

OVERBURDEN ANALYSES; SOME IMPORTANT FACTORS

by

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I. BACKGROUND

The prediction of acid mine drainage (AMD) depends, in part, upon an accurate assessment of the chemical weathering attributes of the overburden that will be disturbed by the mining process. These assessments are generally performed through overburden analyses and can be grouped into two categories, static or dynamic techniques. In the static tests, whole rock analyses are used to predict mine drainage quality; with the assumption that specific minerals (i.e., pyrite, carbonate, etc.) comprising the strata will react directly to produce varying degrees of alkalinity and acidity (the amounts of which are numerically balanced to determine the anticipated chemistry of the drainage). Alternatively, dynamic tests empirically determine leachate quality by subjecting the overburden samples to a variety of simulated weathering tests while monitoring the quality of the effluent thus produced. Within these two categories a variety of mine waste/overburden analytical techniques is available, each having advantages and disadvantages relative to one another.

Many questions exist with regard to the accuracy of the testing procedures and their usefulness in mine drainage quality predictions. Recognized are the host of natural environmental conditions pertaining to climate, methods of mining and reclamation, handling of toxic material (that may affect pyrite oxidation) and the manner by which the weathering products are mobilized from the site. The latter concerns are best addressed by a field oriented study whereby the backfill is instrumented to assess the hydrologic and geochemical parameters affecting the drainage chemistry. Even at this level, refinements in overburden analytical techniques are still required before the laboratory derived results can be accurately translated to field conditions and/or physical models.

As a part of this paper, the independent variables affecting leachate quality (i.e. particle size, temperature, bacteria, etc.) were isolate in four of the most commonly used dynamic overburden analytical techniques. These variables were related to the physical mechanisms introduced by the techniques to identify the effect these variables had on the quality of the leachate produced. Through this strategy the strengths and shortcomings of the static tests

and four of the dynamic tests can be evaluated and used to formulate and/or improve the accuracy of the analytical method.



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II. OVERBURDEN ANALYSES

The overburden analytical methods most commonly used in the coal sector include the static tests whole rock analysis (i.e., acid/base accounting, modified acid/base accounting, and cold acid digestion). For facility of discussion these tests, unless otherwise described, are collectively referenced as whole rock analysis. The dynamic tests or simulated weathering tests include 1) humidity cells, 2) large columns, 3) small columns and 4) soxhlet reactors. Of these tests, the column tests (large and small) are used occasionally, the majority of the dynamic tests being distributed among the other two. As will be shown below, particle size plays an important role in the quality of leachate produced from column tests and should be in a separate group.

The overburden analytical techniques that are most commonly used in the coal mining sector, as well as the advantages and variables associated with each test are summarized below.

Static Tests

1. Acid/Base Accounting and Modifications

This most widely used technique digests the sample, which is pulverized to pass 60 mesh, in a hot acid solution of known normality. The amount of acid consumed is determined by titrating the acid digested solution to pH 7 using sodium hydroxide solution of the same normality. The amount of acid consumed is related to the rock's neutralization potential (NP and is usually expressed as tons of CaCO_3 /1000 tons). Generally, the NP is understood to be comprised of calcium-magnesium carbonate (collectively referred to as calcareous material). A split of the pulverized sample is also analyzed for pyritic sulfur and the sulfur percentage is related to acid potential by multiplying this value by 31.25 (to yield tons of acid produced/1000 tons). The NP is numerically balanced by the acid potential to predict the acid/alkaline production potential (Sobek et. al., 1978).

Advantages: Easy to perform, standard technique, relatively rapid analysis, inexpensive and has been successful in predicting situations that are decidedly acid or alkaline prone.

Disadvantages: Assumes linear kinetics of acid and alkaline production, not accurate in predicting drainage quality in "gray" areas and, most important, the hot acid solution digests siderite (iron carbonate) and clays that report high NP values, but provide minor alkalinity. For this reason, several researchers have modified the acid/base accounting method to circumvent the "hot" acid problems. (Geidel, 1980) proposed sample digestion using cold acid

in the formulation of the Alkaline Production Potential (APP) technique. To address the siderite problem, Meek (1981), Brady et. al., (1990) and Shelton et. al. (1990), suggest modifying the acid/base accounting method by the addition of hydrogen peroxide in the final titration to oxidize all available free iron to complete the hydrolysis and reduce the NP accordingly. In addition, Brady et. al. (1990) propose that the acid potential (S%) be multiplied by 62.5 to improve the assessment.

Independent Variables: 1) temperature of acid digestion solution and 2) presence of pyrite, calcareous material and siderite.

Dynamic Tests

1. Humidity Cell Weathering Tests

Samples are crushed to pass 4 mm, weighed and placed onto a filter paper in separate chambers constructed of a plastic cylindrical pan, 7 cm deep and 15.5 cm in diameter. A plastic lid prevents contamination and minimizes evaporation. Humidified air is circulated into the chamber to maintain saturated conditions. The chamber is opened only to add water during the leaching cycle. Samples are leached periodically by pouring an aliquot of deionized water onto the sample and collecting the leachate produced. The leachate is analyzed and the various parameters are plotted versus time to derive quality production trends (Caruccio, 1968, 1969, and Sobek, 1978).

Advantages: Leachate quality is empirically determined, natural conditions are simulated and leachant can be modified, parameters versus time plots provide kinetic data that can also be used to determine loads per unit weight of sample, effect of siderite is assessed and the effects of bacteria can be evaluated. Best utilized in "gray" areas.

