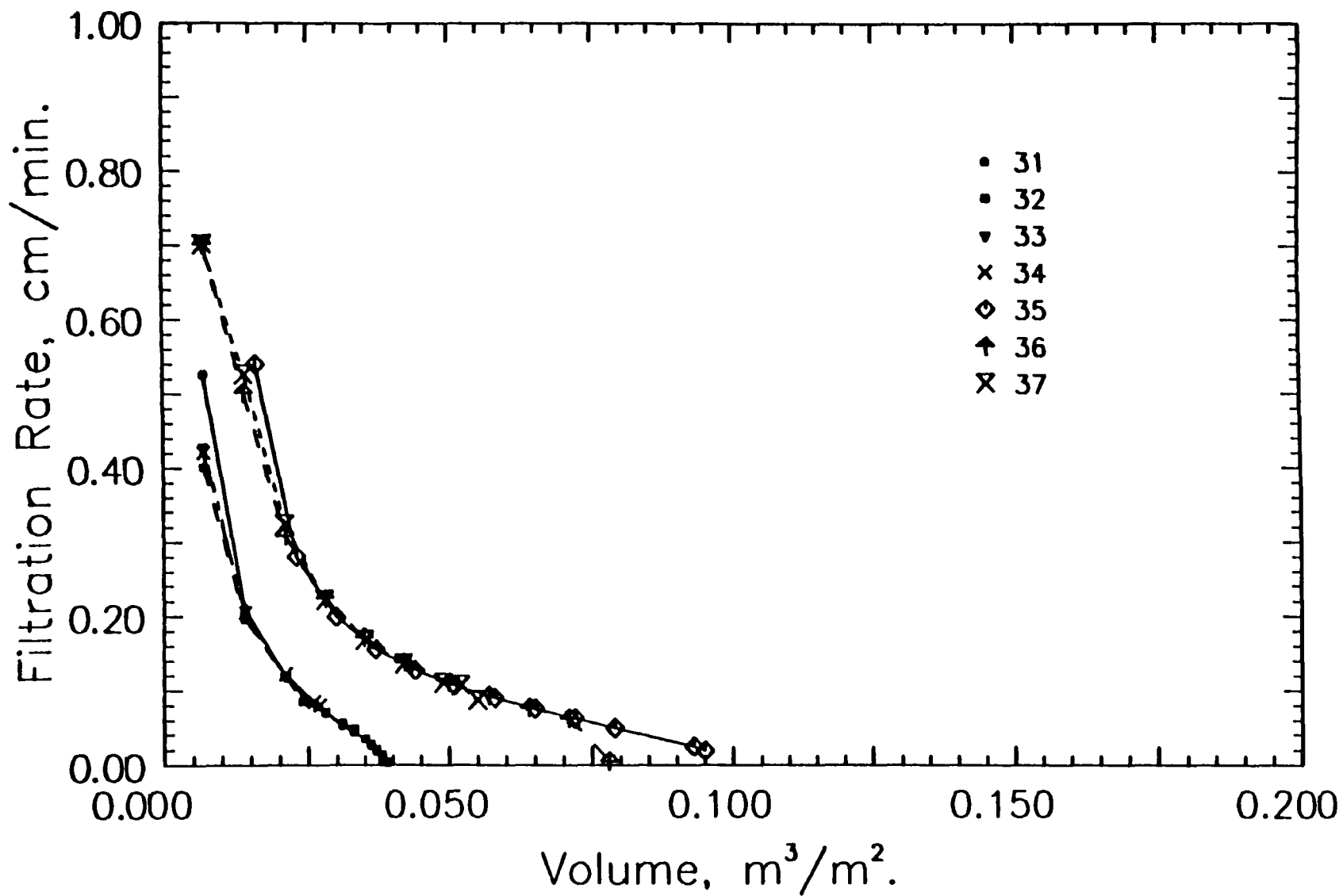
Figure 35. Filtration rate data for series-30 runs with AC3 suspensions for projection of ultimate filtrate volume.
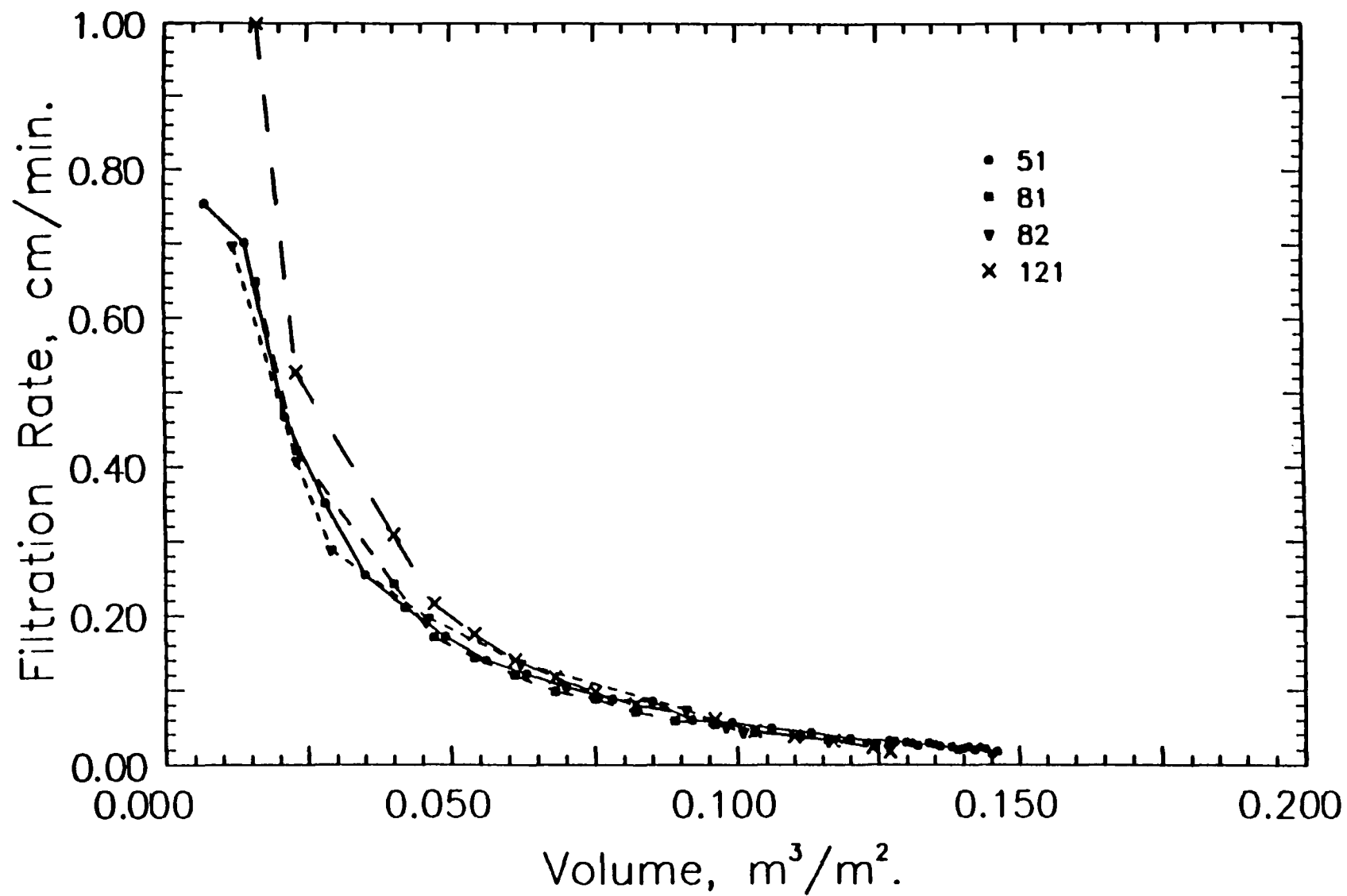
Figure 36. Filtration rate data for selected runs with XC3 suspensions for projection of ultimate filtrate volume.
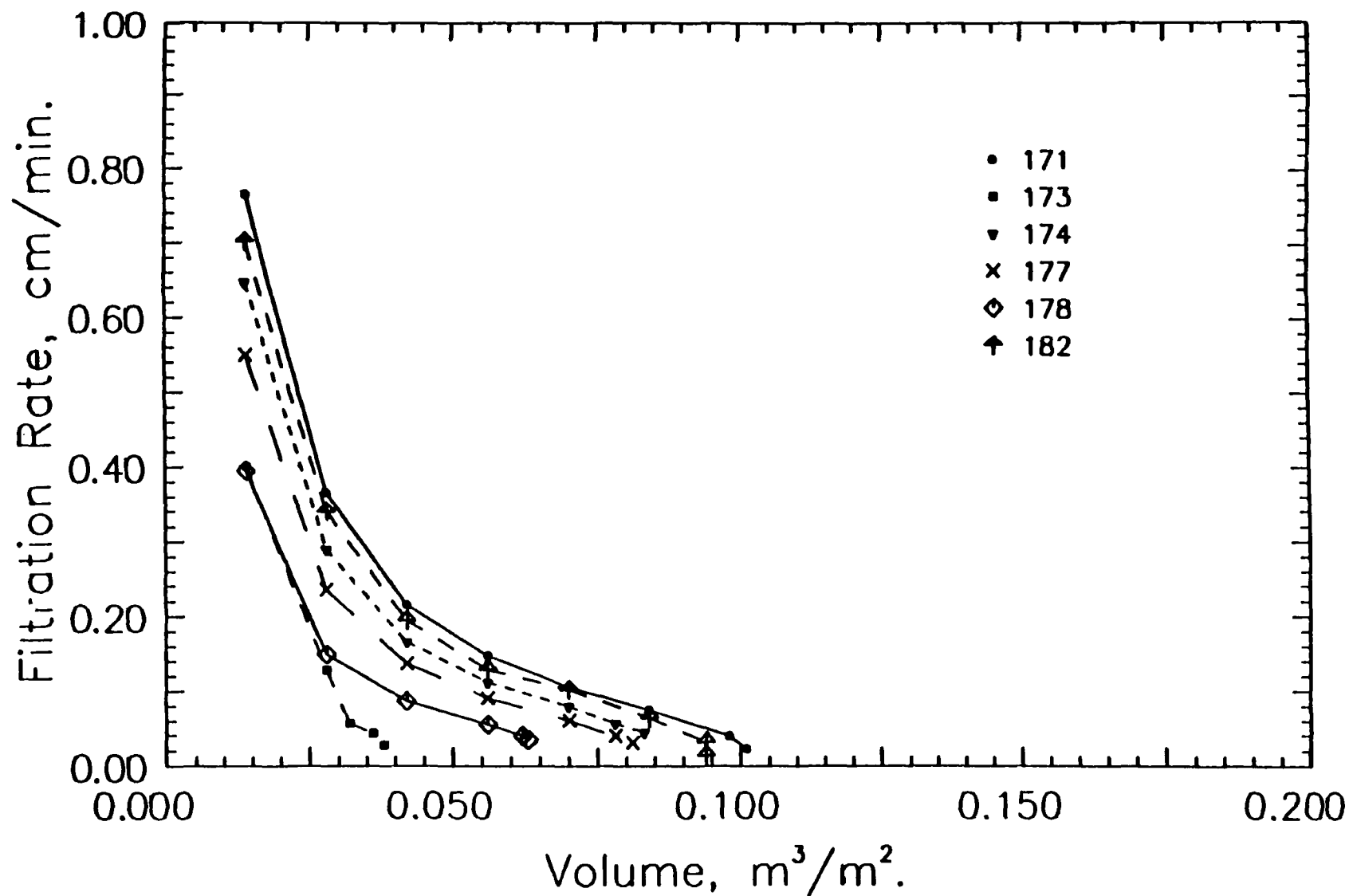
Figure 37.   Filtration rate data for selected AC3- and AS7-suspensions and blends of each for projection of ultimate filtrate volume.

