

Acid Mine Waste Treatment Using Reverse Osmosis



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Acid Mine Waste Treatment Using Reverse Osmosis

by

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for the

ENVIRONMENTAL PROTECTION AGENCY
WATER QUALITY OFFICE

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EPA Review Notice

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ABSTRACT

The basic objectives of this test program were to demonstrate the applicability of reverse osmosis to the demineralization of acid mine drainages, both high-ferrous and high-ferric types, and to reclaim the maximum percentage of such feedwater in purified form suitable for domestic or industrial purposes, or as stream discharge. These goals included the attainment of maximum water recovery while maintaining the required product water quality and the determination of pretreatment requirements necessary to maximize water recovery and membrane life.

Two reverse osmosis test units were operated during the course of these tests: a nominal 10,000-gpd unit equipped with eighteen 50-ft² modules and a nominal 4,000-gpd unit equipped with nine 50-ft² modules. The modules used in these units consisted of both high-selectivity and high-flux cellulose acetate membranes.

The test program was carried out at three different mine drainage sites. The mine drainage water at the first site, Norton, West Virginia, contained greater than 98 percent of the iron present in the ferric form; at the other two sites, Morgantown, West Virginia, and Ebensburg, Pennsylvania, the drainage water contained predominantly ferrous iron. Discharges at the second site were so concentrated that recoveries were limited to 50 percent; recoveries of 80 to 90 percent were attained at the first and third sites. No iron fouling was encountered at any of the three sites. Specific salt rejections were >97 percent at all sites.

This report was submitted in fulfillment of Contract No. 14-12-525 between the Environmental Protection Agency and Gulf Environmental Systems Company.

Key Words: Reverse osmosis, acid mine drainage, demineralization, calcium-sulfate solubility, ferrous-ferric iron ratios, water recovery, brine treatment.

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SECTION I

CONCLUSIONS

The basic objective of the test program, demonstration of the ability of reverse osmosis to demineralize acid mine drainage water (both high-ferrous and high-ferric types), was successfully demonstrated at two of the three experimental test sites. The feedwater at Site 2 was so polluted that reverse osmosis demineralization does not seem feasible. The quality of the product water produced at Site 1 and Site 3 would be suitable for domestic or industrial purposes with the exception of pH or dissolved iron content. If the pH of the water were adjusted to neutral, the iron would precipitate and could be easily removed by sand filtration or settling.

Environmental Protection Agency (EPA) personnel carried out neutralization and decantation operations followed by recycling of the supernatant therefrom through the reverse osmosis unit at Site 1. This resulted in effective 98 percent water recovery based on feed volume, with maintenance of excellent quality in the recovered permeate water. The details of this work are reported elsewhere (Ref. 1) by EPA personnel.

The ability to operate with no iron fouling on acid mine discharge waters similar in type to water with which other investigators (Ref. 2) had experienced fouling was also successfully demonstrated at Site 3. The method used to control this type of fouling was pH adjustment of the raw feedwater.

Laboratory studies of the effectiveness of compounds in retarding calcium sulfate precipitation yielded no successful candidates. Similar studies aimed at neutralizing the reverse osmosis brine indicated that lime or MgO would be satisfactory, with lime being chosen because of its lower cost.

A method of evaluating an acid mine drainage water with regard to the maximum water recovery possible was developed. The limiting factor in recovery is the calcium sulfate concentration. While the CaSO_4 solubility limit can be exceeded, probably because of the short residence time, low pH, other ionic interferences, etc., there does seem to be a fairly definite limit of approximately 300 to 400 percent of saturation.

The standard pretreatment of sand filtration followed by cartridge filters proved to be adequate with respect to feed clarification, so membrane surface fouling from suspended materials in the feed stream was not encountered.

The high-pressure feed pumps used on both units during these tests failed on numerous occasions, resulting in time-consuming delays. These pumps, although different types, contained materials that were not compatible with the acid mine drainage water.

SECTION II

RECOMMENDATIONS

It is recommended that to further evaluate the ability of reverse osmosis to demineralize various acid mine waters, a larger unit, where no or minimal brine recycle is required, be utilized. The larger units can be economically equipped with automatic features that are prohibitive on smaller units. Cost studies of demineralizing acid mine water with units of 1 mgpd and larger should be included in future studies.

Recent field experience has indicated that a spiral module should be operated at pressures required to produce 10 to 15 gallons per square foot per day of actual permeate, even though the membrane is capable of much higher productivity. Preferably this pressure should be below 600 psi and in most cases will be, except for feeds of abnormally low temperatures or those having very high osmotic pressures.

More work is required on different types of acid mine drainage waters to fully evaluate reverse osmosis capabilities. Long-term testing (in excess of six months) is required to accurately predict membrane lifetime. Further testing is required to examine the calcium sulfate solubility limitations and the validity of adjusting the pH of ferrous-type feeds.

It is further recommended that all the pumps used in reverse osmosis plants be either stainless steel, plastic, or a combination of these two materials. Stuffing-box-type packings are not advisable.

The method of recycling the treated brine (Ref. 1) back to the feed remains to be evaluated for operation on ferrous-type acid mine drainage.

Further studies are required to fully ascertain what actually occurs in the modules that results in flux increases during periods of low recovery operation and accidental or planned shutdowns. The apparent discrepancies in the temperature-flux normalization curves used in these tests need to be resolved.

SECTION III INTRODUCTION

This is the final report on the reverse osmosis field testing on acid mine water carried out by Gulf Environmental Systems Company for the Environmental Protection Agency, Water Quality Office, under Contract 14-12-525. The work done under this contract followed prior work performed at the EPA Norton Mine Drainage Treatment Laboratory with EPA site support under Office of Saline Water Contracts 14-01-0001-1243, 14-01-0001-1836, and 14-01-0001-1836 Amendment No. 3, which has been previously reported (Refs. 3-5).

Work done under these referenced contracts had demonstrated that a reverse osmosis unit using the Gulf Environmental Systems ROGA[®] spiral-wound modular concept could successfully be operated on acid mine drainage at up to 75 percent recovery levels (and for several limited times, as high as 92 percent) using either high-flux, lower-selectivity or standard-flux, high-selectivity modules. The purpose of the present contract was to determine the maximum water recovery possible while producing water suitable for domestic or industrial purposes or for discharge into streams. Also to be investigated were the pretreatment requirements necessary to maximize water recovery and membrane life. These tests were carried out at three different locations where the mine discharges were markedly different: (1) Norton, West Virginia, Grassy Run Creek; (2) Morgantown, West Virginia, mine discharge; and (3) Ebensburg, Pennsylvania, mine discharge. Typical analyses of the acid mine discharges are shown in Table I. Based on past evaluations (Ref. 3), the osmotic pressure of these various feeds was assumed to be 10 psi per 1000-micromho specific conductance.

The water was taken from Grassy Run Creek (Site 1) about 2 miles downstream of the mine discharge. The water in this shallow, cascading creek was well aerated before it reached the intake lines, and in addition contained

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TABLE I
TYPICAL WATER ANALYSES OF TESTED ACID MINE DRAINAGE

	pH	Cond (μ mhos/cm)	Ca as Ca (mg/l)	Mg as Mg (mg/l)	Acidity	Total Fe (mg/l)	Fe ⁺⁺ (mg/l)	SO ₄ (mg/l)	Al (mg/l)	TDS (mg/l)
Site 1, Grassy Run Creek, Norton, West Virginia - Winter	2.86	1660	114	33	732 ¹	146	6.6	1040	32	1604
Site 1 - Spring	2.98	1083	54	19	372 ¹	73	1.5	600	16	875
Site 1 - Summer	2.7	1350	115	38	644 ¹	153	<1	936	38.5	--
Site 1 - Fall	2.8	850	72	25	357 ¹	74	<1	610	26	--
Site 2, Arkwright Mine, Morgantown, West Virginia	2.3	7600	650	450	5380	2960	1180	12285	380	--
Site 3, Mines 32-33, Ebensburg, Pennsylvania	3.6	1480	214	157	360	126	101	1638	35	--

¹Acidity as CaCO₃ (mg/l).

the raw domestic wastes discharging from about 40 homes upstream of the test site. This water contained 50 to 150 ppm of iron, depending on climatic conditions, which was present almost totally in the ferric state.

The water at Site 2 was taken directly from a pump discharge of the Arkwright mine of the Christopher Coal Company. The iron content of this water was about 65 percent ferrous, but the total amount was about 30 times that present in the water at Site 1.

The water at Site 3 also was taken directly from the pump discharge of an active mine. This discharge was from Mines 32-33 of the Cambria Division of the Bethlehem Mines Corporation. The total iron content of this water was approximately the same as that at Site 1, but was approximately 80 percent in the ferrous state.

The reverse osmosis equipment, test program, site selection, operation, and maintenance were supported by and under the direction of the Mine Drainage Pollution Control Section of the Environmental Protection Agency, Water Quality Office. Field test engineering personnel were provided by Gulf Environmental Systems at the beginning of each set of experiments and subsequently as required.

SECTION IV

DESCRIPTION OF TEST EQUIPMENT

The reverse osmosis equipment used in these tests consisted of a 10,000-gpd unit and a 4,000-gpd unit. The 10,000-gpd unit was composed of five 4-in.-diameter, 10-ft-long Schedule 40 pressure vessels, a high-pressure pump, and instrumentation and controls. A piping and instrumentation diagram of the unit is shown in Fig. 1. The five pressure vessels contained a total of 15 ROGA® modules, with each module containing 50 ft² of modified cellulose acetate membrane. The pressure vessels were arranged in a 2-2-1 array in order to maintain as uniform a brine flow as possible at required velocities. In this arrangement, vessels 1 and 2 are in parallel. The brine from these vessels is combined to be the feed for vessels 3 and 4, which are in parallel. The brine from vessels 3 and 4 is combined to be the feed for vessel 5. An instrument panel provides for measurement of the feed pressure, the feed channel pressure drop, and the product and brine flow from each vessel. The unit is plumbed so that a portion of the concentrated brine can be recycled back to the feed. This arrangement was required to obtain high recovery levels while maintaining an adequate brine flow of about 3 gpm or greater. This brine flow is necessary because it minimizes the concentration polarization at the membrane surface, which, in turn, minimizes the probability of membrane fouling. Units of larger size, i.e., 50,000 gpd or larger, are capable of high recoveries without brine recirculation. The 4,000-gpd unit had only three pressure vessels, but was similar in controls and operation to the 10,000-gpd unit.

Both high-selectivity, standard-flux modules and high-flux, lower-selectivity modules were used in these tests. In addition, for operation at Site 3, modules manufactured by a different technique were used. These modules

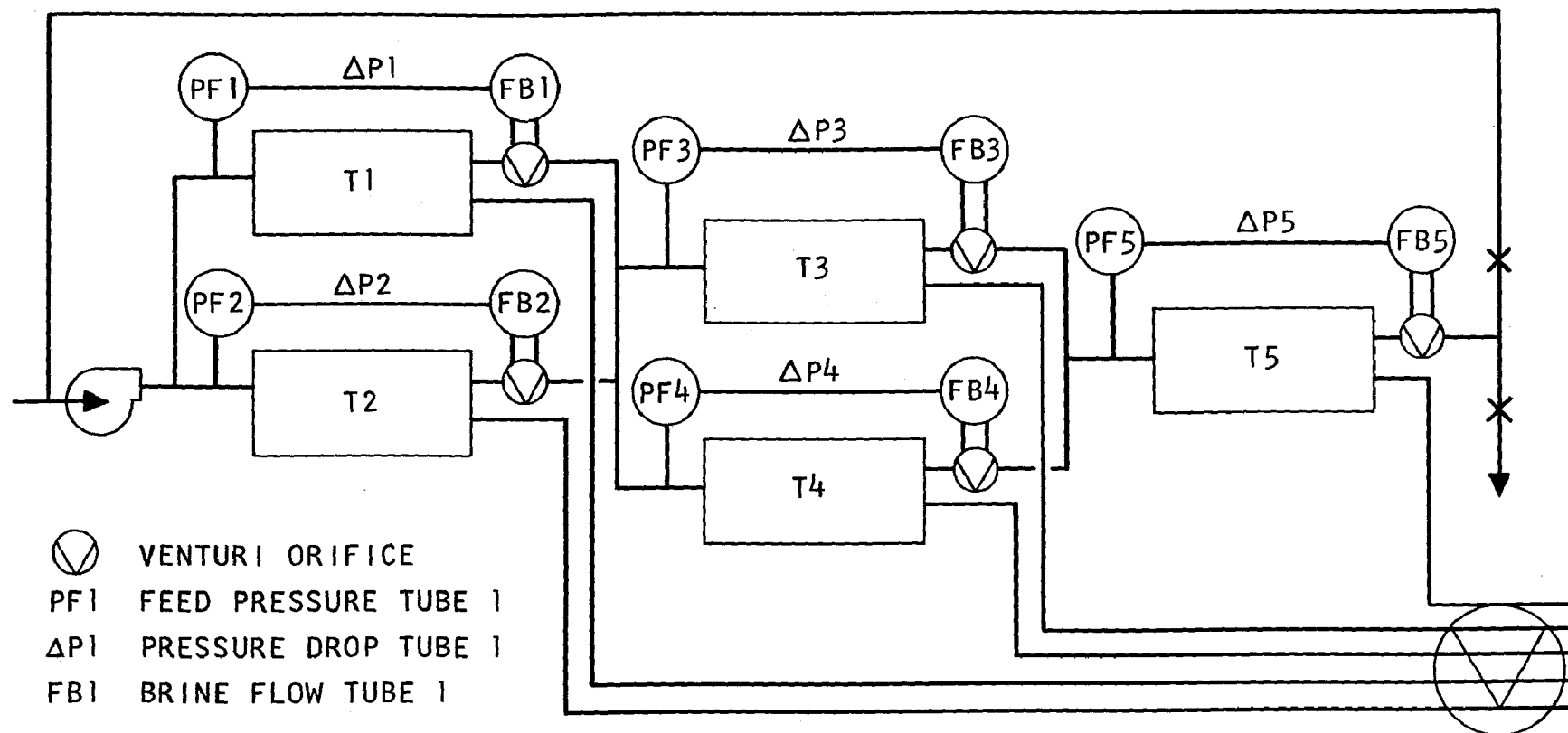


Fig. 1. Piping and instrumentation diagram for 10,000-gpd reverse osmosis unit with brine recycle

combine both the high-flux and high-selectivity properties of the other modules and are more rugged physically. A comparison of performance for typical modules of these three types is as follows:

	Module Membrane Coefficient, ¹ A x 10 ⁵ (g/cm ² -sec-atm)	Percent Sodium Chlorine Rejection ²
High-selectivity module.	≥1.3 < 2.0	≥94.5
High-flux module.	≥2.0 < 3.0	≥90
High-flux, high- selectivity module.	1.9-2.2	≥95

¹A = 1.0 x 10⁻⁵ g/cm²-sec-atm = 8.65 gal/ft²/day at 600 psi net driving pressure and 77°F.