Disadvantages: Frequency of leaching affects the acid/alkaline production (Geidel, 1980), long leaching times are required (on the order of 8-12 weeks), comparatively expensive, quality production affected by particle size (Geidel et. al., 1983).

Independent Variables: 1) frequency of leaching, 2) presence of bacteria, 3) particle size, 4) humidity, and 5) presence of pyrite, calcareous material and siderite.

2. Column Tests (Large)

Samples of any size are weighed and loaded into plastic columns of varying diameters and heights. Periodically the column is rinsed with a leachant and the leachate collected is analyzed for quality parameters (Hood, 1984). Where columns are generally used, the diameter of the traditional columns used in these tests varies from 2 to 6 cm and average about 1 to 2 meters in length. The relatively large size of these columns can accommodate large volumes of overburden sample with large particle sizes. The physical configuration of the columns provides for adequate drainage and as long the sorting coefficient is reasonable, a small portion of the leachant is held in capillarity. The data base is handled similar to the weathering cells technique.

Advantages: Same as with Humidity Cell Weathering tests and, in addition, large particle sizes can be accommodated, more closely approximates field conditions (Caruccio and Geidel, 1986), and can be made to simulate various hydrologic elements (Leach, 1991).

Disadvantages: Large volume of sample required, logistical problems regarding sample preparation and loading, particle size affect, flow may channelize and, contingent upon samples' sorting coefficient, air locks production (Bradham and Caruccio, 1990).

develop and inhibit acid

Independent Variables: 1) particle size, 2) presence of bacteria, 3) sorting coefficient of samples, 4) air locking, and 5) presence of pyrite, calcareous material and siderite.

The ability of the leachate to drain from a column is a function of the degree of sorting of the sample, the rapidity with which field capacity is achieved and the size of the sample used in the test. Should the sample be well sorted and of a relatively large particle size, i.e., 2 to 4 mm, the leachate will drain easily, entraining the soluble weathering products and effectively draining the column of water. Alternatively, should the sample be poorly sorted and comprised primarily of small sized fractions, i.e., 0.5 mm, a substantial amount of the leachant will be retained by capillarity, the sample will remain saturated (due to lack of achieving field capacity) and in the process create air locks which inhibit the migration of oxygen into the inner part of the column and create anaerobic conditions. In this environment, pyrite oxidation is suppressed and alkalinity production is enhanced.

3. Column Tests (Small)

Alternatively, small column tests use 500 to 1000 ml plastic bottles whose bottoms are cut off, inverted and the neck used as a drainage outflow. The neck of the bottle is packed with glass wool and the sample is generally crushed to pass 1/2 mm in order to be adequately loaded into the bottle. Several studies, in attempting to use column tests to reflect anticipated drainage quality from reclaimed mines, have scaled the thicknesses of the various strata found in the highwall down to equivalent proportions of crushed sample whereby the stratigraphic order is mimicked and the entire highwall stratigraphy is loaded proportionally in the plastic bottle. In so doing, the samples are finely crushed and layered in stratigraphic order (as many as 8 to 10 layers might be represented in the small columns). The fine grained nature of the rock samples required for the packing of the bottles creates high capillary forces, causing air locks in which oxygen is excluded from the reactions. In so doing, the small columns generate, irrespective of the overburden chemistry, non-acid leachates. Obviously, because of the airlocking phenomena, abnormal conditions develop within the columns that are artifacts of the testing scheme and not due to the samples' chemical make-up.

The physical nature of this analytical technique and because of the small size of the container used, coupled with the small particle size of the sample, combine to enhance capillarity action. As a result, specific capacity is difficult to achieve and the net effect is the formation of tight air locks which preclude the permeation of oxygen to the system. Under these conditions, acid production is minimized, if not prevented, while alkalinity production is enhanced. To extrapolate these artifacts and/or conditions further onto a mimicked scaled-down stratigraphic sequence, contained in a 500 ml plastic bottle, is even more absurd and to extent leachate qualities produced by the small columns, as a means of predicting field condition response, violates the most basic geochemical principles upon which weathering tests are designed.

4. Soxhlet Reactions

Pulverized samples (to pass 125u) of known weight are placed in separate cellulose extraction thimbles and stored in a drying oven at 105' C. Periodically, the sample (in the thimble) is placed in a soxhlet reactor and an aliquot of deionized water is heated to 100' C and circulated through the sample. After 4 hours, the soxhlet reactor is cooled to room temperature. The thimble containing the sample is returned to the drying oven and stored at 105' C (Renton et.al., 1988). The effluent collected in the soxhlet is analyzed for a variety of chemistries.

Advantages: Rapid oxidation provides total acid production potential in short time frame, standard procedure with little operator error, complete leaching of oxidation products.

Disadvantages : High temperatures of storage and extractions not representative of natural conditions, pulverized sample provides large surface area of reactivity, hot leachant selectively removes calcareous material enhancing acid production while inhibiting alkalinity formation (Bradham and Caruccio, 1991).

Independent Variables: 1) Particle size, 2) sample storage temperature and conditions, 3) frequency of leaching, 4) temperature of leachant, 5) presence of pyrite, calcareous material and siderite.