TABLE 30. PREDICTED SOLIDS CONTENTS OF DEWATERED CAKES FOR SUSPENSIONS
FROM PLANT A DURING HIGH- AND LOW-PRESSURE FILTRATION

Average Suspended Solids g/L	Predicted Cake Solids Content		Number of Runs Included in Analysis (run numbers)
	Ultimate %	at 90% Completion %	
High-Pressure Filtration			
20.6	27	24.9	1 (71)
30.9	27.2	25.2	1 (16)
36.3[a]	28.4	25.6	3 (27-29)
56.9	26.5	24.7	4 (6-9)
72.8[a]	29.0	26.1	3 (24-26)
93.0[a]	34.9	32.7	3 (21-23)
104.0[b]	45	42.4	3 (18-20)
Low-Pressure Filtration			
16.9	24.8	22.7	1 (102)
20.8	24.6	22.6	1 (72)
32.5[a]	23.8	21.4	3 (35-37)
54.0	20.7	19.3	4 (10-13)
63.9[a]	21.8	19.6	4 (31-34)

[a]AC3 suspensions collected from clarifier underflow during period
(Days 303-314) in which etch dump of Day 303 affected sludge
properties (see Table 1)
[b]Suspension collected directly from neutralization basin during
etch dump on Day 303 (see Table 1)

g/L and 93 g/L) were for AC3 suspensions which were influenced by an etch
dump (see Table 1). Therefore, ignoring these data, high-pressure
filtration of typical AC3 suspensions should produce an ultimate dewatered
cake solids content of approximately 27 percent and, at 90 percent
completion of a filtration run (i.e., collection of 90 percent of projected
ultimate filtrate volume), a solids content of approximately 25 percent.
Therefore, high-pressure filtration should be adequate for producing a
dewatered cake suitable for production of liquid alum.

The influence of the etch dump of Day 303 on sludge dewatering
properties is apparent from the data for a suspended solids concentration of
104 g/L. This AC1 suspension was collected directly from the neutralization
basin during the etch dump and was not polymer conditioned. The ultimate
solids content of 45 percent was 1.67-fold higher than that typical of AC3
suspensions and would result in a 40 percent reduction in volume of wet
sludge for disposal.

The ultimate solids content projected for low-pressure filtration of AC3 suspensions ranged from 20.7 to 24.8 percent. Again, ignoring those data influenced by the etch dump, low-pressure filtration would produce a sludge with an ultimate solids content of approximately 23.4 percent and, at 90 percent completion of a filter-press run, a value of 21.5 percent. Therefore, low-pressure filtration would theoretically be suitable for use in conjunction with alum production but has no margin for variation in solids content if a minimum solids content of 21 percent is required.

Data for the single, XC3 suspension from Plant X are presented in Table 31. The data for high-pressure filtration were slightly lower than those for AC3 suspensions, i.e., ultimate values of 27 percent (AC3) vs 24.5 percent (XC3). Low-pressure filtration was similarly lower than that for AC3, i.e., 23.5 percent (AC3) vs 17.6 percent (XC3). Therefore, the XC3 suspension could not be as effectively dewatered as could the AC3 suspension and only high-pressure filtration could be used with an alum production system.

TABLE 31. PREDICTED SOLIDS CONTENTS OF DEWATERED CAKES FOR XC3 SUSPENSIONS DURING HIGH- AND LOW-PRESSURE FILTRATION

Average Suspended Solids g/L	Predicted Cake Solids Content		Number of Runs Included in Analysis (run numbers)
	Ultimate %	at 90% Completion %	
High-Pressure Filtration			
20.7	24.5	22.5	4 (51, 81, 82, 121)
Low-Pressure Filtration			
20.7	17.6	16.2	3 (52, 73, 74)

To indicate the impact of implementation of segregated neutralization on overall sludge dewatering performance, data for the 170-series (i.e., runs 171, 173, 174, 177 and 178) are presented in Table 32. First, the conventional-neutralization suspension examined in run 171 had excellent dewatering properties, as compared to those for other AC3 suspensions, with a projected ultimate solids content of 28.7 percent. As the volumetric percentage of segregated-neutralization suspension increased, the ultimate solids content increased from 33.0 to 39.2 percent, with a projected ultimate value of 51.3 percent for the neat segregated-neutralization suspension. Therefore, segregated-neutralization solids have a dramatic impact on the ultimate volume of sludge produced and can enhance the potential of dewatering systems with respect to alum production.

TABLE 32. PREDICTED SOLIDS CONTENTS OF DEWATERED CAKES FOR AN AC3
SUSPENSION AND BLENDS OF AC3 AND AS7 SUSPENSIONS DURING
HIGH-PRESSURE FILTRATION

Suspended Solids g/L	Portion as AS7 Suspension by Volume %	Predicted Cake Solids Content	
		Ultimate %	at 90% Completion %
37.9	0	28.7	26.6
47.0	5	33.0	30.7
59.9	15	36.6	34.2
78.0	30	39.2	36.7
180.1	100	51.3	48.6

Variable-Volume Pressure Filtration

The limited studies conducted with the diaphragm press indicated
excellent performance in dewatering all aluminum-finishing sludges. For AC3
suspensions, the solids content of dewatered cakes, following removal of all
filtrate at a squeeze pressure of 15.5 bar, ranged from 25.4 percent to 31.2
percent and averaged 29.4 (see Table 27). This was slightly higher than the
average value of 27 percent achieved with high-pressure (14-15 bar)
filtration of AC3 suspensions, indicating the limited utility of the more
complex diaphragm press over a high-pressure, fixed-volume press.

With blends of conventional (AC3) and segregated (AS7) neutralization
suspensions, the diaphragm press produced sludges with solids contents
ranging from 31.1 percent (5 percent by volume of AS7) to 42.9 percent (30
percent by volume of AS7), while the ultimate solids contents projected for
high-pressure filtration were 33 and 39.2 percent, respectively (see Tables
29 and 30). The diaphragm press, therefore, may have potential for
application when overall system design is examined. However, based on
potential for production of a high-solids-content sludge it appears to have
only minimal benefit.

SECTION 6

LIQUID ALUM PRODUCTION FROM DEWATERED SLUDGE SOLIDS

Aluminum-finishing plants produce numerous wastewater residues in conjunction with the etching, anodizing and associated surface-treatment processes used to apply finishes to extruded aluminum products. These residues are produced in several distinctively different ways; three of the most common residues were examined herein.

Gelatinous aluminum hydroxide suspensions are conventionally produced by neutralization of dilute plant rinsewaters at ambient temperatures in a treatment facility. The pH of rinsewaters are typically adjusted with spent caustic etch and finishing-acid suspensions. These conventional neutralization (CN) sludges are produced at virtually all aluminum finishing plants.

An innovative treatment process developed by Saunders et al. (1984) to reduce sludge volumes is referred to as segregated neutralization. Spent caustic etch is neutralized with spent finishing acid to an alkaline pH value (e.g., pH = 8-10) at elevated temperatures producing crystalline, aluminum-hydroxide precipitates such as pseudoboehmite. These crystalline, segregated-neutralization (SN) solids can be dewatered separately or combined with conventional neutralization suspensions and collected as a mixture.

Finally, the introduction of etch recovery systems by Alcoa, Inc. and Fugi Sash, Ltd. has resulted in a third type of sludge residue. Etch recovery solids are produced at elevated temperatures (e.g., 60°C) following seeding of caustic-etch solutions with aluminum trihydrate crystals. The etch recovery (ER), or aluminum-trihydrate, suspension produced is periodically removed from the crystallizer and dewatered. The sludge suspension can be mechanically dewatered to solids contents in excess of 90 percent.

Conventional-neutralization, segregated-neutralization and etch-recovery sludges were examined separately and in mixtures as indicated below. The conventional neutralization sludges were obtained from clarifier under-flow (i.e., AC3) at Plant A and were dewatered at low-pressure (6-7 bar) on the JWI press, due to the unavailability of the high-pressure Netzsch press during this portion of the study. Segregated neutralization suspensions were produced by batch neutralization of caustic etch solutions from Plant A with conventional anodizing acid and dewatered on the JWI press. Etch recovery solids were provided by personnel from Plant A through a companion aluminum-finishing facility. The identification codes used for these dewatered sludges in subsequent tables and figures are: CN = conventional neutralization sludge; SN = segregated neutralization sludge; and ER = etch

recovery sludge. In addition to each individual sludge, two blends were examined, i.e., CN/SN and CN/ER. A blend of segregated neutralization (SN) and etch recovery (ER) sludges was not examined since these sludges are produced in processes which are focused on treatment of spent caustic etch and are not mutually compatible.

ACIDIC EXTRACTION OF ALUMINUM FROM DEWATERED SLUDGE CAKES

Acidic extraction of dewatered sludge samples was accomplished in a covered batch reactor with a temperature-control system. Sulfuric acid, water and sludge additions were carefully controlled and the extraction mixtures were closely monitored throughout the extractions. The results from all extraction runs completed are presented below.

Conventional Neutralization Sludge Cakes (CN)

Two separate samples of dewatered conventional-neutralization sludge cakes were examined. The characteristics of these cakes are presented in Table 33. The solids contents of the two cakes were well below 21 percent but were typical of those produced by low-pressure filtration of conventional neutralization sludges (see Table 8). The fixed (550°C) solids contents were 73 and 80 percent of the dry (103°C) solids and the aluminum contents were 35.6 and 39.2 percent (on a fixed-solids basis). By comparison, aluminum hydroxide precipitates represented by the formula Al(OH)$_3$ theoretically contain 34.6 percent aluminum. The dewatered cakes are, therefore, rich in aluminum and the fixed solids were similar in aluminum content to Al(OH)$_3$. The 20 to 27 percent of dry solids lost following ashing at 550°C was attributed to moisture retained by the gelatinous aluminum precipitates; was not indicative of volatile organic matter; and was typical of aluminum-finishing sludges examined previously by Saunders et al. (1982, 1984).

TABLE 33. CHARACTERISTICS OF DEWATERED CONVENTIONAL NEUTRALIZATION SLUDGE CAKES CN-1 AND CN-2

Parameter	Sludge Cake	
	CN-1	CN-2
Dry (103°C) Solids Content, g dry solids/100 g wet cake......	18.1	17.4
Fixed (550°C) Solids Content, g fixed solids/100 g wet cake...	13.3	13.9
Aluminum Content, g Al/100 g fixed solids.....................	35.6	39.2

A total of three extractions, or runs, were made with the CN-1 sludge cake and two runs were made with the CN-2 cake. The initial experimental conditions employed with cake CN-1 are presented in Table 34. Between 0.54 and 0.74 kg of wet sludge was employed in the runs. The dose of acid added to each run was based on the fixed solids at the rate of 1.89 g H$_2$SO$_4$/g fixed solids

TABLE 34. INITIAL EXPERIMENTAL CONDITIONS FOR EXTRACTIONS
OF CONVENTIONAL NEUTRALIZATION SLUDGE CAKE CN-1

Parameter	Run		
	1-1	1-2	1-3
SLUDGE ADDITION			
Total Mass, g	709.5	541.0	744.5
Fixed Solids, g	94.4	56.8	98.7
Moisture, g	615.1	484.2	645.8
SULFURIC ACID			
Sulfuric Acid Addition, g	163.5	107.1	186.1
Percent of Stoichiometric Acid Dosage, Percent	89.2	97.2	97.2
CONTROL TEMPERATURE, °C	90	70	50
ESTIMATED PRODUCT CONCENTRATION, Percent as Al_2O_3	7.3	5.9	7.1

(see equation (1) in Section 4). After conducting these initial runs,
aluminum-content values were obtained and the acid dose was then evaluated
relative to the stoichiometric requirements for acid (i.e., an acid dose of
5.44 g H_2SO_4/g Al). Based on these post-experiment measurements, the
percent of the stoichiometric acid dosage was calculated. As indicated in
Table 34, the sludges were dosed with 89 to 97 percent of the stoichiometric
quantity of acid, indicating a slight acid deficiency. The nominal control
temperatures for the reactors ranged from 50°C to 90°C. Furthermore, the
estimated product concentrations, based on the fixed solids and acid
quantities employed, ranged from 5.9 to 7.3 percent as Al_2O_3. These values
were well below the desired concentration of 8 percent as Al_2O_3 and were
reflective of the low solids content of the sludge cakes employed. Although
this could not be improved upon with the available low-pressure press, the
purpose was to monitor the kinetics of the extraction and develop a material
balance for it. In this light, the low solids content (i.e., high moisture
content) was not viewed as a deterrent to the study.