²At 600 psi and 2000 ppm feed

Neutralization of the concentrated brine with hydrated lime at Site 1 was carried out by EPA personnel using their equipment and facilities (Ref. 6). Because of the fixed nature of this equipment, no neutralization was possible at the other two test sites.

SECTION V
PERFORMANCE CALCULATION METHODS

The product water flow through a semipermeable membrane may be expressed as

$$F_W = A(\Delta P - \Delta \pi) ,$$

where F_W = water flux ($\text{g}/\text{cm}^2\text{-sec}$),

A = water permeability coefficient ($\text{g}/\text{cm}^2\text{-sec-atm}$),

ΔP = pressure applied to the membrane (atm),

$\Delta \pi$ = osmotic pressure across the membrane (atm),

g = grams.

For these tests, the module or unit water permeability coefficient A (in $\text{g}/\text{cm}^2\text{-sec-atm}$) was calculated by dividing the product water flow, corrected to 77°F, by the membrane area and the net pressure. The net pressure is the applied pressure minus the osmotic pressure. The osmotic pressure was assumed to be directly proportional to the conductivity. A value of 10 psi was used for the osmotic pressure of the acid mine water when the specific conductance of the feed equaled 1000 micromhos/cm.

The percent rejection of the membrane is expressed as

$$1 - \frac{\text{Concentration Product}}{(\text{Concentration Feed} + \text{Concentration Brine}) \div 2} \times 100$$

In cases where the unit was operating with brine recycle, the blended feed concentration was used in lieu of the raw feed concentration.

SECTION VI
HIGH-PRESSURE-PUMP PROBLEMS

A significant amount of time was lost on this program because of the problems encountered with the high-pressure pumps on both units. The 10,000-gpd unit was equipped with a Moyno SSQ-9P4 progressive-cavity type pump, while the 4000-gpd unit was originally equipped with a Gardner-Denver PQ-2 triplex positive-displacement pump.*

The Moyno pump failed in several areas. Both the pumping rotor and the connecting rod between the rotor and drive shaft were broken several times. In addition, the original connecting rods were made from carbon steel, which corroded severely with the resulting corrosion products entering the process water, which tended to foul the modules. Attempts at replacing the carbon steel rods with rods of stainless steel were not successful because the stainless steel did not have sufficient tensile strength. A special precipitation-hardened steel was employed to fabricate several rods which were installed during the latter part of the last run. However, not enough operating hours were accumulated to evaluate this type of steel. No specific answer to this material problem is evident. The Moyno pump also had a stuffing box seal arrangement which both leaked badly and allowed packing to work its way into the process water. Because of these problems, it is recommended that this type of pump not be used in future reverse osmosis testing on acid mine waters.

The Gardner-Denver triplex pump originally was fabricated with an aluminum-bronze fluid end, ceramic plungers, and Monel valves and seats. The Monel valve seats corroded within 2 days of operation on brine from the 10,000-gpd unit. The Monel valves were replaced with readily available aluminum-bronze valves, but this set lasted less than 12 hr. At this point, a new aluminum-

*Mention of commercial products does not imply endorsement by the EPA.

bronze fluid head, complete with stainless steel valves, was installed on the pump. After about five days of operation on brine from the 10,000-gpd unit, the plunger stuffing boxes were leaking very badly, and therefore all testing was suspended until new parts could be obtained. When the new stuffing boxes were installed, the pump was operated only on neutralized brine and performed satisfactorily.

Further use of this type of pump seems feasible only if the complete fluid end is furnished in stainless steel.

SECTION VII

FEEDWATER PRETREATMENT STUDIES

The feedwater to a spiral-wound reverse osmosis unit requires pretreatment for three significant reasons. First, particles in the water capable of plugging the brine channels must be removed from the water before it reaches the modules. During preceding test programs at the Norton site, rapid sand filters followed by 10-micron cartridge filters had proven satisfactory for this purpose. Therefore, this method was used at all three sites.

Second, biological growths of algae or molds inside the modules must be prevented. Continuous addition of 5 ppm copper sulfate during the warmer summer months at Site 1 was used for this purpose, while no additions were required during the winter months. Because the feedwater at Sites 2 and 3 was taken directly from the mine discharge, and not from a creek at a considerable distance from the mine discharge, no chemical additions were deemed necessary at those sites. Postoperational examination of modules supported this assumption.

Third, the precipitation of sparingly soluble salts in the concentrated brine with subsequent plugging or fouling of the modules must be prevented. Indeed, it has been demonstrated that the limiting factor in system recovery is the concentration of these salts. Previous experience on other types of waters has shown that calcium sulfate is the most significant sparingly soluble compound present. In other tests, where the feed was concentrated with respect to calcium sulfate, it was found that the water could be further concentrated in a reverse osmosis system by a factor of two or more if "threshold inhibitors" (sodium hexametaphosphate, Cyanamer P-35, etc.) were added to the feed in the range of 5 to 10 mg/l. To investigate these and other inhibitors, a synthetic acid mine water was prepared at Gulf Environmental Systems, based on suggested formulations in the literature (Ref. 7). The composition of this synthetic acid mine water was as follows:

Magnesium (Mg).	116 mg/1
Calcium (Ca).	385 mg/1
Iron (Fe ⁺⁺⁺).	500 mg/1
Aluminum (Al)	150 mg/1
Manganese (Mn ⁺⁺).	50 mg/1
Sulfate (SO ₄)3615 mg/1
Conductivity.3800 micromhos/cm at 25°C
pH.	2.5

This solution was roughly comparable to the concentrate produced at 75 to 90 percent recovery by a reverse osmosis unit at the Norton site. This value was deemed to be close to the limit of concentration based on calcium sulfate concentration. The achievement of further concentration by inhibiting CaSO₄ precipitation through various inhibitors was investigated and proved to be unsuccessful. Either the low pH or the high concentration of iron, or both, prevented the inhibitors from performing normally. The inhibitors tried included:

Calgon C-55	Magnafloc 837A
Calgon CL-45	Polyvinylpyrosilicone K-90
Cyanamer P-35	Silspend 180
Dequest 2000	Sodium Hexametaphosphate
Dequest 2010	Sodium Xylene Sulfonate
Dequest 2041	Surfynol 82
Eltan 382-E	Tetrasodium Pyrophosphate
Gantrey AN-119	Toranil B, 97D
Hercules Klucel H	Trasitan SPM
	Zimmite AM-100

To determine if the laboratory studies on the synthetic water provided valid conclusions in the light of field performance, sodium hexametaphosphate was added at Site 1 during one series of tests. As in the laboratory studies, no appreciable effect was observed.

SECTION VIII

TEST PROGRAM

The test program at Site 1 was to involve five phases, as outlined in Table II. Operations at this site were to include the use of both the 4,000-gpd and 10,000-gpd reverse osmosis units. The 10,000-gpd unit was to be used to concentrate the feedwater by a factor of 4 to 5; then the 4,000-gpd unit was to be used to further concentrate the brine so that an overall concentration factor of 10 or more was reached. The inability of the particular high-pressure pump (aluminum-bronze fluid end) on the 4,000-gpd unit to handle the acid mine water necessitated a change in program, so that all further testing was accomplished in the 10,000-gpd unit.

Since the previous successful testing on acid mine drainage (Refs. 3-5) was on water containing predominately ferric iron, and since other investigators (Ref. 2) had encountered difficulty when operating on high-ferrous-iron waters, it was decided that the second site should be one where a large amount of ferrous iron was present. Site 2 was selected on this basis. When it became evident that testing was limited at this site owing to the high concentration of dissolved salts, which prohibited high recoveries, a third site was sought. The discharge at this third site contained about the same amount of dissolved salts as the discharge at Site 1, except that it contained approximately 80 percent ferrous iron. At Site 3, the adjustment of the pH of the feedwater was employed to prevent or minimize the ferrous-ferric conversion and ferric precipitation. Although experimental limitations precluded comparison over a complete range of pH and recovery rates, no significant iron hydrate fouling was encountered.

TABLE II
PROPOSED TEST PLAN, SITE 1

Phase I - Unit Shakedown and Operational Check

1. Run on untreated 10,000-gpd-unit product water for approximately 1000 hr.
 - a. Run at various pressures and recoveries up to 600 psi and 80 percent recovery using brine recirculation.

Phase II - 90 Percent Recovery Operation

1. Run on untreated 10,000-gpd-unit brine to approximately 90 percent total recovery, operating at several combinations of recovery, such as:
 - a. 10,000 gpd - 60 percent recovery, no brine recirculation
4,000 gpd - 75 percent recovery, brine recirculation
 - or
 - b. 10,000 gpd - 70 percent recovery, brine recirculation
4,000 gpd - 70 percent recovery, brine recirculation
2. If 90 percent recovery is successful, increase total recovery as far as possible by increasing the recovery in the 4,000-gpd unit.

Phase III - 90 Percent Recovery Operation with Use of Inhibitor

1. Run 4,000-gpd unit on treated 10,000-gpd-unit brine, adding a precipitation inhibitor to the brine.
 - a. The minimum recovery would be that which was found to be the maximum under Phase II and would be increased as far as possible.
 - b. Precipitation inhibitors may be Cyanamer P-35, sodium hexametaphosphate, Dequest 2000, or others.

Phase IV - 90 Percent Recovery Operation, Neutralizing Brine, Use of Inhibitor

1. Run 4,000-gpd unit on neutralized 10,000-gpd-unit brine, approximately 90 percent recovery using precipitation inhibitors.
 - a. Unit conditions would be similar to those in Phase II.
 - b. The brine from the 10,000-gpd unit would be neutralized to pH 5 using CaO, CaCO₃, MgO, or others.

Phase V - Maximum Recovery

1. Run 4,000-gpd unit on treated 10,000-gpd-unit brine to maximum practical recovery, optimizing conditions of either Phase III or Phase IV.
 - a. Determine maximum recovery.
 - b. Determine minimum addition of precipitation inhibitor.

SECTION IX
OPERATION AND RESULTS AT SITE 1

Operations at Site 1 began on November 6, 1969. The mine drainage water at this location is taken from Grassy Run Creek about two miles downstream of the mine discharge. The water contains 50 to 150 ppm of iron, almost totally ferric. The overall system flow diagram for operation at this site is shown in Fig. 2.

Phase I of the test plan, i.e., checking out the pumps, instruments, etc., of the 4,000-gpd unit, continued until November 14, 1969. The high-pressure pump for this unit was a Gardner-Denver PQ-2 triplex positive displacement piston pump with an aluminum-bronze fluid head, ceramic pistons, and Monel valves and seats. Nothing unusual was experienced during this test phase.

Phase II began on November 14, with the 4,000-gpd unit operating on untreated brine from the 10,000-gpd unit. Almost immediately problems were encountered with the high-pressure Gardner-Denver pump. These pump problems continued to plague the test program until this pump was used only for neutralized feeds. This required modification of the test plans for the remainder of the contract.

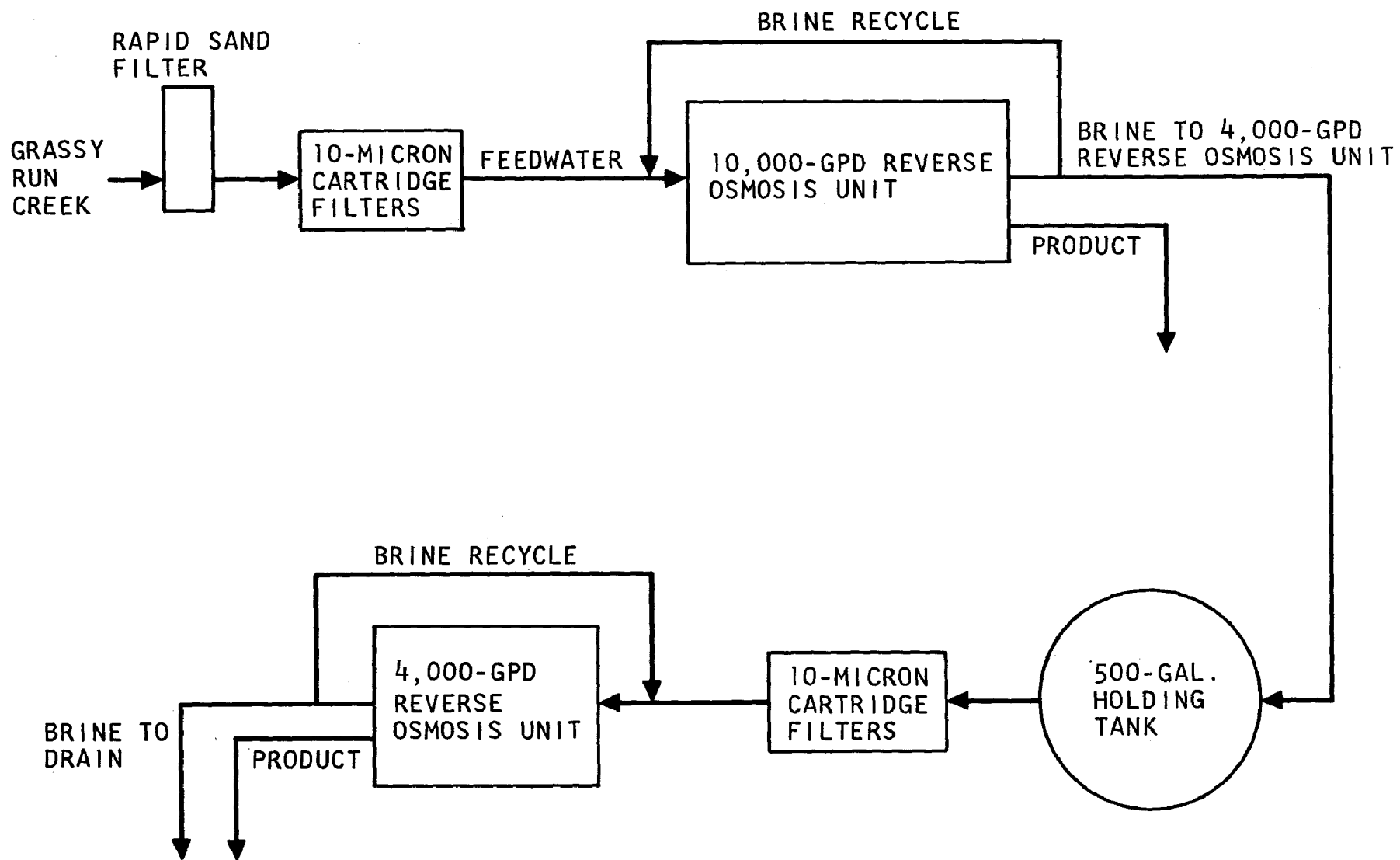


Fig. 2. Schematic of the 10,000-gpd and 4,000-gpd units at Norton, West Virginia

Up to this time, the 10,000-gpd unit had been loaded with modules that were in the unit during the last experimental contract (Ref. 5). New modules were installed as shown in Table III. Phase IV of the program began and the 10,000-gpd unit was operated at 91 percent recovery for five days, when the high-pressure pump on the unit failed.

