EPA-600/2-78-002
January 1978

Environmental Protection Technology Series

COMBINATION LIMESTONE-LIME NEUTRALIZATION OF FERROUS IRON ACID MINE DRAINAGE

Industrial Environmental Research Laboratory
Office of Research and Development
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Cincinnati, Ohio 45268

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by

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FOREWORD

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

This report documents the investigations of the feasibility of combination limestone-lime treatment of acid mine drainage containing soluble iron in the ferrous form. Conclusions drawn from this study indicate that increases in reagent utilization efficiency and reduction in materials usage costs can be achieved in specific situations of mine drainage treatment. In this regard, the report will be of interest to researchers in the acid mine drainage treatment field as well as to the coal industry and regulatory agencies involved with treatment situations. Inquiries on this subject and related subjects should be directed to the Resource Extraction and Handling Division, Extraction Technology Branch.

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ABSTRACT

Studies were conducted on ferrous-iron acid mine drainage (AMD) treatment by a two-step neutralization process in which rock-dust limestone was mixed with the influent AMD and then hydrated lime was added in a polishing reactor. This combination treatment process resulted in reagent consumption cost reductions as high as 30 percent as compared to those for single-stage hydrated lime treatment of the same AMD. Later data indicated that an equal cost reduction (compared to single-stage lime treatment) could be achieved by a two-stage hydrated lime process in which the AMD and recycled sludge were mixed in the first reaction vessel and hydrated lime was added in the second reactor. No cost advantage for the combination process over straight hydrated lime treatment was felt to exist in situations where sludge recycling was not employed.

This report covers the period from November 1975, to August 1976, and work was completed as of the latter date.

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ACKNOWLEDGMENTS

Thanks are extended to Ronald D. Hill, Eugene F. Harris, Robert B. Scott, James L. Kennedy, J. Randolph Lipscomb, Harry L. Armentrout, Robert M. Michael, Ralph S. Herron, Walter E. Grube, Jr., Loretta J. Davis, and Cathy J. Scott for their assistance during these studies.

The cooperation of Ray Henderson, Vince Ream, Bill Light, Mike Ryan, and Hershel Travis of Consolidation Coal Company is greatly appreciated.

SECTION 1
INTRODUCTION

The U.S. Environmental Protection Agency's (EPA's) Crown Mine Drainage Control Field Site facility, located near Morgantown, West Virginia, was established in 1972 to investigate the treatment requirements of predominately ferrous iron acid mine drainage. This was a partial follow-up of earlier studies[1] by the same staff at Norton, West Virginia, where the iron in the acid mine drainage (AMD) was in the ferric state.

EPA employees designed the Crown facility and performed most of the construction work. Contracts were awarded for the plant building shell, drying beds, lagoon, storage bins, and miscellaneous earthwork, and EPA personnel completed the construction including wiring, plumbing, instrumentation, and fabrication.

At present, lime neutralization is the commonly accepted method for treatment of AMD discharges. Lime treatment has several disadvantages:

- It produces a low-density, high-volume sludge.
- Its highly reactive nature provides high potential for accidental overtreatment.
- As a refined product, it is moderately expensive and is in increasingly short supply.

Earlier studies by EPA and others have indicated that limestone treatment and combination limestone-lime treatment offered advantages over lime in ferric iron situations. Most important of these advantages are:

- Production of a low-volume, high-density sludge.
- Cheaper raw-materials costs.
- Cheaper usage costs in some instances.

These processes needed to be investigated under ferrous iron conditions before they could be fully evaluated. This report documents the results of research conducted to evaluate combination limestone-lime treatment of ferrous iron acid mine drainage.

Prior to initiating the combination limestone-lime studies, investigations were completed on lime treatment and limestone treatment of the Crown AMD.[2] Excerpts from the conclusions from those studies are given below for the purpose of providing background comparisons for the current investigation of combination treatment:

ROCK-DUST LIMESTONE TREATMENT OF FERROUS IRON AMD

- Limestone utilization efficiencies were below 30 percent for ferrous iron acid mine drainage neutralization to pH 6.5, which was about the highest pH obtainable without sludge recycling. Effluent quality from the thickener was terrible under the pH 6.5 conditions.

- Recycling sludge at a rate of 20 percent of the influent flow rate increased the utilization efficiency of limestone to the 50-percent range. Effluent quality was still poor.

- The use of two aerators in series provided adequate detention time for iron oxidation. At least 4-hr detention time was necessary to reduce the ferrous iron level from 270 to 2.4 mg/l at pH's near 7. The combination of increased aeration time, better mixing, and sludge recycling enabled limestone treatment to pH's as high as 7.4. Effluent suspended solids (40 mg/l) and turbidity (60 JTU) were still high. Efficiencies ranged from 50-80 percent.

- Addition of coagulants reduced effluent iron levels (from 20 to 1 mg/l), lowered turbidities (from 60 to 12 JTU), and improved suspended solids (from 40 to 9 mg/l). The coagulants greatly diminished pH probe fouling problems that were generally severe below pH 7.5.

- The most satisfactory process operation involved incorporating a 20-percent sludge recycling rate, maintaining pH's near 7.4, furnishing 30-min detention time with vigorous mixing in the reactor, providing between 4 and 6 hr of detention time in the aeration systems, and injecting coagulants at about a 7-ppm rate. Reagent costs for this process were approximately 1.3 cents/cu m (5 cents/1000 gal) for limestone plus approximately 2.6 cents/cu m (10 cents/1000 gal) for coagulant.

Coagulant addition was not optimized. The thickener was most effective in clarification around 0.05 liter/sec/sq m (0.07 gpm/ft^2) of surface area.

LIME TREATMENT OF FERROUS IRON AMD

- Optimum overall operation of the EPA treatment system was at pH 8. At this pH, probe fouling problems were almost non-existent, effluent clarity was improved, and iron oxidation was complete. Reagent cost was increased over 25 percent by treating to pH 8 rather than pH 7; a 40-percent cost increase was incurred by treating to pH 8.5 rather than to pH 8.

- The thickener appeared to function most satisfactorily when the influent suspended solids were between 5,000 and 10,000 mg/l. Sludge recycling was employed to increase the influent solids to this range and to slightly improve utilization efficiency. Thickener effluents contained between 4 and 10 mg/l of suspended colloidal iron.

- Coagulants were very effective in improving the thickener effluent quality. Iron values were consistently below 2 mg/l using coagulants at pH 8.

- Precipitating the iron without oxidation offered few advantages over the oxidized version of the neutralization process. For ferrous precipitation, pH's had to be elevated to around pH 9 resulting in an increase in lime cost of 30 percent as compared to ferric treatment to pH 8. The ferrous hydroxide sludge occupied more than 1.5 times the volume of the ferric sludge. Thickener effluent quality was significantly better in the ferric situation.

- Lime treatment to pH 8 required approximately 7-min reaction time and less than 2-hr oxidation time (utilizing a sludge recycling rate below 20 percent of the influent rate) and required coagulant addition at a rate under 5 ppm. Reagent costs for this process were approximately 2.9 cents/cu m (11 cents/1000 gal) for lime plus 1.9 cents/cu m (7 cents/1000 gal) for coagulant. Coagulant addition was not optimized.

Sludge settling rates with coagulants were greater than .07 cm/s (8 ft/hr) for unhindered settling.

COMPARISON OF LIMESTONE VS LIME

- Limestone (rock dust) treatment of ferrous iron acid mine drainage involves reagent usage costs somewhat less than those required for lime treatment. Overall costs of the limestone process must include the very large reaction and oxidation vessel size and increased power requirements. These and other operational considerations render the limestone process to be considered unfeasible for general application to acid mine drainage waters containing over 50 mg/l of ferrous iron.

IRON OXIDATION

- An average oxidation rate design factor of 2 mg/l per min is suggested for lime neutralization aeration facilities with influent ferrous levels below 300 mg/l. On-site tests should be made to better determine the actual rate for the specific conditions before proceeding to final design. It is imperative that sufficient mixing capability be incorporated to provide sufficient top-to-bottom turbulence to prevent solids accumulation in the aeration tank.

- The stoichiometric oxygen requirement can be calculated from the equation: Oxygen (kg/hr) = 4.16×10^{-4} X (Flow, liter/min) X (Fe^2, mg/l); or, Oxygen (lb/hr) = 7.15×10^{-5} X (Flow, gpm) X (Fe^2, mg/l).

SECTION 2
CONCLUSIONS AND RECOMMENDATIONS

Combination first-stage limestone and second-stage lime treatment of ferrous iron acid mine drainage resulted in better than a 30-percent reduction in reagent usage cost as compared to single-stage hydrated lime treatment of the same water. Effluent quality parameters from both processes were comparable.

A 30-percent reagent usage cost reduction was obtained using a two-stage hydrated lime treatment process in which the sludge was recycled into the first-stage reactor to mix with the influent acid mine drainage and hydrated lime was added to the sludge/AMD mixture in the second stage. The 30-percent reduction was relative to a single-stage hydrated lime process where the recycled sludge, hydrated lime, and acid mine water were combined in a single reactor.

Based upon the success of the two-stage lime process, combination limestone-lime treatment did not appear to be a feasible alternative in ferrous iron situations where sludge recycling capabilities existed or could be provided. Moreover, in comparing the results of combination treatment without sludge recycling with the results of earlier lime treatment studies without sludge recycling, no cost advantage appeared to exist.

Because of the varying characteristics of mine drainage waters, bench-scale feasibility studies could be made to determine the initial potential for the applicability of combination limestone-lime treatment for each specific situation. In general, the greater the ferrous iron content, the less applicable any limestone process.

SECTION 3
PROCEDURES

DEFINITIONS

Definitions and an explanation of the calculations used in this report are presented in the Glossary.

DESCRIPTION OF FACILITY

Acid mine water was obtained from the Stewarts Run borehole pump of Christopher Coal Company that pumped water from the Pittsburgh Coal Seam located approximately 85 m (280 ft) below the surface. This pump removed water from an abandoned section of Mine 93 to prevent the drainage from entering an active section of the mine. The borehole pump discharged approximately 32 liters/sec (500 gpm) into a 3.8-cu m (1000-gal) concrete reservoir constructed from a burial vault. The water used by the EPA facility was pumped from the reservoir to the treatment plant--a distance of about 366 m (1200 ft)--through a 102-mm (4-in) plastic pipe at a pressure of 480 kN/sq m (70 psi).

The AMD flowed through the neutralization facility as illustrated in Figure 1. Processes A and B were identical in every respect. By having a dual system such as this, it was possible to use one side as a control and the other side as the variable and thus expedite the research efforts. The dual system allowed immediate evaluation of the isolated variable under study. All AMD transfer lines were 25-mm (1-in), Schedule 40 PVC. The sludge lines were 13-mm (½-in) plastic roll piping. PVC in-line strainers removed large particulate matter from the AMD prior to its entering the pressure regulators. Plast-O-Matic Model PR075V pressure regulators were used to maintain relatively constant downstream pressures to reduce flow fluctuations. C-E Invalco Model W3/1000 turbine flow meters with Model W315 totalizers and rate indicators totalized gallonage treated and displayed instantaneous rate of flow. Accuracy of these meters was better than 1 percent of the full-scale flow of 4 liters/sec (64 gpm).

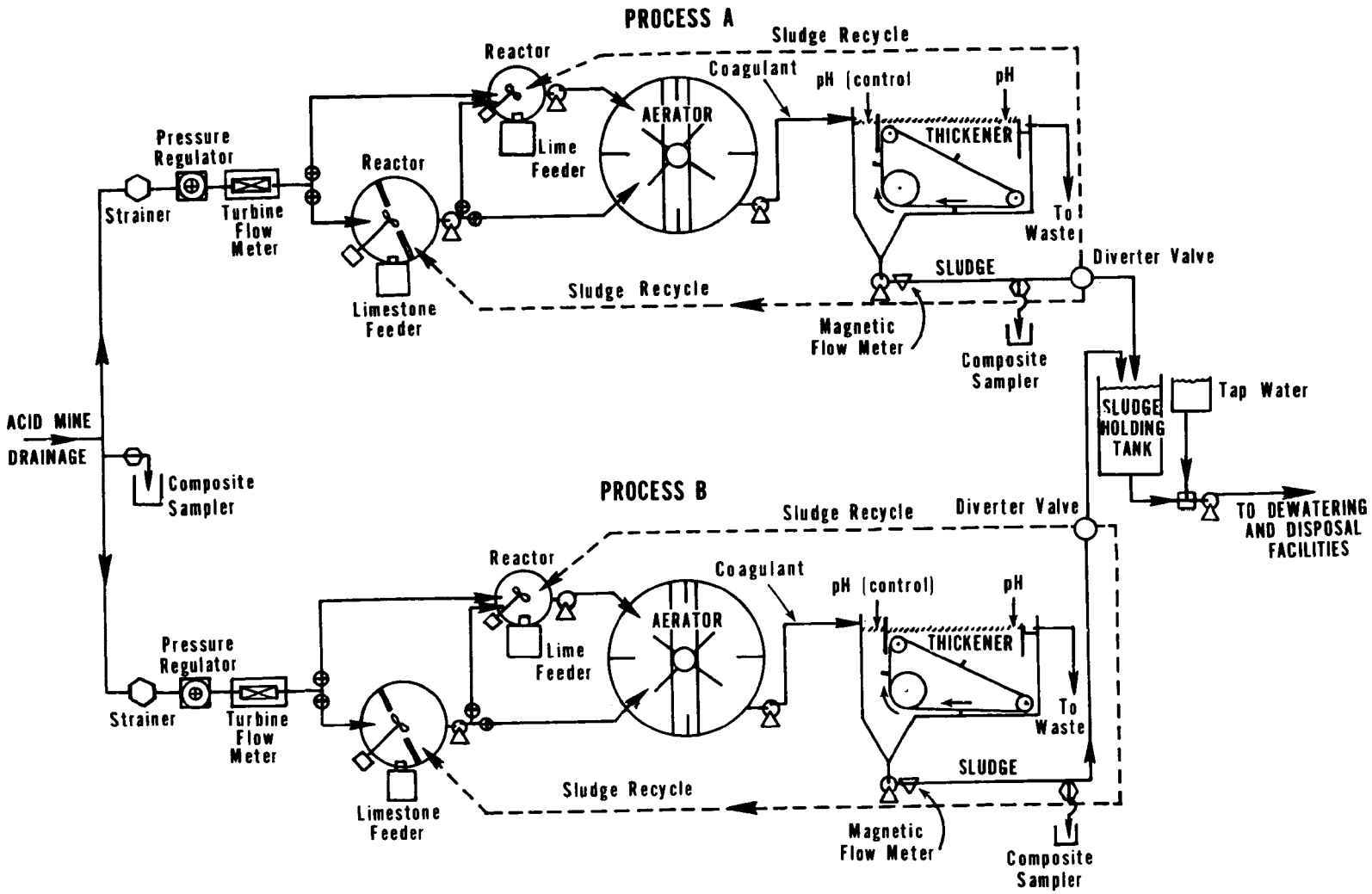

Figure 1. Schematic flow diagram for the EPA neutralization facility.

