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Combined Reverse Osmosis and Freeze Concentration of Bleach Plant Effluents

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COMBINED REVERSE OSMOSIS AND FREEZE CONCENTRATION OF BLEACH PLANT EFFLUENTS

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FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that the new and increasingly more efficient pollution control methods be used. The Industrial Research Laboratory - Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

This report describes the evaluation of two technologies for renovation of bleach plant effluents from three different wood pulp mills. Bleach effluents invariably contain chlorides which render the water too corrosive for reuse. Technologies for removal of chlorides from these effluents are expensive and energy consuming. Two relatively new methods of chloride concentration, reverse osmosis and freeze concentration, have advanced to the stage where their demonstration appeared timely. They are low energy consumers but susceptible to problems from chemicals which precipitate, aggregate or accumulate at interfaces. The results of the project carried out by the Institute of Paper Chemistry·at three mill sites summarize the problems encountered and suggest changes which could overcome some of the obstacles. The information will be of value to other segments of the industry, consultants and reverse osmosis equipment suppliers. For further information please contact the Food and Wood Products Branch of the Industrial Environmental Research Laboratory, Cincinnati.

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ABSTRACT

Reverse osmosis (RO) and freeze concentration (FC) were evaluated at three different pulp and paper mills as tools for concentrating bleach plant effluents. By these concentration processes, the feed effluent was divided into two streams. The clean water stream approached drinking water purity in some instances, and could potentially be recycled to the mill with minimal problems. The concentrate stream retained virtually all the dissolved material originally present in the feed. Typically, reverse osmosis removed 90% of the water from a stream containing 5 g/l of total solids to give a concentrated stream with 50 g/l solids. Freeze concentration further concentrated the reverse osmosis concentrate to about 200 g/l. Thus, each 100 liters of feed resulted in about 98 liters of clean water and 2 liters of concentrate. Schemes for the ultimate disposal of this final concentrate were not tested.

Based on data collected at the three mills, estimates of the process economics were made. Reverse osmosis alone, or combined with freeze concentration, is quite expensive. At current levels of water usage for bleaching, costs ranged from $18 to $27 per metric ton of bleached pulp [approximately $3.50/1000 gallons (M gal) of bleach plant effluent]. Reduction in fresh water usage in the bleach plant and increased membrane life could significantly lower these costs.

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ACKNOWLEDGMENTS

In a project such as this one, which involved pilot scale operations at three mills and the cooperation of two membrane suppliers, it is impossible to acknowledge all those individuals who helped make this project a success. In particular, mill operating personnel at each of the three mills were extremely cooperative and were invaluable aids in operating the pilot scale equipment.

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SECTION 1

SUMMARY AND CONCLUSIONS

Reverse osmosis (RO) and freeze concentration (FC) were evaluated at three different pulp and paper mills as tools for concentrating bleach plant effluents. By these concentration processes, the feed effluent was divided into two streams. The clean water stream approached drinking water purity in some instances, and could potentially be recycled to the mill with minimal problems. The concentrate stream retained virtually all the dissolved material originally present in the feed. Typically, RO removed 90% of the water from a stream containing 5 g/l of total solids to give a concentrated stream with 50 g/l solids. Freeze concentration further concentrated the reverse osmosis concentrate to about 200 g/l. Thus, each 100 liters of feed resulted in about 98 liters of clean water and 2 liters of concentrate. Schemes for the ultimate disposal of this final concentrate were not tested.

Based on data collected at the three mills, estimates of the process economics were made. Reverse osmosis alone, or combined with freeze concentration, is quite expensive. At current levels of water usage for bleaching, costs ranged from $18 to $27 per metric ton (t) of bleached pulp [approximately $3.50/1000 gallons of bleach plant effluent]. These high operating changes confirmed early speculation that RO and FC would be expensive if they were used to treat the entire bleach effluent under current mill operating practices. Further economic studies indicate that if the bleach plant water systems were closed to release about 21 m^3/t (5000 gal/ton) operating charges would drop to the $14-18/t ($15-20/ton) range. Reduction in bleach plant water usage from 42 m^3/t (10,000 gal/t) to 21 m^3/t (5000 gal/ton) would also reduce capital requirements for the RO/FC processes by about 50%.

The <u>first field demonstration</u> was conducted at Flambeau Paper Company, Park Falls, Wisconsin. This mill is a calcium based acid sulfite mill using a two-stage hypochlorite bleaching system. Approximately 38 m^3 of bleach water are used per metric ton of bleached pulp (9100 gal/ton). The trailer mounted, pilot scale RO unit was designed to process about 190 m^3/day (50,000 gpd) of the effluent and supply about 1.9 m^3/day (500 gpd) of concentrate to the trailer mounted FC unit. Membrane fouling problems because of talc and pitch, were overcome, although not completely.

The RO unit functioned well with flux rates ranging from 22.9 l/m^2-hr [13.5 gallons per square foot per day (gfd)] on the feed effluent containing 5 grams total dissolved solids (TDS) per liter down to 13.2 l/m^2-hr (7.8 gfd) on concentrated solutions at 21 g TDS/l. This was less than the desired 90% water removal, but flux rates dropped as the osmotic pressure climbed rapidly for solutions with solids levels greater than 50 g TDS/l. Talc fouling problems necessitated frequent washups. The washups could probably be greatly reduced with improved bleach washing facilities.

The first stage of the pilot scale FC unit functioned well, but the second stage was plagued with mechanical problems. Limited data indicated that the RO concentrate could be further concentrated to 160 to 220 g TDS/l. Due to the mechanical problems in the second stage freezer, much of the later work had to be done in the Avco laboratories.

Economic studies indicate that a reverse osmosis plant to treat the total sulfite bleach effluent (4200 m^3/day – 1.1 M gpd) could cost about $3,650,000 with operating cost about $30.00/t of pulp ($27.00/ton). The FC unit would cost an additional $940,000 and add about $1.32/t ($1.20/ton) to the operating cost.

The <u>second field trial</u> took place at The Continental Group's mill in Augusta, Georgia. This kraft mill discharges about 42 m^3 of water per ton (10,000 gal/ton) from its CEHD (Chlorination-Extraction-Hypochlorite-Chlorine Dioxide) bleach plant. Both the RO and FC mobile laboratories were moved to Augusta for the field trial.

Again, the RO unit functioned well, with far fewer problems than were encountered in the first field trial, although the mill itself suffered several short term shutdowns which caused interruptions in the RO/FC processing. Flux rates ranged from 24 l/m^2-m (14 gfd) at 4.5 g TDS/l down to 20 l/m^2-hr (12 gfd) at 15 g TDS/l. A major accidental mechanical failure prevented further concentration of the effluent in the mobile pilot plant. Subsequent testing, at the IPC laboratories indicated that RO concentration to the 40 to 50 g TDS/l level was feasible.

The FC unit continued to require much operator attention and could not be operated in a continuous manner like the RO unit. However, both stages could be tested and final product water quality was excellent, with total dissolved solids around 0.1 g/l. A six to tenfold increase in concentration was possible, with the concentrate from the second stage freezer averaging around 100 g TDS/l.

Cost evaluation indicates that a RO plant to treat the entire kraft bleach plant effluent (30,300 m^3/day – 8 M/day) would cost about $25,500,000 with an operating cost of $32.00/t ($29.00/ton). The FC plant would add about $3,000,000 to the capital requirement and increase operating costs by $2.43/t ($2.20/ton).

The <u>third field trial</u> took place at Chesapeake Corporation's West Point, Virginia mill. This kraft mill uses a relatively new oxygen-chlorine dioxide bleach sequence. Effluents from the bleach plant average about 29 m^3/t (6900 gal/ton), which closely approached the project goal of field testing at a mill utilizing 21 m^3/t (5000 gal/ton).

Due to mechanical damage during the second field trial, the RO trailer was not moved to West Point. A small test unit was developed which could operate at a maximum feed rate of 30 m^3/day (8000 gpd). No FC runs were attempted on site; all FC work was done on a small scale in Avco's laboratory.

The RO test unit performed satisfactorily and gave the same type of information as could be obtained from the larger trailer unit. The feed solutions averaged about 5 g TDS/l and were concentrated to about 40 g TDS/l. Fluxes ranged from 20.4 l/m^2-hr (12 gfd) when treating the dilute solutions down to 15 l/m^2-hr (8.8 gfd) when treating the more concentrated solutions.

The concentrate (approximately 40 g TDS/l) was then shipped to Avco for FC work. These samples had to be held for some time and apparently precipitation took place, as Avco analyses indicated the concentrate to be about 10 g TDS/l. Avco could fairly readily concentrate the 10 g TDS/l solutions to 100 g TDS/l but the laboratory equipment was limited to 10:1 concentrations.

Cost estimates for RO and FC systems for the third trial at the Chesapeake Corporation's mill are more difficult to make than for the other trials as smaller scale equipment was utilized and not all the necessary data are available. However, based on data from the other trials, plus that accumulated in the third trial, the RO system is estimated to cost $6,200,000 for 7950 m^3/day (2100 M gpd), with an operating cost of $22.00/t ($20.00/ton). The FC unit is projected to cost $1,400,000 with an additional operating cost of $2.00/t ($1.80/ton).

Based on these field trials, it can be concluded that:

- Reverse osmosis is a relatively expensive, but an energy efficient way to concentrate dilute bleach effluents.
- Freeze concentration is technically feasible but needs much work to overcome many mechanical problems. It also is energy efficient relative to evaporation.
- Water usage in bleach plants needs to be reduced considerably if RO/FC is to be economically viable.
- Much work needs to be done to extend RO membrane life, as short life is a major contributor to the high operating cost.

Unlike freeze concentration equipment, the reverse osmosis equipment was reasonably trouble free. Advances in membrane technology may, in the future, brighten the economic picture for RO in the pulp and paper industry, but at the present time, it is an expensive method to concentrate wastes prior to final disposal. Reduction in water usage to at least the 21 m^3/t (5000 gal/t) level will also be necessary if reverse osmosis is to be economically viable.

SECTION 2

RECOMMENDATIONS

Reverse osmosis and freeze concentration are technically feasible means of concentrating bleach plant process waters at reasonable energy consumption levels. High capital and operating cost prohibits their use for economically treating the large bleach plant effluent volumes which prevailed in most of the industry in 1975-76. To make these processes economical, work in the following areas is necessary:

- Development and application of technology to reduce bleach plant water consumption to levels of 21 m^3/t (5000 gal/ton) or less;
- Development of membranes which have long life (greatly in excess of 2 years) and can withstand high temperature conditions;
- Development of membranes which can withstand large pH variations;
- Improvement in the reliability of the multi-stage freeze concentration processes.

SECTION 3

INTRODUCTION

New ways of achieving high efficiency processing systems, using less water for bleaching of wood pulps, and for better and less expensive methods of treating bleaching effluents are the subject of intensive research and engineering development programs within the pulp and paper industry. This project evaluates reverse osmosis (RO) and freeze concentration (FC) systems as new tools for concentration, separation, and disposal pretreatment of the dissolved materials in bleaching process waters. It is also directed to the recovery of high quality water for reuse with some potential in energy savings.

The bleaching of cellulose pulp for the manufacture of paper and the various other products requiring refined cellulose fiber has traditionally used large volumes of water to dissolve and wash away the residual lignin and other components remaining in the washed brownstock from pulping processes. Usage has ranged to 50,000 gallons of water per ton (200 m^3/t)* of bleached pulp, although 10,000 to 20,000 gallons per ton (38-76 m^3/t) may be considered more representative for bleaching systems constructed or modernized since 1965. The development of methods for substantially decreasing this requirement for such large volumes of water has become an important objective in improving the efficiency and economics of bleaching technology. This has become especially critical since 1970 when standards for effluent quality were established.

A typical CEDED (chlorine-extraction-chlorine dioxide-extraction-chlorine dioxide) sequence for bleaching kraft, softwood pulp, with 7% loss in yield (shrinkage), dissolves about 140 (63 kg) pounds of wood derived organics, plus roughly equivalent quantities of inorganic residues from bleaching chemicals, in the 10,000 to 20,000 gallons (38-76 m^3) of bleaching process effluents for each ton of bleached pulp. The large monetary expenditures for construction and operation of equipment which may be required to achieve effective treatment and disposal of high volume dilute effluent waters are critical in the economics of the bleaching process.

Various ways of treating these dilute bleaching effluents have been under development in recent years. Such development studies have usually first been directed toward reducing or eliminating specific environmental quality problems resulting from these waste waters. Treatment to remove the

*For the reader's convenience, standard English units are used, with SI units in parentheses.

dark colored compounds, particularly those from the caustic extraction stage of bleaching, has been one of the first organized research objectives to reach commercial-scale installation and practice. Removal of components contributing to suspended solids and biochemical oxygen demand (BOD), and the elimination of materials toxic to aquatic life have been other specific areas for research and development. Processes for removing color, such as lime precipitation, provide only partial removal of the BOD. Conventional primary clarification and secondary biological treatments are capable of substantially reducing the content of suspended solids, the BOD, and may also reduce some toxicity, but these treatment systems have little effect upon removal of inorganics and of color associated with lignin derived organics contained in these waste flows.

Another objective in developing improved methods for treating bleach effluents is achieving reductions in the cost of chemicals and of energy used in the bleaching process. A typical 500-ton/day (453 t/day) kraft mill, employing the CEHDED (chlorine-extraction-hypochlorite-chlorine dioxide-extraction chlorine dioxide) bleach sequence for softwood, in 1971 was estimated to use chemicals costing $6,945 each day (Dr. F. Kraft, personal communication). Data derived from a nomogram prepared in April 1976 by Heitto (1) indicates this daily chemical cost for a 500 tpd (453 t/day) bleaching operation would have increased to $13,850 at lower levels of chemical use and to $17,700 per day for bleaching systems having higher levels of chemical use. Heitto's nomogram also estimated the total energy range for heat and power from $5,500 to $9,850 daily in the 500 tpd (453 t/day) mill. The continuing rise in the cost of energy is expected to substantially increase the costs for both chemicals and energy, since about 50% of the cost of chemicals derives directly or indirectly from the use of energy.

The energy based cost savings which may derive from in-plant recovery and regeneration of chemical residues from bleaching (and also pulping chemicals carried over in the brownstock) provide one route to cost reduction. Substantial economics in energy usage may arise from further increases in the recirculation of process waters within the bleaching system, and also from reduced requirements for out-plant treatment processing of the bleaching effluents.

SECTION 4

OBJECTIVES AND ORGANIZATION

OBJECTIVES FOR THIS PROJECT

This project evaluated reverse osmosis and freeze concentration as new tools for achieving the objectives of effective treatment and disposal of bleaching process residues by:

(a) Concentration of the dissolved solids contained in bleach process water flows.
(b) Reclamation and recycle of clean, reusable process water.
(c) Increasing the degree of recycle and closure of bleaching process water systems.
(d) Possible reduction in the overall requirements for use of energy.

THE PROJECT PLAN — CONCEPTUAL DEVELOPMENT

Exploratory studies of reverse osmosis concentration of dilute pulping spent liquors had been under way since 1968, and were reported for EPA Project 12040 EEL-02/72 (2). Preliminary discussion and evaluation with mill representatives were initiated in 1971. In this new treatment concept, water volume reduction within the bleach plant, already a growing trend within the industry, was considered to be an important first step. A desirable preliminary goal for achieving the objective of this project was based upon reducing water usuage to about 6000 gallons per ton (25 m^3/t) of bleached pulp. This would give a total dissolved solids content of approximately 0.5% in the total effluent discharged from a kraft bleaching system. The flow sheet then incorporated a reverse osmosis concentration step to recover reusable water and to reduce the volume of the bleach effluent by a factor of about 10 to 1. The resultant preconcentrate of the recycled bleach effluent, in the range of about 5% dissolved solids, would then be concentrated to over 30% solids by standard evaporation systems to obtain a combustible product. Fluid solids incineration was considered to be an especially promising route to recovery of an ash having a high content of NaCl. The crystalline salt could then be separated and made sufficiently pure for use in regeneration of bleaching chemicals. The logic of this approach continues to be of interest, but interviews with experienced bleach plant operators at several mills in 1971 and 1972 indicated the need for substantial levels of process refinement to reduce both capital and operating charges for these concepts.

This project has been developed especially to obtain more complete information about the capabilities of RO systems for concentrating bleach effluents, with inclusion of FC, as alternatives to conventional evaporation and combustion systems. Field trials were undertaken for concentrating bleach effluents produced at three pulp mills, each utilizing different methods of chemical pulping and bleaching. The first field trial was conducted at an older mill in Northern Wisconsin utilizing the calcium-base acid sulfite process with a 2-stage, H-H, bleaching system. The mill cooks and bleaches both hardwood and softwood separately. The second trial was conducted at a modern kraft pulp mill in Augusta, Georgia, for which the CEHD sequence is used on a softwood pulp bleaching line. The third field trial was conducted on a hardwood bleach line at an alkaline kraft mill at West Point, Virginia, which employs the D/C-O-D bleach sequence. This oxygen bleach process comprises one of the more recent and important advances in bleaching technology.

Substantial reduction in the volumes of process water used in bleaching is an essential step preliminary to the use of any of the relatively expensive systems available for concentrating and removing the daily input of wood organics and chemicals solubilized in the bleaching process. Preference was originally directed to process water volume reduction by in-plant, jump stage, recycle of the more dilute flows from the later stages of bleach washing back to the corresponding preceding stages of bleach washing. Histed and coworkers (3) have developed advanced concepts for this important first step of countercurrent process water recycle to achieve bleach process water volume reduction. With the volume of fresh water input and effluent outflow reduced to the order of 6000 gallons for each ton (25 m^3/t) of bleached pulp, it becomes feasible to undertake development of a secondary step of water volume reduction and for concentration and separation of the solubilized wood and chemical residues. This project has been principally directed to laboratory and field trial studies for the secondary step of concentration of the solubles by use of tight, high rejection RO membranes. Freeze concentration was then evaluated as an additional third step of concentration beyond the osmotic pressure limitations for reverse osmosis and as an alternative to the conventional multistage evaporation systems.

Concentration of the volumes of flow to one-tenth of the recycled volume being fed to the RO plant has been extensively studied in these field trials. Ninety percent of the water content of the Bleach Plant Effluent (BPE) feed to the membrane system could readily be recovered as a clear, colorless product water of quality readily capable of being reused in the mill operations. Subsequent processing of the resulting concentrate at 5 to 10% solids content was then undertaken to achieve further concentration by the innovative use of the principles of freeze concentration. This final concentration step seems capable of producing a product ranging to 25% solids or even more. Such a concentrated product could, of course, be burned as in the process developed for the effluent-free process conceived by Dr. Howard Rapson (4). However, an additional step of FC to remove additional water up to 30% solids or more has been evaluated in laboratory studies. Still another step of FC to the point of eutectic freeze crystallization of a clean salt product has been proposed as subject for further study in a following research effort. Other routes to concentration and recovery of clean salt or heavy brine of sufficient purity for electrolytic recovery of the bleaching chemicals comprise additional areas for evaluation and development in proposed follow-up research programs.

This report concludes with a preliminary evaluation of the various alternative methods of concentrating these dilute bleach wastes, and for possible disposal of the final concentrates. More detailed studies and cost evaluations require further studies in the areas of particular promise. Possibilities for recovery of NaCl for regeneration of bleaching chemicals and of pulping chemicals from brownstock carryovers are suggested. Recovery of organic residues or derivatives such as oxalic acid from the bleaching process reactions, could comprise additional and significant routes to cost reduction and to economic feasibility for use of these new processing tools in the bleach plant.

DISCUSSION OF THE LOGIC FOR USE OF VARIOUS TYPES OF MEMBRANE SYSTEMS

Reverse osmosis, sometimes referred to as hyperfiltration, has been chosen as a logical first stage for dewatering of the bleach recycle waters and in achieving the complete degree of treatment of bleach plant effluents desired in this research study. The choice is based on several years of experience (5-13) with not only RO but also with UF and electrodialysis systems in the laboratories of The Institute of Paper Chemistry. The Institute experience specifically on pulp and paper process waters supplements the experience on salt water conversion, concentration of fruit juices, dairy products, pharmaceuticals, and other substrates being developed in other research centers.

Ultrafiltation is well recognized to have advantages of processing large volumes of feed liquor at high rates of permeation per square foot of membrane. In the case of bleach liquors, however, the low molecular weight compounds, and particularly the chloride salts, pass through the membrane. There are situations where the loss of salt may actually be advantageous, or at least of no concern from the pollution standpoint, for example, the discharge of salt containing effluents directly to the sea or to tidal waters and estuaries. Dissolved salt has little or no adverse effect on flux rates of an UF system. But in the case of RO, direct losses in flux rates occur with rising osmotic pressure of salt solutions being concentrated. However, in our experience, fouling problems by large molecular weight lignin products have been found to be substantial and, at times, nearly irreversible, with open ultrafiltration membranes. Table 1 summarizes and compares some of the advantages and disadvantages inherent in the two membrane systems of RO and UF.

Reverse osmosis of bleach liquors can best be accomplished with membranes having relatively high levels of rejections for salt. This is particularly so when starting with solutions below 1% salt content, such as recycled bleach effluents which range from 0.4% solids to 2.0% solids. Universal Oil Products #520 and closely equivalent Rev-O-Pak #95 membranes were chosen for use in this project. It had been found that up to 90% of the water could be removed relatively salt free when concentrating up to about 5% solids. Such permeates were clear, colorless, and capable of being reused within the mill. Salt could be concentrated by RO to levels of 2% to as much as 5%, but at decreasing efficiency in terms of rejection and flux rates as the concentration of salt rose above 3% NaCl. The tight membranes, capable of rejecting 95% salt or better, also have an interesting characteristic of remaining relatively clean. These are not easily fouled by lignin and other organics present in these wastes.

TABLE 1. APPROXIMATE PERFORMANCE CHARACTERISTICS FOR UF AND RO MEMBRANES*

	Ultrafiltration (UF)		Reverse Osmosis (RO)				
	Open →	Tight	Open →				Tight
NaCl rejection, %	0 →	0-20	Less than 50%	80	90	95	96-98
Mol. wt. cutoffs	100,000 →	10,000	1000 →				50
Pressure range, psi	25 →	250	250 →				500-2000
Flux rate, gfd	250 →	25	50 →				5

*This table presents approximations of comparative performance for various types and grades of membranes presently manufactured or under advanced stages of development by several commercial suppliers and development centers. Comparative specifications are in early stages of standardization for these membrane systems. Molecular weight (or size) cutoffs are seldom specified for RO membranes and may not be justified in this attempt at comparison. Membranes commercially available are primarily cellulose acetate and performance estimations are projected for operation at 35°C after 2 hours processing of appropriate substrates.

Further reference to Table 1 discloses several significant advantages of the UF membrane system. The higher levels of water flux through the membrane reduce the capital charges for equipment to process each thousand gallons of feed water. Freedom from the need to use high operating pressures to overcome the osmotic pressure of NaCl or other salts in bleach liquors results from free passage of these low molecular weight (size) molecules through the membrane. Disadvantages result from the inability of the more open UF membranes to reject salt and the tendency to foul.

Electrodialysis, another membrane processing system accomplished with the use of ion selective membranes, has the capability of producing relatively clean solutions of NaCl free from nonionized materials. However, there are limitations to electrodialysis as a first stage concentrating system for processing solutions containing lignosulfonic acids and related wood residues. These organics can contribute to severe fouling and greatly reduce the current density and overall efficiency of the electrodialysis process. The electrodialysis system was not studied in this project but could serve as a possible method for separation and recovery of clean NaCl brines for regeneration of bleach chemicals after RO or UF or both.

As the work proceeded and the concepts further developed, it became increasingly apparent that significant "short cuts and economic advantages" might result from using a combination of these processes for developing concentration and fractionation routes to the complete processing of bleach plant effluents. These concepts are further discussed and developed in the concluding sections of this report.

COOPERATING MILLS AND ORGANIZATIONS

Development of the program of field testing at representative bleach plants was initiated with a preliminary survey especially directed to identifying suitable sources of feed liquors. These liquors were derived from countercurrent recycle operations which had the goal of reducing fresh water usage to 6000 gallons of water per ton (25 m^3/t) of bleached pulp or less. A number of mills had closely approached that criterion, at least experimentally, on short-term runs, but few were in a position to provide feed liquors from such operation on a sustained basis. Two mills were selected for the initial program and a third mill using oxygen bleaching system was later added to the program in an extension of the project.

FUNDING

The program, as initially developed, was undertaken by The Institute of Paper Chemistry in cooperation with the U.S. Environmental Protection Agency and Avco Corporation under a joint funding program at the level of $318,742. Of that total, $150,000 was a grant from the U.S. Environmental Protection Agency, $64,291 was funded by The Institute of Paper Chemistry, $44,451 was funded by Avco Systems, and the two original cooperating mills contributed services of $30,000 each. The original grant award became effective February 12, 1975. Preliminary laboratory studies were initiated to establish performance expectations at each mill and to develop optimum arrangements for processing the liquor at each individual installation. Those preliminary studies indicated that very little pretreatment would be required ahead of the membrane system, based upon testing drum quantities and truck load shipments of bleaching effluents shipped into the laboratory and pilot plant center on the Institute campus.

Subsequently, the project was expanded to include the third mill which has been operating the first oxygen bleaching system in the U.S.A. This is located at the West Point, Virginia bleach plant of The Chesapeake Corporation of Virginia. The funding was increased by approximately $120,000, with $50,000 from the Environmental Protection Agency, $20,000 as the mill services commitment from Chesapeake Corporation and the balance funded by the Institute.

SCHEDULES

The field studies at the first test site were initiated early in June 1975. The first field trial was designed to evaluate the possibilities for concentration processing of the bleach effluent from the two-stage hypochlorite (H-H) sequence of bleaching for softwood and hardwood pulps manufactured at the Flambeau Paper Company, Division of The Kansas City Star Company, Park Falls, Wisconsin. The first operational data for the large trailer mounted reverse osmosis and freeze concentration units were taken June 20, 1975 and the 6-week field test program was completed August 1, 1975.

The second field trial, conducted at the bleached kraft mill of the Continental Group Inc., Augusta, Georgia, was scheduled to start early in September 1975 after the two trailer units had been returned to their home bases in Appleton, Wisconsin and Wilmington, Massachusetts for cleanup and minor alterations indicated to be desirable from experience gained in the first trial. The second trial at the kraft bleach plant in Augusta, Georgia was substantially completed in mid-October 1975, but was later resumed for one week in mid-November to obtain a 5000 gallon (19 m^3) supply of preconcentrate to be further processed in Appleton.

The extension of the field test program to the third mill at West Point, Virginia (Chesapeake Corporation) was initiated early in the month of April 1976. A 3-week run was completed April 28, 1976. One thousand gallons (3.8 m^3) of concentrate from this oxygen bleaching field trial were shipped to the Institute for continuing studies for high level concentration and for recovery of NaCl during the month of May. Laboratory and pilot RO studies were concluded May 28, 1976 and FC studies on a substantial shipment of RO preconcentrate were completed about June 15, 1976 in the Avco pilot facilities at Wilmington, Massachusetts.

A NOTE ON NOMENCLATURE

For the convenience of the reader, the units used throughout this report are those currently used in the industry. SI units, or SI derived units are enclosed parenthetically after the English units. Appendix A contains an abbreviated list of factors for converting the English units to SI or SI derived units. A list of the common abbreviations is also included.

SECTION 5

THE MEMBRANE PROCESS AND EQUIPMENT

GENERAL

The first two large-scale field trials were conducted with two trailer mounted pilot units, one for the reverse osmosis preconcentration and the second for freeze concentration to higher levels of solids and salt concentration. The two trailers are shown on-site in Figure 1.

Several years of experience with the trailer mounted RO unit and the smaller test unit have shown these units usually require no more pretreatment than can be provided by a simple vibrating screen. This proved insufficient in the case of the first field trial at the Flambeau mill due to the high content of suspended talc, which was used at rates of as much as 3 tons/day (2.7 t/d) for pitch control. At that first test site we were forced to set up a make-shift clarifier operation to remove the talc with use of a Sven-Pedersen flotation saveall converted to a settling basin. No pretreatment was required for processing flows from the bleach system at the Continental Group, Inc. plant in Augusta, Georgia. The bleach washers on the softwood line at this mill operated with high levels of fiber retention. A very minor loss of fiber was indicated throughout the 6-week period of operation and no operating problems due to suspended fiber were apparent in the RO system. Some relatively small amounts of fiber and also of a precipitate in the RO preconcentrate held for feed to the FC unit were cause for frequent replacement of small capacity string filter media ahead of the freeze concentration unit.

THE MEMBRANE MODULES

This project benefited substantially from the availability of the large portable RO field test unit constructed in 1968 for processing pulp wash waters in volumes ranging from 20,000 to 70,000 gal/day (76-265 m^3/day). The trailer mounted reverse osmosis unit has been described in detail in prior publications, and particularly in the final report for EPA Project 12040 EEL 02/72 (2). The manifolding and pumping system for this large unit are capable of being adapted to quite a number of different modular concepts for membrane systems. Experience gained in studies over a 10-year period continued to favor the use of the 1/2-inch tubular (1.3 cm) configuration for the membrane support structure. The hollow-fiber, spiral wound or plate and frame configurations experienced fouling problems arising from formation of precipitates and crystalline deposits containing large molecular weight lignin and other wood chemical residues. Suspended solids and sediments develop in these process waters with increasing concentration, but deposition and fouling is

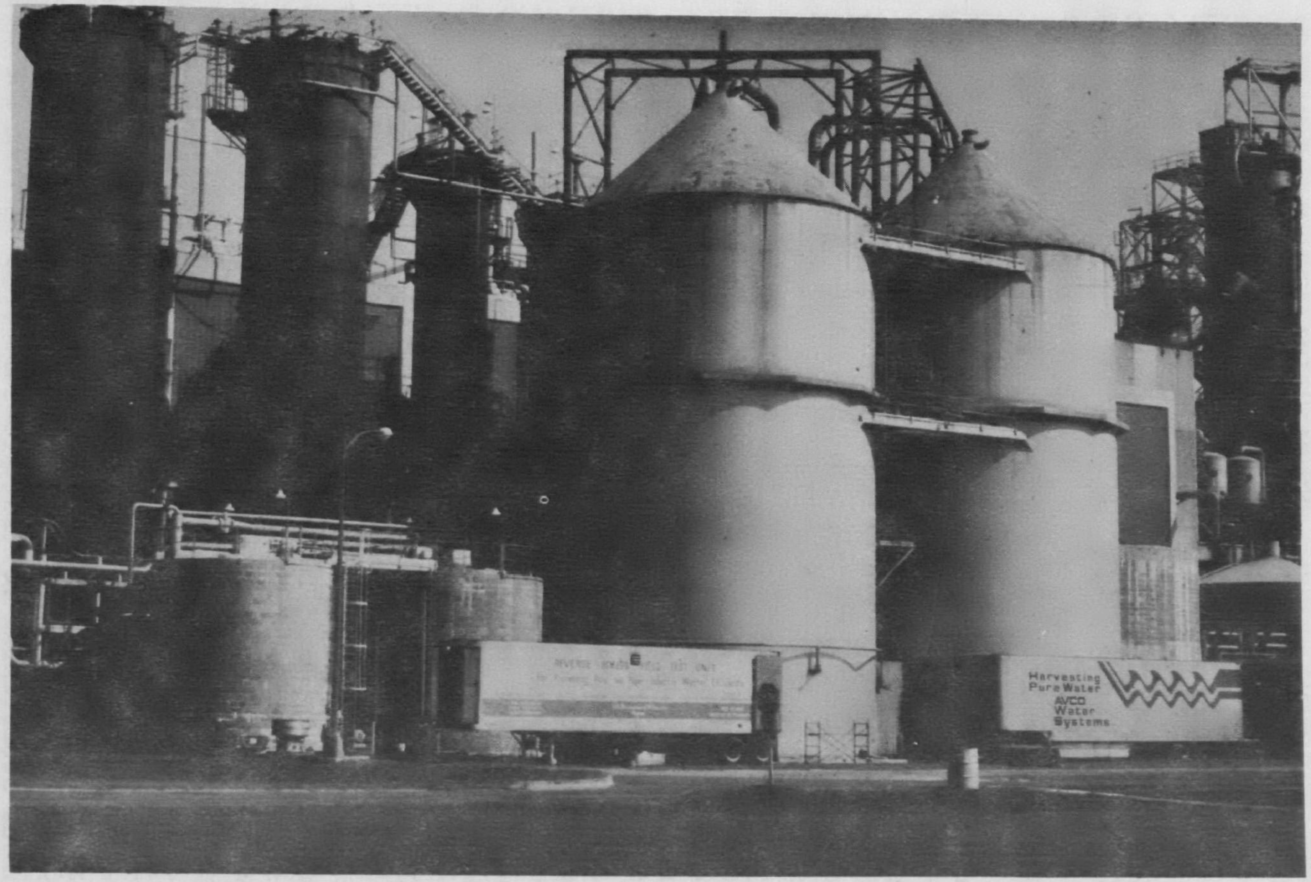


Figure 1. Two trailers on site at Augusta, Georgia.

prevented or minimized by the high velocities maintained across the membrane surface in the tubular design of the reverse osmosis modules. Earlier studies for EPA Project 12040 EEL (2) had well established the need for maintaining velocities of 4 feet per second (1.2 m/s) at higher concentration, particularly above 2% solids.

Two tubular designs had been subject for a continuing membrane life study in independent programs carried out over a 3-year period prior to initiating this project. The 1/2-inch ID (1.3 cm) fiberglass tubular support structure, manufactured by Universal Oil Products Company (UOP), and the 5/8-inch OD (1.6 cm) ceramic tube support structures, designed and manufactured by the Rev-O-Pak Division of Raypak, Inc. (ROP), had proven to be particularly well adapted to maintaining relatively clean membrane surfaces. Design of the UOP tubular module with 16.7 ft^2 (1.55 m^2) of membrane is shown in Figure 2 and the ROP 7 core cell with 10.5 ft^2 (0.98 m^2) of membrane is presented in Figure 3.

Importantly also, these two systems had been improved to the point where they have proven reliable and free from mechanical failures. With the exception of several ceramic tubes broken on the 1100-mile (1800 km) trip to the field test site, at Augusta, Georgia there were no mechanical or membrane failures for any of the 300 modules, nor for any of the nearly 5000 individual tubular cores within the modules over the one year of intermittent service on this project. This is a remarkable improvement over the structural failures so frequently experienced with tubular membrane equipment manufactured and tested prior to 1973.

THE PRELIMINARY LAB TEST UNITS

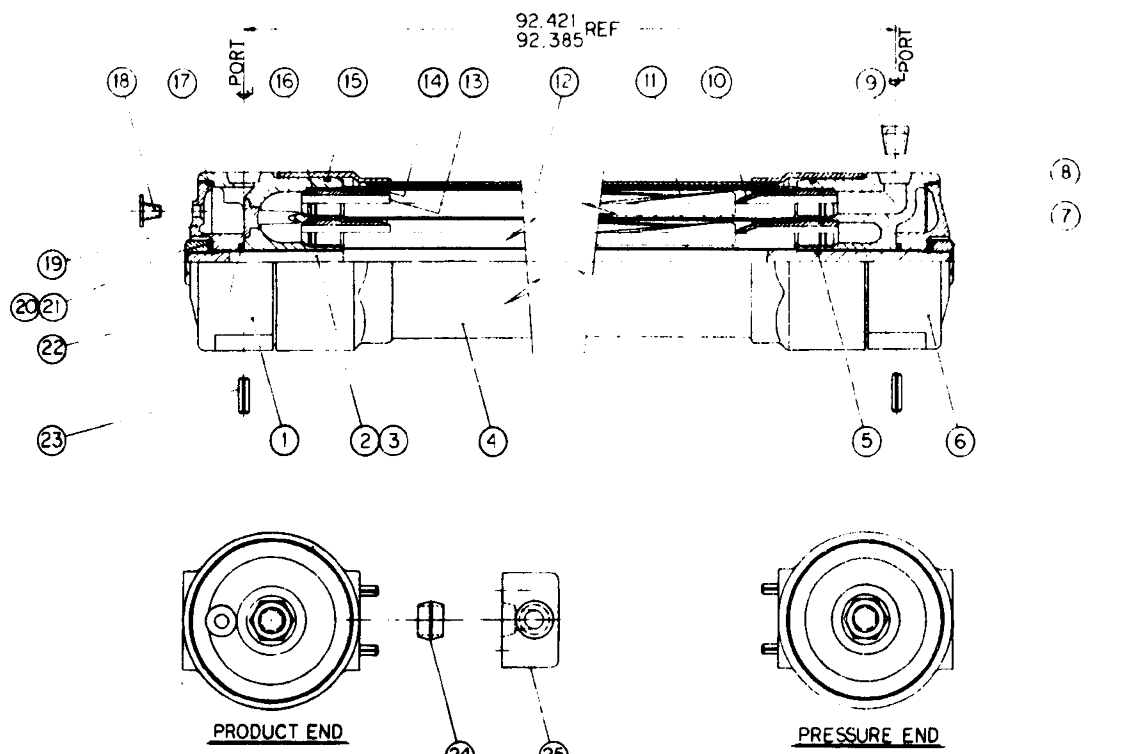
Several different laboratory and small-scale pilot units were utilized in the preliminary testing program to develop a program for the large field test unit. For each trial, 5-gallon (18.9 l) carboys of the bleach liquor were first subjected to laboratory study, with the first membrane test conducted with single UOP or ROP test units and then followed by 50-gallon (189 l) drum-scale tests with several modules operated over one or more days of recycle testing to establish fouling and flux rate patterns. The final large-scale tests, utilizing part of the trailer unit with 10 or more modules, were carried out with a 5000-gallon (18 m^3) truck load of liquor from each of the first two mills participating in the field trials.

The small laboratory units utilized duplex piston pumps capable of operating at closely controlled flow rates in the 1 to 5-gpm (3.8-18.9 l/min) range and at pressures ranging to 800 psi (5.5 MPa) and more. These units have been described in prior publications (2).

For the ROP 7 core cells, it was necessary to use another test stand equipped with a multiple stage centrifugal pump capable of delivering flows of 10 to 25 gpm (37.8-94.6 l/min) and at pressures of 600 to 700 (4.1-4.8 MPa) psi. This unit, as modified for the Chesepeake field tests, is described in a following section.

MODULE ASSEMBLY

No. 100A



Item No.	Description
25	Module connector
24	Grommet connector
23	Roll pin 3/16" dia. x 3/4"
22	"O"-ring
21	Washer, flat 1/2"
20	Washer, flat 1/2"
19	Cover prod. head
18	Plug 201 A
17	"O"-ring
16	"O"-ring
15	"O"-ring
14	Compact sleeve
13	Tube adapter
12	Tube
11	Rod (VDR) volume displ.
10	Tube adapter
9	Plug K-8
8	Cover-press. head
7	Hex nut 1/2"-20-2B
6	Pressure head
5	"O"-ring
4	Shroud assy.
3	Strain rod
2	Strain rod
1	Product head

Figure 2. UOP reverse osmosis module.

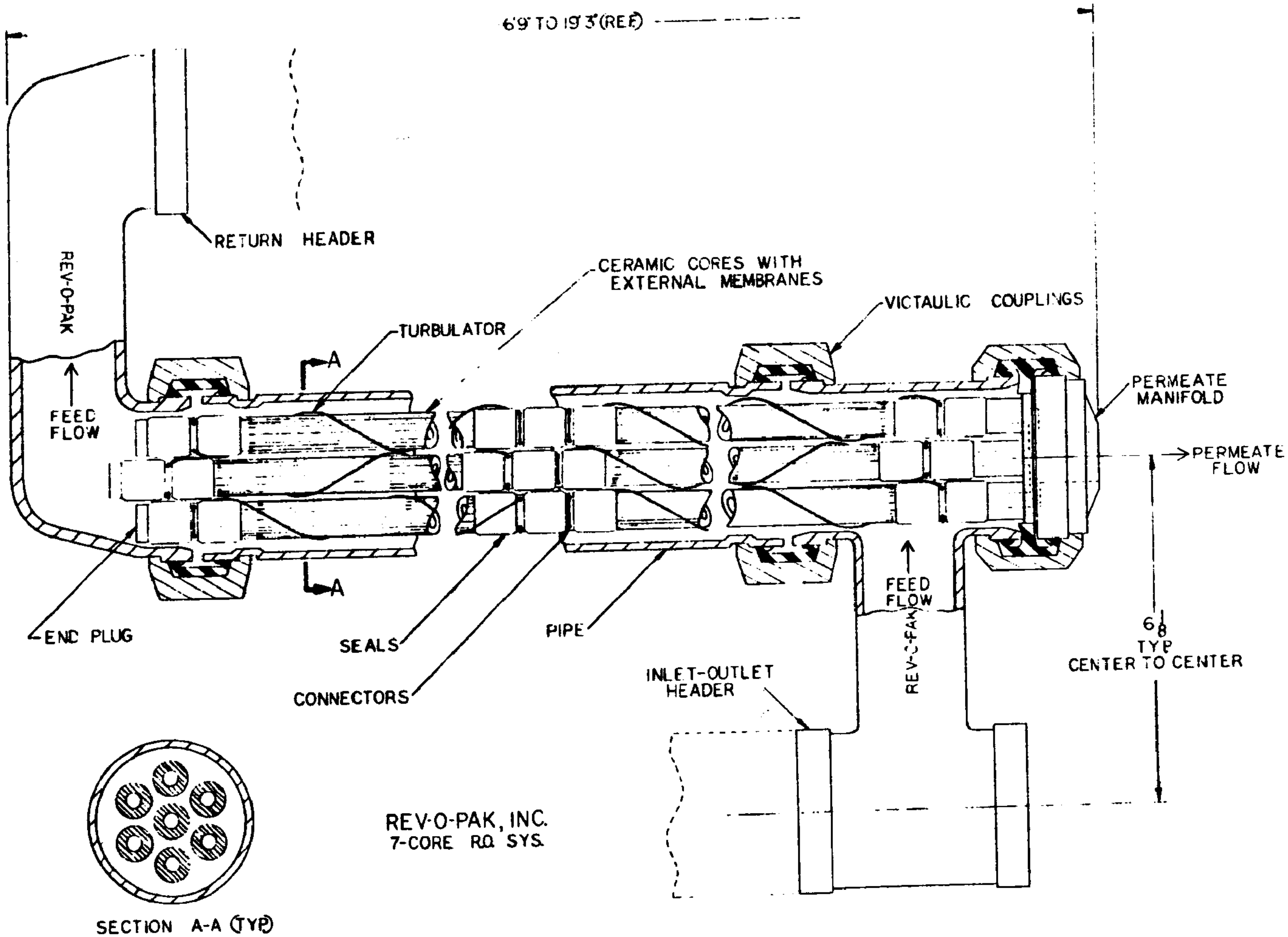


Figure 3. Rev-O-Pak reverse osmosis module.

THE RO TRAILER MOUNTED FIELD TEST UNIT

The RO trailer mounted field test unit was designed around a large, 3-stage, piston pump capable of delivering flows in the range of 10 to 70 gpm (37.8-265 l/min) at pressures to 1200 psi (8.2 MPa) and supplemented by three centrifugal recirculation pumps adapted to inlet pressures above 500 psi (3.4 MPa). The flow pattern in Figure 4 for the manifolding system was adapted to the needs for a combined operation with two different types of tubular modules. The ceramic ROP cells require flow rates in excess of 10 gpm (37 l/min) to each individual cell. These tests were programmed to be operated with pressurized feed flows in excess of 30 gpm (114 l/min) to the several module banks. The ROP cell, with flows external to the tubular membrane support structure, had the advantage of low levels of pressure loss and a large number of modules could be operated in series. Fouling was readily apparent if the flows to these cells were permitted to drop below the 10 gpm (38 l/min) level, but operations were relatively trouble-free at flows ranging above 10 gpm to 20 or more gpm (38-76 l/min).

In contrast, the pressure loss was higher in the UOP tubular conformation which provides for internal flows in tubes of 1/2-inch inside diameter (1.3 cm) and with tight U bends of less than 1/2-inch diameter (1.3 cm). The UOP modules could not be efficiently operated with more than two modules in series because of the high level of back pressure generated at the rates of flow required to maintain velocities of 4 ft/sec (1.2 m/sec). The relatively low rates of flow found to be feasible for operating the UOP modules require a more complex manifolding system, but the overall performance of the two conformations of module design by UOP and by ROP were substantially equivalent when operated in accordance with manufacturer's recommendations.

The less expensive fiberglass tubular structures in the UOP module were found to be especially well adapted to removing 70 to 80% of the permeate water from the feed liquor while processing the more dilute flows having low osmotic pressure (20 to 200 psi — 138 kPa to 1.39 MPa) from around 0.5% solids up to 2.5% solids at operating pressures below 650 psi (4.48 MPa). At levels of concentration above 2.5% solids, operating pressures above 650 psi (4.48 MPa) were required to overcome osmotic pressures ranging to 500 psi (3.45 MPa) or more. The more expensive ROP units, capable of maintaining high levels of performance, were advantageous at the elevated pressures in the final stages of the concentrating process.

THE CHESAPEAKE UNIT

In contrast to the trials at the first two mills, the trial at the third mill, Chesapeake Corporation, was conducted on a smaller RO unit. This was done because extensive redesign of the manifold system of the larger unit was required. This would have led to excessive delays and project costs.

A smaller RO unit using a total of 22 modules, including 12 UOP and 10 ROP, was readily adapted from a basic module life test stand which had been extensively used in prior studies. This unit was equipped with a multiple stage centrifugal pump capable of handling flows in excess of 20 gpm (76 l/min) and at pressures to 750 psi (5.17 MPa). Figure 5 is a photograph of

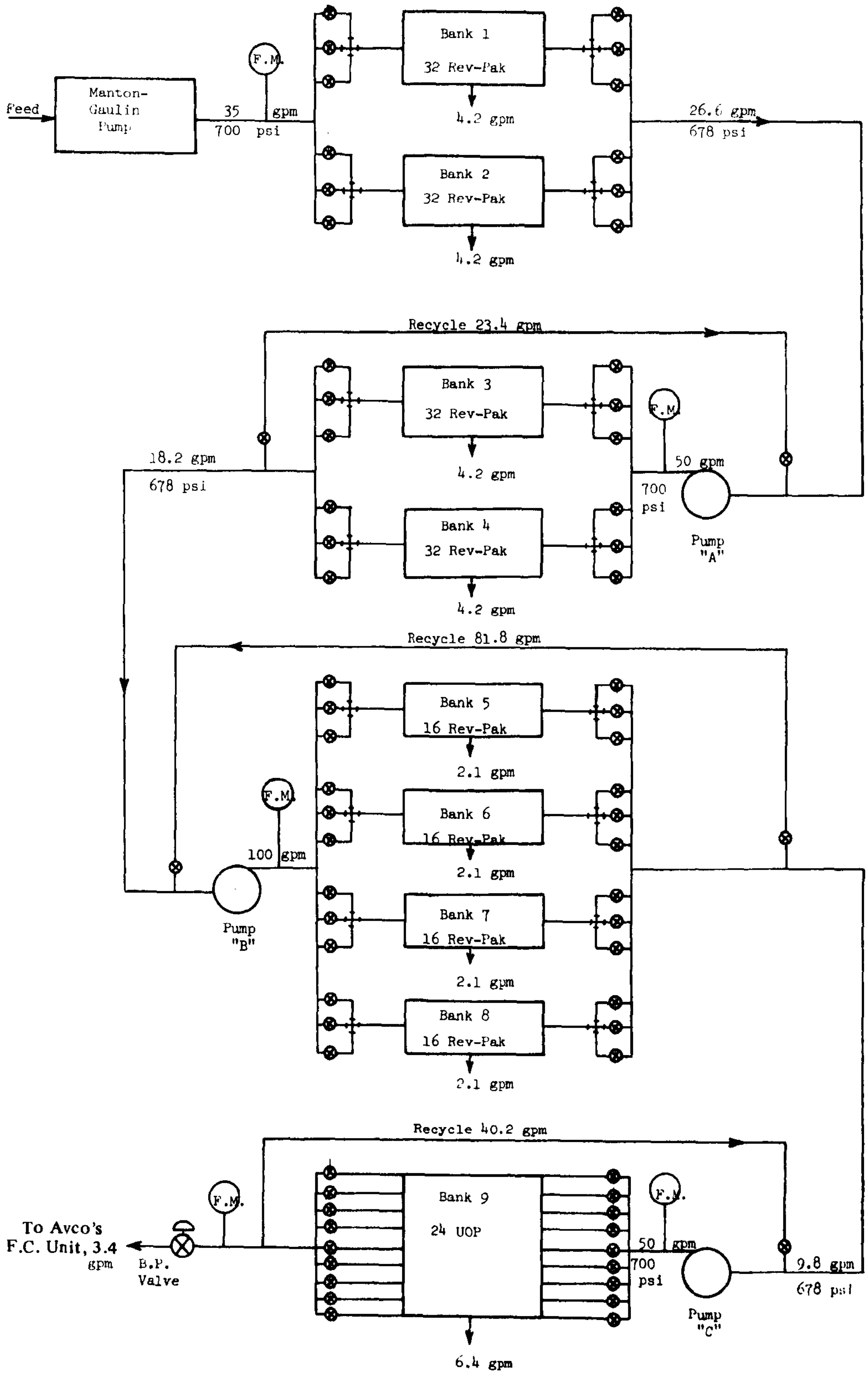


Figure 4. Manifolding system for trailer mounted reverse osmosis unit.

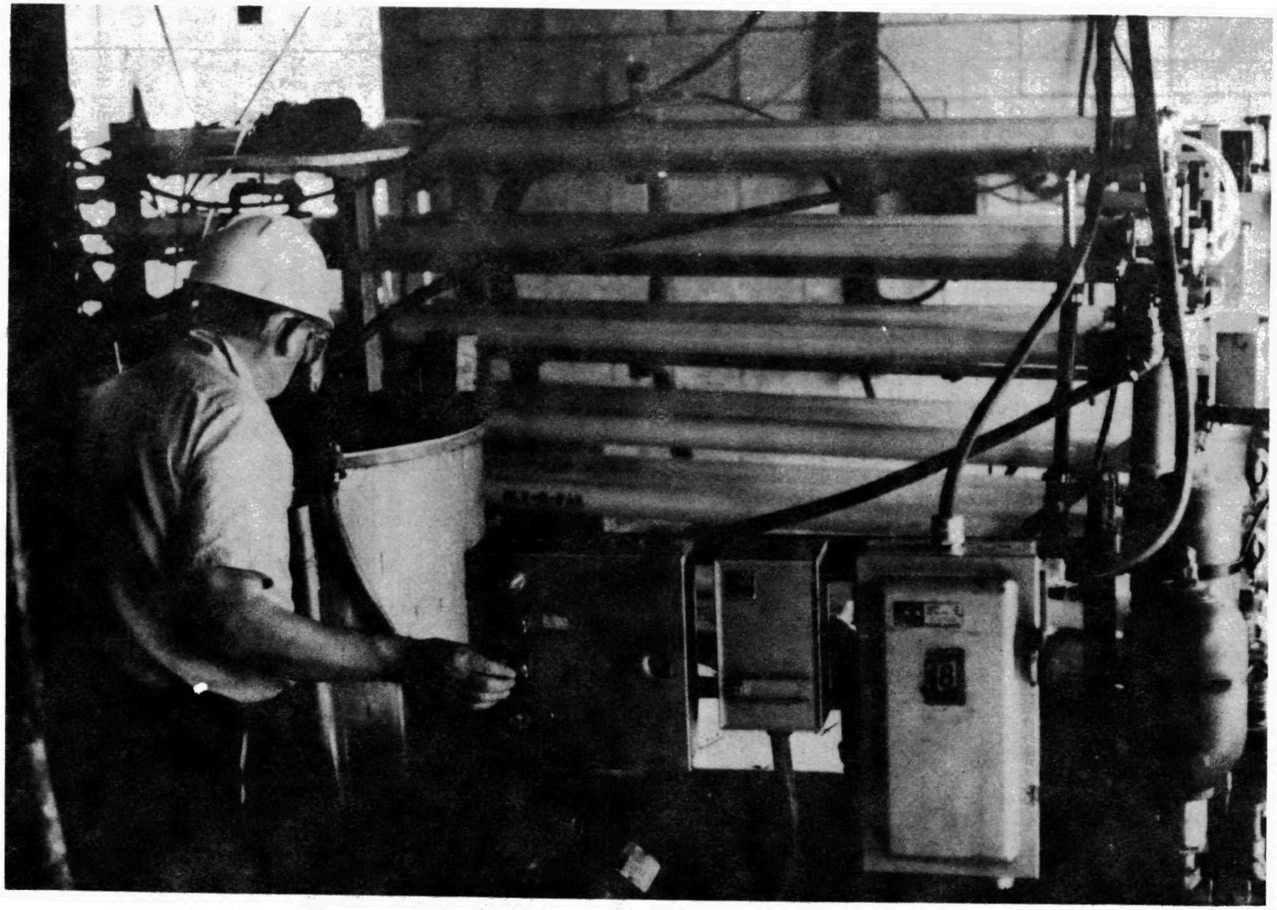


Figure 5. Small RO field test stand used at Chesapeake Corp., West Point, Virginia.

the smaller test unit in operation at the Chesapeake Corporation mill.

Avco also reverted to a more versatile small unit for evaluating the Chesapeake concentrates and permeates forwarded to their Wilmington laboratory for freeze concentration studies.

SECTION 6

THE FREEZE CONCENTRATION PROCESS AND EQUIPMENT*

OVERVIEW

Freeze concentration is based on the principle that when an ice crystal is frozen from an aqueous solution the crystal that is first formed is pure water (14). The impurities in the solution are concentrated in the remaining liquor which surrounds the ice. All freezing processes of a practical nature utilize a direct contact crystallizer (freezer). In the crystallizer, liquid refrigerant is mixed with the solution to be concentrated. The vapor pressure above the solution is reduced below that of the refrigerant causing the refrigerant to flash. By flashing, an amount of heat equivalent to the latent heat of vaporization for the refrigerant is withdrawn from the water to be frozen, thus forming the ice. The ice takes the form of discrete platelets of 50 to 1000 microns in diameter and about two-tenths of that in thickness.

One other very important step is necessary to achieve separation of fresh water and concentrate; that of washing the ice crystals using a portion of the product water. The majority of the energy consumed in the process is associated with ice formation. In order to reduce the energy requirements of the process, a vapor compression cycle is used in which the refrigerant which is withdrawn from the crystallizer is compressed and then condensed by the washed ice. This accomplishes the melting as well as reduces the pressure difference over which the refrigerant must be compressed. Significant energy savings are also affected by utilizing a feed heat exchanger in which the solution to be concentrated is cooled by the outgoing concentrate and fresh water streams. The basic process is illustrated in Figure 6.

The freeze concentration process has several inherent advantages:

1. Low Energy Consumption — Compared to multiple effect evaporators, freezing is equivalent to a 20 effect evaporator.
2. Elimination of Scaling and Fouling — No pretreatment (other than perhaps chlorination or defoamer) is necessary. Since the concentration is accomplished in a direct contact reactor where no heat transfer surfaces are utilized, scaling is eliminated. If crystallization of low solubility salts that would normally cause scaling should occur, they form as very fine salts and are carried out of the system with the concentrate.

*The freeze concentration work was carried out by Avco systems, Wilmington, MA. This section is abstracted from their report to IPC.

3. Low Corrosion — Since the process operates at low temperatures, corrosion is minimized. This allows use of lower cost materials and reduced corrosion. Mild steel and aluminum have been shown to be practical for desalination applications.

