

NEUTRALIZATION OF HIGH FERRIC IRON ACID MINE DRAINAGE

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For the past year and one half, the Norton Mine Drainage Field Site of the Federal Water Quality Administration has been conducting research on the feasibility of acid mine drainage treatment using lime, limestone, and soda ash neutralization.

The Norton research facility is located at Norton, West Virginia, on the banks of Grassy Run--a small, heavily polluted stream of which an estimated 90 percent of the water flow is from abandoned mines. In Table 1, the water quality of Grassy Run is presented. This water was used for most experiments reported in this paper.

Table 1
Grassy Run Water Quality - 1968

<u>Parameter</u>	<u>Units</u>	<u>Max.</u>	<u>Min.</u>	<u>Mean</u>
pH		3.1	2.1	2.8
Specific Conductance	Mmhos/cm	1800	600	1190
Hot Acidity (as CaCO_3)	mg/l	680	137	466
Calcium (as CaCO_3)	mg/l	360	94	211
Magnesium (as CaCO_3)	mg/l	140	90	93
Hardness (as CaCO_3)	mg/l	470	134	304
Iron (Total)*	mg/l	200	10	93
Aluminum	mg/l	70	10	31
Sulfates	mg/l	980	270	621
Flow (Water Year 1968) ⁽¹⁾	cfs	278	1.2	7.52

*Ferric/Ferrous Ratio Greater than 4:1

The advent of stronger pollution laws and the shortage of reliable at-source pollution control techniques makes treatment a required abatement method. Treatment by neutralization appears, at this point, to be the most feasible (cost wise) of the various methods.

Most mine drainage neutralization research efforts have been done on ferrous iron mine waters. In contrast, the studies at Norton have concentrated on water with greater than 80 percent ferric iron.

PURPOSE OF STUDY

The purposes of this study were: 1. To investigate the effluent water quality characteristics of high ferric iron acid mine drainage when neutralized to various pH's by three types of neutralizing agents--lime, limestone, and soda ash, 2. To compare the effectiveness of each of the three neutralizing agents in neutralizing this acid mine drainage to various pH's, 3. To determine optimum basic operating conditions required to neutralize the acid mine drainage in order to produce an effluent water of desirable quality, 4. To acquire relevant operating data at the optimum conditions, derived from actual pilot plant operation, and 5. To enhance the efficiencies, if possible, of the limestone reaction.

FACILITY LAYOUT AND FLOW DIAGRAM

A simplified flow diagram of the neutralization test system is presented in Figure 1.

Water from Grassy Run was pumped into a 500 gallon fiberglass tank which was kept constantly full by a continuous overflow. This tank served as a constant head source and gravity fed a constant flow of raw water into a neutralization reactor. Flow rates through the system ranged from 1-6 gallons per minute. Control of this flow was accomplished by means of a globe valve (other type valves fouled or allowed the water flow rate to vary excessively). The reactor was a 55 gallon stainless steel tank on which a flash mixer was mounted. The rapid mixing was responsible for some short circuiting. Tracer tests yielded a mean logarithmic probability efficiency of 34.8 per cent. Into the reactor, a neutralizing agent could be fed in either the dry or slurry form. The dry feeder used was a volumetric type disc feeder which performed reliably and had a feed accuracy of ± 2 per cent. The slurry feed system consisted of a storage tank exactly like the reactor tank with a flash mixer. Constant agitation was supplied by the mixer to keep the relatively insoluble neutralizing agent in suspension. Small precise chemical feed pumps were used to transfer the slurry from the slurry storage tank into the reactor. These pumps were electrical solenoid type, rapid impulse, diaphragm pumps chosen for their ability to pump the heavy slurry accurately.

The reactor contained a pH probe for a recording pH meter. This meter was used for precise monitoring and control of the system.

Water level in the reactor was maintained by float switch operated pumps which transferred the neutralized water from the reactor into a 1250 gallon upflow settling tank. Tracer tests of the settling tank yielded a mean logarithmic probability efficiency of 80 per cent.

Supernatant from the settling tank, which was considered treated water, overflowed into a sampling line and then was returned to Grassy Run. The sludge from the settling tank could be either periodically or continually withdrawn and transferred to a holding pond for periodic disposal.

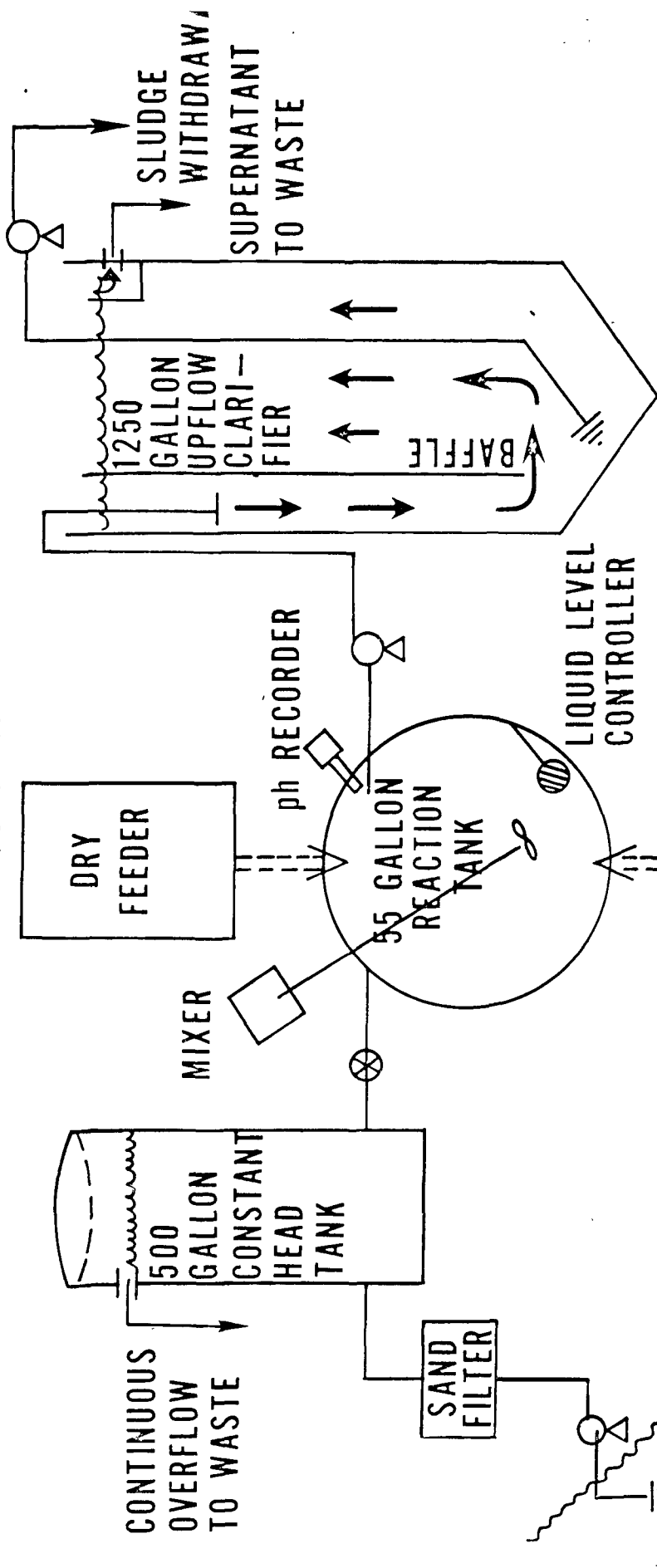
The water was filtered prior to treatment with a pressurized rapid sand filter to remove large particle matter which tended to clog the valves and thus vary the flow. Filtration would not be necessary in full scale plants or large pilot plants.