From the turbine meters, water could be channeled either:

1) Into the lime reactor, the aerator, and the thickener, or
2) Into the limestone reactor, the aerator, and the thickener, or
3) Into the limestone reactor, the lime reactor, the aerator and the thickener.

The lime reactor tanks were type 304 stainless steel with effective capacities of 454 liters (120 gal) and were equipped with Lightnin' Model NS-7, 3-hp, 1750-rpm mixers. High-density polyethylene tanks with 9.5-mm (3/8-in) thick walls and effective capacities of 3785 liters (1000 gal) were used for the limestone reactors. Lightnin' NS-7 mixers were installed on independent mounts for use with the polyethylene tanks. In later studies, the NS-7 mixers on the limestone reactors were replaced by 3-hp, 350-rpm, Lightnin' ND-4B mixers to obtain increased turbulence. The NS-7 system provided a mixing (pumping) rate of 93 liter/sec (1470 gpm); the ND-4B pumped 360 liter/sec (5710 gpm). The limestone reactors were equipped with two baffles per tank. Each baffle consisted of a metal outside support frame surrounding a 168-cm by 36-cm (66-in by 14-in), 14 x 14-mesh, 304-stainless steel screen. The screen baffles served to improve mixing efficiency and to abrade the limestone particles in order to remove reaction coatings.

BIF Model 25-06 helix-type volumetric dry feeders with variable-speed drives were used to feed both hydrated lime and rock-dust limestone into the respective reactors. The hoppers on top of the dry feeders held 0.2 cu m (7.8 cu ft) of material. Syntron Model V-20 vibrators were bolted to the side of each hopper to prevent bridging. The vibrators operated whenever the dry feeders were operating. Typical observed dry feeder accuracies were about ± 5 percent on a weight basis. The operating time for each dry feeder was accumulated on Eagle Model HK300A6 time totalizers reading directly in minutes. These units were equipped with dynamic braking circuitry to prevent the timers from coasting after the dry feeders stopped.

The lime and limestone dry feeders were refilled automatically from 36-tonne (40-ton) capacity Butler bolted-steel silos equipped with Vibra-screw bin activators to prevent bridging. Jeffrey screw conveyors with 15-cm (6-in) screws transported the neutralizer from the silos to the top of the dry feeders. Monitor-brand bin level indicators were mounted in the dry feeder hoppers to activate the conveyors when the level dropped.

The storage silos were periodically pneumatically filled from 18-tonne (20-ton) supply trucks. Butler Model CS9-90 bin-vent filters were mounted on top of the silos to prevent dust from escaping while the tanks were being loaded.

The neutralized water was pumped from the reactors into the aerators. The aeration tanks were 3 m (10 ft) in diameter by 1.8 m (6 ft) high with a nominal water depth of 100 cm (3.25 ft); they contained 6800 liters (1800 gal). Four 25-cm (10-in) solid baffles were installed at equal spacing around the periphery of the tank. The tank itself was constructed of 6.4-mm (¼-in) hot-rolled steel coated with epoxy. Lightnin' Model LAR-10, 1 hp, fixed-mount, surface aerators were installed on top of the aeration tanks. These aerators were designed to transfer 1.8 kg/hr (4 lb/hr) of oxygen under standard conditions.

Although the aerators were quite satisfactory in oxygen transfer, they lacked sufficient top-to-bottom turbulence to keep heavier particles suspended. This problem was most prevalent during limestone treatment studies where a sizable accumulation of solids occurred in the bottom of the aeration tank and thus decreased the effective detention time. A bottom-mixing prop was installed on the aerator shaft later in the studies to successfully decrease the solids accumulation problem.

After aeration, the treated water was pumped into the thickener for clarification and sludge removal. FMC Link-Belt, Type-H thickeners were used. These thickeners were 5.7 m (18.75 ft) long, 2.4 m (8.0 ft) wide, and 2.7 m (9.0 ft) high (excluding sludge hoppers) and had an effective capacity of 34.8 cu m (9200 gal). Adjustable effluent weirs allowed the effective depth to be varied from 2.7 m (9 ft) to 1.8 m (6 ft). Rakes moving at 0.005 m/sec (1 fpm) pushed the sludge into a collection hopper on the influent end of the thickener. The sludge hopper extended approximately 1.6 m (5.25 ft) below the floor level of the thickener and held 3.4 cu m (900 gal) of sludge. At 1 liter/sec (15.85 gpm), the surface overflow rate was 0.07 liter/sec/sq m (0.11 gpm/sq ft).

Sludge was removed from the thickeners by timer-controlled centrifugal pumps. Continuous removal was not possible because the lines became plugged at low-flow rates. It was therefore necessary to pump the sludge for short durations at high-flow rates. A Tork Model 12M8001 repeating-cycle timer

with a 12-min full-cycle time and 60 adjustable tabs to regulate on/off time controlled the pumping duration and frequency. Typically, in non-recycling situations, the sludge pumps operated 10 percent of the time (on 0.6 min and off 5.4 min). Sludge pumping rates were about 0.3 liter/sec (5.5 gpm) through a 13-mm (½-in) line corresponding to a linear flow velocity of 2.7 m/sec (9 fps).

Sludge recycling was accomplished by utilizing Eagle Signal Series HP700, adjustable-duration pulse timers to control a pneumatically operated diverter valve in the sludge line. When the Tork timer activated the sludge pump, it simultaneously activated the Eagle pulse timer. Sludge was recycled into the appropriate reactor until the Eagle timer timed-out and switched the sludge diverter valve in the waste direction.

Initially, Fischer X404 diverter valves were used for recycling sludge. These valves had a 316-stainless steel body and ball with teflon seats. During limestone sludge recycle studies, the tiny unreacted limestone particles wedged between the teflon seats and the ball and scored both the seat and the ball in just a couple of weeks of operation. Later, these valves were replaced with Model 2600 NPT Red Valves with hypalon liners and PVC end caps.

Sludge flow was measured by Brooks Model 7185FB11KA 13-mm (½-in) magnetic flow meters that totalized gallonage and displayed instantaneous rate-of-flow (Model 7300B1A2C signal converters and Model 5540B1A4A1A1 totalizers). Accuracy of these magnetic flow meters was 0.5 percent of the full-scale indication of 0.63 liter/sec (10 gpm).

Since each magmeter was placed before the diverter valve, the output was indicative of total sludge flow. To independently totalize the sludge quantity being recycled and the quantity going to waste, the magmeter signal converter output was channeled through a relay that switched (at the same time the diverter valve switched) from one totalizer to another.

The waste sludge from both thickeners was pumped into a 1.5-cu m (400-gal) reservoir located immediately adjacent to the thickeners. When the sludge level reached the top of the reservoir, an automatic sequencer-controlled pump transferred the sludge to disposal facilities and then flushed the sludge lines with approximately 150 liters (40 gal) of tap water. Sludge flow to the disposal facilities was initially measured by volumetrically determining the flow rate and multiplying by pumping time. Operating time of

the pump was recorded on an Eagle elapsed-time meter. Later, this flow was totalized electronically by remote slave counters on each magmeter waste totalizer. Typical pumping rates to the disposal facilities were 1.1 liter/sec (17 gpm) through a 25-mm (1-in) roll plastic line corresponding to a linear flow velocity of 2.1 m/sec (6.8 fps).

Both lime and limestone feed could be controlled either manually or automatically. Automatic control was accomplished by Universal Interloc (Uniloc) Model 1001 pH recorder-controllers that utilized on/off type control. The pH controllers turned the dry feeders on when the pH dropped below the set point and off when the pH increased beyond the set point. In order to reduce probe fouling, the pH probes (Uniloc Model 321 immersion-type) were placed in the influent area of the thickener. Probes that were placed in the reactors fouled within a few hours. Even in the thickener, the probes fouled severely at pH's below 7.5. It was necessary to clean the probes with HCl as often as three times per day at the lower-pH conditions. It took the probes approximately 1 hr to recover from each acid cleaning. Neutralizing the probes in a pH 10 Na_2CO_3 solution hastened the probe recovery. At pH levels above 7.5, probe fouling was not a problem and cleaning was routinely done once a day.

Effluent pH was monitored by similar Uniloc equipment with the pH probes placed immediately in front of the thickener effluent weir.

Both influent (control) and effluent pH's for processes A and B were recorded on Hewlett-Packard Model 7100B strip chart recorders. In the normal mode of operation, the control set-point on the influent pH meter was adjusted until the desired effluent pH was achieved. Generally, the entire system could be changed from one pH to another and stabilized within 36 hr. The pH control system worked extremely well, as the effluent pH could be maintained within $\pm$ 0.1 pH for extended periods of time. The design flow rate through each process was 1 liter/sec (15.9 gpm).

All process equipment was located indoors to isolate the variable of weather from the data evaluation.

PHYSICAL MEASUREMENTS

Flow and Reagent Usage

Water and sludge flow rates and quantities were measured by turbine meters and magnetic flow meters, respectively, as previously described. Reagent usage was calculated by collecting and weighing a 3-min sample from

the dry feeders at the beginning and end of each data set and averaging the values. The average rate per minute was multiplied by the number of minutes of dry feeder operation to determine the quantity of neutralizing agent used. These reagent data were then related to the water quantity and influent and effluent acidity and alkalinity values to calculate the various parameters of interest such as usage, stoichiometric addition rate, and utilization efficiency. Definitions and details of these calculations are presented in the Glossary.

Water Sampling

The water quality from the borehole pump was quite variable. Fluctuations in influent acidity were as large as 40 percent in a 24-hr period. Initially, the beginning and ending acidities for each data set were averaged before being used in data calculations. Later, an automatic composite sampler was built and installed to provide more reliable influent water quality data.

Sludge Settling Rates

Sludge samples were placed in 1000-ml graduates and the sludge/supernatant interface was recorded as a function of time. In the case of limestone, as well as lime at low pH's, a definite interface never clearly developed during settling. In these cases, it was possible to observe the buildup of solids in the bottom of the graduate.

Sludge Sampling

Representative sludge samples were quite difficult to obtain from the process. Generally, the sludge was pumped in short-duration intervals on the timed-basis previously described. Considerable variation in sludge density was observed from the beginning to the end of the pumping cycle. All attempts were made to sample near the midpoint of the cycle. Later, automatic composite samplers were built and installed in the sludge lines at the discharges of the thickener hoppers.

The clarifier rakes moved the sludge consistently to the edge of the 3400-liter (900-gal) sludge hopper. Movement of the sludge from the top edge of the hopper down the steep sides to the pump intake was often erratic and thus created difficulties in obtaining representative samples. This was not an operational problem but merely presented difficulties in data evaluation.

Detention Times of the Various Vessels

Detention time determinations were made by instantaneously injecting either salt or acid into the inlet of the vessel and monitoring the effluent conductivity until it returned to a base level. The mean probable detention time was calculated from a plot on probability paper (see Glossary for explanation of calculation) and represents the point in time when the centroid of the tracer mass passed through the vessel. Results of these studies are in Table 1. The tracer appeared almost immediately in the effluents from all reactors and aerators because of the mixing capability of these vessels. In the thickeners, however, the first visible trace occurred at 120 min at both 0.32-liter/sec and 0.95-liter/sec (15-gpm) flow rates with the sludge rakes operating. The sludge rakes significantly affected mixing in the thickeners. In a static thickener with the rakes off, it took 50 hr after tracer injection for the tracer to diffuse sufficiently to increase the conductivity at the discharge end of the thickener to 500 micromhos/cm; with the rakes on, the same conductivity was attained in 14 hr.

PROCEDURES FOR CHEMICAL ANALYSES

Conductivity and pH were measured potentiometrically. Total iron, aluminum, magnesium, manganese, and calcium were determined by atomic absorption spectrophotometry.[2] Sulfate was measured by adding barium to precipitate the sulfate and analyzing for residual dissolved barium in the supernatant by atomic absorption.[3] Precipitation of the barium was accelerated by centrifuging. EPA methods[2] were used for total solids, suspended solids, alkalinity, and turbidity determinations. Ferrous iron was determined by colorimetrically titrating potassium dichromate against a p-Diphenylamine sulfonic acid sodium salt indicator.[4] A YSI Model 51 dissolved oxygen (D.O.) meter was used for D.O. measurements. The Salotto acidity method[5] was used, in which hydrogen peroxide is added to oxidize the metals; a room-temperature titration is then made to pH 7.3.

