FINAL REPORT

ACCELERATED WEATHERING OF TOUGH SHALES

by

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ABSTRACT

The purpose of this study was to find or develop a test that would identify a very tough but relatively rapid weathering type of shale that has caused problems when used in embankments as rock.

Eight shales, including the problem shale, were collected and tested by the slake, slake durability, and modified sulfate soundness tests, and a new sulfuric acid test that was thought to simulate the type of weathering that takes place in the field.

Quantitatively, the slake and slake durability tests did not differentiate between the shales very well. However, observations of the physical condition of the shales after testing did provide some useful information. The relatively rigorous modified sulfate soundness test and the new sulfuric acid test did differentiate between the shales, and the orders of responsiveness of the shales to the tests compared quite favorably.

The clay mineral chlorite was dissolved from the shales by the sulfuric acid and the shales underwent considerable physical distress. Thus, it is recommended that dark colored shales should be checked first for the presence of iron sulfide; if it is found to be present, then the shales should be checked for chlorite. If both minerals occur in the same shale, that shale should be considered to have the potential for relatively rapid weathering.

The variability determined by the more rigorous tests for shales that appeared to be of the same toughness suggests the need for a classification system for Virginia shales.

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INTRODUCTION

Shale is defined as "A laminated sediment, in which the constituent particles are predominantly of the clay grade. Shale includes the indurated, laminated or fissle claystones and siltstones. The cleavage is that of bedding and such other secondary cleavage or fissility that is approximately parallel to bedding. The secondary cleavage has been produced by the pressure of overlying sediments and plastic flow" (Howell 1957).

Shale is one of the most commonly occurring soil or rock materials on the earth's surface (Chapman 1975). Because of the wide geographic distribution of shale, it is commonly used in highway construction (Noble 1976). Factors such as jointing, cementation, mineralogical composition, and compaction affect the physical properties of shale. There is potential for considerable variation in such factors, thus it is understandable that the physical properties of shale have a very wide range. The properties of those shales that are frequently used within a specific geographic region become known to local engineers, who develop guidelines for their use. Because of the variability in shales, a particular set of local guidelines cannot freely be used in a different area; and even within a locality a shale may be encountered that differs from the norm to an extent that it cannot be properly handled under the established guidelines. Inasmuch as a shale that posed a particularly vexing problem was encountered in Virginia, it was investigated with the aim of developing a method for dealing with it.

PROBLEM SHALE

The problem shale, a very tough, dark gray material, was placed in an embankment 100 feet (30.5 meters) high on I-64, in the folded Appalachian Mountains in the vicinity of

Clifton Forge, Virginia. Field observations of the shale cored during a preliminary investigation and the need to blast it during excavation attested to its toughness. Because of its apparent toughness, the shale was placed in an embankment according to the specifications for the construction of embankments with rock (Virginia Department of Highways 1966). Approximately three years after construction, the embankment began to settle and move. In drilling augur holes through the embankment to bedrock for the placement of slope inclinometers, only a few holes had to be abandoned, moved a few feet, and restarted because of encountering anything too tough to drill through. Most of the material penetrated was soil-like.

PREVIOUS WORK ON PROBLEM SHALE

The Federal Highway Administration (FHWA) contracted with the U. S. Army Corps of Engineers at the Waterways Experiment Station (WES) in Vicksburg, Mississippi, to conduct a thorough investigation of the use of shale in embankments. In the early stages of the investigation, the Corps surveyed various states that had a history of problems with shale to determine the state of the art and to compile a list of either good and/or bad experiences that the states might have had with shale in embankments (Shamburger 1975). Later in the investigation a slake durability test was run on some fresh problem shale from Clifton Forge (Lutton 1977), and the high durability numbers determined would normally be interpreted as indicating that the shale was suitable for emplacement in an embankment according to the construction specifications for rock.

X-ray diffraction analyses made at the Research Council to supply information for the survey conducted by the Corps showed that a sedimentary chlorite was one of the major clay mineral components of rock sampled from the cut faces, and that very little of this chlorite was in the soil-like material taken from the embankment. The work done at the WES disclosed that pyrite, an iron sulfide, was disseminated throughout the shale and that the water used in the slaking test became acidic (Shamburger 1975). Inasmuch as chlorite will dissolve in acid and sulfuric acid is one of the products of the oxidation of iron sulfide, it is possible that when the shale was placed in the embankment the iron sulfide was oxidized, which produced sulfuric acid that contributed to the rapid weathering of the shale by dissolving the chlorite.

APPROACH TO HANDLING PROBLEM SHALE

While this particular problem shale may never be encountered again within Virginia's interstate system, it or a similar one may be involved in work on the state's primary or secondary road systems, and a method for handling it was thought to be needed. In preparation for the study reported here, the approach outlined below was suggested.

1. Locate or develop a test that will identify the problem shale.

2. Conduct a laboratory evaluation of techniques designed to prevent oxidation of the pyrite and subsequent weathering of the shale.

3. Apply one or more of the preventive techniques in the field and monitor the long-term condition of the embankment.

PURPOSE

The specific purpose of the study reported here was limited to the accomplishment of item one above, which was to locate or develop an accelerated weathering test that would identify shale that can be expected to break down in such a manner and length of time as to create settlement and/or movement in the embankment.

SHALES TESTED

Geologic Setting

The shales collected for this study were part of a Paleozoic sedimentary sequence deposited in the Appalachian geosyncline. They are from the Devonian Millboro and Brallier formations, which were deeply buried in the above mentioned order and subsequently folded. The dark color (carbon) of the Millboro and the occurrence of pyrite in it suggest that its burial was rapid enough that a reducing rather than an oxidizing environment was maintained. The combined effect of

the depth of burial (compaction through pressure), the folding (additional compaction through pressure and possible diagenesis through heat and pressure),and the mineralogy (a large proportion of silt size quartz) of the materials from both the Millboro and Brallier formations explains their toughness.

Geographical Setting

Geographically, these shales were taken from two general locations in the folded Appalachians. One area was along the recently completed sections of I-64 at the failing embankments near Clifton Forge and along a segment of I-64 that was under construction in a synclinal valley roughly paralleling Simpson's Creek. The second area was 0.5 mile (.81 km) into Highland County from the Augusta County line along Rte. 250.

Location and Description of Shales

1. Shale #1 was taken from the south side of the cut west of the bridge over Commerical Avenue where I-64 passes to the north of Clifton Forge. It is from the Millboro formation. This shale is dark gray, with alternating dark and light bands of variable thickness. During collection, it broke into slabby pieces 1 to 2 inches (25.4 to 50.8 mm) thick. It appears to be tough and well indurated. A freshly broken surface has a slightly rough, silty texture.

2. Shale #2 was taken from the north side of the above mentioned cut, and appears to be from the Brallier formation. This shale is greenish gray with indistinct bedding. It has limonite stain on joint surfaces and a few thicker, crusty deposits of limonite along what appear to be bedding surfaces. The texture is not quite as abrasive as that for shale #1.

3. Shale #3 was taken from close to the centerline of the eastbound lane of I-64 at station 787 + 00 during construction. It is from the Brallier formation. This shale is grayish green with indistinct, nonuniform bedding. Rounded, elongated blebs of deformed,slightly darker colored material are strung out along a roughly

linear pattern. It appears that pene-
contemporaneous erosion and rapid redeposi-
tion of coherent but relatively soft muds
occurred. The material sampled was
excavated by means of a scraper aided by a
bulldozer.

4.   Shale #4 was taken from close to the
      centerline of the westbound lane of I-64
      at station 633 + 00 during the excavation
      of a ditch that would hold a drain pipe
      under a proposed embankment. It is from
      the Millboro formation. This shale is
      dark gray to black with indistinct bed-
      ding in the size particles sampled.
      Breakage that occurred along some of the
      bedding planes exposed shiny black, car-
      bonaceous parting with striations indi-
      cating that movement had occurred along
      these planes at the time of deformation.
      Pyrite was on joint surfaces, and a very
      few thin white veinlets of calcite cut
      through the particles. The excavation
      was being drilled and blasted.

5.   Shale #5 was taken from along I-64 at the
      base of the north cut slope at station
      483 + 00. It is from the Millboro formation.
      This shale is dark gray to black with the
      bedding — or shaly, shiny, black carbona-
      ceous parting — tightly folded. There is
      much lensing and pinching out of the bed-
      ding. Numerous thin veinlets of white
      calcite traverse the particles. Pyrite
      occurs on both joint and bedding surfaces,
      as does limonite stain.

6.   Shale #6 was taken from a cut on a sharp
      U shaped curve 0.5 (0.81 km) mile into
      Highland County from the Augusta County
      line along Rte. 250. It is from the
      Brallier formation. This shale is grayish
      green to tannish green. The bedding is
      quite pronounced in the face of the cut
      but is indistinct in hand size particles.
      The beds vary in toughness; some silty
      members maintain their entity and stand
      out, while the more fissle members present
      an indistinct image of bedding. Jointing
      surfaces are covered with limonite stain.
      The bedding surface is gently rippled and
      has a slight sheen in direct light; magni-
      fication shows that many discrete mica flakes
      parallel the surface.

7. Shale #7 was taken from the same locality as shale #6 and, it too, is from the Brallier formation. This shale is grayish green to tannish green, thicker bedded than most of the other materials and seems siltier and tougher. None of its surfaces have the kind of sheen possessed by shale #6, but there are some discrete bright reflections from mica flakes.

8. Shale #8 was taken from the same locality as shales #6 and #7 and, like them, is from the Brallier formation and is grayish green to tannish green. Its extreme fissility makes obtaining large pieces difficult. Like shale #6, it has a slight sheen, and magnification discloses many discrete reflections of individual mica flakes parallel to the bedding planes.

TEST PROCEDURES

General

All matter attempts to be in equilibrium with its environment. Thus the shales investigated were affected by their depositional, burial, and tectonic environments. As mentioned earlier, the rapid deposition of silts and clays rich in organics probably resulted in a reducing environment. They also underwent deep burial beneath thousands of feet of Mississippian and Pennsylvanian sediments, thus being subjected to a great deal of compaction. Burial was followed by folding with the application of pressure and heat; and lastly they were elevated, weathered, and eroded.