As indicated in Table 35, two runs were conducted with cake CN-2 at
nominal control temperatures of 90°C (run 2-1) and 50°C (run 2-2) to confirm
results obtained with cake CN-1 and to conduct runs at 100-percent,
stoichiometric-acid doses. As indicated in Table 33, the aluminum content
of cake CN-2 was higher than that for cake CN-1, allowing for runs with
estimated product concentrations of 7.9 to 8.0 percent as Al_2O_3. Further-
more, the acid doses used for runs 2-1 and 2-2 were based on aluminum
content and not on fixed solids, allowing for the 100-percent, stoichio-
metric-acid dose.

TABLE 35. INITIAL EXPERIMENTAL CONDITIONS FOR EXTRACTIONS
OF CONVENTIONAL NEUTRALIZATION SLUDGE CAKE CN-2

Parameter	Run 2-1	Run 2-2
SLUDGE ADDITION		
Total Mass, g	611.5	641.5
Fixed Solids, g	84.9	89.4
Moisture, g	526.6	552.1
SULFURIC ACID		
Sulfuric Acid Addition, g	181.4	191.0
Percent of Stoichiometric Acid Dosage, Percent	100.0	100.0
CONTROL TEMPERATURE, °C	90	50
ESTIMATED PRODUCT CONCENTRATION		
Percent as Al_2O_3	7.9	8.0

As indicated in Tables 34 and 35, the control temperatures (i.e., the desired reaction temperatures) for the runs with sludge cakes CN-1 and CN-2 were either 50°C, 70°C or 90°C. These temperatures were to be maintained throughout each run with the exception of the initial reaction period in which sulfuric acid was added to dewatered sludge cakes. During this initial period, temperature could not in all cases be controlled due to the highly exothermic reaction resulting from the addition of sulfuric acid to the moist sludge cakes. Data presented in Figures 38 and 39 indicate the temporal variation in temperature for runs with sludge cakes CN-1 and CN-2, respectively. Although not presented in the figures, the sludge cakes were at ambient temperature (e.g., 25°C ± 5°C) prior to acid addition. As shown in Figure 38, initial reaction temperatures for runs 1-1 and 1-3 were between 83 and 85°C, while run 1-2 was at 66°C. The temperature of runs 1-1 and 1-2 remained near the initial values, while run 1-3 cooled to the desired control temperature of 50°C over a 1.1-h period before the temperature control system was activated.

As indicated in Table 36 for the time interval over which temperature could be controlled at the desired level, average temperatures were 89°C, 71°C and 51°C for runs 1-1, 1-2 and 1-3, respectively. Within the limits of one standard deviation of the average value, the desired control temperatures were achieved. Data in Figure 39 for runs 2-1 and 2-2 indicate that both runs were initially at temperatures of 89 to 90°C. Run 2-1 was effectively controlled near 90°C throughout the run, while run 2-2 cooled to and was controlled at near 50°C after a 1-h period. The average temperatures for the time period of control were 88°C and 52°C, respectively, for runs 2-1 and 2-2, as shown in Table 36. In addition, the desired control temperature was within the limits of one standard deviation
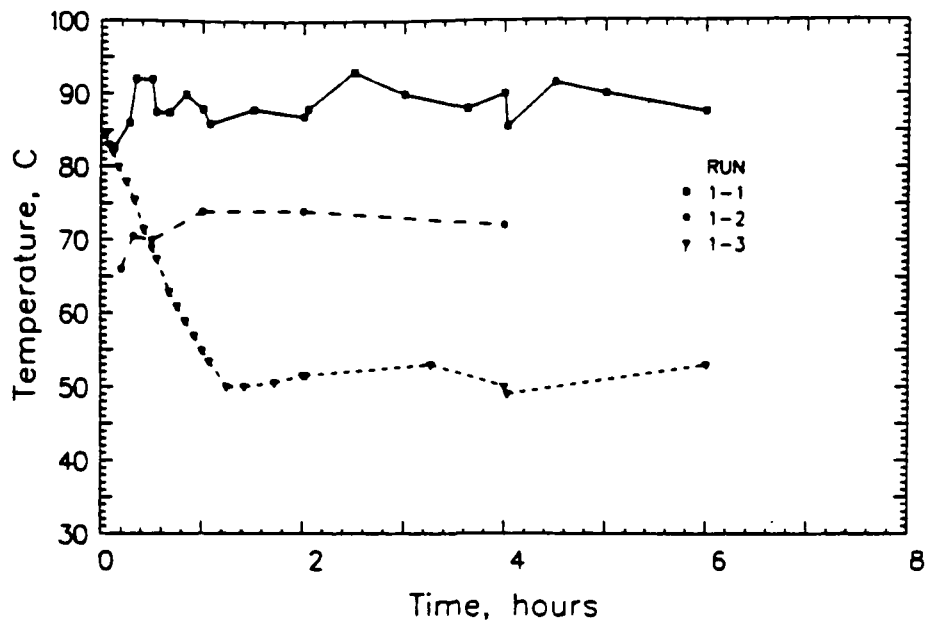
Figure 38. Temperature of reactor contents during acidic extraction of sludge cake CN-1 in runs 1-1 through 1-3.
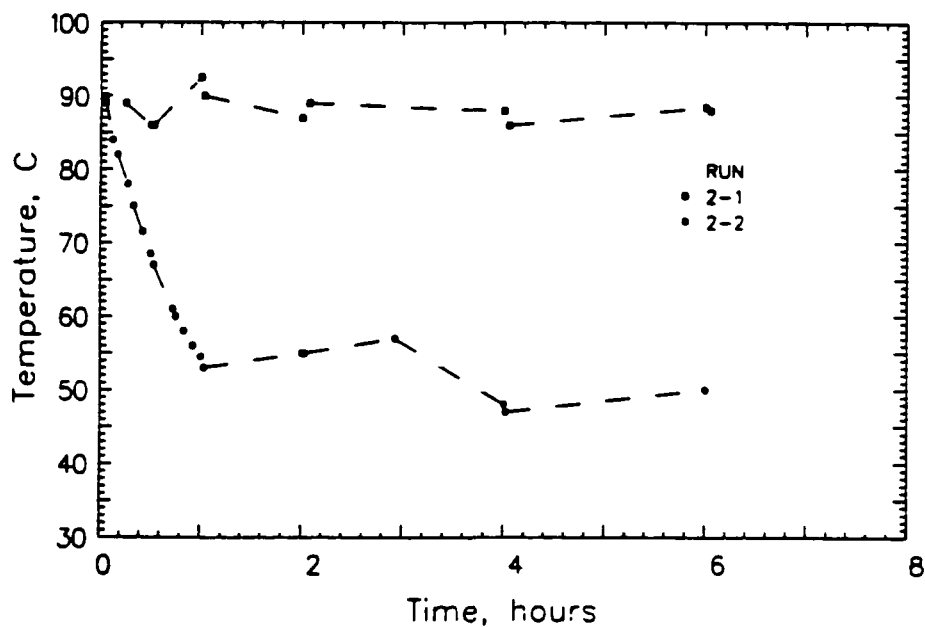
Figure 39. Temperature of reactor contents during acidic extraction of sludge cake CN-2 in runs 2-1 and 2-2.

TABLE 36. ACTUAL AND CONTROL TEMPERATURES FOR EXTRACTIONS
OF CONVENTIONAL NEUTRALIZATION SLUDGE CAKES
CN-1 AND CN-2

Parameter	CN-1 Run			CN-2 Run	
	1-1	1-2	1-3	2-1	2-2
TIME INTERVAL, hours.......	0.13-6	0.2-6	1.1-6	0.1-6	1-6
CONTROL TEMPERATURE,[a] °C...	90	70	50	90	50
ACTUAL TEMPERATURE[b]					
Average, °C..............	89	71	51	88	52
Standard Deviation, °C...	2.7	3.0	1.6	1.9	3.7

[a]Desired temperature selected for temperature-control system.
[b]Temperature data for indicated time interval.

of the actual average temperature for runs 2-1 and 2-2. For this primary reason, desired control temperatures will be used in all subsequent runs to indicate reactor temperatures and will also be used for runs with sludges CN-1 and CN-2.

The aluminum concentration of extract filtrates (filter pore diameter = 0.45 μm) was used to monitor the progress of each run, as presented for CN-1 and CN-2 cakes in Figures 40 and 41. In each of the extraction runs, the initial sample was collected after approximately 0.5 h. This interval was selected since it was virtually impossible to collect a representative sample earlier because of the initial time required to produce a uniformly mixed slurry from the dewatered-cake and acid mixture. However, the extraction of aluminum from cakes CN-1 and CN-2 was, in all instances, near completion after 0.5 h. Over the remaining period of 5.5 h, filtrate aluminum concentration increased gradually, further indicating the rapid rate and extent of the initial reaction.

The effects of temperature on the extractions were relatively minor. The runs at control temperatures of 90°C, however, resulted in the highest rates of extraction although those for control temperatures of 50°C and 70°C were only slightly lower. This apparent insensitivity to the effects of temperature was attributable, in part, to the initial robust reaction which occurred upon the addition of acid. As shown in Figures 38 and 39, initial reaction temperatures actually measured ranged from 82°C to 90°C, with the exception of 66°C for run 1-2. Localized temperatures at the point of acid addition presumedly exceeded these values. Furthermore, reaction temperatures after 0.5 h ranged from a low of 69°C (runs 1-2, 1-3 and 2-2) to 92°C (run 1-1), indicating that during the period of initial reaction, temperatures remained relatively high. Therefore, since filtrate aluminum concentrations, as shown in Figures 40 and 41, indicated the extractions were at or near completion within the initial 0.5 h, temperatures at or

93
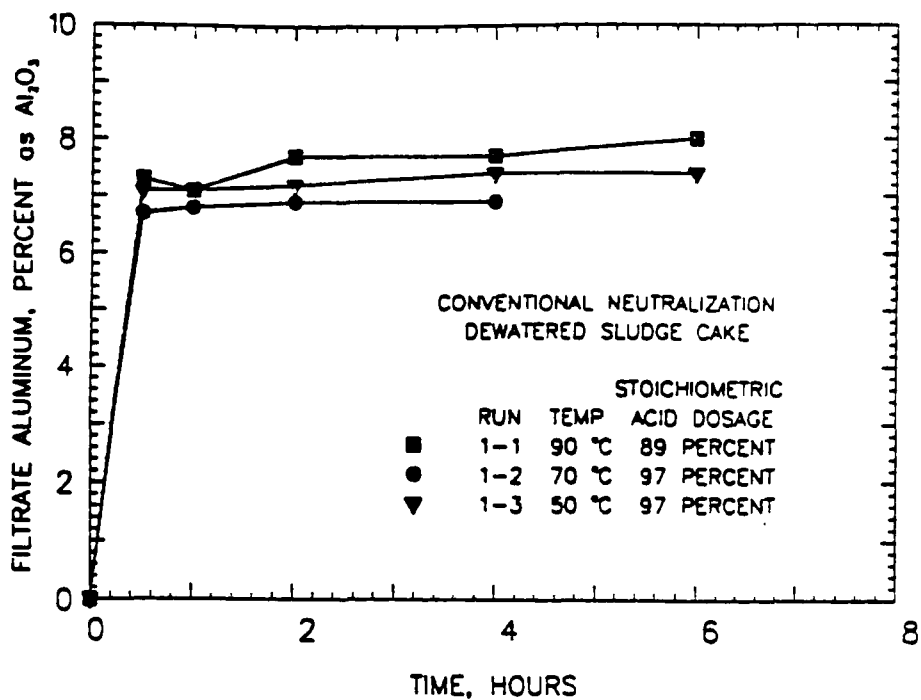
Figure 40. Filtrate aluminum concentration for sulfuric-acid extractions of sludge cake CN-1 during runs 1-1, 1-2 and 1-3.
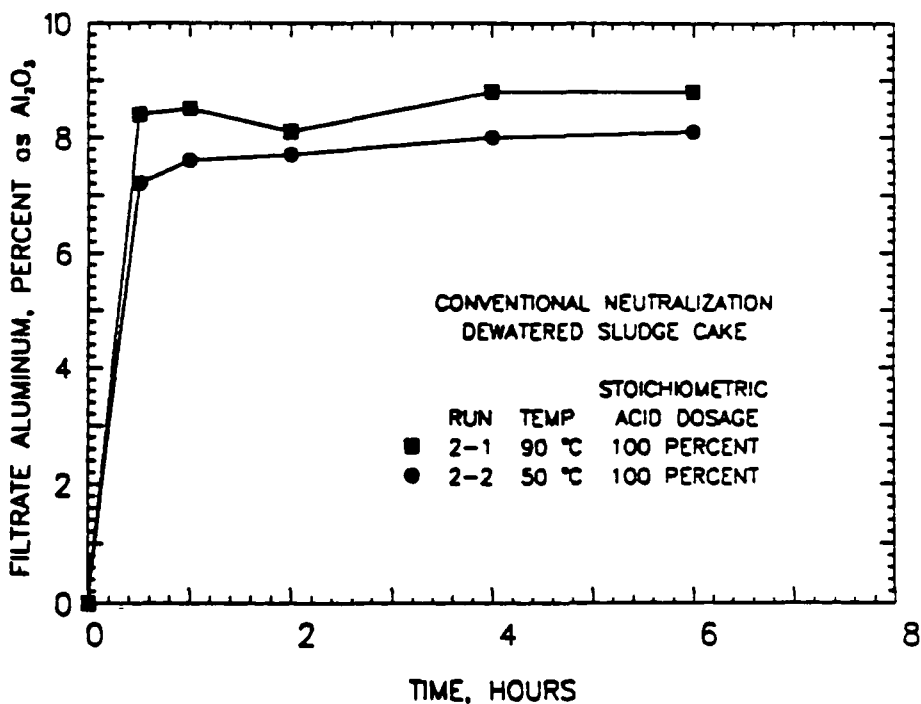
Figure 41. Filtrate aluminum concentration for sulfuric-acid extractions of sludge cake CN-2 during runs 2-1 and 2-2.