CHARACTERISTICS OF REACTANTS

Mine 93 Borehole Discharge

Table 2 presents the typical chemical quality of the AMD used for these studies.

Table 1. TYPICAL DETENTION TIMES

Test vessel	Effective volume at time of test		Flow rate		Theoretical detention time, min	Mean probable detention time, min	Efficiency (T/To), percent
	liters	gal	liter/sec	gpm			
Limestone reactor A	2460	650	0.32	5	130	78.6	61
Limestone reactor B	2460	650	0.95	15	43	28.8	67
Lime reactor B	454	120	0.32	5	24	16.6	69
Lime reactor B	454	120	0.95	15	8.0	6.1	76
Aerator A	6810	1800	0.32	5	360	273	76
Aerator B	6620	1750	0.95	15	117	92	79
Thickener B	34820	9200	0.32	5	1840 (30.7 hr)	1435 (23.9 hr)	78
Thickener A	34820	9200	0.95	15	613 (10.2 hr)	515 (8.6 hr)	84

Table 2. CROWN WATER QUALITY DATA, 7/74 THRU 6/75

Parameter	Unit	Mean	Maximum	Minimum	Standard deviation
pH	pH	5.04	5.9	4.7	--
Specific conductance	µmhos/cm	3760	4000	3400	260
Acidity as $CaCO_3$	mg/l	640	1070	155	120
Calcium	mg/l	370	450	300	40
Magnesium	mg/l	110	150	55	20
Total iron	mg/l	300	380	250	39
Ferrous iron	mg/l	270	340	160	34
Sodium	mg/l	480	670	280	95
Aluminum	mg/l	15	36	6	9
Manganese	mg/l	6	8	4.3	1
Sulfate	mg/l	3040	3600	2300	300
Alkalinity	mg/l	17	100	0	25
Total dissolved solids	mg/l	4320	5170	3250	480
Temperature	^{o}C	13.8	17	9	2.3

Lime and Limestone

The limestone and hydrated lime used in this study were obtained from Germany Valley Limestone Company, Riverton, West Virginia. In order to obtain the smallest particle size commercially available, the rock-dust form of limestone was used. All tests in this study were made using the hydrated form of lime.

Table 3 presents the manufacturer's chemical analyses and cost for lime and limestone.

Table 3. MANUFACTURER'S CHEMICAL ANALYSES OF LIME AND LIMESTONE

Parameter	Hydrated lime,[a] minimum % composition	Rock-dust limestone,[b] minimum % composition
CaO	72.00	53.0
MgO	0.70	0.38
$CaCO_3$ equivalent	130	98.6
SiO_2	-	0.49

[a]Lime cost $38.58/tonne ($35.00/ton) or 3.86 cents/kg (1.75 cents/lb) delivered in bulk.

[b]Limestone cost $12.13/tonne ($11.00/ton) or 1.21 cents/kg (0.55 cents/lb) delivered in bulk.

A spectrochemical analysis of the limestone was made by Bituminous Coal Research and is reported in Table 4. A dry sieve analysis of as-received lime and limestone is presented in Table 5.

Table 4. SPECTROCHEMICAL ANALYSIS OF GERMANY VALLEY LIMESTONE
(percent)

Loss on ignition	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	TiO_2	Na_2O	K_2O	MnO_2
43.0	1.0	0.43	0.15	1.16	97.0	0.04	0.02	0.1	0.03

Table 5. SIEVE ANALYSES (DRY) OF AS-RECEIVED LIME AND LIMESTONE

Screen size	Hydrated lime, percent passing	Rock-dust limestone, percent passing
50 mesh (0.297 mm)	92.9	97.7
100 mesh (0.149 mm)	62.5	83.6
200 mesh (0.074 mm)	35.0	56.6
400 mesh (0.037 mm)	13.3	15.7
Percent not passing sieve	4.4	2.1

SECTION 4
RESULTS

BATCH-SCALE NEUTRALIZATION TESTS

A laboratory-scale study was performed to directly compare lime, limestone, and combination limestone-lime neutralization to pH 6.5 and pH 9.5. Limestone treatment was tested only at the 6.5 pH level. Since the tests were almost simultaneously conducted, results can be directly compared.

Figure 2 shows the sludge settling trends. Neutralization to pH 6.5 resulted in very-low-volume sludges because of incomplete reactions at that pH. Increasing the pH to 9.5 significantly increased sludge volumes as the iron oxidized, hydrolyzed, and precipitated. Combination-treated sludge settled more rapidly than lime sludge but the 24-hr volumes were the same. This result is not in agreement with later observations that combination sludge settled to a lesser final volume than lime sludge.

Chemical and physical data for the tests are presented in Tables 6 and 7. The data are the same in both tables but are presented differently to make comparisons easier.

Very little iron oxidation occurred at pH 6.5 in 24-hr. At pH 9.5, complete oxidation occurred in a matter of minutes. Unexpectedly, very little loss in calcium and sulfate was detected by the chemical analyses.

Sludge Characteristics

Bench-scale studies were made to investigate sludge characteristics as related to the neutralizing agents used and the treatment pH.

Figure 3 presents settling trends as a function of pH for combination limestone-lime treatment. No clear sludge/supernatant interface developed with any of the neutralizing agents below pH 6.5. Figure 4 is a composite drawing to compare the relative settling trends of lime, limestone, and combination treatment at pH 7. Since the tests for each neutralizing agent were made at separate times on separate AMD samples, Figure 4's direct comparison is not strictly valid but may be useful as a guide. At the end of

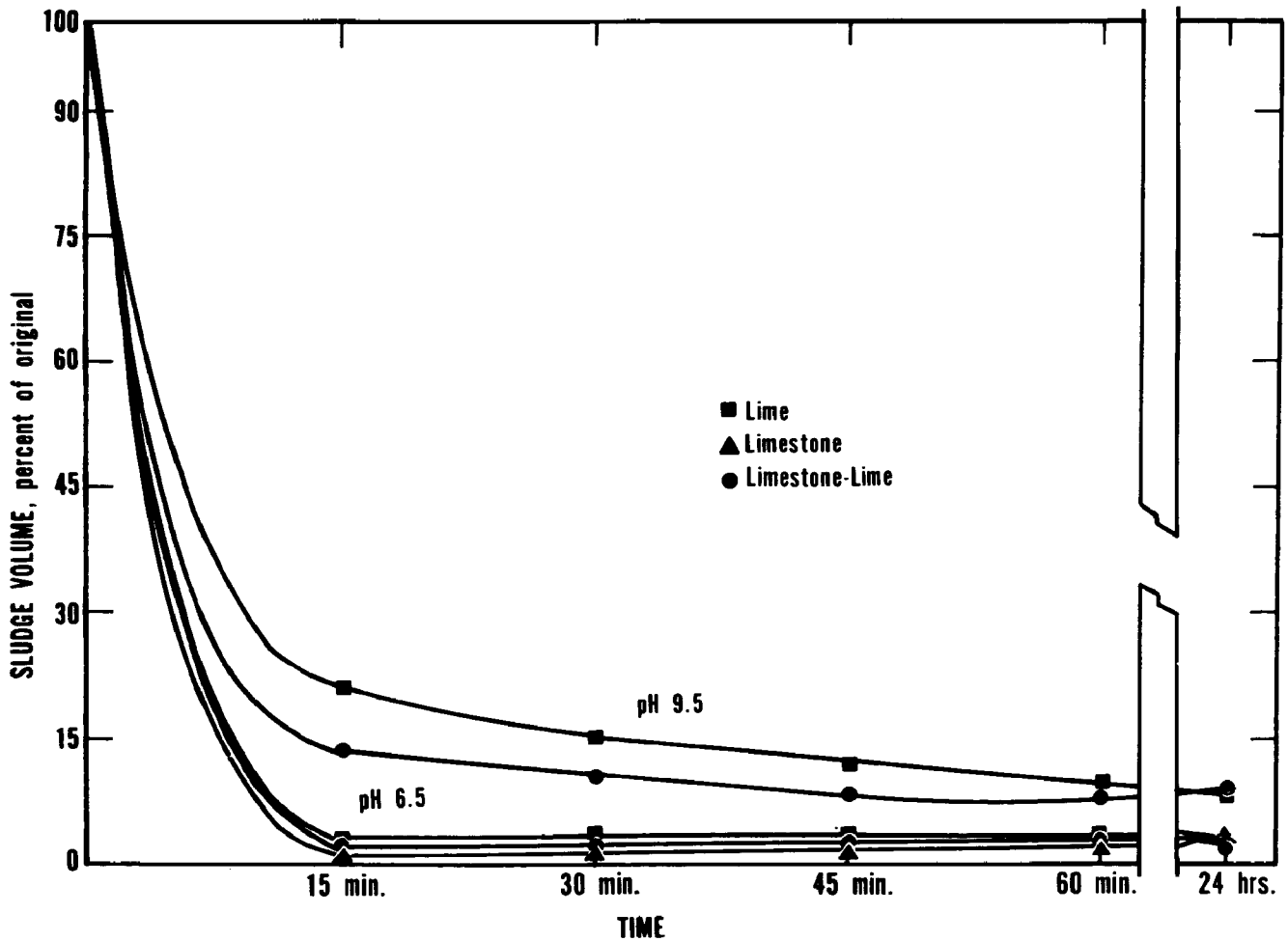

Figure 2. Comparison of settling trends at pH 6.5 and pH 9.5.

Table 6. RESULTS COMPARISON AS RELATED TO TREATMENT pH[a]

Parameter	Raw water	Lime pH 6.5	Lime pH 9.5	Limestone-lime pH 6.5	Limestone-lime pH 9.5
			1-hr Settling		
Turbidity	7	62	25	80	30
Ferrous iron	530	350	0	390	0
Total iron	550	360	0.54	390	0.28
Aluminum	50	3.1	0.7	3.2	0.9
Manganese	9.2	11	0.31	12	0.37
Magnesium	180	200	150	200	150
Calcium	450	790	1020	740	1010
Sodium	620	640	610	620	610
Sulfate	4050	3780	3690	3900	3900
Conductivity	4800	5200	5050	4900	4950
Acidity	1450	---	0	760	0
Alkalinity	0	---	---	---	---
Sludge % solids		3.6	6.5	4.4	9.3
			24-hr Settling		
Turbidity	7	34	6	42	7
Ferrous iron	530	340	0	380	0
Total iron	550	340	0.07	380	0.09
Aluminum	50	1.1	0.9	1.2	1.0
Manganese	9.2	11	0.19	12	0.26
Magnesium	180	180	130	180	130
Calcium	450	720	1010	730	1050
Sodium	620	600	590	590	590
Sulfate	4050	3940	3690	3940	3900
Conductivity	4800	4750	5000	4950	5100
Acidity	1450	717	0	780	0
Alkalinity	0	5	79	5	81
Sludge % solids		5.2	6.5	4.3	6.1

[a]All units are mg/l except for turbidity (JTU) and sludge percent solids.

Table 7. RESULTS COMPARISON AS RELATED TO SETTLING TIME[a]

Parameter	Raw water	Lime 1-hr	Lime 24-hr	Limestone 1-hr	Limestone 24-hr	Combination 1-hr	Combination 24-hr
				At pH 6.5			
Turbidity	7	62	34	120	49	80	42
Ferrous iron	530	350	340	470	450	390	380
Total iron	550	360	340	490	450	390	380
Aluminum	50	3.1	1.1	3.7	1.3	3.2	1.2
Manganese	9.2	11	11	12	12	12	12
Magnesium	180	200	180	200	190	200	180
Calcium	450	790	720	710	640	740	730
Sodium	620	640	600	630	575	620	590
Sulfate	4050	3780	3940	3940	3780	3900	3940
Conductivity	4800	5200	4750	5100	4950	4900	4950
Acidity	1450	--	717	950	940	760	780
Alkalinity	0	--	5	--	8	--	5
Sludge % solids		3.6	5.2	4.5	6.7	4.4	4.3
				At pH 9.5			
Turbidity	7	25	6	---[b]	---[b]	30	7
Ferrous iron	530	0	0	---	---	0	0
Total iron	550	0.54	0.07	---	---	0.28	0.09
Aluminum	50	0.7	0.9	---	---	0.9	1.0
Manganese	9.2	0.31	0.19	---	---	0.37	0.26
Magnesium	180	150	130	---	---	150	130
Calcium	450	1020	1010	---	---	1010	1050
Sodium	620	610	590	---	---	610	590
Sulfate	4050	3690	3690	---	---	3900	3900
Conductivity	4800	5050	5000	---	---	4950	5100
Acidity	1450	0	0	---	---	0	0
Alkalinity	0	--	79	---	---	--	81
Sludge % solids		6.5	6.5	---	---	9.3	6.1

[a]All units are mg/l except for turbidity (JTU) and sludge percent solids.

[b]Not applicable.