PROCEDURES

Normal operational procedures during a test run involved the following:

The pH was the only meaningful parameter that could be continuously and immediately monitored and was used for operational control. The raw water flow was controlled by the globe valve which maintained a constant flow of feed water into the reactor. By adjusting the neutralizer feed rate, a constant pH was established in the reactor. Samples were taken from the reactor after a few hours operation, when the system had come to equilibrium. However, several days operation was required to allow the formation of an active sludge blanket before the settling tank effluent was sampled. Once the sludge blanket was established, normal sampling procedures were begun. Samples were taken from the inflow (raw Grassy Run water), treated water (immediately from the discharge of the reactor), and from the settling tank effluent. Rates of the raw water inflow and neutralizing agent feed were measured and the temperature of all three water samples was recorded. A sample was also taken to

DRY FEED SYSTEM



SLURRY FEED SYSTEM

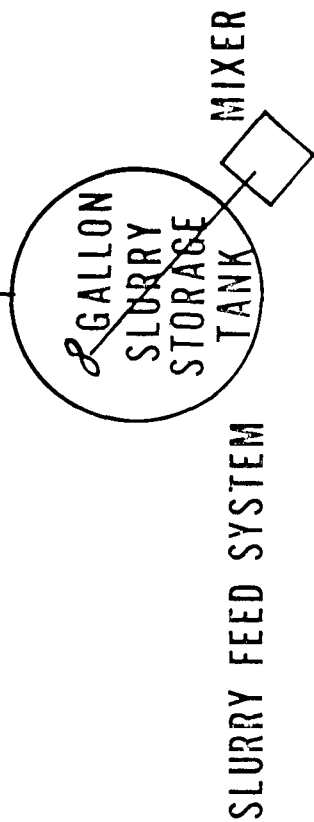


FIGURE 1

SIMPLIFIED FLOW DIAGRAM

measure the sludge settling rate and volume. This sample of treated water was taken immediately from the reactor discharge and placed in a 1000 ml graduate. The height of the precipitated sludge was recorded every fifteen minutes for a period of one hour. A few long term settling tests were made with periodic readings for total time lengths of 24 hours and longer.

If the slurry feed method of introducing the neutralizing agent was used, the slurry mixture was prepared on a batch basis in a separate tank and then dumped into the slurry storage tank. A flash mixer constantly agitated the slurry to keep the relatively insoluble neutralizing agent in suspension. Electronic impulse diaphragm pumps, whose flow could be varied electronically, transferred the slurry into the reactor. Even with these specialized pumps, a five per cent (by weight) limestone slurry was the maximum concentration (of limestone) the pumps were able to transfer. Although lime slurries were much easier to keep in solution (as calcium hydroxide is much more soluble than calcium carbonate), fouling problems were also experienced with it. Studies were not performed on soda ash slurries.

When the dry feed method of neutralization was used, control of the neutralizer feed rate was accomplished by adjusting a variable speed transmission on the dry feeder.

During aeration studies in the reactor, a 3/4" PVC plastic pipe 'U' was inserted in the reactor tank. The sides of the 'U' fit vertically against the sides of the reactor tank and the bottom of the 'U' laid near the bottom of the tank. The bottom of the 'U' was drilled with a number of 1/16" holes spaced to provide an equal distribution of air throughout the reactor. An air compressor supplied a regulator which, in turn, controlled the air flow through the aerator. The compressor provided excellent intermittent service, but overheated in continuous duty situations.

When aeration of the settling tank was studied, a rectangular aerator, made from four lengths of 3/4" PVC piping, was suspended above the sludge blanket. Holes 1/16" in diameter were drilled in the rectangular aerator and spaced to provide maximum dispersion. Sufficient air pressure was used to maintain adequate agitation of the supernatant. Initially, the rectangular aerator was placed eight inches above the sludge blanket, but the turbulence disturbed the blanket. The aerator was then moved to a height of fifteen inches above the blanket.

Analytical Procedures

As pH was found to vary with time following neutralization and as the solubility of metal salts in the water is dependent upon pH, it was critical to establish a standard time of analysis following neutralization.

A study of pH change following neutralization was conducted to determine the optimum time for analysis. Neutralized samples of acid mine water were agitated by flash mixers for ten minutes. Then the samples were allowed to set undisturbed and the supernatant pH periodically recorded. Figure 2 indicates the pH change with time after the addition of lime, limestone, and soda ash to identical samples of acid mine drainage.

In the case of lime, the reaction was rapid and the pH was stabilized after one hour, thus indicating that the reaction had gone to completion. However, where limestone was the neutralizing agent, the pH had not stabilized even after 96 hours. In the case of soda ash, the pH change curve closely resembles the limestone curve and the reaction required a comparable time to go to completion. Therefore, for our use, a standard procedure of analyzing

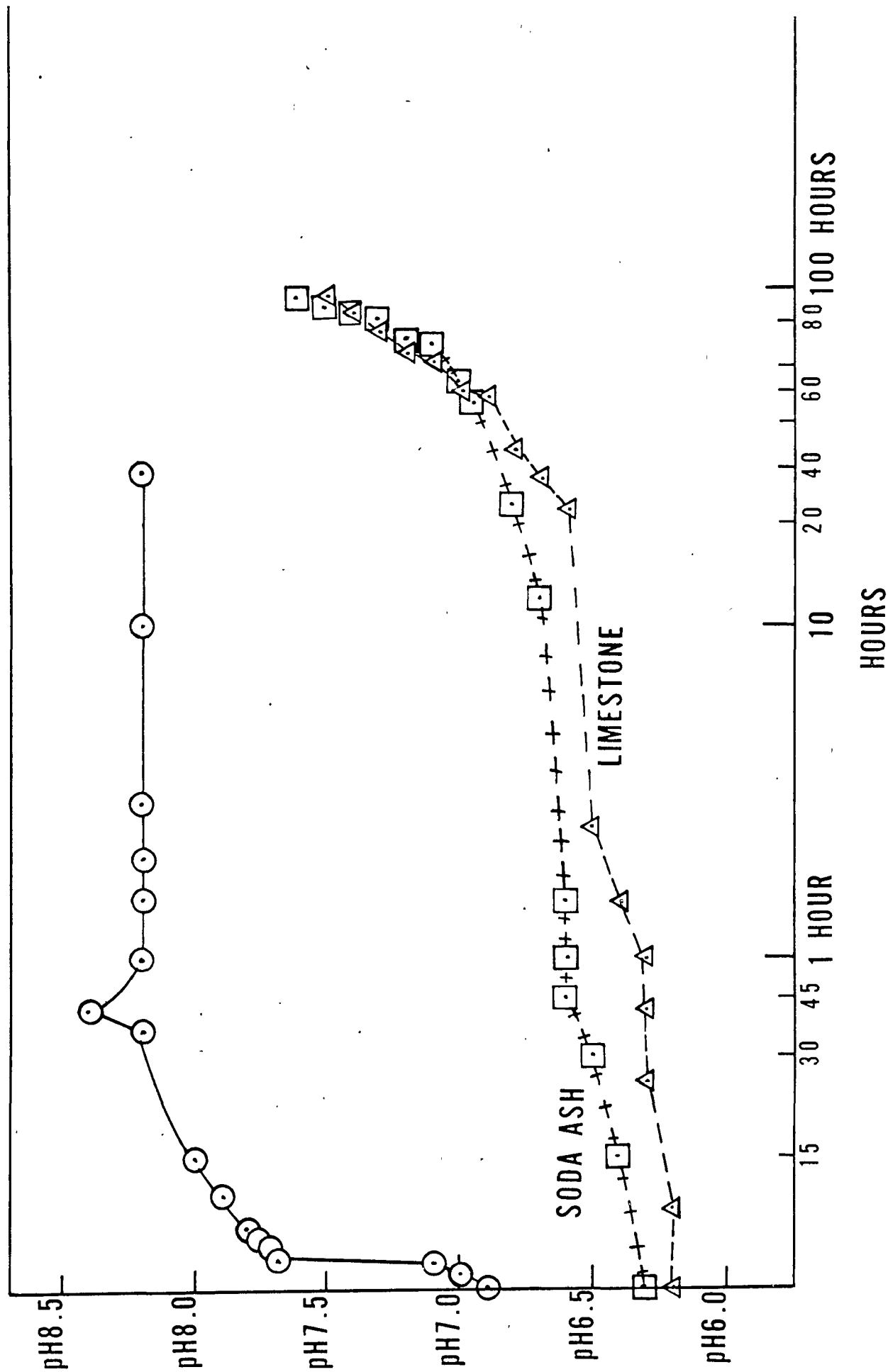


FIGURE 2

pH CHANGE (SAMPLES TAKEN IMMEDIATELY FROM REACTOR)

samples just prior to 24 hours after sampling was adopted. At this time, the lime reaction had gone to completion and the limestone and soda ash reactions have had what we felt to be the maximum detention time which could be found in any operational neutralization plant. Also, this 24 hour standard conformed well to the work schedule of the chemistry laboratory.