After the pump was repaired, the 10,000-gpd unit was operated at approximately 91 percent recovery for 100 hr, during which time the brine was collected and stored. The operational history of the unit during this run is shown in Table IV. Note that during the last half of the run, the ΔP of tube 5, the last tube in the array, was increasing steadily. It was felt that this was caused by calcium sulfate precipitation. The unit recovery was lowered to 50 percent, and in about 3 hr the ΔP was back to its initial value. Thus, while CaSO_4 precipitation does occur at high recoveries, it apparently can be flushed easily from the system if caught early enough. Mean operating values for this run are shown in Table V, and representative chemical analyses are shown in Table VI.

The next run consisted of operating the 10,000-gpd unit at 91 percent recovery during the normal working hours and at 85 percent recovery at all other times. Small changes in the operating parameters, caused by valve vibration, temperature, etc., could cause major changes in recovery due to the small amount of brine being discharged. Since the unit was unattended except during working hours, it was safer to operate at 85 percent than at 90 percent during these times. This run continued for 100 hr, at which point the high-pressure pump failed. The pump was repaired, and the test continued another 80 hr before the run was terminated. Operating values and chemical data for this run are shown in Tables VII and VIII.

The stored brine was neutralized using hydrated lime and pumped into a settling tank overnight. The supernatant was sand-filtered, and the 4,000-gpd unit operated at approximately 50 percent recovery on this neutralized brine. Only enough filtered water for a 2-hr run could be stored, but during

TABLE III
MODULE LOADING ARRANGEMENT FOR 10,000-GPD UNIT
AT SITE 1, 2/5/70

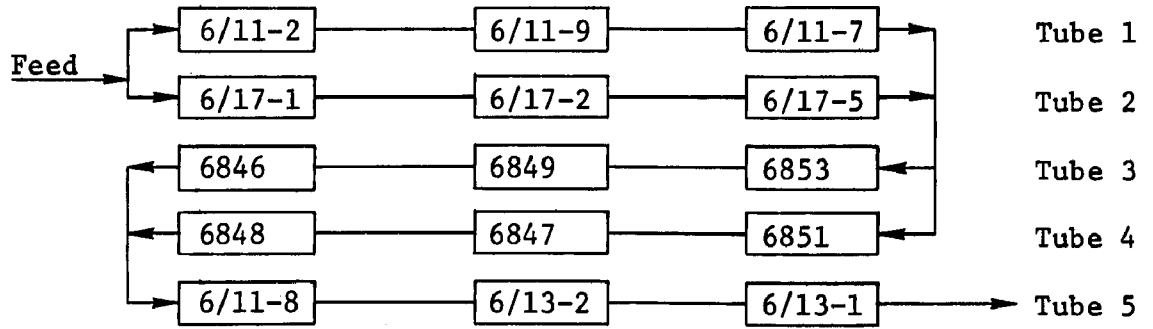


TABLE IV
OPERATIONAL HISTORY OF 10,000-GPD UNIT
AT TEST SITE 1, 3/9/70 - 3/13/70

Date	Elapsed Time (hr)	Pressure (psi)	Recovery (percent)	Product Flow (gpm)	Brine Flow (gpm)	ΔP , Tube 5 (psi)	Temp (°F)	Unit Water Permeation Coefficient, $A \times 10^{-5}$ at 77°F
3/9/70	5.4	600	92.2	5.88	0.50	3.4	49	2.15
	9.5	600	90.8	5.80	0.59	3.2	40	2.48
	12.6	600	91.0	5.55	0.55	3.2	37	2.47
	16.8	600	90.9	5.40	0.54	3.2	41	2.26
	20.9	600	90.9	5.26	0.53	3.3	41	2.20
3/10/70	24.9	600	91.0	5.30	0.52	3.2	45	2.08
	28.7	600	91.5	5.55	0.52	3.1	48	2.06
	33.0	600	89.9	5.26	0.59	3.3	38	2.31
	36.6	600	90.6	5.26	0.54	3.3	36	2.38
3/11/70	41.0	600	91.0	5.26	0.52	3.3	42	2.17
	44.8	600	91.4	5.00	0.47	3.4	42	2.06
	49.4	600	91.3	5.00	0.48	3.4	47	1.89
	53.1	600	93.1	6.20	0.46	3.1	59	1.88
	56.8	600	91.4	5.88	0.55	3.3	52	2.03
	61.0	600	90.8	5.00	0.50	3.5	40	2.13
3/12/70	64.9	600	91.3	5.00	0.47	3.6	42	2.06
	68.9	600	91.4	5.00	0.47	3.6	42	2.06
	72.7	600	91.2	5.00	0.48	3.6	46	1.93
	76.9	600	91.2	5.26	0.51	3.6	51	1.85
	81.0	600	91.7	5.26	0.47	3.7	44	2.10
	84.8	600	90.6	5.00	0.51	4.0	41	2.10
3/13/70	88.9	600	91.2	5.00	0.49	4.1	43	2.03
	92.8	600	91.3	4.76	0.46	4.1	42	1.96
	96.8	600	90.9	4.35	0.43	4.4	43	1.76
	100.0	600	90.9	4.54	0.46	4.4	44	1.81

TABLE V
MEAN OPERATING VALUES OF 10,000-GPD UNIT AT SITE 1,
3/9-70 - 3/13/70

Pressure	600.6 psi
Unit recovery	91.2 percent
Recovery variation, max.-min	93.1-90.6 percent
System recovery	91.2 percent
Feed flow	5.75 gpm
Brine flow	0.51 gpm
Brine flow, tube 5	4.12 gpm
Recycle brine flow	3.69 gpm
Temperature	44°F
Product flow	5.24 gpm
Product flow, 50°F	5.86 gpm
Flux rate, 50°F	11.25 g/f ² d
Flux rate, 77°F	18.00 g/f ² d
Length of test.	100 hr

TABLE VI
CHEMICAL ANALYSES¹ FOR 10,000-GPD UNIT OPERATION AT SITE 1, 3/9/70 - 3/13/70

Sample Designation	Date	Time	Percent Recovery	pH	Conductivity (μmhos/cm)	Acidity, pH 7.3	Hardness as CaCO ₃	Ca as Ca (mg/l)	Mg as Mg (mg/l)	Fe (mg/l)	Al (mg/l)	SO ₄ (mg/l)
Raw feed	3/9/70	1700	92.1	2.7	1200	585	390	102	32	100	33	819
Blended feed				2.2	4200	2478	1936	475	180	465	151	4026
Brine				2.0	8500	5864	4296	1050	395	1025	360	9555
Product				3.3	450	140	14	4.0	1.0	3.2	1.2	22
Percent rejection					92.91	96.64	99.55	99.48	99.65	99.57	99.53	99.68
Raw feed	3/11/70	2000	91.4	2.5	1200	533	425	112	35	114	37	874
Blended feed				2.0	4400	2098	1989	475	192	530	180	4232
Brine				1.7	10,000	5397	4849	1200	444	1200	450	10,784
Product				3.3	240	145	10	2.8	0.7	3.0	1.0	19
Percent rejection					96.67	96.13	99.71	99.66	99.78	99.65	99.68	99.75
Raw feed	3/13/70	1100	90.9	2.8	1200	559	379	102	30	110	32	628
Blended feed				2.4	4500	2239	1854	438	182	550	180	3549
Brine				2.3	10,000	5210	3938	1000	345	1120	380	7644
Product				3.4	210	107	8	2.4	0.5	2.5	1.3	11
Percent rejection					97.10	97.13	99.72	99.67	99.81	99.70	99.54	99.81
Raw feed	3/9 - 3/13/70 means	--	91.2	2.7	1190	633	405	106	32	110	35	810
Blended feed				2.2	4210	2584	1920	482	180	528	172	4024
Brine				2.0	9542	5914	4385	1102	401	1190	398	9542
Product				3.4	248	116	10	3.0	0.7	2.8	1.1	16.8
Percent rejection					96.39	97.27	99.68	99.62	99.76	99.67	99.61	99.75

¹These analyses were made on-site by EPA personnel.

TABLE VII
MEAN OPERATING VALUES OF 10,000-GPD UNIT AT SITE 1,
3/16/70 - 3/30/70

	<u>91 Percent Recovery</u>	<u>85 Percent Recovery</u>
Pressure, psi.	600	602.5
Unit recovery, percent	91.1	84.4
Feed flow, gpm	5.42	5.63
Brine flow, gpm.	0.48	0.69
Brine flow, tube 5, gpm.	4.07	4.11
Recycle brine flow, gpm.	3.62	3.44
Temperature, °F.	45	44
Product flow, gpm.	4.94	4.94
Flux rate, 50°F, g/f ² d	10.48	10.52
Flux rate, 77°F, g/f ² d	16.77	16.83
Length of test, hr		192

TABLE VIII
CHEMICAL ANALYSES¹ FOR 10,000-GPD UNIT OPERATION AT SITE 1, 3/16/70 - 3/30/70

Sample Designation	Date	Time	Recovery	pH	Conductivity (μmhos/cm)	Acidity pH 7.3	Hardness as CaCO ₃	Ca as Ca (mg/l)	Mg as Mg (mg/l)	Fe (mg/l)	Al (mg/l)	SO ₄ (mg/l)
Raw feed	Means	--	90.7	2.8	1100	494	407	102	36	80	31	819
Blended feed				2.5	3600	1990	2010	500	182	380	160	2594
Brine				2.3	7000	4266	4423	1050	432	860	360	8463
Product				3.1	240	40	14	4.0	1.0	2	1	14
Percent rejection ¹					95.47	98.72	99.56	99.48	99.67	99.68	99.62	99.75
Raw feed	Means	--	84.8	2.7	1200	539	422	107	37	80	31	846
Blended feed				2.5	3000	1730	1689	412	158	320	126	1775
Brine				2.3	6000	3887	3759	887	370	680	290	7098
Product				3.1	240	37	11	3.2	0.7	2	1	12
Percent rejection					94.67	98.68	99.60	99.51	99.73	99.60	99.52	99.73

¹These analyses were made on-site by EPA personnel.

this time, even with 10 mg/l sodium hexametaphosphate addition, an increase in the last vessel ΔP indicated that calcium sulfate precipitation was occurring. The run was repeated with similar results. Overall system recoveries of approximately 94 percent were achieved during this short test. With this work, Phase IV of the test plan was completed. Results of Phase IV testing can be summarized as follows:

1. Recovery levels of 94 percent or so were attainable during short-term operations, but not over a longer time period because of calcium sulfate precipitation.
2. Again, the CaSO_4 precipitation inhibitor appeared ineffective.

After completion of Phase IV testing, discussions with the contracting agency were held, and a decision to try a new approach was made. This was to blend the neutralized brine directly back to the feed of the 10,000-gpd unit. This work was done by the EPA and is being reported (Ref. 1) elsewhere by EPA personnel.

The work described above concluded the testing under this contract at Site 1. Environmental Protection Agency personnel continued to operate the 10,000-gpd unit until May 5, 1970 or until the modules had accumulated 770 hr. The modules were then removed from the unit, and a module from each tube was returned to San Diego for postoperational inspection while the remainder were stored in copper sulfate solution. The results of tests performed using the five modules are given in Table IX.

One module, No. 6/11-7, was opened and visually inspected and was found to contain a large amount of debris, probably pump packing. Debris samples were collected for identification and samples of the backing material were taken for evaluation.

Results of analyses of the deposits on the backing material from module 6/11-7 are shown in Table X. The major constituent is iron, as would be expected, since if iron ions do get through the membrane or enter via leaky areas, the

TABLE IX
POSTOPERATIONAL PERFORMANCE OF MODULES FROM SITE 1

Module Number	A		M	B		Percent Rejection	
	Orig.	As Tested		Orig.	As Tested	Orig.	As Tested
6/17-5	2.43	2.17	-0.02	6.26	12.17	93.84	87.52
6/11-7	2.04	1.40	-0.06	6.8	7.09	92.2	88.56
6/13-1	2.06	1.50	-0.05	6.55	8.62	92.52	87.23
H-6847	1.73	1.65	-0.01	7.31	4.95	95.37	92.84
H-6853	1.48	1.24	-0.03	2.38	3.33	96.06	93.51

Note: All tests were run on 2000 mg/l NaCl solution.