III. COMPARATIVE STUDY

Experimental Design

The experimental matrix and study design were based on a statistical package which provided for analyses of variance, both among and between the independent variables, to identify those that are most important for, and common to, each analytical method used. In so doing, the critical elements of each analytical technique could be isolated and evaluated. Leachate quality as affected by particle size, temperature of leachant, temperature and conditions of sample storage, humidity, presence of bacteria, and sample composition were related to whole rock analyses, humidity cell weathering tests, column tests and soxhlet reactors.

All data collected were stored on electronic worksheets using WINDOWS. The data base was loaded onto a mainframe to derive statistical relationships between the independent variables, plots of parameters as required and derivation of equations expressing kinetic data. All data were normalized to accommodate variations in sample weight, pyritic sulfur, and leachate quality. Using a variation of ANOVA, each variable was tested against each other to determine the degree to which that variable impacts leachate quality. Upon completion of the study the independent variables with the greatest influence on leachate quality were identified (Bradham, 1992).

Results of the study

The objectives of the study were to determine the significant factors of each prediction technique, the relative importance of each factor, and the manner in which each factor affects leachate quality. The conclusions of the study are as follows:

1. The temperature of the leachant (water) has virtually no effect on either pyrite oxidation or calcium carbonate dissolution.
2. The important factors affecting pyrite oxidation and calcium carbonate dissolution are: lithology, particle size, and storage conditions.
3. The storage conditions that resulted in the lowest sulfate production rates were abiotic, room conditions and humidity chamber.
4. Weathering the samples in a drying oven increased the sulfate production rates from 15% to 344%, depending on lithology.
5. The presence of the bacteria *Thiobacillus ferrooxidans* had the effect of increasing sulfate production rates by approximately 200%.
6. Increasing the length of storage time in the drying oven had the effect of increasing sulfate production per leach, but decreasing sulfate production per day.
7. Pyrite oxidation reactions do appear to occur at high temperatures in the absence of water, but the nature of these reactions is unknown.
8. Calcium carbonate dissolution in high NP samples occurred, in some part, as a response to pyrite oxidation. Enhanced pyrite oxidation in the drying oven also resulted in a commensurate increase in calcium production.
9. In pyrite rich, high NP samples, that are stored in the drying oven, the weathering products of the pyrite oxidation and calcium carbonate dissolution may be stored until the next leaching period in the form of calcium sulfate minerals such as gypsum or anhydride (Bradham, 1992)

IV. ARTIFACTS OF TESTING PROCEDURE

The physical configuration of the testing method may impact the leachate quality more so than the chemistry of the sample. These artifacts in leachate production may be more directly related to the mechanics of the test rather than reflect the chemistry of the rock. Several artifacts of leachate quality have been related to testing procedures and are outlined below.

Weathering Cells

Ambient T within natural range (storage conditions) - between leaching intervals, the rocks contained in the weathering cells are stored in the laboratory complex. Within the rooms, samples in the weathering cells are stored at ambient conditions similar to those found under naturally weathering conditions.

Humidified air (natural conditions) - during the testing procedure, the samples are continuously purged with humidified air. It is reasonable to assume that the interior of a backfill in the humid east should be partially saturated and that the pore space of the reclaimed mine has some humidity. Accordingly, samples tested in the weathering cells, are done so under conditions approaching natural systems.

Specific yield - weathering fronts - under natural conditions, rain of low intensity and long duration would saturate the surface of the reclaimed mine, permeate into the backfill, and achieving field capacity to produce infiltrating wetting fronts. This pattern of exposure to periodic leaching episodes, which is mimicked by the flushing of the sample in the weathering cell and approximates natural field conditions.

Crushed particles - need field calibration - in order to provide results in a short time samples tested by weathering cells must, by necessity, be crushed to pass 4 mm. This size range minimizes capillary attraction, does not allow the particles to float-off, affords relatively rapid weathering and lends itself to leaching the weathering products from the samples. A large unknown remains regarding the amount of the backfill that is represented by this size fraction and underscores the need to scale up or scale down leaching test data as a function of the particle size distribution in the field.

Temperature of leachant - ambient - weathering cell tests are leached with deionized water, the temperature of which is typically in the range of about 20' C and which approximates field conditions.

Columns - Large

Ambient temperature within natural range (storage conditions) - Most closely approximates storage conditions in the vadose zone.

Particle size - if well sorted best approximates field conditions and may accommodate relatively large particle sizes. If poorly sorted capillary forces will tend to accumulate with the formation of airlocks.

Leachant temperature - natural.

Humidity - available.

Columns - Small

Ambient temperature within natural range (storage conditions) - most closely approximates conditions below the water table.

Particle size - must of necessity be fine-grained which leads toward airlocks and could approximate field conditions below the water table.

Leachant temperature - normal.

Humidity - available.

Soxhlets

High Temperature - abnormal conditions - The configuration of the soxhlet apparatus and its use in weathering tests creates a condition whereby the leachant temperature is close to boiling and precludes the ability to translate laboratory data to field conditions.

Between leaching intervals, samples contained in the cellulose thimbles are stored at 105' C. Measurements made on weight loss through time showed that the samples dry out within two days, rendering the samples inactive. Again, these are abnormal conditions compared to what we expect to see in the coal fields.

Particle size - pulverized and even less representative than the natural system.

Leachant temperature - 105° C and not representative of natural system.

Humidity - depleted after two days.