above 70°C were apparently sufficient to assure completion of the extraction. As shown in Figures 38 through 41, this was valid for runs 1-3 and 2-2 which were set at control temperatures of 50°C, although this temperature was not achieved until approximately 1.0 to 1.2 h into the extraction runs. Therefore, the effect of reaction temperature within the limits of the experimental conditions used were minimal due, in part, to the lack of temperature control in the initial reaction period. It is therefore possible that temperature could play a significant role in the extraction if, for example, the environmental conditions associated with the reaction vessel suppressed reaction temperature throughout an extraction. While this may be possible under some conditions, it is not expected to occur within the context of the environmental conditions associated with typical industrial process and wastewater-treatment facilities. Furthermore, in a larger batch reactor system employed in an industrial plant with a lower surface-area-to-volume ratio, less heat of reaction would be lost (compared to that for the laboratory beaker) and elevated reaction temperatures would be expected for a longer time period than that indicated in Figures 38 and 39.

Material balances for total mass of the reactor contents and aluminum mass for all runs with cakes CN-1 and CN-2 are included in Table 37. During

TABLE 37. MATERIAL BALANCES ON TOTAL MASS AND ALUMINUM FOR EXTRACTIONS OF CONVENTIONAL NEUTRALIZATION SLUDGE CAKES CN-1 AND CN-2

Parameter	Run			Run	
	1-1	1-2	1-3	2-1	2-2
TOTAL MASS					
Input:					
Fixed Solids, g...	94.4	56.8	98.7	84.9	89.4
Moisture, g.......	615.1	484.2	645.8	526.6	552.1
Acid, g..........	163.5	107.1	186.1	181.4	191.0
Total, g.........	873.0	648.1	930.6	792.9	832.5
Output, g...........	837.0	522.5	913.3	667.2	814.4
Percent Recovered.....	96	81	98	84	98
ALUMINUM					
Input, g.............	33.6	20.2	35.1	33.3	35.1
Output:					
Soluble, g........	33.8	19.5	35.3	-	33.7
Residual, g.......	1.0	1.5	1.4	-	1.0
Total, g..........	34.8	21.0	36.7	-	34.7
Percent Recovered.....	104	104	104	-	99

each run, mixed aliquots of reactor contents were withdrawn at 0.5- to 1-h intervals. The mass and aluminum content of these aliquots were determined and accumulated with similar values for the volume remaining at the end of the run. The data for runs 1-1, 1-3, and 2-2 indicated that the material balance on total mass closed (i.e., output mass was equal to 96-98 percent of input mass). Material balances for runs 1-2 and 2-1 were not as complete, as indicated by recoveries of 81 to 84 percent. A major portion of the mass loss could be attributed to evaporation. Although the reactor was covered, it was not permanently sealed, allowing for the loss of water at the elevated temperatures employed. With respect to an aluminum balance, within the typical limits of detection for aluminum analyses, the aluminum recoveries were excellent, ranging from 99 to 104 percent.

The extent of the extractions are graphically indicated in Figures 40 and 41. To more precisely indicate the quality of the extracted product, the characteristics of the terminal sample taken for each run are presented in Table 38. Commercial-strength, liquid alum has a nominal aluminum concentration of 8 percent as Al_2O_3. This minimal criterion was met or exceeded in runs 1-1, 2-1 and 2-2 and was approached in runs 1-2 and 1-3. Given that the moisture contents of the initial sludge cakes were noted previously as being higher than desired (i.e., desired moisture content of <79 percent) and that the estimated product concentrations ranged between 5.9 and 8.0 percent as Al_2O_3 (see Tables 34 and 35), these results were considered to be excellent and indicative of successful extractions. The data for free Al_2O_3, free acid and Congo Red measurements indicated the products were near neutral or slightly alkaline, relative to Al_2O_3 dissolution, and additional acid was required to completely extract the remaining aluminum. Given that the acid doses for runs 1-1, 1-2 and 1-3 were less than the stoichiometric dose, the results indicating excess free aluminum (i.e., free Al_2O_3 > 0; free acid = 0; and a light-brown color with the Congo Red test) were expected. Furthermore, the results for runs 2-1 and 2-2 were indicative of stoichiometric addition of acid as confirmed by the near neutral conditions, relative to Al_2O_3 dissolution, for each run.

Total iron in the extracts ranged from 0.03 to 0.09 percent as Fe_2O_3. While no standards exist for iron in commercial alum, a value of 0.2 percent as Fe_2O_3 is typical. These alum products were then of excellent quality relative to iron.

The suspended solids concentrations of 1.9 to 9.1 g/L in Table 38 were for terminal extraction samples which had not been gravity clarified and no attempt was made to remove suspended matter. Informal studies indicated that these samples could be clarified by gravity settling but this was not quantified. Comparison of the final suspended solids concentrations with projected initial values indicated 95 to 99 percent of the initial suspended matter was destroyed by acidic extraction, leaving a suspended residue of only 1 to 5 percent of the initial mass.

Acidic extraction of conventional-neutralization sludge cakes was, therefore, rapid and virtually complete within a 1- to 2-h period. Product strengths were near commercial levels and indications are that commercial

TABLE 38. CHARACTERISTICS OF LIQUID ALUM PRODUCED BY ACIDIC EXTRACTION OF CONVENTIONAL NEUTRALIZATION SLUDGE CAKES CN-1 AND CN-2

Parameter	CN-1 Run			CN-2 Run	
	1-1	1-2	1-3	2-1	2-2
Total Al_2O_3, percent.................	8.0	7.5	7.4	8.8	8.1
Free Al_2O_3, percent.................	1.2	1.0	0.5	0.1	--
Free Acid, percent....................	--	--	--	--	0.3
Total Iron, percent as Fe_2O_3.........	0.09	0.03	0.07	0.07	0.08
Suspended Solids:					
Concentration, g/L................	5.6	7.1	5.9	1.9	9.1
Percent Reduction................	97	95	97	99	95
Specific Gravity.....................	1.3	1.3	1.3	1.3	1.3
pH (2% dilution).....................	3.8	4.0	3.7	3.4	3.0
Congo Red Test, color*...............	light brown	light brown	light brown	light brown/ purple	light purple

*Qualitative acidity test: light brown = free alum; purple = neutral; blue = free acid.

97

strengths could be routinely achieved with an adequate dewatering system producing cakes with solids contents of approximately 21 percent and higher.

Segregated-Neutralization Sludge Cake (SN-1)

As indicated in Table 39, the segregated neutralization cake had a solids content of 36.8 percent following low-pressure (6-7 bar) dewatering and a fixed solids content of 29.9 percent, both well above the minimal acceptable solids content of 21 percent. The aluminum content of 31 percent on a fixed solids basis further indicated the high potential for alum production.

TABLE 39. CHARACTERISTICS OF DEWATERED SEGREGATED-NEUTRALIZATION
SLUDGE CAKE SN-1

Parameter	Sludge Cake
	SN-1
Dry (103°C) Solids Content, g dry solids/100 g wet cake......	36.8
Fixed (550°C) Solids Content, g fixed solids/100 g wet cake...	29.9
Aluminum Content, g Al/100 g fixed solids...................	31.0

Three extractions of SN-1 cakes were conducted, as indicated in Table 40, and a total of 0.3 to 0.33 kg of wet sludge solids were extracted.

TABLE 40. INITIAL EXPERIMENTAL CONDITIONS FOR EXTRACTIONS
OF SEGREGATED NEUTRALIZATION SLUDGE CAKE SN-1

Parameter	Run		
	3-1	3-2	3-3
SLUDGE ADDITION			
Total Mass, g.....................................	333.5	300.0	307.0
Fixed Solids, g.................................	99.7	89.7	107.9
Moisture, g.....................................	233.8	210.3	199.1
WATER ADDITION, g.....................................	264.5	238.0	340.0
SULFURIC ACID			
Sulfuric Acid Addition, g...........................	188.0	169.1	182.2
Percent of Stoichiometric Acid Dosage, Percent.....	111.6	111.6	100.0
CONTROL TEMPERATURE, °C...............................	90	70	90
ESTIMATED PRODUCT CONCENTRATION, Percent as Al_2O_3.....................................	7.4	7.4	7.6

Because the solids content was well above 21 percent, it was necessary for water to be added to the extraction mixture. Since liquid alum has a maximum solubility near 9 percent as Al_2O_3 and will crystalize at higher concentrations at ambient temperatures, water addition was necessary to prevent formation of a crystalline alum. The quantities of water added resulted in estimated product concentrations of 7.4 to 7.6 percent as Al_2O_3. Sulfuric acid addition was at, or slightly in excess of, the stoichiometric dose, as indicated as being necessary from previous experience.

Filtrate aluminum data in Figure 42 indicated the extraction of aluminum in runs 3-1, 3-2 and 3-3 was rapid but that extraction at 70°C (run 3-2) was slightly slower than those at 90°C, although similar levels of filtrate aluminum were ultimately achieved. An abrupt increase in aluminum between 4 and 6 hours for runs 3-2 and 3-3 was unexplained but was attributed, in part, to evaporational losses. The extractions of SN-1 solids were therefore very similar to those for CN-1 and CN-2 solids (see Figures 40 and 41), indicating that, although SN-1 solids were highly crystalline and CN solids were highly amorphous and gelatinous, acidic extraction of both types of sludge cakes proceeded rapidly to completion, producing a commercial-strength product.