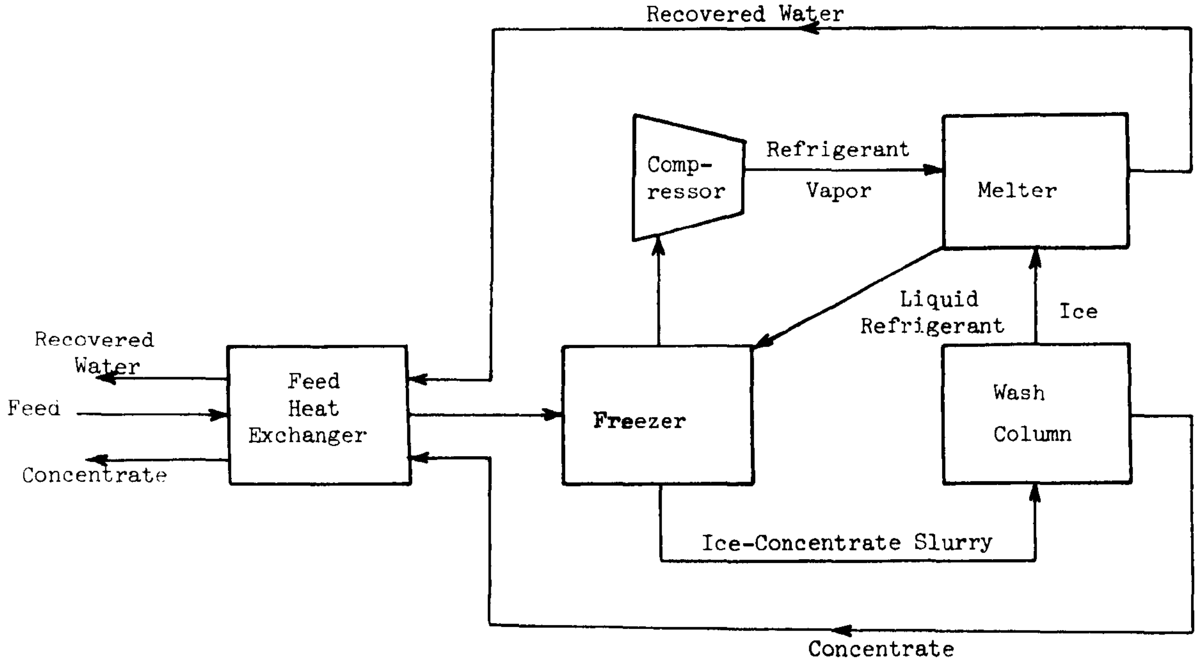


Figure 6. Simplified freezing process.

HISTORICAL EVOLUTION

Serious development of the freezing process began in the mid-1950's, principally by the Office of Saline Water (OSW). Initial process work was carried out on an absorption process (15) in which the refrigerant was water vapor, which, rather than being compressed, was absorbed by an absorbent (lithium bromide). This resulted in the first published work on a wash column for ice, although the device was simultaneously and independently developed by Weigandt (16) and Colt Industries (17). The idea was originally used for the washing of crystals in other chemical processes (18).

As shown in Figure 7, a slurry of ice and concentrate enter the bottom of the column. The slurry, about 15% ice, proceeds upward through the column. At approximately the mid-point of the column, the ice is dewatered by extracting the concentrate from screens located in the column walls. The resulting ice pack, about 50% ice, proceeds upward through the column until it is harvested at the top by a scraper. The ice moves upward through the column, not due to bouyancy, but rather due to the difference in pressure at the two ends of the ice column. This pressure difference results because the concentrate flows through the ice in the lower part of the column at a greater velocity than the ice is moving upward. This causes a pressure drop to be created between the bottom of the ice pack and the point where the concentrate leaves through the screen. This is counteracted by the friction on the walls and the restaining force of the scraper. Washing of the ice is accomplished by applying fresh water (a small portion of the melted ice) to the top of the column. This wash water displaces the concentrate from the interstices of the ice crystals which, when melted, result in nearly pure water. The washing is very efficient, approaching ideal plug flow, using less than 5% of the product. The rate at which the ice can be moved through the column and successfully washed is limited by the permeability of the ice pack, which is proportional to the square of the crystal size. If the ice crystals are too large or too small, problems occur.

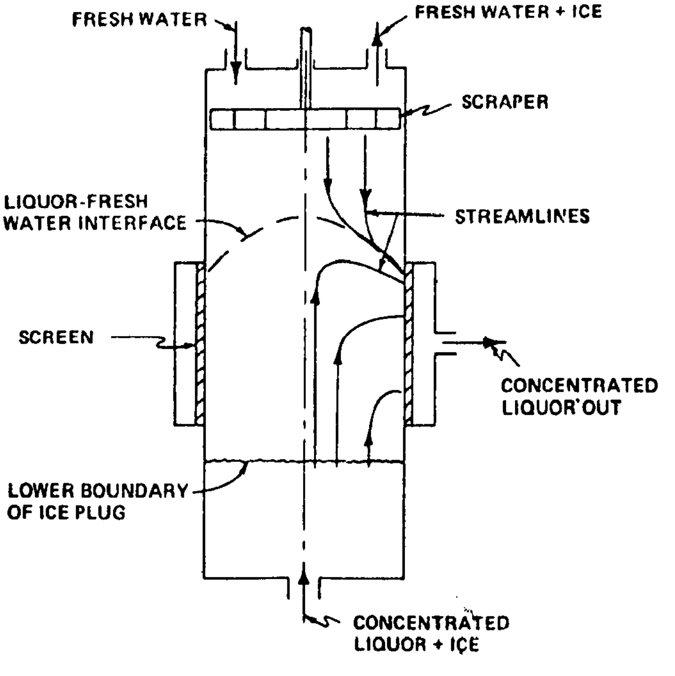


Figure 7. Pressurized counterwasher.

Blaw-Knox and Colt carried out initial development of their processes independently of the Office of Saline Water and there are little published data on their early work. Colt developed the Vacuum Freezing Vapor Compression (VFVC) process while Blaw-Knox developed a secondary refrigerant process. The VFVC process used the water as the refrigerant and therefore operated at relatively low pressures, 3-5 mm Hg absolute. This resulted in the handling of extremely large volumes of vapor and a special compressor was developed (17,19). The necessity to handle the large volumes of vapor limited the practical size of the process to plants of perhaps 1-2 mgd (158-315 m^3/hr). In addition to the development of the compressor, two other significant developments came from the Colt work: 1) a large scale wash column was developed to handle 125,000 gpd (19.7 m^3/hr), and 2) the freezing process was demonstrated to be a practical, reliable, low energy consuming process. Energy consumption of 43 kw/hr/1000 gallons (11 kw/m^3-hr) fresh water was shown on a plant operating at 125,000 gpd (19.7 m^3/hr). Automatic operation was shown over a 2000 hr run (19). Since Colt was considering only the desalination market, which was quite small, further work was dropped in 1970.

The Blaw-Knox process was the first successful refrigerant process. Rather than using the water vapor as refrigerant, a "second" fluid was introduced into the crystallizer. This reduced the volume of refrigerant to be handled by a factor of nearly 100 and enabled much larger plants to be considered practical at least from the vapor handling viewpoint. Butane was used as the refrigerant because of its low cost and desirable vapor pressure properties. They also developed a wash column similar to the one of Colt. Their work was carried out on a pilot plant of 10,000 gpd (1.6 m^3/hr) capacity and never extended to larger sizes.

During the same time period, Struthers-Wells was also developing a secondary refrigerant process under OSW sponsorship (20). They developed a low capacity crystallizer which produced crystals of quite large size, 1000 microns compared to the 200-300 microns of other processes. Their initial work utilized a centrifuge for washing the ice crystals. This approach never succeeded and they switched to a wash column in later years.

Other similar processes have been investigated in England (21), Israel (22), and Japan (23) but no significant differences are noted from the limited literature.

Avco, who performed the freeze concentration work under this contract, has developed a secondary refrigerant process (24) which differs from earlier processes in three areas:

1. Use of a Freon Refrigerant — All previous secondary processes used butane which is toxic and flammable — these are significant limitations especially in relatively small plants where the explosion proof equipment adds significantly to the cost and the hazard is likely to be of concern. The higher cost of the refrigerant (70¢/lb — $1.54/kg) is not of great concern because in either case the refrigerant must be well contained and stripped out of the effluent streams, — in order to meet discharge or safety standards.
2. Indirect Melting — For applications where volatiles are contained in the feed, it is important not to contact the ice with the refrigerant vapor in order to prevent contamination of the product with the volatiles. All previous processes utilized melting of the ice by direct contact of vapor on the ice. This is a satisfactory application for desalination, but not for many industrial applications. The Avco process uses a shell and tube heat exchanger for the melter with a fresh water slurry passing through the tubes and the refrigerant condensing on the outside.
3. Pressurized Wash Column — By applying a higher differential pressure to the wash column the throughput of the column can be increased by up to an order of magnitude. Probstein (25) proposed this approach and Avco has utilized this approach in its process. This results in smaller wash columns.

Avco operates a 75,000 gpd (11.8 m^2/hr) pilot plant at Wrightsville Beach, North Carolina under OWRT sponsorship (26). This plant has demonstrated the features of the process and is providing data for design and commercial plants. Avco is the only company to investigate large scale use of freezing for applications other than desalination and has conducted tests on several industrial solutions (27). These tests have shown suitability of the process to operate on a wide variety of wastes. As a result of this work a two-stage process has been developed (28) which enables higher concentrations to be achieved than in the original single stage process. This has been demonstrated in a 500 gpd (0.08 m^3/hr) laboratory unit and a 5000 gpd (0.79 m^3/hr) pilot plant.

SECTION 7

THREE FIELD TRIALS

I. FIELD TRIAL AT FLAMBEAU PAPER COMPANY, PARK FALLS, WISCONSIN

The Flambeau Paper Company, Division of The Kansas City Star Company, located in Park Falls in Northern Wisconsin, is an integrated pulp and paper manufacturing operation. Production averages about 120 tpd (109 t/day) of bleached calcium sulfite pulp. Cooking and bleaching of hardwood pulps are alternated with softwood pulps in separated flows. The bleaching is carried out in a two-stage hypochlorite (H-H) sequence. The normal flow of bleaching process effluent at this mill was estimated to total about 1,100,000 gallons daily (173 m^3/hr), or about 760 gpm (2.9 m^3/min), and averaging about 9,165 gal/ton (38 m^3/t) of bleached pulp production. Such flows in terms of gal/ton of pulp are substantially higher than would be required for an economical commercial installation and operation of an expensive membrane processing system. However, the solids concentration of the feed liquors available for the field trials was shown to average closely around the desired minimum level of 5 g/liter.

Description of Flambeau Bleach Plant and Material Balance

The two-stage bleaching operation at the Flambeau mill may be described with review of the flow sheet and balance sheet provided in Figure 8. Brownstock is conveyed to the unbleached decker at a rate of 167 pounds (75.6 kg) of fiber per minute, with a moisture content equivalent to 63 gallons of water per minute (0.2 m^3/min). This is slurried with 287 gpm (1.1 m^3/min) of fresh water to provide a flow of 350 gpm (1.3 m^3/min) to the first-stage bleacher. With the addition of 28 gpm (0.1 m^3/min) of bleach liquor, the first-stage bleacher delivers 156 pounds (70.8 kg) of first-stage bleached pulp in 378 gallons (1.4 m^3) of bleach effluent per minute to the drop chest. Two hundred and forty-three gpm (0.92 m^3/min) of first-stage wash water are added to the drop chest, giving a combined flow of 621 gpm (2.4 m^3/min) to the consistency regulator, which received an additional 931 gpm (2.5 m^3/min) of recycled wash water from the first-stage washer seal tank.

The first-stage washer receives 245 gpm (0.93 m^3/min) of dilute recycled second-stage wash water and discharges 1700 gpm (6.4 m^3/min) of first-stage wash, plus recycled second-stage wash to the first-stage seal tank. The overflow from this first-stage seal tank comprises the principal volume of discharge to the mill outfall. This overflow from the first-stage seal tank served as the source of feed to the RO and freeze concentration systems.

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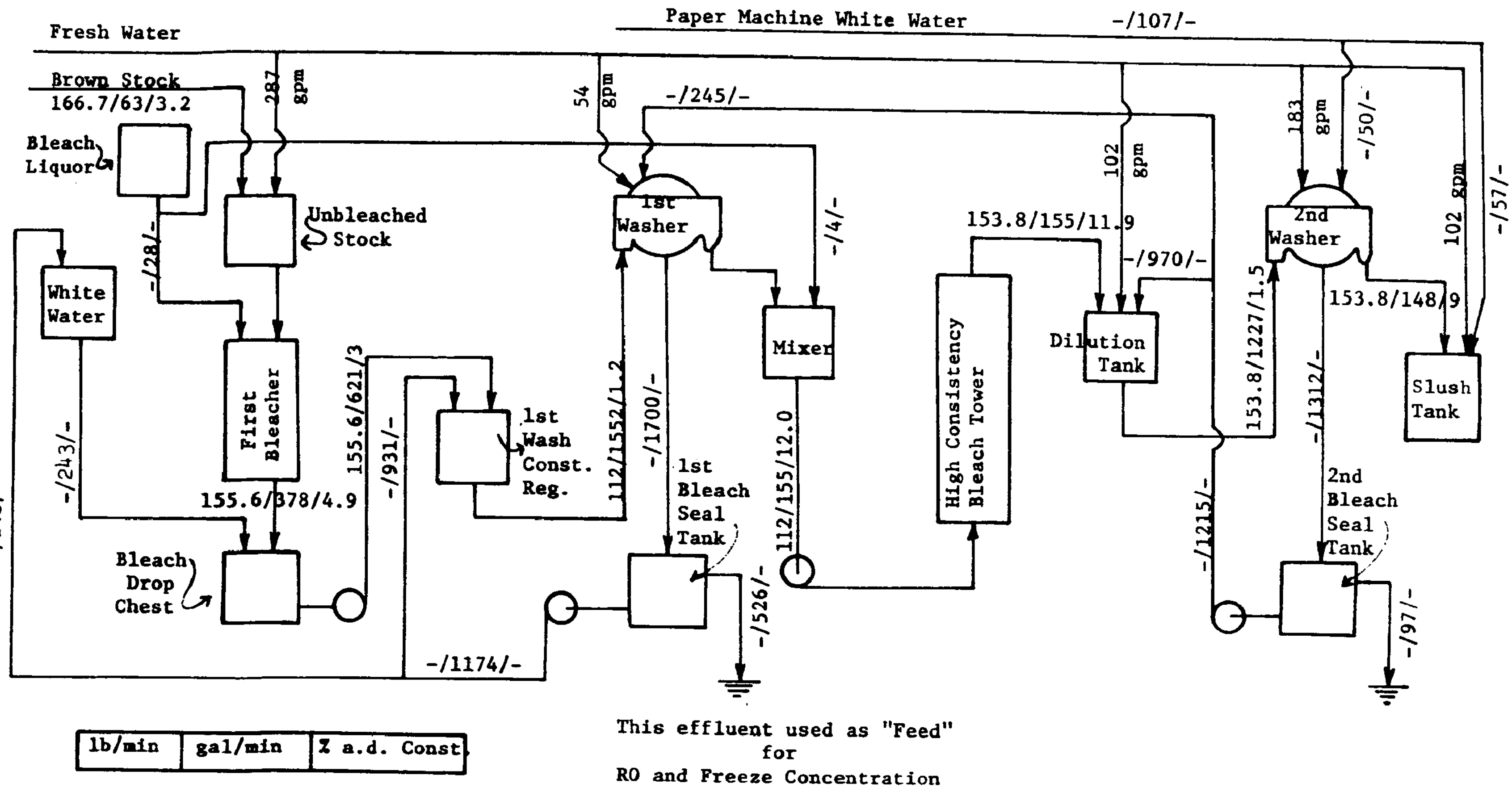

Figure 8. Flow sheet and material balance. H-H bleach sequence for Ca base sulfite pulp mill Flambeau Paper Company, Park Falls, Wisconsin June-August, 1975.

The pulp from the first-stage washer at 156 lb/min (70.8 kg/min) and 151 gpm (0.57 m^3/min) of entrained bleach effluent flow to the second-stage bleacher. Four gallons per minute (15 l/min) of bleach liquor were added in this second bleacher which discharges second-stage bleached pulp, totaling 154 lb/min (69.9 kg/min), with 155 gpm (0.59 m^3/min) of entrained second-stage effluent to a dilution tank receiving 102 gpm (0.39 m^3/min) of fresh water and 970 gpm (3.67 m^3/min) recycled second-stage bleached liquor. This flow to the second-stage washer is washed with 183 gpm (0.69 m^3/min) of fresh water and 50 gpm (0.19 m^3/min) of white water from the paper machine. The final product gives 154 lb/min (69.9 kg/min) of bleached pulp, with 148 gpm (0.56 m^3/min) of entrained second-stage wash water to the paper mill.

The second-stage washer delivers 1,312 gpm (4.97 m^3/min) of bleach wash water to the second-stage seal tank. This seal tank provides 970 gpm (3.67 m^3/min) to the second-stage dilution tank, 245 gpm (0.93 m^3/min) to the first-stage washer, and 97 gpm (0.37 m^3/min) to the mill outfall.

It was not possible to obtain a detailed balance for the bleach liquor effluent solids and the chlorides in the Flambeau bleach liquor effluent.

Preliminary RO Laboratory Scale Tests

Prior to the field installation, laboratory and pilot tests were conducted on a large volume sample of the Flambeau bleach effluent shipped to the Institute in Appleton. Flux rates were at satisfactory levels in these preliminary tests (8 to 15 gal/sq ft/day – 13 to 25 l/m^2-hr). The development of heavy precipitates or crystalline deposits were not apparent until after the process materials had stood for some time. The small samples and the 5000 gallon (18.9 m^3) truck load did not show evidence of unusual amounts of suspended matter nor of colloidal talc which would require pretreatment ahead of the field test unit. A small amount of sediment, characteristic of fiber, was found in the final drainage from the tank truck load of liquor processed in the principal test run in Appleton.

Samples of lab concentrate were subsequently forwarded to the Avco laboratories in Wilmington, Mass., for preliminary freeze concentration tests.

Reverse Osmosis Field Trial at Flambeau

Description of RO Field Installation--

The RO field installation was designed in cooperation with the mill staff to include a preliminary vibratory screening of the spent liquors close to the source of the feed liquor coming from the first-stage washer seal pit. The liquor was then piped to a 4000-gallon (15.1 m^3) trailer mounted storage tank parked on-site for the duration of this run. A second trailer tank was added to increase the settling capacity after the first week.

The complete layout, with placement of the RO and FC trailer units, is shown in Figure 9.

Approximately one week was required to hook up the trailers and to conduct preliminary flow tests after arrival at the mill site. From the

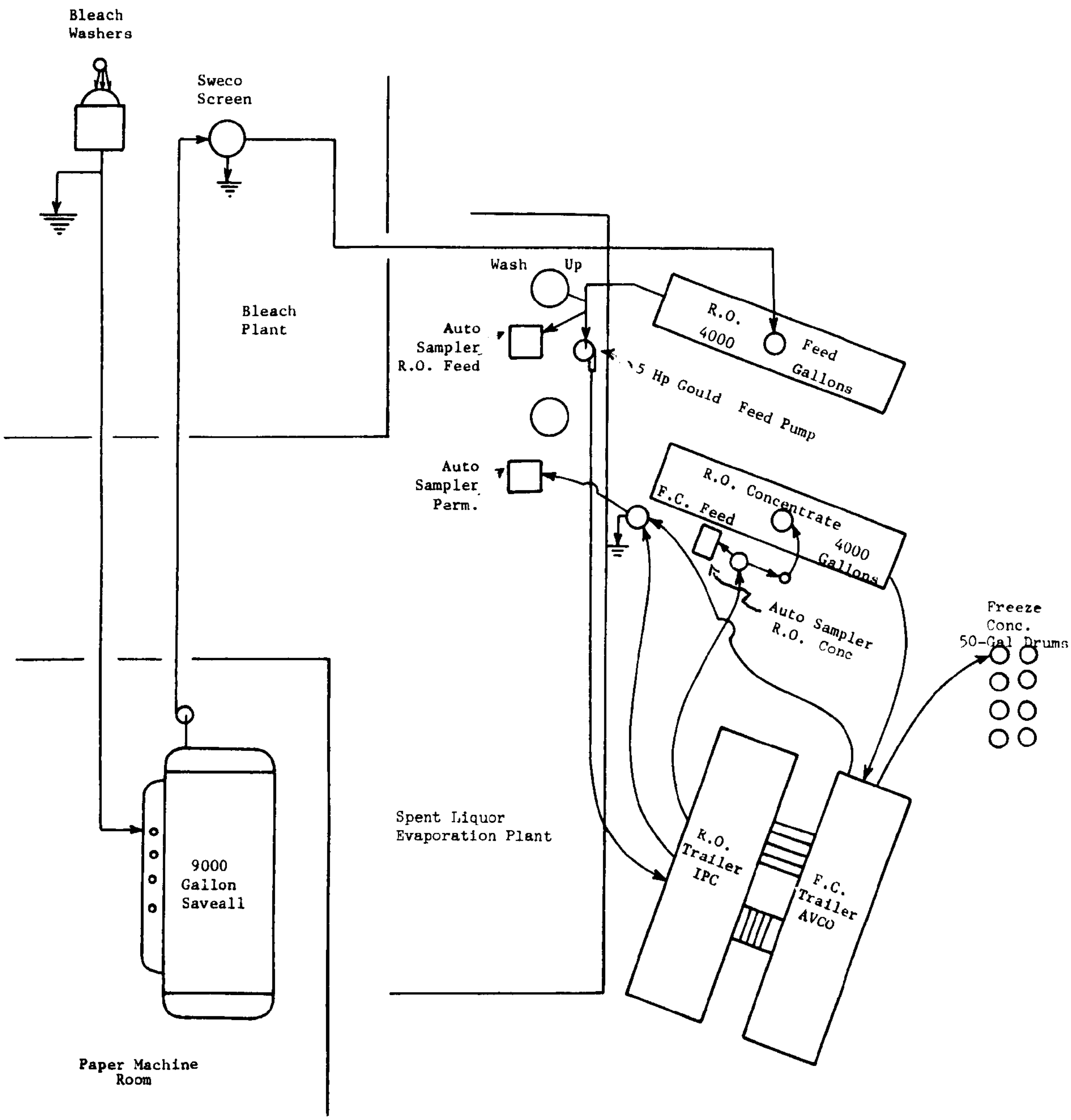

Figure 9. RO-FC setup, Flambeau Paper Company, Park Falls, Wisconsin.

beginning of the preliminary test operations at the mill, it was apparent that the unit was receiving much more suspended material than had been apparent in the drums and truck load test samples sent to Appleton. Preliminary batch type tests of the field unit indicated the operations might be conducted satisfactorily with clarified liquor. Various ideas for achieving clarification of the feed liquor were tried, with only the following approach appearing to have sufficient promise of being made available on such short notice.

Because one paper machine was being operated on a reduced schedule during the course of the trial at this mill, the mill staff was able to hook up the Sveen-Pedersen flotation type saveall from this paper machine as a makeshift settling basin. Even this saveall was too small in terms of surface area and volume to provide a fully successful settling basin at the rates of flow required for the RO unit. Effective volume available for clarification and sedimentation on a continuous flow-through basis was calculated to be 9,850 gallons (37.3 m^3), but this was reduced by a dead volume of 1,910 gallons (7.2 m^3) for batch operation. It was possible to achieve approximately 70% removal of the suspended solids with the use of this clarifier, and the remaining 30% had to be borne as a tolerable load to the RO unit for the duration of the field trail.

Analysis of the suspended matter showed the bulk to be talc, used for pitch control within the mill. Although talc proved to be an effective method for removing pitch and no pitch deposits could be found in the membrane system, the amount of suspended solids (including talc) passing through our 100-mesh screen (149 μ), and not settled out in the makeshift clarifier, gave higher than normal rates of fouling. This resulted in flux rate reductions of 10-20% and required daily backwashes at the end of each 23 hours of operation. More frequent backwashes were also tried (at the end of each 8-hour shift), principally with the use of an enzyme type home laundry detergent (BIZ). In addition, several backwashes with a 3% solution of EDTA (Versene 100) were carried out to remove calcium deposited from this calcium-base bleaching operation. The evidence for fouling by calcium deposits and particularly by calcium oxalate was difficult to establish in terms of their relative importance in the presence of so much talc. The presence of calcium oxalate was definitely established but reliable quantitative assays for total oxalate in the presence of large amounts of lignin type organics did not become available until late in the third field run at Chesapeake and long after completing the Flambeau field trial.

With addition of the saveall as a clarifier in the flow plan, the piping for the field trial at the Flambeau mill provided for pumping the raw feed liquor from the first-stage bleach washer seal tank to the saveall clarifier. The partially clarified liquor from the saveall was passed through a Sweco vibrating screen before being piped to the first of the two trailer mounted storage tanks ahead of the RO unit. Attempts were made to minimize the holding period in these storage and surge tanks in order to prevent precipitation of the inorganic and organic compounds and to maintain the liquor in the freshest possible state.

A Goulds centrifugal pump was used to feed the trailer mounted high pressure pump at a minimum inlet pressure of 20 psi (138 kPa), with the feed rates ranging from 25 to 35 gpm (94-132 l/min). In order to maintain optimum temperatures for these studies at about 40°C, a stainless steel shell and tube type heat exchanger, with 250 sq ft (23.2 m^2) of surface area, was placed in the line between the feed pump and the trailer. It is to be anticipated that high levels of recycle of process waters within the bleaching system will result in heat build-up, with temperatures rising to 50°C or more. However, the membranes available for this project were of cellulose acetate composition, for which temperatures were limited to 40°C. Cooling was required where temperatures exceeded 40°C. On the other hand, the operations at times required small levels of heating to bring cool feed liquors up to the 35°-40°C temperature level which we attempted to maintain. The heat exchanger was readily operated for heating or cooling as required in these test runs. However, it is to be recognized that a minimum, if any, of heating and cooling would be expected in a commercial operation. Some new types of membranes are becoming available which could operate at temperatures of 50° or more. Much higher flux rates can be anticipated with each significant increase in the temperature of operation.

First Stage Intermittent Operation of RO Unit Without Recycle--

The first 12 days of operation were conducted intermittently on the day shifts between June 16 and July 22. Delays were encountered with the time required to develop and test the saveall clarification system before and after the July 4 holiday shutdown. The paper machine had a 5-day run requiring normal use of the saveall which accounted for additional downtime of the RO unit. Table 2 summarizes the operating logs for the period, June 16-23. Table 3 summarizes the analytical data obtained from 12 composited samples in the 3-week period, June 20 to July 22, 1975. For more detailed operating data, the reader should refer to Appendix Table B-1. Complete analytical data are provided in Appendix Table B-2.

Flux rates for this 3-week period of intermittent operation of the RO unit without recycle ranged from 10 to 18 gal/sq ft/day (gfd) (17-31 l/m^2-hr) for the short runs each day. Rejections ranged from 0.80 to 0.90 for total solids, calcium and inorganic chlorides and 0.95 to 1.00 for soluble oxalates and color. Total carbon and BOD rejections ranged from 0.50 to 0.80. The total solids content of the feed liquor averaged 4.95 g/liter and this was concentrated to an average level of 24.14 g/liter. The permeate contained 0.7 g/liter of total solids, thus providing the solids rejection ratio of 0.86. The rejection ratio for calcium was 0.87 and for inorganic chloride 0.84. Only minor amounts of sodium were present in these liquors. Some soluble oxalate was present in minor amounts but was shown to have been rejected at a high (0.98) level. The color was also highly rejected at 0.96 but the rejection for the BOD_5 was only 0.45.

TABLE 2. DAILY RO OPERATING LOG AT FLAMBEAU — JUNE 16-23, 1975
CONCENTRATION OF ACID SULFITE BLEACH LIQUORS

Date	Time (hr)	Operating hours	Feed, gpm	Concentrate, gpm	Flux rate, gfd	Comments
6/16/75	14:00	0				
"	14:45	3/4	21.5	4.9	9.8	
"	15:00	1	19.1	3.3	9.4	
"	15:40	1 2/3	29.7	12.5	10.2	Increased motor speed
"	16:30	2 1/2	Shutdown			
6/17/75	Data not available for Tues., June 17, but unit apparently ran for 4 hours.					
6/18/75	09:30	6 1/2	Startup			
"	10:00	7	33.3	7.7	15.2	
"	10:30	7 1/2	30.8	9.4	12.7	Measurements of flows (and
"	11:00	8	30.0	10.0	11.9	flux rate) are subject to
"	11:30	8 1/2	30.7	10.7	11.9	significant experimental
"	12:00	9	29.2	11.5	10.5	errors
"	13:30	10 1/2	30.6	13.0	10.5	
"	14:15	11 1/4	29.2	12.5	9.9	
"	14:15	11 1/4				Decreased main pump speed
"	14:55	12	23.8	8.8	8.9	
"	15:30	12 1/2	25.3	9.5	9.4	
"	16:00	13	25.2	9.4	9.4	
"	16:00	13	Shutdown			
6/19/75	08:30	13	Startup			
"	09:10	13 2/3	29.3	2.9	15.7	
"	09:30	14	30.2	4.8	15.1	
"	09:50	14 1/3	Shutdown — allowed liquor to settle in storage tanks 4 hours			
"	13:30	14 1/3	Startup			
"	14:30	15 1/3	32.4	3.3	17.3	
"	15:00	15 5/6	32.4	3.7	17.0	
"	15:30	16 1/3	32.1	4.2	16.6	
"	16:00	16 5/6	32.4	6.0	15.7	
"	16:20	17 1/6	32.2	6.1	15.5	
"	16:30	17 1/3	Shutdown			
6/20/75	09:15	17	Startup			
"	09:45	17 1/2	31.5	1.5	17.8	Using liquor clarified
"	10:15	18	32.7	5.9	15.9	overnight
"	10:15	18	Shutdown			
6/23/75	08:30	18	Startup — liquor clarified over weekend			
"	09:00	18 1/2	31.1	3.7	16.3	
"	09:30	19	30.6	5.6	14.9	
"	11:05	20 1/2	31.4	8.1	13.8	
"	11:45	21 1/4	31.3	8.8	13.4	
"	11:50	21 1/3	Shutdown — liquor supply interrupted			
"	14:20	21 1/3	Startup			
"	15:00	22	31.6	8.6	13.7	
"	15:15	22	Shutdown — turbid feed			

TABLE 3. AVERAGE ANALYTICAL DATA*

Preliminary Intermittent RO Operation
Sulfite Bleaching Effluent

	Feed	Permeate	Concentrate	Rejection ratio†
Specific gravity‡	0.999	0.996	1.013	--
pH	6.43	5.29	6.49	--
Total solids, g/ℓ	4.95	0.70	24.14	0.86
COD, mg/ℓ	1,043	--	4,564	--
Soluble calcium, mg/ℓ	1,326	179	6,345	0.87
Sodium, mg/ℓ	3.1	0.8	16.7	0.74
Inorganic Cl^-, mg/ℓ	2,000	330	10,218	0.84
Soluble oxalate§, mg/ℓ	20.3	0.5	53.6	0.98
BOD_5, mg/ℓ	161	88	--	0.45
Color#, mg/ℓ	285	10	--	0.96

*Average of 12 sampling periods June 20-July 22, 1975 (see Appendix Table B-2).

‡At temperature of — feed 28.9°C; permeate 28.4°C; concentrate 29.1°C.

†Rejection ratio = 1 — (concentration of permeate/concentrate of feed).

§As sodium oxalate.

#In terms of platinum by Standard Methods chloroplatinate color standard.

The BOD_5 data, along with the total carbon and chemical oxygen demand data, indicate that the small molecular size, colorless, organic compounds which pass through the membranes might be recycled back with the clear permeate water to be reused in the bleach plant. Some build up of these low molecular weight compounds would be expected from such recycle, but to a limited extent, since oxidation and related degradation reactions apparently take place in the various stages of bleaching. Experience in other operations indicates the chief effect of recycle of the permeate with these low molecular weight materials would probably appear as a nominal increase in chlorine consumption for the additional oxidation loading.

Continuous Operation of RO Unit with Recycle--

The operation of the trailer mounted RO field unit in the continuous mode was conducted with substantial levels of recycle in order to achieve concentration levels approaching 5 times or more. The rate of recycle averaged about 50% of the total feed rate to the system. This recycle was necessary to provide a continuous, minimum feed of 3.5 gal/min (13 l/min) of the membrane preconcentrate for effective operation of the Avco freeze concentration unit. Because the automatic sampling system could not be extended beyond the three principal streams (feed to the RO system and the permeate and the concentrate from the RO system), it was difficult to provide routine evaluations of the flux rates for individual stages of the recycle system. The flux rates for continuous recycle flow were based upon higher levels of solids concentration in the recycled feed. The osmotic pressure was 3 to 4 times higher for the recycled feed than for the fresh feed coming into the system from the mill. The effective driving force was, therefore, substantially reduced which adversely affected the flux rates.

These disadvantages of recycled flow would not be expected to occur in a properly designed and operated full-scale RO unit, since most of the water would be removed in the first stages being fed at low levels of solids concentration and lower osmotic pressure. Subsequent stages would be designed to operate under optimum conditions, with increases in the operating pressure to overcome higher levels of solids and osmotic pressure. Operation of the first stages on dilute feeds, giving flux rates at the 10 to 18 gfd (17-31 l/m^2-hr) level as reported in the previous section, contrast sharply with the reduced rates of flux from recycle operations.

Table 4 provides a summary of the hydraulic data for continuous recycled RO operation over a total period of 189 operating hours between July 22 and July 31. One hundred and seventy-nine thousand gallons (677 m^3) of fresh feed liquor were processed to yield a concentrate of slightly less than 30,000 gallons (114 m^3).

The RO unit processed more than 397,000 gallons (1502 m^3) of liquor in that period, having recycled about 211,000 gallons (799 m^3) of partially concentrated liquor at a recycled rate of 54% and averaged a flux rate of 7.8 gfd (13.2 l/m^2-hr). The average analytical data for this continuous period of operation at the Flambeau mill are presented in Table 5. Reference should be made to Appendix Table B-3 for the more detailed analytical data obtained during this continuous run. Reference should also be made to the operating log provided in Appendix Table B-1, which records the gradual elimination of operating problems as the second stage achieved proficiency in operation during the period July 23 through August 1, 1975. The average analytical data for the period of continuous recycle operation provided in Table 5 are based upon ten sampling periods. Rejection ratios were computed from composited samples from each day of operation. Rejection ratios averaged 0.79 for total solids, 0.81 for COD, 0.48 for BOD and nearly 1.00 for color. Sodium levels are again shown to be relatively low at 4 mg/liter in the Flambeau feed as compared to more than 1480 mg/liter of calcium and nearly 2500 mg/liter of inorganic chloride. The rejection ratio for the small amount of sodium was 0.54 but the soluble calcium and inorganic chloride were rejected at the 0.75--0.77 level.

Reference to the hydraulic data in Table 4 and to the loading and rejection summary provided in Appendix Table B-4, show that for the 189 hours of operation, 179,000 gallons (678 m^3) of raw feed liquor from the mill resulted in processing 8854 pounds (4016 kg) of total solids, of which 6341 pounds (2876 kg) were recovered in the concentrate and 1538 pounds (698 kg) passed through the membrane with the permeate water at an average rejection of 83%. There was an apparent loss in washup of 11%, or 975 pounds (442 kg) of total

TABLE 4. SUMMARY OF HYDRAULIC DATA

Second-Stage Continuous RO Operation at Flambeau

Date	Sample no.	Trailer operation, hours	Total flows, gallons				Recycled, gallons	Recycled, %	Av. flux* rate, gfd
			Feed	Perm.	Conc.	Main pump			
7/22/75	14	20.83	17,750	15,345	2,405	44,254	26,504	59.9	7.29
7/23/75	15	12.00	13,212	11,257	1,955	28,846	15,634	54.2	9.29
7/24/75	16	22.25	20,529	17,596	2,933	53,597	33,068	61.7	7.83
7/25/75	17	20.00	20,748	17,861	2,887	45,549	24,801	54.4	8.84
7/26/75	18	7.50	9,396	8,330	1,066	17,730	8,334	47.0	11.00
7/27/75	19	20.50	22,191	17,964	4,227	37,748	15,557	41.2	8.68
7/28/75	20	22.50	24,505	19,379	5,126	42,734	18,229	42.6	8.53
7/29/75	21	22.50	22,992	17,942	5,050	42,368	19,376	45.7	7.90
7/30/75	22	19.75	13,760	11,720	2,040	37,932	24,172	63.7	5.88
7/31/75	23	21.25	14,241	11,953	2,288	39,376	25,135	63.8	5.57
Total		189.08	179,324	149,347	29,977	390,134	210,810		
Average								54.0	7.82

*Based on total permeate flows; 2,424 ft^2 membrane.

solids. The detailed data for the internal sampling program are available in Appendix Tables B-5 and B-6.

TABLE 5. AVERAGE ANALYTICAL DATA*

Second-Stage Continuous RO Operation at Flambeau Sulfite Bleaching Effluent

	Feed				
	To set. tank	RO	Permeate	Concentrate	Rejection ratio†
Specific gravity‡	1.001	1.001	0.997	1.015	
pH	6.48	6.74	6.29	6.87	
Total solids, g/ℓ	6.32	6.00	1.28	26.62	0.79
COD, mg/ℓ	--	1,125	209	5,124	0.81
Soluble calcium, mg/ℓ	--	1,483	335	6,886	0.77
Sodium, mg/ℓ	--	4.1	1.9	17.2	0.54
Inorganic Cl^-, mg/ℓ	--	2,496	626	11,264	0.75
Soluble oxalate§, mg/ℓ	--	8.7	1.6	8.7	0.82
BOD_5, mg/ℓ	--	235	122	--	0.48
Color#, mg/ℓ	92	95	0	--	1.00
Suspended solids, mg/ℓ	326	100	--	--	--

*Average of 10 sampling periods July 22-31, 1975.

‡At temperature of — feed to settling tank 28.3°C; feed to RO 27.9°C; permeate 28.6°C; concentrate 29.2°C.

†Rejection ratio = 1 — (concentration of permeate/concentration of feed).

§As sodium oxalate.

#In terms of platinum in Standard Methods chloroplatinate color standard.

Review of Table 6 shows 85% rejection of COD and a 10% loss of COD in the washup. One thousand six hundred sixty-five pounds (755 kg) of calcium, 3.4 pounds (1.5 kg) of sodium, and 2,691 pounds (1220 kg) of inorganic chloride were recovered in the concentrate. The best available methods for determination for soluble oxalates showed 4.3 pounds (2.0 kg) of this type of material recovered from the 12.4 pounds (5.6 kg) in the feed liquor. This discrepancy needs to be reevaluated with the development of better methods for an assay on oxalic acid in the presence of lignin residues, but it was apparent that a substantial proportion of the oxalates were being lost as precipitates of insoluble calcium oxalate. Our methods of collecting samples and of analysis could not provide a good balance for effectively tracing the pathways whereby the content of oxalic acid is lost in the system. Some calcium oxalate was apparent in the fouling of the membranes as could be ascertained from regenerating fouled membranes with an EDTA chelating agent (Versene 100). The residual EDTA solution contained appreciable amounts of Ca but no quantitative data were established. However, the amounts of calcium lost as shown in the balance sheets for Table 6 were not adequately accounted for in these studies. An energy dispersive x-ray analysis with electron microscopic examination of the membranes (with and without regeneration treatment with Versene), positively identified the presence of calcium oxalates in small amounts. However, this preliminary study failed to account for the amounts of calcium oxalate shown in the balance sheets. Further study of the formation of oxalic acid and of the problems it may generate in high level recycle operations may be required to document this point.

TABLE 6. PRODUCT BALANCE DATA

Continuous RO Operation
Sulfite Bleaching Effluent

	Feed	Permeate	Concen-trate	Rejection* ratio	Lost in washup Pounds	Lost in washup %
Total solids, lb	8854	1538	6341	0.83	975	11.0
COD, lb	1674	254	1251	0.85	169	10.1
Soluble calcium, lb	2199	403	1665	0.82	131	6.0
Sodium, lb	5.04	1.80	3.42	0.64	+0.18	+3.6
Inorganic Cl^-, lb	3690	754	2691	0.80	245	6.6
Soluble oxalate†, lb	12.40	2.12	2.22	0.83	8.06	65.0
BOD_5, lb	346	151	--	0.56	--	--
Color‡, lb	142.3	0.0	--	1.00	--	--

*Rejection ratio = 1 — (concentration of permeate/concentration of feed).

†As sodium oxalate.

‡In terms of platinum in Standard Methods chloroplatinate color standard.

Performance Summary for RO Concentration With and Without Recycle--

Comparison of the performance of the RO concentrating system under first-stage intermittent periods of operations without recycle and with the second period of continuous recycle modes of operation are presented in Table 7. Data averages for the solids content to the overall system during each mode of operation ranged from 4.95 g/liter without recycle to 5.91 g/liter with recycle. However, the mixed feed during recycle operation, which was the actual concentration of solids being processed in the first stages of the RO system, ranged from 11 to 21 grams solids per liter, or 2 to 4 times the concentration being processed without recycle. The solids content in the final concentrate was 24.1 g/liter without recycle and 25.3 g/liter with recycle. The data for concentration by each mode ranged from 4.8 times without recycle and 4.28 with recycle. The water product recovery ranged from 75.5% of the feed volume without recycle to 83.3% with recycle.

TABLE 7. PERFORMANCE SUMMARY FOR RO CONCENTRATION WITH AND WITHOUT RECYCLE*

	Staged intermittent operation (no recycle)	Recycle operation
Solids in feed to overall system, av. g/ℓ	4.95	5.91
Solids in feed to first membrane stage, g/ℓ	4.95	11.0-21.0
Solids in concentrated product, g/ℓ	24.1	25.3
Degree of concentration of feed to system	4.8	4.3
Water product recovery (permeate), % of feed volume	75.5	83.3
Indicated overall flux rate, gfd (after 3 hours of operation)	13.7	7.82
Osmotic pressure of feed to first stage, psi	35	98
Osmotic pressure of concentrate, psi	--†	175

*The above data are drawn from the more detailed tabulations of operating data provided and described in greater detail within Tables 3 to 7 of the main text of this report and in the Appendix Tables B-1, B-2, and B-3.

†No data.

These data show a flux rate of 13.7 gfd (23.2 l/m^2-hr) without recycle and 7.8 gfd (13 l/m^2-hr) with recycle. The osmotic pressure of the feed to the first stage at 35 psi (241 kPa) without recycle was about one-third that of the recycled feed. The osmotic pressure was a very apparent factor in reducing the flux rate, but other characteristics of process liquors being fed at higher concentration are also known to reduce the flux rate as the concentration rises. This is especially true of substrates with an increasing concentration of high molecular weight, viscous, organic polymers, which are characteristic of lignin residues in bleach liquors.

The effect of the continuous recycle RO operation is shown in Figure 10. A rapid fall-off in flux rates is characteristic of sustained high level concentration operation immediately after a washup. The objective for the study of the continuous recycle was to better establish the overall flux rates and to learn more of the possibilities for gaining sustained high rates of flux at higher levels of concentration. Ways to reduce down time for washups and to regain optimum flux rates of the fouled membrane were also the subject of development in this program of study.

Table 8 provides further interpretative data helpful in establishing the performance of the membrane system as the concentration advances. The fresh mill feed at 5.91 grams total solids per liter, comprising 50% of the recycled feed at 16.98 g/liter, was concentrated overall by a factor of 5.35. In Stage 1, the concentration advanced to an average of 19.5 grams solids per liter, in

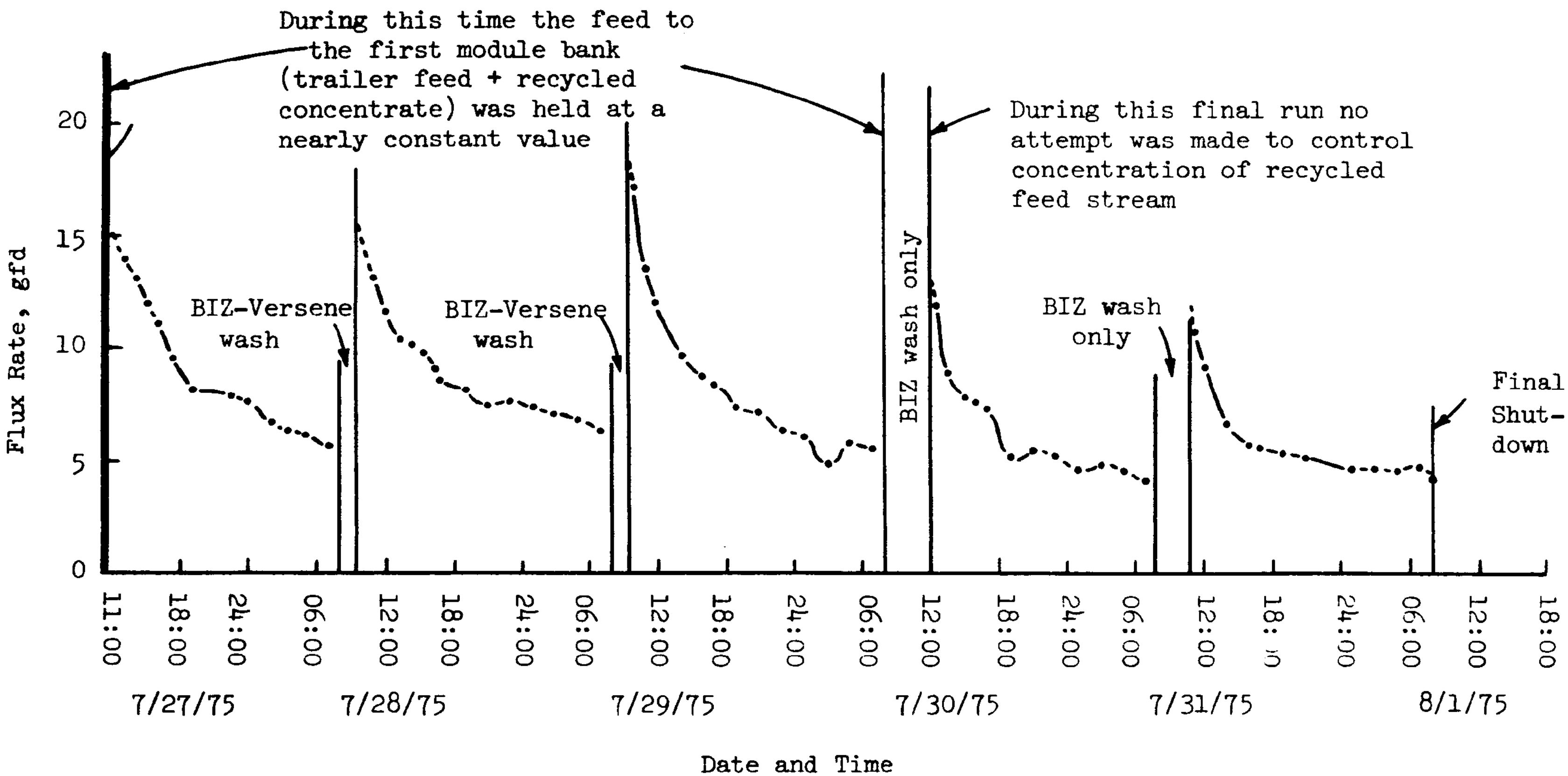

Figure 10. Flux rate vs. time for continuous recycle operation.

TABLE 8. PERFORMANCE OF FOUR SUCCESSIVE MEMBRANE CONCENTRATION STAGES

Sulfite Bleach Process Water — Flambeau Paper Company, Park Falls, Wisconsin
(Data Averaged from Internal Grab Samples on 3 Different Days)*,†

Stage	Total solids, g/ℓ	Rejection ratios — $\left(1 - \frac{\text{permeate}}{\text{feed}}\right)$						Viscosity, centipoises 25°C	Osmotic pressure, psi
		Total solids	COD	Na	Soluble Ca	Inorganic Cl	Color		
Recycled‡ feed	16.98	--	--	--	--	--	--	0.752	98
Stage I	19.47	0.93	0.93	0.82	0.93	0.92	1.0	0.752	113
Stage II	22.53	0.93	0.94	0.87	0.93	0.92	1.0	0.761	139
Stage III	25.01	0.91	0.94	0.82	0.89	0.89	1.0	0.764	156
Stage IV	31.65	0.96	0.95	0.88	0.96	0.95	1.0	0.769	175

*Overall flux rate averaged 8.8 gfd with water recovery at rate of 86%.

†Grab samples taken on 3 days:
July 25, 1975 at 3:30 p.m.
July 29, 1975 at 3:00 p.m.
July 31, 1975 at 3:00 p.m.

‡Fresh feed from mill to recycle system — 5.91 g total solids per liter.

Stage 2 to 22.5 g/liter, in Stage 3 to 25.0 g/liter, and in Stage 4 to 31.6 g/liter. The averages are for each stage on 3 separate days of operation.

The average rejection levels of solids, COD, soluble calcium, inorganic chloride, and color, were all 90% or better. Sodium rejections were also excellent at levels from 82 to 88%. The increasing osmotic pressure accounts for the progressive reduction in flux rate from a starting level at 12 to 15 gfd (20-25 l/m^2-hr) in the first stages of RO concentration of dilute feeds at 5 g/liter to flux rates on the order of 5 to 8 gfd (8-14 l/m^2-hr) at concentrating levels above 25 g/liter. The overall average flux rate shown in this table for the recycled mode of operation was 8.8 gfd (15 l/m^2-hr).

These data show that much of the water removal to be achieved can be accomplished advantageously at high rates of membrane flux within the first stages of operation at the lower levels of solids concentration. More than 70% of the total volume of water to be removed can be accomplished at flux rates approaching 12 to 15 gfd (20-25 l/m^2-hr).

Freeze Concentration Field Trial at Flambeau Mill

The Operating Plan for Freeze Concentration (FC) at Flambeau--

Operation was begun on June 20 to check out the equipment. Pressures in the heat removal system were relatively high due to high temperature cooling water and fouling of the condenser. The condenser was cleaned with an alkaline solution to remove any oily deposits, followed by an acid cleansing to remove rust and scale. Operation was resumed on June 27, but high condenser pressures still hampered operations. The cooling water source was switched from river water at 25°C to well water at 16°C. No further problems with high condenser pressures due to lack of cooling water were encountered.

Testing on July 1, 2, and 7, 1975 established that concentrations corresponding to a freezing point of -4°C could be achieved in a single stage while producing fresh water of a few hundred micro mhos/cm conductivity. Initially, excessive foaming in the first-stage freezer interferred with operation, and could only be controlled with massive injections of defoamer but as higher concentrations were reached, foaming was only intermittent and could be controlled through the moderate use of defoamer. No foaming in the second-stage freezer occurred at any time during these or following tests.

Testing with two stages began July 8. Initial results were very encouraging with temperatures as low as -11°C being reached on 7/9 and 7/10. These were the two best runs obtained at Park Falls. Fresh water quality continued to be a few hundred micro mhos. On 7/13 operation of the system was stopped due to blockage of the slurry line conveying the ice from the second-stage wash column to the first-stage freezer. Small pieces of screen were found in the line blocking the inlet to the control valve. The wash column was subsequently disassembled for inspection of the screen. No damage to the screen was found. A screen failure had occurred about 4 weeks prior to testing at Flambeau and apparently it had taken that long for the screen pieces to work their way through the system and become lodged in the valve. Several other pipes were also taken apart and inspected for screen pieces but no others were found.

Operation was resumed on 7/25 (the downtime included a 1-week scheduled shutdown during which data were reviewed). Operation of the second-stage wash column was unstable during 85 hours of continuous testing. The instability contributed to many upsets of the first stage and fresh water quality was very erratic, generally ranging from 3,000 to 6,000 micro mhos/cm. Second-stage freezer temperatures ranged from -7 to -5°C during this period. Lower temperatures could not be obtained due to the instabilities.

The wash column was disassembled and inspected on 7/29 to see if there were any mechanical damage which could account for the problems, but none was found. The column was constructed with an 8-inch (20 cm) core in its center so as to reduce its cross-sectional area and match its capacity to the expected production. Since some of the instabilities had been associated with high pressures in the column, this core was removed in hope that the pressures would be reduced and better operation could be achieved. Testing during the period of 7/30 through 8/3 gave results essentially the same as that obtained prior to removing the core.

On August 4, 1975 single-stage tests were resumed in order to collect some concentrate for further evaluation. Slightly higher concentration was obtained than during initial tests, but this was at the expense of product quality. The conductivity went up to 3,000-5,000 micro mhos/cm. During this period several upsets occurred, apparently due to the accumulation of noncondensables in the heat removal condenser. This had not occurred previously and has not been fully explained. It may have been due to CO_2 produced by the microbial degradation of stored liquor.

Table 9 is a summary of the freeze concentration operating log at Flambeau.

After testing at Flambeau, the trailer laboratory was returned to Wilmington, MA for some modifications prior to testing at Continental Group mill, Augusta, GA. The second-stage wash column was disassembled for installation of a screen heater. At this time, it was observed that there was a buildup of a slimy cake of solids (dirt) on the screen of the second-stage wash column. This dirt could have contributed to the poor operation of the column. However, this dirt was not observed when the wash column was disassembled two times at Flambeau. In addition to installing the screen heater, extensive modifications were made to the heat removal system to permit operation with the higher temperature cooling water anticipated at the Continental Group mill.

Operation and Results of FC Unit at Flambeau

Figure 11 shows the correlation between freezing point and concentration. As expected, depression of freezing point occurs with increase in solids concentration. Table 10 is a summary of the important FC data gained at Flambeau. Based on an initial concentration of 5 g/liter and a final concentration of 160 g/liter this indicates an overall water recovery of nearly 97% for the combined RO-freeze concentration system. Eighty percent of the water recovered by the freeze system is obtained in the first stage where the energy requirements are lower. The product water quality of 0.2 g/l (200 ppm),

TABLE 9. AVCO DAILY OPERATING LOG SUMMARY FOR FREEZE CONCENTRATION

Avco Mobile Laboratory
Flambeau Paper Company
June 27 — August 6, 1975

Date	Hours operation	Hours* open loop	Single (I) or two (II) stage	Conc.† temp., °F 1st/2nd	Product cond., μ mhos/cm²	Comments
6/27	8	--	I	29.5	260	Check out
6/30						Connect cooling water to city supply
7/1	6.5	0.3	I	29.5		Foaming
7/2	9.8	1.5	I	24	300-800	
7/7	10.5	2	I	25	450-4,000	Filling second stage with conc.
7/8	7.5	2.3	II	26/21	40-60	Start 2-stage tests
7/9	10.0	3.2	II	24/11.3	60-650	Highest concn. achieved in testing
7/10	7.6	1	II	22/11	2,000	1st stage temp. too low couldn't wash well
7/13	11.8	0.5	II	27/17	400	Found pipe blocked with scrap
7/25	13	--	II	27/27	--	Restart after shut-down
7/26	24	1.1	II	23/20	--	2nd stage column not stable
7/27	24	1.3	II	26/23	3,000-6,000	"
7/28	24	2	II	24/20	3,000-6,000	"
7/29						Removed inner core from second column
7/30	8	0.8	II	27/15	500-1,400	2nd stage wash column not stable
7/31	17	0.9	II	24/20	--	"
8/1	24	0.4	II	25/22	900-10,000	"
8/2	7	1.1	II	25/25	150-350	"
8/3	4	--	II			"
8/4	24	1	I	24	50-5,000	Resume single stage tests
8/5	21.5	2.7	I	23.5	3,000	
8/6	24	5	I	23.5	5,000	

*Hours open loop — period when feed is being brought in system and concentrate and product are being discharged. Other periods of operation are termed closed loop when concentrate and product are mixed together to form feed.

†Concentrate temperature is temperature of concentrate in freezer and corresponds to concentration as shown on curves.

although not quite as color free as that obtained from the RO system, had lower dissolved solids than that obtained from the RO system.