TABLE 1 - Matrix of Artifacts and Associated Testing Modes

	WX	SOX	C-L	C-S
Amb.Temp.	Approximates most closely approx.field conditions	Abnor. situation	Most closely approx. field conditions in the vadose zone	most closely approx. field conditions
Particle Size	crushed spl. may approx. a portion of natural system	pulverized spl. approx. even smaller portion of natural system	if well-sorted of approx. field conditions, if poorly sorted, airlocks	necessity, fine gravel particles high capillarity, completely airlocked
Leachant Temp.	natural below surface	105° C	natural	natural
Humidity (water)	available	depleted after 2 days	available	available

V. CAUSE AND EFFECT RELATIONSHIPS

Effect of Airlocks

Within the acid mine chemical reaction a necessary component for the complete oxidation of pyrite is oxygen. Several studies have shown that immersion of an acid producing sample in water was sufficient to arrest the oxidation of pyrite (Ed Leach, 1991). This was also demonstrated in the Canadian Study which used fine grained tailings materials. Thus, airlocking phenomenon and the subsequent exclusion of oxygen ingress is even more dominant in the column tests using small (<0.5 mm) particle size and small column tests in particular.

In a comparative study of tailings analysis using acid/base accounting, cells, columns and soxhlets, the columns using fine grained samples did not correlate as well with field conditions as the humidity cells (Bradham and Caruccio, 1991). Tailings samples, from a variety of mines in Canada, were used in the study to normalize the effect that particle size has on leachate quality. The samples used had a variety of neutralization potential/sulfur ratios. The results of the study showed that samples with high NP's and high S percentage gave similar results in all testing procedures. However, for samples with low NP's and low S percentage, the column tests performed least accurately in comparison to field conditions.

Referring to Figures 1a, 1b and 1c the acid or alkaline production of the samples is cross-

correlated between leaching test data (using columns, soxhlets and weathering cells) and whole rock analyses. The NP and sulfur ratio of a sample is plotted on the appropriate axis and if the sample produced acidity, it is designated by an asterisk, and if the sample produced alkalinity it is designated by an open circle. In the weathering cell tests and the soxhlet reactors, the chemical weathering attributes of the various tailings samples were more closely aligned to the whole rock composition than were the samples used in the column tests which showed random correlations.

Measurements of column porosity provided an explanation for apparent contradictory behavior of two samples and the suppression of acid production in a third column. Of the ten samples analyzed, the three sample above, as well as two others, had much lower porosities than the other columns. When these five columns were leached during the experiment, water was noted to pond on top of the samples and the leachant required up to seven days to permeate down through the columns. Also when the columns were dismantled, the low porosity samples exhibited mottling and cracking and, in one sample, a hard rind of oxidized sample adjacent to the column wall, incasing a core of relatively unoxidized sample.

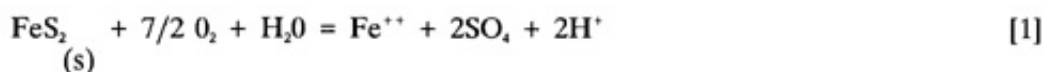
The data are consistent with the existence of "airlocks" within columns marked by such low porosities and permeabilities that had the effect of isolating the samples from oxygen infiltration and, thus, pyrite oxidation. In samples of high neutralization potential, the dissolution of carbonates is favored by water retention, the resulting leachate was alkaline. For samples with high sulfur and low NP, the result was reduced acid loads.

Therefore, while column tests utilizing well-sorted, coarse-grained samples (i.e., greater than 0.5 cm) most accurately resemble field conditions, when fine grained samples (i.e., less than 1 mm) are used, the presence of "airlocks" limits the transfer of oxygen required for pyrite oxidation and restricts acid production to the exterior surface of the sample. In this manner, pyrite oxidation in high sulfur samples may be suppressed or arrested completely.

Electro-chemical oxidation of pyrite

As an alternative to accepting the non-reactivity of pyrite oxidation by lack of oxygen, some researchers have called for the anaerobic oxidation of pyrite (electrochemical school of reactions) and have advocated the production of acid mine drainage under oxygen-free conditions.

The oxidation of pyrite generally follows the pathway expressed in equation whereby oxygen is the oxidizer.



Alternatively, it has been proposed that pyrite could be oxidized by ferric or ferrous iron and the dominant pathway is expressed by equations 2 and 3.

An examination of the complete reaction expressed in equation # 1 shows that under aerobic conditions, 1 mole of sulfate is produced for each mole of acidity producing a one to one equivalent of sulfate and acidity. Under the conditions expressed in equations 2 and 3 the complete reaction expresses an equivalent of 8 moles of sulfate for each mole of acidity, i.e., a mole equivalent ratio of 8:1. Plots of sulfate versus acidities have been compiled and are presented in Figures 2a, 2b, 2c, 2d and clearly show 1 to 1 mole ratios for a variety of field situations. This data base suggests that the electro-chemical oxidation of pyrite plays a minimal role in coal field environments.

Further, and as additional testimonial, several researchers have shown that submersion of acid producing material renders the samples inactive. The configuration of the columns and the sample size used in this study, were such that airlocks could not develop and, in fact, the observed stability of the pyrite was a result of isolation from the atmosphere. Under these conditions the samples were under negative Eh environments and afforded pyrite a mark in the stability field.