Chemical Analytical Procedures

Water - Cold acidity was measured by automatic potentiometric titration to an end point of pH 8.3. Hot acidity was done by potentiometrically titrating a boiling sample to an end point of pH 8.3. Iron, aluminum, calcium, and magnesium determinations were by atomic absorption spectrophotometry. Hardness was the summation of calcium and magnesium. Standard Methods (2) were used for the alkalinity, total residue (TR), and sulfate determinations. The supernatant was used for these analyses rather than the total sample. Sludge alkalinity and sludge total residue were also determined by Standard Methods procedures.(2)

Utilization Efficiency

In order to compare the different methods of neutralization, the efficiency of usage of each chemical was computed. This Utilization Efficiency was defined as the ratio of Neutralizing Agent Used/Neutralizing Agent Added. This concept includes not only the alkalinity used to counteract acidity, but also includes the excess alkalinity imparted to the supernatant (which will be discharged into a stream), thus it was felt that a benefit was derived from not only the acidity removed but also the alkalinity added to the water. The alkalinity precipitated with the sludge was considered a loss. Therefore, the formula for determining efficiency was:

$$\text{Utilization Efficiency (Per Cent)} = (H + A) \times 100 / (N \times P)$$

Where H = Hot Acidity Removed (as CaCO_3)

A = Alkalinity of Supernatant (as CaCO_3)

N = Neutralizing Agent Used (CaCO_3 equivalent)

P = Per Cent Purity of Neutralizing Agent

Particle Size

The importance of particle size in the neutralization reaction has been emphasized in studies by Ford, Young, and Glenn,(3) which indicated that decreasing the particle size of limestone increased both the rate and efficiency of the neutralization reaction. They recommend a particle size of 400 mesh or smaller. Ford and Young tested thirteen limestones from the Eastern United States and rated the limestone used during these studies (Germany Valley Limestone) as the most efficient in reference to reactivity with acid mine drainage. A spectrochemical analysis of this limestone was made by Bituminous Coal Research and is presented in Table 2.

Since particle size is so important, sieve tests were made on the three neutralizing agents used. These size determinations were made on a standard sieve shaker with 50, 100, 200, and 400 mesh screens. All tests were made by shaking the sample for one hour and then weighing the portion of the sample retained on each screen.

Initial results from the sieve analyses indicated a larger particle size than was advertised by the suppliers. It was suspected that water had been absorbed by the neutralizing agents and was causing the material to agglomerate. Therefore, the tests were rerun after drying the neutralizing agents by the direct application of heat. Table 3 presents the results of these tests. It is evident that water had been absorbed.

Table 2
Spectrochemical Analyses of Germany Valley Limestone⁽³⁾
Reported as Per Cent by Weight of Ignited Sample (900° C)
BCR Sample No. 2177

Loss on Ignition	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	TiO ₂	Na ₂ O	K ₂ O	MnO ₂
43.0%	1.0%	0.43%	0.15%	1.16%	97.0%	0.04%	0.02%	0.1%	0.03%

Per Cent CaCO₃ = 96.5%*

X-Ray analysis of the limestone identified only one compound - CaCO₃

*This figure was used in limestone utilization efficiency calculations as P

Table 3
Lime, Limestone, and Soda Ash Particle Size

Per Cent Passing

Screen Size	Lime		Limestone		Soda Ash	
	Before Drying	Dried	Before Drying	Dried	Before Drying	Dried
50 Mesh	92.1%	89.7%	98.1%	99.2%	49.1%	49.0%
100 Mesh	36.0%	55.1%	42.2%	57.7%	9.2%	10.0%
200 Mesh	5.6%	43.5%	26.6%	34.4%	0.01%	1.5%
400 Mesh	1.5%	3.8%	4.5%	14.8%	0%	0.6%
% Not Passing 50 Mesh	7.9%	10.3%	1.9%	0.8%	50.9%	51.0%

The limestone and 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 at a reasonable cost, "rock dust" was used. The soda ash was from Diamond Shamrock Corporation, Cleveland, Ohio.*

In Table 4, the chemical analyses as quoted by the manufacturers are presented along with the respective costs of the chemical.

Table 4
Manufacturer's Chemical Analyses

Hydrated Lime		Limestone		Soda Ash	
CaO	72.00% Min.	CaO	53.0 %	Na ₂ O	58%
MgO	00.40% Min.	MgO	0.38%	Na ₂ CO ₃	99-100%
CaCO ₃ Equivalent	130% Min.	CaCO ₃	98.8 %		
		SiO ₂	0.49%		

Cost

\$17.00 per ton
in bags

\$6.00 per ton
in bags

\$100.00 per ton
in bags

* Mention of commercial products does not imply endorsement by the Federal Water Pollution Control Administration.

Concentration vs. pH Tests

Tests to indicate the relative concentrations of the chemical constituents at various pH's were run by the following procedure: First, a five gallon sample of water from Grassy Run was taken into the lab and allowed to reach ambient temperature. The water was then divided into one liter portions. A separate sample was used for each pH point in the test. The neutralizing agent was added to the sample in sufficient quantity to bring the sample to the desired pH. A laboratory mixer was used to agitate the sample. Once the sample pH had stabilized, the supernatant was withdrawn and analyzed. As discussed earlier, the time required for pH stabilization was dependent on the neutralizing agent used. A 24-hour maximum limit was placed upon the reaction times, thus the limestone and soda ash pH readings and supernatant analyses were made at the 24 hour point. By running all the concentration tests from one large water sample, many variables were eliminated and direct comparisons could be made between the results of each of the three neutralizing agents.

RESULTS

Concentration vs. pH Tests

Tests were run on identical samples of Grassy Run water to determine the quality of the supernatant when the sample was neutralized to various pH's. Lime, limestone, and soda ash were each tested as the neutralizing agents. The results of the tests are presented in Table 5.

When lime was used, the acidity was removed between a pH of 4.6 and 5.0. At this pH, the iron, sulfate, and aluminum had been decreased to values near minimum levels and the calcium, hardness, and magnesium had increased as a result of the lime addition.

It was not until a pH of 9.2 that the magnesium was reduced to a low level. The addition of more lime, beyond a pH of 5.9 only served to increase the pH and decrease the magnesium since there were no further significant increases in the alkalinity or calcium concentrations. Thus, above pH 5.9, the lime was being lost to the sludge. It should be noted that the iron and aluminum were never reduced below 2 mg/l. Referring to our definition of utilization efficiency, it can be seen that the greatest efficiency would be obtained between a pH of 4.6 and 5.0, because at this pH the demand by the acidity had been satisfied. When more lime was added, at first a small amount was used to increase the alkalinity and the rest was lost to the sludge. Eventually all of the further addition of lime would be lost to the sludge.

In these tests, limestone was not able to increase the pH beyond 6.5, because of the formation of carbonic acid, which acted as a buffer. An indication of the carbonic acid is seen in the cold acidity analysis as the difference between the hot and cold acidity is mostly due to carbonic acid. At a pH of 3.9, the hot acidity was removed but it wasn't until pH 5.5 that the iron was reduced to a minimum of 2 mg/l. Aluminum reached its minimum at pH 4.3. Greatest utilization efficiency, as defined in this paper, occurred at pH 3.9. Above this pH, some of the limestone is utilized to increase the alkalinity, but the majority is lost to the sludge.

Soda ash removed all the acidity at a pH slightly above 4.7. The carbon dioxide liberated during the reaction caused the continued presence of cold acidity. Although iron results were erratic, it can be concluded that a concentration of approximately 3 mg/l is the minimum value. Aluminum reached its minimum at approximately 2 mg/l at pH 5.4. Continued increase of soda ash addition beyond pH 4.7 served primarily to increase the pH, alkalinity, and sodium concentration. Maximum utilization efficiency occurred at pH 4.7.

Table 5
Concentration vs. pH*

pH	Conductivity	Hot Acid	Calcium	Magnesium	Total Hardness	Cold Acid	Sulfate	Iron	Aluminum	Alkalinity	Sodium
<u>RAW WATER</u>											
2.8	1300	594	396	174	570	680	1542	130	44		2.0
<u>LIME</u>											
3.5	1100	261	816	180	996	300	1192	10.6	40		3.9
3.8	1060	168	938	182	1120	205	1192	4.3	34		2.9
4.2	1060	34	1031	185	1216	50	1205	3.0	7		2.7
4.6	1100	2	1181	185	1366	0	1156	2.8	1.6	6	3.0
5.0	1100	0	1181	182	1363	0	1180	8.5	3.1	14	2.7
5.3	1100	0	1181	170	1351	12	1192	4.3	3.2	20	3.3
5.6	1100	0	1266	167	1433	7	1180	5.5	4.0	22	3.1
5.9	1100	0	1312	152	1464	0	1192	4.9	4.6	30	2.7
6.1	1100	0	1312	144	1456	0	1192	3.7	4.2	38	3.0
6.4	1100	0	1312	142	1454	0	1192	2.5	4.1	30	3.2
6.7	1100	0	1312	134	1446	0	1192	2.4	3.9	32	2.8
7.0	1100	0	1312	113	1425	0	1192	2.7	3.3	32	2.8
7.3	1100	0	1500	52	1552	0	1192	3.0	2.7	30	3.4
8.3	1100	0	1500	16	1516	0	1192	3.3	2.8	36	3.2
9.2	1100	0	1575	8	1583	0	1180	2.5	3.5	40	3.3
<u>LIMESTONE</u>											
3.6	1160	24	890	178	1068	269	1132	5.8	36		2.4
3.9	1100	0	998	179	1177	160	1144	5.5	24		3.2
4.3	1100	0	1096	179	1275	35	1156	4.5	2.6		3.1
4.8	1200	0	1331	182	1513	16	1192	3.3	2.1	44	2.9
5.0	1200	0	1500	185	1685	8	1192	4.1	2.2	58	3.1
5.5	1200	0	1181	185	1366	11	1084	1.9	1.9	64	3.1
6.5	1200	0	1235	192	1427	12	1110	2.0	2.0	71	3.1