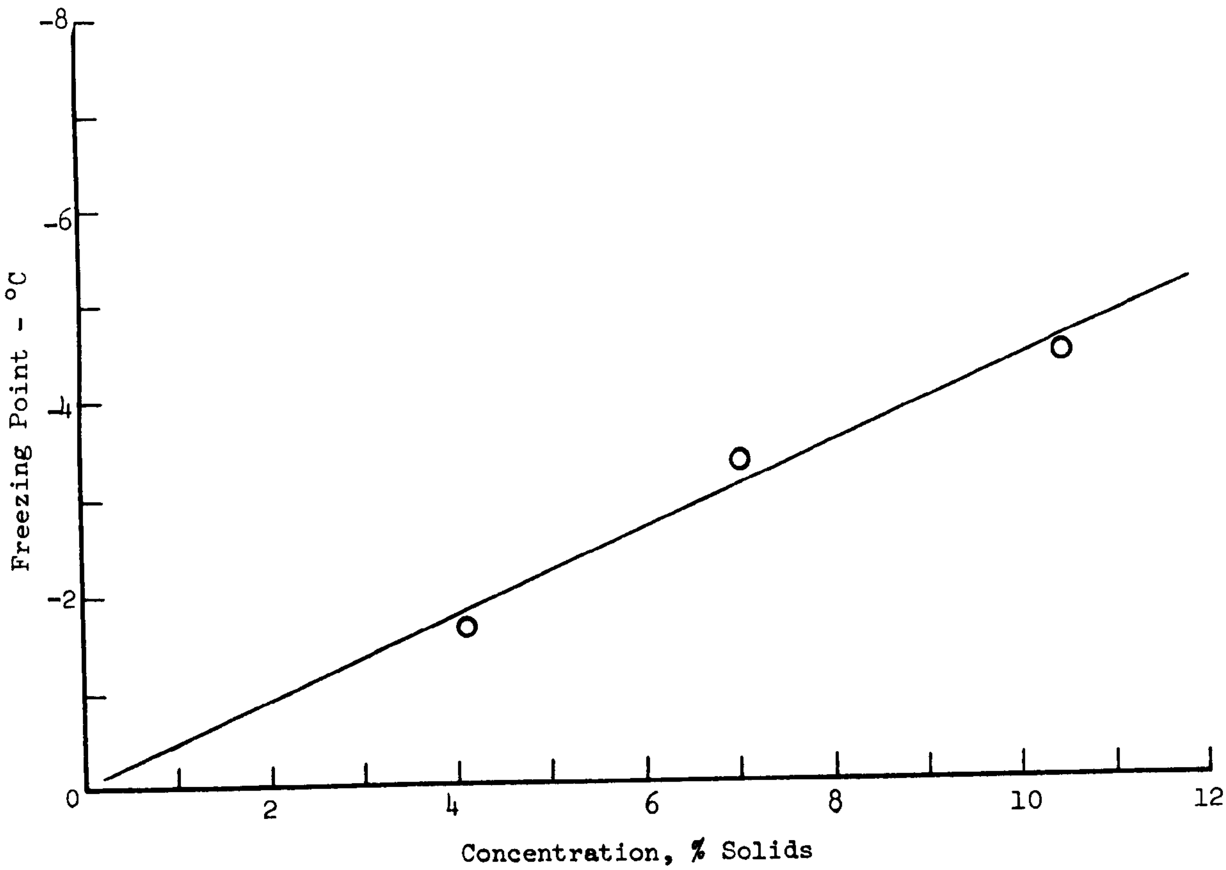

Figure 11. Freezing point correlation for Flambeau concentrate.

TABLE 10. SUMMARY OF PRINCIPAL DATA AVCO MOBILE LABORATORY FLAMBEAU TEST RUN

Solids in feed, g/ℓ	18-26
Solids after first stage, g/ℓ	100
Solids after second stage, g/ℓ	160
Degree of concentration	6X-9X
Solids in recovered water, ppm	200
Freezing point, first stage, °C	-4
Freezing point, second stage, °C	-5.5
First stage recovery, %	8
Overall freezing recovery, %	88

Appendix Table B-7 gives some analytical data for grab samples. In addition, samples taken on 7/2/75 were analyzed for sulfate; results are shown in Table 11. Most of the samples from the Flambeau run were lost in transit due to sample containers bursting from the pressure generated by vaporization of the refrigerant retained in the samples. This resulted in much less analytical data being obtained than had been anticipated. Significant amounts of suspended solids were found in the concentrate from the freezing process. Sulfate data indicate that a large percentage of these solids might have been $CaSO_4$.

TABLE 11. AVCO ASSAY OF FREEZE CONCENTRATION GRAB SAMPLES FROM FLAMBEAU

Sample	Total solids, g/ℓ	Freezing point, °C	SO_4, g/ℓ
RO concentrate	16.8	--	0.3
Brine	40.9	-1.67	0.9
Brine	80.2	-3.33	1.8
Brine	105.0	-4.44	2.5

The Institute's Appleton laboratory received samples from the daily operations of RO and FC units during the course of the two test operations at Flambeau. Data for the two best freeze concentration runs are summarized in Table 12, with a more complete analytical data for the entire run provided in the Appendix, Table B-7. In the run on July 9, the RO concentrate with 19.8 grams solids per liter was concentrated to 108.08 g/liter in the first stage of freeze concentration and to 127.45 g/liter in the second stage of freeze concentration. In the second trial on 8/6/75, the RO concentrate at 26.14 g/liter was concentrated to 153.36 g/liter in a single stage of freeze concentration.

The melted water recovered from both of these operations was very clean and contained only 0.16 to 0.19 grams of solids per liter. It is interesting to learn that the second-stage concentrate from July 9 apparently contained 141.6 grams of soluble oxalate; however, the reliability of the soluble oxalate assay continued to be in question due to interference by lignin residues in the best practical analytical procedure available at that time.

Complete analyses of the feed and first-stage concentrate were available only for the August 6 freeze concentration run. The recovered melted ice water showed low levels of all components. High levels of all soluble materials were present in the concentrate. High levels of $CaCl_2$ (up to 50% of the total solids) were apparent. This is to be expected as the hypochlorite bleach chemical, CaOCl is converted to the chloride salt.

Operation of the first stage of the freeze concentration unit at Flambeau was quite good. Solids concentration of 10% (freezing point of -4°C, 25°F) were quite readily achievable. Even though significant amounts of suspended

TABLE 12. ANALYTICAL DATA — TWO BEST RUNS

Avco Freeze Concentration Unit at Flambeau
Sulfite Bleaching Effluent

	7/9/75				8/6/75		
	FA*	CAI	CAII	MA	FA	CAI	MA
Hours operation	10	--	--	--	24	--	--
Stages	2	--	--	--	1	--	--
Concentrate temp., °F	--	24	11.3	--	--	23.5	--
Specific gravity†	1.011	1.083	1.094	0.996	1.015	1.081	0.997
pH	6.40	7.10	7.10	6.71	6.48	5.95	6.89
Total solids, g/ℓ	19.80	108.08	127.45	0.16	26.14	153.36	0.19
Soluble oxalate‡, mg/ℓ	--	--	141.6	--	9.0	28.9	9.3
COD, mg/ℓ	--	--	--	--	4,375	23,299	81
Soluble calcium, mg/ℓ	--	--	--	--	4,580	26,500	32
Sodium, mg/ℓ	--	--	--	--	13.4	68	Trace
Inorganic Cl^-, mg/ℓ	--	--	--	--	11,006	54,092	39
Viscosity§, cp.	--	--	--	--	0.760	0.964	--
Color#, mg/ℓ	--	--	--	--	1,780	9,700	22

*FA - feed; CAI - Stage 1 conc.; CAII - Stage 2 conc.; MA - recovered water.

†At temperatures of 29.0°C; 27.0°C; 27.0°C; 29.0°C; 29.0°C; 29.0°C; 27.0°C, respectively.

‡As sodium oxalate

§At 35°C.

#In terms of platinum in Standard Methods chloroplatinate color standard.

solids were found in this concentrate, no operational problems were attributed to it. Except for operation on July 8, 9, and 10, the second-stage wash column was very erratic. This erratic operation was characterized by 1 or 2 hours of stable operation, followed by a stoppage of the wash column. The column pressures would rise and eventually reach a value in excess of the capability of the slurry pump feeding the column. Pressure taps located along the lower portion of the column indicated that the ice pack in the column was gradually growing in length and eventually reached the bottom of the column, at which time the pressures would be so high [about 115 psig (825 kPa), compared to a normal value of 45 (411 kPa)] that no flow could be forced through the column. Even after the core of the column was removed on July 29, no improvement was noted, indicating that friction was not a problem. This left two other possible explanations for the stoppages: 1) freezing of the screen, or 2) accumulation of solids on the screen. Although no solids were noted on the screen when it was inspected on July 13 and 29, the significant accumulation found on disassembly between the tests at Flambeau and Continental Group Inc. raises serious question as to this possibility. The screen in the second stage is much finer than that in the first stage. This is because the ice produced in the second stage is finer than that made in the first stage and difficulty had been encountered in retaining this ice with the coarser screen. The possibility of freezing was investigated during the testing at Continental Group mill and is discussed in that portion of this report.

Control of the second-stage wash column was difficult, even during periods of otherwise stable operation. The level control in the second-stage freezer is coupled to the washing in the second-stage wash column. As the pressures in the wash column changed, the amount of water used as wash changed drastically. Because the amount of water being processed in the second stage was only 20% of the feed, it was relatively easy to have a large wash water loss which would be in excess of the required feed to the second stage. This resulted in overfilling of the second-stage freezer. Conversely, the freezer, on occasion, became starved due to carryover of concentrate with the ice from the wash column. Although these are the extremes, minor problems of this type required considerable attention. In order to alleviate this problem, less than the maximum amount of water was recovered in the first stage which did relieve the problem to some extent.

Although concentrations of over 22% were achieved in the laboratory tests and, as indicated by temperature, exceeded in the field tests, there was no indication that this value was maintained for any significant period of time. It was observed that operation about -5.5°C corresponding to a concentration of 16%, was better than lower temperatures and thus somewhat arbitrarily this has been defined as the current limit of concentration for the Flambeau liquor.

<u>Further Concentration and Disposal Studies of FC-stage Concentrate</u>

Six hundred gallons (2.3 m^3) of the freeze concentrate produced by the Avco trailer unit at Flambeau were shipped to Appleton for further study.

The entire 600-gallon (2.3 m^3) shipment was concentrated to 200 gallons (0.76 m^3) at about 30% solids in the Struthers-Wells crystalizer type of pilot evaporator. There were no apparent problems in conducting this higher level of concentration, but the run was of much too short duration (about 4 hours) in this large evaporator unit to have any indication of scaling or corrosion problems. Some additional turbidity settled out slowly over a period of weeks in cold storage. The high level solubility of $CaCl_2$ hydrate ($CaCl_2 \cdot 6H_2O$) is such that no crystals were apparent at that concentration. The concentrated material appeared to be in a state that could be readily handled. Relatively small volumes (a few tank truck loads per day) might be disposed in such outlets as dust laying on gravel roads. Local highway and street maintenance crews in the area of this mill have extensive experience with the use of sulfite roadbinder, during summer months. The question arises as to whether the 30% solids level would be high enough to act as a source of road salt for deicing operations on roadways during winter months.

About 35 gallons (132 l) of the 30% solids were further concentrated to 50% in a large lab vacuum evaporation loop. There was no immediate deposit of crystalline material, but examination after storage at room temperature showed substantial deposits of large crystals typical of the $CaCl_2$ hydrate. These crystals were found to contain 18% calcium, which is quite close to the theoretical calcium content. Therefore, we can safely assume that the crystalline material was substantially, if not entirely, composed of $CaCl_2 \cdot 6H_2O$.

Discussion of the Flambeau Field Trial--

Evaluation of the overall data summaries and of the daily operating logs provide a base for reassessment of the objectives and goals for this research project. The capabilities of RO and FC to concentrate the materials solubilized in the older H-H bleaching sequence at this Ca base acid sulfite mill were demonstrated. The substantial flows of recovered clean, clear product water and of the concentrates of dissolved BPE solids from each trailer were impressive.

Various mechanical and hydraulic operational problems requiring improvement in design were disclosed. The ever present problems associated with fouling were less troublesome than in prior experience, and appear capable of being successfully surmounted, but will require continuing study and improvement. However, much more critical to success in achieving practical and economically feasible systems of RO and FC concentration, is the readily apparent and growing need for substantial reduction in the volumes of flow for the bleaching process effluents to be fed to these concentrating systems.

The first-hand experience gained in close and excellent cooperation with the Flambeau technical staff in the course of conducting the field test operations disclosed need for innovative studies on liquor collection. All tests of flow reduction for this field trial were necessarily based upon existing operations requiring collection of highly diluted flows coming from the bleach washers. Recycle of secondary wash waters to the first-stage bleach washer was the principal route to flow volume reduction. Although the use of white water instead of fresh water is helpful in reducing overall consumption of fresh water, it did little to reduce the volume of flow from which the feed to the RO system was drawn. The conventional bleach washers still require the same high levels of wash and rinse water flows to the showers. The discharge of highly diluted wash waters from the washers was the only feasible source of feed of the RO system in this mill at the time of conducting this field trial, and also this was the case in the other bleach demonstration sites for this project.

The need for reduced bleach effluent flows is apparent in the flow data summarized in Table 13. The normal levels of bleach plant effluent flow at the Flambeau mill in recent prior years, 1970-75, has varied around 850 gallons per minute (3.2 m^3/min), equivalent to 10,200 gallons per ton (14.6 m^3/t) pulp of dilute bleach plant effluent overflowing from the seal tanks of the first- and second-stage washers. This would have a calculated solids concentration of about 3.9 grams per liter. To obtain a concentrate at 5% solids from the RO system, it would be necessary to remove about 9,385 gallons of water for each ton (39.2 m^3) of pulp produced. With extreme flow reduction, only 2785 gallons of water need to be removed for each ton of pulp (11.6 m^3/t)

TABLE 13. VOLUME OF WATER TO BE REMOVED BY RO TO ACHIEVE 5% SOLIDS PRECONCENTRATE

(At Various Levels of Collecting Bleach Process Effluent)

Basis for Calculations:	
Bleached pulp production, ton/day	120
Shrinkage in bleached pulp yield @ 7.7%, lb/day	18,500
Total bleach effluent solids (55% inorganics), lb/day	40,800
Average analysis RO feed samples (6-week run), g/ℓ	5.4

	Normal operation (1970-75)	Reduced flow (field trial)	Minimum flows (new washers)	Probable maximum flow for process feasibility
BPE flow, gallons/min*	850	625	450	300
BPE flow, gallons/ton pulp	10,200	7,500	5,400	3,600
Total solids, g/ℓ	3.9	5.4	7.5	11.3
Permeate water to be removed, gallons/ton	9,385	6,605	4,585	2,785

*BPE = bleach plant effluent.

Cost evaluations for this project under 1976 price levels for equipment, energy, and man power are subject for computerized study and discussion in a later section of this report. This project was set up with the full realization that costs have inflated but that there have been improvements in membrane performance which may partially compensate for the rising costs. Preliminary estimates based on the range of costs developed in an earlier study in 1972 (2), at levels ranging from $1.50 to $2.00 per thousand gallons of water removed ($0.40-0.53/m³), would indicate a membrane concentration charge of from $15.00 to $20.00 per ton ($16.50-22.00/t).

The program for organizing this research project sought test sites in bleach plants which had reached levels of 6,000 gallons of BPE for each ton (23 m³/t) of bleached pulp produced. The Flambeau staff were not able to attain the 6,000 gallons per ton (23 m³/t) figure but did arrange to recycle their second-stage washer effluent back to the first-stage washer and were able to include several other water saving practices, such that we were able to have a feed flow to the RO system based on 625 gallons (2.4 m³) of combined flow from the first-stage washer, equivalent to 7,500 gallons of BPE per ton (28 m³/t) of pulp production. This substantially improved the volume of flow at 25% reduction over normal practice and was very helpful to development and execution of this field trial. The solids concentration averaged 5.4 g/liter from the many feed samples collected and analyzed during the six weeks of active field operations. At this level of operation, we could anticipate having to remove 6,600 gallons (25 m^3) of permeate water to achieve a 5% solids concentrate.

In conversations with the mill staff, they estimated that a substantially greater reduction in flows, to a level of about 450 gallons per minute (1.7 m^3/min), might be possible if the mill could later afford the installation of more efficient multiple-stage washers. The flow from a rebuilt washing system might be on the order of 250 gallons per minute (0.95 m^3/min), equivalent to 5,400 gallons per ton (22.5 m^3/t) of pulp having 7.5 g/liter of total solids. The permeate flow to achieve 5% solids under such conditions would be expected to remove 4,585 gallons of water per ton (19.1 m^3/t) of pulp production.

Although the greatly reduced flows which could be anticipated from improved washers would substantially reduce the costs of a concentration system on the order of one-half of that for the flows coming from conventional practices of prior years, the cost of concentration would still be considered far in excess of the probable range of practical feasibility for treating bleach plant effluents.

Similar problems have been experienced in developing liquor collection systems for the spent pulping liquors, which are now almost universally collected for evaporation or other methods of concentration processing, but it seems desirable to undertake an innovative search for ways in which the bleach plant effluents could be collected from each individual bleaching sequence prior to dilution on the washer. Discussions with mill representatives and also with equipment representatives, having prior experience with the liquor collection problems, indicate there may be possibilities for accomplishing such collection of strong liquor ahead of the washers. Facilities available at the Flambeau mill did not permit an actual trial of liquor collection from the bleach towers, but we can speculate that it might be possible to collect as much as 300 gallons per minute (1.1 m^3/min) of strong bleach liquor flow from the No. 1 bleach tower containing upwards of 80% of the total dissolved solids in bleach plant effluents discharged from this mill. Displacement washing within the bleach towers, such as has commonly been used in the blow pits of sulfite pulp mills, is one possible route. Substantial experience is available on the use of presses to dewater pulp throughout the industry, and indeed The Chesapeake Corporation presently uses the pressing operation to remove excess chlorides ahead of the oxygen bleaching stage at their mill, which served as the third field test site for this project. The Norwegian mill at Halden is known to have been using a press for removing the bleach liquors from their soda base bleach pulp for more than 20 years.

The final column of Table 13 shows that at a collection rate of 300 gallons per minute (1.1 m^3/min) of the strong flow from the No. 1 bleach tower could be expected to yield 3,360 gallons of flow per ton (14 m^3/t) pulp, with 11.3 g/liter of solids. About 2,800 gallons (11 m^3) of permeate water would have to be removed by RO to give a 5% concentrate of the bleach plant effluent solids. Under such conditions, both the capital and operating charges could be expected to be reduced to a fraction of that required for much higher levels of very dilute flow coming directly from the bleach washers. Obviously, a first route to process feasibility lies in collecting the bleach plant effluent flows prior to dilution on the washer. The equipment manufacturers are well aware of need for reduced use of water and in washing pulp, and various types of equipment can be expected to become available for new plants and for renovation of older systems. Possibilities for using a modified displacement liquor collection system within the bleach towers may greatly reduce the capital investment required for liquor collection, and may also have a positive benefit in greatly reducing the amount of washing required on conventional bleach pulp washers. Discussion of this line of reasoning will be further developed in the final sections, based upon computerized cost evaluations for this project.

II. FIELD TRIAL AT CONTINENTAL GROUP, INC., AUGUSTA, GEORGIA

The Pulp Mill and Bleach Process

Continental Group, Inc. (formerly Continental Can Company) operates a large kraft mill with two pulping, bleaching, and paper machine process lines on softwood and hardwood. The mill was producing a total of about 800 tons (726 t) daily of semibleached and bleached pulp, principally for food container board at the time of the field trials. Substantial improvements to the existing bleaching system were being programmed in 1975 as shown in Figure 12. The washed brownstock entering the bleach plant at 3% consistency with a calculated 66,667 lb of water per ton (32.5 m^3/t) of unbleached fiber was to be processed through the CEHD bleaching system. The bleached fiber slurry issuing at 12% consistency would have a water content reduced to 15,556 lb per ton (7.8 m^3/t). The normal yield of 45% unbleached fiber from kraft pulping of wood at this mill would be further reduced to 42% (6% loss in bleaching). This bleaching loss of about 133 lb (60 kg) of the dissolved wood organics with bleaching chemical residues of about 155 lb (70 kg) of chlorine and 69 lb (31 kg) of NaOH would discharge in the 10,000 gal (38 m^3) of bleaching effluent to the mill sewer. The dissolved solids content of the bleaching effluent, calculated to be about 0.43% under the planned program, would approach the 0.5% dissolved solids (DS) level established as a goal within this field demonstration project as a minimum concentration of RO feed needed to attain an economically feasible application of the RO preconcentration step for bleach process waters.

Further modifications and extension of process water recycle within limits of the corrosion resistance of the existing metallurgical components of the pulp washing system may be expected to reduce water usage to about 8,000 gal per ton (33 m^3/t) but these further improvements could not be completed on a mill scale for this trial. Still further reductions in water usage could only be accomplished with major reconstruction of the bleaching system.

Although the flows available were substantially above the volumes desired for the RO feed in this demonstration project, a meeting with the mill staff on February 20, 1975 disclosed capabilities for collecting, mixing and storage of selected flows from individual stages of the CEHD bleach sequence on the No. 2 softwood bleach line (400 tons/day — 363 t/day). This bleach line was originally operated as a five-stage CEHDP bleach sequence but the peroxide stage had been discontinued. This left the large P stage bleach tower available as a mixing and storage tank for volumes well in excess of the desired 50,000 gallons (189 m^3) of RO feed each day. The seal tank for the P stage was also available for use as a short term storage tank to provide surge capacity for 4,000 gallons (15 m^3) or more of RO preconcentrate as feed to the freeze concentration system.

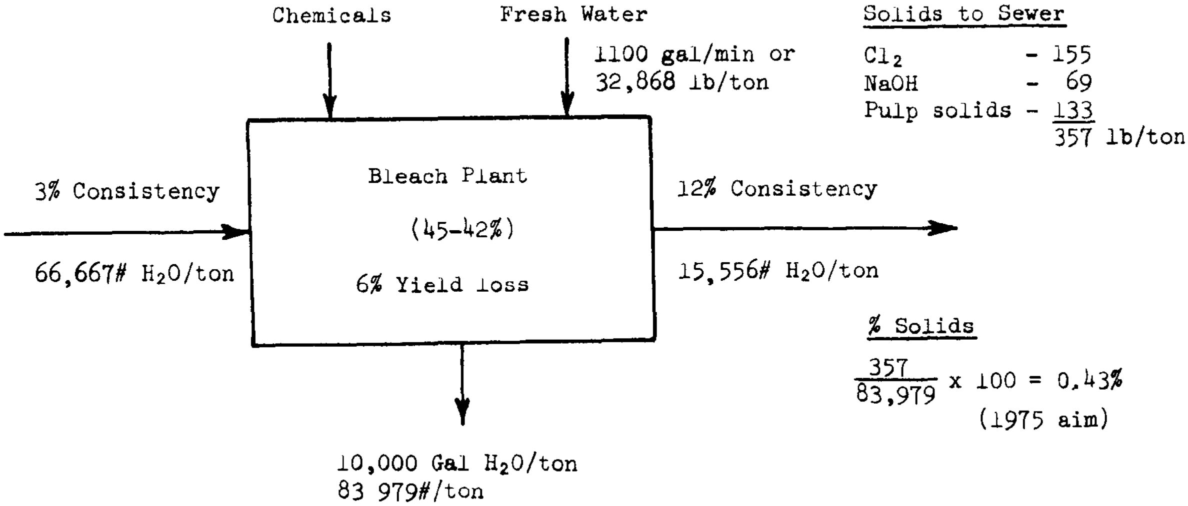

Figure 12. Calculated flows and balances — No. 2 softwood bleach line (400 tons/day). 1975 Goals — Augusta, Georgia mill — Continental Group.

A carefully planned program of sampling and analysis was undertaken by the mill staff with suggestions from Dr. Ferdinand Kraft for the purpose of establishing an effluent collection strategy to provide 60,000 gallons per day (9.5 m^3/hr) of flows from the C, E & H stages equivalent to a 6000 gallons per day (0.95 m^3/hr) usage of water and with a solids content on the order of 0.5%. It was also desired to obtain a feed flow having a pH in the range of 4.5 to 7.5 and as closely as possible equivalent to a stoichiometric balance of Na and Cl. Several blends of the flows from the C, E & H seal boxes were tried experimentally in the mill laboratory and a 15-gallon (57 l) sample was sent to Appleton for a small scale RO trial. A blend comprising 12.5% by volume of Cl_2 stage, 25% by volume of caustic extraction stage and 62.5% hypochlorite bleach flow was finally established as best capable of providing a reliable and reproducible flow for a large field trial. It was decided to proceed with development of the field trial at this mill on the base of having storage facilities and the necessary flow of 50,000 gallons per day (7.9 m^3/hr) simulating a representative kraft bleach plant effluent.

A 4800-gallon (18 m^3) tank truck load of this blend was collected May 20, 1975 and shipped to Appleton for conducting a final confirming trial before undertaking the large scale RO and FC field trials. This preliminary truck load test on kraft bleach effluent, and also the earlier truck load test for the 1st field trial on sulfite bleach effluent from the Flambeau mill, were both completed with use of older, more open membranes available on the trailer prior to installation of new and much tighter membrane equipment early in June 1975. Analytical characterization of the tank truck load and the performance of the RO concentrating system are summarized in Table 14.

TABLE 14. RO CONCENTRATION OF TRUCK LOAD OF BLEACH LIQUOR FROM CONTINENTAL GROUP

Feed volume processed, gallons	4,340
Volume of concentrate, gallons	380*
Total volume pumped (recycled), gallons	39,368
Stoichiometric ratio of feed, Na:Cl	1.25
Total solids (24 hours)	
Feed to RO, g/ℓ	5.57
RO concentrate, g/ℓ	42.19*
Flux rate	
Initial, gfd	18.15
Final, gfd	7.46
Rejections overall	
Total solids, %	76.0
Inorganic chloride, %	67.8
Membrane area (UOP Type 320), ft^2	744
Pressure, psi	600
Operating time, hr	20.5

*Avco laboratories received 200 gallons of RO concentrate for freeze tests.

The preliminary truck load trial confirmed the capability for collecting and processing of a representative kraft bleach effluent from a mill practicing partial recycle (C&E stages). Operation of the RO concentrating system was free from operating problems. Volumetric concentration by a factor in excess of 8X carried the total solids content of the feed liquor from about 0.5% solids to about 4.0% in the final concentrate. Rejections were on the order of 90% for each pass of the relatively open, 4-year old, #320, UOP membrane units available for this run. This rejection was reduced on an overall basis, after the equivalent of 10 or more passes, to about 76% for total solids and 68% for inorganic chlorides. The permeate passing these elderly type #320 membranes carried appreciable amounts of low molecular weight solubles but remained completely clear and colorless. Planning for the second field trial was advanced on the base of these preliminary tests. It should be noted that preliminary testing had resulted in decision to use the relatively tight UOP #520 and closely equivalent ROP #95 membranes with NaCl rejections at the 95% level or better.

Installation of Field Units at Augusta, Georgia

The two trailer mounted field test units were cleaned and rechecked at their respective home base in Appleton, WI (RO unit) and at Avco Systems in

Wilmington, MA (FC unit) during the several weeks intervening between completion of the first field trial in Park Falls, WI July 31, 1975 and the transfer to the second test site at the Continental Group mill in Augusta, GA during the second week of September.

The field test site immediately adjacent to the peroxide bleach tower was especially convenient with all needed facilities close at hand. Cooperation from the mill operating staff and maintenance crews was well coordinated for connecting all utility lines and equipment within the time schedule for the field trial. The layout for the RO and FC units alongside the No. 2 bleach line is presented in Figure 13. The two units are shown operating on site at the mill in Figure 14.

The two trailers were moved to Augusta, GA as scheduled. Utility connections and preliminary tests were completed Monday, September 22, 1976. Brief trial runs for the purpose of training and familiarizing the field crew with the operating program at this location required two additional days. Five ROP ceramic cores found to have been broken in transit during the 1100-mile (1770 km) shipment to Augusta were readily identified and replaced in the crew-training program. This breakage seemed to be at an acceptably low and practical level considering that nearly 5000 of these ceramic cores were mounted on the RO trailer during the long trip to Augusta under usual and normal conditions of commercial trucking.

Provision for Pretreatment of Feed Flows

The preliminary test runs made in Appleton with carboy and truck load quantities of the Continental Group's bleach effluent had shown this product to be remarkably clear with low levels of suspended matter and in other respects readily processed by RO with little need for pretreatment other than temperature control. The RO unit was, however, shipped complete with several auxiliaries (vibrating screen to remove fiber, pH controller, shell and tube heat exchanger for cooling or heating, and temperature controlling instrumentation). Only the temperature controlling equipment and heat exchanger were actually required for operation of the RO field unit at this bleach plant. However, the RO preconcentrate prepared as feed for the FC unit did throw down a small amount of precipitate (probably $CaSO_4$ and Ca oxalate) plus minor accumulations of fiber which required frequent changes of the small string filter cartridges in the feed line to the FC unit. There was little evidence of precipitates or suspended matter in the fresh RO concentrate. But 4000-gallon (15 l) quantities, accumulated in the seal tank as feed for the freeze concentration unit, did show evidence of precipitation after several hours of storage. Analysis and more detailed discussion of these precipitates are provided in the following section covering the freeze concentration tests. Reduction in the small amounts of suspended fiber noted in the FC feed was accomplished by a midtrial change in draw off piping of feed for the RO unit. The take-off line was raised about 7 feet (2.1 m) above the bottom of the cone on the main storage tank and addition of a purge valve to the bottom of the cone permitted draw off of a few gallons of very dilute settlings from the daily charge of 60,000 gallons (227 m^3) of mixed bleach effluent feed.

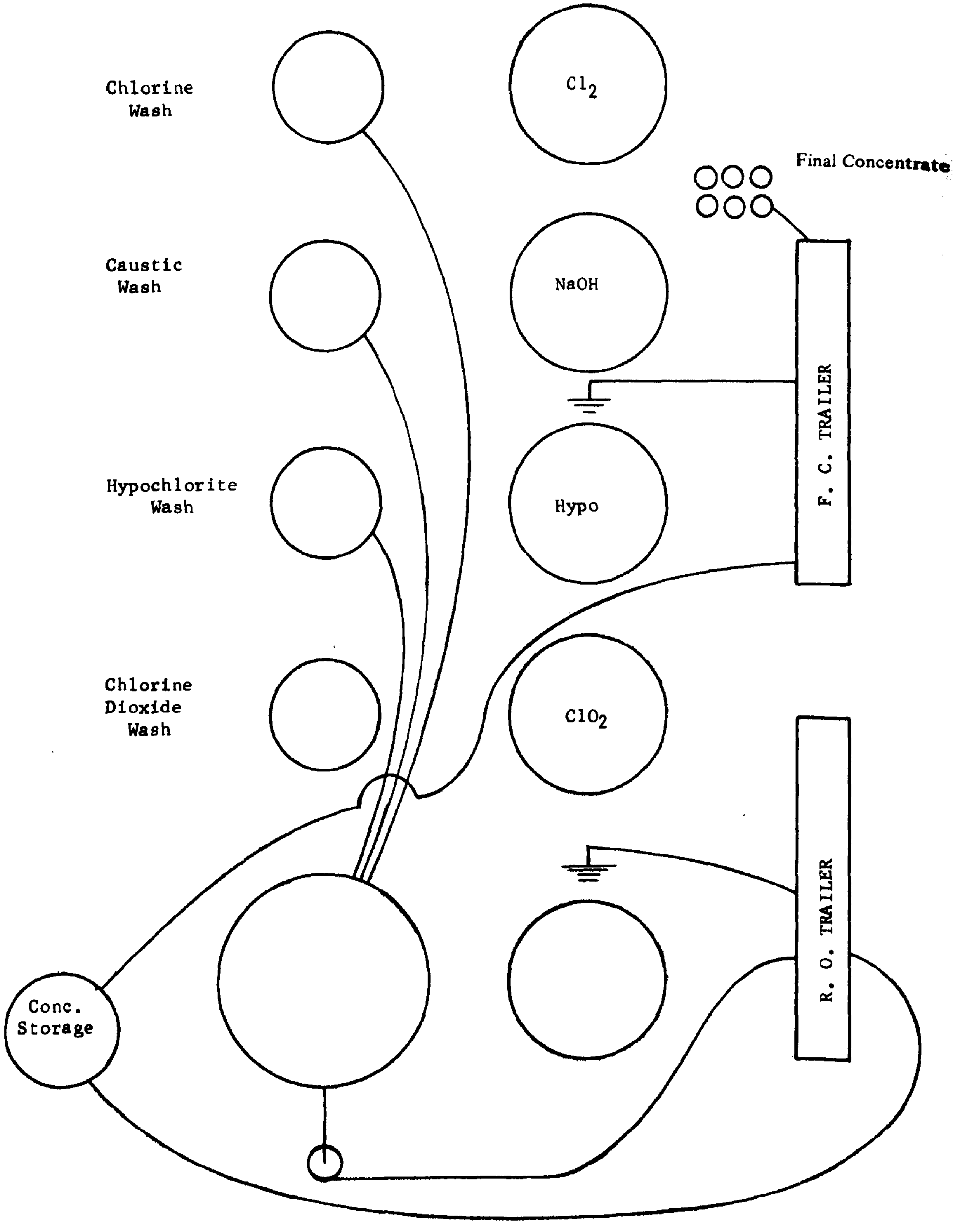

Figure 13. Layout — Continental Can Company, Augusta, Georgia.

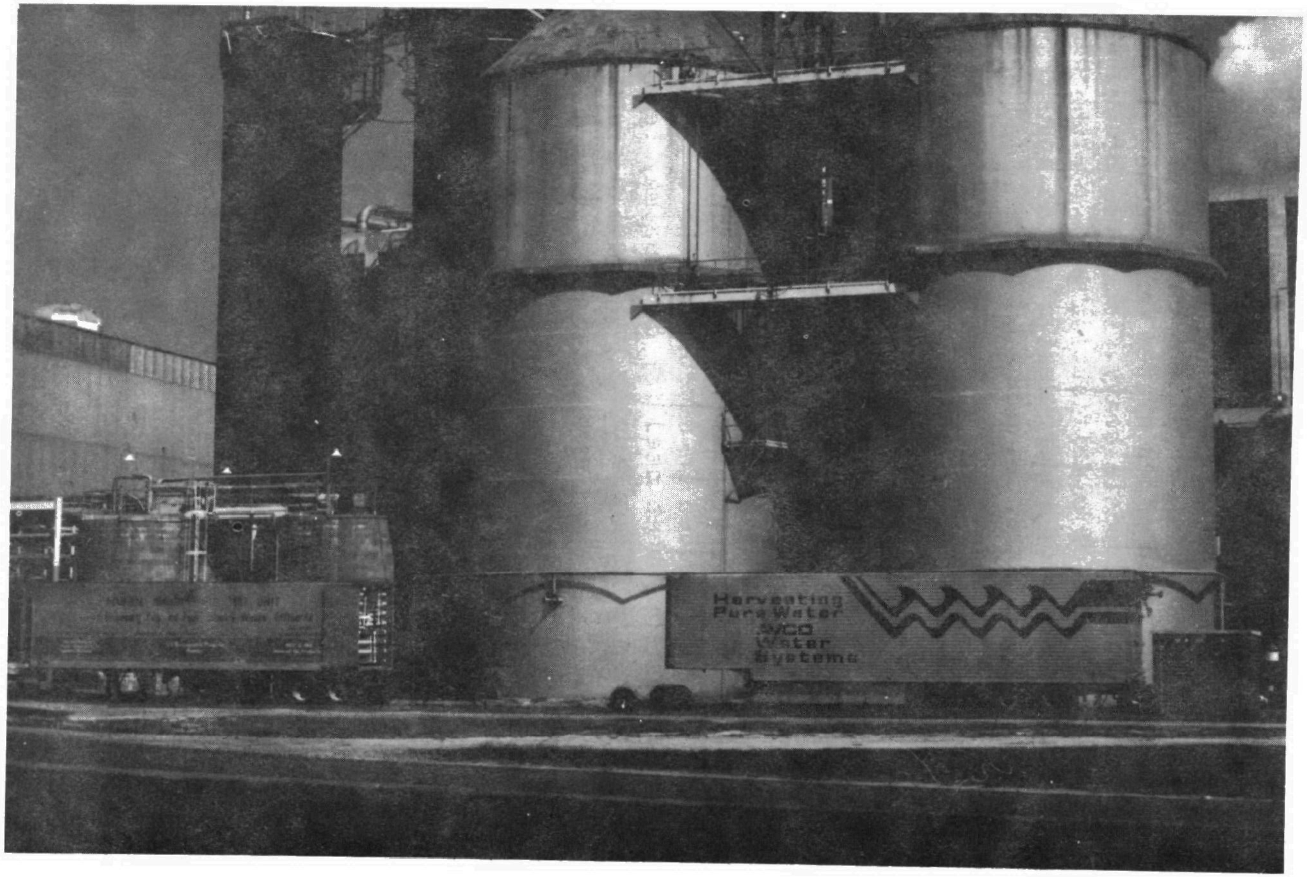

Figure 14. Photograph of trailer units at Augusta, Georgia.

The favorable experience, with little apparent need for pretreatment of the kraft softwood bleach process waters ahead of the RO concentrating system at this mill, contrasted sharply with the critical need for removal of talc and fiber passing the overloaded washers processing hardwood pulp in the Flambeau bleach plant during the field trial. The shorter and finer fibers from hardwood generally result in greater losses of suspended fiber than for softwood process lines and this may continue to be a factor to be contended with in design of bleach liquor concentrating systems. However the field experience in this second field trial at the Augusta mill of the Continental Group indicated the design and manner of operation of the washers was far more significant in affecting the degree of clarification pretreatment required ahead of the RO system. The overloaded older system of bleached pulp washing practiced at the Flambeau mill resulted in heavy discharges of suspended matter which needed substantial levels of clarification even for the RO system. But the kraft bleached pulp washers of modern design at the Augusta mill were operating well within their recommended range of loading and produced clear flows of feed liquor to the RO system. No clarification problems requiring pretreatment ahead of the RO system were apparent. Subsequent need for minor levels of clarification of the preconcentrated RO product may be required ahead of the FC final concentration equipment, especially when the preconcentrate is held in storage for any length of time in a tank provided only with bottom draw off.

Temperature control of the RO feed was practiced throughout the field trial at Augusta but did not show evidence of presenting a high cost pretreatment problem. As had been concluded in discussing the prior Flambeau trial, there were positive indications that need for cooling may be greatly reduced and very probably eliminated with continuing progress in research and development for RO membrane systems.

There was little evidence of need for pH adjustment of the kraft bleach effluent in the preliminary trials or during the on-site operation of this field trial at the Augusta mill. The mixed feed liquors collected from the three bleaching stages were generally in the pH range of 6.0 to 7.5. Readings outside that range occurred occasionally, but briefly, during periods of charging the feed storage tank and before mixing was complete. Design of a commercial operation could be expected to achieve proper mixing and freedom from slugging of pH levels outside the safe range for sustained operation of membrane systems.

Reverse Osmosis Preconcentration--

Operation of the RO unit for data and sample collection was initiated with 10 hours of operation on September 24, 1975. Records for the three week field test program which followed and for a two day extension, November 18 and 19, 1975, are provided in the operating log (Appendix Table C-1). Essential hydraulic data are summarized in Table 15.

Extensive levels of recycle within the RO system, ranging from 27 to 54%, were practiced throughout the main run to permit delivery of adequate and continuous flows of preconcentrate to the FC unit. The need to practice recycle of flows for most of the available operating time handicapped the development of optimum flux rate data. This results from the higher solids

TABLE 15. SUMMARY OF HYDRAULIC DATA FOR RO TRAILER

Continental Group, Augusta, Georgia

Date	Sample no.	Trailer operation, hours	Total flows, gallons: Feed	Perm.	Conc.	Main pump	Recycled, gallons	Recycled, %	Av. flux* rate, gfd
9/24/75	101	10	12,240	10,032	2,208	22,320	10,080	45	9.9
9/25/75	102	7	8,508	7,002	1,506	15,570	7,062	45	9.9
9/29/75	103	5 3/4	8,924	7,620	1,303	12,255	3,332	27	15.4
9/30/75	104	6	9,430	8,107	1,323	14,115	4,684	33	15.6
10/1/75	105	16 1/4	21,232	18,910	2,372	34,845	13,563	39	13.0
10/2/75	106	11	25,405	20,254	5,151	45,395	19,990	44	18.3
10/3/75	107	7 1/4	7,997	6,507	1,490	16,230	8,233	51	10.9
10/6/75	108	5	7,020	5,784	1,236	12,600	5,580	44	13.9
10/7/75	109	8	7,872	6,402	1,470	14,790	6,918	47	9.7
10/8/75	110	17	17,874	15,222	2,652	34,572	16,698	48	11.1
10/9/75	111	23 1/2	19,476	16,158	3,318	40,020	20,544	51	9.4
10/10/75	112	14	22,356	17,640	4,716	33,510	11,154	33	12.5
10/11/75	113	20 3/4	26,316	20,850	5,466	46,290	19,974	43	12.6
10/12/75	114	21 3/4	22,369	18,550	3,819	47,160	24,790	53	10.1
10/13/75	115	22 1/4	22,240	18,696	3,544	48,750	26,510	54	9.9
10/14/75	116	16 3/4	15,236	12,390	2,845	33,135	17,899	54	9.6
11/18/75		5	10,545	6,505	3,960	10,545	0	0	12.9
11/19/75		7.9	17,000	10,602	6,398	17,000	0	0	13.3
Average		12.51						44.4	12.1
Total gallons processed			282,013	224,231	54,777	499,092	217,011		

*Based on total permeate flow with 2,424 ft^2 membrane for period with samples.

concentration and especially the higher osmotic pressures resulting from the approximate 50% NaCl content of the dissolved solids in kraft bleach liquors. The flux performance was however quite favorable in spite of this handicap.

The sixteen operating days during the main field trial included eight days of day shift operation, four days with 2 shifts and 4 days with round-the-clock 3-shift runs with an overall average of 12.5 hours per daily run. Fresh feed from the bleach storage tank processed in the RO concentrating system totaled about 282,000 gallons (1067 m^3). About 224,230 gallons (848 m^3) (80%) of clear, clean permeate water were recovered at an average flux rate of 12.1 gfd (20.6 l/m^2-hr) through 2424 sq ft (225 m^2) of membrane. The soluble solids were concentrated to a volume of about 54,780 gallons (207 m^3).

Mechanical operations for the RO trailer unit were relatively free from interruption and equipment failure during the three programmed weeks of operation. However, near the end of the program, a burst in the concentrate hose line flooded and burned out the DC power supply to the Manton-Gaulin main pressurizing pump. This accident, apparently caused by a mill forklift left parked over the pressurized hose line while millwrights were repairing severe storm damage within the mill system, necessitated termination of the main RO run for this field trial 3 days ahead of schedule.

The premature shut down occurred just prior to a planned conversion to straight through feeding and operation of the RO system without recycle. Data from straight through operation were needed to better confirm the flux rates and rejections to be expected without recycle. The field unit was, therefore, retained on the mill site for a six-week period while factory representatives rebuilt the burned out rectifier for the power supply. The RO field crew returned to Augusta November 10, 1975 and, after reinstallation and testing of the rectifier, resumed operations to obtain the needed flux rate data on two final days of operation, November 18 and 19, 1975.

Sample Collection, Transportation and Analysis--

Refrigerated and automated samplers were employed to collect composites of the feed, permeate and concentrate flows to and from the RO field test unit. One gallon quantities of the precooled composite samples plus additional grab samples collected to evaluate specific membrane performance capabilities were shipped daily to the Appleton laboratory in insulated containers by air freight. Prompt analysis was routinely scheduled for specific gravity, pH, total solids, COD, BOD_5, Na, soluble Ca, inorganic Cl^-, and color. Viscosity, osmotic pressure and suspended solids were also analyzed for selected samples. The detailed analytical data for the 48 composited samples from daily recycled operations of the RO field unit are recorded in Appendix Table C-2. Analyses of the 13 sets of grab samples collected hourly from the two days of straight through operation without recycle are recorded in Appendix Table C-3. Grab samples were also collected for evaluating internal performance of the RO system for which detailed analytical data are tabulated in Appendix Table C-4. Advanced RO concentration data for solids levels ranging from 2 to 4% are recorded in Appendix Table C-5.

Loading and Rejection Performance of the RO Field Unit at Augusta--

Table 16 summarizes and evaluates the extensive analytical data for the 16 days of sustained concentrating runs for this field trial on CEH stage bleach waters processed at the Continental Group mill. Rejections were well in excess of 90% for the important categories of COD and color and also importantly for soluble calcium. These are soluble components which would be particularly of concern in developing closed recycle systems for bleach process waters. Rejections were found to be on the order of 70 to 80% for the total solids, inorganic chlorides and sodium.

The daily runs, processing an average of 15,000 gallons (57 m^3) of CEH bleach liquor feed and containing from 265 to more than 1200 pounds (120-544 kg) of total solids, did lose 20 to 30% of the low molecular weight solids (chiefly Na and Cl) in the permeate. Losses in this category were greater than might be desired but appear to be well within the range of acceptability. This is especially true in view of the degree of recycle (2X) within the RO system necessary to deliver the necessary 3.5 gpm (13.2 l/min) flow of RO preconcentrate to the FC trailer unit for the final stage of concentration. Reduction or elimination of internally recycled flows in design and operation of RO concentrating systems may make possible substantial increases in the overall rejection and recovery of these smaller molecular sized components in the concentrates. However, subsequent trials of straight through operation were handicapped by equipment limitations and not fully conclusive in this respect.

The wash-up losses recorded in the material balances developed in Table 16 ranged widely from less than 5% for total solids in some of the better runs to as much as 73% loss of calcium in other trial runs. Wash-ups were undertaken at the end of each daily run as a precaution to avoid the possibility that irreversible fouling of the membranes might occur during shutdown periods. These precautions were probably much more elaborate than actually needed and reflected the concern developed in the prior experience with foulding by the nearly colloidal suspensions of talc in the Flambeau field trial. RO concentrating systems for commercial operation would be designed to substantially reduce or eliminate losses in this category.

The data in Table 16 account for the overall material balance in these runs and serve to show the need for proper design and operation of a membrane concentrating system to avoid dilution and losses when cleaning and regenerating membranes. These data are of primary significance for demonstrating that the RO membrane concentrating system can be effectively employed for recovery and substantially complete removal of those bleaching components (COD, color, Ca, and possibly also oxalates and pitch) which are of primary concern in increasing the degree of recycle within a bleach process water system. There was no evidence of pitch and talc fouling problems at the Augusta mill trial. The data also demonstrate the capabilities of the RO process for effectively rejecting 60 to 80% or even more of the Na and Cl ions and for concentrating and removing this major fraction from the bleach process water system. It is to be expected tha the 20 to 40% fraction of Na and Cl components passing through the membrane with the permeate water would result in a substantial build-up of Na and Cl components within a recycled bleach process water system. However, withdrawal of the 60 to 80% slice of the Na and Cl input from the bleaching process should provide a leveling off of the build-up at

TABLE 16. RO LOADING AND REJECTION SUMMARY

(Data Available only for Recycle Operation)
CEH Kraft Bleach Run – Augusta, Georgia

Date	Sample no.	Sample	Total solids Pounds	Total solids Rej.	Total solids Lost in washup Pounds	Total solids Lost in washup %	COD Pounds	COD Rej.	COD Lost in washup Pounds	COD Lost in washup %	Sodium Pounds	Sodium Rej.	Sodium Lost in washup Pounds	Sodium Lost in washup %	Soluble calcium Pounds	Soluble calcium Rej.	Soluble calcium Lost in washup Pounds	Soluble calcium Lost in washup %	Inorganic chloride Pounds	Inorganic chloride Rej.	Inorganic chloride Lost in washup Pounds	Inorganic chloride Lost in washup %	BOD$_5$ Pounds	BOD$_5$ Rej.	Color Pounds	Color Rej.
		Feed	506				134.0				161.4				2.9				198.3				23.0		167.5	
9/24/75	101*	Perm	152	.70	+17	+3	15.2	.89	2.1	1.6	54.4	.66	+3.0	+1.9	0.1	.95	1.5	54	76.9	.61	2.8	1.4	5.9	.75	4.7	.97
		Conc	371				116.7				110.0				1.2				118.5				--		--	
		Feed	351				94.4				114.3				1.8				134.5				15.2		103.0	
9/25/75	102*	Perm	148	.58	+59	+17	8.5	.91	7.9	8.4	37.4	.67	+4.3	+3.7	0.1	.94	0.9	50	50.8	.62	+3.1	2.3	3.6	.77	1.3	.99
		Conc	262				77.9				81.2				0.8				86.8				--		--	
		Feed	354				86.7				113.2				1.7				142.2				13.2		70.4	
9/29/75	103*	Perm	57	.84	147	42	5.7	.93	36.5	42	20.7	.82	47.4	42	Trace	.99	1.3	73	3.1	.98	85.8	60.3	2.2	.84	0	1.00
		Conc	150				44.5				45.1				0.5				53.3				--		--	
		Feed	284				66.4				90.8				1.7				106.4				12.7		52.3	
9/30/75	104*	Perm	88	.69	43	15	6.4	.90	16.9	25	31.9	.65	19.6	22	Trace	.99	1.3	73	45.1	.58	8.3	7.8	3.0	.76	1.0	.98
		Conc	153				43.1				39.3				0.5				52.9				--		--	
		Feed	719				183.6				235.1				3.9				292.5				30.5		108.3	
10/1/75	105	Perm	230	.68	194	27	19.6	.89	67.6	36	89.3	.62	52.6	22	0.5	.87	2.3	59	119.8	.59	75.9	26	9.0	.71	1.3	.99
		Conc	295				96.5				93.2				1.1				96.8				--		--	
		Feed	926				261.8				301.9				4.7				363.8				44.3		107.1	
10/2/75	106*	Perm	223	.76	+27	+2.9	19.1	.93	38.2	15	87.7	.71	+20.1	+6.7	0.3	.94	2.0	43	120.7	.67	+25.6	+7.1	10.1	.77	0	1.00
		Conc	730				204.5				234.3				2.4				268.8				--		--	
		Feed	302				78.1				97.4				1.5				120.7				11.2		67.7	
10/3/75	107*	Perm	74	.75	16	5.3	6.4	.92	12.2	16	29.4	.70	0.3	0.3	0.1	.92	0.7	46	40.8	.66	0.2	0.06	2.7	.76	0.4	.99
		Conc	212				59.5				67.8				0.7				79.8				--		--	
		Feed	265				68.5				85.5				1.3				105.9				9.8		58.6	
10/6/75	108*,†	Perm	70	.74	19	7.2	5.6	.92	13.2	19	27.5	.68	1.1	1.3	Trace	.99	0.8	60	39.0	.63	1.0	0.91	2.9	.71	0	1.00
		Conc	176				49.7				56.9				0.5				66.0				--		--	
		Feed	297				76.9				95.9				1.5				118.8				11.0		65.7	
10/7/75	109*,†	Perm	85	.71	+18	+6.1	6.3	.92	4.7	6.1	34.0	.65	+11.5	+12	0.1	.93	0.7	46	46.4	.61	+11.0	+9.2	2.9		0	1.00
		Conc	230				65.9				73.4				0.7				83.3				--		--	
		Feed	888				228.1				296.9				4.6				360.3				35.9		194.9	
10/8/75	110‡	Perm	255	.71	174	20	2.9	.99	87.1	38	100.5	.66	55.3	19	0.5	.89	2.7	58	137.0	.62	60.6	17	9.2	.73	1.3	.99
		Conc	459				138.1				141.1				1.5				162.7				--		--	

(continued)

TABLE 16 (continued).

Date	Sample no.	Sample	Total solids Pounds	Total solids Rej.	Total solids Lost in washup Pounds	Total solids Lost in washup %	COD Pounds	COD Rej.	COD Lost in washup Pounds	COD Lost in washup %	Sodium Pounds	Sodium Rej.	Sodium Lost in washup Pounds	Sodium Lost in washup %	Soluble calcium Pounds	Soluble calcium Rej.	Soluble calcium Lost in washup Pounds	Soluble calcium Lost in washup %	Inorganic chloride Pounds	Inorganic chloride Rej.	Inorganic chloride Lost in washup Pounds	Inorganic chloride Lost in washup %	BOD_5 Pounds	BOD_5 Rej.	Color Pounds	Color Rej.
10/9/75	111‡,§	Feed	882				217.3				285.0				4.5				352.4				48.9		135.3	
		Perm	216				15.5	.93	77.0	35	84.4				0.5				112.1	.68	126.5	36	8.2	.83	1.5	.99
		Conc	—				124.8				—				—				113.9				—		—	
10/10/75	112#	Feed	1.209				297.9				390.6				6.2				483.0				67.0		185.4	
		Perm	297	.75	203	17	20.6	.93	86.2	29	103.3	.74	63.3	16.2	0.4	.93	3.6	57	146.5	.70	80.8	17	10.1	.85	22.9	.98
		Conc	709				191.0				224.0				2.2				255.8				—		—	
10/11/75	113	Feed	596				145.6				193.3				3.2				245.0				29.3		189.5	
		Perm	138	.77	144	24	10.3	.93	53.8	37	48.9	.75	54.1	28	Trace	.99	2.2	67	69.5	.72	64.4	26	4.8	.84	0	.99
		Conc	314				81.6				90.3				1.1				111.0				—		—	
10/12/75	114	Feed	858				205.0				266.3				4.5				340.1				40.2		117.9	
		Perm	199	.77	300	35	4.9	.98	104.1	51	76.1	.71	80.7	30.2	0.4	.92	2.9	65	107.2	.68	101.5	30	8.6	.79	3.4	.98
		Conc	359				96.0				109.5				1.2				131.4				—		—	
10/13/75	115+	Feed	762				191.2				237.1				4.0				302.8				41.5		240.3	
		Perm																								
		Conc	333				88.9				100.5				1.0				92.1				—		—	
10/14/75	116§	Feed	320				75.9				103.3				1.6				125.1				16.4		94.8	
		Perm	71	.78	75	23	5.2	.93	21.9	29	27.4	.74	24.7	24	Trace	.99			35.4	.72	26.4	21	3.4	.79	0	1.0
		Conc	174				48.7				51.3				0.5				63.3				—		—	
Average				.73				.93				.70				.95				.70				.80		.99

*Computed from grab samples.

†Based on feed of sample No. 107.

‡Computed from composite samples.

§Some flow data missing.

#Feed data taken from No. 111 sample.

+Permeate sampler malfunction.

tolerable levels. The extent to which the build-up would occur in any particular bleach recycle system would require further study of specific bleach recycle systems.

Confirming evidence for the effectiveness of the RO membrane system in rejecting the soluble materials contained in kraft bleach liquors during the Continental Group field trial is further available in Table 17. Grab samples were taken from the individual four stages of the RO field unit on four separate days, including 3 days for which the unit was operated on a straight through feed basis and one with recycled feed. Rejections for COD averaged 96%, color nearly 100%, and soluble Ca nearly 98%.

Removal of soluble calcium ions capable of accumulating and forming scale deposits within the bleaching and papermaking equipment lines is likely to be a critical factor in development of bleach process water recycle systems. The quantities of soluble calcium ion in the bleach feed liquors averaged 20 to 40 mg/liter in this RO field trial which would be indicative of a state of saturation or supersaturation for the less soluble calcium compounds chiefly responsible for scale deposits. The highly effective levels of rejection and removal in the RO concentrating system points to a possibly important area of use for RO in effectively removing precursors responsible for scale formation and thereby increasing the degree of recycle which could be achieved in a bleaching system. The RO concentrates did show increased levels of soluble Ca in proportion to the degree of concentration but with little if any evidence of precipitation being apparent when freshly concentrated at the 2 to 4% solids level during the short periods of hold up in the RO system and under periods of continuous operation. Fouling of the membranes by Ca scale did not seem to take place in this trial at the kraft mill bleach plant in Augusta, as was probably the case at the Flambeau Ca base sulfite pulping and bleaching operation trial. There was little evidence of flux improvement after the trials of sequestrant (Versene 100) wash up at this mill. It was concluded that Ca fouling was much less in evidence in this field trial. The actual need for membrane regeneration by the relatively expensive Ca sequestering agent was difficult to assess within the short period of operation at Augusta. On the other hand, RO concentrates which were stored overnight did show evidence of precipitation and were responsible for plugging the small, string type, filter cartridges ahead of the freeze concentration unit. It remains to be determined whether scaling by Ca compounds would be a problem in the freeze-concentration step.