These materials had ample time to equilibrate through all the conditions mentioned, with the exception of the weathering and erosion. The amount of time spent in the weathering environment was quite short compared to the time spent in burial and deformation. The shale that is exposed or that is near the surface has equilibrated or is in the process of doing so, but burial of 10 or 20 feet(3.05 or 6.1m)is probably quite effective in isolating that shale from the weathering environment. The principal factor affecting the rock used to construct an embankment is that the rock is removed from a state of equilibrium and placed in a state of disequilibrium. The length of time the rock takes to equilibrate is the factor that determines whether man can use it in a structure that is expected to have a reasonable life span.

In planning tests for the durability of the trouble-
some shale on I-64, it was thought that the procedures used
should in some way simulate the state of disequilibrium that
the shale will be in when placed as rock in an embankment.
The most obvious environmental change is that the shale is
exposed to large excesses of free water and air. The slake
tests that have been developed do a reasonable job of simu-
lating this change.   Inasmuch as sulfide is in some of the
shales and oxidation of a sulfide will produce sulfuric acid,
it seemed appropriate to try a test in which the shales were
to be soaked in various concentrations of sulfuric acid.

Premise for Testing

As noted earlier, shale has a very broad geographic
distribution and, because of its very variable physical prop-
erties, it is the source of many highway related problems.
Because many researchers have dealt with the problems,
numerous tests of shale properties and classification schemes
for shale have evolved (Smith 1967; Underwood 1967; Franklin
1972; Wood 1973; Morgenstern 1974; Reidenouer 1974; Bragg
1975; Chapman 1975; Shamburger 1975; Bailey 1976; and Lutton
1977). While the adoption of test methods already in use
elsewhere would have been convenient, and some were used,
the toughness of the shale in Virginia was anticipated to
dictate the need for developing a rigorous accelerated weath-
ering test. Thus, a testing program was planned that would
carry the shales from the less rigorous tests through the
more rigorous tests in the order that the tests are described
in the following section.

Description of Tests

Slake Test

The slake test makes an excess of water available
to a shale by submerging it in water. In the natural state,
a shale with absorbed water can possess a degree of stability
that will not be affected by dropping the shale into water.
However, drying the shale, as is done in the slake test,
destroys that stability and a poorly consolidated shale is
apt to slake quite readily.

A full description of the slake test used in this
study is included in Appendix A. Briefly, the steps in
running a slake test are to dry to a constant weight, weigh
six 150 gram-particles, submerge in water, observe over a 24-
hour period at various intervals, dry and weigh to determine

amount of loss, and repeat the cycle four times. The slake index is calculated according to the quantification developed by the Indiana State Highway Commission (Chapman 1976) by dividing the amount of loss times 100 by the original weight.

Slake Durability Test

The slake durability test varies from the slake test in that agitation is introduced by placing 10 particles of shale into a wire mesh (2.00 mm) drum and rotating it with its axis 20 mm above the level of the water. The greater energy involved in this test, and the control over the amount of energy expended by varying the total number of rotations, increases the resolution as compared to the slake test. Thus this test can differentiate between tougher shales than can the slake test.

A full description of this test as made is also given in Appendix A. (For details of the development of this test and its evaluation see Franklin 1972.) The steps in running the test are to dry to a constant weight, weigh ten 40 to 60 gram-particles, weigh the wire mesh drum, rotate the wire mesh drum and particles in water for 10 minutes at 20 rpm, rinse, dry, and weigh the drum and remaining shale, and repeat the cycle four times. The durability index is calculated by dividing the weight of the remaining shale times 100 by the weight of the original shale.

Modified Sulfate Soundness Test

While in the modified sulfate soundness test the medium in which the sodium sulfate is dissolved is water, the reaction of the water with the clay particles in the shale is not thought to be the principal reaction that takes place as it is in the slake test. The access of the solution to the pore spaces in the shale is important, as is the total effective porosity. The principal disruptive force involved in the modified sulfate soundness test appears to be the physical force exerted by the sodium sulfate crystals that form each time the shale is dried. Thus the critical process is that of alternate wetting and drying.

The standard sulfate soundness test uses a saturated solution of sodium or magnesium sulfate. A saturated solution would make the test much too rigorous for shales. Thus the modification of running the test with a 50% solution of sodium sulfate and with approximately 1,000 grams of only two size fractions was tested and did an adequate job of differentiating between tough shales (Wood 1973).

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A description of the modified sulfate soundness test
used in this study is included in Appendix A. For details
of the standard test see ASTM, part 14, C88-73 (ASTM 1973).
The steps in the test are to prepare the solution, wash all
dust and loose material from the rock, dry shale at 110 ± 5°C,
weigh, immerse specimen for 16 to 18 hours, remove and dry
at 110 ± 5°C, note condition of rock, and repeat the immersion
and drying until five cycles have been completed. After the
last cycle and subsequent cooling, wash specimen free of
sodium sulfate, dry at 110 ± 5°C, weigh total specimen, sieve
through 1/2 inch (12.7 mm) and 5/16 inch (7.9 mm) screens,
weigh the two size fractions, and photograph them. The sound-
ness index is calculated by dividing the final weight times
100 by the original weight.

Sulfuric Acid Deterioration Test

As was mentioned earlier, the presence of pyrite, or
iron sulfide, in some of the shales means that the potential
for the formation of sulfuric acid exists when the shale is
placed in an oxidizing environment. Because some clays are
susceptible to dissolution by acids, subjecting chunks of the
shales to sulfuric acid attack seemed to be a logical test
for simulating accelerated weathering. A preliminary check
on the effect of a very dilute solution (approximately 1%)
of concentrated sulfuric acid (18M) on some of the shales did
not give any visual indication that there was any acceleration
of the weathering process over a period of 1 to 2 months.
For the test used in this study, distilled water (0%) and
solutions of 25%, 50%, and 75% concentrated sulfuric acid
(18M) were prepared as the soaking mediums. As they were for
the sulfate soundness test, access to the pore spaces and the
total effective porosity are important properties of the shales
that affect the rate and total distructive effect of the acid
attack.

A description of the sulfuric acid test as used in
this study is included in Appendix A. Briefly, the steps in
running the test are to choose pieces of shale, wash off dust
and loose material, dry at 105°C, weigh, submerge in acid,
observe deterioration, stop acid attack, and check for its
effect by chemical analysis of the leachate for K, Ca, Mg, Fe
and Al, and by X-ray diffraction of the remaining particulate
matter.

Chemical and X-ray Diffraction Analyses

The clay minerals in shales are differentially sus-
ceptible to acid attack. For the purpose of this study, knowing
which of the clays were most vigorously attacked by the sul-
furic acid would be of benefit because their presence in a shale

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in the field would indicate a potential for accelerated weathering through acid attack. Therefore, chemical analyses of the sulfuric acid leachate and X-ray diffraction analyses of the unleached and leached shales were made.

The chemical analyses for K, Ca, Mg, Fe, and Al were made using standard procedures for the flame atomic absorption technique.

Most of the X-ray diffraction analyses were made on randomly oriented powders. In addition, powder of the untreated shales was suspended in water and aliquots of the suspension were pipetted onto porous ceramic plates. These preparations were irradiated untreated, treated with ethylene glycol, and heated overnight at 270°, 375°, and 500°C.

RESULTS AND DISCUSSION

X-Ray Diffraction — Unleached Shales

This discussion of the results of the X-ray diffraction analyses is presented out of the order previously established because knowledge of the mineralogy of the untreated shales should be valuable support data in the consideration of the other test results. Only the mineralogy of the clays that were not leached is discussed here because discussion of the changes in mineralogy resulting from treatment with sulfuric acid will be more meaningful if discussed along with all the results of the sulfuric acid test.

The mineralogy of the unleached shales is presented in Table 1. Because of the difficulty of dealing with clay minerals quantitatively, the relative abundance of the clay minerals in the shales was estimated using guidelines suggested by Weaver (1958). A semiquantitative terminology such as that used by Lutton (1977) was used here.

The most notable characteristic of these shales is that their principal constituents are quartz, muscovite —illite and chlorite in that order of abundance. The only exception is shale #5, which has only slightly less calcite than quartz. While the general statement concerning the quartz content is factual, it does not allow any distinction to be made between the various shales based on their quartz content. Yet the descriptions of the shales allude to differences in quartz content based on their degree of siltiness. Shale #7 was very silty and more massive bedded than the nearby shales #6

TABLE 1

Semiquantitative Mineralogical Composition of Shales

Shale No.	Clay Minerals			Nonclay Minerals					
	Muscovite-Illite	Chlorite	Kaolinite	Quartz	Plagioclase	Calcite	Dolomite	Siderite	Pyrite
1	C	R	-	A	R	R	R	Tr	Tr
2	C	M	-	A	M	-	-	-	-
3	C	R	-	A	Tr	-	-	-	-
4	C	R	-	A	Tr	Tr	Tr	Tr	R
5	C	M	-	C	-	C	-	-	M
6	C	M	-	A	Tr	-	-	-	-
7	C	R	-	A	R	-	-	-	-
8	C	R	-	A	R	-	-	-	-

Relative abundance determined by X-ray diffraction peak intensities; (A) abundant, greater than 50 percent; (C) common, 25 to 50 percent; (M) minor, 10 to 25 percent; (R) rare, 5 to 10 percent; (Tr) trace, less than 5 percent; (-) not present.

and #8, and might be expected to have a high quartz content. The most intense quartz peak, which is shared with muscovite, was allowed to go off the top of the chart paper so as to exaggerate the clay minerals' peaks and cannot be used for a comparison of the relative quantities of quartz in the shales. However, the 4.26A° quartz peak stayed on the chart and can be used. Based on the intensities of the 4.26A° peak, shale #7 has the highest quartz content, shales #1, #2, #6, and #8 are comparable to each other and have noticeably less quartz than #7, and shale #5 has the least quartz. The quantity of quartz in shales #3 and #4 falls between the values for the group of shales #1, #2, #6, and #8, and that for shale #5. These relative abundances of quartz may be of interest when the quantity of cations dissolved from the clays by the sulfuric acid are considered. The mineral name muscovite was used rather than mica because so many of the muscovite peaks were indexed. In all the shales, chlorite is no more than one-third as abundant as is the muscovite-illite. Nevertheless, this particular constituent is very important because it is extremely susceptible to dissolution in acidic solutions.