Hot Acid, Calcium, Magnesium, Total Hardness and Cold Acid are expressed as CaCO₃ equivalents

Table 5 (Continued)
Concentration vs. pH*

pH	Conduct- ivity	Hot Acid	Calcium	Magne- sium	Total Hardness	Cold Acid	Sulfate	Iron	Alum- inum	Alka- linity	Sodium
<u>SODA ASH</u>											
3.1	1200	240	422	178	600	320	1156	10.0	42		165
3.5	1200	172	404	179	583	251	1192	5.5	38		204
4.0	1200	68	401	179	580	95	650	7.2	16		320
4.7	1300	2	401	182	583	22	1301	7.4	2.9	14	330
5.4	1500	0	401	182	583	26	1638	3.7	2.3	120	450
5.8	1600	0	375	182	557	47	1783	10.6	4.0	166	495
6.2	1600	0	375	185	560	30	1638	4.6	2.3	140	510
6.5	1700	0	372	179	551	11	1350	5.0	2.4	220	645
6.9	1700	0	355	176	531	27	1638	10.0	3.9	246	690
7.3	1700	0	332	172	504	0	1687	2.6	2.3	320	750
7.6	1700	0	315	170	485	0	1422	2.8	2.5	284	1000
8.3	1700	0	232	164	396	0	1398	2.7	3.0	220	1000
9.0	1700	0	161	154	315	0	1350	2.8	3.9	316	960
9.6	2800	0	58	123	181	0	1156	3.0	7.3	1028	1460

All units are mg/l except for Conductivity (Mmhos/cm) and pH.
*All Analyses Made After 24 Hours

Hot Acid, Calcium, Magnesium, Total Hardness and Cold Acid are expressed as CaCO₃ equivalents

One of the pronounced results of these tests was the continued presence of iron, even at high pH levels. The presence of iron is in contrast to published 'ideal' solubility curves, such as those by Hill(4) which indicate complete ferric iron removal above pH 5.0. Similar difficulties in iron removal have been reported by Maneval, et al.(5)

Since the analyses were made on unfiltered samples, it was suspected that possibly the iron was remaining in suspension rather than in solution. Therefore, three 'suspect' samples were analyzed for iron, then filtered through a 0.45 micron filter to remove all particle matter and the iron analysis was repeated. The results of this test are in Table 6. Obviously, the iron concentration was not affected by filtration, thus the iron does not exist in a suspended particle state unless it is smaller than 0.45 microns. The iron may be involved in an organic complex. However, no studies have been made to resolve this question.

Table 6
Effects of Filtration
Upon Iron Concentration

Suspect Sample	pH	Total Iron Concentration	
		Before Filtering	After Filtering
L-6	7.0	6.7	6.6
L-7	7.3	10.1	10.2
L-8	8.0	17.2	17.1
L-9	10.0	2.0	2.0

Filter Size = 0.45 microns

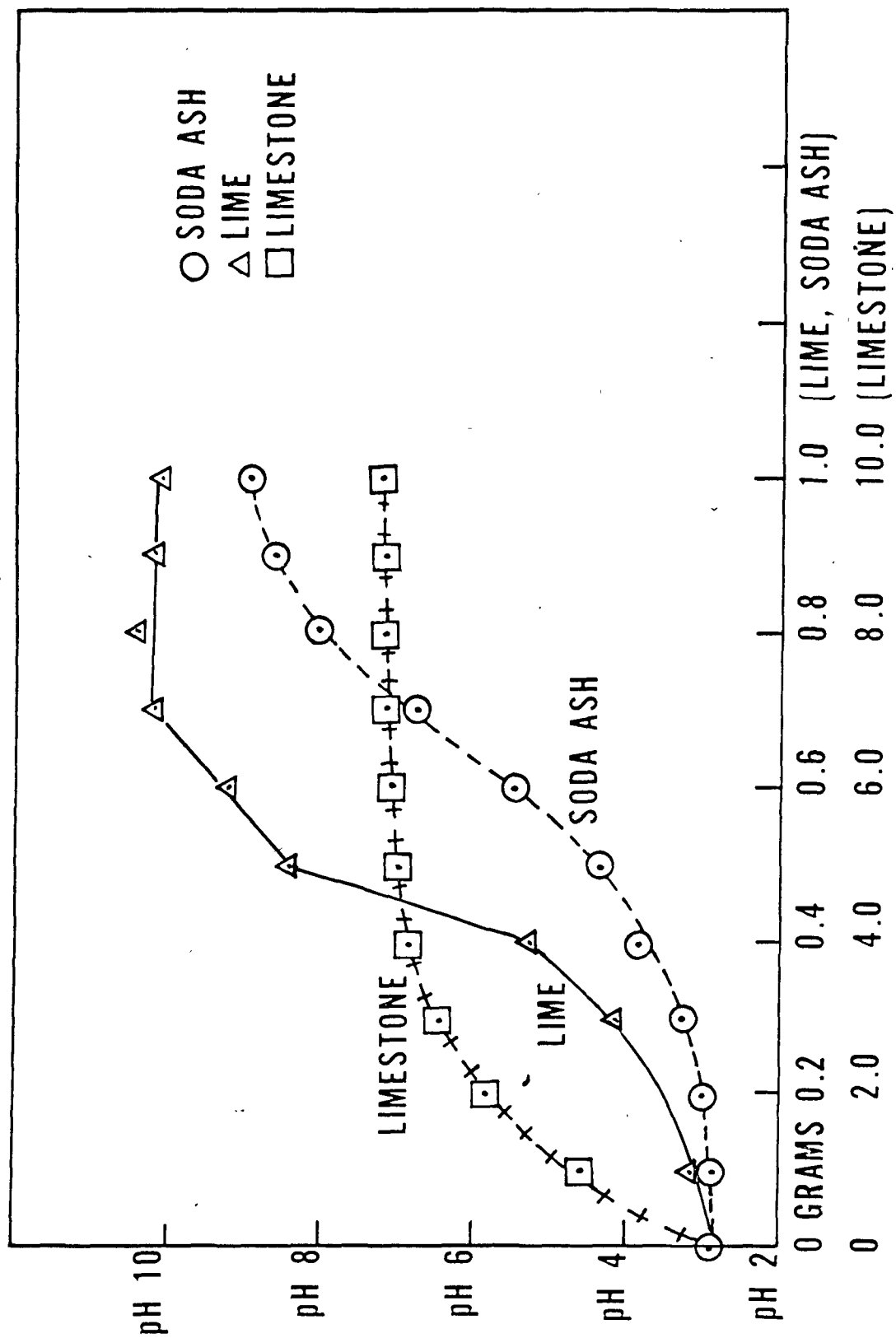
Relative Reactivity Study

The relative reactivities of lime, limestone, and soda ash were compared by the addition of measured amounts of each neutralizing agent to identical separate samples of Grassy Run water. Graphs of the results of these tests are shown in Figure 3. These tests were run allowing five minutes reaction time between incremental addition of the neutralizing agents.

In the lime reaction, the steep slope between pH 4.0 and pH 8.0 is indicative of the quantitative sensitivity of the lime reaction in this portion of the curve. It is obvious that a very small change in the amount of lime added at pH 6.0 can easily shift the pH one to two full points. Thus, in the lime reaction, accuracy of feed is of vital importance.

The slope of the limestone curve decreases rapidly at pH's above 6.0 as the reaction liberates CO₂ which, in turn, buffers the reaction. Consequently, the curve is nearly flat above pH 7.0 and increases in pH above this point are very difficult to attain. However, the shallow slope of the curve in the pH 5.0 - 7.0 range would make the limestone reaction an easy one to control in an operating system as the reaction would be insensitive to small changes in the neutralizing agent feed rate.

The soda ash reaction is quite similar to the lime reaction in the shape of the curve. However, this sodium carbonate reaction, as in the case of limestone, suffers from buffering due to the formation of carbonic acid (H₂CO₃). It is seen that the formation of H₂CO₃ retards the progress of the reaction in both limestone and soda ash neutralization. With time, the H₂CO₃ breaks down to yield carbon dioxide which escapes as a gas, thus the buffering capacity decreases and the pH increases.