The overall performance of the RO preconcentrating system in operation at the Augusta mill is summarized in Table 18 with results of staged, straight through feeding presented in the first column and with the results of recycled feeding to accomplish somewhat higher levels of concentration in the second column. The degree of concentration achieved was much less than desired due to anticipated need for volumes of 3.5 gpm (13 l/min) of preconcentrate to the freeze concentration field unit and also due to the high velocities of feed required for efficient operation of the Rev-O-Pak RO modules. However 62.6% of the feed volume was recovered as clear colorless permeate product water in the straight through feed mode and 79.5% of the bleach feed input was recorded as clear colorless water of similar quality in the recycle feed mode of operation. The overall rejection ratio for soluble solids was at the 0.81 level

TABLE 17. PERFORMANCE OF FOUR SUCCESSIVE MEMBRANE CONCENTRATION STAGES

Kraft Bleach Process Water — Continental Group, Augusta, Georgia

(Data Averaged from Internal Grab Samples on 4 Different Days)*,†

Stage	Total solids, g/ℓ	Rejection ratios — $\left(1 - \frac{\text{permeate}}{\text{feed}}\right)$							Viscosity, cp. 25°C	Osmotic pressure, psi
		Total solids	COD	Na	Soluble Ca	Inorganic Cl	BOD_5	Color		
Feed	6.74	--	--	--	--	--	--		0.730	75
Stage I	8.87	0.88	0.95	0.86	0.99	0.84	0.84	1.00	0.741	85
Stage II	10.78	0.84	0.95	0.83	0.98	0.80	--	--	0.739	101
Stage III	12.68	0.87	0.96	0.84	0.97	0.81	--	--	0.746	121
Stage IV	13.37	0.84	0.97	0.82	0.96	0.77	--	1.00	0.748	138

*Overall flux rate averaged 12.3 gfd with water recovery at rate of 60-65%.

†Grab samples taken:
October 11, 1975 at 2:00 p.m., without recycle
October 14, 1975 at 3:00 p.m., with recycle
November 18, 1975 at 12:30 p.m., without recycle
November 19, 1975 at 9:25 a.m., without recycle.

for staged operation and 0.85 for recycle. The osmotic pressure of the concentrate more than doubled in each mode of operation and was realized to be an important factor in reducing the flux rates of the RO Process.

TABLE 18. ABBREVIATED SUMMARY OF PRINCIPAL DATA FOR RO PROCESS EVALUATION CONCENTRATION OF KRAFT CEH BLEACHING STAGES

	Staged* operation	Recycle† operation
Solids in feed to overall system, av. g/ℓ	5.70	4.51
Solids in feed to first membrane stage, av. g/ℓ	5.70	9.10
Solids in concentrated product, g/ℓ	13.7	15.06
Degree of concentration of feed in system	2.40X	3.34X
Solids rejection from 1st stage feed $1 - \left(\frac{\text{Permeate}}{\text{1st stage feed}}\right)$	0.81	0.85
Water product recovery (permeate), % of feed volume	62.6	79.5
Indicated overall flux rate, gfd (after 3 hours' operation)	13.9	12.1
Osmotic pressure of feed to 1st stage, psi	67	44
Osmotic pressure of concentrate, psi	137	128

* Average of 13 hourly samples (Appendix Table C-3).

† Average of 15 daily samples (Appendix Table C-2).

Figure 15 summarizes the results of concentrating 5 five hundred-gallon (1.9 m^3) batches of the preconcentrate from the 10 to 15 g/l solids level to 60 g/l or higher levels of concentrated solids. Flux rates at the 12 to 13 gfd (20-22 l/m^2-hr) level for 10 to 15 g/l solids preconcentrates dropped to less than 2 gfd (3 l/m^2-hr) at solids concentrations above 50 g/l. At 50 to 60 g/l solids the osmotic pressure increased to between 500 and 650 psi (3447-4481 kPa), thus reducing the RO effective working pressure to practically zero for the UOP type of RO modules and the pressurizing pump available for this concentrating study on the Continental Group bleach liquor. Subsequent experience with the ROP modules operated at pressures to 750 psi (5171 kPa) with preconcentrate from the third field trial served to indicate much higher and more practical rates of the flux were feasible with the ROP equipment, which was designed to operate at pressures ranging above 750 psi to 1000 psi (5171-6890 kPa) or even higher.

The RO studies for the field trial on the kraft CEH bleach process waters were concluded with the production of about 550 gallons (2.1 m^3) of preconcentrate at 4% solids and 280 gallons (1.1 m^3) at 6% solids concentration. These RO products were made available for freeze concentration and for further evaluation of routes to final disposal or for utilization of the solids recovered in concentrating this bleach process water.

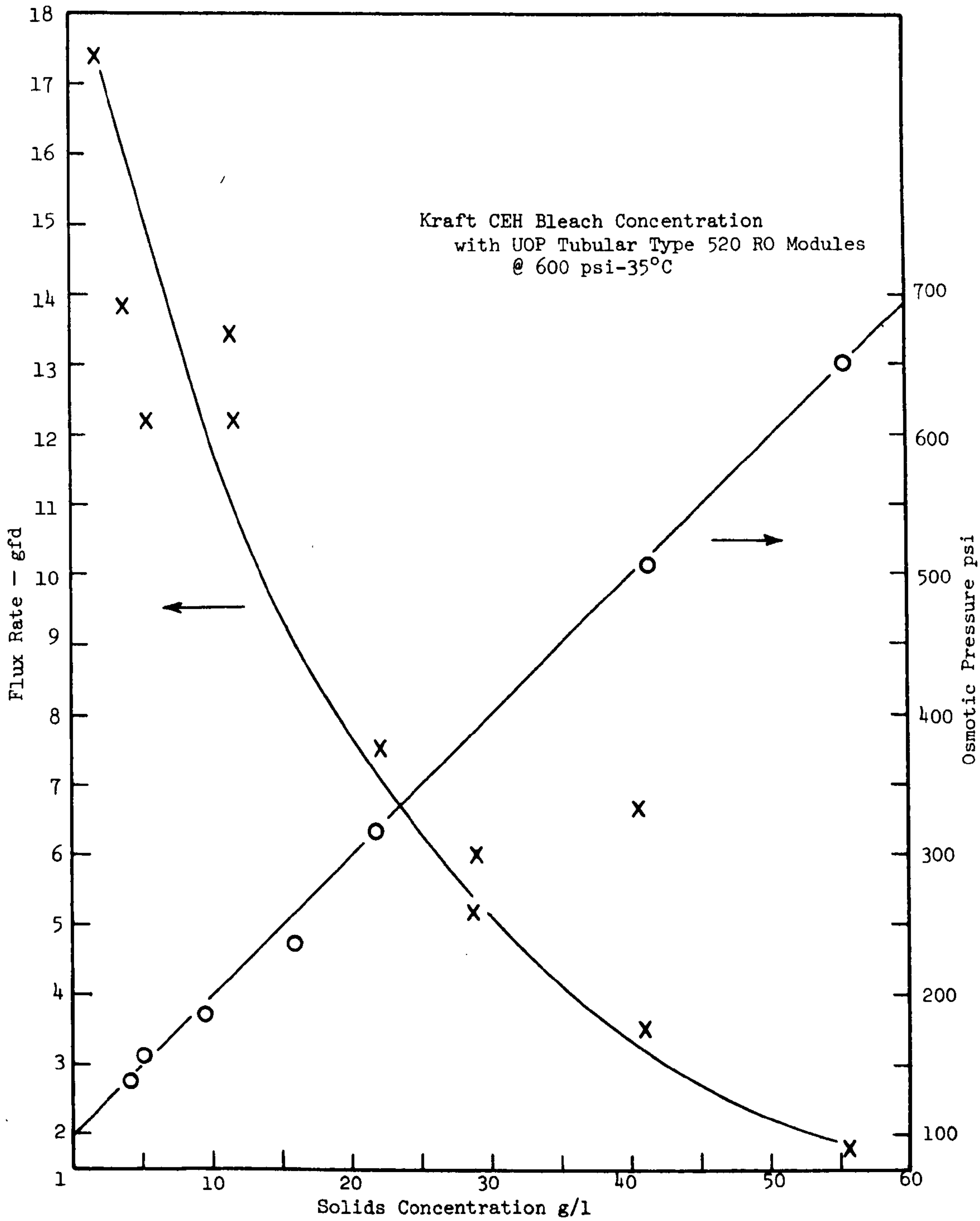

Figure 15. Relation of flux rate and osmotic pressure to solids concentration.

Overall recovery of clean and colorless permeate water exceeded 90% of the original feed volume. The quality of the permeate water recovered in concentrating to at least 4% solids appeared to be highly suited for recycle and reuse within the bleaching system and particularly so for the final stages of washing the pulp in a multistage bleaching sequence. Some color was apparent in the permeates recovered at concentration levels above 4% solids where high levels of recycle were practiced. The volume of the final stage permeate water was very low (less than 5% of the total permeate water volume) and might suitably be returned to the process water recycle system or disposed in other ways without materially affecting the pulp washing efficiency.

Freeze Concentration Trial at Continental Group — Augusta Mill

The operating plan for the freeze concentration field trials at Continental Group was similar to that at Flambeau. Changes made to the heat rejection section were successful, and no problems were encountered due to overloading of the heat rejection compressors. Air was bubbled through the samples taken for analysis, thus stripping out the refrigerant and eliminating the shipping problem encountered at Flambeau. Table 19 is a summary of the daily operation at Continental Group's Augusta mill.

Single stage testing at Continental Group started on September 25. Initial testing indicated that a single stage could operate at a freezing temperature of -3.3°C. Although this temperature was not as low as at Flambeau, the concentration corresponding to this temperature was still considerable — about 6.8%. Foaming was experienced during this trial.

Two-stage FC testing began on October 1 and continued for the remainder of the test period. Freezing temperatures of -5.5°C were reached and could be maintained during the tests. Steady open loop FC operation was achieved on three days of testing at Augusta. Product quality was extremely good during these periods of steady operation. Conductivity under 100 micro mhos/cm was maintained with a conductivity of 40 being maintained during the run on October 10. However, operation of the second stage wash column continued to be erratic during much of the operation. Continuous operation was attempted starting on October 8. Due to the great amount of operator attention required, it was not possible to maintain steady conditions during a continuous test and two-shift operation was resumed on October 13. Excessive loss of refrigerant occurred throughout the testing and the system was shut down on two occasions to check for leaks. No large leaks were found. The largest loss of refrigerant was with the concentrate, as no provision for stripping this stream was provided in the mobile laboratory. The amount of loss in this stream without stripping was not normally significant. With this concentrate, the concentrate decanter was not effective and refrigerant content as high as 2% was measured in the concentrate. This refrigerant could be easily separated from the concentrate in a centrifuge indicating that a larger decanter would solve this problem.

FC Data and Discussion of Results

Table 20 is a summary of the principal data from the Continental Group run (the analytical data are given in Table 21). Freezing point vs.

concentration is shown in Figure 16. Because of the greater freezing point depression, the recovery in the first stage was only 75% even though the initial concentration from the RO was lower (1.5% — 2.0%) than at Flambeau (about 5%). This led to a slightly higher recovery in the second stage even though the final concentration was not as great. Product water quality is the same. The correlation between conductivity and TDS, Figure 17, is not good but clearly indicates quite acceptable values. A final concentration of 11% TDS was attained and it appeared that higher values might be possible. Greater emphasis was paid to steady operation than at Flambeau at a sacrifice of reaching the maximum final concentration.

TABLE 19. DAILY SUMMARY AVCO MOBILE LABORATORY

Continental Group Mill Operation
September 25 — October 16, 1975

Date	Hours operation	Hours* open loop	Single (I) or two (II) stage	Conc.† temp., °F 1st/2nd	Product cond., μ mhos/cm²	Comments
9/25	3	--	I	29.4	11,500	Check
9/26	7.5	--	I	29.7	150	Check
9/29	8.5	3	I	26	150	Concentrating
9/30	11	2.8	I	28	500-5,000	Foaming
10/1	8.5	--	II	29/28	300	Start 2nd stage tests
10/2						General maintenance
10/3	8.8	5.3	II	28/22	100	Steady 5 hours
10/4						Check systems for leaks
10/7	8.8	--	II	29/22	1,100	
10/8	15.5	2.7	II	28/24	50-300	
10/9	17.3	4.2	II	29/23	300-7,000	
10/10	24	11.4	II	29/22	40	Steady 9 hours
10/11	2	2	II	29/23	6,000	
10/13	13	3.5	II	29/22	150-850	
10/14	10.5	3.5	II	29/23	150	
10/15						Check systems for leaks
10/16	10	4.7	II	30/23	70	Steady 4 hours

*Hours open loop — period when feed is being brought in system and concentrate and product are being discharged. Other periods of operation are termed closed loop when concentrate and product are mixed together to form feed.

†Concentrate temperature is temperature of concentrate in freezer and corresponds to concentration as shown on curves.

TABLE 20. SUMMARY OF PRINCIPAL DATA AVCO MOBILE LABORATORY CONTINENTAL GROUP TEST RUN

Solids in feed, g/ℓ	11-17
Solids after first stage, g/ℓ	60
Solids after second stage, g/ℓ	110
Degree of concentration	6X-10X
Solids in recovered water, g/ℓ	0.20
Freezing point, first stage, °C	-3
Freezing point, second stage, °C	-5.5
First stage recovery, %	75
Second stage recovery, %	11
Overall freezing system recovery, %	86

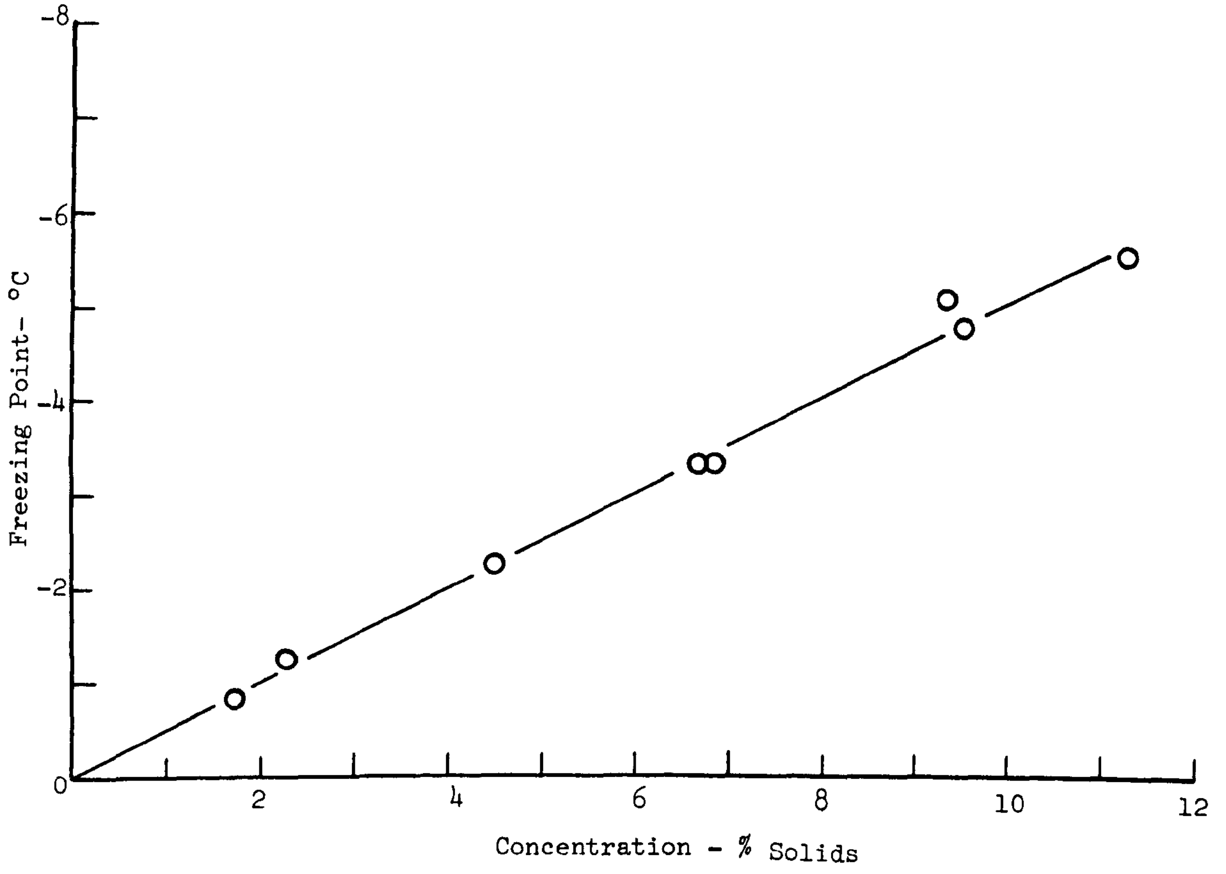

Figure 16. Continental Group freezing point correlation.

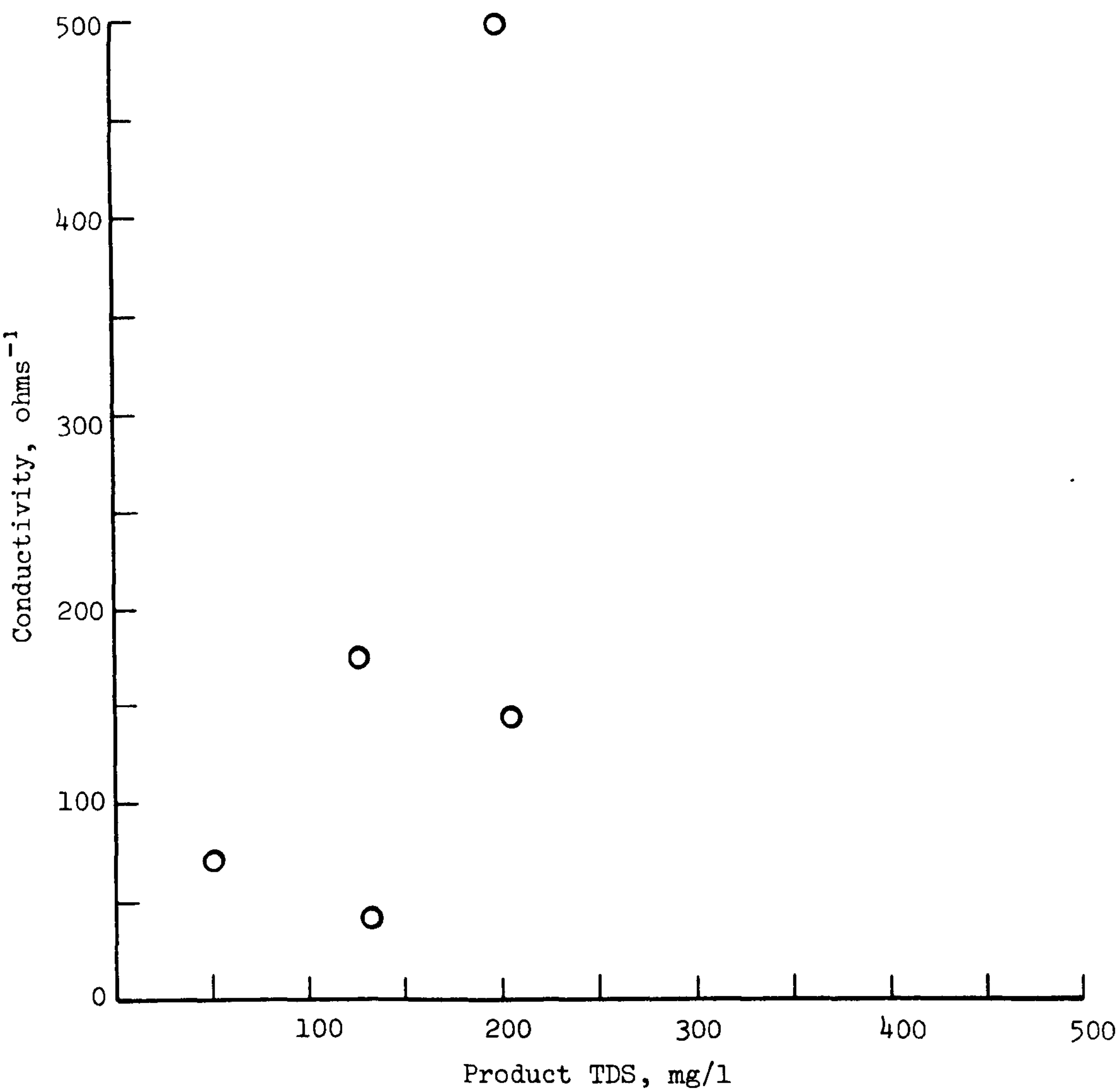

Figure 17. Freeze concentration product water quality correlation.

Suspended solids in the RO preconcentrated feed were noticeably lacking at Continental Group. This is attributed to a cleaner RO feed stock. However, noticeable solids were found in the FC product, which necessitated replacement of the cartridge filters. These suspended solids contained a high oxalate concentration. We think it is not a serious problem and can be quite easily handled in a commercial plant.

Second-stage wash column performance was quite a bit better than at Flambeau. This was perhaps partly due to a slightly higher yield in the second stage, although the more significant difference appears to be the lack of solids in the concentrate. However, the second-stage wash column still required a lot of operator attention and caused several upsets. The addition of a screen heater did not appear to improve the performance in that no noticeable difference in operation could be observed with the heater either on or off.

Foaming both at Flambeau and Continental Group was such that a defoamer was required. At Flambeau, Diamond Shamrock Foamaster VL was used effectively, but at Continental massive doses were required. A defoamer used in the pulp mill, BASSO #894, was found to be effective and used for most of the testing. Dosage rates of 75 ppm based on the feed rate were used at both Flambeau and Continental. This was in excess of the minimum requirements but no attempt was made to minimize the quantity.

Table 21 summarizes analytical data from grab samples collected intermittently during six of the better days of FC operations. These samples were shipped to the Institute laboratories in Appleton for analysis concurrently with corresponding RO samples. The RO preconcentrate ranging somewhat over 1% solids was concentrated by FC about 10X to more than 10% solids. The recovered melt waters were of excellent quality, in these assays. Dissolved solids were substantially less than 200 mg/liter with Na and Cl ions both averaging less than 50 mg/liter. COD and color units were also less than 200 mg/liter.

The field concentration trials were limited in the degree to which the level of concentration could be carried due to capacities of the equipment available for both RO and for FC. These limitations, on the order of 50% of that desired for each system, were extended by further concentration runs conducted in Appleton (RO) and in Avco's Wilmington, MA laboratory for the FC products. Both RO and FC were readily demonstrated capable of reaching the originally programmed levels of 5% preconcentrate for the RO system and 25% solids for the final concentrates from freeze concentration. The highly concentrated products were produced in sufficient quantity for further evaluation of final disposal or utilization of the recovered bleaching residues.

Overview of the Continental Group Field Trial at Augusta

The experience gained in the RO and FC operations in the second field trial substantially improved upon the prior performance in the first trial at the Flambeau mill. Gains were especially apparent in freedom from pretreatment problems arising from need to remove suspended solids. Very little fiber settled out in the feed liquor collection tower and there was no evidence of residual suspensions of talc such as that arising from pitch control operations at the Flambeau mill. Washers operating at design loadings at Augusta appeared capable of delivering feed liquors with quite acceptably low levels of fiber and of other suspended solids to the RO system.

The presence of oxalic acid in the bleach liquors from both trials necessitated a substantial analytical program to better assess the nature of any problem which might arise from formation of insoluble calcium oxalate. Chemical analysis of the concentrates did confirm the presence of oxalic acid and electron microscope studies of the surface of membrane samples removed from several tubes in the assembly showed small accumulations of the calcium oxalate salt and also calcium sulfate and carbonate. However, precautionary routine cleanups with Versene-100 (EDTA) before weekend or other prolonged shutdowns along with high velocity maintained in the tubes during operation of the unit apparently served to control scaling from this source without buildup of a fouling problem. The actual need for including the Versene

TABLE 21. ANALYTICAL DATA

Grab Samples from Avco Freeze Concentration Trailer Unit

Kraft Bleach Process Water — Continental Group, Augusta, Georgia

Sample no.	Sample*	Date	Sp. gr., 35°C	pH	Total solids, g/ℓ	COD, mg/ℓ	Sodium, mg/ℓ	Soluble Ca, mg/ℓ	Inorganic Cl, mg/ℓ	Color, mg/ℓ‡	Viscosity, cp. 35°C	Osmotic pressure, psi
103	FA	9/29/75	1.004	7.30	13.84	4,093	4,150	42	4,906	--	0.750	134
	CAI		1.015	8.45	33.30	35,075	6,700	116	8,765	--	0.824	217
	MA	"	0.994	7.64	0.18	184	21	Trace	33	78	--	--
104	FA	9/30/75	1.003	7.23	13.84	3,905	3,560	41	4,784	--	0.761	130
	CAI		1.044	8.44	98.70	92,150	22,080	218	25,482	--	1.017	729
107	FA	10/3/75	1.005	6.88	17.05	4,786	5,450	56	6,418	--	0.754	143
	CAII		1.023	8.17	112.60	--	37,600	192	39,474	--	0.907	1,086
	MA	"	0.995	7.45	0.13	126	45	Trace	32	130	--	--
115	FA	10/13/75	1.002	7.33	11.74	3,137	3,544	35	3,249	--	0.741	109
	CAII		1.046	8.13	75.73	23,006	23,120	72	27,567	--	0.838	704
	MA-1†	"	0.995	6.76	0.09	129	16	Trace	18	30	--	--
	MA-2†	"	0.994	7.00	0.08	60	19	Trace	20	32	--	--
116	FA	10/14/75	1.002	7.58	11.58	3,251	3,420	34	4,223	--	0.754	99
	CAII		1.066	8.02	109.20	38,311	34,240	218	40,308	--	0.907	1,046
	MA	"	0.994	8.05	0.25	101	72	Trace	78	86	--	--
117	FA	10/16/75	1.002	7.58	11.58	3,251	3,420	34	4,223	--	0.754	99
	CAI		1.012	8.00	21.80	7,324	6,920	60	8,025	--	0.758	204
	CAII	"	1.067	8.11	108.30	35,870	34,640	222	40,230	--	0.916	1,119
	MA	"	0.994	8.51	0.05	12	9	Trace	8	16	--	--

*FA — RO concentrate or feed to Avco unit. CAI — Avco concentrate — Stage I. CAII — Avco concentrate — Stage II.
MA — melt or recovered water from Avco unit.
†MA-1 and MA-2 — before and after filter.
‡In terms of platinum in Standard Methods chloroplatinate color standard.

washups remained as an incompletely answered question.

The RO field unit again failed to reach the programmed levels of concentrate volume and flowrate [3.5 gpm (13 l/min) and 5% solids] when operating at low levels of recycle; the FC unit could not be placed in the continuous two-stage concentration mode. In other respects both of these units did provide impressive flows of clean, clear and colorless product water of high quality upon which a program for substantially increasing the degree of recycle to be achieved in bleaching process water systems could be developed.

Accidental Damage to the RO Main Drive and to the Membranes

The premature shutdown of the RO trailer unit occasioned by overpressurization and bursting of the rubber hose on the final concentrate collection system was the first serious mechanical breakdown of the trailer unit in the 8 years of its operation at various field test sites and intermittently on the Institute campus. This hose burst, apparently caused by parking of a maintenance crew forklift over the line, sprayed concentrate liquor upwards into the otherwise drip-proof ventilation system for the rectifier with resultant electrical shorting out of the AC/DC main motor power supply. Two control modules within the Statohm rectifier unit were destroyed. The resultant emergency shutdown required innovative use of auxiliary pumps to achieve the usual shutdown washup and membrane cleaning routines. The membrane system trailer was placed on standby storage and the operating staff returned to the home base in Appleton for the several weeks required for factory staff repair of the Statohm power converter and controller unit.

Completion of repairs subsequently enabled the unit to be reactivated at Augusta for a brief 3 day run needed to develop additional data on operation at low levels of recycle and to accumulate a truck load of the RO preconcentrate.

The trailer unit and the 5000-gallon (18.9 m^3) tank truck load of preconcentrate were returned to Appleton for continuing studies on higher level membrane concentration and followup FC concentration studies.

However, test runs of the unit after its return to Appleton disclosed the entire system of membranes had been partially hydrolyzed in some manner as a result of the emergency shutdown at Augusta. A critical loss of NaCl rejection was apparent for the entire set of membranes. It was, therefore, not possible to resume use of the trailer unit for the concentrating studies on the 5000-gallon (18.9 m^3) truck load of Augusta preconcentrate.

The membranes which had retained their rated 95% rejection (for a single module) consistently throughout the first and second field trials over the preceding 5 months were found capable of no better than 70% rejection.* They appeared satisfactory in other respects, including high levels of color rejection, freedom from leaks and no apparent accumulation of scale or other foulants. All attempts to restore the rejection such as by developing a dynamically formed surface membrane coating were without success.

*Rejection data given in other parts of the text are for several modules in series.

Thus, a smaller membrane unit with high NaCl rejection membranes was developed to carry out the final concentration of the truck load shipment and to extend the studies on the third field test site at the Chesapeake mill.

Studies to determine the cause for the loss of NaCl rejection failed to disclose a clear definitive answer. The Institute staff and representatives of the membrane equipment suppliers were agreed that alkaline hydrolysis of the membranes seemed to have occurred at some time during the six-week shutdown. High temperature buildup in the stored trailer and high pH levels from emergency washup procedures seem likely causes, individually or together.

The electrically operated heating and ventilation system of the trailer had proven highly reliable during the 8 years of operation but power interruptions during the shutdown may have occurred as a result of the mill reconstruction activities and thus permitted a high temperature buildup in the closed trailer during the still very warm autumn weather in southern Georgia. Further hydrolytic damage to the membranes could have occurred if the emergency washup measures undertaken with auxillary pumps failed to completely neutralize the alkaline BIZ detergent and Versene chelating agents, or if these reagents were incompletely rinsed from the system before the storage period. The operating staff had carried out normal precautions to avoid such eventualities but the substitute pumping assembly was makeshift at best and it proved impossible to determine the exact train of events leading to the loss of rejection.

Insurance coverage was available to reimburse the costs of repairing the clearly defined, accidental damage to the electrical power supply unit, but could not be extended to the supplementary, less well defined, and partial damage to the membrane system. Since the project budget had no provision for the high cost of replacing the entire coat of membranes [nearly 2500 ft^2 (232 m^2)] for the trailer mounted RO field unit, it became necessary to revise the continuing program to permit operations with much smaller scale equipment. Limited sources of supplementary funding and with excellent cooperation from the membrane equipment suppliers enabled equipping a moderately sized test stand with 300 ft^2 (27.9 m^2) of new membrane modules, 12 from Universal Oil Products and 10 from Rev-O-Pak. Much experience had been gained with the smaller unit employed as a membrane life test stand for two prior years.

III. FIELD TRIAL AT CHESAPEAKE CORPORATION

The Chesapeake Corporation's kraft pulp and paper mill at West Point, Virginia was producing about 1150 tons per day (1043 t/day) of chemical pulp at the time of this field trial. About 900 tpd (816 t/day) was unbleached softwood pulp with the remaining 250 tpd (227 t/day) being a hardwood market pulp bleached by an oxygen bleaching sequence. Approximately 250 tpd (227 t/day) of recycled kraft fiber were also used in the manufacture of 26 to 69 lb (127-337 g/m^2) linerboard, which is the chief paper product of this mill.

The oxygen bleaching system provided an opportunity to test, for the first time, membrane and freeze concentration processes on effluents from this new bleaching technology. Additionally, bleach liquors would be more concentrated than at The Continental Group and Flambeau Paper Company mills as this mill uses much less water per ton of bleached pulp.

The Chesapeake oxygen bleaching system, based on the process developed by MoDoCel in Sweden, was put in operation at West Point in 1973. Figure 18 presents the flow pattern of the bleaching system based upon the D/C OD sequence. Brownstock (after dilution with about half of the D/C stage washer effluent) is drawn from the brownstock storage tank. Chlorine and ClO_2 are added in a Kemics mixer ahead of the two chlorine stage towers for the combined first stage of bleaching. The D/C stage washer removes a substantial portion of the soluble residues with highly acid chloride content. Recycle of one half the D/C washer effluent for dilution of the brownstock leaves about 700 gpm (2.6 m^3/min) for discharge to the large new Unox waste treatment plant.

The washed pulp from the D/C stage is pressed to remove excess quantities of chlorine and water. It is then mixed with caustic and steam before injection into the oxygen stage reactor. The pulp, after the oxygen bleach, is blown to a tank and then washed before the final ClO_2 bleaching and washing steps. The oxygen and ClO_2 bleach washers each discharge about 300 gpm (1.1 m^3/min) to the waste treatment plant sewer along with an additional 150 gpm (0.57 m^3/min) of pump seal water and related smaller waste flows from the bleach plant. The total bleach effluent discharge to the waste treatment facility, therefore, totals about 1450 gpm (5.5 m^3/min). We understand that this volume remains relatively constant regardless of the amount of pulp being bleached in the range of 250 to 400 tons of pulp per day (226-363 t/day). Calculations show that water usage in this bleach plant was 6950 gal/ton (29 m^3/t) of bleached pulp for 300 tpd (292 t/day) and 5200 gal/ton (21.7 m^3/t) for 400 tpd (363 t/day) production.

The new oxygen waste treatment plant (Unox) in operation at the Chesapeake mill was achieving high levels of efficiency in terms of BOD and suspended solids removal. That $20 million investment substantially achieved compliance with environmental regulations at the mill. Major additional expense for corrosion resistant bleach washing systems to permit any further reductions in the volume of water usage for added or supplementary bleach waste treatment would necessarily be subject for careful evaluation of costs and benefits. Such added expense would have to be justified in terms of increased bleaching efficiency, improved bleach product yields, substantial reduction in the cost of bleaching or similar significant process and product improvements.

Preliminary RO Lab Trials

Arrangements for a small preliminary test run of RO concentration of the Chesapeake oxygen bleach system effluent were made soon after the project extension to this mill was first suggested in May 1975. A 10-gal (37.9 l) shipment to the Institute laboratories in Appleton was processed July 17 and 18, 1975, using a single Rev-O-Pak test core with a high rejection membrane. Table 22 summarizes the data from the run which started with a feed liquor at 3.95 g/l total solids and a pH of 6.3. The changes in content of Na, inorganic Cl, organic Cl, total organic carbon (TOC) and free Cl_2 were analyzed.

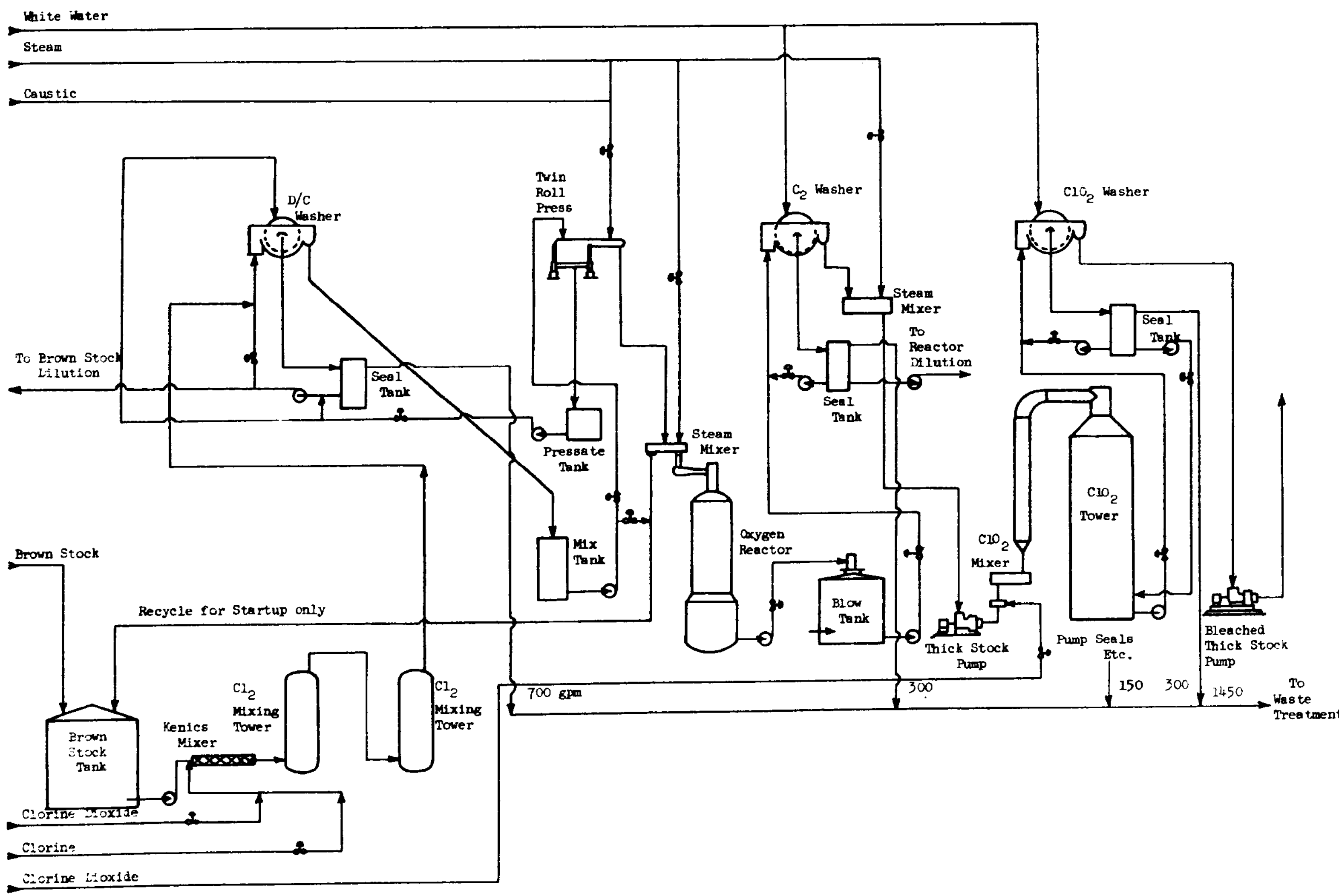

Figure 18. Bleach plant flow diagram — Chesapeake Corp., West Point, Virginia.

TABLE 22. ANALYTICAL DATA — PRELIMINARY RO LABORATORY TRIAL

Concentration of Chesapeake Corporation Bleach Plant Effluent
Single Loop of Rev-O-Pak R.O. Single Core Tube

Sample	Time	Date	Total solids gm/l	Total solids Rej. ratio†	pH	Sodium mg/l	Sodium Rej. ratio	Inorganic chloride mg/l	Inorganic chloride Rej. ratio	Organic chloride mg/l	Organic chloride Rej. ratio	TOC mg/l	TOC Rej. ratio	Chlorine
Feed	11:55 AM	7/17/75	3.95		6.30	938		845		399		625		0
F1	2:54 PM	"	5.04		--	--		--		--		--		--
P1			0.22	0.96	--	--		--		--		--		--
F2	8:25 AM	7/18/75	7.75		--	--		--		--		--		--
P2			0.34	0.96	--	--		--		--		--		--
F3	1:00 PM	7/18/75	18.38		--	--		--		--		--		--
P3			1.13	0.94	--	--		--		--		--		--
Final C	2:35 PM	7/18/75	48.83		6.60	8120		8747		Low		3600		--
Combined F			0.50	0.99	7.05	130	0.98	178	0.98	41	--	50		0.99
				0.87*			0.86*		0.79*		0.90*			0.92*

*Based on original feed. — 10 gallon shipment.

†Rejection ratio = 1 (concentration of permeate/concentration of feed).

The test run concentrated the liquor more than 10X to 48.8 g/l total solids with over 95% rejection of the analyzed components.

Before conducting the field trial at the Chesapeake mill, an additional larger scale test run was conducted in the Institute laboratories. The mill shipped two 50-gal (0.19 m^3) drums of fresh oxygen bleach process effluent for this test which utilized two 18-tube UOP modules with relatively tight No. 5 RO membranes. Table 23 summarizes data (for details see Appendix Table D-1) from this additional run.

This 100-gal (0.38 m^3) run, although relatively brief in duration (5-1/2 hours), confirmed earlier results. Color rejection, although not analyzed on all samples, was excellent. Flux rates were expectably high for the short periods of operation in these preliminary tests. The limited supply of feed liquor did not permit sufficient operation at each level of concentration to accurately determine the effect of concentration polarization and fouling of the membrane surface. These important criteria could only be checked with longer term operation. This 100-gal (0.38 m^3) feed sample had a pH of about 3.9 (as with the first sample). The Na and Cl contents seemed to be in reasonably close balance with no large excess of Cl^-, which would be of concern for membrane stability.

Evaluation of the project data from the two large field runs at the Flambeau and Continental Group mills had raised concerns over the high insoluble oxalate content in the various types of bleach feed liquors to the RO and FC systems. Of particular interest was the fate of precipitated oxalates as concentration advanced.

The expectation that the oxygen stage bleaching reactions might lead to relatively high content of oxalates was confirmed. The feed liquor analysis showed 660 mg/l of Na oxalate [equivalent to 40 lb/ton (20 kg/t)]. The concentration appeared to quadruple in the first stage of concentration. The recovery of precipitated oxalates, however, appeared to fall off rapidly in subsequent stages of concentration. These observations seemed to tie in with prior observations throughout the project. Qualitative tests readily demonstrated the presence of traces of oxalates but quantitative analysis for oxalates seemed to indicate little evidence of scaling or fouling build-ups on the membranes or other critical equipment. Loss of oxalates as deposits on tank walls and piping was not checked. In instances of expected membrane fouling due to oxalates, the problem could be avoided by forming and removing insoluble oxalates ahead of the RO and FC systems.

RO Field Trial at West Point, Virginia

The smaller scale RO field test stand developed and used for the Chesapeake field trial has been described in the equipment section of this report (Section V). The unit was trucked from Appleton to West Point by the two Institute staff members who had been responsible for field trial operations throughout this project. It was set up in and around the bleach plant pumphouse at the mill with the layout shown in Fig. 19. Bleach effluent feed flows to the RO system were pumped to the Sweco 100-mesh (149 μ) vibrating screen mounted on the pumphouse roof. The screened feed liquor was then

TABLE 23. PERFORMANCE OF RO MEMBRANE SYSTEM — PRELIMINARY LABORATORY TRIAL

Concentration of Chesapeake Corporation Bleach Plant Effluent
Single Loop of 2 UOP RO 18-Tube Modules in Series

			Total solids			Sodium		Inorganic chloride		Sodium oxalate,
Sample	Time	Date	g/l	Rej. ratio*	pH	mg/l	Rej. ratio*	mg/l	Rej. ratio	mg/l
Feed #1	11:30	1/26/76	4.784		3.88	1,166		803		
#2			3.564		4.00	760		921		
F-1	11:50	1/26/76	4.89		3.93	960		918		663
P-1				0.90	3.88	27	97.2	24	97.4	1.4
F-2	2:12	1/26/76	17.36		3.80	3,080		3,318		2,512
P-2				0.97	3.67	76	97.6	74	97.8	1.0
F-3	3:30	1/26/76	17.48		3.88	3,120		3,318		
P-3				0.97	3.70	82	97.4	94	97.2	0.1
F-4	3:55	1/26/76	29.76		3.90	5,480		5,643		
P-4				0.98	3.60	129	97.6	177	96.9	2.8
F-5	4:20	1/26/76	39.36		3.89	7,520		6,178		
P-5				0.98	3.57	183	97.6	183	97.0	2.4
FC-6	4:50	1/26/76	43.95	0.98	3.95	7,560		8,715		
FC-6A			39.72		3.95	7,520		7,463		
FC-6B			6.85		4.45	1,280		1,183		
P-6					3.52	498		659	92.4	2.1
CP-6			0.485		3.72	77		86		6.3

*Estimated rejection, based on composited permeate.

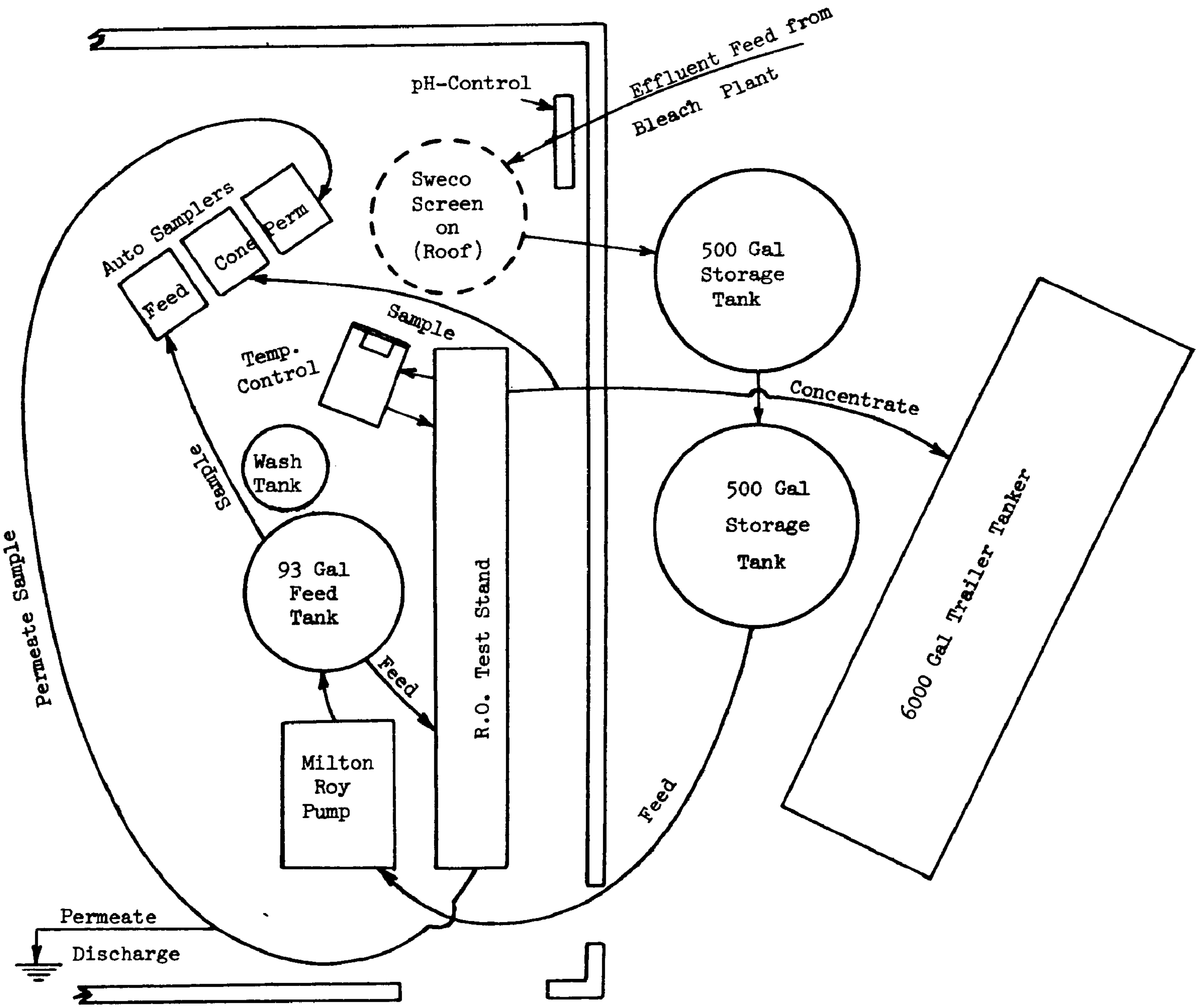

Figure 19. RO setup at Chesapeake Corp., West Point, Virginia.

accumulated in two 500-gal (1.9 m^3) polyethylene tanks ahead of the Milton Roy duplex piston pump which metered the flow to a 93-gal (0.35 m^3) level controlled tank ahead of the Goulds multistage centrifugal pressurizing and recycling pump on the test stand. Feed, concentrate and permeate were automatically sampled. Temperature and pH controllers were available to maintain proper operating conditions throughout the run.

The three flow patterns used in operating the small field RO unit at Chesapeake are outlined in Figure 20a (feed thru mode); Figure 20b (recycle mode); and Figure 20c (concentrating mode). The feed thru mode evaluates the amount of water removed at maximum permeation rates at any stage of concentration, particularly the early stages, in which large amounts of total water removal could be achieved at minimum concentration polarization and fouling effects. However, in order to assess the long term operational behavior at higher levels of concentration, it was necessary to operate the equipment under the recycle and the concentration modes for most of this field trial.

The two pressurizing pumps available for this field trial had limited ranges of flow. The duplex Milton Roy piston pump was rated at 0.5 to 5.9 gpm (1.9-22 l/min) while the multistage Goulds centrifugal pump operated best at 20 gpm (76 l/min) or more. The test stand was set up for comparative evaluation of the performance of the UOP and ROP tubular modules under conditions requiring an in-between flow range of 10 to 15 gpm (38-57 l/min). It was, therefore, necessary to use the larger centrifugal pump with a by-pass as the main pump and to use the excellent metering capabilities of the piston pump to control and measure the feed liquor flow to the test stand. The unit was set up and successfully test operated in the first week of April 1976.

Several unexpected operating problems were soon apparent:

1. Major construction activities underway in the pulp mill area frequently interrupted the bleach plant operation. Shut downs, cleanups and startups of the bleach plant occurred almost daily during the three-week field trial at West Point. The delays and interruptions experienced cut the time available for steady state, continuous, straight through feeding and operation studies.

2. Each shutdown and cleanup resulted in substantial discharges of fiber to the bleach plant sewer. This overloaded and plugged the Sweco screen in the feedline at times. Still, the short and fine hardwood fiber passed through the 100-mesh (149 μ) screen into the RO feed supply. Various remedies to counter this problem were undertaken, including emergency purchase of a finer screen and automated screen cleaning assembly. However, the best solution to the fiber problem was found to be the accumulation of 1000 gallons (3.8 m^3) of clear feed when the bleach plant was in full operation with little or no fiber losses apparent. Two 500-gal (1.9 m^3) polyethylene storage tanks were used for this purpose.

3. The interruptions in bleach plant operation also resulted in slugs of very dilute liquor at times. Accumulating 1000 gal

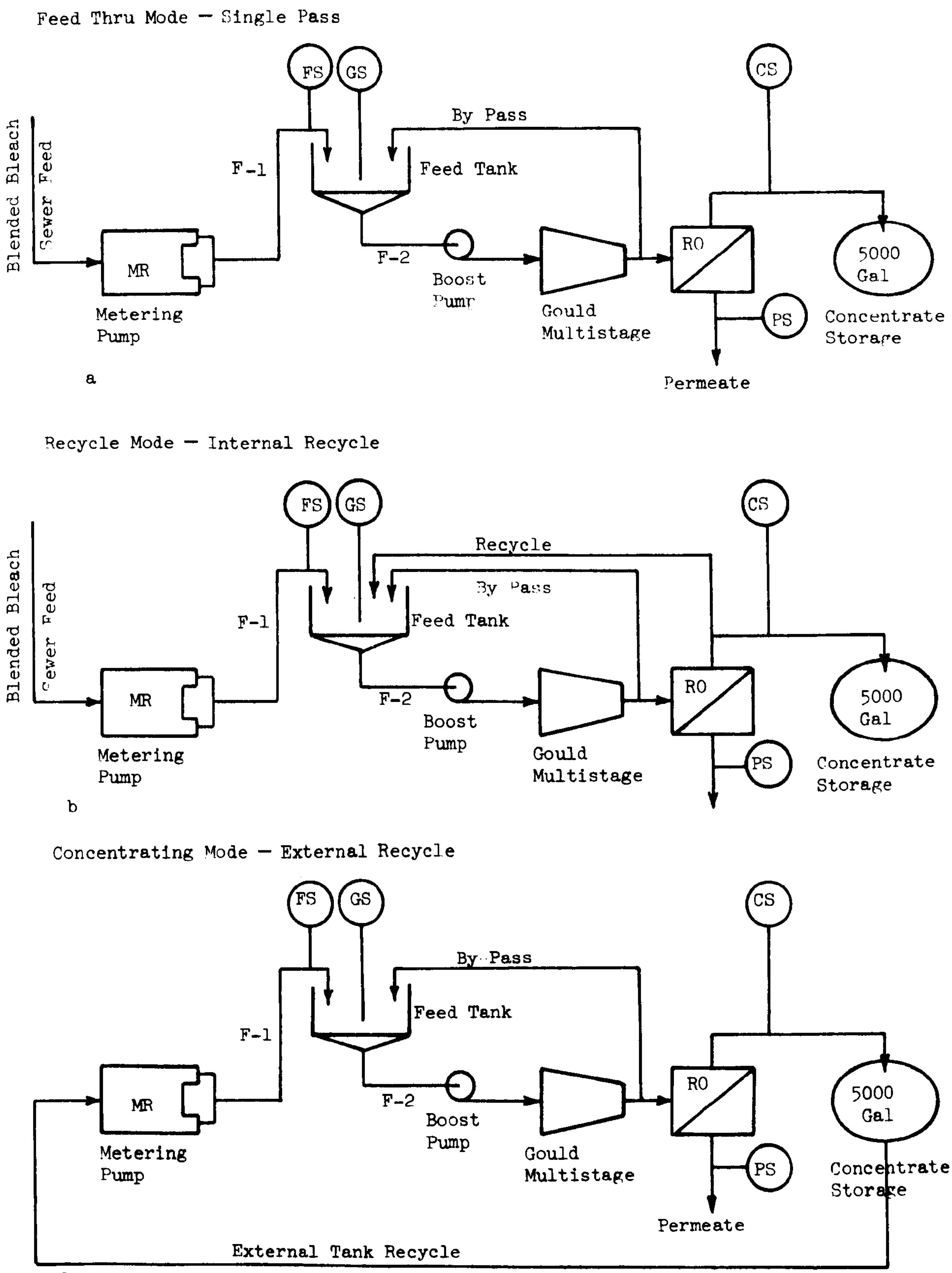

Figure 20. Three modes for operation of small RO field test stand Chesapeake O_2 bleach plant.

($3.8\ m^3$) of normal strength feed liquor when the bleach plant was in full operation minimized this problem.

4. A chief disadvantage of the batch type feed storage system was that the feed liquor to the membrane system was aged (up to 48 hours). Some problems in extrapolating the fouling characteristics of fresh liquor from data on aged liquor were, therefore, to be expected.

5. Another problem arose in the first week of test operations when analysis of the fresh feed showed pH levels of 2.5 to about 4.0 and substantial acid Cl^- content. This also refuted the earlier evidence of an apparent close balance for Na and Cl residues observed in the two samples shipped to the Institute laboratory. Because at pH below 4.0 membrane degradation is known to occur, these adverse, on site findings necessitated revision in the planned program for conducting the field trial. To prevent membrane damage, an auxiliary feedline was, therefore, installed from the bleach plant to supply a small flow of high pH oxygen bleach stage effluent for neutralizing the principal flow of bleach plant sewer feed coming to the RO field unit.

A short 5.8-hour run was undertaken with untreated bleach sewer feed at a time when the pH was relatively high. Analysis of a composite sample of this feed liquor is compared in Table 24 with that for the averaged analytical data from composited samples collected during 4 separate days of subsequent operation utilizing feed liquors neutralized to the range of 6.5 to 7.0. The addition of the alkaline oxygen stage effluent for neutralization did not radically change the character of the feed liquors other than achieving the desired pH.

Operating Data - Three Week Run April 12-30, 1976

Hydraulic data from the daily operating logs during the eleven days of sustained operation for the field run are summarized in Table 25. Operating time varied with the availability of feed liquor from the bleach plant and also within the limitation of the capabilities of the 2-man operating staff to maintain reliable operating routines. The three automated and refrigerated samplers were a much valued asset but still required close supervision with much time required for packing and delivery of the composited samples to the airport for shipment to the Institute. The operating schedule called for the two men to maintain 4 six-hour shifts upon occasion. Daily runs ranged from 5.75 hours on the first day to nearly 26 hours for one around the clock run. The daily average was 17.0 hours.