A = $\text{g/cm}^2\text{-sec-atm} \times 10^{-5}$, water flux; A of $1 \times 10^{-5} = 8.65$ gpd
at 600 psi net at 77°F.

B = cm/sec, salt flux.

M = log-log flux decline slope, based on 770-hr operation.

TABLE X
SPECTROCHEMICAL ANALYSIS OF BACKING
MATERIAL DEPOSIT¹ FROM MODULE 6/11-7

<u>Element</u>	<u>Concentration² ($\mu\text{g}/25\text{ cm}^2$)</u>
Al	40
B	4
Ca	20
Cu	100
Fe	>1000
Mg	60
Mn	<10
Si	100
Ti	4

¹Sample was leached with HCl from a 25-cm² piece of backing material, then diluted for analysis.

²Concentration based on original sample before dilution.

higher pH of the product water accelerates the precipitation of iron hydrates. The small amount of copper present is surprising because previous analysis of the feedwater had not revealed the presence of any copper. However, at several times brass fittings were used in the feed lines and were eventually corroded away, and this could be the source of the copper.

Table XI shows the result of a spectrochemical analysis of material collected by scraping large areas of the membrane surface of module 6/11-7. A large percentage of iron is present in this sample, with copper, silica, chrome, manganese, aluminum, molybdenum, sodium, and nickel also present. The source of the copper is probably as explained above, while pump corrosion and pump packing could account for most of the remainder, except for the sodium. The presence of sodium in this large amount was to be expected, since the modules were tested on NaCl solution. While these elements are present in the sample in rather large quantities, they were present on the membrane surface in only minute quantities, which resulted in little flux decline as shown in Table IX. This material would have an adverse effect on long-term flux values if allowed to accumulate in the module. It is believed that all the iron present was a result of pump corrosion, not precipitation from the feedwater. Calcium sulfate does not appear in Table XI because this module did not have CaSO_4 precipitation present, as it was removed from the reverse osmosis unit when recovery levels were lower than that where CaSO_4 precipitation occurs. Measurements showed that the water-carrying capacity of the backing material was still within specifications for new material.

Two of the five modules were soaked in a neutralized BIZ* solution for 4 days in an attempt to clean the membrane of the fouling material previously described. The modules were drained and replaced in the same solution at least twice daily. After this soaking, the modules were again tested on 2000 mg/l NaCl solution. The results were as follows:

TABLE XI
SPECTROCHEMICAL ANALYSIS OF MEMBRANE DEPOSIT¹
FROM MODULE 6/11-7

<u>Element</u>	<u>Concentration (ppm)</u>
Al	2000
Ba	80
Co	100
Cr	>10,000
Cu	>10,000
Fe	>10,000
Mn	800
Mg	400
Mo	2,000
Na	>10,000
Ni	6,000
Pb	2,000
Si	>10,000
Sn	200
Ti	400
V	200

¹Loss of weight on ignition = 21%.

	Module 6853		Module 6/17-5	
	A	% Rej.	A	% Rej.
Original test data	1.48	96.1	2.43	93.8
After Site 1 testing	1.24	93.5	2.17	87.5
After BIZ soak	1.32	93.4	2.58	85.8

The BIZ cleaning evidently removed most of the fouling material present, which indicates it was probably pump packing or other foreign material, not precipitated salts.

The effect of the cleaning on Module 6853 is significant: the flux decline slope changed from -0.03, calculated after Norton operation, to -0.01, after cleaning, with little or no change in rejection. The results obtained for Module 6/17-5 seem to indicate a small leak. Spiral-wound modules exhibit compaction slopes of -0.01 to -0.03 when operated on reverse osmosis permeate.

When the modules were opened and samples of membrane were tested, the following results were obtained:

	Module 6853		Module 6/17-5	
	A	% Rej.	A	% Rej.
Sample No. 1	1.70	94.6	2.99	86.9
Sample No. 2	1.46	95.0	3.07	89.2

The membranes from Module 6853 seemed comparable to new membrane, while that from Module 6/17-5 showed some degradation. There was no apparent reason for the difference in the condition of the membranes. This work marked the completion of the evaluation of Site 1 modules.

The results of testing at Site 1 indicate that if the module fouling due to CaSO_4 precipitation and pump packing and corrosion products is prevented, flux decline values approaching those obtained in the laboratory as base-line values are obtained. The method of monitoring the last tube or tubes in a

unit for ΔP seems to be adequate for determining when excessive CaSO_4 precipitation is occurring. Low recovery flushes are capable of removing the CaSO_4 and restoring system performance.

No iron fouling of the membrane as a result of operating on this ferric acid mine drainage water was apparent during limited testing.

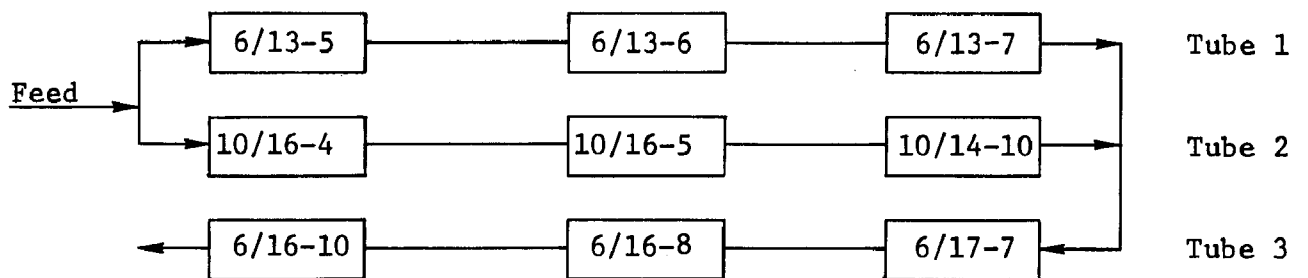
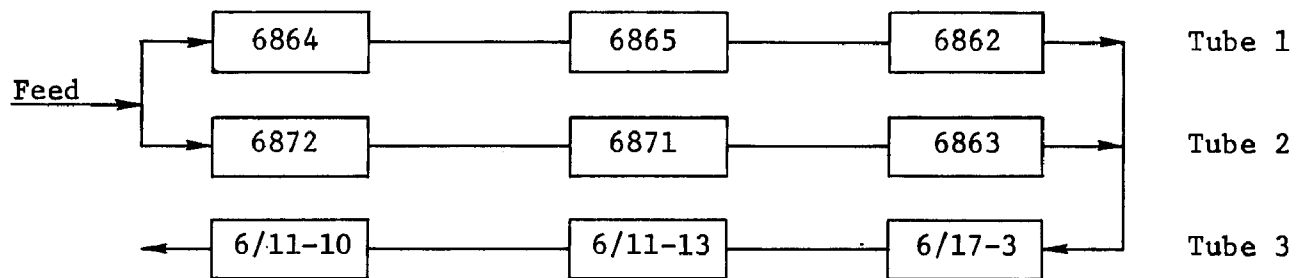
SECTION X

OPERATION AND RESULTS AT SITE 2

In order to investigate the performance of reverse osmosis on acid mine drainages containing a high ferrous iron/ferric iron ratio, the 4,000-gpd unit was moved to Site 2. The mine discharge at this site came from the Arkwright mine of the Christopher Coal Company, about ten miles southwest of Morgantown, West Virginia. The water was pumped from a flooded mine shaft and discharged into a small stream. A 500-gal tank was placed at the discharge, and the water was pumped from this tank through a small sand filter, then through cartridge filters, and from there to the high-pressure pump. The unit was placed inside a small portable storage building, and the pump was left outside. The pump used for this test was a Moyno 9S2 that had been reconditioned by the factory. All the wetted parts were 316 stainless steel or hard rubber. The unit was loaded with modules that had been in the unit during previous testing at Site 1. The loading arrangement is shown in Table XII.

Operations began on May 13 and were temporarily suspended on May 15 while a concrete pad for the Moyno pump, necessitated by excessive vibration, was installed. During this shutdown, the modules were fouled by material, identified as CaSO_4 , which precipitated from the water on standing. Another set of modules was loaded in the unit, and operation was resumed with one of the three tubes performing very poorly. Probing indicated that two of the modules were causing the poor performance, so they were replaced. Later the seal of the booster pump, used to deliver the water from the discharge to the Moyno pump, was corroded through and the unit again lost flow, with resulting precipitation and module fouling. This time, however, much of the material was removed with flushing. A new booster pump was installed, and the unit was then operated at approximately 50 percent recovery over the weekend, during which time a brass valve seat corroded causing low system flow and subsequent module fouling.

TABLE XII
MODULE LOADING ARRANGEMENT FOR 4,000-GPD UNIT AT SITE 2



Attempts to flush the unit were not successful, so it was mutually agreed by EPA and GES personnel to terminate testing at Site 2. The operational history of the unit for these two short runs is shown in Table XIII. Note that the ΔP increases with operating time and the product flux decreases, especially during the last run. Subsequent examination revealed the presence of substantial calcium sulfate precipitation, which accounts for each of these occurrences.

While the incidents described above might seem to indicate that the Site 2 test was not successful, this is not the case. The purpose of this test was to determine the difference between operating on high-ferrous-iron water and operating on the high-ferric-iron water at Norton. The feedwater at Site 2 was about 65 percent ferrous iron, but the total iron concentration was 30 times greater than at Norton. The magnesium, aluminum, calcium, and sulfate concentrations were also higher by factors of 15, 11, 6, and 15, respectively. This means that the second feedwater concentration could be compared with the concentrated brine at 92 percent recovery at Norton (see Table XIV). Further concentrating this type of water is difficult, primarily because of CaSO_4 solubility limitation. The achievement of recoveries of 50 percent during the short time the unit operated seemed possible, with precipitation of CaSO_4 occurring on the brine samples in several hours. This would indicate that this level of recovery is the maximum obtainable. Mean operating values and chemical analyses for this test are shown in Tables XV and XVI.

The salt rejections at this site were somewhat lower than those reported at Site 1. The higher osmotic pressure at Site 2 resulted in less product flow, whereas the salt flow was comparable to Site 1.

It seems apparent that reverse osmosis would not be feasible for demineralizing acid mine waters containing this amount of pollutants. The product water would not meet potable water standards unless treated further. This treatment would entail more than neutralizing and filtration, as was the case at Site 1. The relatively low recovery would create a serious brine disposal problem. Not enough testing was carried out to successfully predict membrane and module

TABLE XIII
OPERATIONAL HISTORY OF 4,000-GPD UNIT AT TEST SITE 2, 5/13/70 - 5/24/70

Date	Elapsed Time (hr)	Pressure (psi)	Recovery (percent)	Product Flow (gpm)	Brine Flow (gpm)	ΔP , Tube 3 (psi)	Temp (°F)	Unit Water Permeation Coefficient, $A \times 10^{-5}$ at 77°F
5/13/70	3.6	400	39.5	2.30	3.52	6.7	68	1.99
5/14/70	22.0	500	43.5	2.86	3.70	7.2	64	1.97
5/14/70	27.8	600	49.0	3.58	3.70	7.1	67	1.90
5/15/70	Unit down for repairs; modules fouled during this shutdown and were removed.							
5/21/70	New load of modules installed.							
5/22/70	2.3	600	51.4	3.84	3.64	7.5	68	1.99
5/23/70	17.8	600	50.0	3.45	3.46	7.0	62	1.95
5/24/70	42.5	600	42.5	3.23	3.57	8.0	64	1.76
5/24/70	46.4	600	46.4	3.13	3.57	8.0	70	1.55
5/24/70	Testing terminated.							

TABLE XIV
COMPARISON OF CHEMICAL ANALYSES¹ FOR OPERATION AT SITE 1 AND SITE 2

Sample Designation	Date	Percent Recovery	pH	Conductivity (μmhos/cm)	Acidity, pH 7.3	Ca as Ca (mg/l)	Mg as Mg (mg/l)	Fe (mg/l)	Al (mg/l)	SO ₄ (mg/l)
Site 1 (Norton, West Virginia)										
Raw feed	3/9/70	92.2	2.7	1200	585	102	32	100	33	819
Blended feed			2.2	4200	2478	475	180	465	151	4026
Brine			2.0	8500	5864	1050	395	1025	360	9555
Product			3.3	450	140	4	1.0	3.2	1.2	22
Percent rejection				92.91	96.64	99.48	99.65	99.57	99.53	99.68
Site 2 (Morgantown, West Virginia)										
Raw feed	5/14/70	43.5	2.5	7250	5120	625	480	1180/ 3360 ²	380	12,968
Brine			2.4	11,900	8890	1030	920	2214/ 5088 ²	700	20,475
Product			3.1	520	180	20	15	56/ 71 ²	12	273
Percent rejection				94.57	97.43	97.58	97.86	98.32	97.78	98.38

¹These analyses were made on-site by EPA personnel.

²Ferrous iron/total iron. Rejection based on total iron.

TABLE XV
MEAN OPERATING VALUES OF 4,000-GPD UNIT AT SITE 2, 5/13/70 - 5/24/70

Pressure	600 psi
Unit recovery	49.0 percent
Feed flow	6.95 gpm
Brine flow	3.54 gpm
Temperature	66°F
Product flow	3.41 gpm
Flux rate, 77°F	13.11 g/f ² d
Tube 3 ΔP	11.0 psi
Length of test	46.4 hr

TABLE XVI
CHEMICAL ANALYSES¹ FOR 4,000-GPD UNIT OPERATION AT SITE 2, 5/13/70 - 5/24/70

Sample Designation	Percent Recovery	pH	Conductivity (μmhos/cm)	Acidity, pH 7.3	Dissolved Oxygen	Ca as Ca	Mg as Mg	Fe ² (mg/l)	Al (mg/l)	SO ₄ (mg/l)
Raw feed	49.0	2.24	7,040	5,230	4.5	526	420	1,280/ 2,300	317	10,920
Brine			11,840	10,050	4.5	933	810	2,450/ 4,460	598	20,480
Product		2.00	423	148	0	9.6	7.6	29/ 39	5.0	191
Percent rejection		3.14	95.52	98.06	0	98.68	98.76	98.84	98.91	98.78

¹These analyses were made on-site by EPA personnel.