GRAMS ADDED TO ONE LITER OF GRASSY RUN WATER
 (INITIAL HOT ACIDITY = 619 MG/I)

FIGURE 3

COMPARISON OF LIME, LIMESTONE, AND SODA ASH REACTIVITIES

Keeping in mind that the scale for limestone is different from that of lime and soda ash in Figure 3, it is clear that lime is the most reactive agent. It requires roughly 7.4 times as much limestone as lime in this case to raise the pH to approximately pH 6.5. One and one-half times as much soda ash as lime is required to reach pH 6.5. It should be remembered that these tests had only five minutes reaction time and any increase in detention time would narrow the gap between lime and the other two agents.

Another comparison that can be made is the amount of neutralizing agent used at the maximum utilization efficiencies developed in the preceding section. As seen in Table 7, four times as much limestone would be required and five times as much soda ash as lime.

Table 7
Maximum Utilization Efficiencies

Neutralizing Agent	pH at Which Maximum Utilization Efficiency Occurs*	Grams Material Used**
Lime	4.6	0.10
Limestone	3.9	0.40
Soda Ash	4.7	0.54

* Data from Table 5

**Data from Figure 3

Optimization

Before optimizing the system, it was necessary to set a goal for the effluent water quality and then to determine the operating conditions which best achieved this goal. The initial intent was to produce the best effluent possible. Considerations of cost paled this view somewhat to the more realistic criteria of meeting and/or exceeding the water quality standards of the mining states which, in themselves, proved quite restricting. Table 8 presents the published criteria relating to mining in West Virginia and Pennsylvania.

The previous studies had shown that the stream standards of West Virginia could be met with the exception of sulfate and that the more stringent effluent standards of Pennsylvania could be satisfied. It also was evident that the iron, aluminum, and net alkalinity requirements could be met at a pH less than 6.0. Therefore, in order to meet the pH 6.0 requirement, the system must be operated at less than maximum utilization efficiency. Referring to Figure 3, it can be seen that the amount of neutralizing agent required for an increase in pH from the optimum values given in Table 7 is more for limestone and soda ash than for lime. Therefore, the limestone and soda ash neutralization efficiencies decrease at a higher rate than lime as the pH increases.

Thus our choice of operating conditions were:

- pH 6.0 to pH 6.5
- Iron removal below 7.0 ppm
- Net Alkalinity (Alkalinity exceeds acidity)

Table 8
Water Quality Standards

State of West Virginia⁽⁶⁾

pH	5.5 Minimum, 8.5 Maximum
Iron	10 ppm
Aluminum	30 ppm
Sulfates	200 ppm

These are stream standards. There are no effluent standards in West Virginia.

Commonwealth of Pennsylvania⁽⁷⁾

pH	6.0 Minimum, 9.0 Maximum
Net Alkalinity	
Iron	7.0 ppm Maximum

These are effluent standards.

Pilot Plant Studies

Actual pilot plant operation supplied the following data. This data has been evaluated and mathematically reduced to eliminate, as much as possible, the effects of variations due to the day-to-day fluctuation of water quality in Grassy Run.

Limestone

Because of the delay in the limestone treated water reaching a final pH, it was necessary for control purposes to establish a lower pH in the reactor in order to obtain the desired resulting effluent pH. For example, for the test reported in Table 9, a pH of 5.8 was maintained in the reactor which resulted in a pH of 6.6 in the plant effluent. The pH maintained in the reactor was called the instantaneous in-reactor pH. This phenomenon causes some difficulties in operating a limestone treatment system.

Tables 9 and 10 show the results of treatment of two different concentrations of Grassy Run water with "rock dust" limestone. In these instances, the limestone was introduced in the slurry form.

The cost of limestone is 1.7 times as great to treat the water in Table 10 as in Table 9 and the water's acidity is 1.7 times as concentrated. Similarly, there is no significant difference in utilization efficiency between the two.

Lime

Table 11 shows data acquired from neutralization with lime. Utilization efficiency of lime approaches 100 per cent and is easily maintained, even at high pH's. Unlike limestone, the pH was slightly less in the plant effluent than in the reactor.

Supernatant water quality from lime is relatively good. However, as in the case of limestone, the water still remains typically high in hardness, calcium, and sulfates. Removal of iron with lime was better than that recorded for limestone as supernatant iron concentrations were consistently below 3.0 ppm. Better iron removal probably is partially attributable to the higher pH's attained using lime.

Table 9

Limestone Slurry Neutralization of Grassy Run Water*

Instantaneous In-Reactor pH = 5.8**

Raw Water Feed Rate = 6402 ml/min (1.69 gpm)

Limestone Slurry Feed Rate = 182 ml/min slurry (9.1 grams/min limestone)

Reactor Detention Time = 33 minutes (theoretical)

Settling Tank Detention Time = 12 hours (theoretical)

MEAN DATA***

Parameter	Units	Feed Water	Reactor Effluent	Settling Tank Effluent
pH (after 24 hrs.)		2.9	6.6	6.6
(after 48 hrs.)			6.8	6.8
(after 72 hrs.)			7.1	7.0
Hot Acidity (as CaCO ₃)	mg/l	362	0	0
Cold Acidity (as CaCO ₃)	"	373	33	25
Iron	"	195	7.3	7.0
Sulfates	"	516	479	479
Hardness (as CaCO ₃)	"	626	1647	1547
Conductivity	Mmhos/cm	927	841	857
Magnesium (as CaCO ₃)	mg/l	171	175	170
Alkalinity	"	0	78	66
Calcium (as CaCO ₃)	"	455	1472	1377
Aluminum	"	33	1.6	0.9
Temperature of Water		10°C	10°C	9°C
Acid Removed (Hot)	"		362	362
Acid Removed (Cold)	"		340	348
Limestone Utilization Efficiency			32.1%	31.2%
Lbs/1000 gal Limestone = 11.8 lbs/1000 gal Grassy Run Water				
Cost per 1000 gallons = \$0.0354/1000 gallons				
Cost per mg/l Acid/1000 gallons = 0.010 cents				

Per Cent Sludge by Volume (1 hour settling time) = 3.5%

Per Cent Sludge by Volume (24 hours settling time) = 3.7%

*All analyses made 24 hours after sampling, except as noted for pH

**pH of water in the reactor during the test run

***Summary of many sets of data collected during the test run

Table 10

Limestone Slurry Neutralization of Grassy Run Water*

Instantaneous In-Reactor pH = 5.7**

Raw Water Feed Rate = 4800 ml/min (1.27 gpm)

Limestone Slurry Feed Rate = 228 ml/min slurry (11.4 grams/min limestone)

Reactor Detention Time = 43 minutes (theoretical)

Settling Tank Detention Time = 16 hours (theoretical)

MEAN DATA***

Parameter	Units	Feed Water	Reactor Effluent	Settling Tank Effluent
pH		2.7	6.7	6.6
Hot Acidity (as CaCO_3)	mg/l	602	0	0
Cold Acidity (as CaCO_3)	"	595	77	79
Iron	"	145	5	5
Sulfates	"	904	663	723
Hardness (as CaCO_3)	"	588	1621	1588
Conductivity	Mmhos/cm	1250	1100	1150
Magnesium (as CaCO_3)	mg/l	188	196	188
Alkalinity	"	0	122	90
Calcium (as CaCO_3)	"	400	1425	1400
Aluminum	"	44	3.8	3.9
Temperature of Water		5°C	7°C	7°C
Acid Removed (Hot)	"		602	602
Acid Removed (Cold)	"		518	516
Limestone Utilization Efficiency			31.5%	30.1%

Lbs/1000 gal. limestone = 19.7 lbs/1000 gal. of Grassy Run Water

Cost per 1000 gallons = \$0.0591/1000 gallons

Cost per mg/l Acid per 1000 gallons = 0.010 cents

Per Cent Sludge by Volume (1 hour settling time) = 5.0%

Per Cent Sludge by Volume (24 hours settling time) = 4.5%

*All Analyses made 24 hours after sampling

**pH of water in the reactor during the test run

***Summary of many sets of data collected during the test run

Table 11

Lime Neutralization of Grassy Run Water*

Instantaneous In-Reactor pH = 7.9**

Raw Water Feed Rate = 5964 mg/l (1.58 gpm)

Lime Feed Rate = 2.69 grams/min - dry feed

Reactor Detention Time = 35 minutes (theoretical)

Settling Tank Detention Time = 13 hours (theoretical)