The three weeks of active field trial studies during April 12 to 30, 1976 included a number of short period special start-up trials and also special concluding studies in addition to 11 days of sustained operations (187.3 operating hours) for developing the operating data.

TABLE 24. COMPARISON OF UNTREATED AND NEUTRALIZED BLEACH SEWER FEED

Chesapeake Field Trial

	Untreated	Neutralized*, av.
pH	4.0	6.7
Total solids, g/liter	5.0	5.6
COD, mg/liter	2265	2560
Soluble Ca, mg/liter	119	62
Na, mg/liter	1090	1300
Inorganic Cl^-, mg/liter	1301	1035
Oxalate, mg/liter	209	80
BOD, mg/liter	830	870
Color, units	2660	2945
Osmotic pressure, psi	43	58
Electrical resistance		
21°C, ohms	201	222
35°C, ohms	131	140

*Average of 4 daily composites (Samples 8, 9, 14, and 15).

Bleach effluent fed to the RO unit, totaling about 51,800 gal (196 m^3) in 11 days, ranged from 1,716 gal (6.5 m^3) of fresh bleach feed during the first day to nearly 8,000 gal (30 m^3) of fresh feed for full operating days and averaged 4,710 gpd (17.8 m^3/day). As much as 26,000 gal (98 m^3) of mixed feed flow per day were actually fed to the system at recycle rates ranging from 66 to 81% of the total flow when operating in the recycle and concentrating modes.

The operating data recorded in the daily logs maintained during the field trial in West Point are summarized in Appendix Table D-1. The unit was operated with the feed temperature maintained at 38-40°C for much of the time and only rarely dropped to 35°C for short periods. The Rev-O-Pak (ROP) modules were fed at flow rates of 10 to 20 gpm (38-76 l/min) and with the feed pressure maintained at 600 to 610 psi (4136-4205 kPa). Under these feed conditions the ROP modules had a uniform 5 psi (34 kPa) pressure drop and delivered a flow to the UOP modules at 595 to 605 psi (4102-4171 kPa). The pressure drop observed with the UOP modules ranged from 35 to 45 psi (241-370 kPa). The overall flux rates for the test stand were around 10 to 12 gfd (17-20 l/m^2-day) during initial operation on fresh feed liquors with 5 to 6 g solids/liter in a straight thru mode and exhibited a progressive drop to about 5 gfd (8.5 l/m^2-day) as the concentration increased above 15 g/liter to a

TABLE 25. SUMMARY OF HYDRAULIC DATA

Third Field Trial — Chesapeake

Date	Sample no.	Mode of* operation	Unit operation, hours	Total flow, gallons: Feed	Perm.	Conc.	Main pump	Recycled, gal	Recycled, %	Av. flux[†] rate, gfd
4-15-76	7	Thru	5.75	1716	851	871	5,907	4,191	70.9	11.50
4-16-76	8	Thru	22.83	6047	2717	3222	23,137	17,090	73.9	9.24
4-17-76	9	Thru	13.13	2517	1122	1280	12,991	10,474	80.6	6.64
4-19-76	10	Conc.	7.58	2447	839	1632	7,641	5,194	68.0	8.60
4-20-76	11	Conc.	25.89	7925	2019	5819	26,097	18,172	69.6	6.06
4-21-76	12	Conc.	21.54	6095	1087	5010	21,712	15,617	71.9	3.52
4-23-76	14	Thru	18.75	5781	1855	3848	18,900	13,119	69.4	7.68
4-24-76	15	Conc.	13.08	3462	1069	2116	13,184	9,723	73.7	6.35
4-25-76	15A	Conc.	13.50	2453	954	1499	13,608	11,155	82.0	5.49
4-26-76	16	Conc.	21.00	7047	1898	5149	21,168	14,121	66.7	7.02
4-27-76	17	Conc.	18.49	6316	1366	4950	18,638	12,322	66.1	5.74

*Thru = no recycle of concentrate; Conc. = recycle of 100% concentrate back to feed supply.

†Based on total permeate flows, 309 ft^2 membrane.

maximum of 40 g/liter in the concentrating modes.

Detailed analytical data for the field test are presented in Table 26. Corresponding loading and rejection data were calculated and summarized in Table 27. The quality of the permeate waters recovered in all modes of operation was exceptionally high throughout the entire 3 weeks of operation. Rejections were on the order of 95-99% for most components routinely analyzed. Even the BOD rejection ranged upwards of 88%, a level much higher than normally experienced. The flux rates were somewhat less than normally experi enced for new membranes. It seems that the membrane equipment suppliers had provided new, very "tight" (high rejection) membranes for the smaller field test stand having substantially higher rejection ratings than the 95% NaCl rejection level for the membranes with which the large trailer unit had been equipped.

The acquisition of field data demonstrating the capabilities for recovery of permeate waters of exceptionally high quality from bleach liquors should prove to be useful under some industrial situations and as such be a positive value coming from this project. But the recovery of such high quality water with the higher rejection grade of membrane probably would not be required or be economically attractive in most commercial bleach plant operations.

The high quality permeate water recovered in the Chesapeake field trial further confirmed the results of the earlier field trials with the large trailer mounted unit. It demonstrated the capability of the RO membrane system to recover excellent quality water for reuse as a bleach wash water. The water recovered in initial stages of concentration (recycle mode with 40% water recovery) approached the standards for potable water with less than 300 mg/liter total solids, 150 mg/liter NaCl, less than 1 mg/liter Ca, and with one rare exception, practically complete removal of color (less than 1 color unit/liter). As expected, water quality deteriorated at higher levels of concentration and with further recycle thru the membranes (up to 90% water recovery). Since a large proportion of the total water recovery occurs in early stages of concentration, the overall permeate water quality was still indicated to be very good for reuse within the pulp mill and bleach plant.

The high level membrane rejection of BOD_5 was sustained over the entire 3 weeks of operation for the field trial. It seems that the oxygen bleaching generates lesser amounts of degraded, low molecular weight, BOD_5 giving residues, such as acetic acid and methanol which readily pass through cellulose acetate membranes. Because of budgetary constraints, the molecular weight estimation of BOD_5 giving material in mill effluents was not attempted. The finding that BOD_5 in oxygen bleach effluents could be due to a higher proportion of large molecular weight carbohydrate residues might have practical importance outside the area of membrane processing. One could remove these materials by physicochemical methods in primary clarifiers in contrast to low molecular weight materials which conventionally require biological methods.

Continuing concern with the possibility of membrane fouling which could result from the presence of relatively insoluble Ca salts and especially the insoluble oxalates necessitated an analytical study of the daily composited

TABLE 26. ANALYTICAL DATA SUMMARY

Third Field Trial — Chesapeake

Sample no.	Sample*	Date	Mode of operation†	Specific gravity‡	pH	Total solids, g/l	COD, mg/l	Soluble calcium, mg/l	Sodium, mg/l	Inorganic chloride, mg/l	Total§ oxalate, mg/l	BOD_5, mg/l	Color units	Osmotic pressure, psi	Elec. res., ohms 21°C	35°C
7	Feed-1	4-15-76	Recycle	--	4.20	5.08	2,265	119	1090	1301	207	830	2,660	43	201	131
	Feed-2			1.0051	4.23	8.04	3,380	224	1744	1910	268	1096	4,494	63	134	87
	Perm			--	3.77	0.26	140	<1	66	58	10	197	<1	--	3362	2185
	Conc			1.0059	4.21	8.97	3,520	254	1968	2340	259	--	5,040	73	--	--
8	Feed-1	4-16-76	Recycle	--	6.73	5.36	2,766	45	1254	910	91	--	3,100	--	247	160
	Feed-2			1.0055	6.88	8.66	4,433	100	2084	1333	127	--	5,240	67	160	104
	Perm			--	6.06	0.30	220	<1	81	74	--	--	67	--	3100	2015
	Conc			1.0061	6.91	9.44	4,681	116	2228	1438	122	--	5,740	69	--	--
9	Feed-1	4-17-76	Recycle	--	6.32	5.51	2,837	60	1244	942	92	891	3,240	--	239	155
	Feed-2			1.0054	6.43	8.52	4,397	118	1968	1411	97	1281	5,160	62	164	107
	Perm			--	5.47	0.24	213	<1	66	66	--	86	0	--	4060	2639
	Conc			1.0075	6.52	9.65	5,000	130	2204	1784	130	--	6,000	66	--	--
10	Feed-1	4-19-76	Conc	--	6.87	10.69	5,342	164	2520	1946	--	--	7,040	75	115	75
	Feed-2			1.0080	6.98	12.38	6,120	194	2916	2377	--	--	8,320	89	111	72
	Perm			--	5.80	0.36	217	<1	112	132	--	--	35	--	3000	1950
	Conc			1.0092	6.94	14.42	7,269	225	3336	2823	--	--	8,580	93	--	--
11	Feed-1	4-20-76	Conc	--	6.93	15.33	7,779	245	3504	2886	125	2241	10,200	105	92	60
	Feed-2			1.0113	7.00	17.63	8,820	278	3944	3389	173	2754	11,500	123	78	51
	Perm			--	5.83	0.52	217	<1	162	191	0	88	0	--	1950	1268
	Conc			1.0122	6.99	19.09	10,202	303	4240	3463	192	--	12,900	126	--	--
12	Feed-1	4-21-76	Conc	1.0214	7.19	33.44	16,986	452	7432	5908	--	--	2,350	--	42	27
	Perm			--	6.57	1.36	295	3	430	1626	--	--	30	--	--	--
	Conc			1.0221	7.16	34.96	17,829	466	7580	6663	--	--	--	233	515	335
	Final conc			1.0254	7.15	40.83	20,737	651	8600	7597	--	--	--	272	--	--

(continued)

TABLE 26 (continued)

Sample no.	Sample*	Date	Mode of operation+	Specific gravity‡	pH	Total solids, g/l	COD, mg/l	Soluble calcium, mg/l	Sodium, mg/l	Inorganic chloride, mg/l	Total§ oxalate, mg/l	BOD_5, mg/l	Color units	Osmotic pressure, psi	Elec. res., ohms 21°C	35°C
14	Feed-1	4-23-76	Recycle	1.0044	6.85	6.31	2,660	76	1460	1,296	--	--	2,930	59	176	114
	Feed-2			1.0051	6.79	7.27	3,160	89	1675	1,364	--	--	3,430	--	155	101
	Perm			--	6.15	0.26	198	<1	76	74	--	--	8	--	2828	1838
	Conc			1.0054	6.45	7.71	3,460	98	1795	1,380	--	--	3,620	62	--	--
15	Feed-1	4-24-76	Recycle	1.0038	6.79	5.42	1,980	66	1240	997	55	854	2,510	56	206	134
	Feed-2			1.0050	6.87	7.08	2,960	100	1625	1,359	73	1152	3,400	--	158	103
	Perm			--	6.16	0.24	176	<1	76	79	3	56	0	--	3000	1950
	Conc			1.0053	6.61	7.64	3,380	105	1825	1,427	109	--	3,700	64	--	--
15A	Feed-1	4-25-76	Conc	1.0050	6.87	7.65	4,220	104	1825	1,448	121	768	3,770	--	168	109
	Feed-2			1.0078	6.91	12.04	5,500	175	2900	2,335	137	1710	5,960	88	98	104
	Perm			--	5.88	0.36	180	<1	118	134	0	60	8	--	1920	1248
	Conc			1.0088	6.74	13.04	6,060	196	3205	2,408	149	--	6,500	100	--	--
16	Feed-1	4-26-76	Conc	1.0061	6.88	9.33	4,640	122	2280	1,826	--	--	4,500	73	164	107
	Feed-2			1.0087	6.97	13.18	6,320	182	3180	2,513	--	--	6,720	101	142	92
	Perm			--	6.03	0.39	197	<1	129	155	--	--	11	--	1945	1264
	Vers. wash			--	--	7.52	--	162	--	--	--	--	--	--	--	--
17	Feed-1	4-27-76	Conc	1.0185	7.19	28.47	13,620	365	6680	55,719	436	--	15,000	200	53	34
	Perm			--	6.39	1.16	266	2	682	460	0	124	5	19	614	399
	Final perm			--	6.13	2.01	300	3	392	798	0	162	5	25	351	228
	Final conc			1.0254	7.03	40.02	19,440	470	9640	75,341	572	--	21,250	269	--	--

*Feed-1 = bleach sewer feed to system; Feed-2 = feed to modules from recycle tank.

+Recycle mode (internal recycle); concentrating mode (external recycle).

‡By pycnometer at 35°C.

§As sodium oxalate.

TABLE 27. LOADING AND REJECTION SUMMARY

Third RO Field Trial – Chesapeake

Date	Mode of operation*	Sample no.	Sample†	Total solids Pounds	Total solids Rejection‡ Based on Feed-1	Total solids Rejection‡ Based on Feed-2	COD Pounds	COD Rejection‡ Based on Feed-1	COD Rejection‡ Based on Feed-2	Soluble calcium Pounds	Soluble calcium Rejection‡ Based on Feed-1	Soluble calcium Rejection‡ Based on Feed-2	Sodium Pounds	Sodium Rejection‡ Based on Feed-1	Sodium Rejection‡ Based on Feed-2
4-15-76	Thru	7	Feed-1	73			32			1.7			15.6		
			Feed-2	396			167			11.0			86.0		
			Perm	1.8	.975	.995	1.0	.969	.994	0.007	.996	.999	0.47	.970	.995
			Conc	65			26			1.85			14.3		
4-16-76	Thru	8	Feed-1	270			140			2.27			63.3		
			Feed-2	1672			856			19.31			402.4		
			Perm	6.8	.975	.996	5.0	.964	.994	0.023	.990	.999	1.84	.971	.995
			Conc	254			126			3.12			59.9		
4-17-76	Thru	9	Feed-1	116			60			1.26			26.1		
			Feed-2	924			477			12.79			213.4		
			Perm	2.2	.981	.998	2.0	.967	.996	0.009	.993	.999	0.62	.976	.997
			Conc	103			53			1.39			23.5		
4-19-76	Conc	10	Feed-1	218			109			3.35			51.5		
			Feed-2	789			390			12.37			185.9		
			Perm	2.5	.988	.997	1.5	.986	.996	0.007	.998	.999	0.78	.985	.996
			Conc	196			99			3.06			45.4		
4-20-76	Conc	11	Feed-1	1014			514			16.20			231.7		
			Feed-2	3840			1921			60.54			858.9		
			Perm	8.8	.991	.998	3.7	.993	.998	0.017	.989	.999+	2.73	.988	.997
			Conc	927			495			14.71			205.9		
4-21-76	Conc	12	Feed-1	1701			864			22.99			378.0		
			Perm	12.3	.993	--	2.7	.997	--	0.027	.999	--	3.90	.990	--
			Conc	1462			745			19.48			316.9		
4-23-76	Thru	14	Feed-1	304			128			3.67			70.4		
			Feed-2	1147			498			14.03			264.2		
			Perm	4.0	.987	.996	3.1	.976	.994	0.015	.996	.999	1.18	.983	.996
			Conc	248			111			3.15			57.6		
4-24-76	Conc	15	Feed-1	157			57			1.90			35.8		
			Feed-2	779			326			11.00			178.7		
			Perm	2.1	.987	.997	1.6	.972	.995	0.009	.995	.999	0.68	.981	.996
			Conc	135			60			1.85			32.2		
4-25-76	Conc	15A	Feed-1	157			86			2.13			37.4		
			Feed-2	1367			626			19.87			329.3		
			Perm	2.9	.982	.998	1.4	.984	.998	0.008	.996	.999+	0.94	.975	.997
			Conc	163			76			2.45			40.1		
4-26-76	Conc	16	Feed-1	549			273			7.17			134.0		
			Feed-2	2328			1116			32.15			561.8		
			Perm	6.2	.989	.997	3.1	.989	.997	0.016	.998	.999+	2.04	.985	.996
4-27-76	Conc	17	Feed-1	1501			718			19.23			352.1		
			Perm	13.2	.991	--	3.0	.996	--	0.023	.999	--	7.77	.978	--

(continued)

TABLE 27 (continued)

				Inorganic chloride			Total oxalate			BODs			Color		
					Rejection‡			Rejection‡			Rejection‡			Rejection‡	
Date	Mode of operation*	Sample no.	Sample†	Pounds	Based on Feed-1	Based on Feed-2	Pounds	Based on Feed-1	Based on Feed-2	Pounds	Based on Feed-1	Based on Feed-2	Pounds	Based on Feed-1	Based on Feed-2
4-15-76	Thru	7	Feed-1	18.6			2.96			11.9			38		
			Feed-2	94.1			13.21			54.0			221		
			Perm	0.41	.978	.996	0.07	.976	.995	1.40	.882	.974	0.007	1.000	1.000
			Conc	17.0			1.88			--			37		
4-16-76	Thru	8	Feed-1	45.9			4.59			--			156		
			Feed-2	257.4			24.52			--			1012		
			Perm	1.68	.963	.993	--	--	--	--	--	--	1.519	.990	.998
			Conc	38.7			3.28			--			154		
4-17-76	Thru	9	Feed-1	19.8			1.93			18.7			68		
			Feed-2	153.0			10.51			138.9			559		
			Perm	0.62	.969	.996	--	--	--	0.81	.957	.994	0	1.000	1.000
			Conc	19.1			1.39			--			64		
4-19-76	Conc	10	Feed-1	39.7			--			--			144		
			Feed-2	151.6			--			--			531		
			Perm	0.92	.977	.994	--	--	--	--	--	--	0.245	.998	.999+
			Conc	38.4			--			--			117		
4-20-76	Conc	11	Feed-1	190.9			8.27			148.2			676		
			Feed-2	738.1			37.68			599.8			2506		
			Perm	3.22	.983	996	0.00	1.000	1.000	1.48	.990	.997	0.000	1.000	1.000
			Conc	168.2			9.32			--			626		
4-21-76	Conc	12	Feed-1	300.5			--			--			120		
			Perm	14.75	.951	--	--	--	--	--	--	--	0.272	.998	--
			Conc	278.6			--			--			--		
4-23-76	Thru	14	Feed-1	62.5			--			--			141		
			Feed-2	215.1			--			--			541		
			Perm	1.15	.982	.995	--	--	--	--	--	--	0.123	.999	.999+
			Conc	44.3			--			--			116		
4-24-76	Conc	15	Feed-1	28.8			1.59			24.7			73		
			Feed-2	149.5			8.03			126.7			374		
			Perm	0.70	.976	.995	0.03	.981	.996	0.50	.980	.996	0.000	1.000	1.000
			Conc	25.2			1.92			--			65		
4-25-76	Conc	15A	Feed-1	29.6			2.47			15.7			77		
			Feed-2	265.2			15.55			194.2			677		
			Perm	1.07	.964	.996	0.00	1.000	1.000	0.48	.969	.998	0.064	.999	.999+
			Conc	30.1			1.86			--			81		
4-26-76	Conc	16	Feed-1	107.4			--			--			265		
			Feed-2	443.9			--			--			1187		
			Perm	2.45	.977	.994	--	--	--	--	--	--	0.174	.999	.999+
4-27-76	Conc	17	Feed-1	2936.8			22.98			--			791		
			Perm	5.24	.998	--	0.00	1.000	--	1.41	--	--	0.057	.999+	--

*Thru = normal recycle of concentrate; conc = recycle of 100% concentrate back to feed supply.
†Feed-1 = feed to system; Feed-2 = feed to modules from recycle tank (Feed-2 is material treated by modules — high value due to recycle).
‡Rejection (ratio) = 1-(concentration of permeate/concentration of feed).

samples sent by air freight to the Institute. All F-1 samples (fresh bleach sewer feed to the RO system) were found to contain 50 to 100 mg/liter of soluble Ca and from 50 to 200 mg/liter of total oxalates. The partially recycled F-2 feed samples consistently showed more of these salts accumulating as concentration advanced ahead of the main bank of membrane modules. The permeate water product samples were substantially lower in both Ca and oxalates. However, analysis of the final concentrates taken from the membrane system showed little evidence of increased concentrations of either soluble Ca or total oxalates. Presumably these products were precipitating out somewhere along the line, either within the membrane system or after withdrawal and prior to analysis. The same picture had been apparent in the prior field trials at the Flambeau and Continental Group mills, but the fate and whereabouts of the insolubilized materials was not at all clear. A Versene (EDTA) wash on Run 17 recovered only a small fraction of the missing Ca. The method of analysis used for oxalates was not reliable on the Versene wash water. There was little evidence from electron microscopic study that these materials were accumulating on the surface of the membranes in quantities sufficient to cause fouling. As a precautionary measure, Versene washes were carried out frequently to avoid any possibility of irreversible fouling with consequent loss of the very limited supply of membrane equipment required to complete this project.

The indications again pointed to a probability that insoluble products were continuing to form as concentration advanced but that deposition on the membranes was being inhibited or prevented to a high degree by the velocity maintained across the membrane surfaces in the tubular UOP and ROP reverse osmosis modules. Such insoluble scale and fouling deposits are often observed in pulp and paper manufacturing systems and can be troublesome to control and costly to remove wherever accumulations develop. Accumulations are especially prevalent in areas of lessened turbulence. The lack of evidence for deposition of scale forming foulants on highly turbulent membrane surfaces was apparent throughout this project but that fortunate situation needs to be proved out with sustained operations over months and years. It seems quite likely that membrane systems will need to be engineered with areas of low turbulence specifically provided for ready removal by deposition of the relatively high levels of scale forming compounds present in recycled bleach liquor as concentration increases. The significance of these observations lies in the positive evidence for complete removal of these scale forming materials from permeate waters recovered for reuse in a bleach process water recycle system. The capabilities for accomplishing increased recycle of bleach process waters should be substantially advanced with incorporation of a tight RO membrane concentrating step for removing insolubles from the recycle system.

Fouling of membrane systems and significant losses in flux rates are of course not confined to formation of insoluble, scale forming materials. The observations reported in the preceding paragraphs provide substantial evidence that flux rate losses from fouling can be greatly reduced and substantially controlled with proper engineering design and particularly with maintaining high velocities across the membrane surface.

Another important cause for loss in flux rates is apparent in the substantial increase in osmotic pressure as concentration of bleach liquors with high levels of salts and other low molecular weight solubles (particularly NaCl) increases. The straight line, direct relationship of the bleach liquor solids concentration to the osmotic pressure of the Chesapeake oxygen bleach liquor effluents is presented graphically in Figure 21. Concentrating the bleach sewer feedstock by a factor of 10X increases the osmotic pressure from about 40 psi (276 kPa) to more than 300 psi (2.07 MPa). For this field trial the initial RO stage pressure of 600 psi (4.14 MPa) provided an effective working pressure of about 560 psi (3.86 MPa) when feeding a bleach process water with 5 g/liter total solids having an osmotic pressure of 40 psi (276 kPa). Concentration to 40 g/liter produced a product with 270 psi (1.86 MPa) osmotic pressure leaving just about 50% of the original effective working pressure. The substantial effect of the increased osmotic pressure in reducing flux rates as concentration increases is very apparent but the exact relationship between fouling and increased osmotic pressure as causes for reduction in flux rate was difficult to determine from the field data for this project. A special laboratory study could not be undertaken within budgetary limitations. However, increasing the working pressure within limitations of the available equipment [up to 700 psi (4.82 MPa) with the ROP modules and multistage centrifugal pump but with reduced flows and velocity] did increase the flux rates proportionately with the increasing pressure.

Special Test for Feed Thru Mode of Operation — Sustained Study

Sustained operation of the ROP modules at 10 to 20 gpm (38-76 l/min) flow rates required use of the Goulds multi-stage centrifugal pump on the small Chesapeake field test stand under less than optimum conditions for developing the data needed in this field trial. Operation of a by-pass with partial recycle of the concentrate was required for around-the-clock sustained studies. Half of the operating time during the three week run resulted from operation in that recycle mode. The degree of concentration achieved was roughly equivalent to operating a larger membrane processing unit having two or three stages of concentration. Such operation in the recycle mode was intended to approximate performance of the larger trailer mounted field unit used for the previous two field trials at the Flambeau and Continental Group mills.

The remaining half of the operating time was carried out in the concentrating mode with external recycle from the concentrate storage tank truck, after operation in the recycle mode had filled that tank with preconcentrate. The two runs in the concentrating mode were sufficient to provide 4% concentrate needed for freeze concentration tests at Avco and for elevated concentration studies with RO at the Institute.

A special 3 1/2-hour run in the feed-thru mode was made on the final day at Chesapeake to accumulate more data needed to confirm the results of short er term trials made at the Institute and Chesapeake before the main field trial began. Table 28 summarizes the operating data and shows a relatively high flux rate averaging 11.7 gfd under the test conditions at 600 to 620 psi (4.13-4.27 MPa) input pressure and at 35° to 38°C. Table 29 summarizes the analytical data showing a rather high solids concentration in the feed liquor at 7.62 g/liter. That feed was further concentrated to an average of 8.95 g/liter and with better than 97% solids rejection. A high quality permeate was produced with only 0.2 g/liter of solids and an inorganic Cl content of 68 mg/liter at 95% Cl rejection. More complete analysis was not attempted but conductivity meter readings further confirmed the high levels of rejection for other components (e.g., Na) in terms of a high resistance permeate water product from a low resistance feedstock.

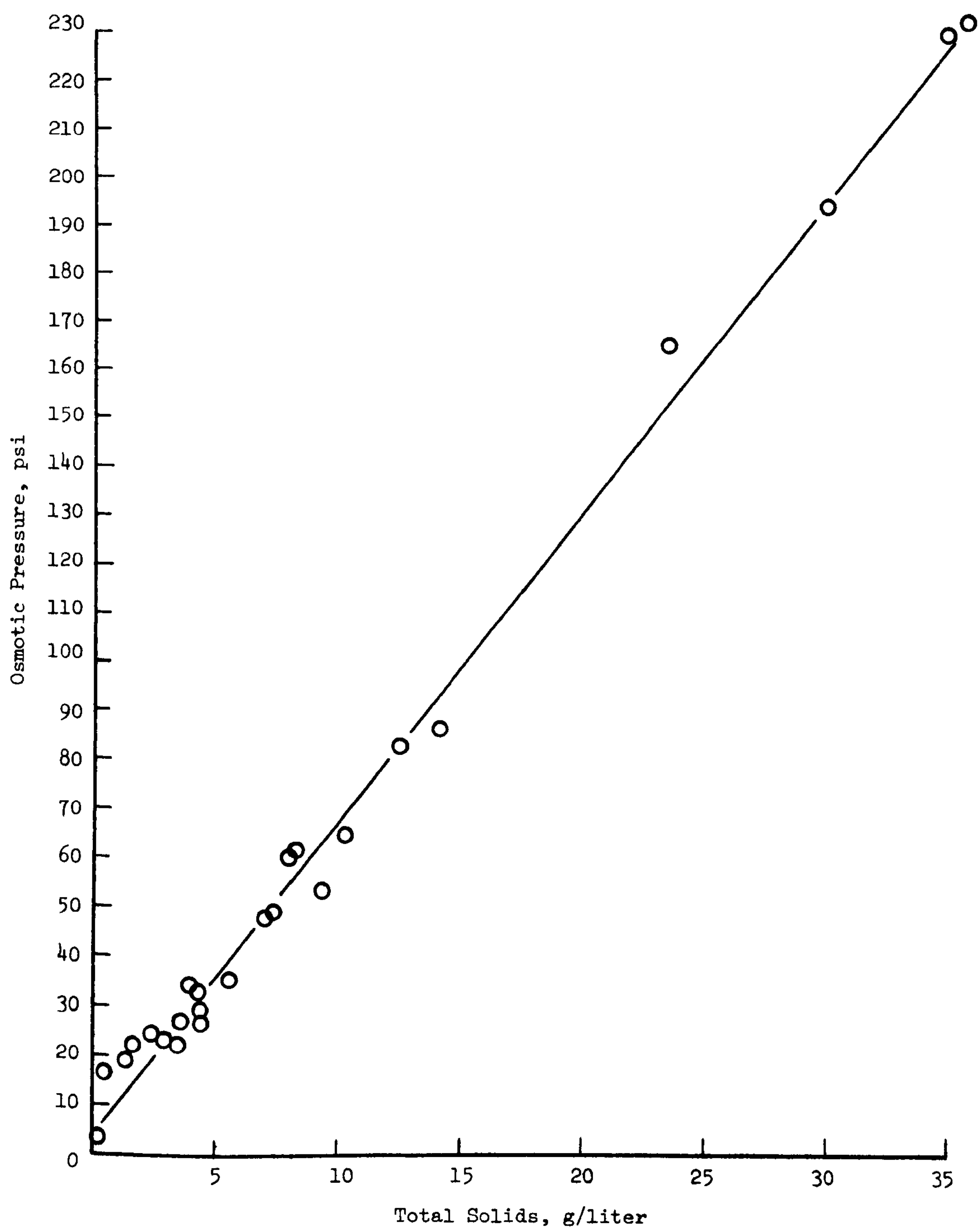

Figure 21. Osmotic pressure vs. total solids for Chesapeake effluent.

TABLE 28. CHESAPEAKE CORPORATION — RO FIELD TRIAL

Membrane Concentration of Oxygen Bleach Process Waters
309 ft^2 — membrane area
(Rev-O-Pak 105 ft^2 — UOP 204 ft^2)

Operating Data — Feed Thru Mode — No Recycle (4-27-76)

Time	Flow rate, gpm			Flux, gfd	Pressure, psi				Temp., °C
					Rev-O-Pak		UOP		
	Feed	Perm	Conc.		In	Out	In	Out	
12:00		2.75		12.82	620	615	615	580	35.6
12:45		2.42		11.28	600	595	595	545	36.0
13:45		2.50		11.65	600	595	595	550	38.0
14:45		2.36		11.00	--	--	--	--	--

TABLE 29. ANALYTICAL DATA
FEED THRU RO MODE — NO RECYCLE

3rd Field Trial — Chesapeake

Time	Sample	Total solids		Inorganic chloride		Elec. Res., ohms	
		g/liter	Rej. ratio*	mg/liter	Rej. ratio*	21°C	35°C
12:00	Feed	7.19		1348		152.	99
	Perm	0.19	0.974	60	0.955	3650	2372
	Conc	8.92		1779		128	83
12:45	Feed	7.72		1359		166	108
	Perm	0.21	0.973	68	0.950	3900	2535
	Conc	9.02		1805		129	84
13:45	Feed	7.87		1495		144	94
	Perm	0.21	0.973	74	0.950	3400	2210
	Conc	8.88		1789		139	90
14:45	Feed	7.68		1406		171	127
	Perm	0.20	0.974	68	0.952	3450	2242
	Conc	8.99		1794		143	93

*Rej. ratio = 1 - (concentration of permeate/concentration of feed).

Avco Laboratory Freeze Concentration Tests

The Chesapeake RO concentrate was concentrated by a factor of 10 from 1% to 10% total solids in the Avco Industrial Waste Laboratory. Although this was not as high a concentration as anticipated, these tests did show that the gravity wash columns could be applied to the process and thus eliminate the control problems that had been encountered with the pressurized columns used previously. It is difficult to demonstrate a concentration factor of greater than 10:1 in the laboratory test loop due to the intermittent nature of operation of the loop. A 10:1 concentration factor is not the limit of the process.

Operation of the equipment was quite smooth, with a minimum of foaming and no evidence of the formation of salt precipitates. Product water quality was quite good with total dissolved solids being below 400 ppm during most tests.

Discussion of FC Process

The feed for the freezing tests, which were run in the Avco laboratory, was preconcentrate from Institute's RO test runs at Chesapeake. Detailed analysis of this material was done by the Institute. It should be noted, however, that this solution was of lower concentration than anticipated. Because of the small membrane field test stand, it was not possible to produce 500 gallons (1.89 m^3) of 5% solids preconcentrate in the available time at Chesapeake.

Difficulties encountered with the pressurized wash columns, especially in second stage, led to the use of gravity wash columns. The gravity column permits precise regulation of the wash water and eliminates coupling of the first and second stages in the Concentrex process which is believed to be the primary source of the instability encountered in the FC mobile laboratory tests.

Freezing point data for the Chesapeake solution are shown in Figure 22. This solution had the highest freezing point depression, at a given concentration, of any of the bleach streams tested. This is probably due to a smaller quantity of organic material (which has less of an effect on the freezing point depression) in this solution than the others.

Analytical data for these tests are summarized in Table 30. Specific gravity is plotted in Figure 23. No salt precipitates were observed during the testing. The sulfate data correlate well with TDS indicating no precipitation of sulfates, which would be expected due to the low calcium content. This solution had less tendency to foam than the other solutions, though occasional additions of defoamer were required.

Operating conditions were similar to those required for the other solutions. Freezer temperature difference was 2-2.5°C and freezer specific capacity 60-90 lb/hr-ft^3 (0.96-1.44 t/hr-m^3). Wash column performance with the gravity columns was quite different than that of the pressurized columns. The total pressure difference between the top and bottom of the column was less than 3 psi (21 kPa) compared to 50-80 psi (344-552 kPa) for the pressurized column. Allowing for static head, this leaves less than 1 psi (6.9 kPa) for friction and restraining force compared to 20 psi (138 kPa) in the pressurized column. Flux rate through the columns was 100-500 lb/hr-ft^2 (1.6-8.0 t/hr-m^3) compared to the 2000 lb/hr-ft^2 (32.1 t/hr-m^3) in the pressurized column. These data were as anticipated and show the advantages of each type of column.

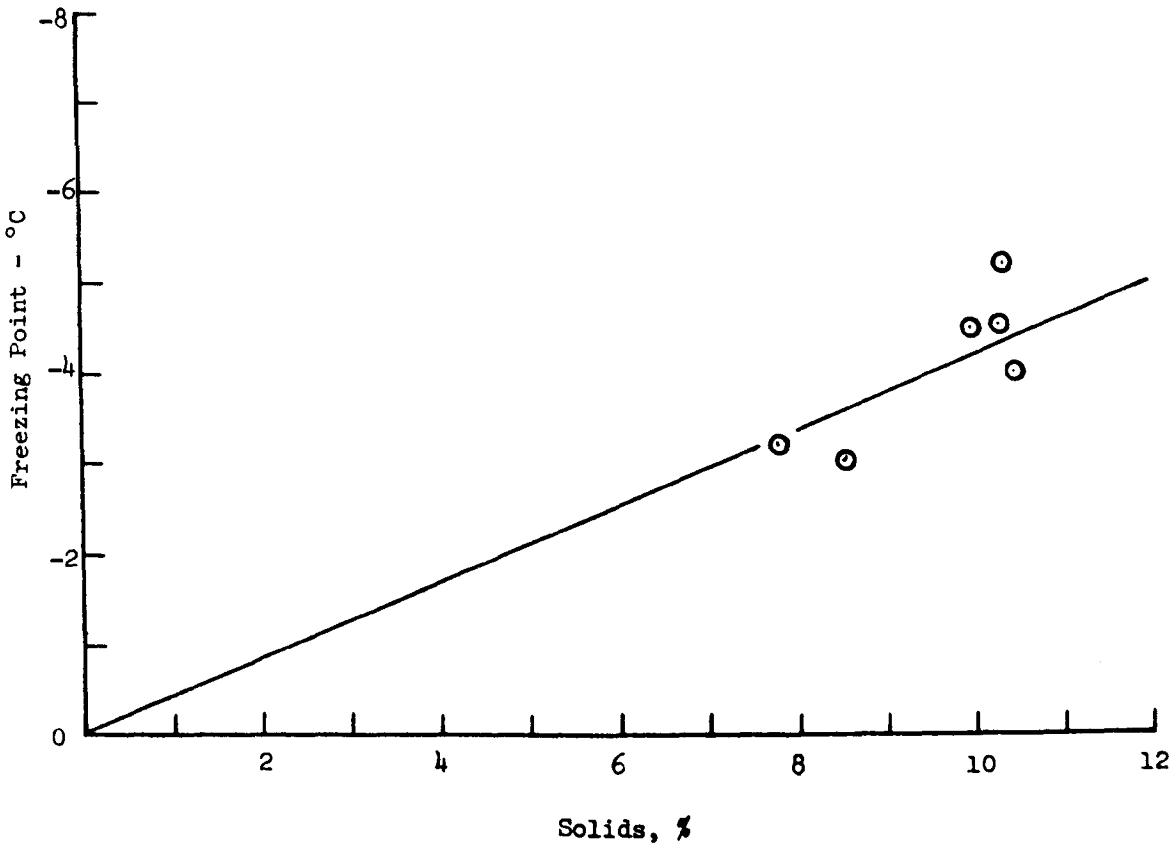

Figure 22. Freezing point correlation for Chesapeake effluent.

Overview of the Chesapeake Field Trial

Essential data for evaluating the capabilities of an RO membrane system for concentration of oxygen sequence bleach plant effluents were developed during this field trial at the Chesapeake mill. Some additional information was also gained in the freeze concentration tests on the RO preconcentrate at the Avco laboratory.

However, excessive problems arose in the planning and implementation of this test program. Because of damage to RO trailer during the second field trial and ultimate loss in membrane quality, the decision was taken to use a smaller scale RO unit of the Institute. The major part of the budget for this trial was spent on manpower for design, assembly, test operation and analytical control. The program also required modification and substantial readjustments to fit the unscheduled bleach plant shutdowns and consequent interruptions in flow and in quality of the supply of bleach feed liquor to the RO field unit. The Avco lab tests were confined to evaluating the gravity wash water column in a single stage unit and confirmed its capability to operate on a bleach liquor substrate to about 16% solids concentration but no further advancement could be made within the available funding for proving out the capability to attain and sustain continuous two-stage freeze concentration.

TABLE 30. AVCO ANALYTICAL DATA — CHESAPEAKE TESTS

Sample no.	Location	TDS, g/l	pH	Specific gravity, 25°C	Freezing point, °C	Conductivity, micro mhos/cm	SO_4, ppm
1	Brine I	38.86	7.02	1.025	-1.5		2200
2	Product	1.12	7.33	1.004		1000	
3	Brine II	85.58	7.50	1.053	-3		4200
4	Product	1.80	7.44	0.994		1050	
5	Brine I	24.14	8.08	1.017	-1		1200
6	Brine I	20.40	7.16	1.020	-0.8		800
7	Brine II	79.60	7.52	1.050	-3.2		3900
8	Brine II	103.00	7.73	1.071	-4.5		3800
9	Brine I	22.16	7.52	1.022	-0.8		700
10	Brine II	100.02	7.72	1.073	-4.5		3400
11	Product	0.18	7.60	0.994		360	
12	Product	0.42	7.50	0.996		445	
13	Brine I	20.92	7.98	1.026	-1		1625
14	Brine II	103.60	7.92	1.074	-5.2		4900
15	Product	--	7.59	0.994		220	
16	Product	0.12	7.35	--		67	
17	Brine I	25.78	7.94	1.028	-1		600
18	Brine II	105.34	8.01	1.080	-4		4500
19	Product	0.35	7.88	0.997		67	

Principal achievements in the Chesapeake field trial arose from the opportunity to prove the capabilities of operating an RO membrane system to process oxygen stage bleach effluent, especially at a mill using substantially less than the usual amounts [10,000 gal/ton (41.7 m^3/t)] of water, which contained relatively large amounts of scale forming Ca, oxalate and sulfate ions.

Data developed are summarized in Table 31. The feed liquors to the RO system averaged 5.5 grams total solids per liter for most of the sustained runs and reached 7.6 g/liter for the short term straight thru feed run but the mill sewer averages 4.5 to 5.0 g/liter when producing about 250 tons (227 t) bleached pulp per day. The RO unit concentrated its feed to about 7.95 g/liter in the straight thru and the internal recycle modes. For the full concentrating mode with external concentrate recycle the unit raised the concentration to 40 g/liter in two sustained runs. Flux rates ranged from 11.7 gfd (20 l/m^2-hr) for the feed thru mode and 8.77 gfd (15 l/m^2-hr) for the recycle mode down to less than 5 gfd (8.5 l/m^2-hr) in the concentrating mode at 40 g/liter solids. The high level of NaCl in the concentrate caused a rapid increase in osmotic pressure as the solids increased such that the effective working force across the membrane dropped from about 550 psi (3.79 MPa) with fresh feed at 5 g/liter solids to less than 230 psi (1.59 MPa) at 40 g/liter. Product water recovery as permeate was of exceptionally good quality for reuse in recycle systems and ranged from 40% recovery in the recycle mode to more than 95% in the full concentrating modes. Freeze concentration also produced high quality product water (less than 400 ppm solids indicated).

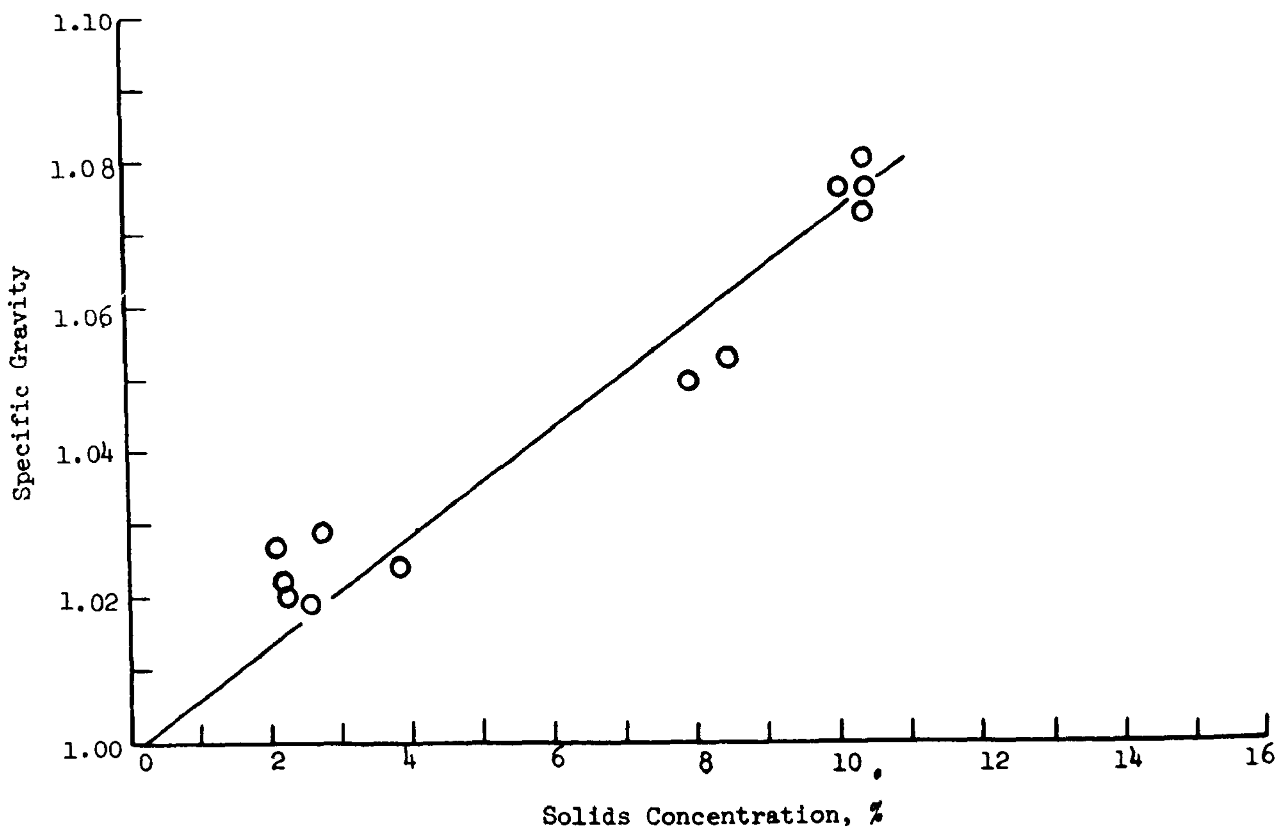

Figure 23. Specific gravity as a function of total solids for Chesapeake effluent.

Although the Chesapeake mill is now equipped with a highly efficient Unox biological secondary waste treatment system which meets the present and forseeable future waste treatment requirements (except possibly for color), this field test achieved its basic objectives of evaluating the possibilities for treating oxygen bleach sequence process waters. New bleach systems and particularly modified older mills using an O_2 bleach sequence could consider RO and possibly also FC concentrating systems for substantially increasing the degree of water recycle. Recovery of concentrates for regeneration of bleach chemicals may be possible with substantial overall cost reduction.

Specifically in the case of the Chesapeake mill, the advantages from adding RO and RC systems would arise in the areas of 1) reducing the discharge of chlorides; 2) reducing water usage; 3) removing or concentrating scale forming ions from the process waters; and 4) possibly in recovering bleach and pulping chemical residues for regeneration and reuse.

TABLE 31. SUMMATION OF PRINCIPAL OPERATING DATA FOR RO FIELD TRIAL CHESAPEAKE O_2 BLEACH EFFLUENT

	Average
Solids in bleach sewer feed to overall RO system (Feed-1), g/l (4 daily composited samples)	5.57 (A)
Solids in recycle mode feed to modules (Feed-2), g/l (4 daily composited samples)	8.12 (B)
Solids in recycle mode concentrate, g/l (4 daily composited samples)	8.94 (C)
Solids in feed thru mode — feed, g/l	7.62 (D)
Solids in feed thru mode — concentrate, g/l	8.95 (E)
Solids in final concentrate — concentrating mode, g/l	40.43 (F)
Degree of concentration in system	
Recycle mode — overall C/A	1.60
Recycle mode — single pass C/B	1.10
Feed thru mode — single pass E/D	1.17
Concentrating mode — full recycle F/A	7.25
Product water recovery (permeate flow/feed flow)	
Recycle mode, percent	40.8
Flux rates	
Feed thru mode, gfd	11.69
Recycle mode, gfd	8.77
Concentrating mode	
Run 10 — 10.69 to 14.42 g/l TS, gfd	8.60
Run 11 — 15.33 to 19.09 g/l TS, gfd	6.06
Run 12 — 33.44 to 40.83 g/l TS, gfd	3.52
Run 17 — 28.47 to 40.02 g/l TS, gfd	7.74
Osmotic pressure	
Bleach sewer feed, Runs 7 & 14 at 5.69 g/l TS, psi	51
Final concentrate, Runs 12 & 17 at 40.5 g/l TS, psi	271

SECTION 8

PROCESS ECONOMICS FOR REVERSE OSMOSIS AND FREEZE CONCENTRATION

OVERVIEW

The field demonstrations were designed to provide pilot scale operating experience and data which could then be used to estimate process economics. Data collected for the reverse osmosis trailer were analyzed and correlated for use in a computer program which developed capital and operating expense estimates (2). Data from the freeze concentration trailer were used in a similar manner by Avco to develop a tentative freeze concentration cost. Institute staff used the design correlation developed by Avco to compute the FC economics.

It became apparent that the cost of replacing the RO membrane would be a significant factor in the operating costs for the RO system. Major factor in the capital cost is the need for high pressure, stainless steel equipment.

The operating costs of the FC unit were significantly affected by two principal items: 1) power consumption; and 2) maintenance (labor, supplies and refrigerant). Refrigerant losses during operation and operating labor were not significant factors.

Total capital costs for treating current levels of bleach plant effluent [10,000 gal/ton (41.7 m^3/t)] range around $35,000 per daily ton of production ($38,600/t), with operating cost between $20 and $30 per ton of production ($22-33/ton). Reduction in bleach plant water usage to about 5000 gal/ton (20.9 m^3/t) reduces capital cost (for the RO plant only) to about $16,000 per daily ton ($17,600/t) and operating cost to around $15/ton ($17/t).

REVERSE OSMOSIS COST ESTIMATION

The computer program developed to estimate RO economics is relatively simple in concept. The program needs information on osmotic pressure vs. solids concentration, flux rate vs. solids concentration and minimum velocity vs. solids concentration. The basic design parameters of the system, such as the feed flow rate and pressure drop vs. velocity for the modules being considered, must also be specified. The program then, on the basis of inlet and desired final solids level, computes the amount of pumping horsepower and membrane area required to achieve the desired result at the selected operating pressure. Manufacturers cost data are then used to estimate the membrane costs. The total installed cost is computed by multiplying the membrane cost by a factor (Lang factor) (29). Operating costs are computed from the power consumption, estimated maintenance, and estimated membrane replacement costs. More refined economics, such as present value or depreciation schedules, are not computed as most mills have their own internal accounting systems. Thus, the costs are strictly out-of-pocket investments for equipment and direct operating charges.

Inputs to the Estimating Programs

The correlations on the physical characteristics of the bleach plant effluents (osmotic pressure and flux rate as a function of TDS) were obtained from the experimental data. The membrane suppliers recommended velocity ranges that they felt should be sufficient to prevent concentration polarization and fouling; IPC staff fitted simple curves to these data to obtain a continuous minimum velocity vs. concentration profile. Additionally the membrane suppliers were asked to estimate membrane cost ($/sq ft), membrane life, membrane replacement cost, and the Lang factor. Their estimates are given in Table 32.

TABLE 32. REVERSE OSMOSIS DESIGN FACTORS

	Membrane Supplier	
	UOP	ROP
Cost/sq ft	15.00	39.68
Membrane life	2 yr	2 yr
Lang factor	2.5	1.5
Module replacement cost (% of original module cost)	68.	
Minimum flow	3-3.5 gpm	

UOP = Universal Oil Products.
ROP = Rev-O-Pak.

Rather than attempt to run the program for each mill's flow, a standard size plant treating 500,000 gpd (79 m^3/hr) of the effluent was selected as a basis for testing the importance of various variables in the program. This allowed the various mills with different bleach sequences to be compared without confounding the comparison by large differences in flow rates.

Each mill was asked to estimate the effluent flow rates under moderate and tight bleach plant closure schemes. These flow rates were then used to scale the 500,000 gpd (79 m^3/hr) plant to the moderate and tight closure cases.

Capital and Operating Costs--

The results of the computer design runs are given in Table 33. The operating costs vary between the mills, but all are over $3.00/M gal ($0.79/m^3) of product water, or in excess of $2.75/M gal ($0.73/m^3) of feed effluent for the 90% water recovery utilized in the design. The table indicates that as the bleach systems are closed, the cost to treat the remaining effluent increases. This is due to the fact that the early stages of concentration require relatively few modules as the flux rates are high. The bulk of the modules and, thus, the cost, are utilized in removing the water at the higher concentration levels. Figure 24 plots the capital and operating costs for the idealized 500,000 gpd (79 m^3/hr) plants at each mill as the total solids change.

TABLE 33. DATA FOR EVALUATING CAPITAL COSTS AND OPERATING CHARGES FOR RO THREE LEVELS OF WATER USE IN BLEACHING

(Computerized Evaluation Based Upon a RO System Sized to Concentrate Dissolved Solids in Equivalent of 500,000 gal of Present Daily Flow)

	Current practice	Moderate closure	Tight closure
	Flambeau Mill		
Use of water, gal/ton	9,165	7,500	3,600
Flow, M gpd	500	409	196
Solids, mg/ℓ	4.95	6.05	12.6
Capital cost, $\bar{M}$ $	1.66	1.37	0.67
Operating cost, $/1000 gal product	3.31	3.43	4.10
	Continental Group Mill		
Use of water, gal/ton	10,000	8,000	5,000
Flow, M gpd	500	400	250
Solids, mg/ℓ	4.87	6.39	9.74
Capital cost, $\bar{M}$ $	1.59	1.30	0.884
Operating cost, $/1000 gal product	3.21	3.35	3.78
	Chesapeake Mill		
Use of water, gal/ton	6,920	5,220	4,000
Flow, M gpd	500	377	289
Solids, mg/ℓ	4.30	5.70	7.44
Capital cost, $\bar{M}$ $	1.45	1.13	0.874
Operating cost, $/1000 gal product	3.07	3.22	3.40

In Figure 24, the feed rate to the system remains constant as the concentration varies. The final total solids content of the concentrate remains fixed. Capital costs are relatively constant, but do show a slight rise as the feed concentration increases. Operating costs per 1000 gallons of feed pass through a rather flat maximum between 6 and 10% total solids. At low feed solids, a combination of module configuration and relatively high flux rates reduces operating cost. At high total solids, the relatively small amount of water that must be removed to reach the final solids level reduces

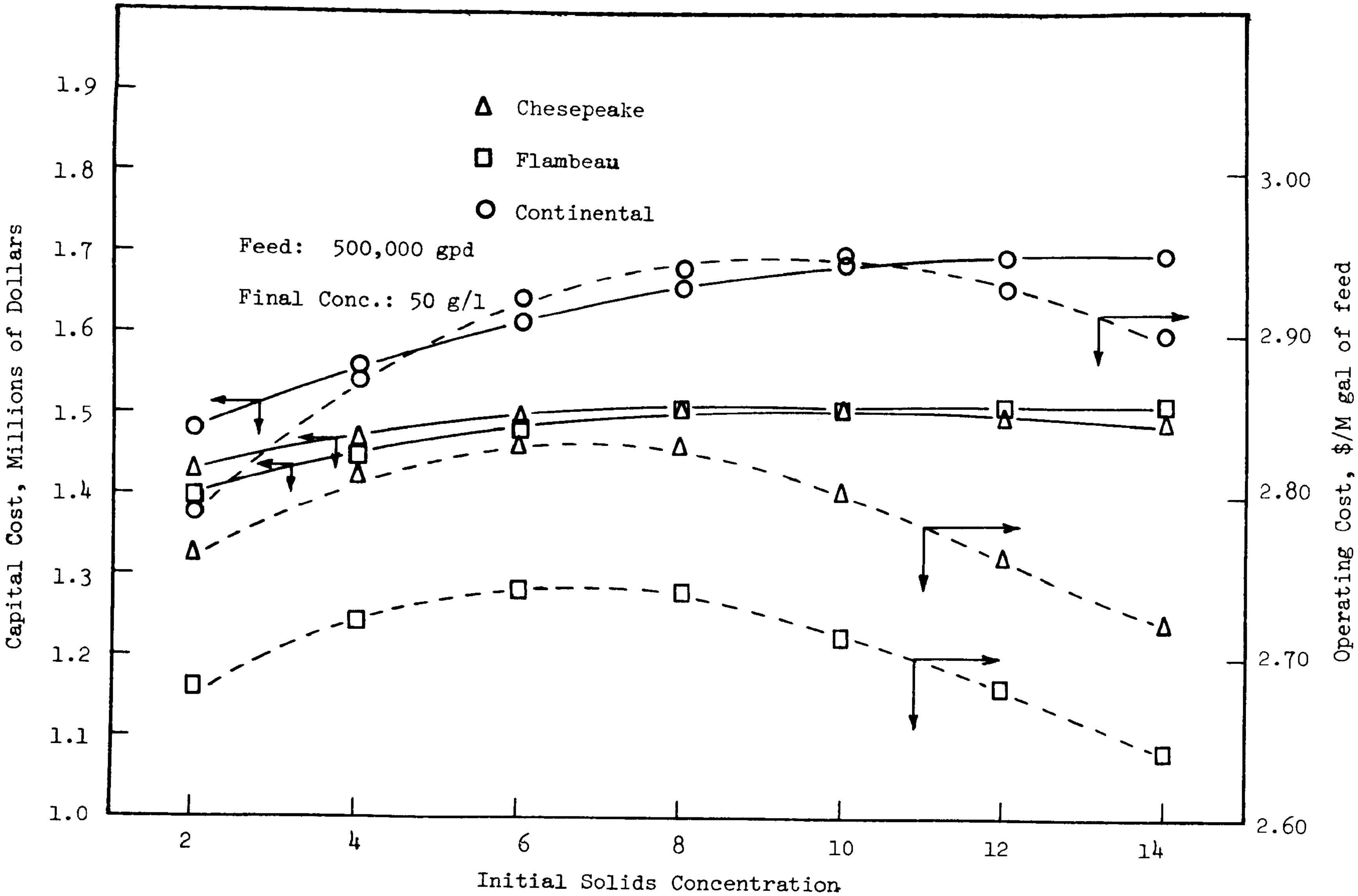

Figure 24. Capital and operating cost at various feed concentrations.

cost. It is in the midrange that lower flux rates combined with high water removal to drive up operating costs.