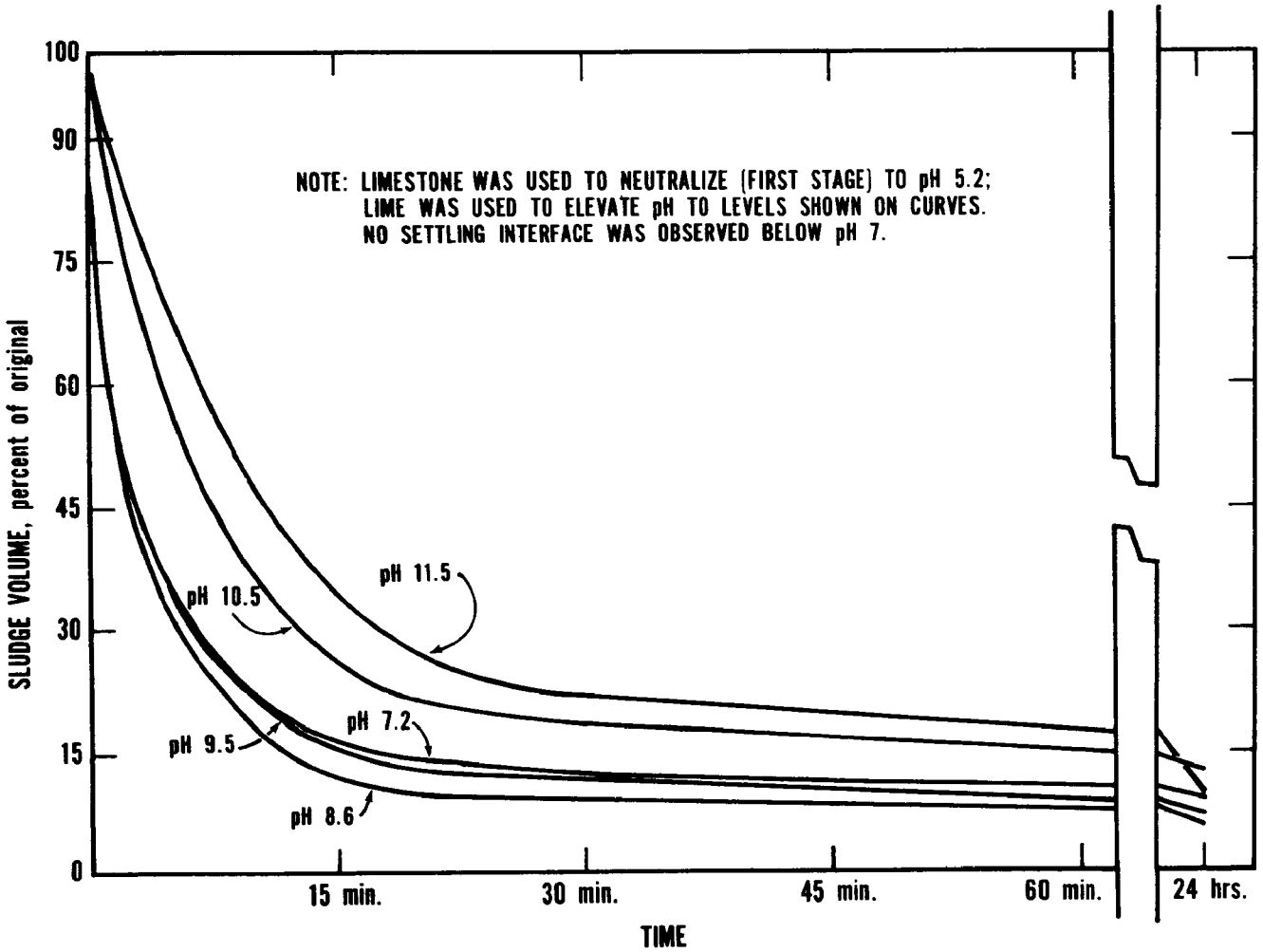

Figure 3. Settling trends for combination limestone-lime neutralization.

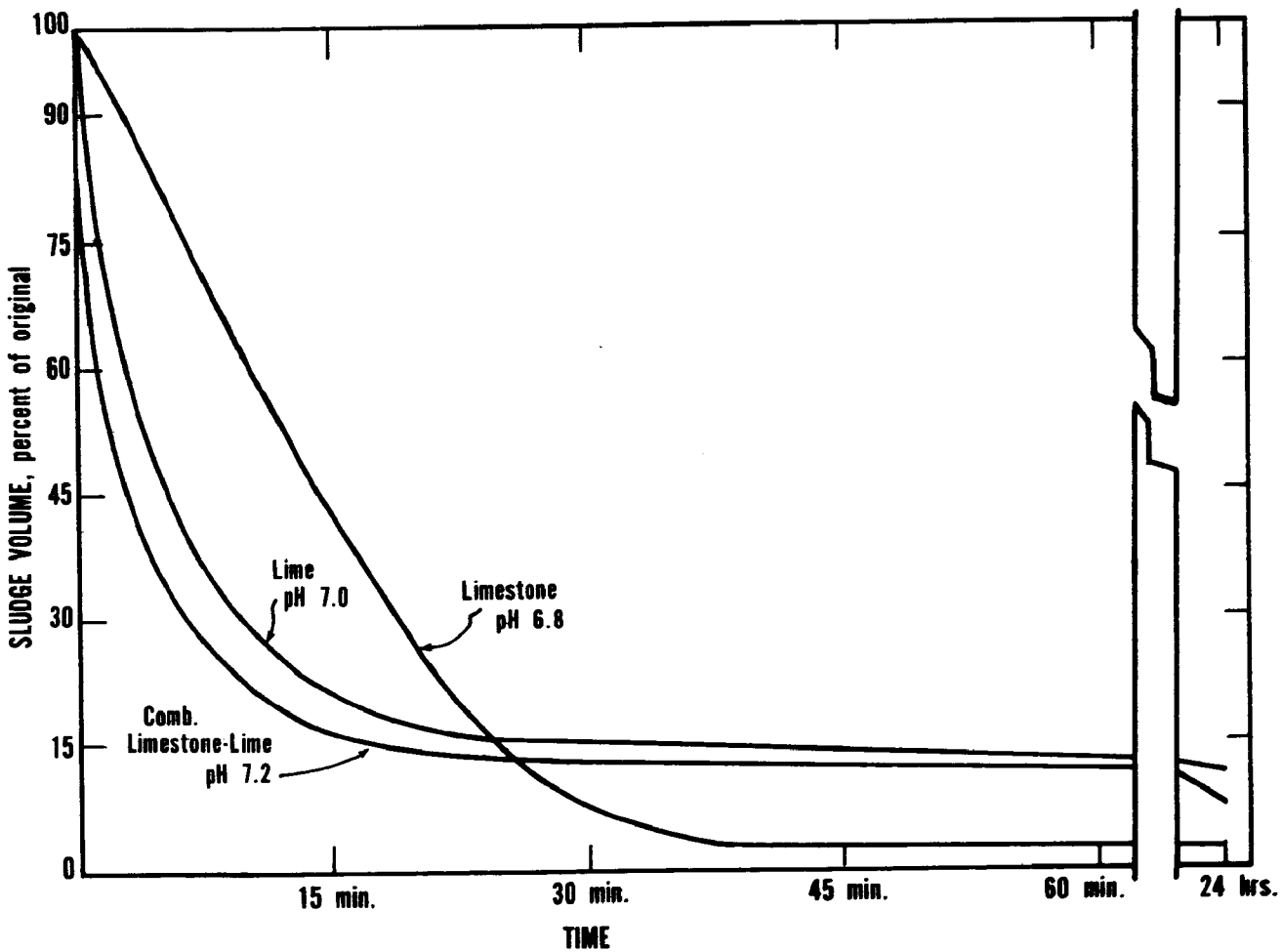

Figure 4. Comparison of settling trends at pH 7.

1 hr, the limestone sludge occupied a significantly smaller volume than the other two sludges. The same situation was observed after 24 hr of settling.

Effluent turbidity and sludge percent-solids determinations were made on each sample after 1 hr and after 24 hr of settling. The results are shown in Figures 5 through 8. Limestone effluent turbidity, after 1 hr, was poor in comparison to that of lime and to combination treatment. At pH 6.8, limestone sludge had a percent-solids content about four times as great as the other two sludges.

Surprisingly, little change occurred in the sludge percent-solids between the 1-hr 24-hr times in all the samples. Supernatant turbidity values were significantly lower for all samples after 24-hr of settling as compared to the 1-hr values.

In summary, limestone sludge settled fastest and compacted to the least volume. Supernatant clarity from limestone treatment, however, was poor in relation to lime treatment and combination treatment. Combination limestone-lime sludge settled more rapidly than lime and settled to a final volume that was on the order of one-half that of lime-treated sludge at pH 7. Supernatant clarity of combination treatment after 24 hr was superior to that of lime treatment.

Reagent Costs For Batch Neutralization

Bench-scale tests were made to determine the feasibility of combination limestone-lime treatment of the Crown acid mine drainage. A group of one-liter samples were neutralized to various predetermined pH's, using limestone and allowing 20 min of reaction time, and then were treated with lime to pH 7.0 and further to pH 9.5 (for ferrous oxidation or precipitation). The quantities of limestone and lime were recorded. For comparison, samples were treated to pH 7.0 using limestone alone. Lime was used alone to treat to pH 7.0 and pH 9.5.

The important consideration in this test was cost. Raw material costs at Crown were: Limestone = $11.00 per ton in bulk, and lime = $35.00 per ton. Table 8 and Figure 9 contain the data from the feasibility test. Combination treatment using a first-stage limestone pH of 5.1 was the most economical method of treating to pH 7.0. This combination (6.4 cents/1000 gal) was 57 percent cheaper than straight limestone treatment (14.8 cents/1000 gal) and 9 percent cheaper than straight lime treatment (7.0 cents/1000 gal).

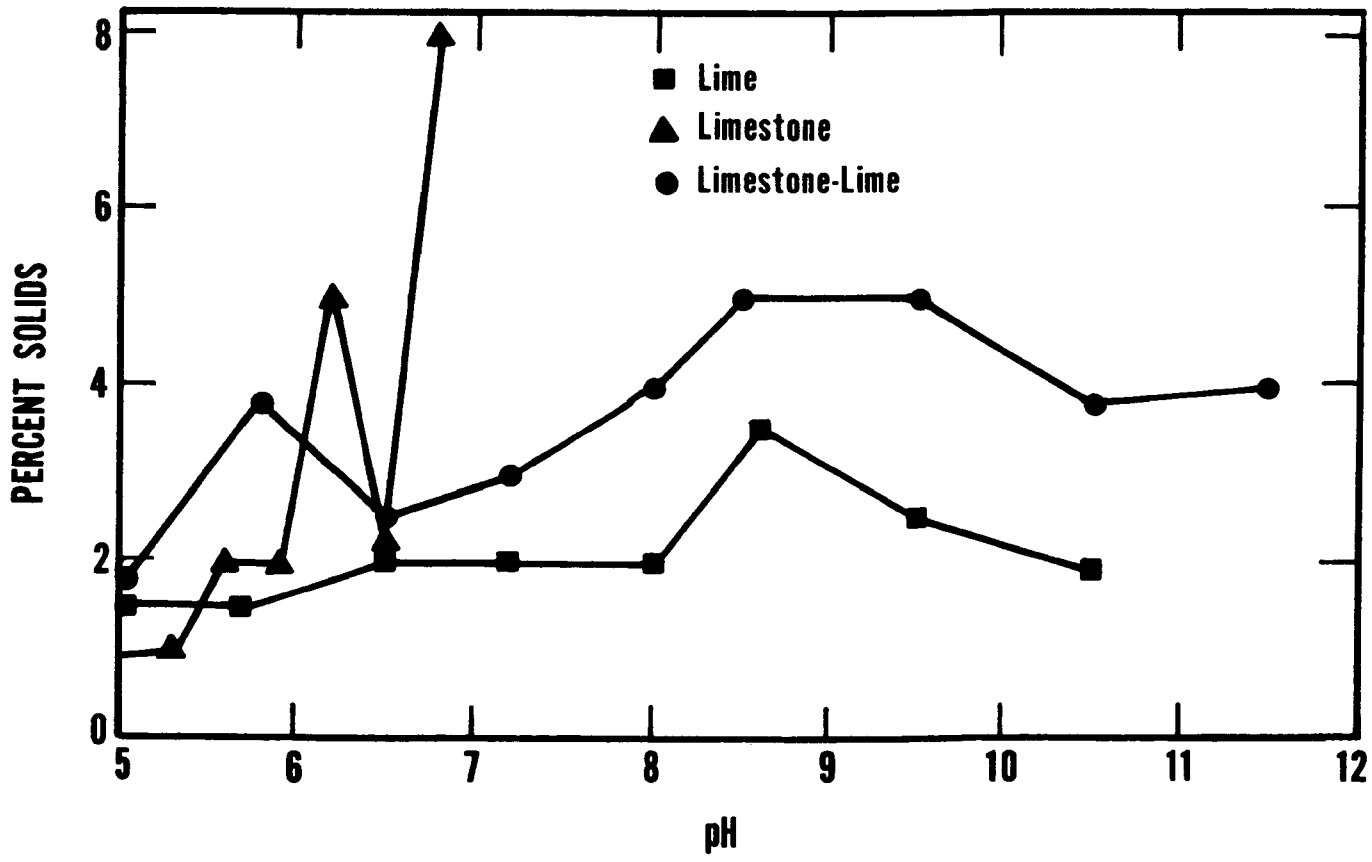

Figure 5. Sludge percent solids @ 1-hr. settling time as a function of pH.