Another distinction to be made between the shales is the occurrence of carbonates, principally calcite, with dolomite and siderite in some, and pyrite in shales #1, #4, and #5. The occurrence of calcite along with illite can portend problems in that the potassium can be leached from the illite during weathering and can be replaced by calcium that is available as the calcite is dissolved. With very little other change in the crystal structure of the illite, it can develop into an expansible clay with a greater affinity for water than the original illite. However, this is not considered to be a problem because of the extremely rapid deterioration of these shales. The occurrence of the pyrite is especially important because of the potential for the formation of sulfuric acid if the pyrite is oxidized.

General

When dealing with material that is as variable in its physical properties as is shale, the likelihood that one specific test can differentiate and thus characterize the individual shales in a group is very slim. Thus, to adequately differentiate individual shales it is necessary to use a classification system based on the results of several tests. Deo devised such a system (Wood 1973) that uses the slake (1 cycle), slake durability (500 revolutions), and modified sulfate soundness (5 cycles) tests, see Figure 1. As part of the discussion of the test results, the classification of the shales according to Deo's system will be presented so that the effectiveness of the system with the Virginia shales may be evaluated. However,
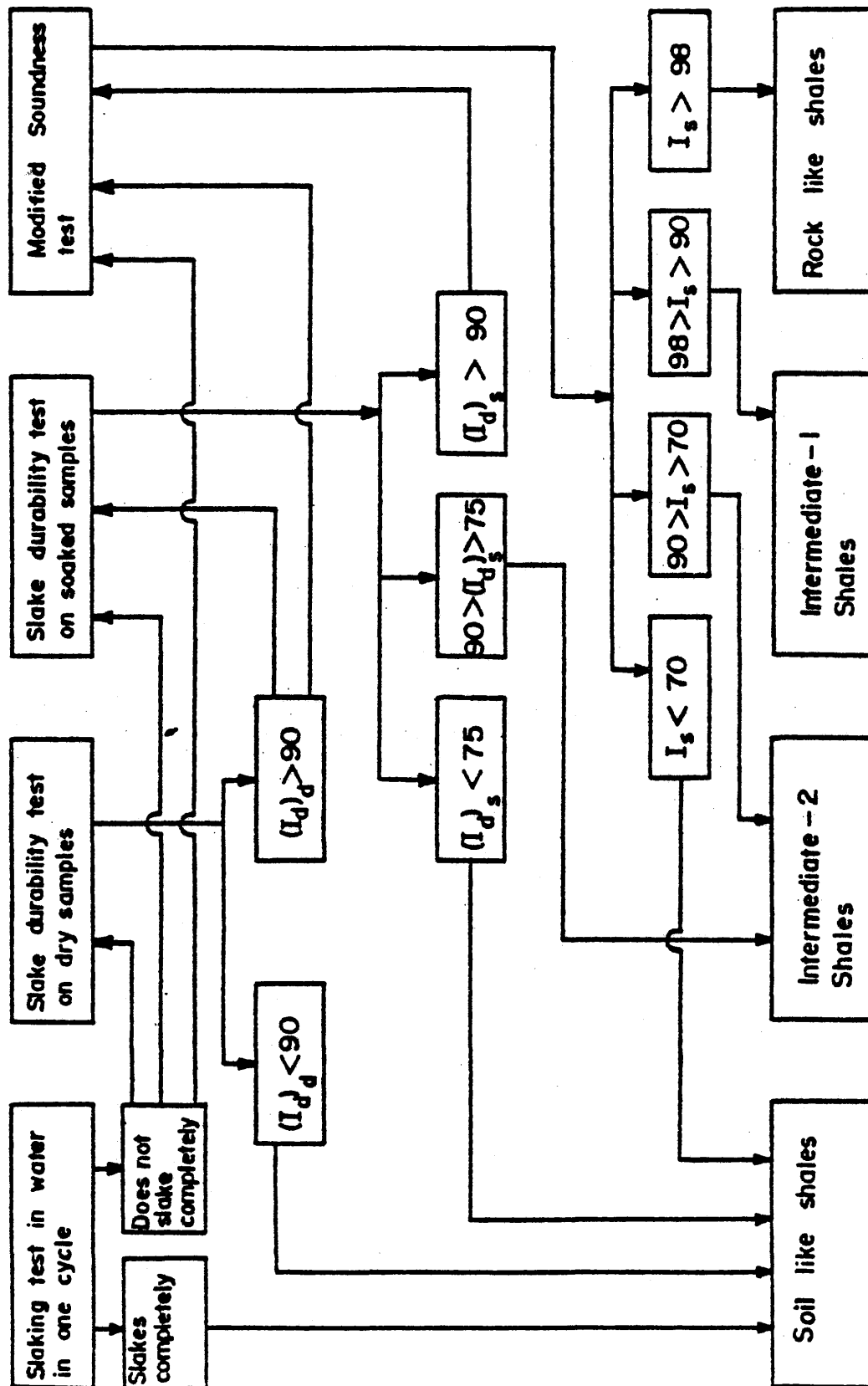
Figure 1. Deo's proposed classification or indiana snales for embankment construction (Wood 1973).

the toughness of the Virginia shales dictated that a large input of energy be applied in the tests on them. Therefore, the Virginia test procedure ran the tests as follows: slake (5 cycles), slake durability (1,000 revolutions), and modified sulfate soundness (5 cycles). Where possible, values that are directly comparable to those obtained by Deo are presented.

Slake Test

The slake test has been used primarily in a qualitative manner, and only recently has the Indiana State Highway Commission developed a quantification of the test (Chapman 1976). The results of the slake test are listed in Table 2. With soundness index (SI) values ranging from 0.3% to 8.2%, the test does not appear to have the potential for differentiating varying durabilities of relatively tough shales based solely on the quantified results. As the test is being run, observations of the physical condition of the shale are supposed to be made and recorded. Though qualitative in nature, such observations can serve to differentiate the reactions of the various shales where the magnitude of the differences is significant. The obvious comparison is the one between those shales that had slake indices that differed by as much as an order of magnitude. Thus the observations for shales #1, #2, and #7 will be compared with those for shales #3, #6, and #8. All the shales gave off air bubbles as they absorbed water. No change was noted for shale #1; bedding plane cracks opened during soaking and heating for #2, but very little material slaked off; and hairline cracks that were more easily seen in the dry condition than the wet developed in #7, which maintained sharp edges through 3 cycles. For shale #3, some slaking started within an hour, hairline cracks appeared after 2 hours, were longer at 8 hours, and the shale had broken down badly at the end of 5 cycles (Figure 2). Particles slaked off in the manner of exfoliation with as many as 30 particles with long dimensions of 1/2 to 1 inch (1.27 to 2.54 cm) developing. Hairline cracks developed in #6, with some slaking of slivers of rock and eventual separation along the cracks into 2 or 3 pieces. Hairline cracks with the slaking of small slivers but no separation of the particles occurred for #8. While shale #3 suffered the greatest apparent distress, it developed fewer particles small enough to pass through a 2 mm screen than did #6, which had a higher SI. No slaking was noted for #4 and only slight slaking and some cracking was noted for #5, which also experienced some chemical activity as shown by the precipitation of a limonite-like stain on the white calcite. It seems probable that iron goes into solution when the iron sulfide is oxidized and forms acidic conditions, and is precipitated out of solution in the slightly alkaline conditions existing in proximity to the calcite.

TABLE 2

Test Data

Shale No.	Slake Index		Slake Durability Index		Modified Sulfate Soundness Index	
	1 cycle	5 cycles	I_{d500}	I_{d1000}	Total Sample	1/2" to 3/4"
1	0.08	0.3	99.0	98.5	84.3	66.9
2	0.12	0.3	98.1	96.7	42.0	16.5
3	1.35	4.0	96.6	91.9	01.3	0.0
4	0.17	1.5	99.0	98.0	64.7	35.3
5	0.41	2.4	98.8	97.4	70.0	54.0
6	0.56	8.2	94.6	87.9	48.3	27.8
7	0.30	0.45	98.7	97.7	86.1	73.0*
8	1.33	4.0	94.6	90.3	46.9	22.7

Note: 1 inch = 25.4 mm

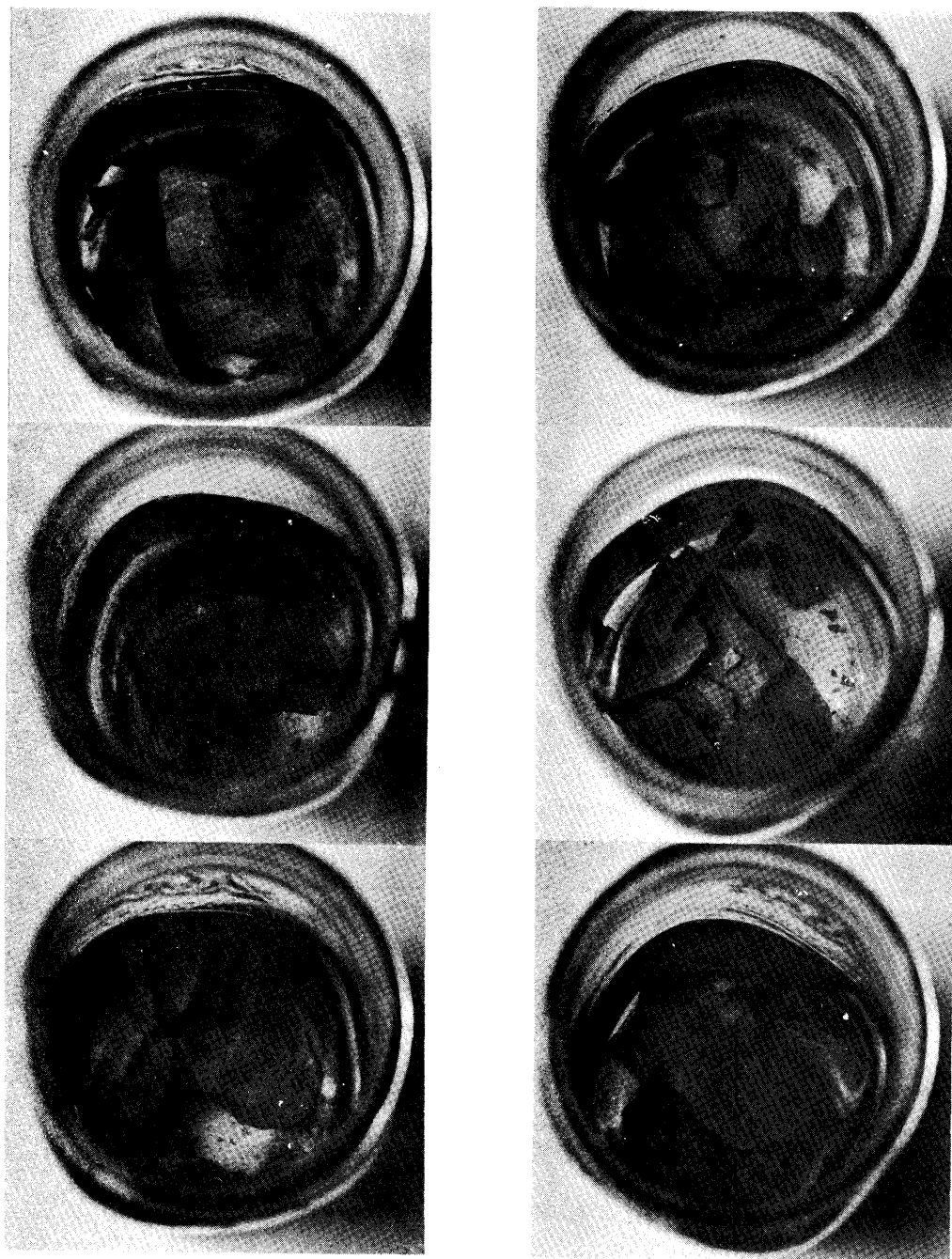* Ten cycles rather than five were run on shale #7.