Data in Table 41 indicate that 95 to 96 percent of total mass was

TABLE 41. MATERIAL BALANCES ON TOTAL MASS AND ALUMINUM FOR EXTRACTIONS OF SEGREGATED NEUTRALIZATION SLUDGE CAKE SN-1

Parameter	Run		
	3-1	3-2	3-3
TOTAL MASS			
Input:			
Fixed Solids, g.....	99.7	89.7	107.9
Moisture, g.........	498.3	448.3	539.1
Acid, g.............	188.0	169.1	182.2
Total, g...........	786.0	707.1	829.2
Output, g..............	743.8	678.9	785.2
Percent Recovered.......	95	96	95
ALUMINUM			
Input, g...............	30.9	27.8	33.5
Output:			
Soluble, g..........	31.0	28.1	35.1
Residual, g.........	0.0	0.0	0.1
Total, g...........	31.0	28.1	35.2
Percent Recovered.......	100	101	105

99
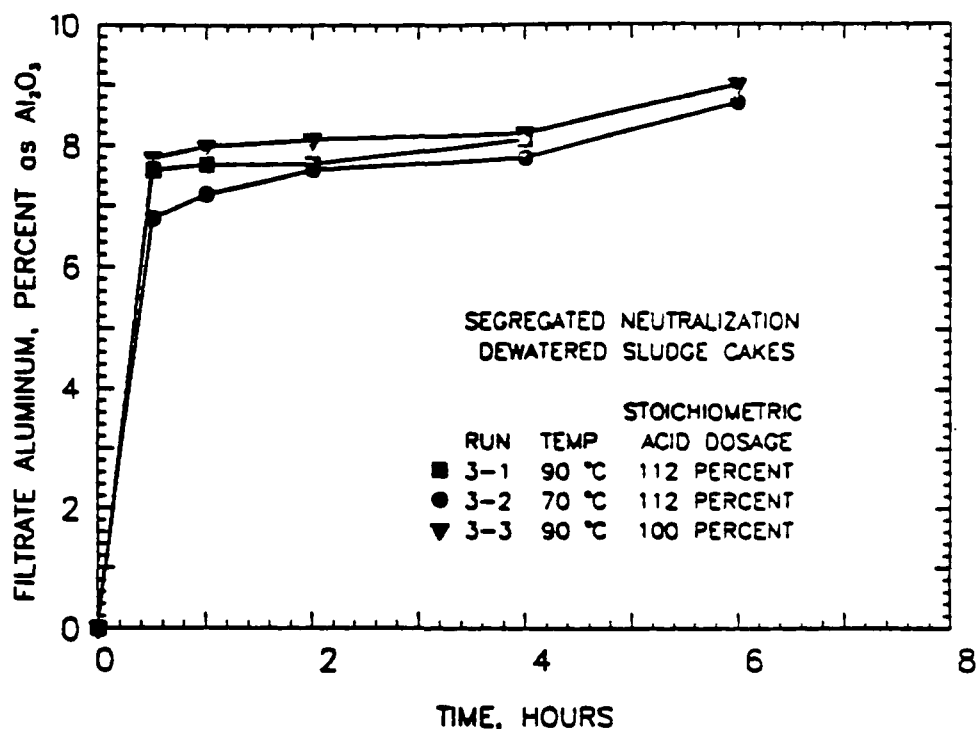
Figure 42. Filtrate aluminum concentration for sulfuric-acid extractions of sludge cake SN-1 during runs 3-1, 3-2 and 3-3.
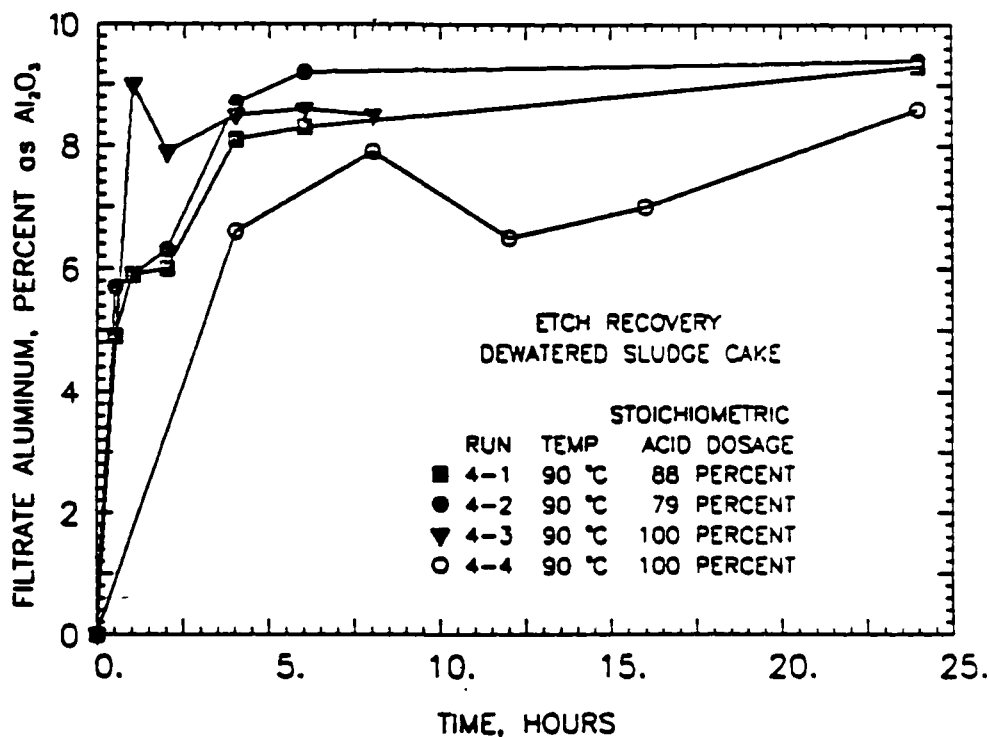
Figure 43. Filtrate aluminum concentrations for sulfuric-acid extractions of sludge cake ER-1 during runs 4-1 through 4-4.

recovered experimentally in runs 3-1, 3-2 and 3-3 and that residual unextracted aluminum was negligible (i.e., 0 to 0.1 g of Al). The acid doses which were slightly in excess of stoichiometric requirements (i.e., runs 3-1 and 3-2) resulted in complete extraction of aluminum (i.e., no residual nonsoluble aluminum in the output), while that conducted at a stoichiometric dose of 100 percent (run 3-3) resulted in virtually complete extraction of available aluminum. In addition, aluminum recoveries, relative to input and output aluminum, were excellent.

Product quality data in Table 42 indicated aluminum concentrations ranged from 8.1 to 9.0 percent as Al_2O_3, and were well within acceptable limits for commercial products. The liquid alum products were virtually free of suspended solids and were near neutral or slightly acidic, with respect to dissolution of Al_2O_3. Total iron was 0.03 to 0.11 percent as Fe_2O_3 and was well within acceptable limits. In summary, the alum products produced were of excellent quality, indicating segregated neutralization solids were excellent sources of extractable aluminum.

TABLE 42.   CHARACTERISTICS OF LIQUID ALUM PRODUCED BY ACIDIC EXTRACTION OF SEGREGATED NEUTRALIZATION SLUDGE CAKE SN-1

Parameter	SN-1 Run		
	3-1	3-2	3-3
Total Al_2O_3, percent	8.1	8.7	9.0
Free Al_2O_3, percent	--	--	0.3
Free Acid, percent	1.4	1.3	--
Total Iron, percent as Fe_2O_3	--	0.03	0.11
Suspended Solids:			
Concentration, g/L	0.0	0.1	0.0
Percent Reduction, %	100	>99.9	100
Specific Gravity	1.4	1.4	1.4
pH (2% dilution)	2.5	2.5	3.7
Congo Red Test, color*	purple	purple	brown

*Qualitative acidity test: light brown = free alum; purple = neutral; blue = free acid.

Etch Recovery Sludge Cake (ER-1)

The etch-recovery solids examined during the study were provided as

dewatered solids by a companion plant of Plant A. The ER solids were produced from a caustic etching process similar in function to that used at Plant A. The characteristics of the etch-recovery solids are included in Table 43 and indicate the solids were exceptionally low in free moisture, as indicated by a dry solids content of 91.6 percent and a fixed solids content of 66.5 percent. Approximately 27 percent of the dry solids were volatilized at 550°C which was typical of the CN and SN solids as well. Because the etch-recovery process must be operated without the addition of organic chelating and sequestering agents, it was assumed that the loss of solids at 550°C was attributable to the loss of bound water. The aluminum content of the fixed solids was 43.6 percent and was the highest of any of the sludge solids included in the study. This value was exceptionally higher than the theoretical value of 34.6 percent for $Al(OH)_3$ but below the theoretical value of 52.9 percent for Al_2O_3. In examination of this point, aluminum hydroxides are frequently characterized as hydrated forms of aluminum oxide. For example, $Al(OH)_3$ is equivalent to $Al_2O_3 \cdot 3H_2O$ in terms of aluminum content. In examination of the 43.6 percent value in Table 43, this aluminum content could be representative of a compound with the formula $Al_2O_3 \cdot 1.21H_2O$. Regardless, the sludge cake was rich in aluminum and an excellent candidate for production of alum. However, because of its apparent crystalline nature and more aluminum-oxide-like form, there was concern that it would be more difficult to extract under acidic conditions.

TABLE 43. CHARACTERISTICS OF DEWATERED ETCH-RECOVERY SLUDGE CAKE ER-1

Parameter	Sludge Cake ER-1
Dry (103°C) Solids Content, g dry solids/100 g wet cake......	91.6
Fixed (550°C) Solids Content, g fixed solids/100 g wet cake...	66.4
Aluminum Content, g Al/100 g fixed solids....................	43.6

Four runs were conducted with the ER-1 cake, as presented in Table 44, in which 0.14 to 0.33 kg of ER-1 cake were extracted. Because of the exceptionally high aluminum content of the sludge cakes it was necessary to add between 0.45 to 0.6 kg of water to the extractions. In all but run 4-3, the water was added prior to the addition of acid. In run 4-3, 0.1 kg and 0.025 kg of water were added to the extraction mixture immediately following the collection of samples at the 1-h and 6-h time intervals. These additions were made to prevent the formation of a crystalline product and decreased filtrate aluminum concentrations slightly. Runs 4-1 and 4-2 were conducted with less than stoichiometric addition of acid while runs 4-3 and 4-4 were conducted at stoichiometric levels. Furthermore, all runs were conducted at 90°C for periods of 8 to 24 h because of the refractory nature of the aluminum precipitates (Harmon and Saunders, 1985). The estimated product concentrations ranged from 6.9 to 10.5 percent as Al_2O_3. The low value of 6.9 percent as Al_2O_3 for run 4-4 was the result of an error made in

TABLE 44. INITIAL EXPERIMENTAL CONDITIONS FOR EXTRACTIONS
OF ETCH RECOVERY SLUDGE CAKE ER-1

Parameter	ER-1 Run			
	4-1	4-2	4-3	4-4
SLUDGE ADDITION				
Total Mass, g	140.5	153.8	139.0	119.1
Fixed Solids, g	93.3	101.0	87.4	74.9
Moisture, g	47.2	52.8	51.6	44.2
WATER ADDITION, g	478.6	451.7	509.9	599.8
SULFURIC ACID				
Sulfuric Acid Addition, g	194.6	190.3	207.6	177.9
Percent of Stoichiometric Acid Dosage, Percent	87.9	79.4	100.0	100.0
CONTROL TEMPERATURE, °C	90	90	90	90
ESTIMATED PRODUCT CONCENTRATION, Percent as Al_2O_3	9.4	10.5	8.4	6.9

the mass of water added to the extraction mixture, creating a dilute product.
The estimated product concentrations for runs 4-1, 4-2 and 4-3 were therefore
all above the desired value of 8 percent as Al_2O_3.

Filtrate aluminum data in Figure 43 indicate that the acidic extraction
of cake ER-1 proceeded at rates slower than those demonstrated for CN and SN
cakes, with the exception of run 4.3. Filtrate aluminum concentration for
runs 4-1 and 4-2 reached a plateau value of approximately 6 percent in one
hour and proceeded to values in excess of 8 percent after 4 hours, while run
4-3 was at approximately 9 percent in one hour. The accelerated rate of
extraction for run 4-3, as depicted in Figure 43, was attributed to the
elevated strength of acid in the extraction mixture during the initial por-
tions of the run. Because 0.125 kg of the total of 0.45 kg of water to be
added was withheld initially, but was added at the 1-h and 6-h intervals,
the effective strength of the sulfuric acid added to the mixture was initi-
ally 28.4 percent as H_2SO_4 (conc.) as opposed to 23.9 percent for runs 4-1
and 4-2 and 19.8 percent for run 4.4. This elevated acid strength enhanced
the extraction of aluminum and would appear to be the preferred means for
extraction where possible. Filtrate aluminum data were not collected for
run 4-4 during the initial 4 h of the extraction. At 4 h, filtrate aluminum
was 6.6 percent and was virtually at completion since the projected
concentration for the run was 6.9 percent. After the initial 4-h sample,
filtrate aluminum concentration varied slightly and trended towards higher
values due to continued extraction of aluminum and evaporation of water.