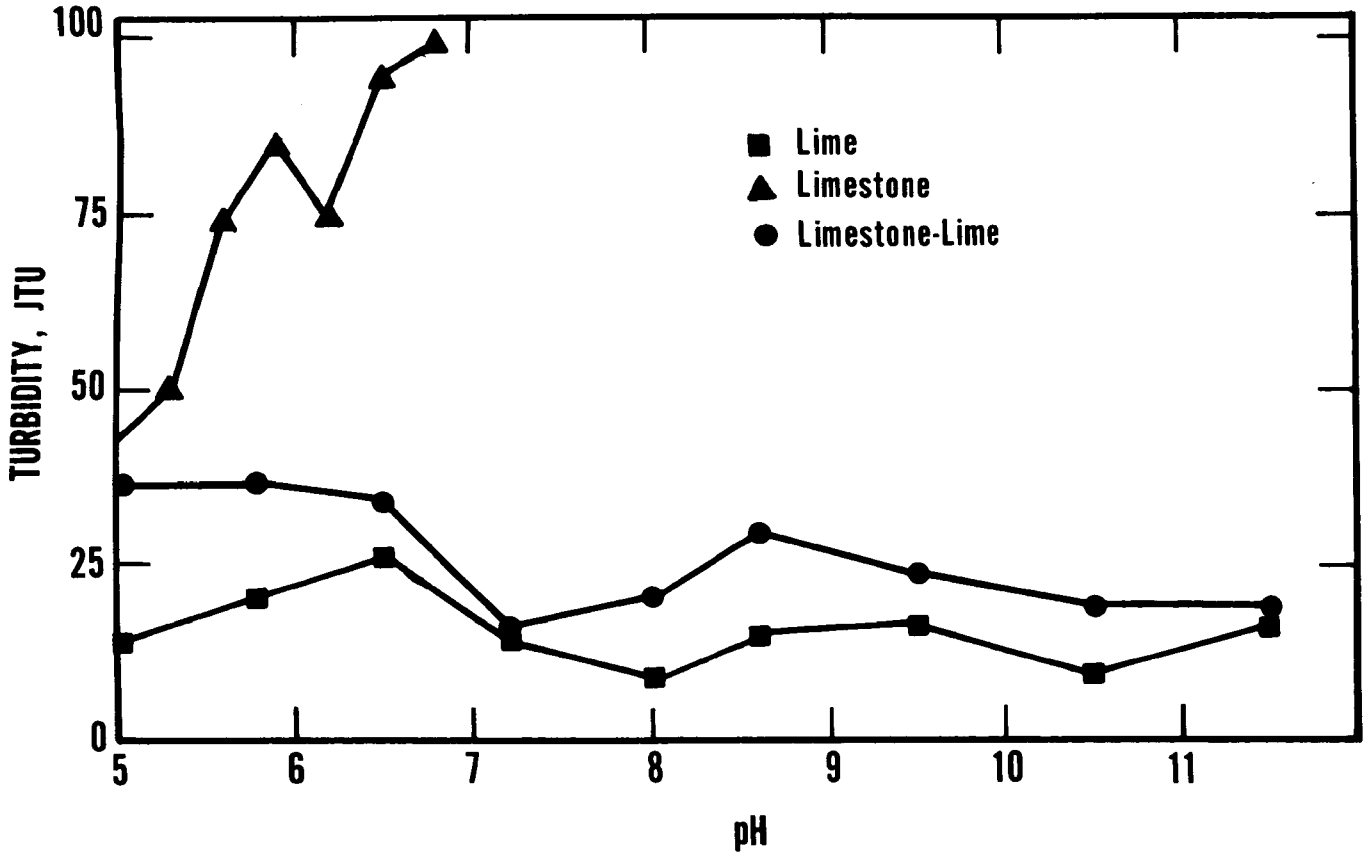

Figure 6. Supernatant turbidity @ 1-hr. settling time as a function of pH.

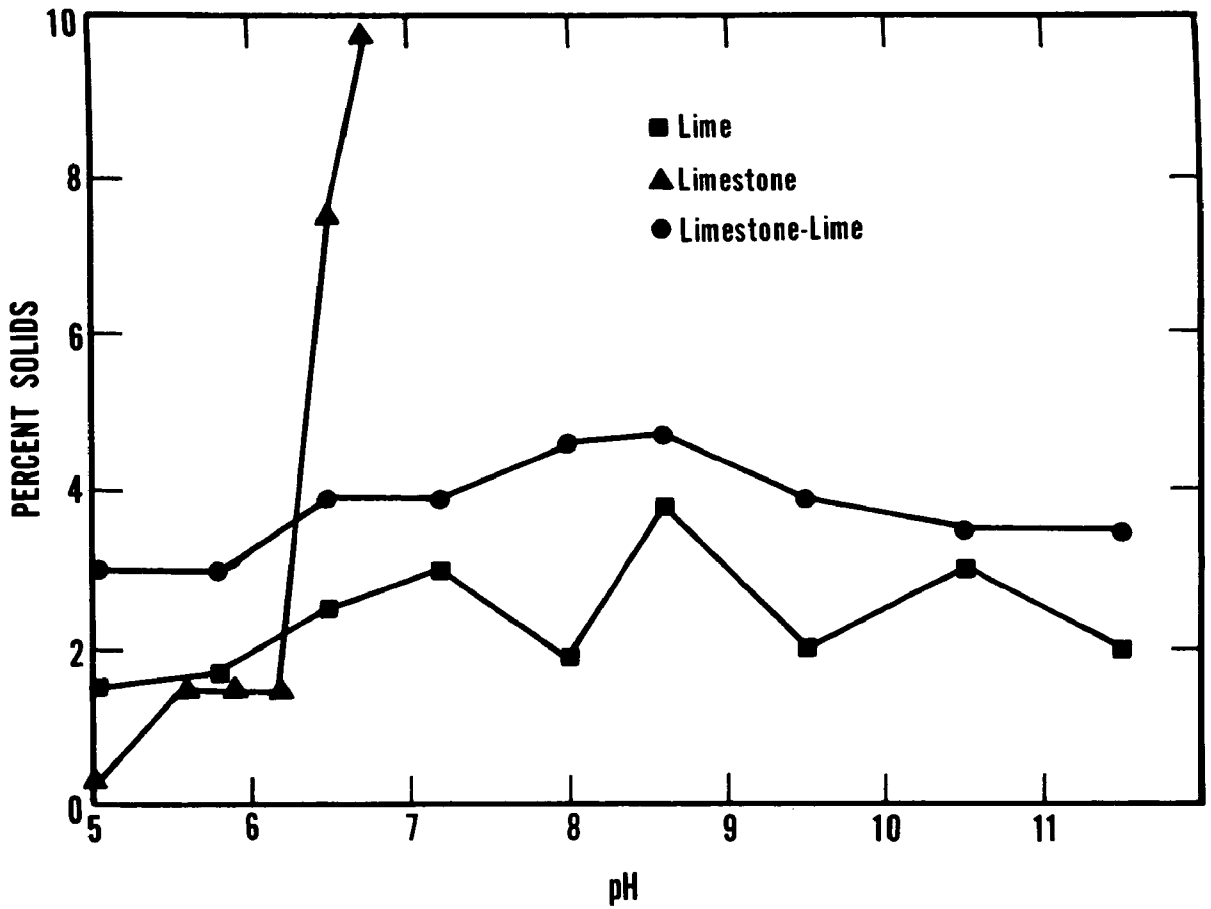

Figure 7. Sludge percent solids @ 24-hr. settling time as a function of pH.

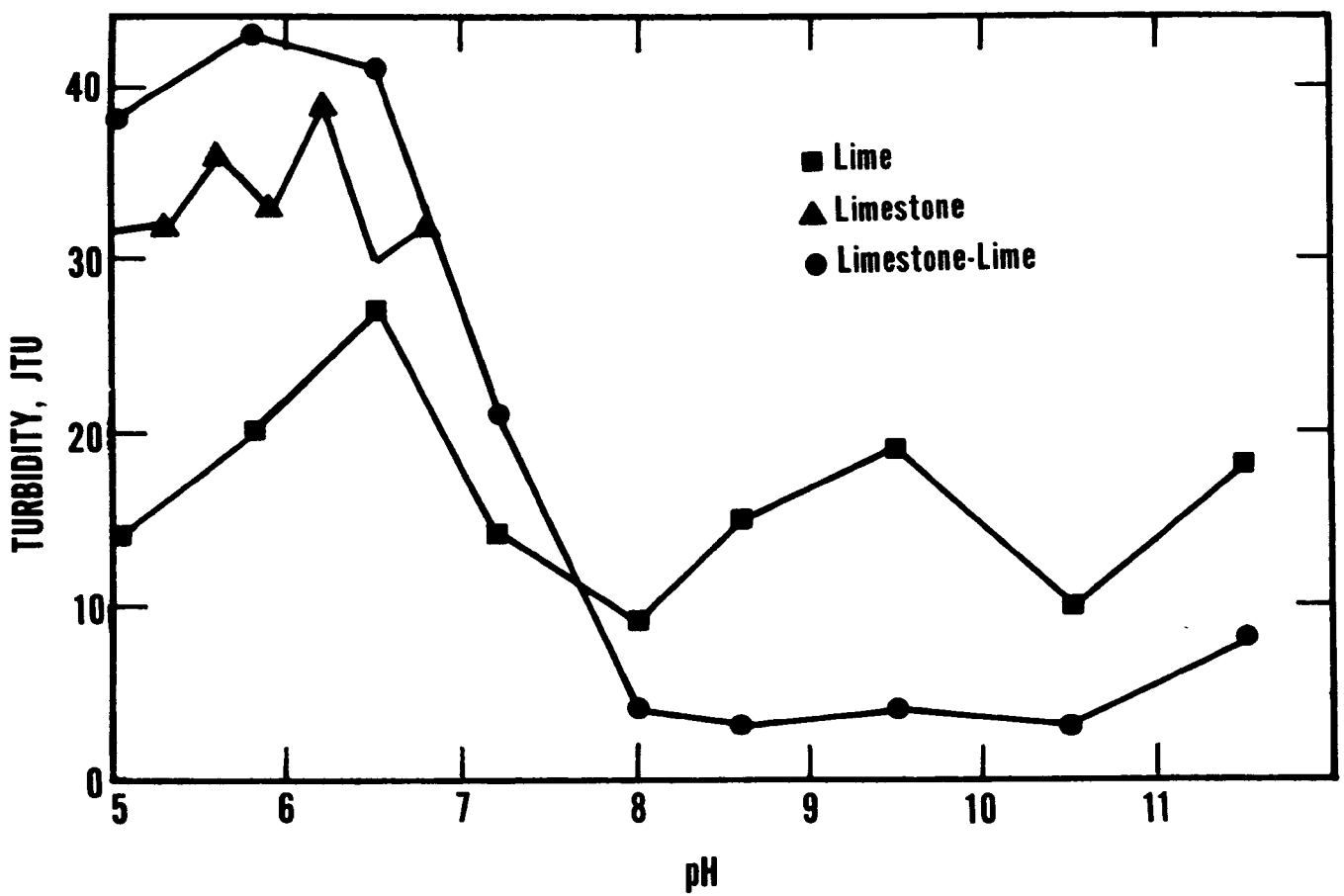

Figure 8. Supernatant turbidity @ 24-hr. settling time as a function of pH.

Table 8. COMBINATION LIMESTONE-LIME LABORATORY STUDY[a]

Treatment	Limestone			Lime		Total	Lime		Total
	First stage pH	Amount, g/l	Cost,[b] ¢/cu m	Amt. to pH 7, g/l	Cost, ¢/cu m	cost to pH 7.0, ¢/cu m	Amt. to pH 9.5, g/l	Cost, ¢/cu m	cost to pH 9.5, ¢/cu m
Limestone	6.8	3.2300	3.917			3.917			
Lime				0.4820	1.860	1.860	0.8280	3.195	3.195
Combination	5.1	0.0240	0.029	0.4310	1.663	1.692	0.6950	2.681	2.710
"	5.3	0.0830	0.101	0.4410	1.701	1.802	0.6660	2.570	2.671
"	5.5	0.1180	0.143	0.4380	1.690	1.833	0.6010	2.319	2.462
"	5.7	0.1500	0.182	0.4800	1.852	2.034	0.7380	2.847	3.029
"	5.9	0.1730	0.210	0.4780	1.844	2.054	0.6980	2.693	2.903
"	6.1	0.1910	0.232	0.4130	1.593	1.825	0.7130	2.751	2.983
"	6.3	0.4020	0.487	0.4110	1.586	2.073	0.7690	2.967	3.454

[a]Raw water pH = 5.0. Raw material cost: lime, $38.58/tonne; limestone, $12.13/tonne.

[b]To convert from cents/cu m to cents/1000 gal, multiply by 3.785.

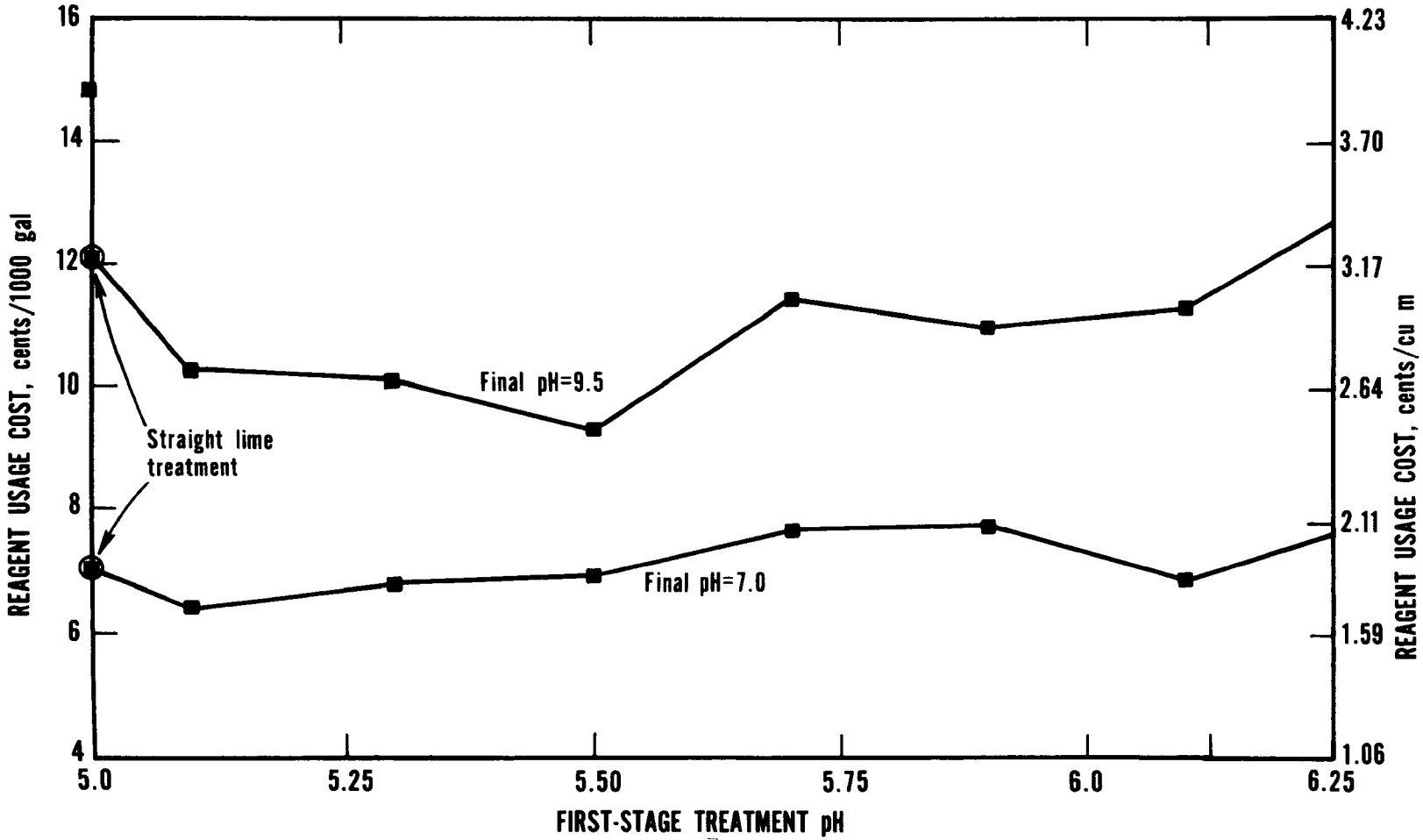

Figure 9. Batch-scale combination limestone-lime feasibility test where influent water pH was 5.0.