Effectiveness of submergence

Leach (1991) proposed a series of weathering tests using columns whereby samples of acid producing sandstone and shale would be weathered under vadose conditions, the zone of fluctuating water table and the zone below the water table. The results are presented in Figures 3a and 3b where accumulative acidity versus time plots are presented. Within the plots the samples designated with a V are vadose, F fluctuating water table and S for submergence. The results clearly show that submerging acid producing material below the water table have a dramatic effect on inhibiting acid production.

As an aside, during our investigation of several mines in Kentucky and Tennessee having a manganous-neutral drainage, we found that under the conditions of immersion and in the presence of organic matter, strongly reducing drainage networks were established which selectively solubilized manganese and produced drainages out of metals compliance. The responsible mechanism for this unique drainage quality was traced to the customary manner of disposing vegetation. In preparation for the stripping operation, vegetation is cleared and at time bulldozed into piles, which are burned. The hardwood ashes contain substantial amounts of manganese in the charred plant tissue (on the order of 10,000 to 15,000 ppm). Should this material be disposed within the backfill or adjacent to drainage networks and waterways, the digestion of the organic matter will further deplete the oxygen concentrations transforming the aquatic system into a reducing environment which will readily extract the manganese.

VI. RESULTS

Two statistical models were used to determine the significant factors of overburden analytical techniques. Sample storage temperature and humidity were evaluated as a single variable called "storage". Other factors tested were lithology, particle size, leachant temperature, the presence of iron oxidizing bacteria, and leaching interval.

Leachant temperature was found to have no effect on either pyrite oxidation or calcium carbonate dissolution. The factors of lithology, particle size, leaching interval, and storage

conditions were found to be significant at the $\alpha = 0.05$ error level. The lowest pyrite oxidation and calcium carbonate dissolution rates resulted from abiotic, low temperature storage conditions. Weathering rock samples at high temperatures (105- Q resulted in increased pyrite oxidation rates, dependent on lithology, accompanied by a concurrent increase in calcium carbonate dissolution in high NP samples. Secondary formation of calcium sulfate minerals from the high temperature storage of pyrite rich, high NP samples, may provide a highly soluble source of Ca^{2+} and SO_4^{2-} at the next leaching event. Pyrite oxidation appeared to occur at high temperatures in the absence of water, and longer periods of weathering at high temperatures resulted in higher pyrite oxidation rates. Smaller sample particle sizes resulted in higher pyrite oxidation rates, dependent on lithology. The presence of iron oxidizing bacteria resulted in higher pyrite oxidation rates in certain shale samples (Bradham, 1991).