²Ferrous iron/total iron. Rejection based on total iron.

TABLE XVII
RESULTS OF POSTOPERATIONAL TESTING OF MODULES OPERATED AT SITE 2

Module	Original Test Data		6/18/70 Test Data	
	A	Percent Rej.	A	Percent Rej.
6844	1.58	95.6	1.42	95.0
6851	1.41	96.5	1.38	95.6
6863 ¹	1.50	95.8	1.24	92.3
6871 ¹	1.47	96.7	1.40	91.9
6872 ¹	1.56	96.4	1.24	93.8
6/11-10 ¹	2.08	95.3	1.82	80.0
6/11-13 ¹	2.10	95.1	1.87	81.5
6/13-3 ²	2.13	91.3	1.84	90.9
6/13-7	2.51	91.3	1.25	48.6
6/16-4A ²	2.04	90.4	1.91	92.7
6/16-8	2.12	95.1	1.58	91.1
6/16-10	2.01	93.4	1.44	70.2
6/17-3 ¹	2.60	93.1	2.44	85.1
6/17-7	2.63	91.9	1.95	89.1

¹This module was operated at Site 1 for approximately 250 hr before the unit was moved to Site 2.

²This module was operated at Site 2 for less than 24 hr. It was removed from unit because of poor performance.

NOTE: All tests were run on 2000 mg/l NaCl solution.

A = $\text{g/cm}^2\text{-sec-atm} \times 10^{-5}$; A of $1 \times 10^{-5} = 8.65$ gpd at 600 psi net at 77°F.

lifetimes. Fouling caused by the ferrous iron present in the acid mine water was not apparent, but due to the short exposure times and massive calcium sulfate fouling, no definitive statement regarding iron fouling can be made.

The EPA has prepared an in-house report (Ref. 9) in which the performance (A values and ΔP) of the individual tubes is reported for the testing at this site.

Since the recovery level was limited at Site 2, it was decided to select a third site, where the water would have a high ferrous iron/ferric iron ratio but a total iron content similar to that at Site 1.

Fourteen of the modules used in this test were returned to San Diego for postoperational evaluation and cleaning. The modules were tested for rejection and water flux on 2000 mg/l NaCl solution. The results of these tests are shown in Table XVII, along with the original quality control test data. All modules experienced a decline in "A" values and also in rejection, with a few undergoing greater declines than others.

Module 6/13-7 is an extreme case, but this module lost the brine seal, probably during loading, and was virtually full of precipitated material. Samples of this material were collected from the pressure vessel when the module was removed and, along with a sample of the precipitate in a brine sample at 50 percent recovery, were analyzed. Results of these analyses are shown in Table XVIII. In both cases, the material is almost pure gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), which again points out that the limiting substance governing recovery was the calcium sulfate, rather than iron hydrate.

Two of the returned modules (6/11-10 and 6/13-7) were run on acidified (pH 3 to 4) San Diego tap water for two days. These modules were known to be loaded with calcium sulfate, with module 6/13-7 being much worse in this respect than module 6/11-10. The modules were then tested on 2000 mg/l NaCl solution. Results obtained were as follows:

TABLE XVIII
CHEMICAL ANALYSIS OF SAMPLES FROM SITE 2

Solid Material Collected from Tube No. 3, 5/17/70

	<u>Concentration (percent)</u>
CO ₃	<1
Fe	<0.3
Mg	1.6
SO ₄	60.8
Ca	21.7

Precipitate from Brine Sample, 5/24/70, ~50 Percent Recovery

	<u>Concentration (percent)</u>
CO ₃	<0.5
Fe	0.70
Mg	<0.5
SO ₄ ¹	63.0
Ca ¹	25.0
Al	<0.3
Clay and silica	1.5

¹Present in stoichiometric amounts found in CaSO₄.

	Module 6/11-10		Module 6/13-7	
	A	Percent Rej.	A	Percent Rej.
Original test data	2.08	95.3	2.51	91.3
After Site 2 operation	1.82	80.0	1.25	48.6
After low-pH flush	1.92	80.7	2.15	87.4

Module 6/13-7 had been severely packed and weighed two or three times more than a new module. As evidenced by the test results, much of this material was removed by the flushing. The low rejection value after Site 2 operation was caused by the extremely high boundary layer concentration in the brine channels, and as this concentration was reduced, the rejection improved. Further flushing might have increased the water flux and rejection even more, but no further flushing was performed.

Modules 6/11-10 and 6/13-7 were opened, and the remaining CaSO_4 was removed by rinsing with distilled water. Membrane samples were taken for testing in the membrane test cells using 2000 mg/l NaCl solution. The results of these tests were as follows:

	Module 6/11-10		Module 6/13-7	
	A	Percent Rej.	A	Percent Rej.
Sample No. 1	2.77	85.66	2.30	91.45
Sample No. 2	2.47	87.42	--	--

These results indicate that the CaSO_4 present in the modules, even after flushing, affects rejection values due to the boundary layer concentration. The membrane from Module 6/13-7 has rejection values equivalent to the original module test data. Module 6/11-10 results indicate a slight deterioration of the membrane as compared with the original test data. This module had been operated at Site 1 for 250 hr and was in the unit when it was moved to Site 2. No reason for this decrease in performance is apparent.

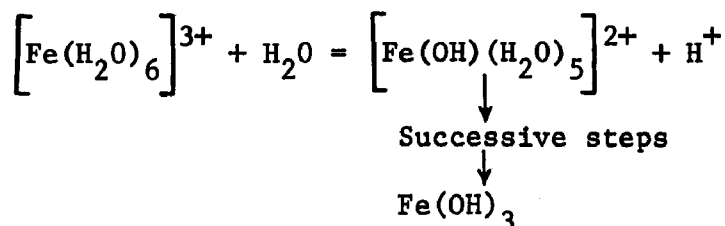
No further work was performed on any of the remaining modules.

SECTION XI

OPERATION AND RESULTS AT SITE 3

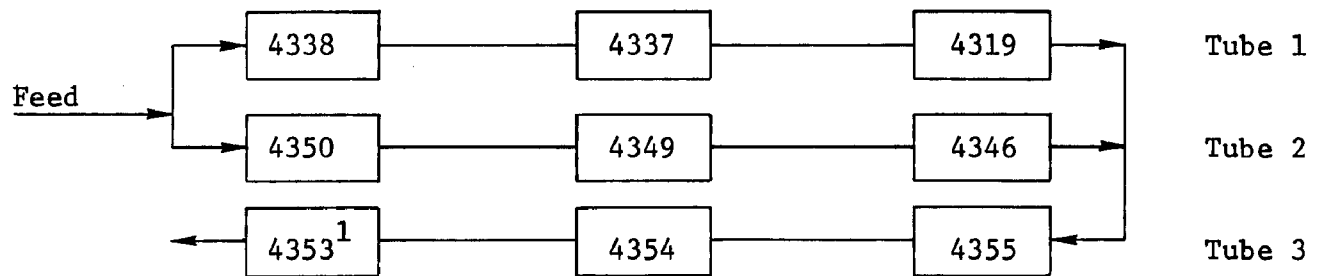
The site selected for the third series of tests was about four miles east of Ebensburg, Pennsylvania. The water was discharged from active mines of the Cambria Division of the Bethlehem Mines Corporation. The discharge at this location was over 4 mgd and contained about 130 mg/l of iron, about 80 percent of which was ferrous.

The 4,000-gpd unit was moved to this site and set up, with the high-pressure pump, 500-gal. feed tank, filters, etc., in an arrangement similar to that used at Site 2. Modules were loaded as shown in Table XIX. Provisions were made to add chemicals to the feed to the reverse osmosis unit. In reviewing data from past successful operations at various locations, it was noted that in all cases, the pH of the feed was below 3, whereas at Site 3, the mine discharge pH was 3.6. No iron precipitation problems had been encountered in any Gulf Environmental Systems previous runs, but other investigations (Ref. 2) had encountered precipitation, and it was observed that this appeared to occur when operating on feeds with a pH above 3. The reaction which results in iron precipitation is



The pH at which there is 50 percent conversion of the $\left[\text{Fe}(\text{H}_2\text{O})_6 \right]^{3+}$ to $\left[\text{Fe}(\text{OH})(\text{H}_2\text{O})_5 \right]^{2+}$ is 2.8, which is the pK value. Below pH 2.8, there is predominantly the trivalent ion, which is highly soluble; above 2.8; there is more of the other species, which results in the favoring of the precipitation of

TABLE XIX
MODULE LOADING ARRANGEMENT FOR 4,000-GPD UNIT AT SITE 3



¹4353 replaced by 4320 on 8/7/70.
 4320 replaced by 4327 on 8/27/70.
 4327 replaced by 4328 on 9/4/70.

$\text{Fe}(\text{OH})_3$. Also, the conversion rate of ferrous (soluble) to ferric iron increases rapidly with increasing pH. It was felt that since this particular discharge was over pH 3, precipitation of iron with the resulting fouling of the modules was a distinct possibility. In order to evaluate the effects of pH, four test runs were conducted at this site. The principal parameters of these runs were:

- Run 1 Sulfuric acid added to feed to lower pH. Unit operated at ~85 percent recovery with brine recycle.
- Run 2 Nothing added.
Unit operated at ~85 percent recovery with brine recycle.
- Run 3 Caustic added to feed to increase pH. Unit operated at ~85 percent recovery with brine recycle.
- Run 4 Nothing added.
Unit operated at ~50 percent recovery with no brine recycle.

In run 1, sulfuric acid was initially added to lower the blended feed pH to 2.5; acid addition was slowly decreased until the blended feed pH was 2.8. The unit was operated at approximately 85 percent recovery for 200 hr. Results of chemical analysis for this run are shown in Table XX and the operational history is shown in Table XXI.

There was an increase of ΔP in the last pressure vessel, accompanied by a decrease in product flow, but since this condition could be corrected by operating at 50 percent recovery for ~3 hr as shown in Table XXI, calcium sulfate precipitation, not iron precipitation, was thought to be the cause.

An inhouse report has been prepared by the EPA (Ref. 10) in which the performance ("A" values and ΔP) of the individual tubes is reported for the testing at this site.

TABLE XX
CHEMICAL ANALYSES¹ FOR 4,000-GPD UNIT OPERATED AT SITE 3, RUN 1

Sample Designation	Date	Elapsed Time (hr)	pH	Conductivity (μmhos/cm)	Acidity, pH 7.3	Ca as Ca (mg/l)	Mg as Mg (mg/l)	Al (mg/l)	Total Fe (mg/l)	Ferrous Iron (mg/l)	SO ₄ (mg/l)	Dissolved Oxygen (mg/l)	Percent Recovery
Raw feed	7/30/70	4.0	3.7	1,600	410	170	48	28	124	101	1,638	2	85.5
Acidified feed			2.7	2,300	560	168	48	27	124	101	1,638		
Blended feed			2.4	6,400	1,680	600	190	101	450	354	6,552		
Brine			2.2	9,700	3,280	1,008	336	180	840	624	12,012		
Product			3.3	205	70	1.7	0.6	0.6	0.8	<1	32		
Percent rejection				97.45	97.18	99.79	99.77	99.57	99.88	>99.8	99.66		
Raw feed	7/31/70	23.5	3.6	1,500	390	168	49	29	128	101	1,665		84.8
Acidified feed			2.7	2,000	540	160	50	28	124	101	1,583		
Blended feed			2.4	5,000	1,540	600	185	100	440	325	6,006		
Brine			2.2	8,000	3,020	1,020	324	168	800	574	11,193		
Product			3.5	148	70	1.6	0.6	0.6	0.8	<1	25		
Percent rejection				97.72	96.93	99.80	99.76	99.55	99.87	>99.8	99.71		
Raw feed	8/3/70	96.0	3.6	1,600	No value reported	200	59	34	156	101	1,638		82.4
Acidified feed			2.75	2,150		196	57	33	188	101	1,638		
Blended feed			2.4	4,900		600	188	106	450	292	4,914		
Brine			2.3	7,300		900	340	192	750	522	8,463		
Product			3.7	102		1.2	0.3	1	2	<2	6		
Percent rejection				98.33		99.84	99.89	99.33	99.67	99.26	99.91		
Raw feed	8/3/70	100.0	3.6	1,480	390	200	57	34	136	101	1,638	2	82.5
Acidified feed			3.1	1,750	400	196	57	35	132	101	1,747		
Blended feed			2.8	4,200	1,160	600	186	104	420	281	4,914		
Brine			2.6	6,800	2,150	900	340	188	700	511	8,463	2	
Product			4.2	63	50	1	0.3	1	2	<2	12	2	
Percent rejection				98.85	96.98	99.87	99.89	99.32	99.64	99.24	99.82		
Raw feed	8/6/70	168.0	3.6	1,460	380	200	55	33	136	101	1,638	2	84.2
Acidified feed			3.1	1,650	420	200	54	33	126	101	1,638		
Blended feed			2.8	4,000	1,200	610	208	104	425	320	4,914		
Brine			2.6	6,200	2,130	870	360	180	700	523	8,463	2	
Product			4.3	60	50	1.1	0.3	1	2	<2	12	2	
Percent rejection				98.82	97.00	99.85	99.89	99.30	99.64	99.30	99.82		
Raw feed	8/7/70	191.0	3.6	1,440	390	190	55	33	140	101	1,638	2	84.7
Acidified feed			3.1	1,600	430	190	54	32	130	101	1,638		
Blended feed			2.9	3,900	1,210	600	204	103	440	308	4,914		
Brine			2.7	5,900	2,010	880	304	168	700	510	8,327	2	
Product			4.5	57	50	1	0.3	1	2	<2	11	2	
Percent rejection				98.84	96.89	99.86	99.88	99.26	99.65	98.53	99.84		

¹These analyses were made by EPA personnel.