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Figure 2.   Shale #3 after the 5th cycle of slaking, from
the top, and left to right 2-1, 2-2, 2-3, 2-4,
2-5, and 2-6.

The variability of the shales' reactions to the slake test based on observations was rather limited for shales #1, #2, #4, #5, and #7. A limited variability might be expected for #1, #2, and #7 because of the closeness of their slake indices. However, #4 and #5, with SI's 5 to 8 times as large as those for #1 and #2, did not seem more distressed than #1 and #2. The shales with the largest SI's appeared to have quite variable reactions which were not directly proportional to their SI's. As mentioned above, shale #3 appeared to break down to a much greater extent than did #6, which has an SI twice that of #3. Because of the inconsistencies between the SI's and observations just mentioned, it is obvious that the slake test cannot be used as the only test in classifying shales.

Inasmuch as none of the shales slaked completely, they would move on in Deo's system of classification to testing by the slake durability test.

Slake Durability Test

The results for the slake durability test are listed in Table 2. The values for a durability index for 500 revolutions (I_{d500}) range from 94.6 to 99.0 and do not make a clear distinction between the shales. On the other hand, the values for $I_{d1,000}$ seem to fall into two groups, one with values from 96.7 to 98.5 and the other with values from 87.9 to 91.9. Thus shales #1, #2, #4, #5, and #7 seem to be tougher than shales #3, #6, and #8. That a distinction can be made between the shales after 1,000 revolutions illustrates the necessity of applying a greater input of energy to the Virginia shales than to shales from less rigorous geologic environments.

The observations of the physical condition of the shales after 1,000 revolutions also tended to divide the shales into two groups. Statements that the particles did not break up but that the corners and edges were rounded characterized the observations for shales #1, #2, #4, #5, and #7. While statements that the particles broke down a good bit (Figure 3) or that some particles remained intact while others broke down completely (Figure 4) characterized the observations for shales #3, #6, and #8.

Based on Deo's classification system and using the I_{d500} values, all the shales should be put through the modified sulfate soundness test. Even if the $I_{d1,000}$ values were used, testing would continue on all the shales except #6. Thus, Deo's classification system indicates that none of the shales used in the study can be easily classified as soil-like.

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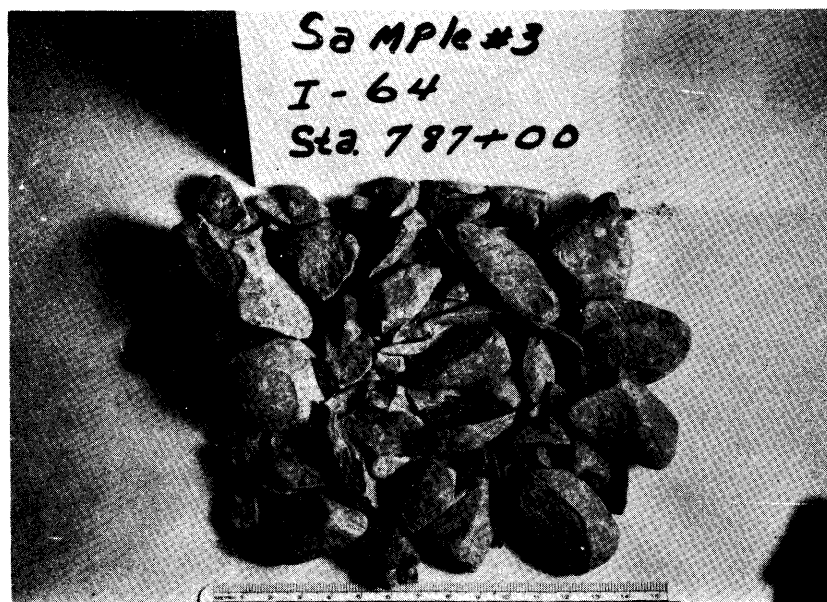
Figure 3.   Shale #3 before test (top), and after 5th cycle
of slake durability.
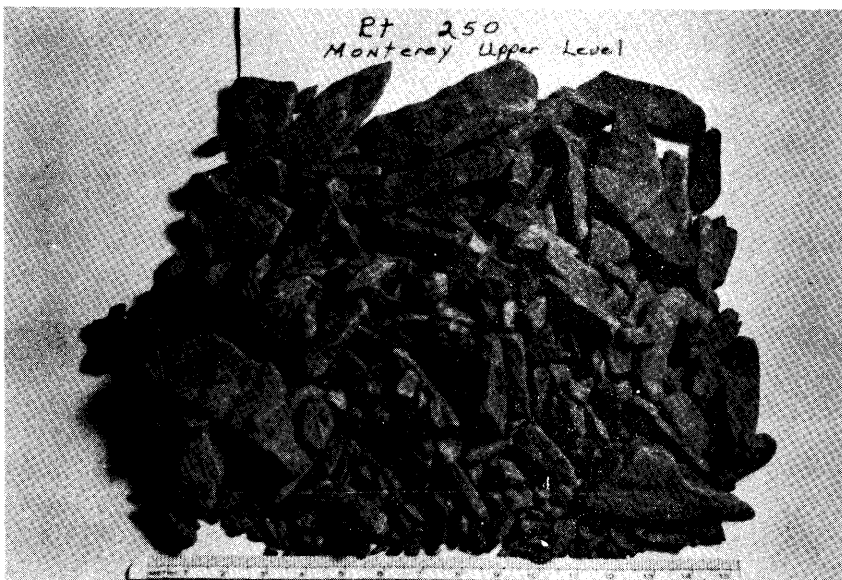
Figure 4.   Shale #8 after the 5th cycle of slaking.

Modified Sulfate Soundness Test

The results of the modified sulfate soundness test are listed in Table 2.   All the shales but #7 showed a distinct effect by at least the end of the third cycle and were tested, as planned, for only 5 cycles. Because shale #7 did not show much of an effect after 5 cycles, it was tested an additional 5 cycles. Thus, in comparing the results listed in Table 2, the reader should keep in mind that the values for shale #7 are for 10 cycles of soaking and drying. The standard eval- uation of the sulfate soundness test combines the quantitative sieving of the sample on a 5/16 inch (7.9 mm) screen and weigh- ing of the fraction remaining on the sieve, along with the observation of the physical condition of the particles of rock. An extra sieving was made before the standard sieving using a 1/2 inch (12.7 mm) screen so that the effect of the test on the larger size fraction could be assessed. Therefore, Table 2 contains values for the total sample and for the 1/2 inch (12.7 mm) to 3/4 inch (19.0 mm) size fraction.

The modified sulfate soundness test has been termed quite severe on soft shales and ineffective on the very hard ones. This evident lack of resolution is a disadvantage of the test (Chapman 1976). The values for both the total sample and the large size fraction seem to indicate that the shales used in this study can be grouped according to their reaction to the soundness test into four groups. Looking at the values for the total sample, the values for the groups would range from 0-40, 40-60, 60-80, 80-98, and 98+. While none of the

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values are 98 or above, it seems appropriate to include such a category for rock-like shale inasmuch as this type of shale has been so categorized by Deo (Wood 1976) and can be expected to be encountered in Virginia. Shale #7 with a 10-cycle value of 86.1 might be expected to have a much higher 5-cycle value, especially inasmuch as the observations state that it started to crack after cycle 4 and only after 8 cycles did it reach a rate of cracking comparable to that reached by shales #4 and #5 after 3 cycles. Unfortunately, the information needed to estimate the 5-cycle value for shale #7 does not exist; so where it falls between 86 and 100 is problematic.

The observations made for the modified sulfate soundness test will not be detailed here. However, they did complement the values listed, in terms of when the rock started to crack and the general appearance of degradation of the rock.

The important observation to be made concerning the data presented is that the modified sulfate soundness test differentiated between the eight shales tested quite well, and as used on these shales the test does not seem to suffer from a lack of resolution. In addition, shales which had not acted as soil-like shales through the slake type tests are now classified according to Deo's system as either soil-like shales or intermediate - 2 shales.