The cost of a reverse osmosis plant to treat each mill's entire bleach effluent can be scaled up directly from the 500,000 gpd (79 m^3/hr) plant used to generate cost comparison. The linear scale-up factor is a result of the membranes being the major cost item and at the 500,000 gpd (79 m^3/hr) plant size, the membranes are being purchased at the lowest possible price. That is, a 5 million gpd (789 m^3/hr) plant would look very similar to ten plants of 500,000 gpd (79 m^3/hr) each.

The cost data for treating the entire bleach effluents are given in Table 34. Under current operating conditions, the cost to generate a concentrate at 5% (50 g TDS/l) will cost between \$20 and \$30 per ton (\$22-33/t). Flow reduction within the mill can reduce these costs to \$11 to \$15 per ton (\$12-17/t). In all cases, it was assumed that no pretreatment was required.

Flow reduction will also have a significant effect on the capital cost. For example, under current practice, a RO plant at the Flambeau mill would cost \$3,650,000 to treat a volume of 1.0 $\overline{M}$ gpd (158 m^3/hr), while with tight closure, the flow drops to 0.43 $\overline{M}$ gpd (68 m^3/hr) and the capital costs drop to about \$1,480,000.

FREEZE CONCENTRATION COST ESTIMATION

Avco developed a correlation to compute the cost of a freeze concentration plant as a function of the feed rate. This correlation is

$$C = 200 + 70\left(\frac{F}{50}\right)^{0.4} + 345\left(\frac{F}{50}\right)^{0.8}$$

where

C = capital cost in thousands of dollars

F = feed rate, in thousands of gallons per day

The correlation is good for feed rates between 50,000 and 150,000 gpd (7.9-24 m^3/hr). Assuming 90% water removal by RO the FC units would range up to 800,000 gpd (126 m^3/hr). Thus, the FC units that would further concentrate the bleach liquors would be outside the limits of the correlation. Rather than extrapolate the correlation, the "six tenths" rule was used to estimate the costs for plants outside the range of the correlation. (An "eight tenths" scale-up rule could easily be justified as the third term in the Avco correlation will dominate the cost at large plant sizes.) The "six tenths" rule was used as it represents the average for many types of plants and because the first two terms in the correlation tend to reduce costs toward the six tenths rule from an "eight tenths" rule.

Operating costs were scaled up directly from Avco's sample calculations. Power consumption was computed from the formula:

$$P = \{13.9 + 2.55\,Y_1\,(6 + \Delta T_1) + 3.42\,Y_2\,(6 + \Delta T_2)\}\cdot\{0.021\,(T_c + 50)\}$$

where

P = power required kw-hr/1000 gallons of feed
Y_1 = fraction of feed water recovered in the first stage
Y_2 = fraction of feed water recovered in the second stage
ΔT_1 = freezing point depression in the first stage, °C
ΔT_2 = freezing point depression in the second stage, °C
T_c = cooling water temperature, °C

TABLE 34. CALCULATED CAPITAL COST AND OPERATING CHARGE FOR RO TREATMENT OF TOTAL BLEACH FLOWS

(Based Upon Computerized Values from Table 33)

	Current practice	Moderate closure	Tight closure
	Flambeau Mill		
Use of water, gal/ton pulp	9,165	7,500	3,600
Bleach plant flow, M gpd	1,100	900	432
Capital cost, $\overline{M}$ \$	3.650	3.015	1.475
Operating charge, \$/ton pulp	27.30	22.60	11.00
	Continental Group Mill		
Use of water, gal/ton pulp	10,000	8,000	5,000
Bleach plant flow, M gpd	8,000	6,400	4,000
Capital cost, $\overline{M}$ \$	25.450	20.800	13.5
Operating charge, \$/ton pulp	28.90	23.50	15.20
	Chesapeake Mill		
Use of water, gal/ton pulp	6,920	5,220	4,000
Bleach plant flow, M gpd	2,075	1,566	1,200
Capital cost, $\overline{M}$ \$	6.150	4.700	3.630
Operating charge, \$/ton pulp	19.55	14.90	11.56

Other operating costs are operating labor, maintenance labor and supplies, defoamer, and refrigerant losses. These charges, either as total charges per year, or as dollars per 1000 gallons of feed, were obtained from the Avco report. Table 35 lists the costs for an FC plant for each of the mills. These costs must be added to those of Table 34 to obtain the total treatment cost per ton of production. As less water is used in the bleach plant, FC capital costs will drop, although the operating costs will remain approximately constant.

TABLE 35. CAPITAL AND OPERATING COSTS OF FREEZE CONCENTRATION PLANTS

	Flambeau	Continental Group	Chesapeake
Feed rate, M gal/day	110	800	208
Feed solids, g/ℓ	18	11	10
First stage solids, g/ℓ	100	60	80
Second stage solids, g/ℓ	160	110	130
ΔT_1, °C	-4	-3	-3
ΔT_2, °C	-5.5	-5.5	-5.2
Capital cost, $M	994	3,110	1,382
Operating cost, $/M gal			
Power, 3¢/kw-hr	1.30	1.19	1.45
Refrigerant	0.057	0.057	0.057
Defoamer	0.095	0.095	0.095
Maintenance supplies	0.497	0.225	0.387
Total labor	0.672	0.672	0.672
Total operating cost			
$/M gal	2.62	2.24	2.66
$/ton	2.40	2.24	1.84

ENERGY CONSIDERATIONS

Reverse osmosis and freeze concentration are relatively energy efficient methods for separating a stream into two component streams. Such a separation can often be achieved by other means, such as electrodialysis or evaporation. Alternatively, the entire stream could be treated by conventional biological and physicochemical methods. Of course, not all streams are amenable to treatment by this range of options, but such a comparison is instructive as it illustrates the energy consumption of RO/FC relative to other possible mechanisms of treatment. Table 36 summarizes a variety of energy requirements for different treatment processes. RO/FC is more energy efficient than many methods which rely on phase separation to treat the stream. On the other hand, biological treatment is much less energy intensive than either RO or FC. However, one major reason for using RO and FC to concentrate the stream is the added advantage of color removal. The removal of BOD or suspended solids can be done by conventional techniques such as secondary bio-oxidation. Thus, the "cost" to remove color is the change in energy usage from bio-oxidation to treatment by reverse ormosis.

TABLE 36. ENERGY USAGE (KW-HR/1000 GAL) TO TREAT WASTE STREAMS

Treatment process	Cooling tower blowdown	Pulp & paper mill effluent	Bleach plant effluent
Primary clarification	1-2*	1-3	
Secondary bio-oxidation		3-10	
Unox		4†	
Zurn Attisholz		7‡	
Reverse osmosis			36-40+
Spent sulfite liquor		14-16§	
NSSC liquor		80#	
Electrodialysis	30*		
Freeze concentration			65-70+
Vapor compression	100*		
Multiple effect evaporators	580*		
Single effect evaporators	2650*		
Drum dryers	3400*		

*Ref. 30.

†Nicholls, W. Personal communication, NCR Corp., Combined Locks, WI.

‡Van Camp, B. Personal communication, Wisconsin Tissue, Menasha, WI.

§Ref. 2.

#Walraven, G. Personal communication, Green Bay Packaging, Green Bay, WI.

+This report.

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APPENDIX A

BRIEF LIST OF CONVERSION FACTOR

To convert from			To convert to	
Unit	Abbreviation	Multiply by	Unit	Abbreviation
inch	in	2.54	centimeter	cm
foot	ft	0.3048	meter	m
gallon (US)	gal	3.785	liter	l
gallon (US)	gal	3.785×10^{-3}	cubic meter	m^3
pound	lb	0.4536	kilogram	kg
ton (short)	ton	0.9072	metric ton	t
$\frac{\text{gallons}}{(\text{foot})^2\text{-day}}$	gfd	1.698	$\frac{\text{liters}}{(\text{meter})^2\text{-hour}}$	$\frac{l}{m^2\text{-hr}}$
$\frac{\text{pounds}}{(\text{inch})^2}$	psi	6894.7	Pascals	Pa
gallons/ton	gal/ton	4.173×10^{-3}	$(\text{meter})^3/\text{ton}$	m^3/t
gallons/day	gpd	1.577×10^{-4}	$(\text{meter})^3/\text{hour}$	m^3/hr
$\frac{\text{pounds}}{1000(\text{foot})^2}$[basis wt]	#	4.88	$\frac{\text{grams}}{(\text{meter})^2}$	g/m^2

NOTE. In common US engineering usage, M implies a multiplier of 1000, $\overline{M}$ is a multiplier of 1,000,000. In the SI-metric system, the following symbols are used for multipliers:

Multiplier	Name	Symbol
1,000,000	mega	M
1,000	kilo	k
100	hecto	h
10	deka	da
1	--	--
0.1	deci	d
0.01	centi	c
0.001	milli	m
0.000001	micro	μ

TABLE B-1. DAILY R.O. OPERATING LOG AT FLAMBEAU PAPER CO., PARK FALLS, WI

Date	Time/ operating hours	Energy used, kwh	Suction/discharge pressure, psig: Main pump	Pump A	Pump B	Pump C	Feed from main pump: Temp., °C	Sp.gr.	Flow, gpm	pH	Concentrate: Temp., °C	Sp.gr.	Flow, gpm	Trailer feed, gpm	Flux rate, gfd	Remarks
7/18/75	10:45/65															Start up
	11:45/66	98270	33/580	530/580	520/580	410/440	37	--	37.9	--	40	1.015	1.9	21.6	11.7	Batch operation, testing recycle
	12:15/66¼	98287	33/550	490/540	500/530	410/440	37	--	39.8	--	40	1.017	1.8	17.9	9.56	system, grab samples #12
	12:45/67															Shut down
7/22/75	09:00/67	98382														Start up, continuous operation
	10:05/69	98419	31/700	670/700	650/700	530/560	34	--	42.8	--	37	1.015	3.7	28.4	14.7	
	11:10/69	98453	32/470	450/490	460/480	360/390	34	--	40.9	--	37	1.015	1.45	14.9	7.96	Liquor supply cut off @ 09:00
	13:10/71	98511	32/520	490/530	490/530	390/410	38	--	41.3	--	43	1.018	1.04	13.7	7.54	Lowered pressures and de- creased concentrate flow to reduce feed flow and conserve liquor supply
	14:00/72															increased Pressures were increased to normal levels @ 14:00
	14:10/72	98543	32/680	630/670	650/680	530/550	37	--	39.8	--	41	1.023	2.12	17.7	9.27	
	16:05/74	98618	33/700	650/690	670/700	550/580	37	--	39.8	--	40	1.021	2.26	17.2	8.85	
	18:05/76⅔	68673	33/690	650/680	670/700	530/550	37	--	39.4	--	40	1.018	2.38	16.2	8.20	
	19:40/77	98727	33/700	660/700	670/700	540/570	38	--	40.3	--	41	1.017	2.33	15.8	8.02	
	21:10/79	98778	33/690	650/690	670/700	530/550	38	--	40.3	--	40	1.017	2.34	15.2	7.66	
	23:00/81	98841	33/700	660/700	670/700	530/550	38	--	40.3	--	39	1.016	2.40	14.8	7.36	
7/23/75	01:00/83	98919	33/700	700/720	680/720	540/560	38	--	40.3	--	39	1.015	2.40	14.6	7.24	Bleachers shut down 01:10
	02:00/84	98942	36/650	610/650	620/650	460/480	37	--	28.7	--	38	1.018	1.75	10.7	5.88	Liquor supply interrupted 3:30
	03:00/85	98968	35/650	720/730	670/700	530/550	37	--	30.1	--	38	1.017	2.06	10.9	5.23	Reduced main pump speed to
	04:00/86	98994	37/560	550/580	480/500	480/500	36	--	23.3	--	37	1.017	1.43	9.9	5.06	decrease feed flow rate and
	05:00/87	99015	37/620	680/700	490/510	500/530	35	--	23.3	--	36	1.018	1.25	10.6	5.52	conserve supply left in
	06:00/88	99040	37/530	650/670	560/570	550/600	35	--	22.3	--	36	1.020	1.25	10.2	5.29	storage tank trailer
	07:00/89	99063	37/540	660/680	570/580	570/600	34	--	22.3	--	36	1.019	1.24	10.0	5.23	
	08:00/90															Shut down, bleach plant did not begin operation until 18:00. Liquor
	20:00/90															supply restored 20:00
	21:30/90¼	99148	33/700	650/700	650/690	520/545	33	--	39.4	6.8	36	1.019	3.12	21.4	10.9	Start up, automatic Samples started 21:00
	23:00/93	99202	33/700	640/690	640/680	525/550	37	--	40.3	7.1	37	1.012	3.05	19.4	9.60	(during shutdown a mild BIZ
7/24/75	01:00/95	99272	33/700	670/730	690/730	560/580	34	--	39.8	7.2	37	1.014	2.72	18.1	9.15	wash was performed, however,
	03:00/97	99342	33/700	630/700	680/710	540/570	39	--	39.8	7.0	40	1.010	2.75	17.6	6.79	the raw water supply was off
	05:00/99	99412	33/700	630/700	650/700	510/530	37	--	40.9	7.0	39	1.013	2.40	19.0?	9.86?	from 17:00-20:00, so the system could not be given a good fresh
	07:00/101	99483					38	--	40.3	6.7	39	1.014	2.30	14.6	7.31	water flush)
	09:00/103	99554	33/730	670/730	690/730	540/570	38	--	40.3	6.5	40	1.014	2.06	13.6	6.83	Composite samples #15 collected
	11:00/105	99625	33/720	660/720	680/720	550/580	40	--	40.3	6.4	41	1.014	1.95	13.1	6.59	08:00, may be contaminated with BIZ
	11:10/105															Shut down, system was subjected to preliminary BIZ wash followed by Versene wash and fresh water flush
	13:45/105															
	14:00/105															Start up
													3.5	35.0	17.8	High flux rate, membranes
	15:00/106	99684	33/750	710/750	710/750	560/590	34	--	38.9	6.6	37	1.016	3.05	25.4	13.2	regenerated
	16:00/107	99719	33/740	700/740	700/730	530/560	34	--	40.3	6.7	37	1.019	2.05	20.6	11.0	
	17:00/108	99758	33/690	645/690	650/690	525/550	35	--	39.4	6.8	37	1.021	1.89	17.4	9.21	
	19:00/110	99827	33/700	650/700	660/710	530/560	37	--	39.4	6.8	39	1.021	2.06	16.5	8.55	
	21:00/112	99893	33/700	650/700	660/700	530/580	37	--	39.4	6.8	39	1.020	2.07	14.9	7.60	
	23:00/114	99963	33/700	655/700	660/700	545/575	37	--	40.3	6.8	39	1.018	2.26	14.4	7.19	

(continued)

TABLE B-1 (continued)

Date	Time/ operating hours	Energy used, kwh	Suction/discharge pressure, psig: Main pump	Pump A	Pump B	Pump C	Feed from main pump: Temp., °C	Sp.gr.	Flow, gpm	pH	Concentrate: Temp., °C	Sp.gr.	Flow, gpm	Trailer feed, gpm	Flux rate, gfd	Remarks
7/25/75	01:00/116	00013	33/700	650/700	650/700	525/550	37	--	41.3	6.8	38	1.019	2.13	12.9	6.42	
	03:00/118	00102	33/700	650/700	640/680	550/580	37	--	40.9	6.8	38	1.017	2.19	12.5	6.12	
	05:00/120	00173	33/700	660/700	650/690	550/590	37	--	40.3	6.7	39	1.016	2.21	12.2	5.94	
	07:00/122	00243	33/720	670/710	660/700	560/600	37	--	40.9	6.6	38	1.017	2.22	11.9	5.76	Attempted pressure pulse cleaning. Note: After pressure pulse, concentrate was sewered. Thus feed to R.O. unit was less concentrated
	08:15/123															
	09:00/124	00310	33/730	680/720	660/700	570/600	36	--	39.9	6.5	37	1.010	1.97	12.9	6.47	Composite samples #16 collected
	09:30/124½															Shutdown; BIZ-Versene wash
	11:00/124½															Start up
	11:30/125	00342	33/750	710/750	720/750	570/600	33	--	38.4	6.6	38	1.009	2.95	31.4	16.9	
	12:30/126	00377	33/750	710/750	700/740	550/580	34	--	38.9	6.7	38	1.017	2.94	23.9	12.5	
	14:30/128	00447	33/720	670/720	680/720	530/570	36	--	39.9	6.7	39	1.019	2.83	19.9	10.2	
	15:30/129															Grab samples #17 collect
	17:00/130½	00534	33/700	660/700	660/700	520/545	36	--	40.3	6.8	38	1.018	3.16	17.9	8.73	
	19:00/132½	00603	33/700	665/710	660/710	540/570	37	--	40.3	6.8	39	1.016	3.23	17.3	8.37	
	21:00/134½	00675	33/700	655/700	655/700	570/600	37	--	40.3	6.8	39	1.015	2.56	15.6	7.72	
	22:00/135½															Shutdown for 1 hr, BIZ wash
	23:00/135½															Start up, automatic samplers
7/26/75	01:00/137½	00783	34/720	670/710	680/720	600/630	37	--	36.0	6.8	38	1.018	1.01	15.5	8.61	Continued to operate during this wash period
	03:00/139½	00846	34/700	670/700	650/700	570/600	38	--	35.5	6.6	39	1.021	2.06	14.6	7.13	
	05:00/141½	00911	34/730	690/720	650/700	570/600	37	--	35.5	6.5	39	1.021	2.16	13.5	6.71	
	07:00/143½	00975	34/720	680/710	650/700	550/580	37	--	35.5	6.6	37	1.020	2.16	12.6	6.18	
	08:00/144½	01009	34/730	690/720	650/690	550/580	36	--	35.0	6.6	38	1.019	1.97	12.2	6.06	Composite samples #17 collected
	08:30/145															Shutdown; BIZ-Versene wash
	10:00/145															Start up
	11:00/146	01057	33/700	650/700	670/710	540/570	33	--	39.4	6.7	35	1.013	3.00	25.1	13.1	
	12:00/147	01091	33/720	660/710	680/720	540/580	33	--	39.4	6.8	35	1.019	1.96	20.4	10.9	
	14:00/149	01160	33/740	670/730	680/730	570/600	36	--	39.4	6.7	38	1.022	1.90	17.3	9.15	
	16:00/151	01228	33/750	690/740	690/740	560/590	37	--	39.4	6.6	40	1.023	2.02	16.1	8.38	Automatic samplers shut off
	16:30/--		33/750	700/750	710/750	550/580	32	--	38.9	6.8	40	1.011	1.92	38.5	11.5	Beginning shortly after 16:00 all concentrate was sewered After ½ hr, because of the greatly decreased concentration of feed to the trailer, the flux rate increased significantly
							↑ These temperatures were taken from trailer gages; they are not accurate (see below)									
	16:30															Shutdown, overnight BIZ soak
7/27/75	10:30															Flushed system, washed with Versene
	11:30/151						Temperatures from trailer gage ↓ / Temperatures recorded by thermometer ↓									Start up, composite samples #17 were collected, but were apparently contaiminated during BIZ wash
	11:30/151						30	1.002			30	1.003				
	13:00/152	01301	32/710	670/720	660/710	550/560	34 35	1.005	37.9	6.6	39	1.016	3.30	26.9	14.0	All concentrate was recycled for the 1st hr, then the concentration of feed to the 1st banks (recycled feed) was held nearly constant during the remainder of the run
	14:00/153	01333	33/740	700/750	700/750	550/580	34 36	1.004	35.0	6.7	39	1.017	2.93	25.0	13.1	
	15:00/154	01367	33/740	700/750	700/740	540/570	35 35	1.005	35.5	6.8	39	1.016	3.42	23.5	11.9	
	16:00/155	01400	33/750	710/760	710/750	560/590	36 36	1.0045	35.5	6.9	39	1.014	3.69	22.3	11.0	
	17:15/156¼	01442	33/730	690/740	680/730	520/550	37 37	1.0055	34.5	6.9	39	1.013	3.10	19.2	9.56	

(continued)

TABLE B-1 (continued)

Date	Time/ operating hours	Energy used, kwh	Suction/discharge pressure, psig: Main pump	Pump A	Pump B	Pump C	Feed from main pump: Temp., °C	Sp.gr.	Flow, gpm	pH	Concentrate: Temp., °C	Sp.gr.	Flow, gpm	Trailer feed, gpm	Flux rate, gfd	Remarks
7/27/75	19:00/158	01508	34/730	680/730	670/720	540/570	37 36	1.007	32.1	6.9	40	1.012	3.25	16.8	8.02	
	21:00/160	01561	35/720	690/740	680/730	570/600	37 38	1.0055	30.1	6.9	40	1.012	3.49	17.2	8.14	(Rate of flux loss de-
	22:30/161½	01605	35/720	690/740	680/730	560/580	37 38	1.004	29.2	6.9	39	1.011	4.37	17.5	7.78	creases, or flux rate
	24:00/163	01656	35/720	710/750	690/740	570/600	37 38	1.004	29.2	6.9	40	1.010	3.84	16.7	7.66	even increases, as feed
7/28/75	02:00/165	01708	36/710	660/705	660/705	565/690	37 39	1.0045	26.2	6.7	39	1.010	3.13	14.4	6.71	concentration drops)
	03:30/166½	01775	36/710	660/710	660/700	560/585	37 39	1.005	26.2	6.8	39	1.010	3.17	13.9	6.36	
	05:00/168	01797	36/720	665/715	665/710	565/590	37 38	1.004	26.2	6.8	38	1.009	3.42	13.9	6.24	
	07:00/170	01859	36/710	650/700	650/700	540/570	37 38	1.0045	26.2	6.8	39	1.008	3.50	13.1	5.67	Composite samples #19 @ 08:00
	08:00/171															Shutdown; BIZ-Versene washup
	09:30/171															Start up
	11:00/172½	01933	33/740	690/730	690/740	560/590	35 37	1.0055	38.9	6.8	39	1.016	3.02	25.0	13.1	
	12:00/173½	01966	34/730	680/720	690/730	550/580	36 37	1.0055	35.5	6.8	40	1.017	3.40	23.0	11.6	
	13:30/175	02014	35/700	670/710	660/700	550/580	36 38	1.005	31.6	6.8	41	1.017	3.30	20.6	10.3	
	14:30/176	02045	35/740	710/750	700/740	570/600	37 37	1.005	30.6	6.8	41	1.017	3.29	20.5	10.2	
	15:30/177	02077	35/740	720/750	700/740	570/600	38	1.0045	30.1	6.8	41	1.016	3.34	19.7	9.74	
	16:30/178	02118	35/720	700/730	680/720	540/570	38	1.0045	30.1	6.8	41	1.014	3.62	18.9	9.09	
	17:00/178½	02134	35/700	650/700	640/680	500/530	38	1.003	31.1	6.7	41	1.012	3.90	18.2	8.49	
	19:00/180½	02183	35/720	690/730	680/730	540/560	37	1.006	30.6	6.8	40	1.014	2.50	16.0	8.02	
	21:00/182½	02246	35/730	700/750	680/730	540/560	37	1.0055	30.6	6.9	40	1.013	3.58	16.1	7.43	
	23:00/184½	02308	35/700	670/720	680/720	520/550	30	1.0045	31.1	6.8	39	1.010	4.30	17.1	7.60	
7/29/75	01:00/186½	02360	35/680	660/700	660/710	555/580	36	1.0045	30.6	6.8	38	1.006	4.55	16.8	7.31	
	03:00/188½	02433	35/710	660/710	660/710	560/580	37	1.0045	30.6	6.8	37	1.0075	4.56	16.4	7.01	
	05:00/190½	02495	35/710	660/710	660/720	560/590	37	1.004	30.1	6.8	38	1.007	4.48	16.0	6.83	
	07:00/192½	02556	35/710	660/710	660/710	550/575	37	1.004	30.6	6.9	37	1.006	4.44	15.0	6.30	Composite samples #20 taken 08:00
	08:00/193½															Shutdown; Versene washup
	09:30/193½															Start up, flux rate = 17.1 gfd @ 10:00
	11:00/195	02642	33/750	710/750	710/750	570/600	37	1.004	38.4	6.8	40	1.015	3.54	26.3	13.5	Bleach plant shutdown from
	12:00/196	02673	33/730	700/740	700/740	550/580	37	1.0045	34.0	6.6	40	1.015	3.29	23.3	12.0	10:00-12:15; feed liquor
	14:10/198	02740	34/720	680/720	680/710	540/570	38	1.0045	31.6	7.0	40	1.013	3.59	20.0	9.74	flow through saveall increased
	16:00/200	02808	35/710	670/710	670/700	540/570	39	1.004	31.1	6.9	40	1.011	3.84	18.5	8.73	to catch up; the resulting
	17:00/201	02828	35/720	690/730	690/730	560/590	39	1.004	31.1	6.9	41	1.011	3.07	17.4	8.49	feed contained higher amounts of suspended solids
	19:00/203	02888	35/700	660/710	670/700	510/530	39	1.004	31.1	6.8	41	1.010	3.43	15.9	7.43	Grab samples #21 collected
	21:00/205	02949	35/710	680/720	680/720	530/550	40	1.004	31.1	6.8	42	1.010	3.52	15.6	7.19	at 15:00
	23:00/207	03010	35/710	670/710	660/700	490/520	40	1.004	31.6	6.7	40	1.008	4.00	14.7	6.36	Bleach plant shutdown, 22:00
7/30/75	01:00/209	03069	35/700	680/730	650/700	540/570	39	1.005	26.2	6.6	37	1.010	3.05	13.5	6.18	Feed liquor cut off from
	03:00/211	03127	35/720	680/715	650/700	490/525	39	1.0045	30.1	6.6	37	1.009	4.46	12.8	4.94	23:45 to 01:45
	05:00/213	03180	35/715	655/705	650/700	540/565	39	1.004	30.1	6.8	39	1.007	4.62	14.4	5.82	
	07:00/215	03253	35/720	655/710	660/710	540/570	38	1.004	30.6	6.9	37	1.0065	4.45	13.8	5.55	Composite samples #21, 08:00
	08:00/216															Shutdown, BIZ washup only
	12:00/216															Start up (delayed because
	13:30/217½		35/690	650/700	650/700	540/570	39	1.004	32.1	6.8	40	1.009	1.19	17.2	8.91	repiping), flux rate 11.9
	15:00/219	03410	35/720	680/730	680/730	550/580	39	1.0055	32.1	6.8	41	1.011	2.79	15.9	7.78	gfd at 12:30
	16:00/220	03440	35/730	680/730	690/730	560/590	40	1.0045	30.1	6.8	42	1.010	3.22	16.1	7.66	
	17:00/221	03472	37/730	690/730	690/740	550/575	36	1.008	30.6	6.7	40	1.013	1.50	12.9	6.77	No longer is an attempt being
	19:00/223	03534	37/750	700/750	710/760	480/500	36	1.009	31.1	6.7	38	1.018	1.85	10.5	5.11	made to hold feed to 1st module
	21:00/225	03598	37/750	720/760	700/750	540/560	37	1.009	30.1	6.6	39	1.015	1.77	11.0	5.46	bank at a constant conconcentration
	23:00/227	03663	36/750	710/750	700/750	480/500	37	1.010	34.0	6.6	39	1.015	1.71	10.5	5.20	Concentrate is being pump to 2nd storage tank for Avco

(continued)

TABLE B-1 (continued)

Date	Time/ operating hours	Energy used, kwh	Suction/discharge pressure, psig Main pump	Pump A	Pump B	Pump C	Feed from main pump Temp., °C	Sp.gr.	Flow, gpm	pH	Concentrate Temp., °C	Sp.gr.	Flow, gpm	Trailer feed, gpm	Flux rate, gfd	Remarks
7/31/75	01:00/229	03728	36/710	660/700	650/700	535/565	37	1.012	33.0	6.6	38	1.015	1.51	9.4	4.66	
	03:00/231	03792	36/725	690/735	660/720	560/590	40	1.011	32.6	6.6	39	1.016	1.54	9.7	4.86	
	05:00/233	03859	36/720	670/710	650/700	550/570	40	1.012	32.6	6.6	40	1.016	1.50	9.2	4.54	
	07:00/235	03923	36/730	690/730	670/725	550/580	36	1.013	32.6	6.7	37	1.016	1.48	8.4	4.11	Composite samples #22 @ 08:00
	07:45/235 3/4															Shutdown; BIZ washup only
	10:45/235 3/4															Start up, flux rate was 10.7 gfd at 11:15 (½ hr after start up)
	12:00/237	04034	36/760	730/760	730/760	580/610	39	1.006	33.5	6.8	40	1.011	2.06	17.6	9.21	
	14:00/239	04096	36/700	660/700	660/700	540/570	39	1.008	30.6	6.8	41	1.013	2.06	13.2	6.59	
	16:00/241	04168	36/720	680/720	680/710	550/580	36	1.0085	30.6	6.8	38	1.013	1.86	11.4	5.64	Grab samples #23 collected
	17:00/242	04180	36/730	690/740	690/730	550/580	36	1.0085	31.1	6.8	38	1.013	1.87	11.3	5.58	at 15:00
	19:00/244	04249	37/730	700/750	700/750	560/590	37	1.008	31.1	6.7	39	1.012	1.71	10.7	5.35	
	21:00/246	04313	37/740	710/750	710/750	560/590	38	1.0095	30.1	6.7	40	1.013	1.68	10.2	5.08	
8/01/75	01:00/250	04438	37/710	660/710	660/710	525/550	38	1.010	30.6	6.7	39	1.013	1.71	9.5	4.63	
	03:00/252	04505	36/710	660/720	670/725	530/560	37	1.010	30.6	6.7	38	1.013	1.71	9.6	4.67	
	05:00/254	04565	36/710	665/710	670/720	540/570	36	1.010	30.6	6.7	37	1.013	1.71	9.3	4.53	
	07:00/256	04626	36/700	650/700	650/700	510/535	39	1.008	30.1	6.7	41	1.011	1.65	9.7	4.78	
	08:00/257						38	1.008	30.1	6.8	38	1.011	1.72	9.0	4.32	Composite samples #23
	08:00/257															Shutdown; system given 3 hr BIZ wash followed by 3 hr Versene wash

TABLE B-2. ANALYTICAL DATA

Preliminary Intermittent Operation

Sample	Date	Time	Sp. gr.	Temp., °C	pH	Total solids g/l	Total solids Rej. ratio*	Total carbon, mg/l	COD, mg/l	Soluble calcium mg/l	Soluble calcium Rej. ratio*	Sodium mg/l	Sodium Rej. ratio*	Inorganic chloride mg/l	Inorganic chloride Rej. ratio*	Soluble oxalate† mg/l	Soluble oxalate† Rej. ratio*	BOD_5 mg/l	BOD_5 Rej. ratio*	Color mg/l	Color Rej. ratio*	Susp. solids, mg/l
1 Feed	6/20/75	10:15 AM	.998	31.0	-	3.92		-	-	-	-	-	-	-	-	16.0		-				
1 Perm	"	"	.995	32.0	-	0.77	.80	-	-	-	-	-	-	-	-	0.3	.98	-				
1 Conc	"	"	1.006	30.0	-	14.87		-	-	-	-	-	-	-	-	21.0		-				
2 Feed	6/23/75	11:30 AM	1.000	29.0	6.62	4.57			1237	1500		2.7		1957		15.4		153				
2 Perm	"	"	.997	28.2	5.81	0.53	.88	74	-	170	.89	T	.93	251	.87	0.4	.97	140	.08			
2 Conc	"	"	1.003	29.0	6.79	17.30		-	3522	5600		6.6		6946		81.3		-				
3 Feed	6/24/75	9:00 AM	.999	30.5	6.55	4.48		-	1184	1320		6.4		1495		17.0		132				
3 Perm	"	"	.996	27.8	5.81	0.40	.91	47	-	120	.91	1.3	.80	184	.88	0.3	.98	105	.20			
3 Conc	"	"	1.007	30.1	6.65	15.40		-	3417	4330		23.5		6044		26.2		-				
4 Feed	7/1/75	9:10 AM	.999	29.0	6.20	4.26		-	866	1292		3.0		1716		21.0		156				
4 Perm	"	11:40 AM	.996	30.0	5.60	0.51	.88	54	-	130	.90	1.0	.67	235	.86	0.8	.96	96	.38			
4 Conc	"	12:05 PM	1.012	30.0	6.41	24.09		-	4051	7030		14.2		8271		55.4		-				
5 Feed	7/2/75	11:00 AM	.999	28.0	6.81	4.71		-	973	1216		2.7		1931		21.7		165				
5 Perm	"	"	.995	28.0	6.40	0.71	.85	60	-	160	.87	1.0	.63	334	.83	0.3	.99	88	.47			
5 Conc	"	"	1.013	28.5	6.64	26.22		-	4907	6410		35.5		10834		59.6		-				
6 Feed	7/3/75	10:30 AM	.998	28.0	6.28	4.53		-	910	1136		2.7		1747		19.5		147				
6 Perm	"	"	.995	28.0	6.08	0.61	.86	48	-	135	.88	0.8	.70	210	.88	0.2	.99	88	.40			
6 Conc	"	"	1.015	27.5	6.29	26.80		-	5312	6520		14.5		10745		84.8		-				
8 Feed	7/8/75	9:50 AM	.999	29.0	6.15	6.49		-	779	1380		2.8		2261		10.5		186				
8 Perm	"	"	.997	27.0	3.30	0.69	.89	46	-	198	.86	T	.93	392	.83	T	.99	38	.80			
8 Conc	"	"	1.021	30.0	6.45	33.09		-	4615	8670		17.4		14958		70.1		-				
9 Feed	7/9/75	11:00 AM	.997	30.0	6.63	4.30		-	1075	1120		2.5		1716		28.0		145				
9 Perm	"	"	.996	27.0	4.86	0.39	.91	40	-	81	.93	T	.92	190	.89	0.0	1.00	35	.76			
9 Conc	"	"	1.008	30.0	6.70	17.86		-	4256	4280		10.1		7083		105.1		-				
10 Feed	7/10/75	9:40 AM	.999	26.5	6.39	4.09		-	893	1106		2.5		1710		28.0		106				
10 Perm	"	"	.998	26.5	4.60	0.56	.86	44	-	120	.89	0.7	.72	244	.86	T	.99	50	.53			
10 Conc	"	"	1.018	26.5	6.38	30.15		-	5452	7420		19.5		12560		77.1		-				
11 Feed	7/11/75	10:10 AM	0.999	26.8	6.10	4.70		-	995	1222		2.3		1946		6.8		122				
11 Perm	"	"	0.996	27.5	3.68	0.48	.90	35	-	104	.91	0.8	.65	243	.88	0	1.00	43	.65			
11 Conc	"	"	1.012	28.0	6.33	24.42		-	4772	6310		14.0		9832		21.0		-				
12 Feed	7/18/75	12:25 PM	1.002	29.8	6.35	7.33		-	1335	1794		4.0		3032		35.0		216		334		78
12 Perm	"	"	.996	29.5	5.78	1.31	.82	96	-	364	.80	1.3	.68	664	.78	2.8	.92	151	.30	8	.98	
12 Conc	"	"	1.021	29.5	6.38	33.15		-	5893	33720		16.2		13856		21.0		-		-		
13 Feed	7/22/75	10:55 AM	1.000	29.7	6.62	6.07		-	1229	1504		3.0		2490		24.5		244		236		88
13 Perm	"	"	.997	29.7	6.33	1.40	.77	80	255	382	.75	2.0	.33	686	.72	1.0	.96	136	.44	13	.94	-
13 Conc	"	"	1.016	29.8	6.38	26.40		-	5093	6880		12.3		11264		21.0		-				-

*Rejection ratio = 1 - (concentration of permeate/concentration of feed).

†As sodium oxalate.

TABLE B-3. ANALYTICAL DATA

Continuous Operation

Sample No.	Sample	Date	Sp. gravity Sp. gr.	Temp.	pH	Total solids, g/l	Total carbon, mg/l	COD, mg/l	Soluble calcium, mg/l	Sodium, mg/l	Inorganic chloride, mg/l	Soluble* oxalate, mg/l	BOD_5, mg/l	Suspended solids, mg/l	Color units	Osmotic Pressure, psi
14	RO Feed	7/22/75	1.001	28.5	7.35	6.05	-	1198	1520	3.2	2503	14.0	255	168	156	
	Perm		.997	28.8	7.40	1.44	73	175	394	1.7	699	1.4	138	-	0	
	Conc		1.018	29.8	7.72	31.01	-	6263	7600	15.2	12765	7.0	-	-	-	
15	RO Feed	7/23/75	1.002	29.2	7.72	5.74	-	968	1432	12.6	2316	21.0	250	91	62	
	Perm		.997	29.8	7.00	1.28	62	228	347	5.2	652	1.4	123	-	0	
	Conc		1.012	29.5	7.92	22.18	-	3922	6220	43.4	9675	7.0	-	-	-	
16	Set. T. Feed	7/24/75	1.000	30.0	6.15	6.07	-	-	-	-	-	-	-	254	55	29
	RO Feed		1.000	30.0	6.54	6.27	-	1202	1548	4.2	2558	14.7	274	84	92	
	Perm		.996	30.0	6.09	1.20	62	195	343	1.3	593	2.8	147	-	0	
	Conc		1.019	30.3	6.70	30.87	-	6068	8040	27.2	13353	17.5	-	-	-	201
17	Set. T. Feed	7/25/75	1.000	31.0	6.40	6.66	-	-	-	-	-	-	-	294	93	
	RO Feed		1.000	30.0	6.49	6.33	-	1202	1554	3.0†	2623	7.0	263	104	89	
	Perm		.996	30.5	6.05	1.12	64	221	308	4.3†	552	4.2	138	-	0	
	Conc		1.018	30.5	6.59	29.80	-	5639	7800	14.0	12786	10.5	-	-	-	
18	Set. T. Feed	7/26/75	1.001	25.2	6.72	5.61	-	-	-	-	-	-	-	314	132	
	RO Feed		1.001	25.2	6.51	6.64	-	1112	1642	3.3†	2754	7.0	268	105	101	
	Perm		.997	25.0	5.38	1.20	62	234	319	8.0†	569	Trace	126	-	0	
	Conc		1.021	26.2	6.68	33.83	-	5650	8500	16.8	14349	7.0	-	-	-	
19	Set. T. Feed	7/27/75	1.001	24.5	6.65	6.25	-	-	-	-	-	-	-	255	98	
	RO Feed		1.000	24.6	6.60	5.46	-	1066	1402	3.2	2240	7.0	186	89	118	
	Perm		.997	25.0	6.30	1.05	48	213	260	1.3	458	1.4	88	-	0	
	Conc		1.014	25.2	6.74	23.29	-	5095	6310	12.0	9255	8.8	-	-	-	
20	Set. T. Feed	7/28/75	.999	31.0	6.37	5.70	-	-	-	-	-	-	-	536	70	
	RO Feed		1.000	25.0	6.40	5.27	-	1033	1392	3.0	2251	1.4	197	86	74	
	Perm		.996	28.0	5.69	0.83	51	174	238	1.2	431	0.7	84	-	0	
	Conc		1.013	29.0	6.49	23.36	-	4352	6390	11.3	10037	2.8	-	-	-	
21	Set. T. Feed	7/29/75	.999	32.0	6.78	6.36	-	-	-	-	-	-	-	363	104	
	RO Feed		1.000	30.0	6.75	5.58	-	989	1350	3.0	2395	5.8	198	104	81	
	Perm		.995	30.5	6.41	1.02	55	188	256	1.1	533	1.3	96	-	0	
	Conc		1.011	31.0	6.58	19.28	-	4315	5380	10.8	8699	13.5	-	-	-	
22	Set. T. Feed	7/30/75	1.002	28.0	6.25	7.61	-	-	-	-	-	-	-	297	86	
	RO Feed		1.001	29.0	6.41	6.42	-	1189	1570	3.2	2792	6.3	240	103	89	
	Perm		.996	30.0	6.05	1.95	73	230	473	1.7	966	1.1	141	-	0	
	Conc		1.014	31.5	6.65	26.85	-	5055	6520	10.7	11201	6.4	-	-	-	
23	Set. T. Feed	7/31/75	1.002	25.0	6.55	6.32	-	-	-	-	-	-	-	291	95	
	RO Feed		1.000	27.5	6.60	6.20	-	1294	1422	2.6	2525	2.6	217	64	89	
	Perm		.999	28.0	6.51	1.73	73	232	412	1.8	810	1.4	136	-	0	
	Conc		1.014	28.5	6.65	25.71	-	4880	6100	10.1	10521	6.3	-	-	-	

*As sodium oxalate.

†Sampler left on during washup.

TABLE B-4. LOADING AND REJECTION SUMMARY

Continuous Operation

			Total solids				COD				Soluble calcium				Sodium			
				Rejection				Rejection				Rejection				Rejection		
	Sample			Perm	Lost in washup			Perm	Lost in washup			Perm	Lost in washup			Perm	Lost in washup	
Date	No.	Sample	Pounds	1 - Feed	Pounds	%	Pounds	1 - Feed	Pounds	%	Pounds	1 - Feed	Pounds	%	Pounds	1 - Feed	Pounds	%
7/22/75	14	Feed	896				177				225				.47			
		Perm	184	.79	90	10.0	22	.88	29	6.4	50	.78	22	9.8	.23	.51	+.07	+14.9
		Conc	622				126				153				.31			
7/23/75	15	Feed	633				107				158				1.39			
		Perm	120	.81	151	23.8	21	.80	22	20.5	33	.79	24	15.2	.49	.65	.19	13.7
		Conc	362				64				101				.71			
7/24/75	16	Feed	1074				206				265				.72			
		Perm	176	.84	142	13.2	29	.86	29	14.1	50	.81	18	6.8	.19	.74	+.14	+19.4
		Conc	756				148				197				.67			
7/25/75	17	Feed	1096				208				269				.52*,†			
		Perm	167	.85	211	19.2	33	.84	39	18.8	46	.83	35	13.0	-	-	-	-
		Conc	718				136				188				.37			
7/26/75	18	Feed	521				87				129				.26*,†			
		Perm	83	.84	137	26.3	16	.82	21	24.1	22	.83	31	24.0	-	-	-	-
		Conc	301				50				76				.15			
7/27/75	19	Feed	1011				197				260				.59			
		Perm	157	.84	32	3.2	32	.84	+15	+7.6	39	.85	+2	+0.8	.19	.68	+.02	+3.4
		Conc	822				180				223				.42			
7/28/75	20	Feed	1078				211				285				.61			
		Perm	134	.88	+55	+5.1	28	.87	+3	+1.4	38	.87	+26	+9.1	.19	.69	+.06	+9.8
		Conc	999				186				273				.48			
7/29/75	21	Feed	1071				190				259				.58			
		Perm	153	.86	105	9.8	28	.85	+20	+10.5	38	.85	+6	+2.3	.16	.72	+.04	+6.9
		Conc	813				182				227				.46			
7/30/75	22	Feed	737				137				180				.37			
		Perm	191	.74	89	12.1	22	.89	29	21.2	46	.74	23	12.8	.17	.54	.02	5.4
		Conc	457				86				111				.18			
7/31/75	23	Feed	737				154				169				.31			
		Perm	173	.77	73	9.9	23	.85	38	24.7	41	.76	12	7.1	.18	.42	+.06	+19.4
		Conc	491				93				116				.19			
Totals		Feed	8854				1674				2199				5.04†			
		Perm	1538	.83	975	11.0	254	.85	169	10.1	403	.82	131	6.0	1.80	.64	+.18	+3.6
		Conc	6341				1251				1665				3.42			

(continued)

TABLE B-4 (continued)

Date	Sample No.	Sample	Inorganic chloride Pounds	Inorganic chloride Rejection 1 - Perm/Feed	Inorganic chloride Lost in washup Pounds	Inorganic chloride Lost in washup %	Soluble oxalate*,† Pounds	Soluble oxalate*,† Rejection 1 - Perm/Feed	Soluble oxalate*,† Lost in washup Pounds	Soluble oxalate*,† Lost in washup %	BOD_5 Pounds	BOD_5 Rejection 1 - Perm/Feed	Color‡ Pounds	Color‡ Rejection 1 - Perm/Feed
7/22/75	14	Feed	371				2.07				38		23.1	
		Perm	90	.76	25	6.7	.18	.91	1.75	84.5	18	.53	0.0	1.00
		Conc	256				.14				-		-	
7/23/75	15	Feed	255				2.32				28		6.8	
		Perm	61	.76	36	14.1	.13	.94	2.08	89.6	12	.57	0.0	1.00
		Conc	158				.11				-		-	
7/24/75	16	Feed	438				2.52				47		15.8	
		Perm	87	.80	24	5.5	.41	.84	1.68	66.7	22	.53	0.0	1.00
		Conc	327				.43				-		-	
7/25/75	17	Feed	454				1.21				46		15.4	
		Perm	82	.82	64	14.1	.63	.48	.33	27.3	21	.54	0.0	1.00
		Conc	308				.25				-		-	
7/26/75	18	Feed	216				.55				21		7.9	
		Perm	40	.81	48	22.2	.007	.99	.48	87.8	9	.43	0.0	1.00
		Conc	128				.06				-		-	
7/27/75	19	Feed	415				1.30				34		21.9	
		Perm	69	.83	20	4.8	.21	.84	.78	60.0	13	.62	0.0	1.00
		Conc	326				.31				-		-	
7/28/75	20	Feed	460				.29				40		15.1	
		Perm	70	.85	+39	+8.5	.11	.62	.06	20.7	14	.65	0.0	1.00
		Conc	429				.12				-		-	
7/29/75	21	Feed	460				1.11				38		15.5	
		Perm	80	.83	13	2.8	.19	.83	.35	31.5	14	.63	0.0	1.00
		Conc	367				.57				-		-	
7/30/75	22	Feed	321				.72				28		10.2	
		Perm	94	.71	36	11.2	.11	.85	.50	69.4	14	.50	0.0	1.00
		Conc	191				.11				-		-	
7/31/75	23	Feed	300				.31				26		10.6	
		Perm	81	.73	18	6.0	.14	.54	.05	16.1	14	.46	0.0	1.00
		Conc	201				.12				-		-	
Totals		Feed	3690				12.40		†	†	346		142.3	
		Perm	754	.80	245	6.6	2.12	.83	8.06	65.0	151	.56	0.0	1.00
		Conc	2691				2.22				-		-	

*Sampler left on during washup.

†Runs 17 and 16 excluded from totals and averages.

‡In terms of platinum in Standard Methods chloroplatinate color standard.

TABLE B-5. AVERAGE ANALYTICAL DATA

R.O. Processing of Sulfite Bleaching Effluent at Flambeau

	MF*	MP	MC	AP	AC	BP	BC	CP	CC
Specific gravity	1.008	0.996	1.010	0.995	1.014	0.996	1.014	0.995	1.016
Temp., °C	29.0	29.3	30.3	30.3	30.3	30.2	30.3	30.3	30.3
pH	6.45	5.27	6.40	5.26	6.41	5.95	6.38	5.24	6.37
Total solids, g/l	16.98	0.94	19.47	1.30	22.60	2.09	25.01	1.06	28.31
Rejection ratio†	--	0.94	--	0.93	--	0.91	--	0.96	--
COD, mg/l	3154	200	3500	193	4029	224	4541	210	5203
Rejection ratio	--	0.94	--	0.94	--	0.94	--	0.95	--
Soluble calcium, mg/l	4097	272	4770	309	5587	561	6177	257	7067
Rejection ratio	--	0.93	--	0.94	--	0.90	--	0.96	--
Sodium, mg/l	7.4	1.4	8.8	1.1	11.4	2.0	13.0	1.6	14.4
Rejection ratio	--	0.81	--	0.88	--	0.82	--	0.88	--
Inorganic Cl^-, mg/l	7105	524	8272	609	9658	1020	10,548	492	12,069
Rejection ratio	--	0.93	--	0.93	--	0.89	--	0.95	--
Soluble oxalate, mg/l‡	7.4	1.2	6.3	1.5	7.1	1.2	5.7	1.0	5.9
Rejection ratio	--	0.84	--	0.76	--	0.83	--	0.82	--
Color	483	0	--	0	--	0	--	0	--
Rejection ratio	--	1.00	--	1.00	--	1.00	--	1.00	--
Osmotic pressure, psi§	98	--	113	--	139	--	157	--	175
Viscosity, cp#,+	0.752	--	0.752	--	0.761	--	0.764	--	0.769

*MF, MP, MC feed, permeate and concentrate of banks fed by Manton Gaulin pump.
AP, AC, permeate and concentrate of banks fed by Pump A.
BP, BC, permeate and concentrate of banks fed by Pump B.
CP, CC, permeate and concentrate of banks fed by Pump C.

†Rejection ratio = 1 - (concentration of permeate/concentration of feed).

‡As sodium oxalate.

§Osmotic pressure of feed to system = 35.

#Viscosity taken at 35°C.

+Viscosity of feed to system = 0.733.

TABLE B-6. ANALYTICAL DATA

Internal Samples (Grab)

Sample*	Date	Time	Sp. gr.: Sp. gr.	Sp. gr.: Temp., °C	pH	Total Solids: g/l	Total Solids: Rej. ratio	Total carbon, mg/l	Soluble calcium: mg/l	Soluble calcium: Rej. ratio	Sodium: mg/l	Sodium: Rej. ratio	Inorganic chloride: mg/l	Inorganic chloride: Rej. ratio	Soluble oxalate: mg/l	Soluble oxalate: Rej. ratio	Color	COD: mg/l	COD: Rej. ratio	Osmotic pressure,† psi	Viscosity‡,§ centipoises
17 MF	7/25/75	3:30 PM	1.010	31.0	6.35	20.65		-	5780		9.4		8877		4.2		660	3934		122	0.762
17 MP	"	"	.995	31.0	5.79	0.98	.95	68	275	.95	1.7	.82	466	.95	1.1	.73	0	221	.94	-	-
17 MC	"	"	1.013	31.0	6.31	24.32		-	6570		11.0		10241		6.0		-	4411		141	0.756
17 AP	"	"	.995	31.0	5.59	0.79	.97	60	226	.97	0.8	.93	385	.96	3.0	.50	0	205	.95	-	-
17 AC	"	"	1.016	31.0	6.35	27.98		-	7500		14.0		12020		5.6		-	5090		178	0.772
17 BP	"	"	.995	30.5	5.96	1.82	.93	81	490	.93	2.0	.86	900	.93	0.8	.86	0	239	.95	-	-
17 BC	"	"	1.018	31.0	6.29	31.19		-	8120		16.7		13116		3.5		-	5689		196	0.781
17 CP	"	"	.996	31.0	5.80	1.04	.97	75	310	.96	1.7	.90	525	.96	0.6	.83	0	230	.96	-	-
17 CC	"	"	1.021	31.0	6.28	35.38		-	8980		18.6		15062		4.2		-	6587		215	0.780
21 MF	7/29/75	3:00 PM	1.004	27.0	6.49	11.81		-	2330		5.7		4987		12.2		300	2116		74	0.746
21 MP	"	"	.996	28.0	4.05	.43	.96	45	220	.91	1.1	.81	449	.91	1.3	.89	0	164	.92	-	-
21 MC	"	"	1.006	31.0	6.39	13.64		-	3180		7.1		6060		7.8		-	2505		76	0.742
21 AP	"	"	.995	31.0	4.00	.83	.94	47	210	.93	0.9	.87	440	.93	0.2	.97	0	153	.94	-	-
21 AC	"	"	1.013	31.0	6.42	16.64		-	4030		9.8		7303		9.7		-	2974		104	0.750
21 BP	"	"	.995	31.0	5.65	1.57	.91	67	417	.90	1.8	.82	801	.89	1.0	.90	0	161	.95	-	-
21 BC	"	"	1.010	31.5	6.45	19.05		-	4760		11.4		8279		7.7		-	3443		124	0.757
21 CP	"	"	.994	31.0	3.78	.34	.98	43	72	.98	1.3	.89	173	.98	1.3	.83	0	168	.95	-	-
21 CC	"	"	1.012	31.0	6.42	23.23		-	5960		12.5		9906		7.9		-	4042		148	0.765
23 MF	7/31/75	3:00 PM	1.009	29.0	6.50	18.49		-	4180		7.2		7452		5.8		488	3401		97	0.749
23 MP	"	"	.996	29.0	5.96	1.42	.92	73	322	.92	1.3	.82	658	.91	1.1	.81	0	215	.94	-	-
23 MC	"	"	1.011	29.0	6.51	20.44		-	4560		8.4		8515		5.1			3583		122	0.757
23 AP	"	"	.996	29.0	6.18	2.27	.89	71	490	.89	1.7	.80	1003	.88	1.3	.74	0	222	.94	-	-
23 AC	"	"	1.013	29.0	6.45	23.18		-	5230		10.4		9651		6.0			4022		135	0.760
23 BP	"	"	.997	29.0	6.25	2.87	.88	96	775	.85	2.2	.79	1358	.86	1.7	.72	0	272	.93	-	-
23 BC	"	"	1.014	28.5	6.41	24.79		-	5650		11.0		10250		5.8			4491		150	0.754
23 CP	"	"	.996	29.0	6.13	1.81	.93	73	390	.93	1.7	.84	778	.92	1.2	.79	0	232	.95	-	-
23 CC	"	"	1.016	29.0	6.40	26.33		-	6260		12.2		11240		5.5			4980		162	0.763

*MF - Feed to banks fed by Manton Gaulin pump.
MP - Permeate from banks fed by Manton Gaulin pump.
MC - Concentrate from banks fed by Manton Gaulin pump.
AP - Permeate from banks fed by Pump A.
AC - Concentrate from banks fed by Pump A.
BP - Permeate from banks fed by Pump B.
BC - Concentrate from banks fed by Pump B.
CP - Permeate from banks fed by Pump C.
CC - Concentrate from banks fed by Pump C.

†Osmotic pressure of feed to system = #17 - 38; #21 - 34; #23 - 34.

‡Viscosity of feed to system = #17 - 0.732; #21 - 0.729; #23 - 0.738.

§Viscosity taken at 35°C.

TABLE B-7. ANALYTICAL DATA

Grab Samples from Avco Freeze Concentration Trailer Unit

Sample*	Date	Sp. gr. (Sp. gr.)	Sp. gr. (Temp., °C)	pH	Total solids, g/l	Soluble oxalate, mg/l	Susp. solids, mg/l	COD, g/l	Soluble calcium, g/l	Sodium, mg/l	Inorganic chloride, g/l	BOD_5, mg/l	Total carbon, g/l	Osmotic pressure, psi	Viscosity†, centiposes	Color units
4 FA	7/1/75	1.009	29.5	6.27	18.31	37.5	-	3.64	5.56	16.2	5.53	-	-	-	-	-
8 FA	7/8/75	1.010	29.5	6.33	18.68	-	-	-	-	-	-	-	-	-	-	-
9 FA	7/9/75	1.011	29.0	6.40	19.80	-	-	-	-	-	-	-	-	-	-	-
9 CAI		1.083	27.0	7.10	108.08	-	-	25.76	24.00	111	49.48	-	7.26	906	-	11610
9 CAII		1.094	27.0	7.10	127.45	141.6	8155	37.36	28.40	131	55.40	-	3.06	982	-	10393
9 MA		0.996	29.0	6.71	0.16	-	-	0.05	-	-	0.03	7	0.02	-	-	-
10 FA	7/10/75	1.010	26.5	6.51	18.99	35.0	-	3.58	4.81	11.0	7.71	-	-	-	-	-
11 FA	7/11/75	1.007	27.0	6.52	18.29	17.5	-	3.58	4.57	11.4	7.62	-	-	-	-	-
15 CAII		1.063	38.3	8.22	98.26	21.0	-	-	-	-	-	-	-	-	-	-
16 FA		1.016	30.0	6.32	27.29	10.5	-	-	7.01	25.0	11.72	-	-	-	-	-
20 CAI	7/28/75	-	-	7.17	128.90	-	-	-	-	-	-	-	-	-	-	-
20 CAII		1.136	33.0	5.39	181.49	21.0	-	-	-	-	-	-	-	-	-	-
20 MA		0.996	29.0	7.95	0.14	18.9	108	0.25	0.01	Trace	0.01	-	-	0	-	293
23 CAIIA		1.110	27.0	6.30	144.53	27.3	-	32.35	27.60	109	62.49	-	2.92	1193	-	11704
23 CAIIB		1.097	27.0	6.20	79.56	27.0	-	23.42	28.10	102	59.34	-	6.98	1073	-	12172
23 CAIIC		1.063	27.5	6.58	87.18	18.7	-	20.06	19.20	56	38.46	-	1.25	643	-	7772
24 FA	8/6/75	1.015	29.0	6.48	26.14	9.0	148	4.38	4.58	13.4	11.01	-	-	165	0.760	1780
24 CAI		1.081	29.0	5.95	153.36	28.9	-	27.20	26.50	68	54.09	-	1.83	911	0.964	9700
24 MA		0.997	27.0	6.89	0.19	9.3	36	0.08	0.03	Trace	0.04	-	0.03	-	-	22

*FA - Feed to Avco unit.
CAI - Avco concentrate - Stage I.
CAII - Avco concentrate - Stage II.
MA - Melt or recovered water from Avco unit.