TABLE XXI
OPERATIONAL HISTORY OF 4,000-GPD UNIT, TEST SITE 3, RUN 1

Date	Elapsed Time (hr)	Pressure (psi)	Recovery (percent)	Product Flow (gpm)	Brine Flow (gpm)	ΔP , Tube 3 (psi)	Temp (°F)	Unit Water Permeation Coefficient, $A \times 10^{-5}$ at 77°F	Raw Feed pH	Blended Feed pH
7/30/70	0.25	400	48.3	3.33	3.57	8.8	59.5	2.76	3.5	2.5
	1.5	420	81.3	3.45	0.80	10.8	62.5	2.76	3.6	2.5
	4.0	410	85.5	3.17	0.54	10.8	64.5	2.68	3.7	2.4
	7.5	410	85.7	3.22	0.54	11.2	64.5	2.69	3.7	2.4
7/31/70	23.5	395	84.8	3.13	0.56	10.6	63.5	2.70	3.6	2.4
	28.0	390	84.9	3.13	0.56	10.4	66.5	2.58	3.6	2.5
8/1/70	57.7	390	84.3	3.06	0.55	11.1	65.0	2.60	3.7	2.4
8/3/70	96.0	390	82.4	2.63	0.56	12.5	62.5	2.32	3.6	2.4
Ran for 2-3/4 hr at 50 percent recovery.										
8/3/70	100.0	400	82.5	3.12	0.66	10.3	66.0	2.45	3.6	2.8
8/4/70	120.0	375	81.2	2.86	0.66	9.2	59.0	2.67	3.6	2.8
	125.0	405	83.4	3.12	0.67	10.4	66.5	2.38	3.6	2.7
8/5/70	144.0	400	83.7	2.86	0.56	10.0	59.5	2.46	3.6	3.1
	147.0	400	84.0	2.94	0.56	10.2	67.5	2.27	3.6	2.7
	150.0	400	84.0	2.94	0.56	10.3	67.5	2.27	3.6	2.6
8/6/70	168.0	400	84.2	2.70	0.55	11.1	60.5	2.32	3.6	2.8
	171.0	400	83.4	2.70	0.56	11.5	62.0	2.27	3.6	2.7
	174.0	400	83.6	2.79	0.56	11.8	66.0	2.20	3.6	2.7
8/7/70	191.0	400	74.0	2.50	0.55	12.8	61.0	2.12	3.6	2.9
Unit down, last module of Tube 3 removed and replaced with a new one.										
Calcium sulfate present in Tube 3. System flushed with acidified permeate.										

One module, No. 4353, was removed at the end of 200 hr. An increase in the ΔP of the last pressure vessel had occurred, and it was assumed that this module would have precipitated material present. When the module was opened, it was indeed full of white, flaky precipitate. Analysis of this material showed it to be 23.7 percent calcium and 55.1 percent sulfate, with a 22.6 percent loss of weight at 600°C. The theoretical amount of CaSO_4 in gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) is 79.1 percent. Thus, this material was almost pure gypsum. The precipitate could easily be washed from the membrane, which is what takes place in the unit when the recovery level is lowered. No presence of iron precipitate was noted.

With the relatively small reverse osmosis unit, a large amount of the brine is recycled in order to achieve the high recovery. This recycled brine is of a lower pH than the raw feed, so the blended feed is still lower in pH than the raw feed. During run 2, the unit was operated in this manner with no feed additives for 86 hr.

Chemical analyses and operational history for this run are shown in Tables XXII and XXIII. Note that although there was an increase in the ΔP of Tube 3, the unit water flux remained relatively constant. In fact, after each shutdown due to pump failure, the "A" value increased. This phenomenon has been noted many times in the operation of reverse osmosis units containing spiral-wound modules and is not readily explained. No iron precipitation was noted during this run.

In run 3 an attempt was made to bring the blended feed pH up to the raw feed pH by adding sodium hydroxide to the feed stream. This resulted in mass precipitation of iron in the sand filter, which plugged rapidly. The chemical analyses are shown in Table XXIV and the operational history is shown in Table XXV. Because of the massive amounts of iron precipitation throughout the system, the only conclusion reached was that if the pH of this feed is increased by caustic, iron precipitation will surely occur.

TABLE XXII
CHEMICAL ANALYSES¹ FOR 4,000-GPD UNIT OPERATED AT SITE 3, RUN 2

Sample Designation	Date	Elapsed Time (hr)	pH	Conductivity (μmhos/cm)	Acidity pH 7.3	Ca as Ca (mg/l)	Mg as Mg (mg/l)	Al (mg/l)	Total Fe (mg/l)	Ferrous Iron (mg/l)	SO ₄ (mg/l)	Dissolved Oxygen (mg/l)	Percent Recovery
Raw feed	8/11/70	3.0	3.7	1460	390	185	46	27	114	101	1583	2	84.3
Blended feed			3.1	3900	1200	650	148	85	390	286	4505		
Brine			2.9	7200	2160	1200	300	180	690	545	9282	2	
Product			4.6	78	50	1.5	0.6	1	2	>2	21	2	
Percent rejection				98.59	97.02	99.84	99.73	99.24	99.63	98.56	99.70		
Raw feed	8/17/70	2.8	3.6	1480	400	220	69	40	140	101	1693	2	84.5
Blended feed			3.1	4200	1160	680	200	124	460	292	6006		
Brine			2.9	6800	2280	1260	425	255	900	570	10238	2	
Product			4.5	108	60	2.4	1.6	1	3	>2	27.3	2	
Percent rejection				98.03	96.51	99.25	99.49	99.47	99.56	98.61	99.66		
Raw feed	8/18/70	24.7	3.6	1490	400	230	69	38	154	101	1747	2	84.4
Blended feed			3.1	4100	1220	600	222	114	450	314	4914		
Brine			2.9	7600	2200	1100	425	200	890	600	10238	2	
Product			4.4	91	60	1.8	1.2	1	2	>2	1	2	
Percent rejection				98.44	96.49	99.79	99.63	99.36	99.70	98.69	99.72		
Raw feed	8/19/70	48.5	3.7	1460	380	210	67	38	150	96	1693	2	82.8
Blended feed			3.3	4000	1090	630	216	110	470	292	5324		
Brine			3.1	6400	2020	1170	390	225	900	567	10238	2	
Product			4.7	81	60	1.6	1.0	1	1.7	< 2	23	2	
Percent rejection				97.62	96.14	99.82	99.67	99.40	99.75	98.60	99.70		
Raw feed	8/20/70	66.0	3.6	1460	370	210	66	40	170	96	1693	2	84.3
Blended feed			3.1	4000	1150	640	204	120	490	292	4778		
Brine			3.0	6600	2210	1180	380	230	900	534	10101	2	
Product			4.7	84	60	1.8	1.2	1.2	1.8	< 2	25	2	
Percent rejection				97.60	96.43	99.80	99.59	99.31	99.74	98.55	99.66		
Raw feed	8/21/70	86.3	3.6	1460	370	200	66	40	168	96	1747		84.4
Blended feed			3.1	3800	1140	690	222	116	500	275	5870		
Brine			3.0	6000	2010	1280	390	220	950	500	10238		
Product			4.6	71	60	1.6	1	1.2	1.1	< 1	22		
Percent rejection				98.55	96.19	99.84	99.67	99.29	99.85	98.45	99.73		

¹These analyses were made by EPA personnel.

TABLE XXIII
OPERATIONAL HISTORY OF 4,000-GPD UNIT TEST SITE 3, RUN 2

Date	Elapsed Time (hr)	Pressure (psi)	Recovery (percent)	Product Flow (gpm)	Brine Flow (gpm)	ΔP , Tube 3 (psi)	Temp (°F)	Unit Water Permeation Coefficient, $A \times 10^{-5}$ at 77°F	Raw Feed pH	Blended Feed pH
8/11/70	0.5	400	84.7	3.33	0.60	9.2	59.5	2.85	3.6	3.1
	3.0	400	84.3	3.33	0.60	9.2	67.0	2.57	3.7	3.1
	6.5	400	86.0	3.33	0.55	9.2	68.0	2.57	3.6	3.1
Unit down; main high-pressure-pump failure.										
8/17/70	0.7	400	84.5	3.33	0.62	8.7	66.5	2.56	3.6	3.1
	2.8	400	84.5	3.33	0.62	8.8	67.5	2.55	3.6	3.1
	5.9	400	84.0	3.23	0.62	8.8	67.0	2.49	3.6	3.1
Unit down; main high-pressure-pump failure.										
8/18/70	22.9	400	84.0	3.22	0.62	8.9	64.0	2.67	3.6	2.5
	24.7	400	84.4	3.22	0.60	8.9	67.5	2.49	3.6	3.1
	27.8	400	84.0	3.13	0.60	8.9	67.0	2.44	3.7	3.2
Unit down; main high-pressure-pump failure.										
8/19/70	46.5	400	83.9	3.33	0.64	8.9	64.5	2.63	3.7	3.3
	48.5	400	82.8	3.13	0.65	8.9	65.0	2.48	3.7	3.3
	51.0	400	83.8	3.18	0.62	8.9	68.0	2.41	3.6	3.3
Unit down; main high-pressure-pump failure.										
8/20/70	63.5	400	83.9	3.13	0.61	8.9	62.5	2.58	3.6	3.2
	66.0	400	84.3	3.13	0.59	9.1	64.5	2.51	3.6	3.1
	68.5	400	85.2	3.13	0.55	9.2	67.0	2.41	---	---
Unit down; main high-pressure-pump failure.										
8/21/70	70.4	400	83.5	3.22	0.64	9.2	64.0	2.60	---	---
	72.5	400	83.4	3.22	0.65	9.3	65.0	2.56	---	---
	86.3	400	84.4	3.03	0.57	9.8	60.0	2.60	---	---
Unit recovery lowered to 50 percent; unit then flushed with acidified permeate and shut down.										

TABLE XXIV
CHEMICAL ANALYSES¹ FOR 4,000-GPD UNIT OPERATED AT SITE 3, RUN 3

Sample Designation	Date	Elapsed Time (hr)	pH	Conductivity (μmhos/cm)	Acidity pH 7.3	Ca as Ca (mg/l)	Mg as Mg (mg/l)	Al (mg/l)	Total Fe (mg/l)	Ferrous Iron (mg/l)	SO ₄ (mg/l)	Dissolved Oxygen (mg/l)	Percent Recovery
Raw feed	8/25/70	16.4	3.8	1460	350	225	62	38	165	96	1583	2	82.6
Blended feed			3.3	3400	990	660	180	104	400	253	3822		
Brine			3.1	5300	1790	1188	350	200	740	478	7644	2	
Product			4.7	90	40	2	1	1.4	1	< 1	27.3	2	
Percent rejection				97.93	97.12	99.78	99.62	99.08	99.82	99.18	99.52		
Raw feed	8/26/70	29.6	3.6	1460	390	200	58	32	136	107	1474	2	83.0
Caustic feed			4.1	1500	360	184	59	38	150	101	1529		
Blended feed			3.6	4000	870	600	195	78	425	281	3549		
Brine			3.4	6400	1700	960	380	145	765	551	7098	3.5	
Product			4.6	105	60	2.2	1.2	1.4	1.2	< 1	27	4.5	
Percent rejection				97.98	95.33	99.72	99.58	98.74	99.80	98.56	99.49		

¹These analyses were made by EPA personnel.

TABLE XXV
OPERATIONAL HISTORY OF 4,000-GPD UNIT, TEST SITE 3, RUN 3

Date	Elapsed Time (hr)	Pressure (psi)	Recovery (percent)	Product Flow (gpm)	Brine Flow (gpm)	ΔP , Tube 3 (psi)	Temp (°F)	Unit Water Permeation Coefficient, $A \times 10^{-5}$ at 77°F	Raw Feed pH	Blended Feed pH
8/25/70	1.5	400	79.9	3.22	0.90	8.5	64.0	2.59	--	--
	16.4	400	82.6	3.13	0.66	8.5	59.0	2.68	3.6	3.3
	17.6	Started adding caustic.								
8/26/70	24.1	400	83.5	3.33	0.67	8.1	65.0	2.61	3.7	3.3
	27.7	400	83.4	3.22	0.65	8.1	65.5	2.52	3.7	3.6
	29.6	400	83.0	3.13	0.64	8.4	64.0	2.52	3.6	3.6
	Run terminated; system full of iron and calcium sulfate precipitate. Module removed from Tube 3 and replaced with new one. System flushed with acidified permeate.									

The Moyno high-pressure pump had failed just prior to this run and was repaired using a precipitation-hardening stainless steel in place of the 316 stainless steel previously used. It was felt that the 316 stainless steel did not possess adequate tensile strength for this use.

The final test run was conducted with no recycled brine and no chemical addition, so that the natural raw feed pH was not changed. The system performed very satisfactorily, as expected at the low recovery level, with no plugging or fouling observed. This is different from the results of runs 1 and 2, where fouling is quite evident. The results of chemical analyses for this run are shown in Table XXVI, and the operational history is given in Table XXVII. The conductivity rejection averaged 98 percent or better throughout the tests performed at Site 3, and the salt rejections were greater than 99 percent except for aluminum.

The presence of 2 mg/l of dissolved oxygen in the feedwater had no apparent effect on the reverse osmosis operation at this test site. The dissolved oxygen was present in both the brine and product in amounts equal to the feed, which would indicate no rejection for this module.