Sulfuric Acid Deterioration Test

A large quantity of data and information was accumulated from the sulfuric acid tests. A total of 640 chemical analyses were made and the soaking particles were observed once a work day through 16 days, then once every third day through the 42nd day, and finally on the 56th day. The observations were made less frequently as it became more difficult to distinguish differences in the physical condition of the particles. As the changes in the particles developed, it became obvious that a photographic record of the changes would be valuable. However, it was too late to start with the particles being soaked for the chemical analyses because a great deal of change had already occurred. Therefore, it was decided to start a fresh group of particles soaking in acid so that a photographic record could be obtained of the changes in the physical appearance of those particles. Observation of the first group of particles up to the time that it was decided to start a new group demonstrated that the 25% and 50% concentrations of acid had the greatest effect on the shales. Thus for the photographic record, only those two concentrations of acid were prepared. The record that was obtained, when compared with the observations of the particles treated for the chemical analyses, closely parallels the written descriptions of the deterioration.

All the information accumulated will not be discussed in detail here because such a discussion would contribute little to the fulfillment of the purpose of this study. Only the results of the tests of the leached shales and leachate of the 25% concentrated sulfuric acid solution will be discussed for reasons stated later. However, all the results of the chemical analyses are in Appendix B, Tables A,B,C,D,E,F,G, and H. Two series of photographs, one showing the deterioration of a severely distressed particle, 1-P-25 (P for photographed), and the other showing the deterioration of one of the least distressed particles, 7-P-25, are included in Appendix C as Figures A and B. No scale is included in the photographs because of the difficulty of including one and because the image of the particle photographed through the side of a beaker is somewhat distorted. However, the beaker and some of the markings on the beaker can be used as reference points for comparing the changes in the particles with time.

General Comments

Based on observations of the physical changes that occurred in the acid solutions and in the particles of shale, it was obvious that the 25% solution of concentrated sulfuric acid reacted quicker and had a much more deleterious effect on the shales than did the other solutions. Of the three concentrations of acid used, the 75% solution seemed to have the least effect.

The responsiveness of the clays to the acid solutions in terms of which shale was affected the most and which the least was rather easy to determine based on the comments about the samples soaked for only 1 day in the 25% solution of acid.

Shale #3 imparted a green color to the solution and the particle had many cracks both spheroidal and longitudinal. The particle appeared ready to crumble but it maintained its integrity when tapped with a glass rod.

Shale #2 colored the solution green and the particle had 1 crack parallel to the bedding and through the particle and 1 starting, plus 2 spheroidal cracks.

Shale #8 colored the solution green and the particle had 3 to 4 cracks parallel to the bedding starting on the front side.

Shale #6 colored the solution green and the particle had a distinct bedding plane crack from front to back.

Shale #5 had a slight crack parallel to the bedding near the bottom of the particle.

Shale #4 had an open crack along the bottom edge of the particle. The crack may have occurred during the crushing operation.

Shale #7 showed no apparent change.

Shale #1 showed no apparent change.

As a check on the order established based on 1 day of soaking, the comments on the appearance of the particles that were soaked for 14 days in a 25% solution of concentrated sulfuric acid were reviewed through the 6th day of soaking to determine what order of responsiveness would be established. The order, in decreasing degree, was shale numbers 3, 8, 2, 6, 4, 5, 1, and 7. Basically, there is very little difference between the two orders. The differences, which are considered to be minor, are that the positions in the order were reversed for shales #8, and #2, #4 and #5, and for #1 and #7.

The correlation of the data from a test being developed with the data from an established test is important in that the extent to which they correlate can either lend credence to or take credence from the test being developed. The data for the modified sulfate soundness test in Table 2 indicate a decreasing order of responsiveness for the shales as follows: #3, #2, #8, #6, #4, #5, #1, and #7. This order of responsiveness compares very favorably with the orders established by observation of the sulfuric acid tests.

Being able to establish the order of responsiveness of the shales as was done above is important, because observation of the physical condition of the shale-acid system would be the principal analytical tool if the sulfuric acid test was eventually used to identify tough shales that are susceptible to relatively rapid weathering. A test for this purpose should be simple and should require a minimum of any other type of support testing.

The physical appearance of the particles of shale resulting from acid attack suggests that the mode of attack takes various paths depending on the dominant properties of the shale. In most of the particles the bedding planes seemed to be the dominant feature along which the acid attack progressed, because the severely affected particles had the appearance of a sheaf of somewhat disarranged sheets of paper (Figure 5). In shales #6 and #8, the jointing seemed to have a very strong effect along with bedding because the particles had the appearance of
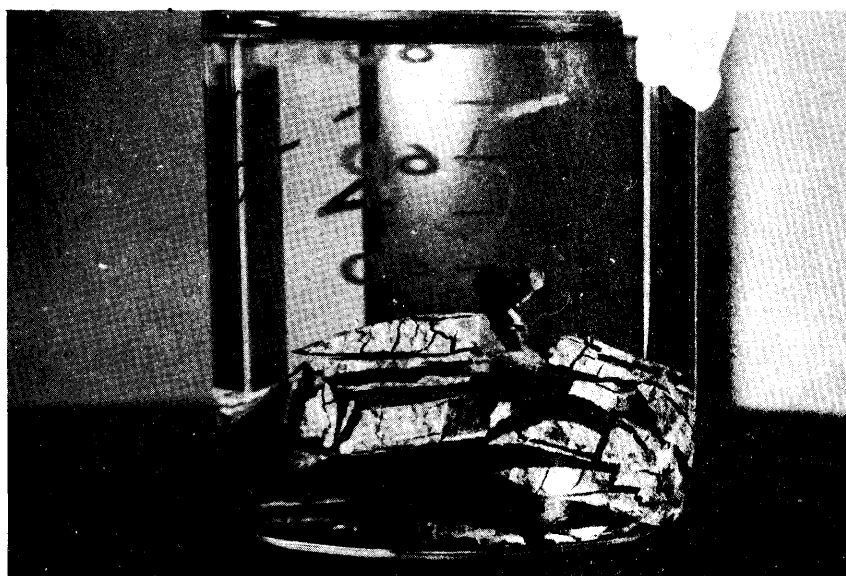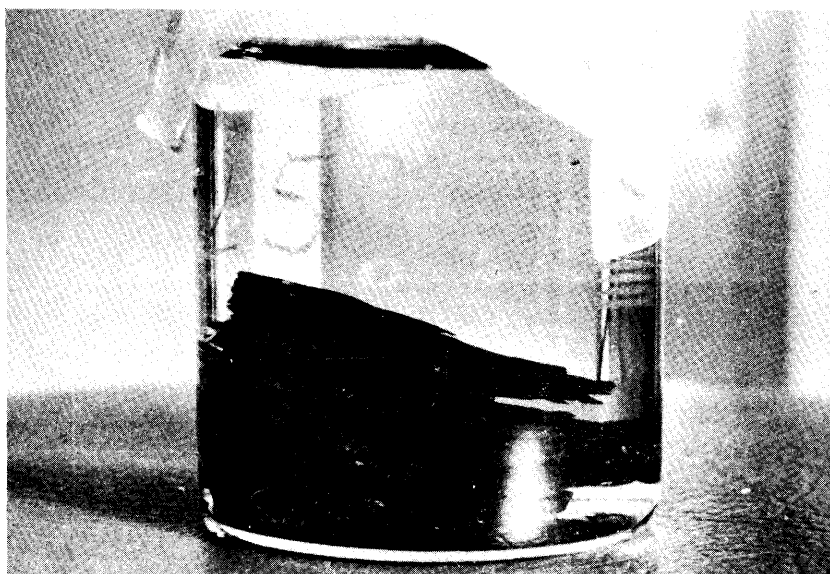
Figure 5.   Shale #5 and Shale #6 after 56 days in a 25%
            solution of concentrated sulfuric acid.

a stack of 2 x 4's as a result of the intersecting planes
(Figure 5). The particles of shales #3 and #7 were rather
equidimensional and much of the cracking had a spheroidal
pattern. Deterioration also progressed along the bedding
in shale #3, and some particles developed a clam shell appear-
ance with a major crack almost through the particle to a
hinge line. The attack of discrete beds on the inner sur-
faces thus exposed to the acid caused progressive expansion
of the major crack.

X-ray Diffraction — Leached Shales

As was mentioned earlier, all the information accu-
mulated during this study will not be discussed here. Be-
cause the 25% solution of concentrated sulfuric acid appeared
to be the fastest acting solution, most of this discussion
will be focused on the particles of shale leached with that
concentration.

The X-ray diffraction test allows a look at the
mineralogy of the shales. Any change in the relative quantities
of minerals in the shales can be determined by comparing the
X-ray diffraction patterns of the unleached shales with the
patterns of the leached shales. Knowing that chlorite occurs
in the shales studied and that it is susceptible to dissolution
by acid solutions, one would expect that as the acid comes in
contact with the chlorite, the chlorite would dissolve and less
of it would be detected on the X-ray diffraction patterns with
longer leaching periods.

Changes in the chlorite content of the shales detected
by X-ray diffraction indicated that the shales were affected
the most by the 25% solution of acid. Shales soaked in dis-
tilled water did not experience any changes in clay mineralogy,
and some of the shales soaked in the 50% and 75% solutions of
acid maintained chlorite for a much longer time than they did
in the 25% solution of acid. The interpretation of the X-ray
diffraction patterns coincided with the observations reported
on earlier in that the 75% solution of acid had less of a de-
structive effect on the shales than did the other concentrations.

With quartz being used as an internal standard, all
the quantitative estimates of changes in mineralogy were made
based on changes in the intensity of the clay peaks related
to any changes in the 4.26A° quartz peak. Muscovite-illite
did not deteriorate appreciably in any of the leaching solutions.
In fact, for some of the longer leaching periods the 9.95A°
peak increased in intensity as did the 4.26A° peak for quartz.

This increase in intensity is attributed to a concentration
of these minerals by the removal of the chlorite. All the
comments that follow relate to the diffraction patterns of
the shales that were leached with a 25% solution of acid.

The amounts of chlorite remaining in the shales
leached with a 25% solution of concentrated sulfuric acid
are listed in Table 3. Deterioration of the shales progressed
rapidly. After just one day of leaching the chlorite content
of shales #1, #2, #3, #4, and #5 decreased from 67% to 80%, and
only a trace of chlorite remained in shale #6. By the 28th
day of leaching no chlorite remained in the shales, except
for the questionable indication of a trace in shale #3 soaked
for 56 days.