Treatment to pH 9.5 was most economical using combination treatment with a first-stage limestone pH of 5.5. In this case, combination treatment (9.3 cents/1000 gal) was 23 percent cheaper than straight lime treatment to pH 9.5 (12.1 cents/1000 gal). Limestone was incapable of treating to pH 9.5.

The raw-materials costs greatly influence the cost advantage of combination treatment. Any consideration of combination treatment would need to include the delivered cost of bulk lime and rock-dust for the plant site in question.

FULL-SCALE, CONTINUOUS-FLOW STUDIES ON COMBINATION TREATMENT

Approach

The basis for these studies (i.e., pH, detention time, reagent addition mode, reactor baffling system, etc.) was the earlier investigations by EPA on the ferric iron AMD at Norton, West Virginia[1] and at Crown.[2] In these earlier studies, it was determined that 20-30 minutes of detention time were required in the mixing reactor, that wire-mesh reactor baffles increased both mixing and utilization efficiencies, and that the rock-dust form of limestone yielded utilization efficiencies in the range of 50 percent treating to pH 6.5 on ferric iron water (Norton), 30 percent on ferrous water (Crown), and 50 percent on ferrous water using sludge recycling (Crown).

The basic avenue of approach toward the continuous-flow studies was that the first-stage limestone addition rate would be initially high for the first comparison and would incrementally diminish to cover a full spectrum of contingencies; thus, the optimum combination parameters could be identified, with the critical consideration being cost.

Test One

Basic data were obtained on combination treatment using a first-stage limestone pH near 6 followed by lime addition to pH 8. The limestone addition rate in Process B was approximately 40 percent higher than Process A although the first-stage effluent pH's were the same for both processes. The first-stage pH of 6 was chosen because the influent pH's during the test averaged 5.6. Limestone was fed semi-continuously by incorporating cycle timers to control the on/off frequency of the dry feeders. This was necessary because the small quantity of limestone required was below the minimum adjustment on the dry-feeder rate control. Typically, the limestone dry feeder ran one

minute out of three. The AMD flow rate was 1 liter/sec (16 gpm). Sludge recycling was not used for this test. No coagulants were used.

The results of the first study (Tables 9 and 17) indicated that although more limestone was being added to Process B than to A, Process B required more lime to reach the same end pH. Oddly, the higher level of limestone addition in Process B is not evidenced in either the limestone reactor suspended solids or the lime reactor suspended solids levels; both of which were lower for B than A.

Problems had been encountered during the test with plugging of the limestone feeders because the limestone storage bin was practically empty and the material was somewhat damp and difficult to control. A new load of limestone was obtained. In view of the unusual suspended solids results, some question must fall on the reliability of the limestone usage figures.

Effluent chemistry values were virtually identical for both processes with the exception that B had a slightly lower effluent suspended solids and turbidity level.

Test Two

In the second test, the rate of limestone addition was increased in Process B to provide a wider differential between process conditions in order to more closely evaluate the unexplained results of Test One. The damp limestone was eventually exhausted from the storage bin and more confidence could be placed in the limestone usage data obtained during this test.

Process B was receiving over 2.8 times the limestone quantity of Process A as shown in Table 10. The limestone reactor suspended solids levels were higher for B than for A in this test as contrasted with the results of Test One. The lime reactor effluent suspended solids, however, were about equal. As in Test One, a greater quantity of lime was required for Process B than for A. This is an unexpected and unexplained result. Total reagent cost for Process B was 46 percent greater than for A. Effluent quality parameters were virtually identical. An unusual observation was that the sludge percent solids from Process B averaged 10 percent as compared to 6.2 percent for Process A. This result suggested that the gross majority of the limestone failed to be utilized but passed directly to the sludge. Total (limestone and lime) reagent utilization efficiency was 51 percent for Process A and 30 percent for Process B. Effluent quality parameters are shown in Table 17.

Table 9. NEUTRALIZATION DATA SUMMARIES FOR COMBINATION TREATMENT: TEST ONE (BASIC DATA)

		Mean	Std. dev.
First-stage effluent pH	A	6.10	0.1
	B	6.12	0.1
Final effluent pH	A	7.96	0.1
	B	7.97	0.1
Limestone usage, kg/cu m	A	0.36	0.1
	B	0.49	0.1
Lime usage, kg/cu m	A	0.48	0.1
	B	0.57	0.1
Cost, cents/1000 gal	A	8.59	1.1
	B	9.58	1.4
Cost, cents/cu m	A	2.27	0.3
	B	2.79	0.2
Cost, cents/10^3 cu m/ppm influent acidity	A	4.32	0.6
	B	5.33	0.7
Utilization efficiency, percent	A	62	9.8
	B	49	5.8
Stoichiometric factor (influent acidity)	A	1.90	0.3
	B	2.40	0.3
Sludge to waste, % of influent AMD	A	1.65	0.1
	B	1.60	0.1
Sludge recycled, % of influent AMD	A	0	-
	B	0	-
Dry solids recycled, lb/1000 gal	A	0	-
	B	0	-
Dry solids to waste, lb/1000 gal	A	7.8	0.9
	B	6.8	0.7
Dry solids to waste, kg/cu m	A	0.94	0.1
	B	0.82	0.1
Underflow solids, percent	A	5.7	0.6
	B	5.1	0.5
Effluent turbidity, JTU	A	33	5
	B	28	3
Limestone reactor suspended solids, mg/l	A	280	54
	B	240	37
Lime reactor suspended solids, mg/l	A	1250	240
	B	880	150
Effluent suspended solids, mg/l	A	40	7
	B	26	8
Difference in reagent usage cost between Process A and B, percent		23	

Table 10. NEUTRALIZATION DATA SUMMARIES FOR COMBINATION TREATMENT WHERE PROCESS B UTILIZED HIGHER LIMESTONE ADDITION RATE THAN A

		Mean	Std. dev.
First-stage effluent pH	A	6.13	0.1
	B	6.14	0.1
Final effluent pH	A	8.04	0.1
	B	8.04	0.1
Limestone usage, kg/cu m	A	0.37	0.03
	B	1.04	0.2
Lime usage, kg/cu m	A	0.52	0.01
	B	0.60	0.02
Cost, cents/1000 gal	A	9.27	0.2
	B	13.5	0.7
Cost, cents/ cu m	A	2.45	0.06
	B	3.57	0.2
Cost, cents/10^3 cu m/ppm influent acidity	A	5.39	0.9
	B	7.87	1.4
Utilization efficiency, percent	A	51	8.1
	B	30	5.8
Stoichiometric factor (influent acidity)	A	2.36	0.4
	B	4.07	0.8
Sludge to waste, % of influent AMD	A	1.65	0.1
	B	1.56	0.03
Sludge recycled, % of influent AMD	A	0	-
	B	0	-
Dry solids recycled, lb/1000 gal	A	0	-
	B	0	-
Dry solids to waste, lb/1000 gal	A	8.6	0.7
	B	13	1.6
Dry solids to waste, kg/cu m	A	1.04	0.1
	B	1.56	0.2
Underflow solids, percent	A	6.2	0.4
	B	10	1.3
Effluent turbidity, JTU	A	31	4
	B	29	3
Limestone reactor suspended solids, mg/l	A	260	30
	B	460	64
Lime reactor suspended solids, mg/l	A	1060	260
	B	1150	240
Effluent suspended solids, mg/l	A	31	8.9
	B	33	11
Difference in reagent usage cost between Process A and B, percent		46	

Earlier studies[2] at Crown on straight lime neutralization to pH 8 without sludge recycling resulted in reagent usage costs around 2.4 cents/cu m (9.1 cents/1000 gal). The 2.45 cents/cu m cost observed in Process A in this study indicates similar costs for combination treatment and straight lime treatment in situations without sludge recycling.

Test Three

The third study involving combination limestone-lime treatment investigated the relative effectiveness of adding 2.6 times as much limestone in the first step (Process B) as was added in Process A while incorporating sludge recycling in both processes. Results of this study are presented in Tables 11 and 17. A slight improvement was noted in utilization efficiencies by employing sludge recycling as compared to the previous study. Even though considerably more limestone was added to B than to A, the effluent pH's from the first stage were approximately equal. Also, Process B required more lime than Process A to reach pH 8. As would be expected, total utilization efficiency for Process B was considerably lower than that for A (33 vs 57 percent). The excessive limestone consumption of Process B was similarly reflected in underflow solids levels significantly higher than those of Process A (7.1 vs 4.5 percent) because of the unused limestone. Effluent quality of Process B (Table 17) was slightly better than that of Process A. Coagulants were injected into both processes during this study at 6-ppm rates (Dowell M144 anionic flocculant). Reagent cost (excluding coagulants) for Process B was 1.5 times that of Process A.

Several operational problems were noted during this study. First, pH probe fouling was much more severe (without coagulants) than had been observed in earlier lime studies at pH 8. The use of coagulants was necessary, therefore, to improve pH probe performance as well as to improve clarity and settling characteristics. Secondly, erroneous reactor suspended solids values were obtained in the earliest phases of the test because the suspension settled so rapidly that representative aliquots were not being obtained for suspended solid analyses. This was corrected by modifying the lab technique in obtaining the aliquot. It was necessary during the course of these studies to enlarge the coagulant reservoirs from two separate 450-liter (120-gal) tanks to a single 1900-liter (500-gal) tank equipped with a Lightnin' variable

Table 11. NEUTRALIZATION DATA SUMMARIES FOR COMBINATION LIMESTONE AND LIME WHERE PROCESS B UTILIZED 2.6 TIMES AS MUCH FIRST-STAGE LIMESTONE

		Mean	Std. dev.
First-stage effluent pH	A	6.68	0.1
	B	6.70	0.1
Final effluent pH	A	7.95	0.1
	B	7.99	0.06
Limestone usage, kg/cu m	A	.41	0.05
	B	1.07	0.14
Lime usage, kg/cu m	A	0.53	0.06
	B	0.66	0.07
Cost, cents/1000 gal	A	9.67	0.9
	B	14.54	1.6
Cost, cents/cu m	A	2.56	0.3
	B	3.84	0.4
Cost, cents/10^3 cu m/ppm influent acidity	A	4.77	0.6
	B	7.27	1.7
Utilization efficiency, percent	A	57	6.6
	B	33	6.2
Stoichiometric factor (influent acidity)	A	2.12	0.3
	B	3.71	0.9
Sludge to waste, % of influent AMD	A	1.41	0.1
	B	1.40	0.2
Sludge recycled, % of influent AMD	A	19.9	1.4
	B	17.3	0.9
Dry solids recycled, lb/1000 gal	A	78	42
	B	100	60
Dry solids to waste, lb/1000 gal	A	5.4	2.3
	B	8.1	4.5
Dry solids to waste, kg/cu m	A	0.64	0.3
	B	0.96	0.5
Underflow solids, percent	A	4.54	2.1
	B	7.10	4.3
Effluent turbidity, JTU	A	17	2
	B	6	1.4
Limestone reactor suspended solids, mg/l	A	15000	3300
	B	18000	2800
Lime reactor suspended solids, mg/l	A	34000	-
	B	41000	-
Effluent suspended solids, mg/l	A	14	3.0
	B	9.0	3.5
Difference in reagent usage cost between Process A and B, percent		50	

speed mixer. This change eliminated virtually all the coagulant pumping, mixing, and service time problems that had been experienced earlier.