MEAN DATA***

Parameter	Units	Feed Water	Reactor Effluent	Settling Tank Effluent
pH		2.8	7.1	6.9
Hot Acidity (as CaCO ₃)	mg/l	601	2.1	1.7
Cold Acidity (as CaCO ₃)	"	612	0.4	2.6
Iron	"	157	2.4	1.6
Sulfates	"	717	709	693
Hardness (as CaCO ₃)	"	582	1261	1220
Conductivity	Mmhos/cm	1450	1241	1245
Magnesium (as CaCO ₃)	mg/l	135	162	145
Alkalinity	"	0	17	11
Calcium (as CaCO ₃)	"	447	1099	1075
Aluminum	"	33	0.8	0.9
Temperature of Water		13°C	15°C	15°C
Acid Removed (Hot)	"		599	599
Acid Removed (Cold)	"		612	609
Lime Utilization Efficiency			103%	102%
Lbs/1000 gal lime = 3.75 lbs/1000 gal of Grassy Run Water				
Cost per 1000 gallons = \$0.032/1000 gallons of Grassy Run Water				
Cost per mg/l Acid/1000 gallons = 0.0052 cents				

Per Cent Sludge by Volume = 6.9% (24 hours after settling time)

Per Cent Solids in Sludge = 1.7%

*Chemical Analyses Made 24 Hours After Sampling

**ph of Water in the Reactor during the test run

***Summary of many sets of data taken during a test run

Table 12

Soda Ash Neutralization of Grassy Run Water*

Instantaneous In-Reactor pH = 6.0**
 Raw Water Feed Rate = 11,000 ml/min or 2.91 gpm
 Soda Ash Feed Rate = 7.1 grams/min
 Reactor Detention Time = 19 minutes (theoretical)
 Settling Tank Detention Time = 7.2 hours (theoretical)

MEAN DATA***

Parameter	Units	Feed Water	Reactor Effluent	Settling Tank Effluent
pH		2.7	6.1	6.5
Hot Acidity (as CaCO ₃)	mg/l	551	0	0
Cold Acidity (as CaCO ₃)	"	586	73	0
Iron	"	126	7.9	2.3
Sulfates	"	581	672	768
Hardness (as CaCO ₃)	"	526	530	541
Conductivity	Mmhos/cm	1300	1425	1500
Magnesium (as CaCO ₃)	mg/l	205	205	208
Alkalinity	"	0	100	103
Calcium (as CaCO ₃)	"	321	325	333
Aluminum	"	41	3.5	2.2
Sodium	"	2.8	555	450
Temperature of water		12°C	12°C	12°C
Acid Removed (Hot)	"		551	551
Acid Removed (Cold)	"		513	586
Soda Ash Utilization Efficiency			106.8%	107.3%
Lbs/1000 Gallons Soda Ash = 5.367 lbs/1000 gallons of Grassy Run Water				
Cost per 1000 gallons = \$0.268/1000 gallons of Grassy Run Water				
<u>Cost per mg/l Acid/1000 gallons = 0.0486 cents</u>				

Per Cent Sludge by Volume = 7.6%

Per Cent Solids = 1.5%

*Chemical Analyses Made 24 Hours After Sampling

**ph of Water in the Reactor during the Test Run

***Summary of Many Sets of Data taken During a Test Run

However, the very low per cent solids of the lime sludge (1.7 per cent) will be a major problem when considering sludge disposal.

Soda Ash

Table 12 contains the results of the pilot plant test using sodium carbonate as the neutralizer.

It is seen that soda ash has only one major advantage over lime or limestone in the low hardness concentration. Utilization efficiency, as in the case of lime, approaches 100 per cent. However, the one major disadvantage of soda ash treatment is the cost. Soda ash costs over nine times as much as lime per mg/l acid per 1000 gallons treated. It costs almost four times as much as limestone.

Even though the hardness is low using soda ash, the sodium concentration in Table 12 was increased from 2.8 mg/l to 450 mg/l.

A comparison of the relative settling rates between lime, limestone, and soda ash sludges is presented in Figure 4. Sludge from the limestone reaction compacts to the smallest volume and all settling is completed in approximately one hour. Lime sludge has a very rapid initial settling rate (due to the rapid lime reaction, high pH's are quickly attained), but suffers from poor compaction. It requires almost five times as long for the lime to reach a state of final compaction as for the limestone. In the final analysis, however, better than 90 per cent of the total settling is completed in all cases in less than one hour. The soda ash settling characteristics are virtually identical to those of the lime.

A test was made to compare the per cent sludge solids of lime, limestone, and soda ash sludges. Using the same water, samples were neutralized to pH 6.5 (lime and soda ash) and pH 6.2 (limestone, allowed to settle 24 hours, and then the sludge was withdrawn and per cent solids determined. Table 13 presents the results of these tests. Obviously, limestone sludge is far superior in per cent solids content.

Table 13
Per Cent Solids of Sludges*

<u>Neutralizer</u>	<u>Supernatant pH</u>	<u>Per Cent Solids of Sludge</u>
Lime	pH 6.5	1.5%
Limestone	pH 6.2	9.5%
Soda Ash	pH 6.5	0.7%

*After 24 hours of undisturbed settling

Lime and limestone settling tests were also run on a Grassy Run water sample which had been concentrated 3.3 times by a reverse osmosis unit. The results of this test on the concentrated water are presented in Figure 5. Comparing the results from the water as opposed to the unconcentrated Grassy Run water, we find that the general settling trend of lime does not appear to vary with increased concentrations. In the case of limestone, however, marked improvements in settling characteristics are apparent in the concentrated water tests. In both of these instances, as in the case of the unconcentrated water, better than 90 percent of the settling is completed in one hour. The sludge volumes were greater for the concentrated water than for the mine drainage due to the larger amounts of iron, calcium, acid, and etc. that were available for precipitation.