The material balances conducted on reaction-mixture and aluminum mass are presented in Table 45. The recovery of total mass varied from 82 to 91 percent. Given that three of the four runs were conducted over a 24-h period and evaporational losses would be expected to be high, these recoveries were acceptable. Aluminum recoveries ranged between 89 to 109 percent and were excellent. Residual aluminum mass, however, ranged from 5 to 12.4 g and accounted for approximately 32 percent of output aluminum for runs 4-1 and 4-2 and 15 to 19 percent for runs 4-3 and 4-4. The lack of complete extraction of aluminum for runs 4-1 and 4-2 was expected since acid doses were 88 and 79 percent of stoichiometric values, respectively. Stoichiometric acid doses were used for runs 4-3 and 4-4, accounting for the lower portion of unextracted aluminum. However, there was apparently additional aluminum that could have been extracted had higher acid doses been employed or had initial acid strength been increased by delayed addition of water.

TABLE 45. MATERIAL BALANCES ON TOTAL MASS AND ALUMINUM FOR EXTRACTIONS OF ETCH RECOVERY SLUDGE CAKE ER-1

Parameter	ER-1 Run			
	4-1	4-2	4-3	4-4
TOTAL MASS				
Input:				
Fixed Solids, g..	93.3	101.0	87.4	74.9
Moisture, g......	525.8	504.5	561.5	644.0
Acid, g.........	194.6	190.3	207.6	177.9
Total, g........	813.7	795.8	856.5	896.8
Output, g...........	742.7	705.0	775.4	737.2
Percent Recovered....	91	89	91	82
ALUMINUM				
Input, g............	40.7	44.0	38.1	32.7
Output:				
Soluble, g.......	26.3	26.9	33.7	29.2
Residual, g......	12.1	12.4	7.9	5.0
Total, g........	38.4	39.3	41.6	34.2
Percent Recovered....	94	89	109	105

The characteristics of selected terminal or near-terminal samples from extraction runs are presented in Table 46. Data presented for runs 4-1, 4-2 and 4-3 were those for samples collected after 6 or 8 h of extraction and were equivalent to those presented in earlier tables. Data for run 4-4 were collected after 24 h. Aluminum concentrations ranged from 8.3 to 9.2 percent as Al_2O_3 and were well within acceptable limits. The samples were all near neutral conditions with respect to free-acid and free-Al_2O_3 concentrations and contained low levels of total iron. Residual suspended solids concentrations were high, ranging from 25 to 94 g/L, and indicated incomplete extraction of aluminum. The free aluminum values of 0.1 to 0.4 percent as Al_2O_3 were not in agreement with this conclusion but the residual aluminum data in Table 45 confirmed the incomplete extraction of aluminum. It therefore appeared that a portion of the aluminum was resistant to extraction and was not measured in the "free Al_2O_3" procedure. No runs were, however, conducted with excess acid doses to establish the extent to which the residual aluminum could be extracted.

TABLE 46. CHARACTERISTICS OF LIQUID ALUM PRODUCED BY ACIDIC EXTRACTION OF ETCH RECOVERY SLUDGE CAKE ER-1

Parameter	ER-1 Run			
	4-1[a]	4-2[a]	4-3[b]	4-4[c]
Total Al_2O_3, percent......................	8.3	9.2	8.5	8.6
Free Al_2O_3, percent......................	0.1	0.2	--	0.4
Free Acid, percent......................	--	--	0.2	--
Total Iron, percent as Fe_2O_3...............	--	--	0.1	0.1
Suspended Solids:				
Concentration, g/L......................	93.7	66.9	38.9	25.0
Percent Reduction, %......................	54	71	81	85
Specific Gravity......................	1.4	1.4	1.3	1.3
pH (2% dilution)......................	2.9	3.2	2.9	3.6
Congo Red Test, color[d]...................	purple	brown	purple	brown

[a]Data from sample collected after a 6-h reaction period.
[b]Data from sample collected after an 8-h reaction period.
[c]Data from sample collected after a 24-h reaction period.
[d]Qualitative acidity test: light brown = free alum; purple = neutral; blue = free acid.

Blended Sludge Cakes

Two runs were made in which sludge cakes were prepared by blending a conventional neutralization sludge cake (CN-2) with either a segregated-neutralization sludge cake (SN-1) or etch-recovery solids (ER-1).

Conventional Neutralization/Segregated Neutralization Sludge Cake (CN-2/SN-1)--

The segregated neutralization cake employed in studies reported previously (see Tables 39-42) was blended with a conventional neutralization sludge cake (CN-2) at a fixed solids ratio of 43 g of CN-2 to 54 g of SN-1. As indicated in Table 47, the net dry (103°C) solids content of the blend was 25.2 percent, fixed solids content was 21.4 percent and aluminum content was 34.6 percent. All of these values were indicative of excellent potential for production of commercial-strength liquid alum. Run 5-1 was made using a 100 percent stoichiometric acid dose at 90°C (Table 48).

TABLE 47.   CHARACTERISTICS OF COMBINED DEWATERED CONVENTIONAL NEUTRALIZATION/ SEGREGATED NEUTRALIZATION SLUDGE CAKES CN-2 AND SN-1

Parameter	Sludge Cake
	CN-2/SN-1
Dry (103°C) Solids Content, g dry solids/100 g wet cake.......	25.2
Fixed (550°C) Solids Content, g fixed solids/100 g wet cake...	21.4
Aluminum Content, g Al/100 g fixed solids....................	34.6

TABLE 48.   INITIAL EXPERIMENTAL CONDITIONS FOR EXTRACTION OF COMBINED CONVENTIONAL NEUTRALIZATION/SEGREGATED NEUTRALIZATION SLUDGE CAKES CN-2 AND SN-1

Parameter	Run
	5-1
SLUDGE ADDITION	
Total Mass, g...	451.5
Fixed Solids, g..	96.8
Moisture, g...	354.7
WATER ADDITION, g...	178.6
SULFURIC ACID	
Sulfuric Acid Addition, g.....................................	182.6
Percent of Stoichiometric Acid Dosage, Percent...............	100.0
CONTROL TEMPERATURE, °C......................................	90
ESTIMATED PRODUCT CONCENTRATION, Percent as Al_2O_3..............	7.8

With the addition of a slight excess of water, the projected alum concentration was 7.8 percent as Al_2O_3. Of the total of 178.6 g of water added, a total of 50 g was added after collection of the fourth sample at 4 h. This resulted in dilution of the remaining extract as shown by a decrease in aluminum concentration in Figure 44.

Filtrate aluminum data in Figure 44 indicated the extraction proceeded to completion rapidly within the initial 0.5 h. This was in general agreement with results obtained previously for CN-1, CN-2 and SN-1 sludges (see Figures 40-42). The recovery of total mass was 90 percent while aluminum was recovered at the 99 percent level, as indicated in Table 49. In addition, the residual aluminum was only 0.6 percent of the total, indicating virtually complete extraction of aluminum from the sludge cake. Data in Table 50 indicated that the concentration of the liquid alum was 8.2 percent as Al_2O_3 and that the product was near neutral with respect to extraction of Al_2O_3. Iron and suspended solids concentrations were low with the suspended solids being reduced by 98.4 percent, relative to the solids placed in the reactor. It was therefore concluded that blended CN

TABLE 49. MATERIAL BALANCES ON TOTAL MASS AND ALUMINUM FOR EXTRACTION OF COMBINED CONVENTIONAL NEUTRALIZATION/SEGREGATED NEUTRALIZATION OF SLUDGE CAKES CN-2 AND SN-1

Parameter	Run 5-1
TOTAL MASS	
Input:	
Fixed Solids, g	96.8
Moisture, g	533.3
Acid, g	182.6
Total, g	812.7
Output, g	730.1
Percent Recovered	90
ALUMINUM	
Input, g	33.5
Output:	
Soluble, g	33.0
Residual, g	0.2
Total, g	33.2
Percent Recovered	99
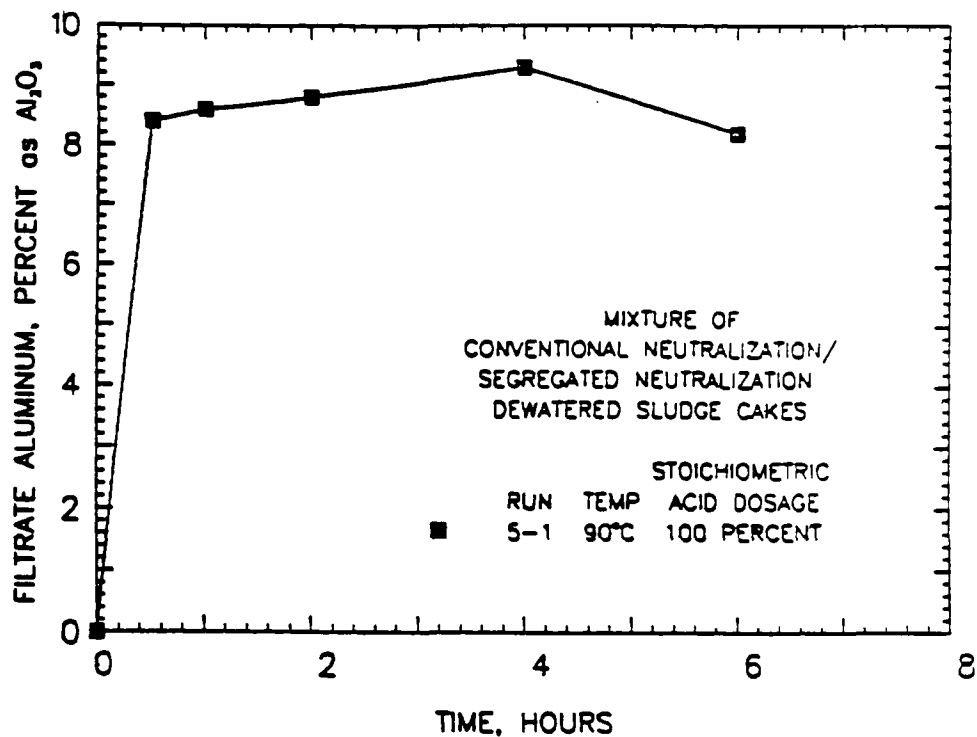
Figure 44. Filtrate aluminum concentrations for sulfuric-acid extraction of sludge cake CN-2/SN-1 during run 5-1.
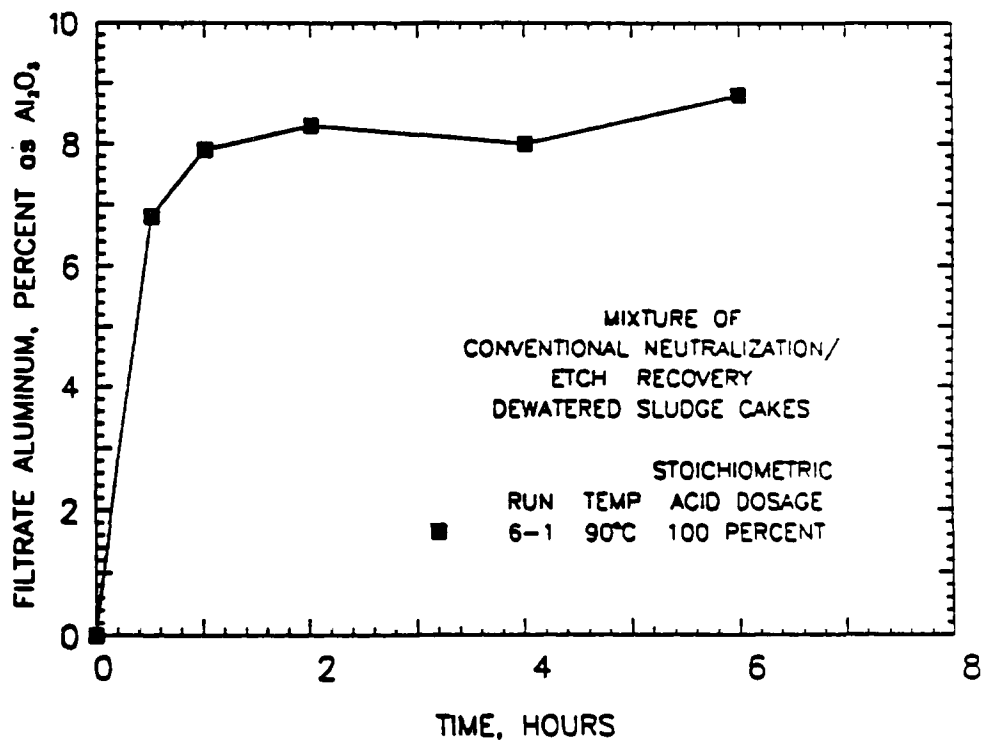
Figure 45. Filtrate aluminum concentrations for sulfuric-acid extraction of sludge cake CN-2/ER-1 during run 6-1.