†Viscosity taken at 35°C.

TABLE C-1. DAILY OPERATING LOG, R.O. TRAILER, CONTINENTAL GROUP, AUGUSTA, GA

Date	Time/ operating hours	Energy used, kwh	Suction/discharge pressures, psi: Main pump	Pump A	Pump B	Pump C	Feed from main pump: Temp., °C	Flow, gpm	pH	Concentrate: Temp., °C	Sp.gr.	Flow, gpm	Trailer feed, gpm	Flux rate, gfd	Remarks
9/24/75	13:30/8	05751	45/715	690/700	660/690	535/560	37.2	37.0	7.0	36.5	1.009	3.7	21.2	10.4	Grab samples: 101 MF, F, C & P were taken @ 08:00
	15:30/10	05815	45/710	690/700	670/700	550/570	37.8	38.0	7.0	37.4	1.008	3.55	17.2	8.1	
9/25/75	08:00/10¼	05835													
	09:00/11¼	05859	45/710	700/715	680/700	560/575	34.4	37.0	7.0	32.0	1.009	4.0	35.6	15.2	Grab samples: 102 MF, F, C & P taken @ 10:00 a.m. @ 15:20 shut down because of broken feed line. Line was repaired and system rinsed with fresh water @ 16:00. Shut down system for the day @ 17:15
	10:00/12¼	05884	45/710	680/710	660/700	575/595	36.7	36.5	7.0	35.1	1.0105	3.7	32.6	12.8	
	11:00/13¼	05915	45/730	720/750	700/730	590/610	36.1	37.0	7.0	35.1	1.010	3.2	27.5	11.2	
	12:00/14¼	05948	45/730	675/710	665/700	560/580	37.2	37.0	7.0	37.0	1.009	3.5	22.4	9.3	
	13:00/15¼	05981	45/720	680/710	670/700	555/575	37.8	37.0	7.0	38.0	1.008	3.7	20.0	8.0	
	14:00/16¼	06018	45/720	655/700	665/700	560/580	38.3	38.0	7.0	38.0	1.007	3.5	17.9	6.9	
	15:00/17¼	06047	45/715	670/700	670/705	590/605	36.7	37.0	7.0	36.0	1.006	3.5	28.9	5.9	
9/26/75															System was washed with 25 oz BIZ @ 08:20-08:45 and shut down to let BIZ soak
9/29/75	08:15														
	09:00/18	06115	35/690	670/700	645/700	570/585	37.7	35.0	6.8	29.0	1.0145	2.7	28.3	18.8	Recycle started @ 10:30 a.m. Grab samples 103 C, F, MF & P taken @ 10:30. Shut down at 14:08 for pressure pulse. Started up again @ 14:18 at which time feed line became disconnected. Start back up after repair @ 14:31. Flux rate increased from 8.55 to 15.1 during this episode. @ 15:32 feed line again became disconnected. Hose was replaced. After repair system was flushed with water and then shut down. There was no re-cycle @ the time of 15:00 readings
	10:00/19	06149	35/700	670/700	635/700	490/505	38.4	35.0	6.8	38.7	1.013	5.2	26.8	17.2	
	11:00/20	06185	35/710	670/700	645/700	540/560	38.6	35.0	6.8	39.0	1.0045	3.56	22.5	14.4	
	12:00/21	06218	35/700	675/705	630/700	500/520	38.6	36.0	6.8	40.0	1.0045	3.85	19.6	11.5	
	13:00/22	06251	36/705	675/710	645/705	510/530	37.8	36.0	6.8	39.0	1.0035	3.5	17.0	9.7	
	14:00/23	06286	37/705	665/700	630/695	500/520	37.8	36.0	6.8	38.0	1.004	3.45	15.1	8.55	
	15:00/24	06305	37/705	680/705	660/720	550/570	39.4	35.0	6.8	--	--	--	--	15.1	
9/30/75	08:15	06340													
	09:00/24¾	06362	41/700	680/705	670/705	520/530	38.3	35.0	6.5	35.0	1.0075	4.2	33.3	17.3	@ 08:45 a.m. grab sample 108F taken. Samples 104 C & P taken @ 11:00 a.m. No MF sample taken. Shut down @ 11:14 for 10 min to try to increase flux. Outside pump left on. Resume @ 11:24. Shut down @ 14:04, outside pump off, feed line disconnected, let sit for 10 min. Start back up @ 14:14
	10:00/25¾	06393	41/720	680/705	660/700	400/410	37.8	35.0	6.5	35.2	1.0065	3.6	25.4	12.9	
	11:00/26¾	06427	43/700	665/700	655/700	490/505	37.2	35.0	6.5	34.0	1.0055	3.5	20.8	10.4	
	11:30/27¼	06435	38/700	670/700	670/705	570/600	37.2	35.0	6.5	--	--	--	--	13.8	
	12:00/27¾	06451	38/690	650/685	650/690	530/550	37.5	35.5	6.5	33.0	1.0075	2.7	24.1	12.7	
	13:00/28¾	06487	42/710	680/710	665/710	500/515	37.6	35.0	6.5	39.0	1.003	3.6	20.6	10.1	
	14:00/29¾	06517	41/715	680/710	660/705	540/560	37.8	35.0	6.5	40.0	1.003	2.6	17.8	9.0	
	14:30/30¼	06526	37/700	675/705	685/715	625/645	37.8	36.5	6.5	39.0	1.0055	--	--	14.3	
	15:00/30¾	06542	40/710	680/715	680/715	560/575	37.7	34.0	6.5	38.0	1.002	3.0	23.5	12.2	
10/01/75	07:05/30¾	06568													
	08:00/31¾	06578	40/710	685/715	665/715	525/540	37.2	35.0	7.5	32.0	1.0065	1.8	35.0	19.8	@ 07:05 washed system with BIZ solution and then flushed with water. Started running feed through system @ 07:35 a.m. Shut down for 5 min @ 09:45 to increase flux. Outside pump remained on. @ 11:30 Cl_2 feed was increased to 12 gpm to lower pH. Shut down @ 11:45 for 5 min to increase flux.
	09:00/32¾	06613	41/710	685/715	655/705	475/490	37.2	35.5	7.5	37.0	1.0085	2.8	28.2	15.1	
	10:00/33¾	06648	41/705	660/700	650/700	530/550	38.3	36.0	7.5	38.0	1.0105	2.6	27.4	14.7	
	11:00/34¾	06677	43/710	670/710	655/705	520/535	38.9	36.0	7.5	39.0	1.0095	2.5	21.7	11.4	
	12:00/35¾	06707	39/700	665/700	645/695	530/550	38.9	35.5	8.2	40.0	1.007	2.4	25.8	13.9	
	13:00/36¾	06741	45/710	665/700	645/695	505/520	39.4	35.5	8.2	41.0	1.006	2.5	21.5	11.3	
	14:00/37¾	06768	44/710	665/700	650/700	510/520	40.6	36.5	8.2	41.0	1.0075	2.3	23.0	12.3	
	15:00/38¾	06801	40/710	675/710	665/710	520/530	40.0	36.5	8.2	42.0	1.0055	2.3	19.8	10.4	
	16:00/39¾	06832	46/700	660/700	650/695	515/545	40.6	36.0	7.9	42.7	1.0055	3.4	20.9	10.4	

(continued)

TABLE C-1 (continued)

Date	Time/ operating hours	Energy used, kwh	Suction/discharge pressures, psi: Main pump	Pump A	Pump B	Pump C	Feed from main pump: Temp., °C	Flow, gpm	pH	Concentrate: Temp., °C	Sp.gr.	Flow, gpm	Trailer feed, gpm	Flux rate, gfd	Remarks
10/01/75	17:15/40¼	06855	37/690	660/690	650/700	550/570	38.9	36.0	7.6	38.0	1.0065	2.5	22.5	11.9	Outside pump on. Shut down @ 13:45 for 5 min to increase flux (outside pump on). Shut down @ 15:10 for 5 min, outside pump on, to increase flux. Shut down @ 16:10 for 5 min, outside pump on. Shutdown @ 16:45 because feed line became disconnected. Started back up after repairs @ 17:10. Shut down @ 18:10, 20:10, 21:10, 22:05 for 5 min each as a pressure pulse with air. Pressure pulses @ 00:30 - 10/2/75. Concentrate storage overflow @ 22:10. At 23:00 started 10 gpm Cl_2 addition to stock to lower pH. Caustic reactants + hypo were not added at this time
	18:00/41	06880	46/710	680/705	660/710	540/560	37.8	35.0	7.6	40.5	1.0060	2.5	19.4	10.0	
	19:00/42	06912	45/705	670/700	655/705	530/550	38.9	35.0	7.6	40.5	1.0070	2.5	17.8	9.1	
	20:00/43	06940	46/720	690/720	670/720	530/550	38.9	36.0	7.8	40.7	1.0070	2.5	18.8	9.7	
	21:00/44	06965	44/700	670/700	660/705	470/500	37.8	37.0	8.0	40.0	1.0075	2.3	16.8	8.6	
	22:00/45	06995	45/690	655/690	650/700	510/540	37.2	35.0	8.0	38.5	1.0070	2.5	17.6	9.0	
	22:55/46	07021	44/710	680/715	650/700	480/500	37.2	36.0	7.75	38.5	1.0070	2.0	16.8	8.8	
	24:00/47	07039	41/700	680/715	650/700	500/520	37.8	35.0	7.55	38.5	1.0080	2.0	17.8	9.4	
10/02/75	01:00/48	07077	42/720	690/730	650/710	500/520	34.4	35.0	7.2	37.0	1.0085			8.1	Pressure pulses without air @ 01:45, 02:45 & 03:45. Pressure pulse consists of shuting down for 5 min leaving outside pump running. Pressure pulses with air @ 04:45 & 05:45. @ 01:00 wind from north, outside temp. dropping 72°F. @ 02:00 outside temp. 68°. Started filling feed tank. @ 03:00 outside temp. 67°F. @ 04:00 raining out, temp. 67. @ 05:00 still raining out, temp. 65°. @ 06:00 still drizzle, temp. 66°. @ 07:15 started rinsing system with 500 gal water. Washed with BIZ @ 08:15 & let soak for 15 min. Then rinsed with 300 gal water. Pulled out some R-O-P to check them after rinsing. Drew samples 106 C, P, MF and F @ 13:00. Shutdown @ 14:35 to rinse with 100 gal of water to help increase flux. A shot of compressed air was added to system. @ 16:00 there was no recycle because recycle hose broke. Hose repaired
	02:00/49	07103	42/720	690/720	650/710	535/550	35.6	34.0	7.0	37.0	1.007	2.0	18.2	9.7	
	03:00/50	07133	42/700	680/710	650/700	530/550	35.6	35.0	6.95	36.5	1.007	2.0	17.8	9.4	
	04:00/51	07166	44/700	660/700	650/700	510/540	36.1	35.0	6.95	37.0	1.007	2.0	17.0	8.9	
	05:00/52	07192	36/700	670/710	660/710	530/560	36.7	35.0	6.75	37.0	1.0065	2.0	19.1	10.2	
	06:00/53	07222	45/700	670/710	660/710	540/560	36.7	35.0	6.65	37.0	1.003	2.0	19.1	10.2	
	07:00/54	07255	47/710	680/710	650/700	480/500	36.7	35.0	6.75	37.0	1.005	2.0	15.0	7.8	
	10:50/55	07284	47/700	690/715	650/700	510/525	36.7	35.0	6.3	30.0	--	--	--	12.5	
	12:00/56	07321	49/700	670/705	650/700	530/540	37.2	35.0	6.7	39.0	1.004	2.5	19.4	10.1	
	13:00/57	07357	50/700	670/705	650/705	530/550	36.7	35.0	6.65	38.0	1.0048	2.1	16.5	8.5	
	14:00/58	07387	50/700	660/700	650/700	530/550	37.8	35.0	6.60	37.5	1.0070	2.6	15.1	7.4	
	14:25/58	07400	50/700	665/700	650/700	520/540	37.8	35.0	6.55	40.0	1.0060	3.0	14.9	7.0	
	15:00/59	07413	50/710	680/715	650/700	520/530	37.8	35.0	6.40	38.0	1.002	16.5	35.0	11.0	
	16:00/60	07445	50/700	670/705	650/700	525/550	38.9	35.0	—	—	—			9.7	
10/03/75	08:15/60														
	09:00/60¼	07472	51/710	690/715	655/705	495/515	36.7	34.0	6.35	37.2	1.0035	3.5	24.5	12.5	Started up @ 08:15 pH 6.4. Sample 107 C, P, & MF taken @ 10:30. Shutdown @ 12:06 to flush with 100 gal water. Resume @ 12:20. @ 15:35 BIZ solution was put into system to soak over the weekend
	10:00/61¼	07503	37/700	670/700	650/700	525/540	37.2	35.0	6.7	39.0	1.0055	2.4	19.9	10.4	
	11:00/62¼	07538	39/710	675/710	650/705	505/520	37.8	35.0	6.45	40.4	1.0060	3.8	18.7	8.9	
	12:00/63¼	07573	42/710	670/705	650/705	540/560	37.8	35.0	6.58	39.0	1.0063	3.6	16.5	7.7	
	13:00/64¼	07601	44/705	665/705	650/705	530/550	36.7	35.0	6.4	38.0	1.0050	2.2	16.6	8.5	
	14:00/65¼	07635	45/705	650/700	645/700	530/550	35.6	35.0	6.45	38.0	1.0055	3.3	14.9	6.9	
	15:00/66¼	07662	46/705	650/700	645/700	520/540	36.7	35.0	6.45	38.0	1.0055	3.6	14.5	6.5	
	15:30/67¼	07688	46/700	645/695	635/695	500/520	36.7	35.0	6.45	39.0	1.0055	3.3	13.8	6.3	

(continued)

TABLE C-1 (continued)

Date	Time/ operating hours	Energy used, kwh	Suction/discharge pressure, psi: Main Pump	Pump A	Pump B	Pump C	Feed from main pump: Temp., OC	Flow, gpm	pH	Concentrate: Temp., °C	Sp.gr.	Flow, gpm	Trailer feed, gpm	Flux rate, gfd	Remarks
10/06/75	07:05														@ 07:05 rinsed with water. @ 07:25
	08:00/68¼	07705	44/700	665/705	670/705	535/550	35.0	35.0	6.35	34.5	1.000	3.4	26.0	13.4	started running feed through system.
	09:00/69¼	07740	46/710	665/705	660/700	520/540	35.6	35.0	6.5	35.0	1.0065	3.5	22.0	11.0	@ 07:30 108 F was taken. @ 08:05
	10:00/70¼	07770	47/700	650/700	660/700	520/540	36.1	35.0	6.6	37.0	1.0075	3.5	19.0	9.2	recycle was started. @ 10:05 sam-
	11:00/71¼	07804	45/715	665/710	670/710	530/550	36.7	35.0	6.3	38.0	1.0070	3.5	17.0	8.0	ples 108 MF, P & C taken. @ 11:30
	12:00/72¼	07830	44/700	645/690	650/690	540/550	36.7	35.0	6.2	37.3	1.0015	3.5	18.4	8.8	flush with 100 gal water to increase
	13:00/73¼	07863	45/705	650/700	650/700	540/550	33.3	35.0	6.2	36.5	1.0035	3.2	14.6	6.8	flux. @ 11:40 feed through system. @ 13:30 rinse with fresh water the washed out with solution of 300 gal water, 2 liters 18M HCl & 3 gal Versene. Let sit for 1 hr then rinsed with water and shutdown @ 15:30
10/07/75	07:00/73¼	07876													Start up @ 0.7:00. Took sample of
	08:00/74¼	07904	45/715	680/710	640/700	500/520	35.0	35.0	6.1	37.0	1.0051	--	--	14.4	feed @ 07:45. Started recycle @
	09:00/75¼	07942	47/700	650/690	630/690	510/530	36.7	36.0	6.87	38.0	1.0070	3.5	23.6	12.0	08:10. Samples 109 C, M, MF taken
	10:00/76¼	07973	45/710	660/700	630/700	510/530	36.7	35.5	6.95	38.3	1.0080	3.5	20.4	10.0	@ 10:00. Flush system with 150 gal
	11:00/77¼	08017	46/710	660/700	630/700	510/530	36.7	35.0	6.78	39.0	1.0075	3.5	17.4	8.2	water @ 11:10. Started running feed
	12:00/78¼	08043	46/710	660/700	640/700	510/520	35.0	35.0	6.50	38.3	1.0038	3.5	20.3	10.0	through @ 11:25. Shutdown @ 15:15
	13:00/79¼	08077	47/715	670.710	640/700	540/550	39.2	35.0	6.55	40.2	1.0050	3.5	17.9	8.6	for the day
	14:00/80¼	08109	47/715	670/710	650/710	540/550	37.8	35.0	6.62	40.5	1.0045	3.5	16.3	7.6	
	15:00/81¼	08132	47/710	655/705	640/700	520/530	38.9	35.0	6.60	41.0	1.0039	3.5	15.3	7.0	
10/08/75	07:10/81¼	08141													Start 24 hr continuous operation @
	08:00/82¼	08165	42/715	690/720	645/700	540/555	33.9	35.0	6.8	36.0	1.0080	3.5	29.8	15.6	07:00. @ 09:10 pressure pulse with
	09:00/83¼	08208	44/735	710/740	670/730	550/570	35.6	35.0	7.0	37.0	1.0090	3.5	26.1	13.4	air. @ 11:10 100 gal of water to
	10:00/84¼	08230	45/720	685/720	650/710	550/565	36.7	35.0	7.2	38.5	1.0090	—	—	11.9	flush system. @ 12:10, 13:15, 14:15
	11:00/85¼	08264	48/700	660/700	630/700	530/550	37.8	35.0	7.4	39.5	1.0100	2.5	19.1	9.9	down for pressure pulse with air.
	12:00/86¼	08290	51/700	645/700	650/700	550/570	37.8	35.0	7.7	39.5	1.0060	3.6	21.0	10.4	Pressure pulse with air consists of
	13:00/87¼	08319	52/700	635/695	645/695	530/550	37.8	35.0	7.9	39.0	1.0040	3.5	20.7	10.2	shütdown of all machinery, draining
	14:00/88¼	08349	52/710	650/705	655/705	550/570	38.3	35.0	7.5	38.5	1.0070	2.5	18.0	9.2	heat exchanger of feed & putting
	15:00/89¼	08376	45/720	705/720	660/715	545/555	37.2	35.0	7.3	37.0	1.0040	2.5	19.9	10.4	compressed air in line for 1 min.
	16:00/90¼	08406	42/710	700/715	655/705	490/510	37.8	35.0	7.5	38.2	1.0060	2.5	15.8	7.9	Total process takes 5-6 min. @
	17:00/91¼	08438	42/725	700/715	655/700	475/500	37.8	36.0	7.55	39.0	1.0068	2.5	15.7	7.8	14:45 down for 100 gal water flush.
	18:00/92¼	08469	44/700	630/700	650/695	460/480	37.8	35.0	7.55	38.7	1.0071	2.5	14.7	7.3	Consists of same as pressure pulse
	19:00/93¼	08495	41/700	645/705	640/700	485/515	36.7	34.0	7.50	37.2	1.0035	2.5	16.4	8.3	except you use water and process
	20:00/94¼	08525	39/710	650/720	650/710	540/570	36.7	35.0	7.40	37.0	1.0060	2.5	16.4	8.3	takes 15 min. This was done ca.
	21:00/95¼	08556	46/700	640/710	640/700	535/560	37.2	35.0	7.27	37.2	1.0060	2.5	16.2	8.1	every 4 hr of operation. Pressure
	22:00/96¼	08587	47/705	650/710	650/705	540/570	37.2	35.0	7.45	37.0	1.0054	2.5	16.1	8.1	every hour except water pulse.
	23:00/97¼	08620	47/700	650/700	640/700	530/560	36.1	34.0	7.30	36.0	1.0030	2.5	16.0	8.0	Pressure pulses with air @ 16:10,
	24:00/98¼	08642	48/710	660/710	650/710	540/570	36.7	35.0	7.30	37.0	1.0038	2.5	16.0	8.0	17:10, 18:45, 20:45, 21:40, 23:45 & 00:45. Water flush @ 18:40 & 22:40. @ 10:30 it was found that rotometer had fibers in tube
10/09/75	01:00/99¼	08671	49/700	650/700	640/700	530/560	36.7	34.0	7.30	38.0	1.0042	2.5	15.4	7.7	Pressure pulses @ 01:45, 03:45,
	02:00/100¼	08702	48/700	650/700	640/700	500/540	36.7	35.0	7.30	39.0	1.0051	2.5	14.9	7.4	04:45, 05:45. Water flush with 100
	03:00/101¼	08731	39/700	650/700	645/705	530/560	35.6	35.0	7.30	36.0	1.0032	2.5	16.4	8.2	gal @ 02:40 & 06:45. Air was also
	04:00/102¼	08753	39/700	650/700	660/710	530/560	35.6	35.0	7.30	37.0	1.0035	2.5	15.9	8.0	introduced into system after water
	05:00/103¼	08783	40/700	650/700	630/690	500/530	36.1	35.0	7.40	37.0	1.0038	2.5	14.9	7.4	flush. @ 07:25 shutdown for wash-uP
	06:00/104¼	08814	39/710	670/710	650/700	520/540	36.7	35.0	7.35	37.0	1.0056	2.5	14.2	7.0	Rinse with fresh water, used 300
	07:15/105½	08840	36/710	665/705	645/705	525/555	35.6	35.0	7.30	31.0	1.0075	2.5	17.4	8.9	gal water with 800 g of gas. Soak
	08:30/106¾	08853	46/710	670720	685/715	55/575	35.0	35.0	7.2	35.0	1.0020	3.0	19.3	9.7	for 10 min & then flush out with
	09:00/107¾	08868	47/700	645/695	660/700	545/550	36.1	35.0	7.2	37.0	1.0040	3.0	17.0	8.3	feed. Start up @ 8:20. Pressure
	10:00/108¾	08898	47/710	660/710	675/710	555/575	36.1	34.5	7.2	36.5	1.0050	3.0	17.0	8.3	pulse with air @ 9:45, 10:45, 11:45,
	11:00/109¾	08929	48/715	665/710	675/710	540/565	36.7	34.0	7.1	35.0	1.0060	2.7	16.0	7.[illegible]	13:45, 14:45, 15:45, 17:45 & 19:45.

(continued)

TABLE C-1 (continued)

Date	Time/ operating hours	Energy used, kwh	Suction/discharge pressure, psi: Main Pump	Pump A	Pump B	Pump C	Feed from main pump: Temp., °C	Flow, gpm	pH	Concentrate: Temp., °C	Sp.gr.	Flow, gpm	Trailer feed, gpm	Flux rate, gfd	Remarks
10/09/75	12:00/110¾	08958	50/710	660/710	675/710	545/565	37.8	34.5	7.4	34.5	1.0070	2.5	16.0	8.0	Shutdown and flushed with gas so-
	13:00/111¾	08978	42/710	660/715	655/710	540/555	37.8	35.0	7.6	34.0	1.0070	2.5	17.4	8.8	lution @ 12:30, 18:30 & 22:30.
	14:00/112¾	09019	47/720	665/715	670/720	565/580	38.3	34.0	7.5	34.5	1.0030	--	--	9.0	Pressure pulse with air @ 20:45,
	15:00/113¾	09038	50/720	665/715	670/720	540/560	--	35.0	--	35.5	1.0037	--	--	8.3	21:45 & 23:45. No recycle running
	16:00/114¾	09067	52/700	660/705	660/705	545/555	37.8	34.5	7.30	38.5	1.0042	2.5	15.7	7.8	at time of 15:00, 19:00 & 23:00
	17:00/115¾	09093	52/730	680/730	680/725	550/570	36.7	35.0	7.50	32.0	1.0092	2.1	15.1	7.71	readings. All concentrate was
	18:00/116¾	09132	53/700	650/700	650/700	505/525	36.7	35.0	7.50	36.0	1.007	2.5	14.1	6.88	being sewered at this time because
	19:00/117¾	09142	40/720	640·720	670/700	570/590	36.7	34.0	7.65	36.0	1.004	--	--	9.04	of possibility of gain in concen-
	20:00/118¾	09167	49/700	665/705	660/710	540/560	36.1	35.5	7.65	37.0	1.004	3.5	17.5	8.29	trate. Sewering began at time of
	21:00/119¾	09196	49/700	660/700	665/705	520/545	35.8	35.0	7.70	37.0	1.005	3.5	16.2	7.55	gain. Flush and ended @ ¼ past the
	22:00/120¾	09224	50/700	660/705	670/710	520/545	35.6	35.0	7.70	35.0	1.0055	3.5	16.2	7.5	hour. Collected composite samples
	23:00/121¾	09253	36/700	660/705	660/710	540/570	36.1	35.0	7.85	--	--	--	--	8.44	from previous 24 hr running.
	24:00/122¾	09277	48/710	660/705	660/710	550/565	35.6	35.0	7.80	36.0	1.0020	3.5	18.0	8.61	Cooled & stored for 03:00 p.m.
															shipment. Samples 75-26 110 C, F,
															MF & P. No Avco samples available
10/10/75	01:00/123¼	09303	48/710	660/705	655/705	530/550	35.6	35.0	7.80	35.0	1.0042	3.5	19.4	9.43	Pressure pulses with air @ 00:45,
	02:00/124¼	09332	45/710	660/705	655/705	540/570	35.0	35.0	7.85	33.0	1.0042	3.5	20.6	10.13	01:45, 03:45, 04:45, 05:45 &
	03:00/125¼	09351	38/700	645/695	640/700	530/550	35.6	34.0	7.80	--	--	--	--	12.02	06:45. Flush with Gain solution
	04:00/126¼	09374	39/700	670/710	650/710	560/580	35.0	35.0	7.90	34.0	1.0029	3.5	23.7	11.98	at 02:30. Shutdown @ 07:10 because
	05:00/127¼	09401	40/690	660/700	640/700	550/570	35.6	35.0	7.90	33.0	1.0040	3.5	23.7	12.02	of color in permeate. No recycle
	06:00/128¼	09427	47/710	665/705	660/715	520/540	35.0	35.0	7.95	33.0	1.0030	3.5	23.7	11.24	@ 03:00 reading because of sewering
	07:00/129¼	09452	40/700	665/705	640/700	490/500	35.0	35.0	7.90	36.0	1.0040	--		11.42	all concentrate. @ 07:10 replaced
	14:30/130¼	09559	48/710	660/715	680/725	545/560	36.7	34.0	7.60	37.0	1.0030	3.5	26.4	13.6	3 Rev-O-Pak tubes. @ 08:15 flush
	15:00/130¼	09575	48/710	660/715	680/715	505/535	37.2	35.0	7.75	39.8	1.0035	3.5	24.6	12.55	out system with fresh water. @
	16:00/131¼	09605	48/710	655/710	675/710	520/540	37.8	35.0	7.50	40.5	1.0050	3.5	24.5	12.46	09:00 wash with Gain. @ 10:00 flush
	17:00/132¼	09633	47/710	660/715	675/710	470/490	37.8	35.0	7.60	40.0	1.0050	3.5	22.3	11.14	with fresh water. @ 10:30 start
	18:00/133¼	09664	44/690	635/690	650/690	510/530	36.7	35.0	7.60	40.2	1.0050	3.5	21.8	10.87	Versene wash (buffer to 7.5). @
	19:00/134¼	09686	43/700	650/710	670/705	545/570	36.1	35.5	7.35	37.0	0.9700	--	--	11.42	13:15 rinse with fresh water. @
	20:00/135¼	09714	46/690	645/700	660/700	510/530	36.1	34.5	7.40	38.0	1.0030	3.4	20.9	10.42	14:00 start back up with feed.
	21:00/136¼	09743	46/715	660/715	670/705	505/530	36.1	35.0	7.50	37.5	1.0035	3.6	20.4	9.96	Pressure pulse with air @ 15:45.
	22:00/137¼	09771	46/710	650/710	660/700	560/580	36.1	35.0	7.50	38.0	1.0035	3.6	20.2	9.85	@ 16:00 color in permeate. Bad
	23:00/138¼	09800	44/720	660/715	670/700	540/560	35.6	35.0	7.50	37.0	1.0015	3.4	18.6	9.05	Rev-O-Pak found and plug to stay
	24:00/139¼	09830	46/700	670/710	670/710	560/580	35.6	35.0	7.55	36.0	1.0052	3.5	18.7	9.05	in operation. Pressure pulses with
															air @ 17:45, 19:45, 20:45, 21:45
															& 23:45. Fresh water rinse @ 18:40
															& 22:40. No recycle @ 14:30 & 19:00
															readings. Collected samples 75-26
															111 C, P, MF, F from previous 24
															hr operation
10/11/75	01:00/140¼	09856	44/700	660/700	660/700	540/560	35.6	35.0	7.50	37.0	1.0042	3.5	18.2	8.74	Pressure pulses with air 01:45,
	02:00/141¼	09888	43/710	665/705	665/705	530/550	35.6	35.0	7.50	37.0	1.0045	3.5	18.1	8.68	03:45,04:45, 05:45 & 06:45. Flush
	03:00/142¼	09910	39/710	670/710	670/710	540/560	35.0	35.0	7.40	33.0	1.0015	18.1	35.0	10.07	with 100 gal water @ 02:40. No re-
	04:00/143¼	09935	43/700	670/710	640/700	530/550	35.0	35.0	7.50	33.0	1.0020	3.5	18.2	8.74	cycle @ 03:00 reading. @ 07:05 rinse
	05:00/144¼	09962	44/700	670/710	650/710	530/550	34.4	35.0	7.50	34.0	1.0049	3.5	19.7	8.53	with fresh water and then wash with
	06:00/145¼	09988	44/710	660/700	640/700	510/530	34.4	35.0	7.50	34.5	1.0040	3.5	17.3	8.20	Gain solution for ½ hr. Flush with
	07:00/146¼	10017	43/700	650/690	630/690	550/570	35.6	35.0	7.60	36.0	1.0045	3.5	17.8	8.49	fresh water. Complete wash-up @
	10:00/147¼	10067	42/700	640/705	650/700	560/575	36.1	35.0	8.2	35.0	1.0010	3.5	23.9	12.1	8:30. Repaired Rev-O-Pak. Start up
	11:00/148¼	10097	43/700	645/710	650/700	540/560	36.7	35.0	8.1	38.0	1.0050	3.5	22.9	11.5	operation again @ 09:30. Pressure
	12:00/149¼	10132	42/700	635/700	640/690	540/565	36.7	35.0	8.1	38.0	1.0060	3.5	20.9	10.4	pulses with air @ 10:45, 11:45,
	13:00/150¼	10162	41/700	645/710	650/700	545/560	37.8	34.0	7.4	35.0	1.0070	3.6	21.7	10.7	12:45, 13:45, 17:45, 18:45, 20:45
	14:00/151¼	10189	43/700	690/705	650/700	545/560	38.3	35.0	7.4	39.5	1.0050	3.5	21.6	10.7	& 23:45. Water flush with 100 gal
	15:00/152¼	10222	44/690	625/695	630/690	495/510	40.0	36.0	7.45	42.5	1.0035	4.0	19.9	9.43	water @ 15:30, 19:30 & 22:35. No
	16:00/153¼	10258	44/700	635/700	640/690	500/510	37.2	35.0	7.4	40.0	1.0010	3.7	19.4	9.33	recycle on @ time of 20:15 reading

(continued)

TABLE C-1 (continued)

Date	Time/ operating hours	Energy used, kwh	Suction/discharge pressure, psi: Main pump	Pump A	Pump B	Pump C	Feed from main pump: Temp., °C	Flow, gpm	pH	Concentrate: Temp., °C	Sp.gr.	Flow, gpm	Trailer feed, gpm	Flux rate, gfd	Remarks
10/11/75	17:00/154¼	10283	45/700	640/710	655/705	550/570	36.7	35.0	7.2	40.0	1.0020	3.6	17.5	8.25	Collected composite samples 112
	18:00/155¼	10312	46/690	630/690	650/700	535/550	36.7	35.5	7.2	38.7	1.0030	3.4	19.2	9.34	C, MF & P from previous day.
	19:00/156¼	10349	47/700	640/700	660/710	540/560	36.7	35.5	7.15	38.3	1.0035	3.7	19.1	9.14	Samples (grab) 113 MC, MP, AC,
	20:15/157½	10376	42/705	645/705	660/710	540/560	37.2	35.0	6.7	35.0	1.000	2.0	16.9	8.87	AP, BC, BP, CC & CD taken. Turn-
	21:00/158¼	10405	47/710	645/705	655/710	525/540	36.7	35.0	6.9	38.5	1.0020	3.5	18.4	8.82	ed off storage tank in coming
	22:00/159¼	10424	48/690	645/695	660/720	560/580	36.1	35.5	6.9	38.5	1.0023	3.5	18.4	8.82	lines @ 21:45 because feed was
	23:00/160¼	10453	48/700	650/700	640/700	510/530	36.1	35.0	7.2	38.0	1.000	3.5	18.4	8.87	backing up into incoming hoses
	24:00/160½	10470	48/700	650/700	640/700	540/560	36.1	35.0	7.25	36.0	1.0018	3.5	17.9	8.57	
10/12/75	01:00/161¼	10518	48/700	650/700	640/700	540/550	36.1	35.0	7.35	38.0	1.0020	3.5	16.8	7.92	Pressure pulses with air @ 00:45,
	02:00/162¼	10535	48/700	650/700	640/700	530/540	35.6	35.0	7.40	38.0	1.0032	3.5	16.8	7.88	01:45, 03:45, 04:45, 05:45 &
	03:00/163¼	10565	40/700	650/700	640/700	520/530	36.7	35.0	7.50	37.8	1.000	3.5	18.2	8.74	06:45. Water flush with 100 gal
	04:00/164¼	10595	47/700	650/700	640/700	510/520	35.6	35.0	7.58	34.0	1.0022	3.5	15.9	7.33	water at 02:30. @ 07:10 wash-up.
	05:00/165¼	10620	46/700	650/700	640/700	510/520	35.0	35.0	7.40	37.5	1.0025	3.5	16.7	7.85	Rinse with fresh water, wash with
	06:00/166¼	10650	45/700	645/695	635/695	540/550	35.6	35.0	7.50	37.0	1.0025	3.5	16.7	7.82	Gain and flush with fresh water.
	07:00/167¼	10677	46/700	650/700	640/700	550/560	35.0	35.0	7.50	37.0	1.0028	3.5	16.5	7.70	Start up again @ 08:10. Pressure
	09:00/168	10727	41/715	670/715	660/715	540/560	39.4	35.0	7.00	38.0	1.0010	3.5	19.5	9.5	pulses with air @ 09:45, 10:45,
	10:00/169	10757	47/710	670/710	655/705	545/565	38.3	35.0	7.10	33.0	1.0050	2.5	17.3	8.8	11:45, 13:45, 14:45, 15:45, 16:45
	11:00/170	10786	48/700	670/710	660/710	540/555	38.3	35.0	7.10	34.5	1.0060	2.5	16.5	8.3	17:45, 19:45, 20:45, 21:45 &
	12:00/171	10813	49/710	670/715	650/705	545/565	38.9	35.0	7.20	33.5	1.0060	2.5	16.0	8.0	23:45. Water flush with 100 gal
	13:00/172	10839	51/710	645/715	655/710	555/580	38.9	33.0	7.20	33.0	1.0010	2.5	18.6	9.6	water @ 12:30, 18:30, 22:30.
	14:00/173	10866	51/710	640/710	650/710	565/580	39.4	35.0	7.30	33.0	1.0050	2.5	17.5	8.9	Collected samples 113 P, MF & C
	15:00/174	10895	52/720	650/715	655/715	580/600	39.4	35.0	7.30	37.5	1.0052	2.5	16.5	8.3	from previous days run
	16:00/175	10925	52/705	640/710	650/705	580/600	38.3	35.0	7.35	40.5	1.0055	2.3	15.7	7.96	
	17:00/176	10953	52/700	635/700	640/700	570/590	38.3	36.0	7.30	40.0	1.0061	2.7	16.0	7.88	
	18:00/177	10982	55/700	640/705	645/700	580/600	38.9	35.0	7.35	39.8	1.0064	2.5	15.4	7.65	
	19:00/178	11013	55/690	620/690	630/680	560/580	37.8	35.0	7.30	38.0	1.0020	2.5	17.2	8.74	
	20:00/179	11032	53/700	630/710	650/700	580/600	37.2	35.0	7.30	39.0	1.0040	2.5	16.1	8.06	
	21:00/180	11057	52/700	630/705	645/700	580/600	36.7	35.5	7.30	39.0	1.0045	2.4	15.3	7.68	
	22:00/181	11088	51/700	620/700	640/695	560/580	36.1	35.0	7.25	38.0	1.0053	2.5	14.9	7.37	
	23:00/182	11127	36/700	650/700	640/700	550/560	37.8	34.0	7.10	38.0	1.0008	2.5	16.6	8.37	
	24:00/183	11141	50/700	650/700	640/700	530/540	35.6	35.0	7.10	38.0	1.0025	2.5	15.2	7.52	
10/13/75	01:00/184	11161	51/700	650/700	640/700	530/540	35.0	35.0	7.10	37.0	1.0045	2.5	14.7	7.21	Pressure pulses with air @ 00:45,
	02:00/185	11198	51/700	650/700	640/700	520/530	35.6	35.0	7.10	37.0	1.0045	2.5	14.5	7.13	01:45, 03:45, 04:45, 05:45 &
	03:00/186	11230	50/710	655/705	645/705	540/550	36.1	35.0	7.00	36.0	1.0010	2.5	15.8	7.92	06:45. Flush with 100 gal water @
	04:00/187	11252	48/700	650/700	640/700	550/560	36.1	35.0	7.00	36.0	1.0025	2.5	15.9	7.96	02:30. Shutdown @ 07:05 to take
	05:00/188	11283	48/700	650/700	640/700	550/560	36.1	35.0	7.00	38.0	1.0035	2.5	15.1	7.46	out a Rev-O-Pak and wash up. Com-
	06:00/189	11314	49/700	650/700	640/700	550/560	36.7	35.0	7.00	39.0	1.0035	2.5	14.9	7.36	posite samples 114 F, MF, P & C
	07:00/190	11342	49/710	660/710	650/710	560/570	35.6	35.0	7.00	38.0	1.0042	2.5	14.6	7.19	taken from previous days run.
	09:00/190¼	11359	47/720	680/720	680/725	550/570	36.7	34.0	6.70	35.0	1.0020	3.5	20.7	10.2	Start up again @ 08:45. Rev-O-
	10:00/191¼	11385	49/700	660/700	655/695	550/565	36.7	34.0	6.70	38.0	1.0040	3.5	18.2	8.7	Pak replaced before start-up.
	11:15/192¼	11418	52/690	655/695	670/700	540/560	36.1	35.0	6.70	34.0	1.0060	2.5	16.1	8.1	Pressure pulses with air @ 09:45,
	12:00/193¼	11436	52/700	655/700	660/695	540/560	36.1	35.0	6.90	35.0	1.0060	2.5	15.9	8.0	10:45 & 12:45. @ 11:45 we pumped
	13:00/194¼	11464	52/700	655/705	665/705	545/560	36.7	35.0	6.90	37.0	1.0060	2.5	16.0	8.0	350 gal of feed through the sys-
	14:00/195¼	11490	51/700	660/695	645/700	545/565	37.2	35.0	6.60	32.0	1.0050	2.5	16.5	8.3	tem @ low pressure (150 psi).
	15:00/196¼	11525	54/710	670/715	655/710	55/575	38.3	35.0	6.80	37.8	1.0040	2.5	16.4	8.3	Pressure pulses @ 14:45, 15:45,
	16:00/197¼	11549	52/700	660/705	650/705	555/580	38.9	35.0	7.0	40.8	1.0043	2.5	16.4	8.25	16:45, 18:45, 19:45, 22:45 & 23:4
	17:00/198¼	11577	53/710	660/710	655/710	565/585	39.4	35.0	7.0	40.0	1.0050	2.2	16.0	8.21	Flush with 100 gal of water @
	18:00/199¼	11612	36/690	650/690	640/690	550/565	38.9	35.0	7.15	41.0	0.9600	2.5	16.3	8.87	13:30, 17:40 & 21:30. No recycle
	19:00/200¼	11631	52/710	670/710	655/715	570/590	37.8	35.0	7.20	40.5	1.0015	2.5	17.4	8.78	at 18:00 reading
	20:00/201¼	11658	52/690	650/695	635/690	525/540	36.7	35.0	7.60	36.0	1.0030	2.6	16.1	8.03	
	21:00/202¼	11683	47/720	670/720	650/715	550/570	36.7	35.0	7.65	39.0	1.0050	2.4	15.2	7.62	
	22:00/203¼	11719	52/710	660/710	650/710	550/570	36.7	35.0	7.63	37.0	0.9900	2.5	16.7	8.41	
	23:00/204¼	11737	47/680	645/690	640/680	530/550	36.1	35.0	7.60	37.0	1.0040	2.5	15.3	7.62	
	24:00/205¼	11765	51/700	650/700	630/690	520/540	36.1	35.0	7.80	39.0	1.0045	2.5	15.0	7.39	

(continued)

TABLE C-1 (continued)

Date	Time/ operating hours	Energy used, kwh	Suction/discharge pressure, psi				Feed from main pump			Concentrate			Trailer feed, gpm	Flux rate, gfd	Remarks
			Main pump	Pump A	Pump B	Pump C	Temp., °C	Flow, gpm	pH	Temp., °C	Sp.gr.	Flow, gpm			
10/14/75	01:00/206¼	11794	52/700	650/700	640/700	530/550	36.1	35.0	7.80	38.0	1.0045	2.5	14.7	7.23	Pressure pulses with air @ 0:45, 01:45, 02:45, 04:45, 05:45 & 6:45. Flush with 100 gal water @ 03:30. Shutdown @ 07:20 to wash up, 2.5 lb Gain to 300 gal water. Start up with feed @ 09:20. Pressure pulses with air @ 10:50, 11:50, 12:50, 13:45, 16:45, 17:45 & 18:45. Flush with 100 gal fresh water @ 15:30. Samples 116 C, MF, F & P taken from previous days operation. @ 19:20 concentrate hose burst under rectifier, blowing all power out. Line was repaired & power restored to trailer but not to main pump. Operator contacted Lyle Dambruch @ 21:00
	02:00/207¼	11824	52/700	650/700	640/700	540/560	36.1	35.0	7.90	38.0	1.0052	2.5	14.0	7.06	
	03:00/208¼	11852	52/700	650/700	640/700	530/550	36.1	35.0	7.90	38.0	1.0042	2.5	14.3	6.99	
	04:00/209¼	11878	50/700	650/700	640/700	530/550	37.2	35.0	7.80	37.0	1.0050	2.5	16.7	8.45	
	05:00/210¼	11906	50/700	650/700	640/700	530/550	35.6	35.0	7.80	38.0	1.0020	2.5	15.7	7.85	
	06:00/211¼	11935	50/700	650/700	640/700	520/540	36.1	35.0	8.00	38.0	1.0032	2.5	15.1	7.49	
	07:00/212¼	11962	49/700	650/700	640/700	540/560	36.7	35.0	8.00	38.0	1.0041	2.5	14.7	7.27	
	10:00/213	12002	52/700	620/680	650/700	550/560	38.9	35.0	7.50	40.0	1.0020	3.5	18.7	9.0	
	11:00/214	12039	54/700	640/705	650/695	570/590	35.6	35.0	7.50	36.5	1.0050	3.5	16.8	7.9	
	12:00/215	12065	54/730	655/725	660/715	575/590	38.3	35.0	7.40	37.0	1.0050	3.5	17.1	8.1	
	13:00/216	12092	54/730	655/725	660/715	590/610	36.7	35.0	7.40	40.0	1.0050	3.0	17.2	8.4	
	14:00/217	12123	55/700	635/705	650/700	560/580	36.1	35.0	7.30	--	--	--	--	7.5	
	15:00/218	12159	56/700	630/700	645/695	545/565	37.2	36.0	7.40	39.0	1.0040	3.2	14.9	6.93	
	16:00/219	12183	54/690	630/690	640/690	560/580	37.2	34.5	7.40	37.0	0.999	3.6	17.4	8.21	
	17:00/220	12207	54/710	645/705	650/700	560/580	37.2	35.0	7.35	40.0	1.0027	3.5	17.4	8.29	
	18:00/221	12235	53/700	650/705	650/700	560/580	36.7	35.0	7.40	37.5	1.0040	3.6	16.9	7.88	
	19:00/222	12264	52/710	660/710	650/710	570/590	36.1	35.5	7.60	38.5	1.0040	3.4	16.6	7.85	
															Pressure pulse with air consists of shuting down trailer, turning off outside pump, opening heat exchanger and draining, then forcing compressed air through heat exchanger to empty. Process takes 5-6 minutes
															Flush with 100 gal of water is done the same way. After heat exchanger is emptied, 100 gal of water is pumped through system without pressure. Process takes 15-17 minutes
11/17/75	Repaired rectifier panel reinstalled and checked out by Ehrinberg of Werner Electric. Operation satisfactory														
11/18/75	Feed tank empty — we had to fill ourselves — most feed lines disconnected or air locked — feed @ 41°C														
	10:00/232	12275	40/705	710/650	640/705	550/570	41	34	8.5	36	1.0085	9.0	34	14.85	Operating @ 41°C, no heat exchanger. Grab samples I-F-1, I-C-1, I-P-1 taken 11:00. Grab samples I-F-2, I-C-2, I-P-2 taken 12:00. Internal samples A-C-MG, A-C-"A", A-C-"B", and A-C-"C" taken 12:20. Internal samples A-P-MG, A-P-"A", A-P-"B", & A-P-"C" taken 12:30. Grab samples I-F-3, I-P-3, I-C-3 taken 13:00. Main pump temp. still high. Grab samples I-F-4, I-P-4, I-C-4 taken 14:00. Grab samples I-F-5, I-P-5, I-C-5 taken 15:00. Started flushing with fresh water after samples taken 15:25. Started BIZ wash up. Ran 300 gal into system. Permitted soaking overnight. Shut down 15:35
	10:30/232½	12292	40/685	685/640	630/690	525/550	41	33	7.85	41.5	1.006	11.3	33	12.90	
	11:30/233½	12326	41/705	715/665	650/710	500/525	41	36	7.7	42	1.005	13.9	36	13.1	
	12:30/234½	12363	41/700	700/645	630/685	530/550	41	36.5	7.68	42.7	1.004	14.1	36.5	13.3	
	13:30/235½	12394	42/700	700/650	630/695	540/560	41	35.5	7.45	42.7	1.0035	14.7	35.5	12.4	
	14:30/236½	12427	42/700	700/650	630/695	540/560	41	35.5	7.35	42.5	1.0033	15.0	35.5	12.2	

(continued)

TABLE C-1 (continued)

Date	Time/ operating hours	Energy used, kwh	Suction/discharge pressure, psi: Main pump	Pump A	Pump B	Pump C	Feed from main pump: Temp., °C	Flow, gpm	pH	Concentrate: Temp., °C	Sp.gr.	Flow, gpm	Trailer feed, gpm	Flux rate, gfd	Remarks
11/19/75	07:05/236½	12452													Start up
	08:00/237½	12479	43/705	700/650	650/695	530/550	37	35.0	7.8	33	1.007	11.6	35.0	13.9	07:05 started rinsing with fresh
	09:00/238½	12501	43/710	700/650	635/690	520/540	37	36.0	8.1	32	1.0062	12.6	36.0	13.9	water, started feed @ 07:25. pH
	10:15/239¾	12537	43/710	710/660	650/710	560/580	36	34.5	7.1	34.5	1.0052	11.2	34.5	13.8	had risen to 8.3 due to boiling
	11:00/240½	12563	42/710	705/655	645/705	540/560	36	36.5	7.0	37	1.0045	13.9	36.5	13.4	off of chlorine by air agitation
	12:00/241½	12596	42/700	700/650	640/700	525/540	36	36.5	7.1	38	1.0033	15.1	36.5	12.7	to help cool tower. During night
	13:00/242½	12631	41/710	705/660	640/700	525/540	36	36.0	7.05	38.5	1.0030	15.3	36.0	12.3	added bleach wash water to lower
	14:30/243¼	12656	41/720	710/660	650/710	545/560	36	36.0	7.0	36.0	1.004	13.9	36.0	13.1	pH. Temp. of process liquor at
	15:00/243¾	12671	41/710	700/650	640/695	530/550	36	35.5	6.9	38.0	1.0032	14.1	35.5	12.7	start up 98°F. Grab samples II-F-1, II-P-I, II-C-1 taken 08:05.

Concentrate storage tower began overflowing 9:00. Grab samples II-P-2, II-F-2, II-C-2 taken 09:05. Internal samples B-C-"MG", B-C-"A", B-C-"B", B-C-"C" taken 09:15; internal samples B-P-"MG", B-P-"A", B-P-"B", & B-P-"C" taken 09:25. 09:55 — concentrate hose beneath rectifier came loose and caused shutdown, no damage, hose replaced, operation resumed 10:10. Grab sample II-P-3 taken 09:50. Grab samples II-C-3 and II-F-3 taken 10:10. Operation smoothly, no damage as result of hose disconnect. Stopped chlorine addition @ 09:30, pH 7.3. Grab samples II-P-5, II-F-5, II-C-5 taken 12:00. Grab samples II-P-6, II-F-6, II-C-6 taken 13:00. Shutdown 13:30 because concentrate hose appeared to be slipping off, cause was a fork lift, was running across concentrate line between towers, thus causing pressure build up, operator feels that this or something similar may have happened to hose when rectifier was damaged, very probable cause. Grab samples II-P-7, II-F-7, II-C-7 taken 14:30. Grab samples II-P-8, II-F-8, and II-C-8 taken 15:00. Shutdown feed liquor 15:05. Started fresh water flush. Started BIZ wash at 15:20 with 300 gal BIZ solution. Let soak over night. Stopped 15:35.

A piece of hose removed for inspection at Appleton, same appearance as original blown out section

11/20/75 Ran system to obtain data for rotometer flow characteristics

08:30 Tanker came to be loaded, had to use trailer feed pump to fill tanker
10:00 Shut down operation; BIZ washed; Versene washed; fresh water rinse; added 55 gal menthanol to trailer using MG pump as circulator
06:30 Trailer scheduled for return to Appleton

TABLE C-2. DAILY ANALYTICAL DATA

R.O. Operation With Recycling at Continental Group, Augusta, GA

Sample	Date, 1975	Sp.gr., 35°C	pH	Total solids g/l	Total solids Rej. ratio*	COD, mg/l	Soluble calcium mg/l	Soluble calcium Rej. ratio*	Sodium mg/l	Sodium Rej. ratio*	Inorganic chloride mg/l	Inorganic chloride Rej. ratio*	BOD_5 mg/l	BOD_5 Rej. ratio	Color mg/l	Color Rej. ratio*	Viscosity, centipoise	Osmotic pressure, psi
101 Feed	9/24	0.998	7.00	4.95		1312	28.4		1580		1941		225		1640		0.7435	48.3
Recycle		1.001	7.23	11.21		3460	46.0		3516		3778		516		4100		0.7412	104
Perm.		0.995	6.61	1.82	0.63	181	1.6	0.94	650	0.59	919	0.53	70	0.69	56	0.97	--	--
Conc.		1.008	7.38	20.15		6335	65.1		5970		6433		--		--		0.7576	151
102 Feed	9/25	0.998	7.15	4.95		1329	25.2		1610		1894		214		1450		0.7354	48.8
Recycle		1.001	7.35	9.08		2578	35.7		2852		3252		395		3150		0.7390	89
Perm.		0.995	6.80	1.61	0.66	145	1.8	0.93	640	0.60	869	0.54	61	0.71	23	0.98	--	--
Conc.		1.009	7.48	20.86		6202	62.4		6460		6910		--		--		0.7578	168
103 Feed	9/29	0.998	7.32	4.75		1164	23.0		1520		1910		177		945		0.7485	45.3
Recycle		0.999	7.39	6.80		1911	27.0		2120		2740		281		1680		0.7482	62
Perm.		0.995	6.92	0.90	0.81	89	Trace	0.99	325	0.79	49	0.97	34	0.81	0	1.00	--	--
Conc.		1.004	7.30	13.84		4093	42.1		4150		4906		--		--		0.7503	134
104 Feed	9/30	0.997	7.00	3.61		844	22.2		1154		1352		162		665		0.7378	34.6
Perm.		0.995	6.51	1.38	0.64	95	Trace	0.99	472	0.59	667	0.51	45	0.72	15	0.98	--	--
Conc.		1.003	7.23	13.84		3905	41.1		3560		4794		--		--		0.7613	130
105 Feed	10/01	0.997	6.73	4.05		1034	21.7		1324		1647		172		610		0.7354	37.3
Recycle		1.000	7.78	8.61		2606	35.0		2672		2947		414		2620		0.7342	78
Perm.		0.995	7.15	1.46	0.64	124	3.1	0.86	566	0.57	519	0.68	57	0.67	8	0.99	--	--
Conc.		1.004	7.65	14.89		4873	53.7		4710		4891		--		--		0.7542	130
106 Feed	10/02	0.997	6.67	4.37		1235	22.2		1424		1735		209		505		0.7260	43.8
Recycle		1.001	6.86	10.70		2972	36.1		3320		4044		491		1680		0.7425	84.6
Perm.		0.995	6.20	1.32	0.70	113	1.7	0.92	519	0.64	714	0.59	60	0.71	0	1.00	--	--
Conc.		1.006	7.03	16.99		4757	55.4		5450		6253		--		--		0.7483	143
107 Feed	10/03	0.997	6.71	4.52		1170	22.6		1460		1808		168		1000		0.7354	45.0
Recycle		1.001	6.83	9.56		2606	35.4		3004		3710		415		1850		0.7389	74.0
Perm.		0.995	6.22	1.36	0.70	118	2.3	0.90	541	0.63	751	0.58	50	0.70	8	0.99	--	--
Conc.		1.005	6.88	17.05		4786	55.9		5450		6418		--		--		0.7542	143
108 Feed	10/06	Same as #107		--		--	--		--		--		--		--		--	--
Recycle		1.001	6.40	10.13		2781	32.8		3268		3864		382		2440		0.7354	78.5
Perm.		0.995	5.97	1.46	0.68	117	Trace	0.99	569	0.61	807	0.55	60	0.64	0	1.00	--	--
Conc.		1.005	6.60	17.04		4814	50.8		5520		6399		--		--		0.7472	140
109 Feed	10/07	Same as #107				--	--		--		--		--		--		--	--
Recycle		1.001	6.83	10.32		3011	36.9		3276		3937		427		2300		0.7448	78.5
Perm.		0.995	6.43	1.59	0.65	118	1.9	0.92	637	0.56	869	0.52	55	0.67	0	1.00	--	--
Conc.		1.006	6.89	18.73		5370	57.1		5980		6874		--		--		0.7519	156
110 Feed	10/08	0.997	7.16	4.65		1194	24.3		1554		1886		188		1020		0.7331	48.3
Recycle		1.000	7.50	9.53		2708	35.0		3032		3481		407		2020		0.7389	95.2
Perm.		0.996	6.90	1.58	0.66	18	3.1	0.87	622	0.60	848	0.55	57	0.70	8	0.99	--	--
Conc.		1.004	7.40	15.59		4689	49.7		4790		5524		--		--		0.7483	137

(continued)

TABLE C-2 (continued)

Sample	Date, 1975	Sp.gr., 35°C	pH	Total solids g/l	Total solids Rej. ratio*	COD, mg/l	Soluble calcium mg/l	Soluble calcium Rej. ratio*	Sodium mg/l	Sodium Rej. ratio*	Inorganic chloride mg/l	Inorganic chloride Rej. ratio*	BOD_5 mg/l	BOD_5 Rej. ratio	Color mg/l	Color Rej. ratio*	Viscosity, centipoise	Osmotic pressure, psi
111 Feed	10/09	0.997	7.42	4.89		1205	25.1		1580		1954		271		750		0.7307	48.6
Recycle		1.001	7.42	9.44		2536	32.7		2912		3504		368		1760		0.7401	86.8
Perm.		0.995	7.07	1.48	0.70	106	3.4	0.86	579	0.63	769	0.61	56	0.79	10	0.99	--	--
Conc.		1.001	7.39	8.86		3608	33.2		2680		3291		--		--		0.7471	82.6
112 Feed	10/10	Same as #111				--	--		--		--		--		--		--	--
Recycle		1.000	7.53	8.13		2092	30.5		2636		3069		273		1500		0.7389	74.8
Perm.		0.995	7.37	1.53	0.69	106	2.1	0.92	532	0.66	754	0.61	52	0.81	15	0.99	--	--
Conc.		1.003	7.45	13.39		3608	42.4		4230		4830		--		--		0.7483	122
113 Feed	10/11	0.997	7.08	3.93		960	21.2		1274		1615		193		590		0.7272	37.6
Recycle		0.998	7.50	6.66		1823	28.4		2140		2501		266		1180		0.7413	63.0
Perm.		0.995	7.53	1.13	0.71	84	Trace	0.99	400	0.69	569	0.65	39	0.80	0	1.00	--	--
Conc.		1.001	7.30	10.64		2764	36.1		3060		3762		--		--		0.7495	95.2
114 Feed	10/12	0.997	7.15	4.69		1121	24.8		1456		1860		220		940		0.7378	44.2
Recycle		1.000	7.50	8.86		2370	34.1		2748		3241		406		1950		0.7401	81.2
Perm.		0.995	7.35	1.29	0.72	32	2.4	0.90	494	0.66	696	0.62	56	0.74	22	0.98	--	--
Conc.		1.002	7.40	12.46		3330	42.6		3800		4560		--		--		0.7425	115
115 Feed	10/13	0.997	7.59	4.28		1074	22.2		1392		1707		233		1350		0.7307	41.8
Recycle		1.000	7.32	8.77		2287	30.0		2700		3247		390		2900		0.7441	78.4
Perm.		No sample																--
Conc.		1.002	7.33	11.74		3137	34.7		3600		4224		604		3700		0.7448	109
116 Feed	10/14	0.998	7.83	4.31		1021	21.9		1390		1683		221		1275		0.7272	41.0
Recycle		1.000	7.95	8.43		2363	28.8		2684		3074		394		2020		0.7401	79.8
Perm.		0.995	7.62	1.20	0.72	88	Trace	0.99	461	0.67	596	0.64	58	0.74	0	1.00	--	--
Conc.		1.002	7.58	11.58		3251	34.5		3420		4223		--		--		0.7542	99.4
Average (omitting #115)																		
Feed		0.997	7.07	4.51		1142	23.5		1455		1790		202		943		0.7346	44.0
Recycle		1.000	7.29	9.10		2558	33.9		2870		3367		388		2160		0.7402	80.5
Perm.		0.995	6.84	1.41	0.69	102	1.7	0.93	534	0.63	693	0.61	54	0.73	11	0.99	--	--
Conc.		1.004	7.26	15.06		4426	48.1		4615		5338		--		--		0.7515	128

*Rejection ratio = 1 - (concentration permeate/concentration feed).