Test Four

The operating parameters for Process A were unchanged from the last test; i.e., combination treatment to pH 8, 0.9-liter/sec (14-gpm) flow rates, 20-percent (of the influent) sludge recycle rate, coagulant addition at approximately a 6-ppm rate, and a 2-percent sludge-to-waste rate. Process B utilized straight lime treatment with identical operating parameters to Process A. An extra mixing chamber (a 2-gal bucket) was installed in the Process A thickener to provide better mixing of the coagulant with the treated AMD. The coagulant was injected into the line to the thickener at a point approximately 3 meters (10 ft) from the thickener inlet. Process B's coagulant injection point was located approximately 12 meters (40 ft) from the B-thickener inlet and provided better contact between the coagulant and the AMD than was provided in Process A. The bucket was suspended beneath the Process A influent pipe so that all flow would first enter the bucket, mix, and then enter the thickener. This modification was quite effective as the effluent turbidities were reduced from as high as 30 JTU prior to use of the bucket to an average of 5 JTU. The combination treatment results (Table 12 and 17) indicated that a reduction in lime requirement was achieved by utilizing first-stage limestone addition followed by lime, in contrast to lime treatment alone. More importantly, the reagent cost for lime treatment (excluding coagulants) was 28 percent higher than for combination treatment (3.21 vs 2.51 cents/cu m or 12.13 vs 9.51 cents/1000 gal). Effluents from both processes were of comparable quality.

Test Five

All operating parameters were the same as those for Test Four except that the first-stage limestone addition rate was cut in half. The decrease in limestone usage resulted in an overall increase in the cost advantage of combination treatment over lime treatment (Table 13) as compared to Test Four (Table 12). Test Four used twice as much limestone as Test Five and exhibited a 28-percent cost advantage over lime, whereas Test Five's cost advantage was 34 percent. No appreciable difference was noted in the underflow solids concentrations (2.6 vs 3.5 percent).

Effluent quality parameters from all processes were comparable (Table 17).

Table 12. NEUTRALIZATION DATA SUMMARIES FOR COMBINATION TREATMENT (PROCESS A) VS LIME (PROCESS B) AT pH 8 USING COAGULANTS

		Mean	Std. dev.
First-stage effluent pH	A	6.72	0.2
	B	-	-
Final effluent pH	A	8.00	0.01
	B	8.00	0.00
Limestone usage, kg/cu m	A	.41	0.08
	B	0	-
Lime usage, kg/cu m	A	.52	0.04
	B	.83	0.12
Cost, cents/1000 gal	A	9.51	0.5
	B	12.13	1.8
Cost, cents/cu m	A	2.51	0.1
	B	3.21	0.5
Cost, cents/10^3 cu m/ppm influent acidity	A	4.70	0.8
	B	5.91	0.7
Utilization efficiency, percent	A	59	10
	B	58	6.6
Stoichiometric factor (influent acidity)	A	2.09	0.4
	B	2.15	0.4
Sludge to waste, % of influent AMD	A	1.92	0.2
	B	1.88	0.1
Sludge recycled, % of influent AMD	A	21.2	2.4
	B	17.8	0.9
Dry solids recycled, lb/1000 gal	A	65	33
	B	67	19
Dry solids to waste, lb/1000 gal	A	6.0	3.0
	B	7.1	1.9
Dry solids to waste, kg/cu m	A	0.71	0.3
	B	0.85	0.2
Underflow solids, percent	A	3.8	2.1
	B	4.5	1.3
Effluent turbidity, JTU	A	5	1.8
	B	5	1.1
Limestone reactor suspended solids, mg/l	A	13600	1500
	B	--	--
Lime reactor suspended solids, mg/l	A	21200	5000
	B	17200	3000
Effluent suspended solids, mg/l	A	7.2	2.7
	B	6.9	2.4
Difference in reagent usage cost between Process A and B, percent		28	

Table 13. NEUTRALIZATION DATA SUMMARIES FOR COMBINATION TREATMENT WITH REDUCED FIRST-STAGE LIMESTONE ADDITION (PROCESS A) VS LIME (PROCESS B) USING COAGULANTS

		Mean	Std. dev.
First-stage effluent pH	A	6.8	0.2
	B	-	-
Final effluent pH	A	8.0	0
	B	8.0	0
Limestone usage, kg/cu m	A	0.21	0.04
	B	0	0
Lime usage, kg/cu m	A	0.53	0.07
	B	0.79	0.1
Cost, cents/1000 gal	A	8.67	0.9
	B	11.60	1.5
Cost, cents/cu m	A	2.29	0.3
	B	3.06	0.4
Cost, cents/10^3 cu m/ppm influent acidity	A	4.15	0.5
	B	5.57	0.9
Utilization efficiency, percent	A	71	8.0
	B	63	9.1
Stoichiometric factor (influent acidity)	A	1.69	0.2
	B	1.95	0.3
Sludge to waste, % of influent AMD	A	1.98	0.1
	B	1.88	0.1
Sludge recycled, % of influent AMD	A	21.7	1.0
	B	17.6	1.0
Dry solids recycled, lb/1000 gal	A	43	22
	B	51	17
Dry solids to waste, lb/1000 gal	A	3.9	2.1
	B	5.5	1.8
Dry solids to waste, kg/cu m	A	0.51	0.2
	B	0.66	0.2
Underflow solids, percent	A	2.6	0.9
	B	3.5	1.1
Effluent turbidity, JTU	A	4	1.2
	B	4	1.4
Limestone reactor suspended solids, mg/l	A	13600	1200
	B	-	-
Lime reactor suspended solids, mg/l	A	21700	2600
	B	16400	2800
Effluent suspended solids, mg/l	A	5.5	2.4
	B	5.4	1.3
Difference in reagent usage cost between Process A and B, percent		34	

Test Six

The limestone addition rate was again reduced by a factor of 2 from Test Five values while all other operating parameters were unchanged. Coagulant addition rates for both processes were approximately 10 ppm. No improvement in the cost advantage of combination treatment was gained by the lower first-stage limestone addition rate of this study as compared to that of Test Five (33- vs 34-percent cost reduction, respectively). Operating parameters for this test are presented in Table 14 and chemical data are given in Table 17. As expected, the effluent quality parameters were comparable to the earlier studies. It was surprising that no change one way or the other was noted in the reagent usage cost advantage.

Test Seven

It was deduced that the cost advantage previously noted in the combination treatment studies may have been more strongly related to the sludge having been recycled into the first-stage reactor than to the effect of limestone addition. To test this, no limestone was added in the first-stage reactor. The recycled sludge contacted the raw AMD in this first step and then the lime was added in the second reactor (Process A). Process B remained the same; i.e., lime treatment and sludge recycling in a single reactor. Both processes used coagulant addition at approximately a 5-ppm rate. AMD flow rates remained at 0.9 liter/sec (14 gpm). Results of the study (Tables 15 and 17) indicated a 54-percent cost difference was obtained by recycling the sludge into a separate reactor for contact with the AMD in this two-stage process. Utilization efficiency averaged 99 percent for the two-stage process as opposed to 65 percent for the single-stage process. It was felt that a portion of the 99 percent figure might have been attributable to utilization of residual alkalinity present in the sludge from previous studies. To insure validity of the results, all process tanks were drained and cleaned and the test was repeated (Test Eight) reversing Processes A and B so that the two-stage process would be Process B rather than A.

Test Eight

As previously described, the process flow schemes were reversed from those of Test Seven. Sludge was recycled into the B limestone reactor (but no limestone was added) where it contacted the raw AMD. The sludge-AMD solution

Table 14. NEUTRALIZATION DATA SUMMARIES FOR COMBINATION TREATMENT WITH MINIMAL LIMESTONE ADDITION VS LIME TREATMENT USING COAGULANTS

		Mean	Std. dev.
First-stage effluent pH	A	6.8	0.2
	B	-	-
Final effluent pH	A	8.0	0.1
	B	8.0	0.1
Limestone usage, kg/cu m	A	0.130	0.01
	B	0	-
Lime usage, kg/cu m	A	0.51	0.05
	B	0.74	0.05
Cost, cents/1000 gal	A	8.06	0.8
	B	10.74	0.7
Cost, cents/cu m	A	2.13	0.2
	B	2.84	0.2
Cost, cents/10^3 cu m/ppm influent acidity	A	3.94	0.4
	B	5.27	0.7
Utilization efficiency, percent	A	79	6
	B	65	6
Stoichiometric factor (influent acidity)	A	1.52	0.2
	B	1.85	0.2
Sludge to waste, % of influent AMD	A	1.98	0.2
	B	1.97	0.1
Sludge recycled, % of influent AMD	A	21.5	2.7
	B	18.2	1.2
Dry solids recycled, lb/1000 gal	A	68	21
	B	58	28
Dry solids to waste, lb/1000 gal	A	6.3	1.9
	B	6.3	3.0
Dry solids to waste, kg/cu m	A	0.8	0.2
	B	0.8	0.4
Underflow solids, percent	A	3.8	1.1
	B	3.8	1.8
Effluent turbidity, JTU	A	5	1.5
	B	5	0.6
Limestone reactor suspended solids, mg/l	A	12000	2100
	B	-	-
Lime reactor suspended solids, mg/l	A	19200	2400
	B	15700	1800
Effluent suspended solids, mg/l	A	5.6	2.1
	B	5.7	2.2
Difference in reagent usage cost between Process A and B, percent		33	

Table 15. NEUTRALIZATION DATA SUMMARIES FOR LIME WHERE PROCESS A RECYCLED SLUDGE INTO A FIRST-STAGE REACTOR VS SINGLE-STAGE LIME

		Mean	Std. dev.
First-stage effluent pH	A	6.63	0.1
	B	-	-
Final effluent pH	A	8.01	0.02
	B	8.01	0.03
Lime usage, kg/cu m	A	0.46	0.04
	B	0.71	0.06
Cost, cents/1000 gal	A	6.71	0.6
	B	10.31	0.9
Cost, cents/cu m	A	1.77	0.2
	B	2.72	0.2
Cost, cents/10^3 cu m/ppm influent acidity	A	3.42	0.4
	B	5.25	0.6
Utilization efficiency, percent	A	99	8.4
	B	65	5.8
Stoichiometric factor (influent acidity)	A	1.20	0.1
	B	1.84	0.2
Sludge to waste, % of influent AMD	A	1.96	0.2
	B	2.23	0.2
Sludge recycled, % of influent AMD	A	19.9	2.3
	B	18.2	0.4
Dry solids recycled, lb/1000 gal	A	49	12
	B	66	19
Dry solids to waste, lb/1000 gal	A	4.9	1.2
	B	8.0	2.1
Dry solids to waste, kg/cu m	A	0.6	0.1
	B	1.0	0.2
Underflow solids, percent	A	3.3	1.2
	B	4.9	1.8
Effluent turbidity, JTU	A	5	1.4
	B	9	2.5
Limestone reactor suspended solids, mg/l	A	10300	2700
	B	-	-
Lime reactor suspended solids, mg/l	A	16200	5200
	B	11000	3200
Effluent suspended solids, mg/l	A	6.0	1.7
	B	10.5	4.1
Difference in reagent usage cost between Process A and B, percent		54	

was then pumped to the lime reactor for lime addition. Recycled sludge and lime were added to the AMD in the same vessel in Process A.

Results of the study (Tables 16 and 17) generally verified the findings of Test Seven. The cost difference between the two-stage process and single-stage process was 30 percent; a slightly lower figure than the 54 percent of Test Seven. The difference between the results was felt to be that Test Seven's sludge bed contained some residual alkalinity from earlier studies as previously discussed. The single-stage process utilization efficiency averaged 61 percent during this test, which compares favorably with the 65 percent observed during the previous test. The two-stage efficiency averaged 81 percent for this study as opposed to 99 percent for Test Seven.

MANGANESE REMOVAL

The Effluent Guidelines Division of EPA was preparing the recommended discharge standards for mine drainage at approximately the same time as the Crown tests were underway. The interim effluent guidelines that were developed by that Division and incorporated in the Federal Register (May 13, 1976) included limits for manganese of 2.0 mg/l on a 30-day continuous average and 4.0 mg/l for a daily maximum.

These manganese restrictions immediately prompted considerable controversy. All the data taken over the two years of neutralization studies at Crown were reviewed to collate the manganese levels of the effluents from a wide variety of test conditions.

Over 320 separate manganese analyses were performed on unfiltered effluents during the study period. Approximately 180 of these were on samples with a pH of 7 or above. An equal number of analyses were performed on filtered samples. The filtered sample data are not presented here but were consistently lower in manganese concentration than the unfiltered samples.

The Crown influent manganese concentration from 106 analyses averaged 5.2 mg/l with a standard deviation of 1.0 mg/l. A plot of the effluent manganese concentrations as a function of treatment pH is presented in Figure 10. The values were grouped in 0.5 pH intervals and are presented in Table 18. Manganese was very effectively removed whenever the effluent pH was above 8.0. Below pH 8, the systems had difficulty meeting the guidelines criteria.