Module 4327 was removed at the end of this run and returned to San Diego for testing. During shipment, the shipping container was broken and the module was fairly dry upon arrival. The module was tested for rejection and water flux, with the following results:

	Module No. 4327	
	A	Percent Rej. on 2000 mg/l NaCl Solution
Original test data	2.05	95.48
After Site 3 operation	2.19	92.58

After this test the module was opened, and inspection revealed a very small amount of foreign material present on the membrane which in no way affected performance. Not enough of the material could be collected for analysis, but it was determined that iron was present. However, it is not clear why traces

TABLE XXVI
CHEMICAL ANALYSES¹ FOR 4,000-GPD-UNIT OPERATION AT SITE 3, RUN 4

Sample Designation	Date	Elapsed Time (hr)	pH	Conductivity (μmhos/cm)	Acidity, pH 7.3	Ca as Ca (mg/l)	Mg as Mg (mg/l)	Al (mg/l)	Total Fe (mg/l)	Ferrous Iron (mg/l)	SO ₄ (mg/l)	Dissolved Oxygen (mg/l)	Percent Recovery
Raw feed	8/31/70	2.5	3.9	1,000	370	170	50	30	130	96	1,297	2	54.8
Brine			3.6	1,900	740	360	110	61	264	219	2,620	2	
Product			5.0	33	45	1.2	0.4	1.2	0.4	<1	0	2	
Percent rejection				97.72	91.89	99.55	99.50	97.36	99.80	99.05	100		
Raw feed	9/1/70	24.4	3.5	1,120	430	160	50	30	128	96	1,338	2	53.2
Brine			3.3	2,250	780	340	115	64	246	208	2,948	2	
Product			4.7	26	60	1	0.4	1.2	0.4	<1	0	2	
Percent rejection				98.46	90.08	99.60	99.52	97.45	99.79	99.01	100		
Raw feed	9/2/70	48.1	3.5	1,140	400	160	52	32	126	96	1,297	2	52.3
Brine			3.3	2,250	760	320	110	64	242	208	2,948	2	
Product			4.8	25	60	1	0.3	1.1	0.4	<1	0	2	
Percent rejection				98.53	89.66	99.58	99.63	97.71	99.78	99.01	100		
Raw feed	9/3/70	71.9	3.6	1,160	380	160	51	28	128	96	1,229	2	53.9
Brine			3.4	2,100	820	320	115	56	246	219	2,893	2	
Product			4.8	26	50	1	0.2	1.0	0.4	<1	0	2	
Percent rejection				98.40	91.67	99.58	99.76	97.62	99.79	99.05	100		
Raw feed	9/4/70	92.0	3.6	1,160	380	160	52	30	126	96	1,338	2	52.3
Brine			3.4	2,300	750	320	105	60	240	208	3,058	2	
Product			4.9	26	50	1	0.3	1.2	0.4	<1	0	2	
Percent rejection				98.50	91.15	99.58	99.62	97.33	99.78	99.01	100		

¹These analyses were made by EPA personnel.

TABLE XXVII
OPERATIONAL HISTORY OF 4,000-GPD UNIT AT TEST SITE 3, RUN 4

Date	Elapsed Time (hr)	Pressure (psi)	Recovery (percent)	Product Flow (gpm)	Brine Flow (gpm)	ΔP , Tube 3 (psi)	Temp ($^{\circ}F$)	Unit Water Permeation Coefficient, $A \times 10^{-5}$ at $77^{\circ}F$	Raw Feed pH
8/31/70	0.5	405	54.8	3.46	2.86	6.9	62.0	2.56	3.8
	2.5	405	54.8	3.46	2.86	6.8	63.0	2.52	3.9
	7.9	405	53.8	3.33	2.86	6.9	61.5	2.51	3.6
9/1/70	21.4	400	53.8	3.33	2.86	6.9	54.0	2.88	3.5
	24.4	400	53.2	3.33	2.94	7.3	62.0	2.52	3.5
	27.4	400	52.9	3.33	2.99	7.4	63.0	2.48	3.5
	30.3	400	52.5	3.33	3.03	7.4	62	2.52	3.5
9/2/70	45.3	400	52.9	3.33	2.99	7.4	55	2.84	3.5
	48.1	400	52.3	3.33	3.03	7.4	57	2.75	3.5
	52.0	400	52.3	3.33	3.03	7.4	63	2.47	3.5
9/3/70	69.4	400	52.3	3.33	3.03	7.4	56	2.79	3.6
	71.9	400	53.9	3.39	3.03	7.4	62	2.56	3.6
	75.2	400	52.3	3.39	3.03	7.4	63	2.51	3.6
9/4/70	92.0	400	52.3	3.33	3.03	7.4	60	2.61	3.6

Run terminated. Module removed from Tube 3 and replaced with new one.

Unit flushed with acidified permeate.

of iron occurred. A certain amount of the mine drainage water was presumably present in the feed channels of the module when it was shipped. Dissolved iron could have been present then, but it may have been deposited when the container broke and the module drained. Since the module was allowed to dry out somewhat, no definite conclusion can be drawn, but the amount of iron was negligible.

Testing at Site 3 concluded the field testing portion of the contract. The 4,000-gpd unit, pumps, tanks, etc., were then moved back to the Norton, West Virginia, site where they will be operated as desired by the EPA.

The test program demonstrated the ability to operate a reverse osmosis system without iron fouling on predominately ferrous iron feed by observing proper pH control and calcium sulfate limitations. Product water quality was good and would meet potable water standards with lime neutralization and filtration.

The high pressure pump performed adequately during this last run, but not enough hours were accumulated to fully evaluate the precipitation hardening steel used for the connecting rod. In any event, this type of pump does not seem adequate for reverse osmosis operation when the feed is acid mine water.

SECTION XII
DISCUSSION OF TEST RESULTS

If the system at Site 1 had been kept free of CaSO_4 precipitation by operating at a recovery level lower than that at which precipitation occurs, and if the system had been equipped with a high-pressure pump suitable for use on acid mine drainage that did not corrode or allow packing to work its way into the process stream, the flux decline values would be similar to those obtained in the laboratory for base-line compaction. For this type of feedwater, module lifetimes and costs can be extrapolated. Reference 5 contains a parametric cost study based on these conditions.

Operations at Site 3 were of too short a duration to make those extrapolations, while the results of the operation at Site 2 cannot be considered because of the CaSO_4 precipitation and pump failures. Treatment of acid mine water by reverse osmosis is clearly feasible at Site 1, probably feasible at Site 3, and not feasible at Site 2.

No difference in reverse osmosis operation was noted when operating on predominantly ferrous or ferric iron acid mine waters. Iron fouling of the membranes as a result of iron precipitation from the acid mine water was not a critical factor during the short-term runs at the three test sites. Figure 3 is a log-log plot of the water permeation coefficient, "A," for the 10,000-gpd unit at Site 1 and the 4,000-gpd unit at Sites 2 and 3. Much of the scatter of these data is caused by apparent discrepancies in the temperature - water flux normalization values used in these tests.

Membrane fouling and compaction usually appear as a straight line when plotted in this manner. The fact that three of the lines plotted tail off rather sharply suggests that module plugging and/or mass fouling was taking place. Pressure drop readings taken at these times indicate plugging was indeed occurring. Modules removed at the end of these runs at Site 2 and Site 3 had calcium sulfate precipitation present.

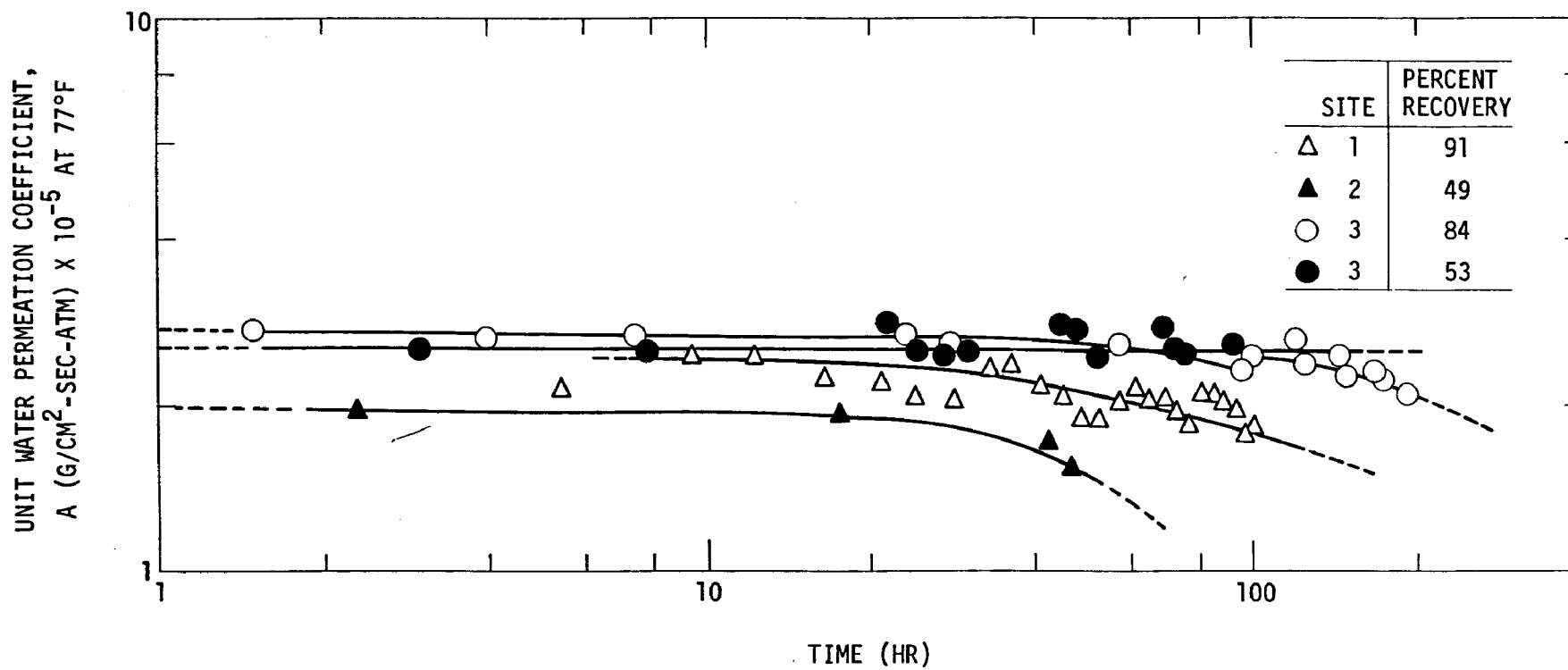


Fig. 3. Membrane performance at Sites 1, 2, and 3

Calcium sulfate precipitation was a distinct problem at all three test sites, with the recovery level being the controlling parameter. These levels are discussed in Section XIII, and further long-term testing would be required to establish the validity of the assumptions made there.

The product water at Sites 1 and 3 would meet potable water standards after being neutralized with lime and filtered. The product water at Site 2 would require even further treatment to meet the potable standards.

The presence of dissolved oxygen at Sites 2 and 3 had no effect on the reverse osmosis operation.

SECTION XIII
CALCIUM SULFATE SOLUBILITY LIMITATION

It was assumed that iron concentration in the brine with resulting precipitation and membrane fouling would be the limiting factor in the total water recovery of a reverse osmosis system operating on acid mine drainage. This has not been the case at locations where the spiral-wound ROGA[®] modules have been operated. Another assumption has been that it is more difficult to operate on ferrous (Fe^{++}) iron water than on ferric (Fe^{+++}) iron water. Early tests in Pennsylvania (Ref. 8) did not support this contention, and no iron fouling was observed. Indeed, the one limiting factor seems to be the calcium sulfate concentration. While tests have shown that the CaSO_4 solubility limit can be exceeded in the concentrated brine, there are indications that a fairly definite limit exists.

Table XXVIII compares results of tests conducted on three very different acid mine drainage waters and a calculated recovery for one of them. Operations at Norton were conducted with no major fouling or plugging of the modules as long as the recovery level was kept below 90 percent. However, if the recovery was allowed to increase to 92.3 percent through only a slight change in operating parameters on that particular reverse osmosis unit (equal to an increase in feed concentration from 10 to 13), plugging of the downstream tube of modules due to CaSO_4 deposition was observed. Tests at the second test site near Morgantown, West Virginia, at the Arkwright Mine, where the discharge was approximately ten times more concentrated than the feed at Norton, resulted in several modules being completely plugged with calcium sulfate when the unit was operated at 50 percent recovery. Tests at the third site near Ebensburg, Pennsylvania, reinforced these previous results.

TABLE XXVIII
COMPARISON OF CALCIUM SULFATE SOLUBILITY FOR DIFFERENT REVERSE OSMOSIS BRINES

Brine Concentration	Norton, 80 Percent Recovery	Norton, 90 Percent Recovery	Calculated Norton, 92.3 Percent Recovery	Arkwright, 50 Percent Recovery	Ebensburg, 84.7 Percent Recovery
Ca, mg/l	551	1,102	1,433	933	870
SO ₄ , mg/l	4,771	9,542	12,405	20,480	8,463
Ca, mol/l	13.78x10 ⁻³	27.55x10 ⁻³	35.82x10 ⁻³	23.32x10 ⁻³	21.75x10 ⁻³
SO ₄ , mol/l	49.69x10 ⁻³	99.40x10 ⁻³	129.21x10 ⁻³	213.33x10 ⁻³	88.15x10 ⁻³
P _{mc}	6.85x10 ⁻⁴	27.38x10 ⁻⁴	46.28x10 ⁻⁴	49.75x10 ⁻⁴	19.17x10 ⁻⁴
$\sqrt{P_{mc}/K_{sp}}$	1.78	3.56	4.63	4.80	2.98

Note: P_{mc} = product of molar concentration = (mol/l Ca)(mol/l SO₄)

K_{sp} = theoretical solubility of product, assuming 2000 mg/l CaSO₄
saturation = 2.16x10⁻⁴

$\sqrt{P_{mc}/K_{sp}}$ = ratio of P_{mc} to K_{sp}

From these test results, a relationship between the product of the molar concentration (P_{mc}) of calcium and sulfate in the brine and the theoretical saturation (K_{sp}) for CaSO_4 (neglecting pH, other ions, etc.) has been established. The solubility of CaSO_4 in distilled water is approximately 2000 mg/l. The solubility product, K_{sp} , is 2.16×10^{-4} , based on this solubility value. Thus, if supersaturation is attained, and the Ca^{2+} and SO_4^{2-} concentrations are known, the percent of saturation, neglecting the presence and strength of other ions, may be calculated as follows:

$$\text{Percent Saturation} = 100 \sqrt{P_{mc} / K_{sp}}$$

when P_{mc} is the product of the molar concentration of calcium and sulfate ions. For reverse osmosis purposes, in determining feasible water recovery rates (feed concentration factors), the calcium and sulfate ion levels in the concentrate stream are used for this calculation. The ability to operate at saturation levels above 100 percent is affected by the combination of short residence time, low pH and other ionic strengths, and temperature.