TABLE C-3. ANALYTICAL DATA

Straight Through R.O. Operation at Continental Group, Augusta, GA

Sample	Date	Time	Sp. gr., 35°C	pH	Total Solids mg/l	Total Solids Rej. ratio*	COD mg/l	Soluble calcium mg/l	Soluble calcium Rej. ratio*	Sodium mg/l	Sodium Rej. ratio*	Inorganic chloride mg/l	Inorganic chloride Rej. ratio*	BOD_5 mg/l	BOD_5 Rej. ratio*	Color mg/l	Color Rej. ratio*	Viscosity, cp.	Osmotic pressure, psi
117 Feed	11/18	11:00	0.998	7.65	6.34		1482	20.8		1900		2538		298		1096		0.7336	72
Perm			0.995	7.00	1.38	0.78	112	Trace	0.99	482	0.76	688	0.73	52	0.82	11	.99	—	—
Conc			1.008	7.90	18.8		5100	39.7		5620		6686		--		--		0.7484	183
118 Feed	11/18	12:00	0.999	7.52	6.40		1446	19.9		2075		2488		293		664		0.7386	87
Perm			0.995	6.95	1.30	0.80	112	Trace	0.99	456	0.78	670	0.73	64	0.78	0	1.00	—	--
Conc			1.007	7.59	17.0		4354	36.0		5190		6196		--		--		0.7558	170
119 Feed	11/18	13:00	1.001	7.65	6.50		1471	19.5		2115		2262		284		614		0.7311	86
Perm			0.995	6.84	1.31	0.80	116	Trace	0.99	461	0.78	662	0.71	64	0.77	0	1.00	—	--
Conc			1.006	7.52	15.7		3882	32.5		4920		5876		--		--		0.7422	155
120 Feed	11/18	14:00	0.998	7.59	6.29		1429	19.5		2015		2480		299		636		0.7348	78
Perm			0.995	6.97	1.25	0.80	116	Trace	0.99	427	0.79	589	0.76	63	0.79	0	1.00	—	--
Conc			1.005	7.55	14.8		3732	30.8		4630		5357		--		—		0.7447	138
121 Feed	11/18	15:00	0.998	7.62	5.93		1113	18.9		1860		2275		278		642		0.7348	72
Perm			0.995	7.03	1.14	0.81	105	Trace	0.99	382	0.79	563	0.75	55	0.80	0	1.00	—	--
Conc			1.004	7.60	13.8		3496	29.6		4230		5112		--		--		0.7447	138
122 Feed	11/19	8:05	0.998	7.50	5.53		1239	16.0		1680		2204		246		888		0.7336	73
Perm			0.995	7.22	1.06	0.81	97	Trace	0.99	369	0.78	565	0.74	50	0.80	0	1.00	—	--
Conc			1.005	7.88	14.3		3496	29.8		4350		5347		--		--		0.7497	133
123 Feed	11/19	9:05	0.997	7.75	5.54		1274	16.3		1665		2196		255		888		0.7336	69
Perm			0.995	7.06	1.10	0.80	100	Trace	0.99	390	0.76	574	0.74	54	0.79	0	1.00	--	--
Conc			1.005	7.86	15.1		4664	30.0		4590		6170		--		--		0.7422	147
124 Feed	11/19	10:10	0.998	7.15	5.30		1204	15.6		1630		2204		232		888		0.7336	54
Perm			0.995	6.45	1.00	0.80	98	Trace	0.99	348	0.79	494	0.78	54	0.77	0	1.00	--	--
Conc			1.000	7.14	7.93		1858	18.0		2280		3017		--		—		0.7361	68
125 Feed	11/19	11:00	0.998	7.02	5.21		1204	14.3		1615		2160		244		888		0.7324	54
Perm			0.995	6.36	0.99	0.81	99	Trace	0.99	335	0.79	488	0.77	52	0.79	0	1.00	—	--
Conc			1.004	7.23	13.0		3177	26.3		3940		5042		--		--		0.7422	122
126 Feed	11/19	12:00	0.998	7.06	5.21		1193	14.3		1595		2170		223		888		0.7336	54
Perm			0.995	6.20	0.92	0.82	98	Trace	0.99	312	0.80	470	0.78	53	0.76	0	1.00	--	--
Conc			1.003	7.20	12.1		2850	23.8		3630		4360		--		--		0.7422	116
127 Feed	11/19	13:00	0.997	7.05	5.24		1168	14.4		1575		2173		223		888		0.7287	58
Perm			0.995	5.99	0.88	0.83	96	Trace	0.99	304	0.81	468	0.78	57	0.74	0	1.00	--	--
Conc			1.003	7.19	11.7		2850	22.9		3460		4554		--		--		0.7361	161
128 Feed	11/19	14:30	0.998	6.94	5.29		1200	14.3		1610		2176		220		912		0.7336	54
Perm			0.995	6.63	0.98	0.81	96	Trace	0.99	323	0.80	482	0.78	52	0.76	0	1.00	—	—
Conc			1.002	6.99	11.5		3010	22.9		3340		4320		--		--		0.7422	121
129 Feed	11/19	15:00	0.998	7.03	5.30		1221	14.7		1695		2157		228		888		0.7311	58
Perm			0.995	6.36	0.96	0.82	88	Trace	0.99	339	0.80	489	0.77	52	0.77	0	1.00	—	—
Conc			1.003	7.20	12.3		3035	27.8		3380		4766		--		--		0.7441	121
Average																			
Feed			0.998	7.35	5.70		1280	16.8		1778		2268		256		829		0.7333	67
Perm			0.995	6.77	1.10	0.81	102	Trace	0.99	379	0.79	554	0.76	56	0.78	0	1.00	—	--
Conc			1.004	7.45	13.7		3500	28.5		3322		5139		--		—		0.7446	137

*Rejection ratio = 1 - (Concentration of permeate/concentration of feed).

TABLE C-4. ANALYTICAL DATA

Grab Samples Collected for Evaluating Interval Performance of RO System
Continental Can Corporation — CEH Bleach Effluent

Sample No.	Sample[a]	Date	Time	Sp.gr. 35°C	pH	Total solids g/l	Total solids Rej. ratio	COD mg/l	COD Rej. ratio	Sodium mg/l	Sodium Rej. ratio	Soluble calcium mg/l	Soluble calcium Rej. ratio	Inorganic chloride mg/l	Inorganic chloride Rej. ratio	BOD_5 mg/l	BOD_5 Rej. ratio	Color mg/l	Color Rej. ratio	Viscosity[†] cp. 35°C	Osmotic[†] pressure psi	Suspended solids mg/l
113	MF	10/11/75	2:00 PM	0.998	7.50	6.66		1823		2140		28.4		2501		266		1180		0.741	63	62
"	MP	"	"	0.995	7.04	0.83	0.88	86	0.95	318	0.85	Trace	0.99	463	0.81	34	0.87	0	1.00	—	—	—
"	MC	"	"	1.001	7.44	9.69		2794		3065		33.1		3522						0.746	87	137
"	AP	"	"	0.994	6.89	1.17	0.88	118	0.96	442	0.86	1.0	0.97	598	0.83	42		10	—	—	—	—
"	AC	"	"	1.002	7.38	11.53		3273		3544		38.7		4053				—		0.747	102	135
"	BP	"	"	0.995	7.05	1.76	0.85	144	0.96	677	0.81	3.1	0.92	930	0.77	55		22	—	—	—	—
"	BC	"	"	1.003	7.34	13.02		3713		4190		42.6		4640				—		0.752	112	156
"	CP	"	"	0.995	6.88	2.15	0.83	113	0.97	800	0.81	4.0	0.91	1166	0.75	53		0	1.00	—	—	—
"	CC	"	"	1.004	7.44	14.37		4086		4480		44.8		5078				—		0.754	126	194
116	MF	10/14/75	3:00 PM	1.000	7.95	8.43		2363		2684		28.8		3074		394		2020		0.740	80	81
"	MP	"	"	0.995	7.88	0.91	0.89	82	0.97	350	0.87	Trace	0.99	480	0.84	57	0.86	0	1.00	—	—	—
"	MC	"	"	1.002	7.71	10.33		2873		3240		34.4		3802				—		0.746	92	101
"	AP	"	"	0.995	7.51	1.11	0.89	94	0.97	420	0.87	1.2	0.97	578	0.85	68		8	—	—	—	—
"	AC	"	"	1.002	7.50	11.46		3326		3424		35.9		4106				—		0.737	101	112
"	BP	"	"	0.995	7.22	1.60	0.86	116	0.97	603	0.82	1.4	0.96	830	0.80	77		10	—	—	—	—
"	BC	"	"	1.003	7.59	12.44		3563		3810		37.0		4515				—		0.745	120	135
"	CP	"	"	0.995	7.12	1.69	0.86	92	0.97	653	0.83	1.6	0.96	949	0.79	73		0	1.00	—	—	—
"	CC	"	"	1.004	7.55	13.84		4101		4150		40.0		4878				—		0.746	123	111
118	MF	11/18/75	12:00 PM	0.999	7.52	6.40		1446		2075		19.9		2488		293		664		0.739	87	
"	MP	"	12:30 PM	0.995	6.63	0.78	0.88	87	0.94	284	0.86	Trace	0.99	391	0.84	57	0.81	0	1.00	—		
"	MC	"	12:20 PM	1.000	7.45	8.59		1910		2720		19.5		3270						0.740	87	
"	AP	"	12:30 PM	0.995	6.74	1.16	0.86	94	0.95	421	0.85	Trace	0.99	590	0.82	56		0		—	—	
"	AC	"	12:20 PM	1.001	6.49	11.29		2832		3480		22.4		4231						0.736	112	
"	BP	"	12:30 PM	0.995	6.80	0.96	0.91	96	0.97	350	0.90	Trace	0.99	497	0.88	52		0		—	—	
"	BC	"	12:20 PM	1.004	6.92	13.77		3628		4580		27.9		5310						0.748	136	
"	CP	"	12:30 PM	0.996	7.05	2.64	0.81	112	0.97	805	0.82	Trace	0.99	1329	0.75	79		0		—	—	
"	CC	"	12:20 PM	1.005	7.60	16.10		3944		5790		32.8		5899						0.747	154	
123	MF	11/19/75	9:05 AM	0.997	7.75	5.54		1274		1665		16.3		2196		255		888		0.734	69	
"	MP	"	9:25 AM	0.995	6.64	0.68	0.88	73	0.94	240	0.86	Trace	0.99	339	0.85	52	0.80	0	1.00	0.735	—	
"	MC	"	9:15 AM	1.000	8.04	6.85		1455		2250		16.3		2733		--		--		—	73	
"	AP	"	9:25 AM	0.996	6.88	1.87	0.73	100	0.93	579	0.74	Trace	0.99	873	0.68	64		0		0.737	—	
"	AC	"	9:15 AM	1.000	7.55	8.85		1998		2900		19.0		3515		--		--		—	90	
"	BP	"	9:25 AM	0.995	6.95	1.44	0.84	99	0.95	464	0.84	Trace	0.99	764	0.78	58		0		0.740	—	
"	BC	"	9:15 AM	1.001	7.72	11.51		2699		3140		24.2		4508		—		--		—	116	
"	CP	"	9:25 AM	0.996	6.64	1.64	0.86	99	0.96	558	0.82	Trace	0.99	882	0.80	66		0		0.744	—	
"	CC	"	9:15 AM	1.002	7.50	9.20		2191		2480		19.2		3476		--		--		—	92	

[a] MF = Feed to banks fed by Manton Gaulin pump.
MP = Permeate from banks fed by Manton Gaulin pump.
MC = Concentrate from banks fed by Manton Gaulin pump.
AP = Permeate from banks fed by Pump A.
AC = Concentrate from banks fed by Pump A.
BP = Permeate from banks fed by Pulp B.
BC = Concentrate from banks fed by Pump B.
CP = Permeate from banks fed by Pump C.
CC = Concentrate from banks fed by Pump C.

[†] Osmotic pressure of feed to system, see No. 113 and 116.

[†] Viscosity of feed to system, see No. 113 an...

TABLE C-5. ADVANCED R.O. CONCENTRATION RUNS IN APPLETON

Experiment 76-15 — Project 3263 — Continental Can Co., Augusta, GA

Advanced R.O. concentration of kraft bleach preconcentrate

Set up: (3) 520 UOP modules on double loop with (2) new units and (1) used unit on separate feed of Milton Roy pump feed varied as indicated
(2) 620 UOP modules on single loop of second side of Milton Roy pump with pumping feed rates as indicated. 620 modules equipped with V.D.R.

Date	Time	Temp., °C	Feed rate, gpm	Pressure 520	Pressure 620	Permeate rate (2)520, cc/min	Permeate rate (1)520, cc/min	Permeate rate (2)620, cc/min	Flux rate (2)520, gfd	Flux rate (1)520, gfd	Flux rate (2)620, gfd	Dissolved solids Feed, g/l	Dissolved solids Conc., g/l	Dissolved solids Perm., g/l	Osmotic pressure, psi	Permeate collected, gal	Chlorides in perm., mg/l
Run #1 — 384 gal of 1% preconcentrate to be concentrated into 192 gal of 2% concentrate																	
2/13/76	8:35	39	3	620/440	610/350	1710	400	810	19.15	8.96	9.07	11.06					
	10:00	42	3	590/420	590/330	1640	390	790	18.37	8.74	8.85		12.7	0.61	125	50	361
	11:10	39.5	3	590/420	590/340	1580	410	780	17.70	9.18	8.74		14.79	0.73		100	384
	12:30	39.5	3	580/395	575/325	1300	315	660	14.56	7.06	7.39		17.49	0.92		150	482
	13:45	39.5	3	580/395	575/325	1280	300	600	14.34	6.72	6.72		20.55	1.26		193	—
	End of run — 173 gal concentrate collected																
Run #2 -- 500 gal of 1% preconcentrate																	
2/16/76	9:00	38	3	590/425	610/340	1570	350	780	17.58	7.84	8.74	11.57					
	10:20	38	3	610/450	610/310	1540	395	680	17.25	8.85	7.61		13.42*	0.71*		65	364
	12:00	38	3	620/450	620/320	1470	375	635	16.46	8.40	7.11		15.39*	0.82	152	130	405
	13:50	38	3	610/430	630/320	1330	330	580	14.90	7.39	6.50		18.25*	1.02*		195	487
	15:30	38	3	620/440	630/310	1200	290	490	13.44	6.50	5.49		21.62*	1.22*		250	672
	End of run — 182 gal concentrate collected																
Run #3 — 500 gal of 1% preconcentrate																	
2/17/76	8:30	32	3	620/450	620/300	1445	380	630	16.18	8.51	7.06	11.90					
	10:10	38	3	590/420	620/300	1410	370	660	15.79	8.28	7.39		13.42*	0.71*		65	371
	11:15	38	3	620/450	620/290	1425	385	550	15.96	8.62	6.16		15.39*	0.82*		130	447
	13:40	38	3	620/450	630/300	1300	335	530	14.56	7.50	5.94		18.25*	1.02*		165	559
	15:25	38	3	620/450	630/290	1180	305	430	13.21	6.83	4.81		21.62*	1.22*		250	687
	End of run — 227 gal concentrate collected																
Run #4 — 500 gal of 1% preconcentrate																	
2/18/76	8:10	31	3	600/440	620/280	1420	370	630	15.90	8.29	7.06	11.82					
	9:55	38	3	610/440	625/290	1460	390	605	16.35	8.74	6.78		13.37†	0.71†		65	372
	11:20	Feed to 620 reduced to 2.25 gpm															
	11:40	38	3 & 2.25	620/460	630/430	1450	395	820	16.24	8.84	9.18		15.33†	0.84†		130	463
	13:25	38	3 & 2.25	620/460	620/400	1330	350	680	14.90	7.84	7.61		18.41†	0.92†	186	19-	480
	15:00	38	3 & 2.25	620/450	620/450	1160	315	700	12.99	7.06	7.84		21.67†	1.19†		250	655
	End of run — 226 gal of concentrate collected																
Run #5 — 500 gal of 1% preconcentrate																	
2/19/76	8:10	41	3 & 2.25	610/450	600/440	1640	440	1025	18.37	9.86	11.48	12.37					
	9:40	39	3 & 2.25	620/450	620/420	1520	410	890	17.02	9.18	9.41		13.37†	0.71†		65	367
	11:15	39	3 & 2.25	620/450	620/400	1420	390	740	15.90	8.74	8.29		15.33†	0.84†		130	420
	13:00	39	3 & 2.25	620/460	620/400	1280	355	700	14.34	7.95	7.84		18.41†	0.92†	186	195	490
	14:38	39	3 & 2.25	620/440	620/390	1220	320	600	13.67	7.17	6.72		21.67†	1.19†		250	674
	End of run — 226 gal of concentrate collected																

Accumulated wash water Run #1 to Run #5: 353.5 lb with sp.gr. of 1.001 = 42.39 gal or 160.43 liters
Dissolved solids in wash water = 5.93 g/liters

(continued)

TABLE C-5 (continued)

Experiment 76-15 — Project 3263 — Continental Can Co., Augusta, GA

Advanced R.O. concentration of kraft bleach preconcentrate
Set up: (3) 520 UOP modules on double loop with (2) new units and (1) used unit on separate feed of Milton Roy pump feed varied as indicated
(2) 620 UOP modules on single loop of second side of Milton Roy pump with pumping feed rates as indicated. 620 modules equipped with V.D.R.

Date	Time	Temp., °C	Feed rate, gpm	Pressure 520	Pressure 620	Permeate rate, cc/min (2)520	Permeate rate, cc/min (1)520	Permeate rate, cc/min (2)620	Flux rate, gfd (2)520	Flux rate, gfd (1)520	Flux rate, gfd (2)620	Dissolved solids Feed, g/l	Dissolved solids Conc., g/l	Dissolved solids Perm., g/l	Osmotic pressure, psi	Permeate collected, gal	Chlorides in perm., mg/l
Run #6 — 350 gal of 2% preconcentrate																	
2/20/76	8:15	38	3 & 2.25	600/440	620/410	1195	315	620	13.38	7.06	6.94	20.84					
	10:20	38	3 & 2.25	620/450	600/360	1040	280	435	11.65	6.27	4.87		25.41	1.66		60	888
	11:40		2.25 & 1.50	Reduced feed pressures													
	12:40	37	2.25 & 1.50	610/500	600/490	850	260	520	9.52	5.82	5.82		32.60	2.35		120	1180
	15:15	37	2.25 & 1.50	620/510	620/490	635	190	390	7.11	4.26	4.37		37.07	3.11		175	1666
	End of run — 162.5 gal of concentrate collected																
Run #7 — 350 gal of 2% preconcentrate																	
2/23/76	8:25	32	2.25 & 1.50	600/520	630/500	1080	320	610	12.09	7.17	6.83	22.03					
	10:25	38	2.25 & 1.50	600/510	630/490	880	245	595	9.86	5.49	6.66		25.79	1.99	262	60	986
	13:00	38	2.25 & 1.50	600/510	610/470	670	240	415	7.50	5.38	4.64		31.85	2.55	316	120	1427
	16:20	36	2.25 & 1.50	610/520	620/470	500	170	305	5.6	3.81	3.41		40.79	4.00		175	2057
	End of run — 162 gal concentrate collected																
Run #8 — 450 gal of 2% preconcentrate — remainder of Runs #1-5																	
2/24/76	8:30	26	3.00 & 1.50	620/500	600/440	915	230	545	10.75	5.15	6.10	21.92					
	11:05	39	3.00 & 1.50	620/440	600/460	740	250	550	8.29	5.60	6.16		25.32	2.19		60	1183
	13:30	39	2.40 & 150	600/475	600/470	640	250	455	7.17	5.60	5.10		34.40	2.48		115	1396
	16:35	37	2.40 & 1.50	610/475	620/480	500	185	365	5.60	4.14	4.04		32.83	3.38		170	1731
	17:00	Shutdown for night															
2/25/76	8:20	39	2.40 & 1.50	610/490	620/470	630	220	400	7.06	4.93	4.48		--	--			
	11:45	39	2.40 & 1.50	610/490	610/470	960	160	285	5.15	3.58	3.19		39.50	4.97		225	2445
	End of run — 225 gal of concentrate collected																
122 Liters of combined wash water with 7.92 g/liters dissolved solids																	
Run #9 — 90 gal of 4% preconcentrate																	
2/26/76	9:30	36	1.50	610/550	610/480	490	190	260	5.49	4.26	2.91	37.05					
	11:35	33	1.50	610/560	610/465	330	130	140	3.70	2.91	1.57		50.43	6.84		25	3116
	14:45	34	1.50	610/520	620/450	175	100	60	1.96	2.24	0.67		65.52	9.12	650	45	4533
	End of run — 45 gal of concentrate collected																

(continued)

TABLE C-5 (continued)

Experiment 76-15 — Project 3263 — Continental Can Co., Augusta, GA

Advanced R.O. concentration of kraft bleach preconcentrate

Set up: (3) 520 UOP modules on double loop with (2) new units and (1) used unit on separate feed of Milton Roy pump feed varied as indicated
(2) 620 UOP modules on single loop of second side of Milton Roy pump with pumping feed rates as indicated. 620 modules equipped with V.D.R.

Date	Time	Temp., °C	Feed rate, gpm	Pressure 520	Pressure 620	Permeate rate (2)520 cc/min	Permeate rate (1)520 cc/min	Permeate rate (2)620 cc/min	Flux rate (2)520 gfd	Flux rate (1)520 gfd	Flux rate (2)620 gfd	Dissolved solids Feed, g/l	Dissolved solids Conc., g/l	Dissolved solids Perm., g/l	Osmotic pressure, psi	Permeate collected, gal	Chlorides in perm., mg/l
Run #10 — 98 gal of 4% preconcentrate																	
3/02/76	8:50	39	1.50	610/550	--	460	175	—	5.15	3.92		39.70					
	9:20	Replaced back pressure regulator on 620 — no control															
	9:33	38			600/395			225			2.52						
	11:20	36	1.50	610/550	620/370	335	130	130	3.75	2.91	1.46		51.06	7.54		25	3919
	14:00	36	1.50	560/485	570/320	185	65	85	2.07	1.46	0.95		63.78	9.92		45	4995
	End of run — 48 gal of concentrate collected																
Run #11 — 90 gal of 4% preconcentrate																	
3/03/76	8:30	38	1.50	610/550	620/375	430	210	195	4.82	4.70	2.18	39.91					
	8:40	Added 15 gal feed															
	9:15	Added 15 gal feed															
	11:50	39	1.50	610/460	620/410	270	90	145	3.02	2.02	1.62		51.52	9.90	505	30	4096
	16:50	37	1.50	610/450	610/370	150	45	75	1.68	1.01	1.84		68.36	11.54		60	6050
	End of run — 60 gal of concentrate collected																
Run #12 — 90 gal of 4% preconcentrate																	
3/04/76	8:25	36	3.00§ & 1.80§	610/380	610/320	365	110	170	4.09	2.46	1.90	40.24					
	9:35	Added 15 gal feed															
	10:30	Added 15 gal feed															
	12:30	39	3.00§ & 1.80§	620/370	630/310	205	70	110	2.30	1.57	1.23		49.99	10.38		30	6002
	Shut down — weather warning																
3/05/76	8:30	37	3.00§ & 1.80§	610/400	600/320	280	75	120	3.14	1.68	1.34						
	13:45	40	3.00§ & 1.80§	600/360	620/280	165	45	85	1.85	1.01	0.95		59.04	13.08		60	7156
	End of run — 60 gal of concentrate collected																
Run #13 — 93 gal of 4% preconcentrate																	
3/08/76	8:30	34	2.25 & 1.50	620/500	610/380	410	150	200	4.59	3.36	2.24	40.21					
	8:35	Added 15 gal feed															
	9:00	Added 22 gal feed (total 130)															
	11:50	39	2.25 & 1.50	600/450	600/345	290	85	120	3.25	1.90	1.34		52.48	9.82		36	5498
	17:00	38	2.25 & 1.50	600/425	600/320	175	50	80	1.96	1.12	0.90		67.98	13.31		63.5	6862
	17:10	End of run — 68.5 gal of concentrate collected															
Collected 50 gal wash water with dissolved solids of 16.57 g/liter																	
Composite concentrate = 67.45 g/liter																	
After wash up with BIZ, water test																	
3/09/76	10:00	39	3.00 & 2.25	600/450	600/275	1600	340	700	17.92	7.62	7.84						

*Composite of Runs #2 and #3.

†Composite of Runs #4 and #5.

§Feed rates as suggested by Dick Walker of UOP.

TABLE D-1. CHESAPEAKE CORPORATION — R.O. FIELD TRIAL

Membrane Concentration of Oxygen Bleach Process Waters
309 ft² — membrane area
(Rev-O-Pak 105 ft² —UOP 204 ft²)

Summary of Operating Data

Date	Time	Feed gpm	Feed pH	Feed Temp., °C	Pressure, psi Rev-O-Pak In	Rev-O-Pak Out	UOP In	UOP Out	Permeate gpm	Permeate gfd (flux)	Permeate Draw off, gpm	Concentrate Draw off, gpm	Concentrate sp.gr.	Concentrate Temp., °C	Gould pump amps	Gould pump gpm	Remarks
									Stage 1, Run 1, Sample 7								
4-15-76	10:00	5.50	4.5	39.0	605	600	600	560	2.75	12.82	2.75	2.70	0.999	35.5	20.0	17.04	Raw feed pH 2.3 (stopped), sp.gr. 0.997
	12:00	5.00	4.3	38.5	605	600	600	560	2.54	11.84	2.54	2.66	0.999	35.5	19.8	16.74	O_2 wash water added, sp.gr. 0.995
	14:00	5.00	4.6	40.5	610	605	605	565	2.38	11.09	2.38	2.37	0.999	35.5	19.8	17.40	Sp.gr. feed 0.995 @ 45°C
	15:45	4.40	4.8	—	605	600	600	555	2.17	10.11	2.17	2.37	0.999	36.0	19.6	17.31	
									Stage 1, Run 2, Sample 8								
4-16-76	8:00	Start up															
	8:30	5.30	6.7	37.0	610	605	605	570	2.69	12.54	2.69	2.75	1.002	33.0	20.0	16.74	
	10:00	5.00	6.8	39.0	610	600	600	560	2.27	10.58	2.37	2.75	1.002	34.5	20.0	16.86	Raw feed, sp.gr. 0.999 @ 32.5°C
	12:00	5.00	6.7	40.5	610	600	600	560	2.04	9.51	2.04	2.75	1.002	36.0	20.0	15.61	
	14:00	5.00	6.8	42.5	605	600	600	560	2.36	11.00	2.36	2.75	1.002	36.0	20.0	17.42	
	16:00	5.00	6.5	39.0	610	605	605	565	1.97	9.18	1.97	2.75	1.000	35.5	19.5	16.25	
	18:00	4.60	6.7	39.0	610	605	605	560	1.92	8.95	1.92	2.75	0.998	35.0	19.0	16.67	
	20:00	4.90	6.7	37.0	610	600	600	560	1.80	8.39	1.80	2.75	0.998	32.5	19.5	16.55	
	22:00	3.64	6.5	38.5	600	590	590	540	1.71	7.97	1.71	1.79	0.998	35.0	19.5	17.10	
4-17-76	24:00	3.56	6.6	38.0	600	595	595	550	1.66	7.74	1.66	1.82	0.998	36.0	20.0	17.43	Shut down 00:35-00:45 to try to
	02:00	3.64	6.5	38.5	605	600	600	560	1.77	8.25	1.77	1.78	0.999	35.5	19.5	17.20	increase flux 1.66 gpm to 3.19 gpm
	04:00	3.22	6.2	38.5	600	600	600	555	1.56	7.27	1.56	1.64	0.999	35.0	19.5	17.48	Down 04:30, replace UDP module
	06:00	3.91	6.2	37.5	600	595	595	550	1.95	9.09	1.95	1.95	1.001	33.0	19.5	17.38	Color in perm., start 05:30
	08:00	End of run															
									Stage 1, Run 3, Sample 9								
4-17-76	08:00	3.91	6.4	39.0	600	590	590	550	1.60	7.46	1.60	1.80	0.999	34.0	20.0	16.03	08:30 shut down for water wash
	09:00	—	—	—	—	—	—	—	2.05	9.55	2.05	2.05	—	—	—	—	
	10:00	3.91	5.8	40.0	600	595	595	550	1.80	8.39	1.80	1.80	0.999	36.0	20.1	17.22	Sp.gr. raw feed 0.995 @ 41°C
	12:00	3.54	4.8	40.5	610	600	600	560	0.98	4.57	0.98	1.20	0.999	35.0	20.3	16.26	Sp.gr. raw feed 0.995 @ 40°C
	14:00	3.00	—	—	605	600	600	555	1.48	6.90	1.48	1.50	0.999	35.5	--	16.61	Press. pulse @ 13:30-2 min
	16:00	2.80	5.5	37.0	600	600	600	550	1.19	5.55	1.19	1.71	1.000	35.5	20.0	16.50	16:10 press. pulse 5 min-18:10 press. pulse
	18:00	2.80	6.1	38.0	600	595	595	550	1.19	5.55	1.19	1.60	1.000	35.0	20.0	16.40	5 min-19:00 press. pulse 5 min
	20:00	2.80	6.3	37.0	600	600	600	555	1.29	6.01	1.29	1.51	1.001	34.0	20.2	16.29	19:00 down, filter full of fiber
	22:15	2.80	6.1	36.0	590	585	585	520	1.24	5.78	1.24	1.45	1.001	33.0	20.2	16.61	Replaced filter, start up 19:05
																	21:15 color in one U of module
																	Cut press. to 580 on UDP
																	Down 22:30, wash up with BIZ
																	pH 7.7, 23:00 rinse with fresh
																	water

(continued)

TABLE D-1 (continued)

Date	Time	Feed gpm	Feed pH	Feed Temp., °C	Pressure, psi Rev-O-Pak In	Rev-O-Pak Out	UOP In	UOP Out	Permeate gpm	Permeate gfd (flux)	Permeate Draw off, gpm	Concentrate Draw off, gpm	Concentrate sp.gr.	Concentrate Temp., °C	Gould pump, amps	Remarks
		Stage 1-A, Run 1, Sample 10														
4-19-76	09:10	Start up														
	10:00	5.57	6.2	34.5	610	600	600	555	2.51	11.70	2.51	3.68	1.002	36.0	19.5	
	10:50	Shut down, no cooling water														
	15:15	Restart														
	15:45	4.90	6.2	35.5	610	605	605	565	1.91	8.90	1.91	3.01	1.002	36.0	19.5	
	17:00	5.50	6.3	35.0	--	--	--	--	1.76	8.20	1.76	3.76	1.002	35.0	19.5	UDP 3 hr 55 min for 5 gal = 1.28 gpm
	18:00	5.70	6.3	36.0	610	605	605	565	1.74	8.11	1.74	3.95	1.002	37.0	19.2	Rev-O-Pak (by diff.) 0.48 gpm
	19:00	5.29	6.4	37.0	610	605	605	570	1.74	8.11	1.74	3.45	1.002	37.0	19.1	
	20:15	5.36	6.5	37.0	610	600	600	560	1.64	7.64	1.64	3.67	1.003	33.0	19.1	UDP — 1.27 gpm
	21:00	5.34	6.5	37.0	610	600	600	560	1.62	7.55	1.62	3.60	1.003	32.5	19.1	UDP — 1.27 gpm
	21:10	End of run, 21:10 start of concentration to 2% solids														
		Stage 1-B, Run 1, Sample 11														
4-19-76	22:00	5.34	6.4	37.0	610	605	605	565	1.59	7.41	1.59	3.65	1.003	33.0	19.0	
	24:00	5.34	6.4	37.5	610	605	605	555	1.55	7.22	1.55	3.67	1.003	32.5	19.1	23:40, color in the same UDP
4-20-76	02:00	5.23	6.4	38.0	605	600	600	555	1.45	6.76	1.45	3.67	1.004	31.5	19.2	Gave nut 1/4 turn
	04:00	5.26	6.5	37.0	602	600	600	555	1.43	6.66	1.43	3.70	1.004	33.0	19.2	1:00, color gone
	05:00							UDP —	1.15	8.12						2:15, pressure pulse 5 min
	05:00						Rev-O-Pak —		0.22	3.02	Conductivity 500					
	06:00	5.17	6.6	37.0	602	600	600	552	1.33	6.20	1.33	3.84	1.005	36.0	19.0	
	06:30							UDP —	1.09	7.69						
	06:30						Rev-O-Pak —		0.23	3.15						
	07:00	4.85	6.6	37.0	605	600	600	555	1.32	6.15	1.32	3.53	1.005	36.0	19.0	
	09:00	4.97	6.4	36.0	610	605	605	555	1.29	6.01	1.29	3.68	1.005	36.0	19.0	
	09:30	Shut down for BIZ wash up									Conductivity 500					Added HCl to pH 7.0 for BIZ
	10:20	Start up														
	11:00	4.75	6.4	36.0	610	605	605	565	1.38	6.43	1.38	3.37	1.006	36.5	19.5	
	11:25							UDP —	1.06	7.48	Sp.gr. liquor in tanker = 1.004 - 14.5 gts/l					Started adding concentrate to truck
	11:25						Rev-O-Pak —		0.27	3.70	Sp.gr. concentrate to tanker = 1.006 = 17.5 gts/l					from Sample 10 run — 1000 gal
	13:00	5.21	6.5	35.5	610	605	605	568	1.26	5.87	1.26	3.95	1.007	35.5	19.3	
	15:15	5.13	6.6	35.5	610	605	605	570	1.18	5.50	1.18	3.95	1.007	35.0	19.5	Tank feed 1.005 @ 35°C, 16 g/l
	16:10	Pressure pulse, 2 min							1.25	5.83						
	17:00	5.00	6.6	35.0	610	607	607	570	1.16	5.41	1.16	3.84	1.008	35.0	19.3	
	19:00	5.08	6.7	37.5	610	602	602	570	1.11	5.17	1.11	3.80	1.009	33.5	19.3	19:30, added the remaining concentrate
	21:00	4.97	6.7	37.0	600	595	595	550	1.09	5.08	1.09	3.82	1.009	33.5	19.5	to truck from Sample 10 run (300 gal)
	23:00	5.12	6.7	37.5	600	595	595	555	1.06	4.94	1.06	3.97	1.010	32.0	19.5	

(continued)

TABLE D-1 (continued)

Date	Time	Feed gpm	Feed pH	Feed Temp., °C	Pressure, psi Rev-O-Pak In	Rev-O-Pak Out	UOP In	UOP Out	Permeate gpm	Permeate gfd (flux)	Permeate Draw off, gpm	Concentrate Draw off, gpm	Concentrate sp.gr.	Concentrate Temp., °C	Gould pump, amps	Remarks
Stage 1-B, Run 2, Sample 12																
4-21-76	01:00	4.51	6.7	39.0	605	600	600	550	1.06	4.94	1.06	3.50	1.010	35.0	19.5	Used different hydrometer for this
	03:00	4.44	6.7	39.0	605	600	600	555	0.97	4.52	0.97	3.52	1.014	33.0	19.5	reading, went from 1.010 at the
	05:00	4.51	6.7	39.0	600	595	595	550	0.92	4.29	0.92	3.43	1.015	33.0	19.5	01:00 reading using the old hydrometer
	07:00	4.32	6.8	35.0	600	595	595	550	0.90	4.19	0.90	3.42	1.015	35.0	19.5	to 1.014 at 03:00 using the higher
	07:15	Shut down for BIZ wash														hydrometer
	08:23	Restart							1.01	4.71	1.01					
	09:00	4.73	6.7	36.0	608	600	600	545	0.97	4.52	0.97	3.76	1.015	38.5	19.3	Sp.gr. tank 1.012 @ 36°C, 26 g/1
	11:00	4.79	6.8	35.0	610	605	605	545	0.84	3.91	0.84	3.95	1.016	34.0	19.5	Sp.gr. tank 1.0135 @ 36°C, 28 g/1
	13:00	4.89	6.9	36.0	610	605	605	550	0.81	3.77	0.81	4.08	1.018	35.0	19.8	Sp.gr. tank 1.0145 @ 34.3°C, 29 g/l
	15:00	4.88	7.0	36.0	610	602	602	545	0.80	3.73	0.80	4.08	1.018	36.0	20.0	Sp.gr. tank 1.015 @ 35.5°C, 29.5 g/l
	17:00	4.83	7.0	36.0	—	—	—	—	0.75	3.50	0.75	4.08	1.020	36.5	20.0	Sp.gr. tank 1.0165 @ 36°C, 32 g/1
	19:00	4.98	7.2	36.0	602	600	600	535	0.69	3.22	0.69	4.30	1.021	34.0	20.0	19:50, sp.gr. tank 1.0185 @ 35°C
	21:00	4.85	7.2	39.0	600	595	595	540	0.62	2.89	0.62	4.20	1.022	35.5	20.3	20:30, sp.gr. tank 1.022 (34.5)
	22:30	4.86	7.2	39.0	605	600	600	540	0.59	2.75	0.59	4.20	1.024	34.0	20.4	22:40, down for wash up
Stage 1-C, Run 1, Sample 14 (concentration of 0.4 to 0.8 – 6000 gal)																
4-23-76	12:45	5.09	5.6	41.0	600	598	598	560	2.34	10.90	2.34	2.75	1.000	35.0	19.5	(Start up 12:30?)
	15:00	6.13	5.3	41.0	605	600	600	555	1.92	8.95	1.92	4.21	1.000	34.5	19.8	
	17:00	5.87	6.4	43.0	605	600	600	558	1.66	7.74	1.66	4.21	1.000	34.5	20.0	
	21:00	—	6.4	38.0	—	—	—	—	1.23	5.73	1.23	—	1.002	32.0	20.1	Sp.gr. of feed 0.999 @ 38°C
	23:30	Unit was shut down														Shut down 3 times, 45 min
	23:45	3.46	6.7	42.0	600	595	595	550	1.36	6.34	1.36	2.10	1.003	32.0	20.0	Plugged screen
4-24-76	10:00	5.16	7.1	37.0	605	600	600	560	1.59	7.41	1.59	3.57	1.002	34.0	19.8	
	12:00	5.12	6.4	39.5	608	600	600	548	1.44	6.71	1.44	3.68	1.001	34.5	19.8	Sample 14-F, P, C, raw feed, R.O.
	12:50	Shut down, lack of feed														unit down about 01:30
	14:15	Restarted after telephone call to Wiley														0700 washing with BIZ (start up? 08:00?)
		Continue Run 2, Sample 15														14-spectral, 1 qt. sample
																Raw feed pH 4.0, sp.gr. 0.995 @ 47°C
Stage 1-C, Run 2, Sample 15																
4-24-76	14:30	4.21	5.6	39.5	600	602	602	560	1.58	7.36	1.58	2.63	1.001	36.5	20.2	
	15:30	Shut down, lack of feed														
	17:30	Start up														
	18:00	5.99	6.4	38.0	605	600	600	560	1.50	6.99	1.50	2.37	1.002	33.6	20.1	
	21:45	Pump down, Sweco plugged, stopped 21:15														
	22:25	Screen cleaned, unit started														
	22:30	5.10	6.2	36.0	605	600	600	555	1.42	6.62	1.42	3.68	1.001	32.0	20.4	Cond. 300 on DS meter (permeate)
	23:30	3.98	6.0	42.0	610	605	605	560	1.35	6.29	1.35	2.63	1.001	35.0	20.2	
	24:00	3.93	6.0	40.5	610	605	605	560	1.30	6.06	1.30	2.63	1.001	35.0	20.0	
4-25-76	Advance time one hour, daylight savings; unit operated unattended from 24:00 to 0.7:00															
	07:00	3.26	6.8	36.0	605	600	600	555	1.02	4.75	1.02	2.24	1.002	33.5	20.0	Permeate DS 300
	07:45	Tanker full, end of run														15-F, C, raw feed – P sampler
		BIZ wash up														malfunctioned – partial sample

(continued)

TABLE D-1 (continued)

Date	Time	Feed gpm	Feed pH	Feed Temp., °C	Pressure, psi Rev-O-Pak In	Rev-O-Pak Out	UOP In	UOP Out	Permeate gpm	Permeate gfd (flux)	Permeate Draw off, gpm	Concentrate Draw off, gpm	Concentrate sp.gr.	Concentrate Temp., °C	Gould pump, amps	Remarks
									Stage 1-D, Run 1, Sample 15A (concentrating run 0.8% to 1.6% — 1200 gal in 3 tanks)							
4-25-76	10:30	Start up														
	11:30	3.97	6.3	37.0	608	600	600	540	1.28	5.97	1.28	2.69	1.005	35.0	20.0	
	12:30	3.99	6.3	35.0	605	600	600	545	1.27	5.92	1.27	2.72	1.005	33.0	19.8	Start collecting 1.6% concentrate
	13:30	2.78	6.3	37.0	605	600	600	550	1.22	5.69	1.22	1.56	1.005	34.2	19.8	
	14:30	3.07	6.3	37.0	605	600	600	550	1.19	5.55	1.19	1.88	1.005	34.0	19.8	
	15:30	2.90	6.3	37.0	605	600	600	545	1.19	5.55	1.19	1.71	1.004	33.0	20.0	
	16:30	2.73	6.3	37.0	605	600	600	545	1.17	5.45	1.17	1.56	1.005	34.0	19.9	
	17:30	2.74	6.3	37.3	600	598	598	540	1.15	5.36	1.15	1.59	1.005	34.0	20.0	
	19:00	2.73	6.3	37.5	605	600	600	545	1.13	5.27	1.13	1.60	1.005	34.5	20.0	
	21:30	2.71	6.3	34.0	605	600	600	545	1.11	5.17	1.11	1.60	1.005	36.0	20.0	DS meter 500
	24:00	2.67	6.3	34.0	605	600	600	545	1.07	4.99	1.07	1.60	1.005	35.0	20.0	DS meter 600
4-26-76	End of Sample 15A, begin recycle of 0.8% in trailer															
									Stage 1-A, Run 2, Sample 16							
4-26-76	00:15	Start of recycle to trailer														
	01:00	4.93	6.3	34.5	610	605	605	550	1.33	6.20	1.33	3.60	1.004	34.5	--	DS meter 450
		Operated for 6 hours unattended														
	07:00	4.86	6.4	--	605	600	600	525	1.11	5.17	1.11	3.75	1.005	33.0	20.0	
	07:30	Washup														
	08:30	Start up														
	09:00	5.23	6.4	36.0	605	600	600	550	1.50	6.99	1.50	3.73	1.005	32.2	20.4	
	11:00	5.21	6.5	37.0	605	600	600	545	1.36	6.34	1.36	3.85	1.005	32.4	20.1	
	12:20	Down for Versene wash up								1.32	6.15					13:15 wash up, Versene 1800 ml
	15:15	Start up														in 60 gal of water, pH 6.3, 1 hr
	15:50	6.05	6.5	36.0	608	600	600	550	1.82	8.48	1.82	4.23	1.006	33.3	20.5	
	16:50	6.01	6.5	36.0	605	600	600	550	1.78	8.30	1.78	4.23	1.006	33.0	20.5	
	17:50	5.91	6.5	36.0	610	605	605	550	1.72	8.02	1.72	4.19	1.007	33.5	20.2	DS 650, sp.gr. feed 1.004 @ 34°C
	20:00	5.95	6.5	37.0	615	610	610	560	1.60	7.76	1.60	4.35	1.008	33.0	20.2	DS 750, sp.gr. feed 1.006 past
	21:00	5.89	6.5	37.0	615	610	610	560	1.53	7.13	1.53	4.36	1.008	33.0	20.2	run added
	22:00	5.82	6.5	37.0	605	600	600	535	1.46	6.80	1.46	4.36	1.009	32.0	20.3	
	24:00	5.66	6.5	37.0	610	605	605	540	1.36	6.34	1.36	4.30	1.010	32.0	20.4	DS 850, sp.gr. feed 1.007
		End of run continue Sample 17														
									Stage 1-A, Run 2, Sample 17 (concentrating run 2% to 4%)							
4-27-76		Operated for 7 hours unattended														
	07:00	5.31	7.0	33.0	605	600	600	538	1.08	5.03	1.08	4.23	1.014	31.0	20.1	Feed 1.011 @ 30°C, DS 1300
	07:20	Shut down for Versene wash up, pH 6.8														
	09:10	Start up														
	10:00	5.60	6.8	26.6	612	608	608	540	1.40	6.52	1.40	4.20	1.015	29.0		Feed 1.012 @ 28°C, DS 1300
	12:00	5.91	6.9	29.0	610	602	602	530	1.43	6.66	1.43	4.48	1.015	35.3		
	14:00	5.85	6.9	32.0	610	605	605	530	1.37	6.38	1.37	4.48	1.016	34.4		DS 1850
	16:00	5.69	--	34.4	613	605	605	530	1.28	5.97	1.28	4.41	1.018	37.2		Feed 1.015 @ 34.4°C, DS 2200
	18:00	5.86	7.2	40.0	608	600	600	520	1.12	5.22	1.12	4.74	1.022	35.0		Feed 1.018 @ 32.2°C, DS 2500
	20:00	5.63	7.2	39.4	608	600	600	530	0.94	4.38	0.94	4.69	1.025	34.0		Feed 1.022 @ 33.2°C, DS 3300
	20:20	End of run														

TABLE D-2. ANALYTICAL DATA

Sample No.	Sample*	Date	Mode of† Operation	Specific‡ Gravity	pH	Total Solids, g/l	COD, mg/l	Soluble Calcium, mg/l	Sodium, mg/l	Inorganic Chloride, mg/l	Total§ Oxalate, mg/l	BOD_5, mg/l	Color Units	Osmotic Pressure, psi	Elec. Res., ohms 21°C	Elec. Res., ohms 35°C
7	Feed-1	4-15-76	Thru	—	4.20	5.08	2,265	119	1090	1301	207	830	2,660	43	201	131
	Feed-2			1.0051	4.23	8.04	3,380	224	1744	1910	268	1096	4,494	63	134	87
	Perm			—	3.77	0.26	140	<1	66	58	10	197	<1	--	3362	2185
	Conc.			1.0059	4.21	8.97	3,520	254	1968	2340	259	--	5,040	73	--	--
8	Feed-1	4-16-76	Thru	--	6.73	5.36	2,766	45	1254	910	91	--	3,100	--	247	160
	Feed-2			1.0055	6.88	8.66	4,433	100	2084	1333	127	--	5,240	67	160	104
	Perm			—	6.06	0.30	220	<1	81	74	--	--	67	--	3100	2015
	Conc.			1.0061	6.91	9.44	4,681	116	2228	1438	122	--	5,740	69	--	--
9	Feed-1	4-17-76	Thru	—	6.32	5.51	2,837	60	1244	942	92	891	3,240	--	239	155
	Feed-2			1.0054	6.43	8.52	4,397	118	1968	1411	97	1281	5,160	62	164	107
	Perm			—	5.47	0.24	213	<1	66	66	--	86	0	—	4060	2639
	Conc.			1.0075	6.52	9.65	5,000	130	2204	1784	130	--	6,000	66	--	--
10	Feed-1	4-19-76	Conc.	—	6.87	10.69	5,342	164	2520	1946	--	--	7,040	75	115	75
	Feed-2			1.0080	6.98	12.38	6,120	194	2916	2377	—	—	8,320	89	111	72
	Perm			—	5.80	0.36	217	<1	112	132	—	—	35	--	3000	1950
	Conc.			1.0092	6.94	14.42	7,269	225	3336	2823	—	—	8,580	93	—	--
11	Feed-1	4-20-76	Conc.	—	6.93	15.33	7,770	245	3504	2886	125	2241	10,200	105	92	60
	Feed-2			1.0113	7.00	17.63	8,820	278	3944	3389	173	2754	11,500	123	78	51
	Perm			—	5.83	0.52	217	<1	162	191	0	88	0	—	1950	1268
	Conc.			1.0122	6.99	19.09	10,202	303	4240	3463	192	—	12,900	126	—	—
12	Feed-1	4-21-76	Conc.	1.0214	7.19	33.44	16,986	452	7432	5908	--	—	2,350	--	42	27
	Perm			—	6.57	1.36	295	3	430	577	--	--	30	—	--	--
	Conc.			1.0221	7.16	34.96	17,829	466	7580	6663	—	—	—	233	515	335
	Final conc.			1.0254	7.15	40.83	20,737	651	8600	7597	--	—	—	272	--	--
14	Feed-1	4-23-76	Thru	1.0044	6.85	6.31	2,660	76	1460	1296	—	--	2,930	59	176	114
	Feed-2			1.0051	6.79	7.27	3,160	89	1675	1364	—	—	3,430	--	155	101
	Perm			—	6.15	0.26	198	<1	76	74	—	--	8	--	2828	1838
	Conc.			1.0054	6.45	7.71	3,460	98	1795	1380	--	--	3,620	62	--	—
15	Feed-1	4-24-76	Conc.	1.0038	6.79	5.42	1,980	66	1240	997	55	854	2,510	56	206	134
	Feed-2			1.0050	6.87	7.08	2,960	100	1625	1359	73	1152	3,400	--	158	103
	Perm			--	6.16	0.24	176	<1	76	79	3	56	0	--	3000	1950
	Conc.			1.0053	6.61	7.64	3,380	105	1825	1427	109	—	3,700	64	--	--
15A	Feed-1	4-25-76	Conc.	1.0050	6.87	7.65	4,220	104	1825	1448	121	768	3,770	--	168	109
	Feed-2			1.0078	6.91	12.04	5,500	175	2900	2335	137	1710	5,960	88	98	104
	Perm			—	5.88	0.36	180	<1	118	134	0	60	8	--	1920	1248
	Conc.			1.0088	6.74	13.04	6,060	196	3205	2408	149	--	6,500	100	--	--
16	Feed-1	4-26-76	Conc.	1.0061	6.88	9.33	4,640	122	2280	1826	--	--	4,500	73	164	107
	Feed-2			1.0087	6.97	13.18	6,320	182	3180	2513	--	--	6,720	101	142	92
	Perm			--	6.03	0.39	197	<1	129	155	--	--	11	--	--	--
	Verg. wash			--	—	7.52	--	162	--	--	--	--	--	--	--	--
17	Feed-1	4-27-76	Conc.	1.0185	7.19	28.47	13,620	365	6680	5572	436	--	15,000	200	53	34
	Perm			--	6.39	1.16	266	2	682	460	0	124	5	19	614	399
	Final perm.			--	6.13	2.01	300	3	392	798	0	162	5	25	351	228
	Final conc.			1.0254	7.03	40.02	19,440	470	9640	7534	572	--	21,250	269	--	--

*Feed-1 = feed to system; Feed-2 = feed to modules from recycle tank (Feed-2 is material treated by modules — high value due to recycle).

†Thru = no recycle of concentrate; Conc. = recycle of 100% concentrate back to feed supply.

‡By pycnometer at 35°C.

§As sodium oxalate.

TECHNICAL REPORT DATA

(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA-600/2-78-132	2.	3. RECIPIENT'S ACCESSION NO.
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15. SUPPLEMENTARY NOTES

16. ABSTRACT Reverse osmosis (RO) and freeze concentration (FC) were evaluated at three different pulp and paper mills as tools for concentrating bleach plant effluents. By these concentration processes, the feed effluent was divided into two streams. The clean water stream approached drinking water purity in some instances, and could potentially be recycled to the mill with minimal problems. The concentrate stream retained virtually all the dissolved material originally present in the feed. Typically, reverse osmosis removed 90% of the water from a stream containing 5 g/l of total solids to give a concentrated stream with 50 g/l solids. Freeze concentration further concentrated the reverse osmosis concentrate to about 200 g/l. Thus, each 100 liters of feed resulted in about 98 liters of clean water and 2 liters of concentrate. Schemes for the ultimate disposal of this final concentrate were not tested.

Based on data collected at the three mills, estimates of the process economics were made. Reverse osmosis alone, or combined with freeze concentration, is quite expensive. At current levels of water usage for bleaching, costs ranged from $18 to $27 per metric ton of bleached pulp (approximately $3.50/1000 gallons (M gal) of bleach plant and increased membrane life could significantly lower these costs.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Water Renovation, Water Pollution, Color, Biochemical oxygen demand, Bleaching	Water reuse, chemical reuse, reverse osmosis, freeze concentration, suspended solids control, product quality	68D

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