Table 16. NEUTRALIZATION DATA SUMMARIES FOR LIME WHERE PROCESS B RECYCLED SLUDGE INTO A FIRST-STAGE REACTOR VS SINGLE-STAGE LIME

		Mean	Std. dev.
First-stage effluent pH	A	-	-
	B	6.5	0.2
Final effluent pH	A	8.0	0.11
	B	8.0	0.05
Limestone usage, kg/cu m	A	0	-
	B	0	-
Lime usage, kg/cu m	A	0.74	0.07
	B	0.57	0.07
Cost, cents/1000 gal	A	10.8	1.0
	B	8.3	1.0
Cost, cents/cu m	A	2.85	0.3
	B	2.19	0.3
Cost, cents/10^3 cu m/ppm influent acidity	A	5.52	0.9
	B	4.23	0.7
Utilization efficiency, percent	A	61	9
	B	81	12
Stoichiometric factor (influent acidity)	A	1.93	0.3
	B	1.48	0.2
Sludge to waste, % of influent AMD	A	2.4	0.5
	B	2.3	0.1
Sludge recycled, % of influent AMD	A	17.3	0.2
	B	16.9	0.5
Dry solids recycled, lb/1000 gal	A	54	20
	B	39	11
Dry solids to waste, lb/1000 gal	A	7.2	2.6
	B	5.3	1.4
Dry solids to waste, kg/cu m	A	0.9	0.3
	B	0.6	0.2
Underflow solids, percent	A	3.7	1.4
	B	2.8	0.8
Effluent turbidity, JTU	A	4	1.2
	B	5	1.6
Limestone reactor suspended solids, mg/l	A	-	-
	B	5500	1300
Lime reactor suspended solids, mg/l	A	8000	1600
	B	7200	1400
Effluent suspended solids, mg/l	A	5.1	2.6
	B	5.2	2.4
Difference in reagent usage cost between Process A and B, percent		30	

Table 17. CHEMISTRY ANALYSES FOR THE COMBINATION TREATMENT STUDIES

Sample	Cond	Acid	pH	Ca	Mg	Total iron	Fe^{2+}	Na	Al	Mn	SO_4	Alk	TDS
TEST ONE - BASIC DATA													
Raw feed	3700	570	5.6	440	120	290	250	480	15	6.3	3300	30	4600
Effluent A	3800	0	8.0	640	110	9.2	0	510	0.8	2.1	3100	110	4400
Effluent B	3800	0	7.9	630	100	8.7	0	440	0.9	1.9	2900	110	4100
Filtered A				640	110	1.3	0	500	0.1	2.0			
Filtered B				580	100	0.1	0	430	0.1	1.9			
TEST TWO - INCREASED DIFFERENCE IN LIMESTONE RATES													
Raw feed	3800	460	5.8	390	120	230	210	500	11	4.3	3000	37	4200
Effluent A	3700	0	8.0	600	110	10	0	500	0.9	1.6	3000	120	4200
Effluent B	3700	0	8.0	570	100	7.8	0	460	0.8	1.5	2800	120	3900
Filtered A				600	110	0.08	0	490	0.2	1.5			
Filtered B				570	100	0.07	0	450	0.1	1.5			
TEST THREE - B UTILIZED 2.6 TIMES AS MUCH FIRST-STAGE LIMESTONE													
Raw feed	3700	480	5.3	390	100	240	210	460	8	4	2900	62	4100
Effluent A	3700	0	7.9	550	100	4.0	0	450	0.1	2.1	2700	150	3800
Effluent B	3700	0	8.0	480	90	1.0	0	430	0.1	1.3	2400	130	3400

(cont'd)

Table 17. CHEMISTRY ANALYSES FOR THE COMBINATION TREATMENT STUDIES (cont'd)

Sample	Cond	Acid	pH	Ca	Mg	Total iron	Fe^{2+}	Na	Al	Mn	SO_4	Alk	TDS
TEST FOUR - COMBINATION VS LIME TREATMENT													
Raw feed	3700	550	5.6	400	120	260	250	470	8.4	6.3	3000	13	4200
Effluent A	3500	0	8.0	610	110	1.3	0	460	0.7	2.7	2900	110	4100
Effluent B	3400	0	8.0	580	110	1.4	0	450	0.3	1.5	2900	110	4000
Filtered A				560	100	0.3	0	450	0.3	2.7			
Filtered B				550	110	0.2	0	450	0.2	1.4			
TEST FIVE - COMBINATION TREATMENT WITH REDUCED FIRST-STAGE LIMESTONE VS LIME TREATMENT													
Raw feed	3600	630	5.2	340	100	290	270	380	15	5.4	2700	4.4	3800
Effluent A	3500	0	8.0	560	83	2.3	0	370	0.5	2.2	2500	100	3500
Effluent B	3500	0	8.0	560	95	1.4	0	360	0.2	1.2	2500	110	3500
Filtered A				550	83	0.1	0	360	0.3	2.1			
Filtered B				540	90	0.1	0	360	0.2	1.2			
TEST SIX - COMBINATION TREATMENT WITH MINIMAL LIMESTONE VS LIME USING COAGULANTS													
Raw feed	4100	600	5.3	350	110	270	250	370	10	6.0	2700	6.4	3750
Effluent A	3900	0	8.0	600	100	0.4	0	350	0.3	2.5	2500	110	3600
Effluent B	3900	0	8.0	550	100	0.6	0	350	0.4	1.1	2500	110	3500
Filtered A				600	95	0.1		350	0.1	2.4			
Filtered B				550	100	0.1		350	0.1	1.1			

(cont'd)

Table 17. CHEMISTRY ANALYSES FOR THE COMBINATION TREATMENT STUDIES (cont'd)

Sample	Cond	Acid	pH	Ca	Mg	Total iron	Fe^{2+}	Na	Al	Mn	SO_4	Alk	TDS
TEST SEVEN - LIME TREATMENT USING TWO STAGES (A) VS SINGLE STAGE (B)													
Raw feed	3300	510	5.1	340	100	240	230	410	9.2	5.5	2700	17	3800
Effluent A	3300	0	8.0	500	90	1.2	0	390	0.4	2.7	2400	110	3300
Effluent B	3300	0	8.0	480	93	1.2	0	380	0.2	1.4	2400	95	3300
Filtered A				460	85	0.1		360	0.2	2.6			
Filtered B				480	93	0.1		380	0.2	1.3			
TEST EIGHT - LIME TREATMENT USING TWO STAGES (B) VS SINGLE STAGE (A)													
Raw feed	3500	630	4.7	400	120	280	260	410	19	7	3000	4	4200
Effluent A	3500	0	8.0	650	100	0.7	0	400	0.4	1.4	2900	81	4000
Effluent B	3400	0	8.0	600	110	0.6	0	390	0.1	3.0	2800	97	3900
Filtered A				650	100	0.1		400	0.2	1.4			
Filtered B				570	110	0.1		380	0.1	3.0			

NOTE: All units are mg/l except for conductivity (micromhos/cm) and pH. Alkalinity and acidity are expressed as $CaCO_3$.

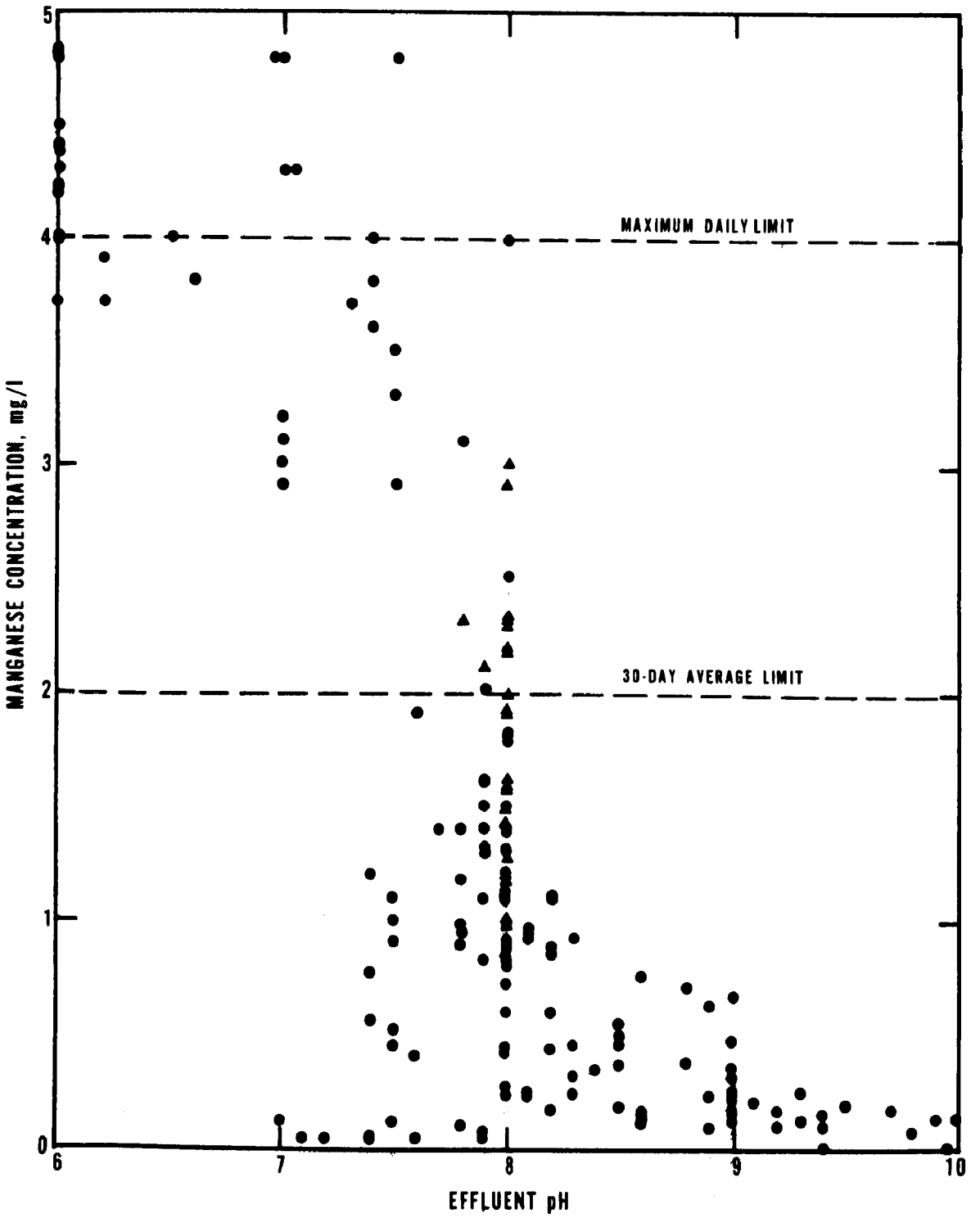

Figure 10. History of the manganese levels throughout the duration of the Crown neutralization studies.

Table 18. EFFLUENT MANGANESE CONCENTRATIONS

pH range	Samples meeting limit, percent	
	≤ 2.0 mg/l	≤ 4.0 mg/l
Below 6.5	0	8
6.5 to 6.99	0	26
7.0 to 7.49	39	83
7.5 to 7.99	81	97
8.0 to 8.49	87	100
8.5 to 8.99	100	100
9.0 to 9.49	100	100
9.5 to 9.99	100	100

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GLOSSARY

CALCULATIONS AND DEFINITIONS

Mean Probable Detention Time

A mathematical approximation of the average actual time a flowing liquid is detained in a vessel. The value is determined by tracer studies in which the tracer concentration exiting from the tank is monitored in respect to time. Two values are plotted on probability paper for each measurement point; i.e., cumulative tracer quantities to each point ÷ total cumulative tracer quantity measured (percent passing) and the ratio of elapsed time of each measurement ÷ theoretical detention time of the vessel. The theoretical time equals the tank volume divided by the flow rate. The resulting probability plot is interpolated to derive the time for 50-percent passing.

Detention Efficiency

The ratio of mean probable detention time to theoretical detention time expressed as a percentage.

Utilization Efficiency

A measure of the proportion of a neutralizer that reacts with the acid water as compared to the amount originally added. Since alkalinity imparted to the water is considered a benefit, the formula for utilization efficiency is:

$$\text{Utilization Efficiency} = \frac{\text{Alkalinity Used}}{\text{Alkalinity Added}}$$

therefore,

$$\text{Utilization Efficiency} = \frac{\text{Influent Acidity} - \text{Effluent Acidity} + \text{Effluent Alkalinity (all as } CaCO_3\text{)}}{\text{Alkalinity Added (as } CaCO_3\text{)}} \times 100$$

Stoichiometric Factor

The ratio of amount of neutralizer required to treat original amount of acid present:

$$\text{Stoichiometric Factor} = \frac{\text{Alkalinity Added (as } CaCO_3\text{)}}{\text{Influent Acidity (as } CaCO_3\text{)}}$$

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA-600/2-78-002	2.	3. RECIPIENT'S ACCESSION NO.
4. TITLE AND SUBTITLE COMBINATION LIMESTONE-LIME NEUTRALIZATION OF FERROUS IRON ACID MINE DRAINAGE		5. REPORT DATE January 1977 issuing date
		6. PERFORMING ORGANIZATION CODE 600/12
7. AUTHOR(S) Roger C. Wilmoth		8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Resource Extraction and Handling Division, Industrial Environmental Research Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268		10. PROGRAM ELEMENT NO. 1BB610
		11. CONTRACT/GRANT NO. In-house
12. SPONSORING AGENCY NAME AND ADDRESS Industrial Environmental Research Lab. - Cin., OH, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268		13. TYPE OF REPORT AND PERIOD COVERED Final 11/75 - 8/76
		14. SPONSORING AGENCY CODE EPA/600/12
15. SUPPLEMENTARY NOTES		

16. ABSTRACT

Studies were conducted on ferrous-iron acid mine drainage (AMD) treatment by a two-step neutralization process in which rock-dust limestone was mixed with the influent AMD and then hydrated lime was added in a polishing reactor. This combination treatment process resulted in reagent consumption cost reductions as high as 30 percent as compared to single-stage hydrated lime treatment of the same AMD. Later data indicated that an equal cost reduction (compared to single-stage lime treatment) could be achieved by a two-stage hydrated lime process in which the AMD and recycled sludge were mixed in the first reaction vessel and hydrated lime was added in the second reactor. No cost advantage for the combination process over straight hydrated lime treatment was felt to exist in situations where sludge recycling was not employed.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
*Limestone *Calcium hydroxides Neutralizing *Drainage	Acid Mine Drainage Coal Mine Drainage Ferrous Iron West Virginia Cost comparison Mine (excavations)	68 O

18. DISTRIBUTION STATEMENT Release to public	19. SECURITY CLASS (This Report) Unclassified	21. NO. OF PAGES 60
	20. SECURITY CLASS (This page) Unclassified	22. PRICE

EPA Form 2220-1 (9-73)

☆ U.S. GOVERNMENT PRINTING OFFICE: 1978— 757-140/6685