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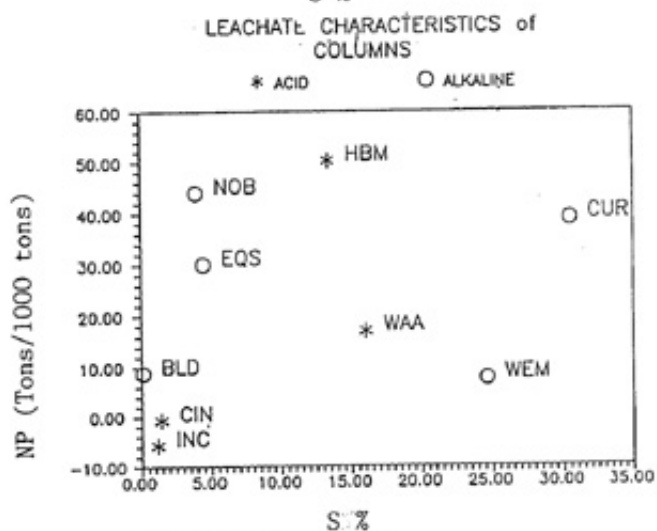
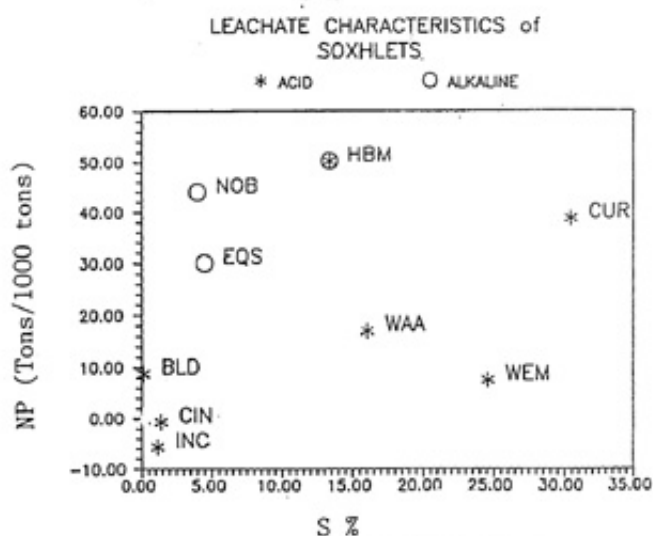
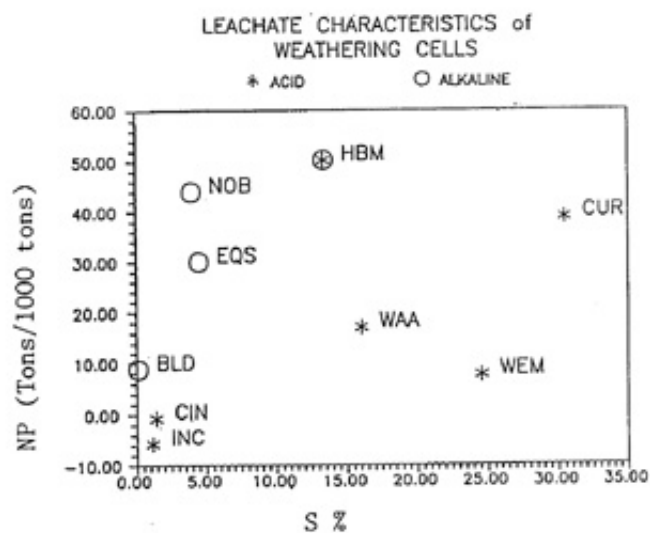
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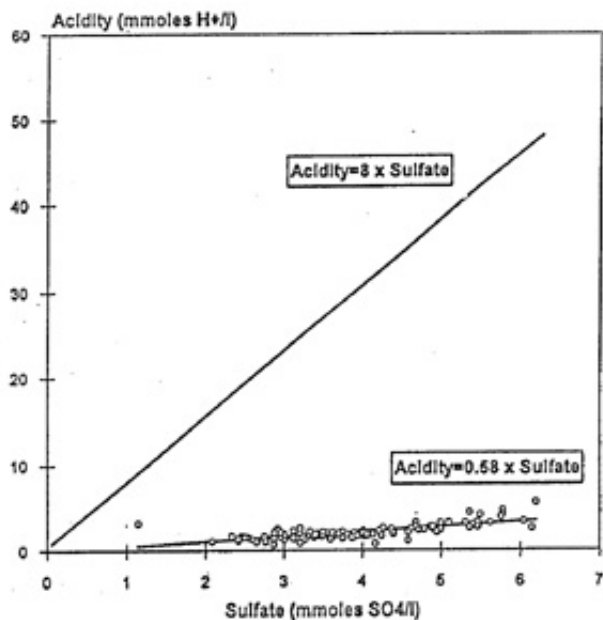