TABLE 50. CHARACTERISTICS OF LIQUID ALUM PRODUCED BY ACIDIC EXTRACTION
OF COMBINED CONVENTIONAL NEUTRALIZATION AND SEGREGATED
NEUTRALIZATION SLUDGE CAKES CN-2 AND SN-1

Parameter	Run 5-1
Total Al_2O_3, percent	8.2
Free Al_2O_3, percent	0.2
Free Acid, percent	--
Total Iron, percent as Fe_2O_3	0.04
Suspended Solids:	
Concentration, g/L	3.0
Percent Reduction, percent	98
Specific Gravity	1.3
pH (2% dilution)	3.5
Congo Red Test, color*	brown/ purple

*Qualitative acidity test: light brown = free alum; purple = neutral;
blue = free acid.

and SN sludges could be effectively extracted with acid to produce
commercial-strength liquid alum.

Conventional Neutralization/Etch Recovery Sludge Cake (CN-2/ER-1)--
A dewatered conventional neutralization cake was mixed with etch-
recovery cake at a fixed solids ratio of 1.1 to 1 (i.e., 50 g CN-2 fixed
solids to 45 g of ER-1 fixed solids). The net characteristics of the
blended sludges are presented in Table 51. The extraction was conducted at

TABLE 51. CHARACTERISTICS OF COMBINED DEWATERED CONVENTIONAL
NEUTRALIZATION AND ETCH RECOVERY SLUDGE CAKES CN-2
AND ER-1

Parameter	Sludge Cake CN-2/ER-1
Dry (103°C) Solids Content, g dry solids/100 g wet cake	30.2
Fixed (550°C) Solids Content, g fixed solids/100 g wet cake	22.5
Aluminum Content, g Al/100 g fixed solids	41.3

90°C using a stoichiometric acid dose with a projected alum concentration of 8.9 percent as Al_2O_3 (Table 52). A total of 50 g of water was withheld from the initial extraction mixture and was added after collection of a sample at the 2-h interval, resulting in a slight dilution of the extraction product.

TABLE 52.   INITIAL EXPERIMENTAL CONDITIONS FOR EXTRACTION OF COMBINED CONVENTIONAL NEUTRALIZATION AND ETCH RECOVERY SLUDGE CAKES CN-2 AND ER-1

Parameter	Run 6-1
SLUDGE ADDITION	
Total Mass, g...	423.5
Fixed Solids, g..	95.1
Moisture, g..	328.4
WATER ADDITION, g...	196.8
SULFURIC ACID	
Sulfuric Acid Addition, g..............................	213.9
Percent of Stoichiometric Acid Dosage, Percent........	100.0
CONTROL TEMPERATURE, °C...................................	90
ESTIMATED PRODUCT CONCENTRATION, Percent as Al_2O_3......	8.9

Filtrate aluminum data in Figure 45 indicated that the extraction proceeded relatively rapidly but was not at completion until about 2 h, due to the presence of the etch recovery solids. Approximately 94 percent of the total mass was recovered during the extraction and aluminum recovery was 102 percent (Table 53). The residual aluminum was, however, equal to 18.8 percent of the initial aluminum added, indicating incomplete extraction of the aluminum. This was consistent with results obtained previously with etch recovery solids (see Table 45).

Finally, the product produced had an aluminum concentration of 8.8 percent as Al_2O_3 and contained a slight excess of free acid, as indicated in Table 54. Iron concentration was exceptionally low but suspended solids concentration was high (i.e., 32.7 g/L). A total of 16 percent of the initial suspended solids remained indicating the incomplete extraction of sludge aluminum.

110

TABLE 53. MATERIAL BALANCES ON TOTAL MASS AND ALUMINUM FOR EXTRACTION
OF COMBINED CONVENTIONAL NEUTRALIZATION AND ETCH RECOVERY
SLUDGE CAKES CN-2 AND ER-1

Parameter	Run 6-1
TOTAL MASS	
Input:	
Fixed Solids, g	95.1
Moisture, g	525.2
Acid, g	213.9
Total, g	834.2
Output, g	780.2
Percent Recovered	94
ALUMINUM	
Input, g	39.3
Output:	
Soluble, g	32.8
Residual, g	7.4
Total, g	40.2
Percent Recovered	102

METAL CONTENT OF SLUDGES AND LIQUID ALUM EXTRACTS

In the initial phase of the study when sludge dewatering properties
were being examined, the metal content of numerous sludges and aluminum
residues were examined in conjunction with studies to determine the extent
of sludge dewatering achieved at various full-scale plants. These data are
presented herein in conjunction with experimental data collected for liquid
alum extracts produced as a part of this study.

Selected Aluminum-Finishing Suspensions and Residues

The metal contents of dewatered sludge cakes provided by seven
aluminum-anodizing plants are presented in Table 55. The solids contents
for the dewatered cakes ranged from 9.8 to 25.9 percent. The high value of
25.9 percent was produced with a high-pressure filter press, while the
remainder were produced with low-pressure presses or rotary vacuum filters.

TABLE 54. CHARACTERISTICS OF LIQUID ALUM PRODUCED BY ACIDIC
EXTRACTION OF COMBINED CONVENTIONAL NEUTRALIZATION
AND ETCH RECOVERY SLUDGE CAKES CN-2 AND ER-1

Parameter	Run 6-1
Total Al_2O_3, percent..	8.8
Free Al_2O_3, percent...	--
Free Acid, percent..	0.8
Total Iron, percent as Fe_2O_3....................................	0.04
Suspended Solids:	
Concentration, g/L...	32.7
Percent Reduction, percent...................................	84
Specific Gravity..	1.4
pH (2% dilution)...	2.6
Congo Red Test, color*...	purple

*Qualitative acidity test: light brown = free alum; purple = neutral;
blue = free acid.

The aluminum content for the cakes varied from 18.4 percent to 48.2
percent of the total dry mass (i.e., 1.84×10^5 to 4.82×10^5 mg/kg of dry
solids). Sodium was the next most prevalent metal and ranged from 0.6 to
8.9 percent, followed by iron, which ranged from 0.17 to 1.13 percent. The
sludge cakes contained measurable quantities of many of the potentially-
toxic metals examined. On an overall basis, the predominant trace metals
detected were chromium (Cr), copper (Cu), lead (Pb), nickel (Ni) and tin
(Sn), although there was considerable variation in the concentrations of
these.

In examination of the metal content of the sludges relative to
production of liquid alum, it was assumed that the alum product was to be
used for the purposes of treating drinking waters. This, however, is a very
strict assumption, since less than 5 percent of the alum produced is used in
treatment of water and wastes. Furthermore, it was assumed that all metals
in a sludge cake would be solubilized during acid extraction and would
appear as a contaminant with the extracted aluminum. In addition, no
consideration was given to the extent to which the contaminant metals would
be removed with the aluminum-hydroxide floc during the coagulation process.
Just as aluminum is removed in an effective coagulation process, trace
metals are commonly removed through adsorption to or complexation with the
aluminum hydroxide formed. This was ignored to provide a conservative
estimate of metal impact.

112

TABLE 55. METAL CONTENT OF DEWATERED CONVENTIONAL NEUTRALIZATION SLUDGE CAKES FROM SELECTED ALUMINUM-ANODIZING PLANTS

Metal	Concentration, mg/kg (dry mass)								
	Plant A		Plant B		Plant C	Plant D	Plant E	Plant F	Plant I
	AC4-3164	AC4-3201	BC4-3181	BC4-3201	CC4-3201	DC4-3059	EC4-3201	FC4-3201	IC4-3201
Aluminum, Al	4.1×10^5	2.2×10^5	2.55×10^5	3.26×10^5	1.84×10^5	3.32×10^5	2.71×10^5	3.02×10^5	4.82×10^5
Calcium, Ca	77	945	3208	574	490	214	181	2605	205
Iron, Fe	2993	1743	6948	11304	20400	3290	4940	2185	2321
Magnesium, Mg	1667	2110	1853	2174	5100	1338	1747	8403	1536
Potassium, K	54	43	201	30	44	177	54	52	45
Sodium, Na	23073	82548	5983	21731	30585	40125	42159	42002	89262
Arsenic, As	1.03	1.65	1.85	1.31	2.35	0.8	1.2	1.0	1.25
Cadmium, Cd	0.37	0.29	0.59	0.77	0.53	0.29	1.85	0.73	0.02
Chromium, Cr	13.9	36.5	108	853	357	56.4	60.1	58.6	40
Copper, Cu	384	108	328	261	240	473	90.2	74.6	70.4
Lead, Pb	26	18	107	9.78	24	91	22.7	26.6	30.9
Mercury, Hg	0.05	0.41	0.1	0.05	0.81	0.04	0.96	<0.01	0.001
Nickel, Ni	6498	8532	12352	8261	418	1552	60	101	107
Selenium, Se	4.38	0.96	1.62	2.74	0.21	<0.1	0.25	0.88	0.19
Silver, Ag	0.28	0.05	0.023	0.035	0.041	0.13	0.03	0.06	0.04
Tin, Sn	13248	16513	7722	9566	<0.005	2132	<0.005	<0.005	<0.005
Zinc, Zn	0.01	32	100	224	102	410	211	28.6	51.8
Total Solids (103°C)	11.7%	–	25.9%	–	9.8%	18.75%	16.6%	11.9%	11.2%

113

To evaluate the impact of liquid alum to be produced from the sludge cakes, a hypothetical aluminum dose was to be selected. Concentrations of aluminum reported as being used in coagulation of drinking waters include: 0.27 to 2.7 mg/L (Stumm and O'Melia, 1968; Mangravite et al., 1975); 0.8 to 4.0 mg/L (Parkham, 1962) and 0.05 to 9 mg/L (Edwards and Amirtharajah, 1985). To be conservative in the evaluation, an aluminum concentration of 10 mg/L was selected for analysis of the sludge metal data. This value is analagous to an "alum" concentration of 111 mg/L as $Al_2(SO_4)_3 \cdot 14.3H_2O$. Using these assumptions and the data in Table 55, metal concentrations in drinking water coagulated with aluminum at a concentration of 10 mg/L were calculated and are presented in Table 56.

The metals controlled through federal drinking water regulations are identified with their maximum contaminant levels (MCL) in Table 56. Projected arsenic, cadmium, lead, mercury, selenium and silver concentrations were all below their respective MCL values. With the exception of two values for lead (i.e., 2.7 µg/L and 4.2 µg/L), all concentrations of these metals were less than 3 percent of the MCL value of 50 µg/L, indicating minimal impact on the quality of the drinking water, even without giving allowance for reduction in contaminant concentrations through sedimentation and filtration processes following coagulant addition. Projected chromium concentrations were all below the MCL of 50 µg/L, although two projected values (i.e., 19.4 µg/L and 26.2 µg/L) were near the limit concentration. The remaining samples, however, had projected concentrations which ranged from 0.3 to 4.2 µg/L and were well below the MCL value.

Copper and zinc are not regulated but values of 1000 µg/L and 5000 µg/L, respectively, have been suggested for use in evaluation of drinking-water quality. All projected concentrations of these metals were well below these suggested values.

The remaining heavy metals, nickel and tin, are not currently (1987) regulated by federal drinking-water limits, although nickel is listed as a contaminant for which a MCL must be established by 1989. Concentrations for these contaminants were highly variable and were the highest for any of the heavy metals examined. Nickel ranged from 2.2 µg/L to 485 µg/L, and tin ranged from <0.0001 µg/L to 751 µg/L. While these are not regulated, the elevated concentrations for nickel (e.g., 158 µg/L to 485 µg/L) and tin (e.g., 293 µg/L to 751 µg/L) in sludge cakes from Plants A and B indicate caution should be exercised in the ultimate use of alum produced from these sludge cakes.