Experience with acid mine water has yielded the following values for CaSO_4 in the recovery range where precipitation was first detected:

	Percent Recovery	Percent Saturation
Site 1, Norton, West Virginia	90	356
Site 2, Morgantown, West Virginia	50	480
Site 3, Ebensburg, Pennsylvania	85	298

Thus, acid mine drainage experience to date indicates that a limit on recovery parameter is CaSO_4 concentration in the range of 300 to 400 percent of saturation when calculated as above. A feedwater can then be analyzed for calcium and sulfate, and a maximum recovery value can be calculated.

SECTION XIV
LABORATORY NEUTRALIZATION EXPERIMENTS

A series of laboratory experiments, both at Gulf Environmental Systems and at the Norton test site, were conducted to determine which alkalies would produce a sludge with the best settling and dewatering qualities and a supernatant with the best properties for further concentration by reverse osmosis.

Alkalies of practical interest and the amounts and costs required to neutralize 100 lb of sulfuric acid are listed below:

Material	Weight (lb)	Cost (\$)
CaO	56	0.56
MgO	40	1.20
NH ₃	34	1.60
Na ₂ CO ₃	106	2.12
NaOH	80	2.40

Other factors that must be considered in the choice of alkalies are the density and settling rate of the sludge, clarity of supernatant, and concentration of sparingly soluble compounds. The actual amounts of alkali required would probably differ from those listed above because of the desirability of neutralizing to the lowest feasible pH rather than to neutrality or beyond.

Bench neutralization experiments were carried out using a typical synthetic mine drainage feed (Ref. 7) whose analysis is quite similar to the brine obtained from the primary 10,000-gpd unit at Norton. This synthetic acid mine water has the following composition and characteristics:

Magnesium (Mg)	116 mg/l
Calcium (Ca)	385 mg/l
Iron (Fe ⁺⁺⁺)	500 mg/l
Aluminum (Al)	150 mg/l
Manganese (Mn ⁺⁺)	50 mg/l
Sulfate (SO ₄)	3,615 mg/l
Conductivity	3,800 μ mhos/cm at 25°C
pH	2.5

Ferric sulfate was used instead of ferrous sulfate since at Norton, West Virginia, almost all of the iron in the feedwater is present as the ferric ion.

In addition to the factors mentioned above, particular attention was given to the iron concentration in the neutralized and clarified mine water. Previous experience indicated that even a fraction of a milligram per liter of particulate ferric hydrate caused significant flux declines in a reverse osmosis unit because it coated the membrane surface. Fortunately, such deposits can be removed by fairly simple and inexpensive cleaning procedures.

If the iron content of the supernatant at pH 4 or 5 is only a few mg/l and consists primarily of ferrous iron, it may be possible to further process the water without additional problems. If the iron is largely present as ferric iron, further processing is possible, such as dropping the pH by using raw acid mine drainage or other acid, or by the use of reducing agents or iron complexing agents.

The laboratory neutralization studies of the acid mine water thus centered around neutralizing only to pH 4 or 5. The principal problem remaining was the calcium sulfate concentration, regardless of the method used, the supernatant was saturated or near-saturated with calcium sulfate and further concentration could result in module plugging. Table XXIX shows the results obtained with the previously listed neutralizing

TABLE XXIX
RESULTS OF ACID MINE WATER NEUTRALIZATION STUDIES

Neutralizing Agent ¹	pH	Amount of Agent Used (ml)	Amount of Fe in Supernatant (mg/l)	Amount of Floc After 30 Min (ml/l)
CaO	4.0	90	9.5	190
	5.4	145	0	535
CaCO ₃	4.0	140	>15	40
	5.1	260	0.95	70
MgO	4.0	60	>15	30
	5.2	110	5.1	48
NH ₄ OH	3.8	90	>15	200
	4.9	147	0	600
NaOH	3.9	100	2	180
	5.0	152	0	720

¹Solutions or suspensions; 10,000 mg/l used.

agents. Some tests were run with soda ash as well, but they showed no particular advantage in cost or improvement in results over those listed. Both calcium carbonate and magnesium oxide produce very dense sludges by comparison with the other agents. The calcium carbonate used was a very fine powder equivalent to chalk or whiting, and ordinary limestone would not serve as well unless some abrading mechanism was available during neutralization. The cost of powdered calcium carbonate is roughly the same as the cost of lime but much more calcium carbonate is required to neutralize the same water.

Magnesium oxide appeared to be the most promising agent with respect to cost, sludge density, and reduction of iron concentration (in the supernatant) to below 5 ppm at a final pH of 5.2. At this pH, and at the prevailing low iron concentration, it was thought that the calcium salt precipitation inhibitors would operate normally. The iron concentration was low enough to make the use of chelating or reducing agents feasible (from a cost point of view) to prevent iron hydrate precipitation when the supernatant was further concentrated.

Since ammonia has been reported to be only moderately rejected at about 80 to 90 percent by reverse osmosis, it was desirable to determine to what extent ammonia would be rejected when present essentially as an ammonium sulfate solution at a pH of about 5. By using an ammonium sulfate solution containing 500 mg/l as ammonia at pH levels between 5 and 6, product water concentrations of 2 mg/l or rejections of 99.5 percent were attained. This work was done with 3.5-ft² spiral-wound modules of the same construction as the standard 50-ft² modules.

Based on the previous information, bench studies were then carried out on the actual acid mine drainage at the Norton test site. A large sample of brine was taken from the 10,000-gpd unit and chemically analyzed. At the time of sampling, the unit was operating at 600 psi and 72.4 percent recovery. Results of the chemical analyses were as follows:

pH.	2.3	Total hardness. . .	2142 mg/l as CaCO ₃
Conductivity. . .	4000 µmhos	Cold acidity. . . .	2728 mg/l as CaCO ₃
Hot acidity . . .	1865 mg/l as CaCO ₃	Aluminum.	150 mg/l
Calcium	510 mg/l as Ca	Iron.	470 mg/l
Magnesium	217 mg/l as Mg	Sulfate	3013 mg/l

In the previous studies using the synthetic acid mine water brine, magnesium oxide appeared to be the most favorable neutralizing agent in terms of cost, settling time and density of sludge, and amount of iron removed from the solution. It was decided to further examine magnesium oxide by testing it on the sample of reverse osmosis brine described above. A one-liter sample of brine was stepwise neutralized using a 10,000 mg/liter suspension of magnesium oxide. An analysis of the supernatant from this neutralization gave the following results:

pH.	5.8	Total hardness. . .	3126 mg/l as CaCO ₃
Conductivity. . .	3000 µmhos	Aluminum.	0.9 mg/l
Hot acidity . . .	4.0 mg/l as CaCO ₃	Iron.	1.4 mg/l
Calcium	375 mg/l as Ca	Sulfate ¹	1325 (?) mg/l
Magnesium	547 mg/l as Mg		

¹Value in question in analysis does not balance.

The amount of iron remaining in the supernatant and the amount of floc formed using the synthetic and actual acid mine water were as follows:

Water	pH	Amount of Agent Used (ml)	Amount of Iron in Supernatant (mg/l)	Amount of Floc after 30 Min. Settling (ml/l)
Synthetic	5.2	110	5.1	48
10,000-gpd unit brine	5.6	105	1.4	30

The fact that the calcium and sulfate ions decreased after neutralization indicated that precipitation of calcium sulfate did take place. This, in turn, indicated that the supernatant was saturated in calcium sulfate.

Further concentration by reverse osmosis would be difficult using this supernatant. While the use of MgO results in considerably less sludge that must ultimately be disposed of, the cost advantage of lime and the better-quality effluent obtained using lime may outweigh the less-sludge advantage of MgO. Therefore, additional tests were conducted using brine from the 10,000-gpd unit to determine whether MgO or hydrated lime should be used for neutralization. The results of these tests are shown in Table XXX. The amount of sulfate remaining after neutralization with MgO cannot be explained readily.

Based on the work described above, it was decided to use lime for the actual neutralization at the Norton site. The brine from the 10,000-gpd unit was neutralized, settled, and filtered before being fed to the 4,000-gpd unit. After two short-term runs, the new process of reintroducing the neutralized supernatant back to the feed of the 10,000-gpd unit was begun. All further neutralization was then done using lime.

TABLE XXX
RESULTS OF BENCH NEUTRALIZATION TESTS ON 10,000-GPD-UNIT BRINE AT 91 PERCENT RECOVERY¹

Agent	Amount (g/l)	Initial pH	Final pH	Mg (mg/l)	Ca (mg/l)	Acidity, pH 7.3	SO ₄ (mg/l)	Fe (mg/l)	Al (mg/l)	Sludge (ml/l)
--	--	2.1	--	444	980	5650	6416	1560	310	--
MgO	1.869	5.0	4.3	1383	162	267	7917	1.6	68	40
MgO	2.000	5.5	4.5	1608	165	185	7371	1.3	48	60
MgO	2.280	6.0	6.7	1899	142	7	7098	0.8	2.1	60
Ca(OH) ₂	3.000	5.0	4.8	444	682	102	2320	2.0	16	125
Ca(OH) ₂	3.575	5.5	5.8	414	710	14	2184	1.0	2.8	130
Ca(OH) ₂	3.614	6.0	7.2	414	730	0	2084	0.8	0.4	185

¹These analyses were made on-site by EPA personnel.

Visual Observations

1. Settling times were about equal.
2. After 24 hr, the effluent from the lime neutralization was clearer and contained less suspended solids.
3. The lime sludge appeared denser.

SECTION XV
ACKNOWLEDGMENTS

The experiments described in this report could not have been accomplished without the aid of EPA personnel at Norton, West Virginia. Project Officer Robert Scott, Roger Wilmoth, James Kennedy, and Alvin Irons are extended special thanks for their cooperation in providing the study sites and facilities, operation and maintenance of equipment, and chemical analyses. Their encouragement, suggestions, and assistance are greatly appreciated.

Gulf Environmental Systems also wishes to thank the Christopher Coal Company and the Bethlehem Mines Corporation for their cooperation in allowing the use of their facilities.

The work was performed by James H. Sleight and S. S. Kremen; counsel and assistance were given by I. Nusbaum, A. Riedinger, C. Mungle, and R. Truby of Gulf Environmental Systems.

SECTION XVI

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Acid Mine Waste Treatment Using Reverse Osmosis,
Final Report FWQA Contract 14-12-525

The basic objectives of this test program were to demonstrate the applicability of reverse osmosis to the demineralization of acid mine drainages, both high-ferrous and high-ferric types, and to reclaim the maximum percentage of such feedwater in purified form suitable for domestic or industrial purposes, or as stream discharge. These goals included the attainment of maximum water recovery while maintaining the required product water quality and the determination of pretreatment requirements necessary to maximize water recovery and membrane life.

Two reverse osmosis test units were operated during the course of these tests: a nominal 10,000-gpd unit equipped with eighteen 50-ft² modules and a nominal 4,000-gpd unit equipped with nine 50-ft² modules. The modules used in these units consisted of both high-selectivity and high-flux cellulose acetate membranes.

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Acid Mine Drainage
Demineralization
Calcium-Sulfate
Solubility
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Ratios
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<div style="border: 1px solid black; padding: 2px;">5</div> Organization Environmental Protection Agency, Water Quality Office		
<div style="border: 1px solid black; padding: 2px;">6</div> Title Acid Mine Waste Treatment Using Reverse Osmosis		
<div style="border: 1px solid black; padding: 2px;">10</div> Author(s) James H. Sleigh S. S. Kremen	<div style="border: 1px solid black; padding: 2px;">16</div> Project Designation Program 14010 DYG <hr/> <div style="border: 1px solid black; padding: 2px;">21</div> Note <div style="border: 1px solid black; padding: 2px;">--</div>	
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<div style="border: 1px solid black; padding: 2px;">23</div> Descriptors (Starred First) Reverse osmosis, acid mine drainage, demineralization, calcium-sulfate solubility, ferrous-ferric iron ratios, water recovery, brine treatment		
<div style="border: 1px solid black; padding: 2px;">25</div> Identifiers (Starred First) West Virginia, Pennsylvania, water recovery, brine treatment		
<div style="border: 1px solid black; padding: 2px;">27</div> Abstract <p>The basic objectives of this test program were to demonstrate the applicability of reverse osmosis to the demineralization of acid mine drainages, both high-ferrous and high-ferric types, and to reclaim the maximum percentage of such feedwater in purified form suitable for domestic or industrial purposes, or as stream discharge. These goals included the attainment of maximum water recovery while maintaining the required product water quality and the determination of pretreatment requirements necessary to maximize water recovery and membrane life.</p> <p>Two reverse osmosis test units were operated during the course of these tests: a nominal 10,000-gpd unit equipped with eighteen 50-ft² modules and a nominal 4,000-gpd unit equipped with nine 50-ft² modules. The modules used in these units consisted of both high-selectivity and high-flux cellulose acetate membranes.</p> <p>The test program was carried out at three different mine drainage sites. The mine drainage water at the first site, Norton, West Virginia, contained greater than 98 percent of the iron present in the ferric form; at the other two sites, Morgantown, West Virginia, and Ebensburg, Pennsylvania, the drainage water contained predominantly ferrous iron. Discharges at the second site were so concentrated that recoveries were limited to 50 percent; recoveries of 80 to 90 percent were attained at the first and third sites. No iron fouling was encountered at any of the three sites. Specific salt rejections were >97 percent at all sites.</p>		
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