TABLE 3

Amount of Chlorite Remaining In Shales Leached
In 25% Concentrated Sulfuric Acid

Shale No.	Days			
	1	14	28	56
1	75	33	0	0
2	67	Tr	0	0
3	67	Tr	0	Tr?
4	75	Tr	0	0
5	80	0	0	0
6	Tr	0	0	0
7	100	90	0	0
8	100	0	0	0

Neither the decline nor the maintenance of chlorite in the shale is, as it was initially thought to be, a particularly good measure of the shale's responsiveness to the acid leaching. The gross physical condition of the shale seems to depend on the presence or absence of major avenues of intrusion, such as bedding planes and joint systems, that the acid solution can follow. Opening and expanding such avenues of intrusion severely distressed the particle. However, aside from the flakes of chlorite exposed along these surfaces, access to the chlorite in the interior of discrete particles is controlled by the porosity and permeability of the shale. Thus some shales may be quite distressed without yielding much of their chlorite content to solution.

Chemical Analyses

The leachates were analyzed for potassium, calcium, magnesium, iron, and aluminum because these are the elements that would be dissolved from those minerals expected to experience the greatest distress from exposure to sulfuric acid. Thus the extent of the acid attack on the clay minerals can be monitored by testing for these elements. The results are expressed in moles. A mole is the "mass numerically equal to the molecular weight. It is most frequently expressed as the gram molecular weight" (Hodgman 1959). Expressing the data in moles allows a comparison on an atom for atom basis. This type comparison is advantageous when the objective of the analyses is to understand what happens to the various minerals.

Calcium and its role in the chemical process are not discussed in detail later because (1) calcite, the principal source of calcium in these shales, was detected in only three of the shales; (2) very little calcium was leached from those shales not containing calcite; and (3) calcium in solution in the various acid solutions decreased with time rather than increased as might have been expected. The decrease in soluble calcium and the disappearance of the mineral calcite suggest that the calcium combined with other elements and precipitated out of solution. That $CaSO_4 \cdot 2H_2O$, or gypsum, precipitated is a possibility, considering the high concentration of the sulfate radical in the sulfuric acid solution.

The results of the chemical analyses are presented in Figures 6 through 9, where the moles of the element leached are plotted against the amount of time the shale was soaked. The interpretation of the results would be simplified if each element could be assigned to a specific mineral. However, this cannot be done unequivocally because the shales are an aggregate of minerals, some of which contain some of the same elements. But not all the minerals in the shales are equally susceptible to acid attack, so the minerals that are the most susceptible were considered as the primary source of the elements in the leachate. Muscovite-illite is not as soluble

in acid as is chlorite and is expected to give up only its interlayer cation, potassium. Leaching of the interlayer cation is typically the first step in the weathering of muscovite-illite. Chlorite is totally broken down by acid attack and was considered to be the primary source for the magnesium and aluminum. Initially some of the iron was attributed to the possible breakdown of the pyrite, but the X-ray diffraction patterns did not indicate any decrease in the quantities of pyrite in the leached shales. Thus the chlorite was considered to be the source of the iron as well, even though a small amount of iron may have come from dis-solution of iron oxide stain.

Dissolution of Clays

Inasmuch as the quantities of muscovite-illite in the eight shales, as estimated using X-ray diffraction, were at least double the quantities of chlorite, the fact that the quantities of magnesium and iron leached from the chlorite were greatly in excess of the quantity of potassium leached from the muscovite-illite is evidence of muscovite-illite's insensitivity and chlorite's extreme sensitivity to sulfuric acid attack. Thus it is easy to understand the results of the X-ray diffraction test showing that the chlorite disap-peared while the muscovite-illite seemed to increase in quantity because of the relative concentration it experienced as the chlorite was dissolved.

Reaction Rate

The rates at which the cations were taken into solution cannot be used for the extrapolation of rates of solubility for either the minerals or the shales, because these rates were affected by the solubility of the minerals, the amount of specific minerals in the shales, the porosity and permeability of the shales, the amount of the minerals exposed on the surface of the particle, and many other factors whose effects would be very difficult to determine.

However, the curves in Figures 6 through 9 clearly show that the chemical reaction between the acid and the shales came very close to peaking by the 14th day of soaking. Only for the very tough and silty shale #7 did the deterio-ration progress so slowly that the chemical reactions started to level off by the 28th day of soaking. The conclusion made concerning the leveling off of the reaction is that with only two data points, 1 day and 14 days, there is no way of knowing whether the reaction was close to leveling off at some time earlier than 14 days. Additional testing to obtain further information would seem to be justified, because of its impor-tance in the development of this type of test.