It must, however, be emphasized that the foregoing analysis does not indicate the products to be produced from these sludge cakes would not meet current standards. For example, the Water Chemicals Codex developed by the National Research Council (1982) has proposed recommended maximum impurity content (RMIC) values for water treatment additives. The RMIC values for alum are, however, based on contaminant concentrations remaining in water samples which have been dosed with an appropriate aluminum concentration (e.g., 10 mg/L of aluminum); adjusted to a pH of 6; flocculated for 1 h; and then filtered with an 0.45µm filter. This RMIC value thereby takes into consideration the removal of alum contaminants during coagulation,

TABLE 56. PROJECTED INCREASES IN METAL CONCENTRATIONS IN COAGULATED DRINKING WATER TREATED WITH ALUM, PRODUCED FROM DEWATERED CONVENTIONAL NEUTRALIZATION SLUDGE CAKES, AT AN ALUMINUM CONCENTRATION OF 10 mg/L

Metal	MCL µg/L	Concentration, µg/L								
		Plant A		Plant B		Plant C	Plant D	Plant E	Plant F	Plant I
		AC4-3164	AC4-3201	BC4-3181	BC4-3201	CC4-3201	DC4-3059	EC4-3201	FC4-3201	IC4-3201
Calcium, Ca	-	2	43	126	18	27	6	7	86	4
Iron, Fe	-	73	79	273	347	1111	99	182	72	48
Magnesium, Mg	-	4	96	73	67	278	40	64	278	32
Potassium, K	-	1.3	2	8	1	2	5	2	2	1
Sodium, Na	-	562.8	3752	235	667	1666	1209	1556	1389	1852
Arsenic, As	50	0.03	0.08	0.07	0.04	0.13	0.024	0.04	0.03	0.03
Cadmium, Cd	10	0.01	0.01	0.02	0.02	0.03	0.009	0.07	0.02	0.0004
Chromium, Cr	50	0.3	1.7	4.2	26.2	19.4	1.7	2.2	1.9	0.8
Copper, Cu	(1000)*	9.4	4.9	12.9	8.0	13.1	14.2	3.3	2.5	1.5
Lead, Pb	50	0.6	0.8	4.2	0.3	1.3	2.7	0.8	0.9	0.6
Mercury, Hg	2	0.001	0.02	0.004	0.002	0.04	0.001	0.04	<0.0003	0.0002
Nickel, Ni	-	158	388	485	253	22.8	46.7	2.2	3.3	2.2
Selenium, Se	10	0.11	0.04	0.06	0.08	0.01	0.003	0.01	0.03	0.004
Silver, Ag	50	0.006	0.002	0.0009	0.001	0.002	0.004	0.001	0.002	0.008
Tin, Sn	-	323	751	303	293	<0.0003	64.2	<0.0002	<0.0002	<0.0001
Zinc, Zn	(5000)*	0.0002	1.5	3.9	6.9	5.6	12.3	7.8	0.9	1.1

*Secondary MCL values; all others are primary MCL values.

115

sedimentation and filtration in a treatment plant. The above analysis did not, however, allow for removal of contaminants by subsequent treatment. The foregoing analysis, however, does highlight concern with respect to selected metals and indicates a need to control or reduce them if alum production is to be pursued. For example, nickel and tin originate from two-step anodizing baths which could be isolated and treated separately to minimize the concentrations of these metals in the sludges produced.

Data in Table 57 are for a segregated neutralization suspension produced at Plant H. The concentrated suspension was produced by addition of spent acids to a spent caustic suspension and, as expected, alumuminum and iron were the major metallic constituents, followed by tin at 1200 mg/L.

TABLE 57. METAL CONCENTRATIONS FOR A SEGREGATED NEUTRALIZATION SUSPENSION PRODUCED AT PLANT H

Metal	Concentration, mg/L
Aluminum, Al	17,500
Calcium, Ca	39
Iron, Fe	50
Magnesium, Mg	120
Potassium, K	49
Sodium, Na	13,100
Arsenic, As	0.06
Cadmium, Cd	0.01
Chromium, Cr	1.58
Copper, Cu	50
Lead, Pb	0.27
Mercury, Hg	0.01
Nickel, Ni	30
Selenium, Se	<0.1
Silver, Ag	0.025
Tin, Sn	1200
Zinc, Zn	1.8
Suspended Solids	83.1 g/L

Liquid Alum Products

Based on initial characterizations of sludge solids to be extracted, selected trace metals were examined in liquid alums produced by acidic extraction of conventional-neutralization (CN-2), segregated-neutralization (SN-1) and etch-recovery (ER-1) sludge cakes. Because copper, mercury, arsenic and selenium were not major constituents, they were not examined. Metal data for three alum samples produced from sludge cakes CN-2, SN-1 and ER-1 are presented in Table 58. In addition, data for two samples of a

116

standard grade of commercial liquid alum, as obtained directly from two
metropolitan Atlanta drinking-water treatment plants, and one sample of an
"iron-free" grade of commercial liquid alum are included in Table 58.

TABLE 58. METAL CONTENT OF LIQUID-ALUM SAMPLES PRODUCED FROM CN-2, SN-1
AND ER-1 SLUDGE CAKES AND THREE COMMERCIAL ALUM PRODUCTS

Metal	Concentration, mg/L					
	Sludge-Cake Alum			Commercial Alum		
	CN-2	SN-1	ER-1	Standard		Iron-Free
				1	2	
Cadmium, Cd	0.4	0.3	0.3	0.03	0.3	0.3
Chromium, Cr	30.0	4.4	3.7	78	0.9	0.9
Iron, Fe	60.0	18.3	21.0	1845	2080	10.1
Lead, Pb	20	19	26	6.6	4.1	5.6
Nickel, Ni	752	8	7	44	44	-
Silver, Ag	0.7	0.2	0.08	0.25	0.2	0.15
Tin, Sn	914	28	17	155	-	2.5
Zinc, Zn	12	6	7	8.5	8.5	1.0

For the three samples of liquid alum produced from sludges, the
concentrations of cadmium (Cd), chromium (Cr), iron (Fe), silver (Ag) and
zinc (Zn) were at, or well below, concentrations contained in standard
commercial liquid alum. The lead (Pb) concentrations ranged from 19 to 26
mg/L in alums from sludges and ranged from 4.1 to 6.6 mg/L in standard
commercial products.

The conventional neutralization sludge (CN-2) produced a liquid alum
with excessive concentrations of nickel (Ni = 752 mg/L) and tin (Sn = 914
mg/L). It was speculated that the two-step (Ni/Sn-based) anodizing process
employed at Plant A resulted in elevated concentrations of nickel and tin in
the alum products.

At an aluminum dose of 10 mg/L for treatment of a drinking water, as
discussed earlier, these levels would result in approximate concentrations
of Ni = 137 µg/L and Sn = 166 µg/L in a coagulated water. These
concentrations could, however, be reduced significantly by gravity
sedimentation and filtration. Despite the fact that no standards are
currently applicable to concentrations of nickel and tin, it is apparent
that the indicated levels warrant further investigation on a plant-by-plant
basis. The levels of nickel and tin in the alum products produced with SN-1
and ER-1 sludges were well below concentrations in standard commercial
liquid alum.

In summary, the trace metal analyses for the liquid alum produced from
conventional neutralization sludge (CN-2) indicated that only lead, nickel
and tin were of concern when compared to standard commercial products.
Concentrations of cadmium, chromium, iron, silver and zinc were of no

concern in this regard. Finally, only lead concentrations in liquid alum were of marginal concern when examining segregated neutralization (SN-1) and etch recovery (ER) sludges. It is recommended that waste segregation be considered when addressing the control of nickel and tin.

SECTION 7

REFERENCES

Allied Chemical Corporation, 1976, "Alum", Allied Chemical Corporation, Syracuse, NY.

APHA, 1985, Standard Methods for the Examination of Water and Wastewater, 15th Edition, American Public Works Association, Washington, D.C.

Edwards, G. A., and Amirtharajah, A., 1985, "Removing Color Caused by Humic Acids," Jour. American Water Works Assoc., 77, 50.

EPA, 1982, Process Design Manual for Dewatering Municipal Wastewater Sludges, EPA-625/1-82-014, U.S. Environmental Protection Agency, MERL, Cincinnati, OH.

EPA, 1986, Design Information Report - Recessed Plate Filter Presses, EPA 600-M-86-017, WERL, U.S. Environmental Protection Agency. Cincinnati, OH.

Harmon, C. B. and Saunders, F. M., 1985, "Production of Liquid Alum from Aluminum-Anodizing Sludges," Water Science and Technology, 17, 541-550.

Johns, P. J., 1987, "Design and Operation of Fixed-Volume Filter Presses," A Special Problem Report to School of Civil Engineering, Georgia Institute of Technology, Atlanta, GA.

Mangravite, F. J., et al., 1975, "Removal of Humic Substances by Coagulation and Microflotation", Jour. American Water Works Assoc., 67, 88.

National Research Council, 1982, Water Chemicals Codex, National Academy Press, Washington, D.C.

O'Connor, J. T., 1984, Environmental Engineering Unit Operations and Unit Processes Laboratory Manual, Third Edition, Association of Environmental Engineering Professors, The University of Texas, Austin, TX.

Parkham, R. F., 1962, "The Theory of the Coagulation Process - A Survey of the Literature II. Coagulation as a Water Treatment Process", Proc. Soc. Water Treat. Exam., 11, 106.

Saunders, F. M., et al., 1982, Characterization, Reclamation and Final Disposal of Aluminum Bearing Sludges, SCEGIT-82-102, Report to The Aluminum Association, Inc., Washington, D.C.

Saunders, F. M., et al., 1984, "Evaluation of Process Systems for Effective Management of Aluminum Finishing Wastewaters and Sludges," U.S. Environmental Protection Agency, IERL, Cincinnati, OH; PB 84-170 661, Springfield, VA.

Stumm, W., and O'Melia, C. R., 1968, "Stoichiometry of Coagulation", Jour. Amer. Water Works Assoc., 60, 514.

1. REPORT NO.	2.	3 RECIPIENT'S ACCESSION NO.

4. TITLE AND SUBTITLE
RECLAMATION OF ALUMINUM FINISHING SLUDGES

5. REPORT DATE

6. PERFORMING ORGANIZATION CODE

7. AUTHOR(S)
F. Michael Saunders

8. PERFORMING ORGANIZATION REPORT NO.

9. PERFORMING ORGANIZATION NAME AND ADDRESS
School of Civil Engineering
Georgia Tech Research Corporation
Georgia Institute of Technology
Atlanta, GA 30332

10. PROGRAM ELEMENT NO.

11. CONTRACT/GRANT NO.
CR110290-01-0

12. SPONSORING AGENCY NAME AND ADDRESS
Water Engineering Research Laboratory- Cincinnati, OH
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, OH 45268

13. TYPE OF REPORT AND PERIOD COVERED
Project Report

14. SPONSORING AGENCY CODE
EPA/600/14

15. SUPPLEMENTARY NOTES
Project Officer, Thomas J. Powers, 513-569-7550/FTS 684-7550

16. ABSTRACT

    The research study of the reclamation of aluminum-anodizing sludges was
conducted in two sequential phases focused on enhanced dewatering of
aluminum-anodizing sludges to produce commercial-strength solutions of
aluminum sulfate, i.e., liquid alum. The use of high-pressure (14 to 15 bar)
and a diaphragm filter press were shown to be effective in dewatering aluminum
anodizing sludges to cake solids contents of 27% to 29% and 25% to 31%,
respectively. These values were well above the 21% value required to justify
pursuit of direct acidic extraction of aluminum. Commercial-strength
solutions of aluminum sulfate with concentrations of 8% as Al_2O_3 were
produced using conventional-neutralization, segregated-neutralization, and
etch-recovery sludge cakes. The trace metal contents of the alum products
were, in general, typical of commercial products.

17 KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS OPEN ENDED TERMS	c. COSATI Field/Group

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS *(This Report)*	21. NO. OF PAGES
RELEASE TO PUBLIC	UNCLASSIFIED	
	20. SECURITY CLASS *(This page)*	22. PRICE
	UNCLASSIFIED	

EPA Form 2220-1 (Rev. 4-77)   PREVIOUS EDITION IS OBSOLETE