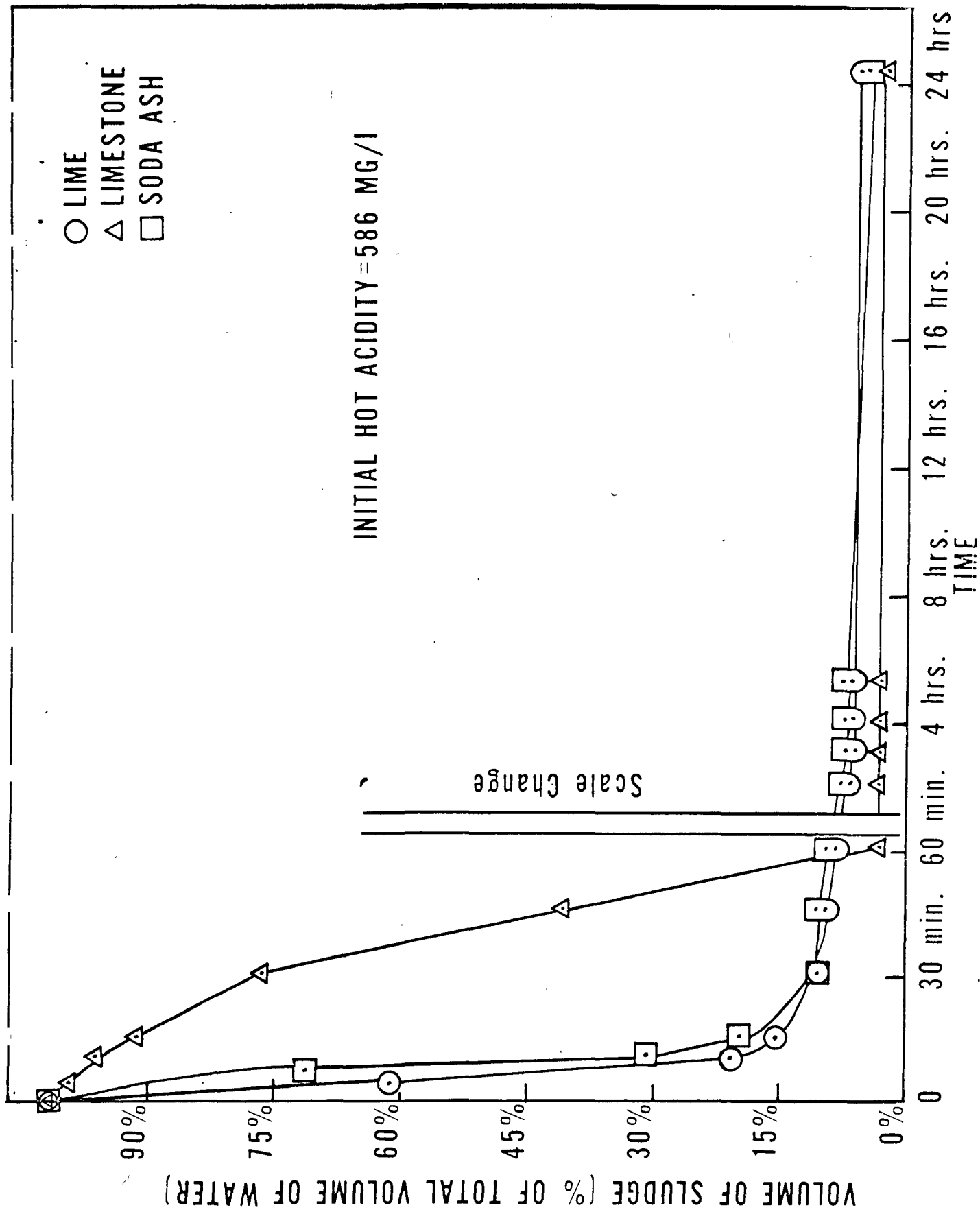


FIGURE 4 COMPARISON OF SETTLING RATE

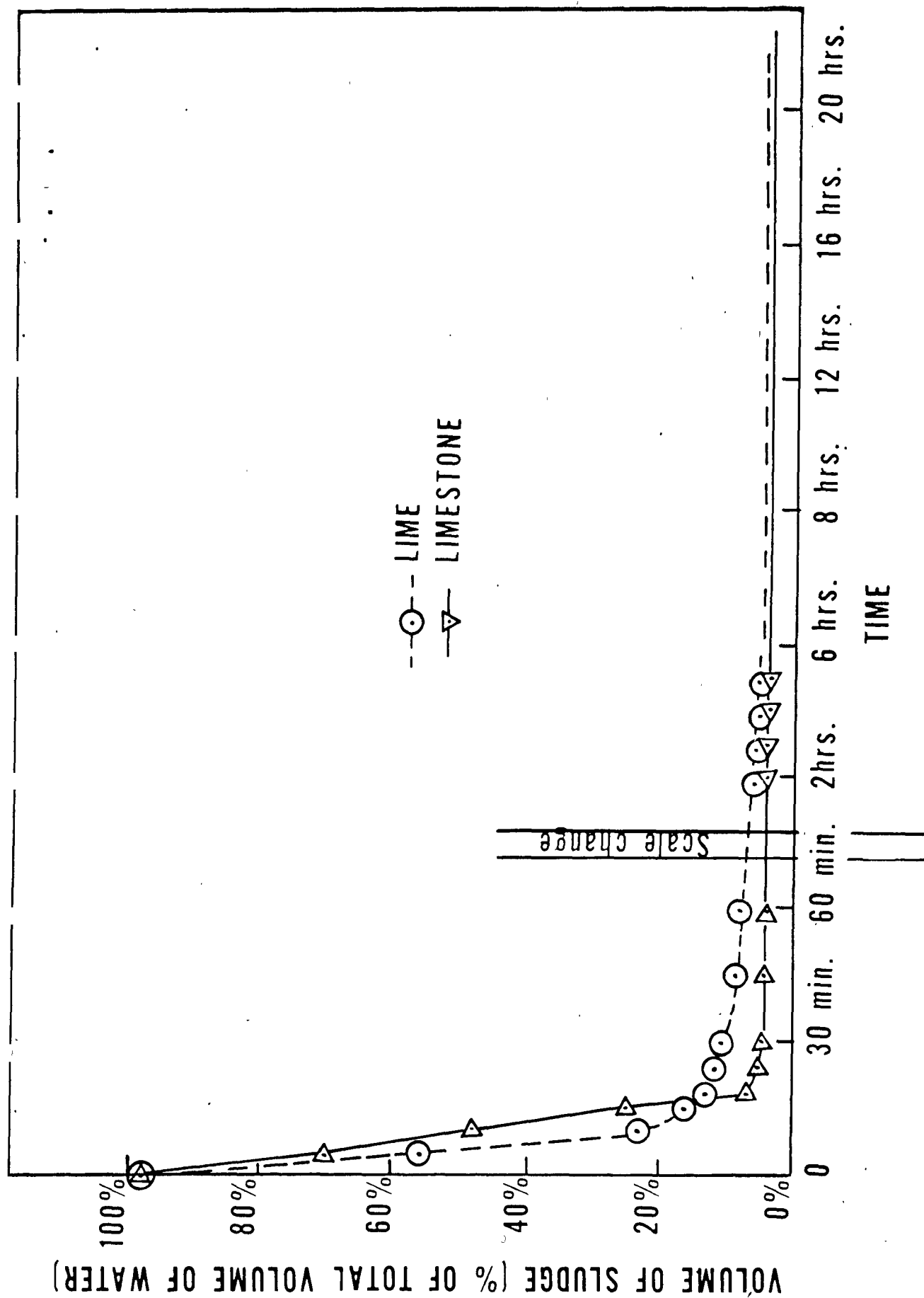


FIGURE 5
COMPARISON OF SETTLING RATE TRENDS
BETWEEN LIME AND LIMESTONE NEUTRALIZATION

Table 14 indicates the per cent reductions or per cent increases in the supernatant quality of neutralized water. Direct comparisons are made between lime, limestone, and soda ash supernatants.

Efficiency Improvement Studies for Limestone

Aeration

The studies up to this point had shown that limestone has a raw material cost advantage over lime (\$6.00 vs. \$17.00) and has a more dense, less voluminous sludge. On the other hand, the limestone utilization efficiency was only 32 per cent while lime was 100 per cent. On this basis, it would appear that it would require three times as much limestone as lime to treat the same water and, therefore, the raw material cost would be 1.1 times higher for limestone. However, as shown in Tables 10 and 11, the cost for limestone was 0.010 cents per 1000 gallons/mg/l acid treated as compared to 0.0052 cents per 1000 gallons/mg/l acid treated for lime or 1.9 times more. There appears to be a conflict in these results when, in fact, there is not. The discrepancy is one of definition.

Assuming supernatant alkalinity to be beneficial to the environment, utilization efficiency was defined to include it. Very little increase in supernatant alkalinity is seen with lime neutralization as opposed to the high alkalinity of limestone treated product water. From a cost of treatment standpoint, this alkalinity could be considered unnecessary and, thus, detrimental.

If the alkalinity term of the utilization efficiency equation were to be excluded and the efficiencies recalculated, then the lime efficiency (Table 11) would be 98.5 per cent and the limestone (Table 9) would be 26.3 per cent. It would appear to be necessary to use 98.5 per cent/26.3 per cent = 1.3 times as much limestone as lime. Referring to the definition of utilization efficiency, it is seen that both lime and limestone efficiencies are expressed as CaCO_3 . Thus to discuss cost, or actual usage, it is necessary to reconvert the lime efficiency from CaCO_3 back to CaO and thus it is multiplied by 1.35 (ratio of molecular weights CaCO_3/CaO). The resulting value for lime is 133.0 per cent as compared to 26.3 per cent for limestone or a ratio of 5.1 times as much limestone is needed as lime. Therefore, on a cost basis, limestone costs 1.8 times as much as lime and this corresponds with the 1.9 times figure derived from the cost per mg/l acid 1000 gallons columns on Tables 10 and 11.

In order to have limestone compete with lime, it is necessary to improve the efficiency of the limestone process. Several methods were initially conceived: 1. Increase the detention time of the water in the reactor, thus providing time for more limestone to become solubilized, 2. Increasing the vigorous mixing action in the reactor to create more complete breakdown of the limestone, as earlier studies indicated the more rapid the mixing, the better the results. 3. Return the sludge with its unused limestone back to the reactor, and 4. Aerate the water to strip off the carbon dioxide and thereby reduce the buffering capacity.

The test equipment on hand was unsuitable to perform tests on items 2 and 3, so a new and larger test facility has been constructed to study these factors. Item number 1 could not be studied directly with the present equipment. However, studies in comparing dry feed and slurry feed gave some indirect insight. Aeration was studied as part of the current study.

First attempts at aeration were performed in the upflow settling tank. The data is presented in Table 15. The effluent pH rose immediately from pH 6.7 to pH 7.1 as soon as aeration began. However, cold acidity barely dropped (from 29 mg/l to 25 mg/l) and the overall rise in efficiency was 0.9 per cent. This insignificant change certainly does not justify the cost of aeration equipment and the involved maintenance.