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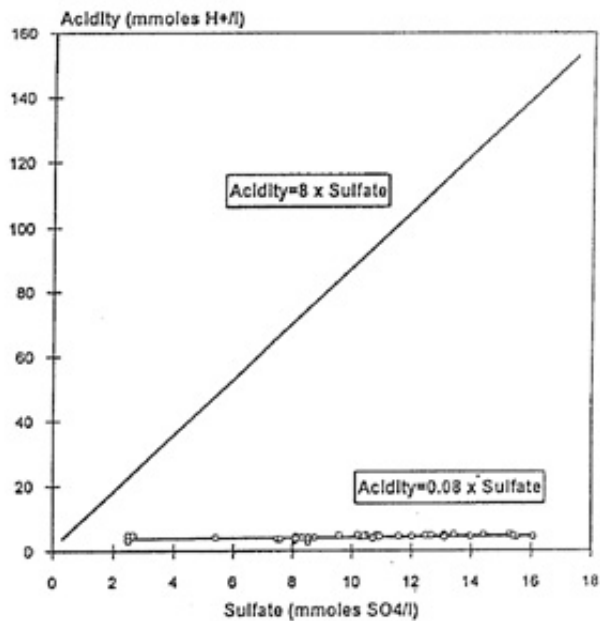


FIGURES 1A, 1B, 1C

Mercer Site, Seep #2



Two Hundred Acre Site, Seep #1



Eight Acre Site, Seep #6

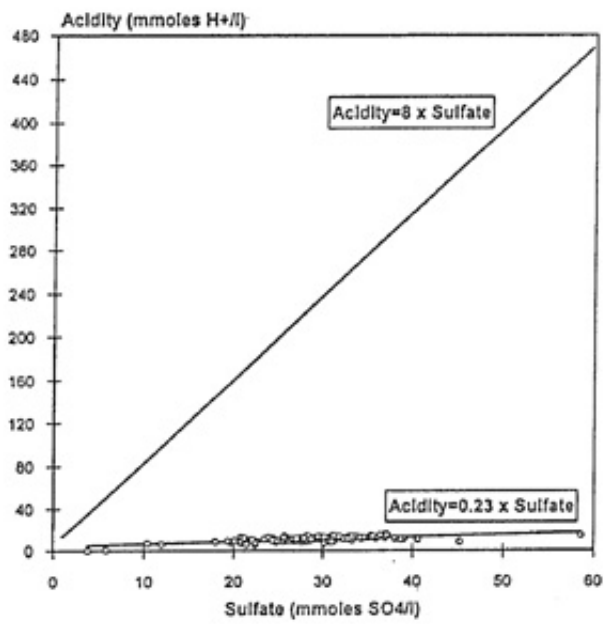
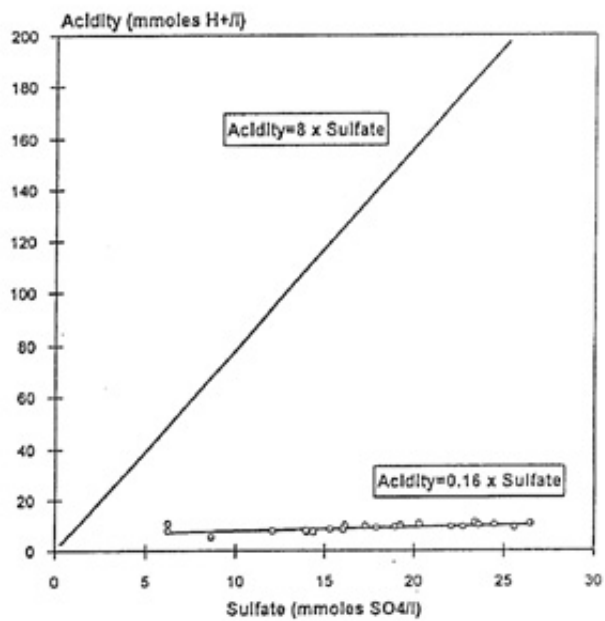
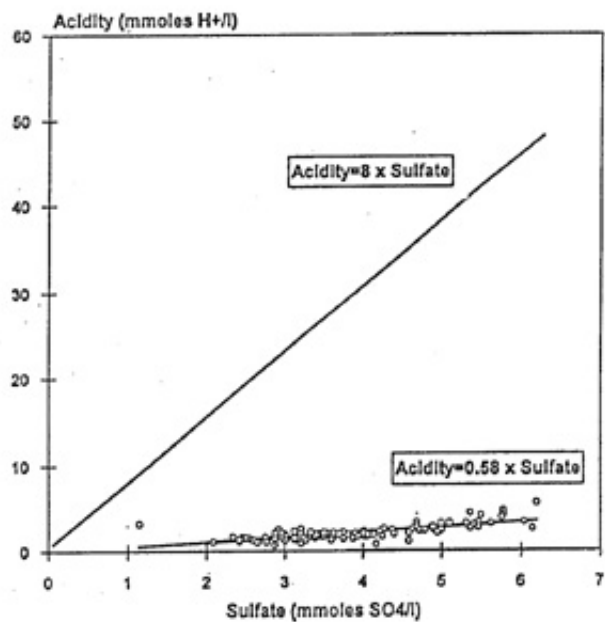