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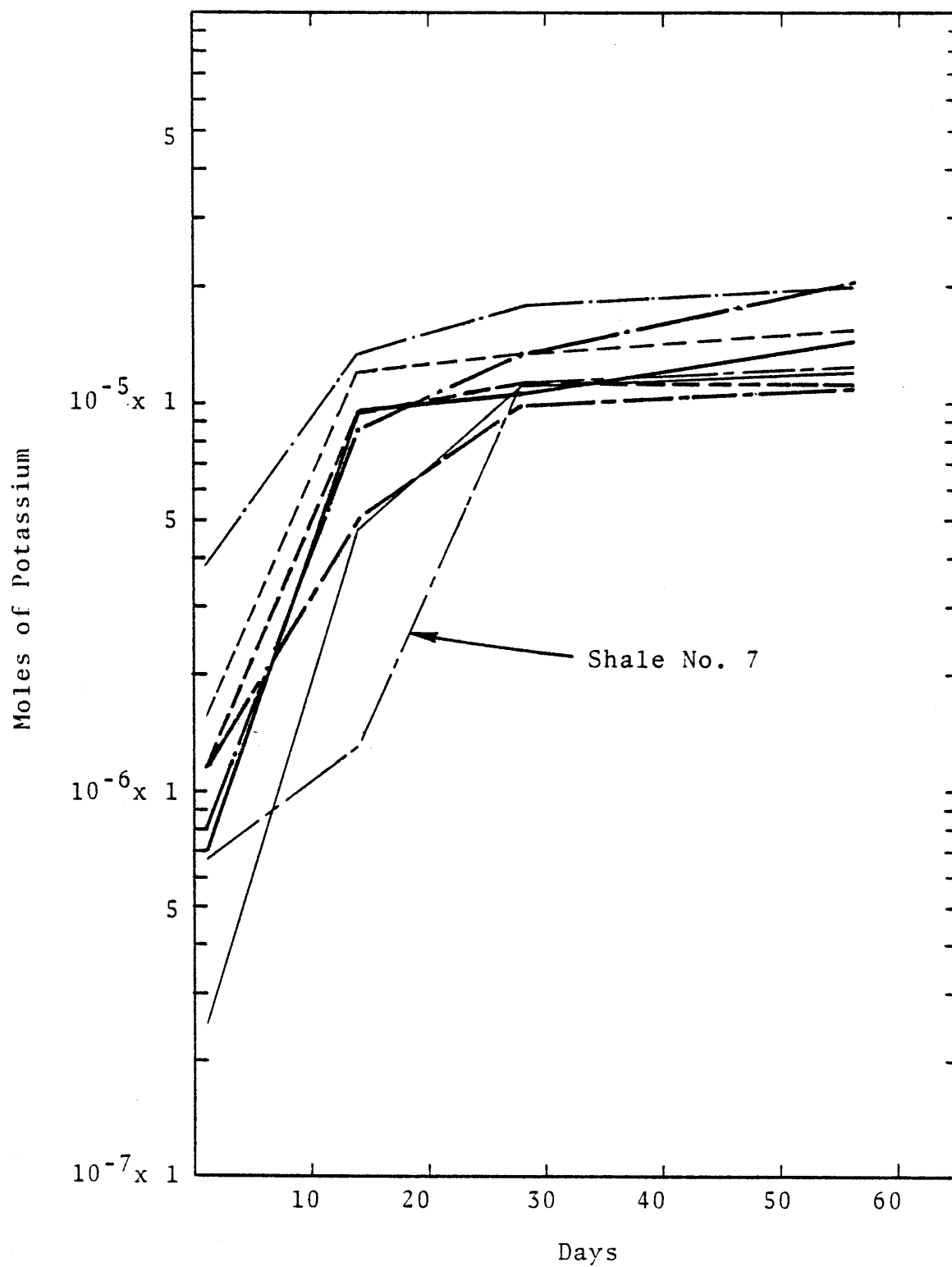
Figure 6. Potassium leached from shales by a 25% solution of concentrated sulfuric acid.
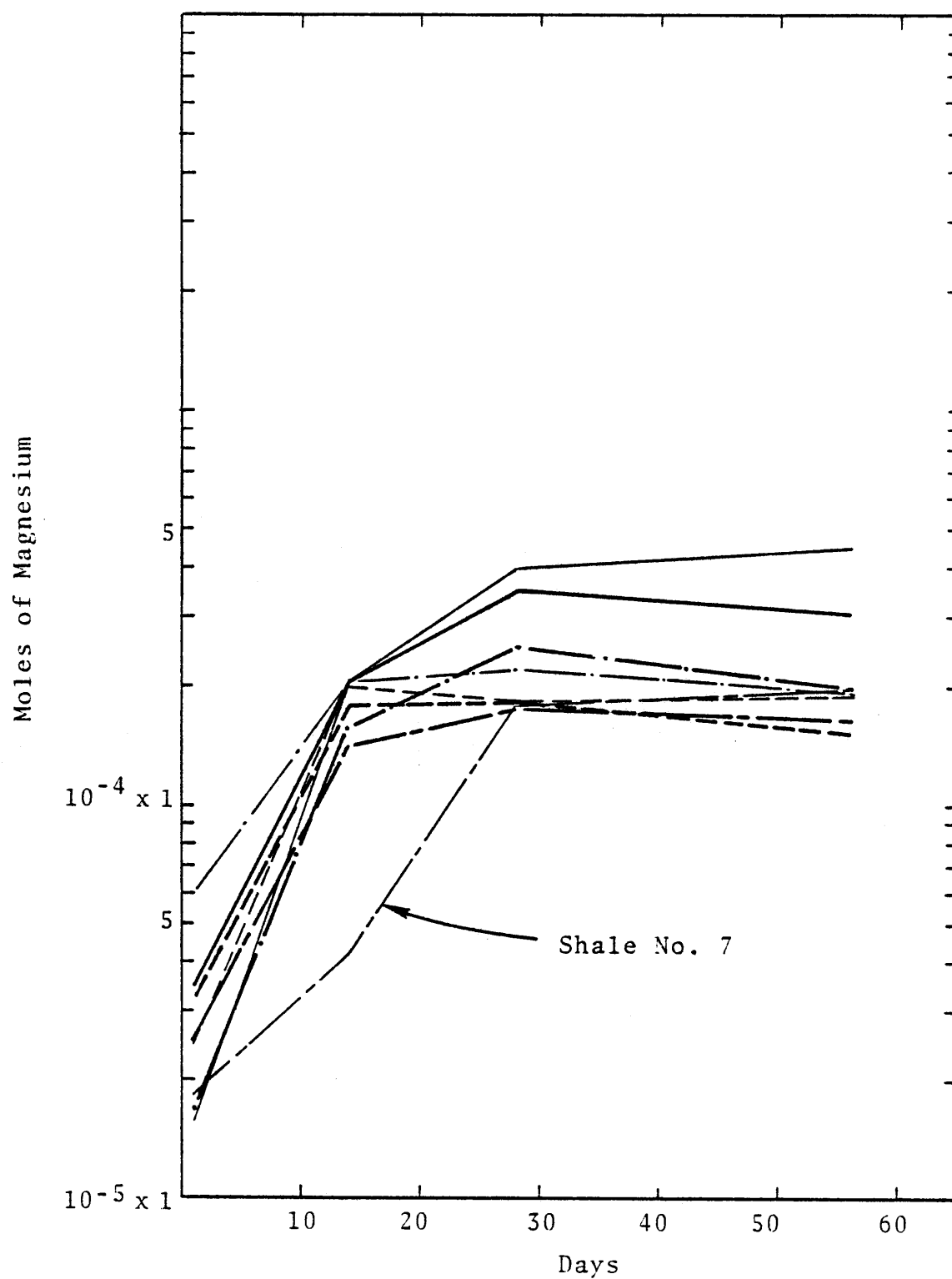
Figure 7.   Magnesium leached from shales by a 25%
            solution of concentrated sulfuric acid.
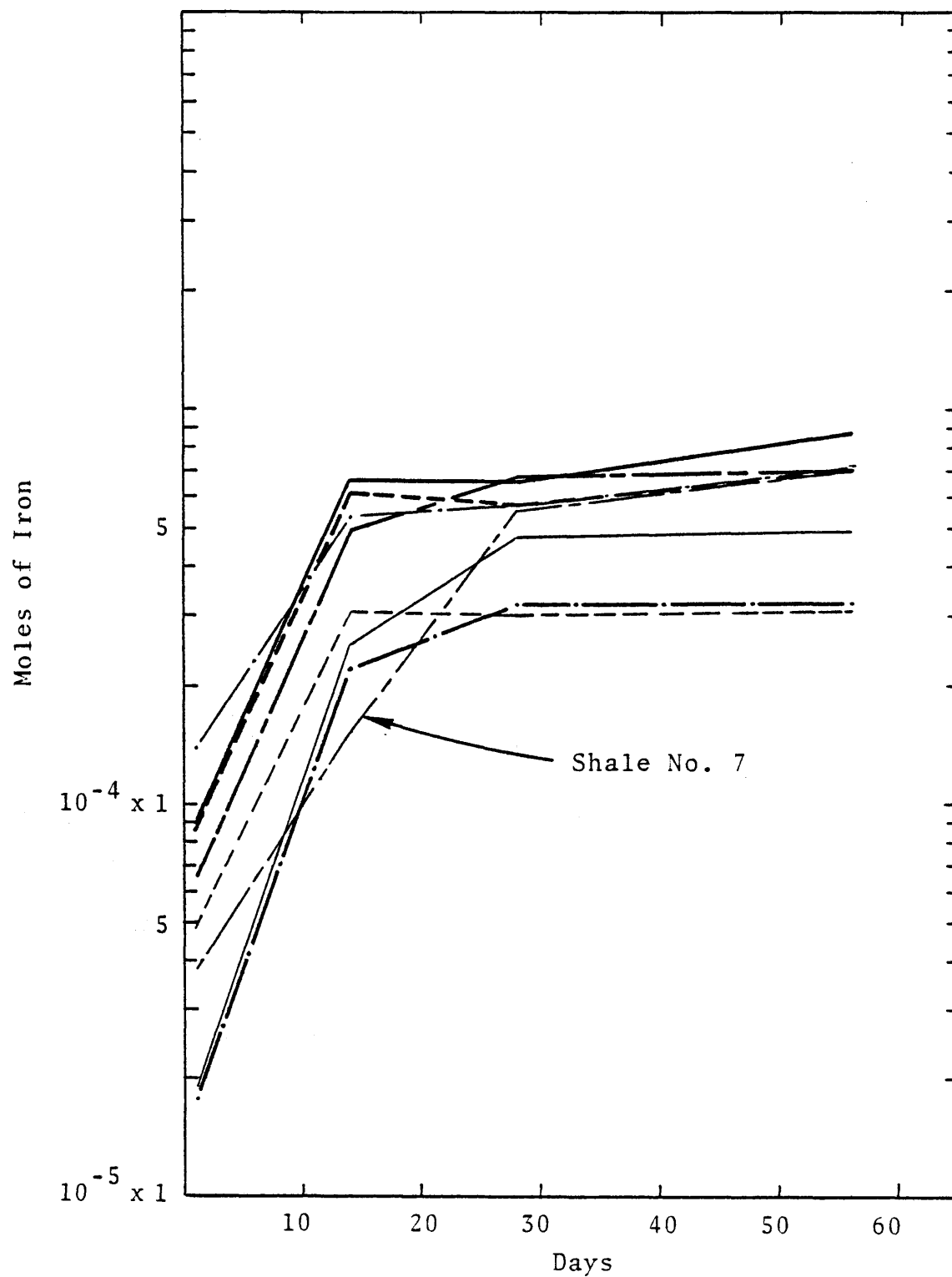
Figure 8.   Iron leached from shales by a 25% solution
of concentrated sulfuric acid.
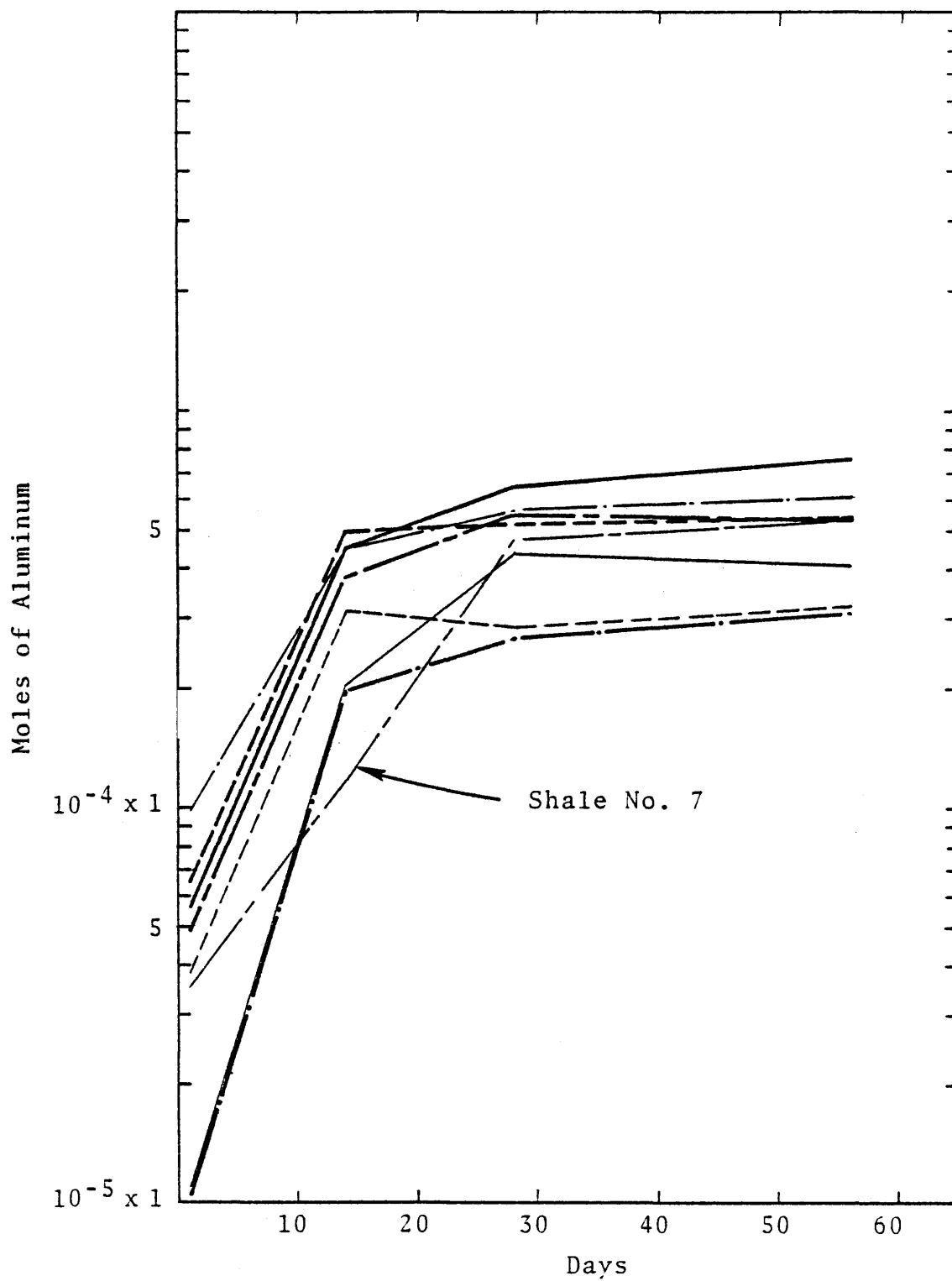
Figure 9. Aluminum leached from shales by a 25%
           solution of concentrated sulfuric acid.

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CONCLUSIONS

1. Interpretation of the quantitative analysis
    of the slake and slake durability tests on
    dry specimens did not distinguish much dif-
    ference between the Virginia shales that were
    tested. However, observation of the change
    in the physical condition of the particles
    suggested that at least shale #3 should be
    considered to be soil-like because of the
    breakage and cracking of the original par-
    ticles.