Table 14

Changes in Water Quality Due to Neutralization

<u>Parameter</u>	<u>Lime</u> (From Table 11)	<u>Limestone</u> (Avg. Tables 9 & 10)	<u>Soda Ash</u> (From Table 12)
pH	Increased to pH 6.9	Increased to pH 6.6	Increased to pH 6.5
Acidity (Hot)	Reduced	Reduced	Reduced
Acidity (Cold)	Reduced	Reduced	Reduced
Iron	Reduced	Reduced	Reduced
Sulfates	Reduced	Reduced	Reduced
Hardness	Increased	Increased	Increased
Conductivity	Reduced	Reduced	Increased
Alkalinity	Increased to 11 mg/l	Increased to 78 mg/l	Increased to 103 mg/l
Calcium	Increased	Increased	Increased
Aluminum	Reduced	Reduced	Reduced
Magnesium	Increased	Increased	Increased
Sodium			Increased to 450 mg/l

Table 15
Grassy Run - Slurry Feed Limestone
Comparison of Data Prior to and After
Aeration of Settling Tank

Instantaneous In-Reactor pH = 6.5*

Parameter	Units	Feed Water	Before Aeration Settling Tank	After Aeration Settling Tank
pH		2.9	6.7	7.1
Hot Acid (as CaCO ₃)	mg/l	375	0	0
Cold Acid (as CaCO ₃)	"	379	29	25
Iron	"	185	2.5	3.1
SO ₄	"	542	506	544
Hardness (as CaCO ₃)	"	564	1375	1263
Conductivity	Mmhos/cm	1100	1100	1100
Alkalinity	mg/l	0	72	87
Calcium (as CaCO ₃)	"	394	1219	1103
Aluminum	"	30	0.7	0.5
Flow Rate (Mean)		1.48 gpm 5600 ml/min	208 ml/min limestone slurry 10.4 grams/min limestone	
Acid Removed (Hot)	mg/l		375	375
Acid Removed (Cold)	"		350	354
Limestone Utilization Efficiency			24.0%	24.9%

Chemistry analyses made 24 hours after samples were taken

Mean Data from settling tank represents five samples taken within 4 hours after aeration was started

*pH of Water in the Reactor during the Test Run.

Table 16
Grassy Run - Slurry Feed Limestone
Comparison of Data Prior to and After
Aeration of Reactor

Instantaneous In-Reactor pH = 6.5 *

Parameter	Units	BEFORE AERATION			AFTER AERATION		
		Feed Water	Imme- diate (1 Hr.)	24 Hrs. Later	Feed Water (Mean)	Imme- diate (1 Hr.)	24 Hrs. Later
pH		2.8	6.3	7.0	2.8	7.0	7.3
Hot Acid (as CaCO ₃)	mg/l	464	0	0	454	0	0
Cold Acid	"	470	41	16	472	30	9
Iron	"	136	23	7.9	139	20	4.3
SO ₄	"	591	566	530	486	466	542
Hardness (as CaCO ₃)	"	439	1184	1168	452	1263	1360
Conductivity	Mmhos/cm	1200	1000	1000	1167	973	967
Alkalinity	mg/l	0	56	62	0	41	45
Calcium (as CaCO ₃)	"	300	1044	1031	310	1113	1200
Aluminum	"	30.8	5	2.2	34	5.0	1.6
Flow Rate (Mean)	6867 ml/min 1.81 gpm	205 ml/min slurry limestone 10.25 grams/min					
Acid Removed (Hot)	mg/l		464	464		454	454
Acid Removed (Cold)	"		429	454		442	463
Limestone Utilization Efficiency			34.8%	35.2%		33.2%	33.4%
Temperature of Water		18°C	18°C	18°C	18°C	18°C	18°C

*pH of Water in the Reactor during the Test Run

Aeration in the reactor was then studied (Table 16). A more pronounced pH change occurred after aeration (from pH 6.3 to pH 7.0), and the cold acidity was reduced 25 per cent. However, product alkalinity was reduced 34 per cent, thus resulting in a 1.6 per cent net loss in efficiency. There were no other significant changes in overall water quality.

In summary, aeration of the limestone reaction proved to be of no value in increasing efficiency. However, as it rapidly strips off CO₂, it could be very useful in drastically reducing detention time before supernatant discharge (discharge occurs when desired pH is attained) and would be especially applicable at sites where installation or presence of large holding lagoons (for the purpose of detention time) would not be feasible.

Dry Feed vs. Slurry Feed

Studies were made to determine if the feeding method--either wet or dry--would affect the reactions of any of the neutralizing agents.

Equal amounts of each neutralizing agent were added to identical samples of acid mine water. The only difference was that one was slurried with distilled water while the other was dry fed. The samples were then agitated by a flash mixer for five minutes each and then allowed to settle. The analyses were made on all samples at identical times after mixing.

The results of these tests are presented in Table 17 and indicate there is no significant difference in water quality or efficiency (as indicated by alkalinity--the only variable in the efficiency equation) between dry feeding or slurry feeding.

These results also indicate that increasing detention time to enhance limestone efficiencies by increasing solubilization of limestone (as mentioned earlier) does not appear promising. The increase in detention time could well increase the efficiencies due to increased acid-base contact time or other factors but the solubility of limestone in water does not appear to have much bearing on the reactions involved.

Table 17
Comparison of Dry Feeding and Slurry Feeding

Parameter	Raw Water	Lime		Limestone		Soda Ash	
		Dry	Slurry	Dry	Slurry	Dry	Slurry
pH	2.7	6.1	6.2	6.6	6.8	6.8	6.8
Conductivity	1650	1460	1460	1460	1460	1750	1750
Hot Acidity (as CaCO ₃)	532	0	0	0	0	0	0
Cold Acidity "	558	69	64	90	12	65	24
Calcium (as CaCO ₃)	318	812	812	862	844	318	300
Magnesium "	212	212	212	206	206	214	201
Hardness "	530	1024	1024	1068	1050	532	501
Sulfates	868	747	723	723	688	723	699
Iron	120	2.3	2.0	2.1	2.2	4.9	6.3
Aluminum	35	1.1	1.2	1.3	1.2	2.7	2.4
Alkalinity	0	17	18	50	50	97	91
Sodium	2	2.3	2.2	2.0	2.0	380	320

All units are expressed in mg/l except conductivity (Mmhos/cm) and pH

SUMMARY

a) All three neutralizing agents--lime, limestone, and soda ash--can meet the pH 6.5, net alkalinity, iron below 7.0 ppm criteria with relative ease.

b) The major differences between soda ash neutralization and lime or limestone neutralization are:

Soda ash produces:

1. Low hardness
2. Low calicum
3. High sodium
4. High cost

All other chemical parameters are comparable between the three neutralizing agents.

c) Soda ash costs nine times as much as lime and four times as much as limestone to treat the same water under the test conditions of this study.

As soda ash is obviously too expensive for other than specialized cases, the remaining part of the summary will be confined to comparisons between use of lime and limestone. The following observations are made, based upon the results obtained during these studies:

d) Lime costs approximately half as much as limestone to treat the same water under the test conditions of this study. This refers only to the cost of the chemical itself.

e) Limestone sludge is more desirable in that it occupies only approximately two thirds of the volume of lime sludge and has a higher solids content. It contains a large residual alkalinity which would be beneficial when disposed into an acid mine environment (although this residual alkalinity is expensive and of questionable value to the treatment plant).

f) Lime is a very reactive material and the neutralization reaction goes to completion in less than one hour. The limestone reaction requires from 24 to 48 hours to go to completion and therefore requires a long detention time before discharge. This detention time can be expensive; however aeration will reduce the detention time to one comparable to lime.

g) The limestone reaction is not very sensitive quantitatively so the accuracy with which constituents are fed into the reactor need not be controlled with the precision required by lime. The cost of the controlling system involved could be somewhat reduced for this corresponding reduction in necessary accuracy. Accidental overtreatment is not the pollution problem with limestone that it would be with lime.

h) Lime is capable of attaining higher pH's which may be necessary in some individual cases for the desired water quality characteristics, whereas limestone efficiencies drop off rapidly at high pH's and correspondingly, pH's above 7.0 are very difficult to attain. In order for limestone to be competitive with lime, methods of improving the utilization efficiency must be found. Sludge return appears to be a promising method.

The following observations are presented relative to the plant design and operation:

i) We found no difference in the efficiency between dry feed methods of introducing the neutralizing agent or slurry feed methods. This applies to lime, limestone, and soda ash.

j) Dry feeding offers fewer operational difficulties than does a slurry feeding and costs less, both initially and in cost of maintenance.

k) PVC (PolyVinylChloride) plastic pipe should be used for all piping, and pumps should be of the open face impeller type with all wetted parts made of 316 stainless steel. This will resist not only acidity but also the extreme abrasiveness of the liquids involved. Initial cost will be higher for this type of equipment but maintenance costs are conservatively estimated to be on the order of 1/16 or less as compared to cast iron or bronze. Selection of low RPM pumps (1750-1800 rpm) will further reduce pump wear.

l) In all cases, almost all the sludge settling is accomplished in one hour.

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