FIGURE 2 - Plots of Sulfate Versus Acidity

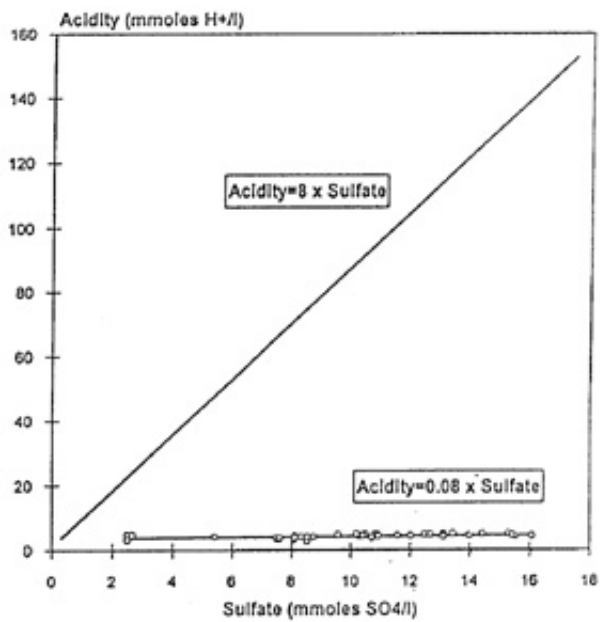
Forty Acre Site, Seep # 7A-1



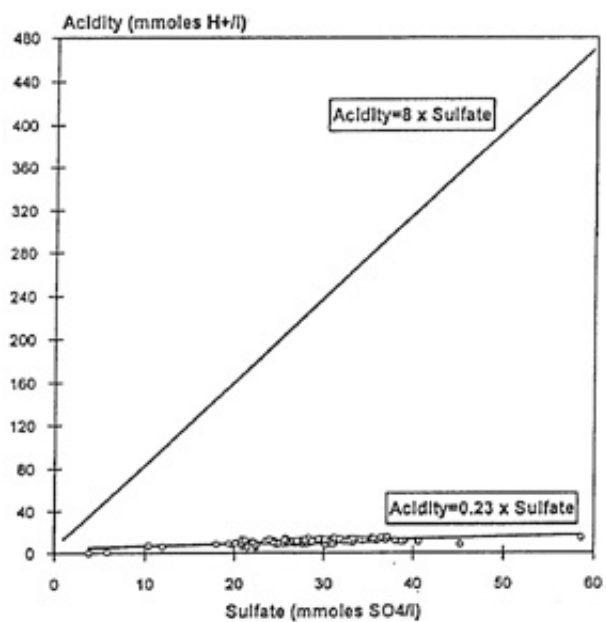
Mercer Site, Seep #2



Two Hundred Acre Site, Seep #1



Eight Acre Site, Seep #6



Forty Acre Site, Seep # 7A-1

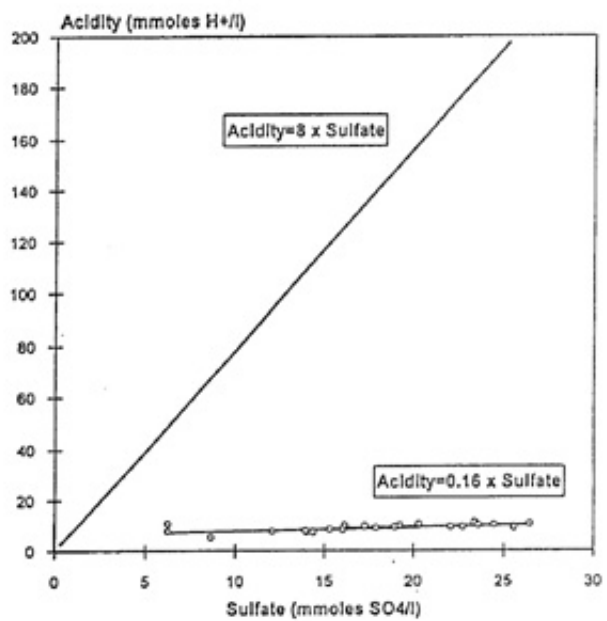
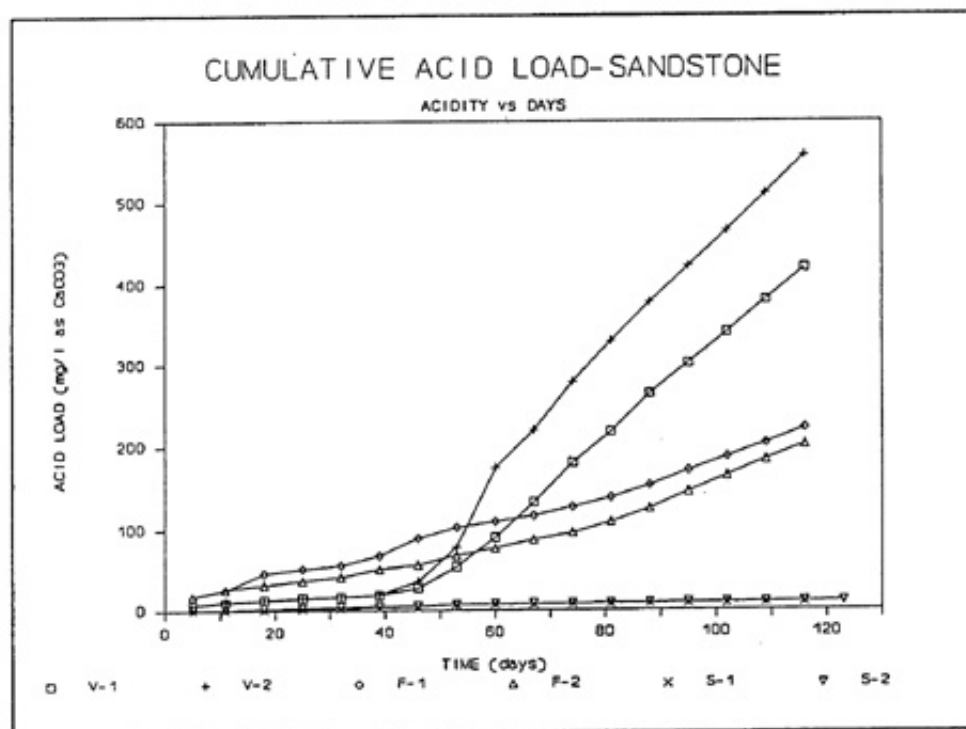
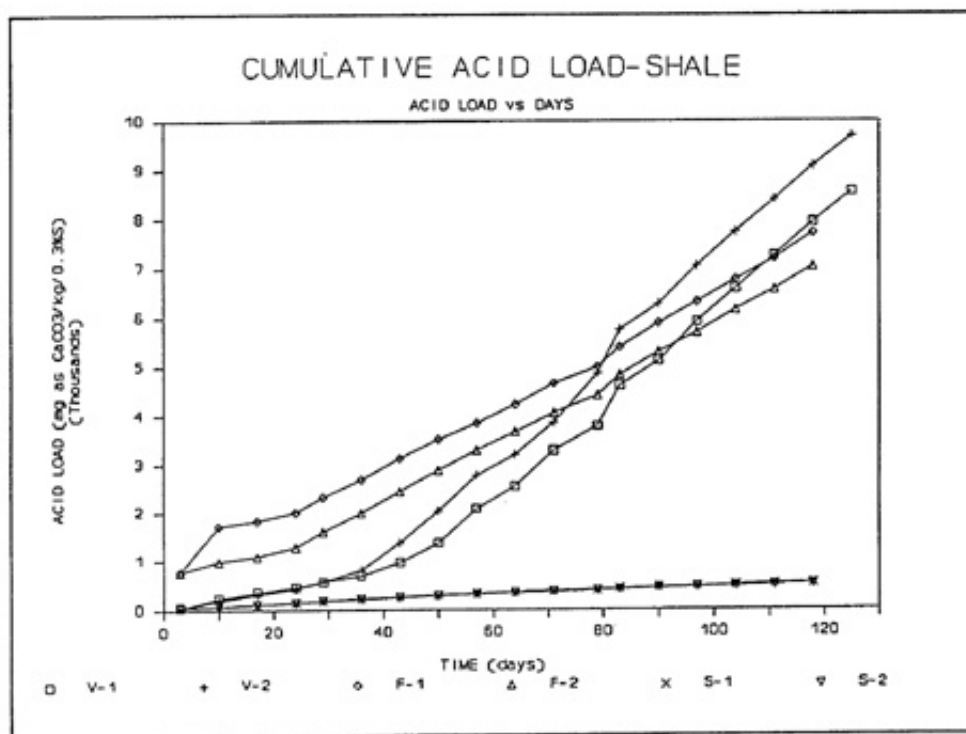


FIGURE 2 - Plots of Sulfate Versus Acidity



FIGURES 3A and 3B