2. The modified sulfate soundness test did a
    good job of differentiating between the Vir-
    ginia shales, which seem to fall into the
    following numerical grouping.

Percent	Shales	Visual Assessment of Shale As Sampled
+98		
80-98	7,1	Did not seem soil-like
60-80	4,5	Did not seem soil-like
40-60	2,6,8	Probably soil-like
0-40	3	Obviously soil-like

    Based on the limited number of shales tested,
    the modified sulfate soundness test would be
    useful in the testing and classification of
    Virginia shales, because the same physical
    properties of the shales that control the
    access of the sulfate to the interior of the
    particles also control the access of nature's
    weathering agents — water and air — to the
    interior of the particles.

3. The sulfuric acid test made with a 25% solution
    is a very rigorous one.

4. The chlorite in the shales was very susceptible
    to dissolution by the 25% solution of concen-
    trated sulfuric acid.

5. The rate at which the chlorite is dissolved
    is strongly dependent on the physical prop-
    erties of the shale which control the access
    of the acid to the chlorite.

6. The order of responsiveness of the shales as
    determined by observation of the acid treated
    shales compared favorably with the order of
    responsiveness as determined by the modified
    sulfate soundness test, thus lending validity

to the sulfuric acid test as a technique
for use in the classification of shales.

7. The 25% solution of sulfuric acid attacked
    the shales more vigorously than the 50% and
    75% concentrations, but no data were
    obtained to determine whether solutions
    slightly stronger or weaker would make a
    more effective test.

8. None of the shales that were tested were
    rock-like.

RECOMMENDATIONS

1. Because the iron sulfide frequently found
    in dark colored shales will produce sul-
    furic acid that will vigorously attack and
    dissolve chlorite, the following procedures
    are recommended.

    a. When dark colored shales are encountered
        in cuts and fills, they should be
        checked for iron sulfide.

    b. If iron sulfide is present, the clay
        minerals should be determined.

    c. If chlorite is present, the shale
        should be considered as having great
        potential for relatively rapid
        weathering.

2. Because shales that are mineralogically sus-
    ceptible to relatively rapid weathering cannot
    always be wasted and thus excluded from use in
    embankments, it is recommended that methods of
    construction and treatment that might impede
    the rapid weathering of these shales should be
    planned, then used in the construction of an
    embankment, and, finally, the effectiveness of
    the methods used to impede rapid weathering
    should be monitored.

3. The variability that was determined in apparently
    equally tough shales suggests the need for a
    classification system for Virginia shales. Thus
    it is recommended that classification systems

such as those used by the Indiana State High-
way Commission and the Pennsylvania Department
of Transportation be tested on a broad repre-
sentation of Virginia shales. Depending on the
ability of these systems to classify Virginia
shales, one should be adopted as is or modified
for use in Virginia.

4. The sulfuric acid test has demonstrated its
   potential for differentiating between the
   shales that were tested; however, it needs
   to be refined. Thus it is recommended that
   the sulfuric acid test be run on a large, repre-
   sentative group of Virginia shales using
   several dilute concentrations of acid and in
   such a manner as to allow the statistical
   variability of the test to be determined.

ACKNOWLEDGEMENTS

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REFERENCES

American Society for Testing and Materials, Annual Book of ASTM Standards, Part 14, Concrete and Mineral Aggregates, C88-73.

Bailey, M. J., 1976, "Degradation and Other Parameters Related to the Use of Shale in Compacted Embankments", JHRP No. 76-23, Purdue University, West Lafayette, Indiana, 230 pp.

Bragg, G. H., Jr., and T. W. Zeigler, 1975, "Design and Construction of Compacted Shale Embankments— Volume 2, Evaluation and Remedial Treatment of Compacted Shale Embankments", Rpt. No. FHWA-RD-75-62, Federal Highway Administration, Washington, D. C., 235 pp.

Chapman, D. R., 1975, "Shale Classification Tests and Systems: A Comparative Study", Purdue University, West Lafayette, Indiana, 90 pp.

Chapman, D. R., L. E. Wood, C. W. Lovell, and W. J. Sisiliano, 1976, "A Comparative Study of Shale Classification Tests and Systems", JHRP-76-10, Purdue University, Indiana State Highway Commission, West Lafayette, Indiana, 27 pp.

Franklin, J. A., and R. Chandra, 1972, "The Slake-Durability Test", International Journal of Rock Mechanics and Mining Sciences, Vol. 9, Pergamon Press, Great Britain, pp. 325-341.

Hodgman, C. D., 1959, Editor in Chief, Handbook of Chemistry and Physics, Chemical Rubber Publishing Co., Cleveland, Ohio, 3,472 pp.

Howell, J. V., Coordinating Chairman, 1957, Glossary of Geology and Related Sciences, American Geological Institute, Washington, D. C., 397 pp.

Lutton, R. J., 1977, "Design and Construction of Compacted Shale Embankments — Volume 3, Slaking Indexes for Design", Report No. FHWA-RD-77-1, Federal Highway Administration, Washington, D. C., 88 pp.

Morgenstern, N. R., and K. D. Eigenbrod, 1974, "Classification of Argillaceous Soils and Rocks", Journal of the Geotechnical Engineering Division, ASCE, Vol. 100, No. GT10, pp. 1137-1156.

Noble, D. F., 1976, "Identifying Shales That Weather Rapidly When Disturbed by Construction" Working Plan, Virginia Highway and Transportation Research Council, VHTRC-76-WP23, Charlottesville, Virginia, 9 pp.

Reidenouer, D. R., E. G. Geiger, Jr., and R. H. Howe, 1974, "Shale Suitability — Phase II, Report No. 2", Pennsylvania Department of Transportation, Harrisburg, Pa., 160 pp.

Shamburger, J. H., D. M. Patrick, and R. J. Lutton, 1975, "Design and Construction of Compacted Shale Embankments — Volume 1, Survey of Problems Areas and Current Practices", Report No. FHWA-RD-75-61, Federal Highway Administration, Washington, D. C., 292 pp.

Smith, R. W., D. R. Reidenouer, and F. G. Dishong, 1967, "Durability of Shales As Determined By Laboratory Tests, Shale Suitability — Phase I", Pennsylvania Department of Transportation, Harrisburg, Pa., 80 pp.

Underwood, L. B., 1967, "Classification and Identification of Shales", Journal of the Soil Mechanics and Foundations Division, ASCE, Vol. 93, No. SM6, pp. 97-116.

Virginia Department of Highways, 1966, Road and Bridge Specifications, E. Waddey Co., Richmond, Virginia, 547 pp.

Weaver, C. E., 1958, "Geologic Interpretation of Argillaceous Sediments, Part I — Origin and Significance of Clay Minerals In Sedimentary Rocks", Bulletin of the American Association of Petroleum Geologists, Vol. 42, No. 2, pp. 254-271.

Wood, L. E., C. W. Lovell, and P. Deo, 1973, "Building Embankments with Shales", JHRP No. C-36-5J, Purdue University, West Lafayette, Indiana, 32 pp.

Appendix A

Test Procedures

SLAKE TEST

a. Choose 6 chunks of shale — each weighing approximately 150 grams. Rock should be free of dust and loose particles; brush lightly, do not wet. If no 150-gram chunks are available, use the largest pieces available and get close to a total weight of 150 grams ($\pm$ 10 grams).

b. Have specimens photographed with identifying number (I-64 sta. 633) and millimeter scale.

c. Dry shale to a constant weight at about 105°C, and record dry weight.

d. Place each chunk in a separate jar and cover with distilled water by 0.5 inch (12.7 mm); allow to soak for 24 hours. Check pH of a daily supply of water before using. The condition of the specimen should be checked for the first 10 minutes, then at 1, 2, 4 or 8, and 24 hours.

e. Remove specimen from water and run pH of the water.

f. If necessary, sieve rock on No. 10 screen. Using the No. 10 screen at this point is desirable so that this test can be compared with Franklin slake durability test.

g. Dry to constant weight, and record weight.

h. Repeat procedure 4 times (make 5 cycles).*

i. Calculate the "Slake Index" for each of the 6 specimens and take the average.

$$SI = \frac{W_o - W_f}{W_o} \quad \frac{(\text{Original Wt.} - \text{Final Wt.})}{\text{Original Wt.}} \times 100$$

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*Note: Depending on how fast the specimen slakes, take additional photographs, if very fast — photograph as it slakes, if very slow — photograph after 5th cycle.

SLAKE DURABILITY TEST

Comment: Rocks that fall apart in the slake test need not be put through this test.

a. Choose 10 chunks of shale (40-60 grams each) with a total weight of approximately 500 grams. Rock should be free of dust and loose particles; brush gently, do not wet.

b. Have specimen photographed with identifying number (I-64 sta. 633) and millimeter scale.

c. Dry shale to a constant weight at about 105°C, and record dry weight.

d. Weigh the drum from the test equipment.

e. With the shale in the drum, weigh the drum and the dry shale.

f. Rotate the wire mesh drum with the axis of the drum 20 mm above the level of the water for 10 minutes at 20 rpm.

g. Remove the drum from the water, rinse, dry, and weigh drum and remaining shale.

h. Repeat the cycle 4 times, but calculate the durability index (I_d) after each cycle.

Photograph the shale at least <u>before</u> the 1st cycle and after the 5th cycle; photograph after the other cycles only if the appearance of the shale is quite different.

i. Calculate the durability index as follows:

$$I_{dn} = \frac{\text{Wt. of shale remaining in drum}}{\text{Original wt. of shale}} \times 100$$

(n = number of cycles)

j. Run at least 2 specimens from each sample of shale and take the average of their durability indices.

| Rock - | | Route - | | Station No. - | | Date - |

Specimen	1	2	3	4	5	6
Original Wt.						
Wt. after 1st. cycle						
Wt. after 2nd. cycle						
Wt. after 3rd. cycle						
Wt. after 4th. cycle						
Wt. after 5th. cycle						
Total Diff. in Wt.						
Specimen Slake Index						

Slake index average -

Comments -

Slake Durability Index

| Rock - | | Route - | | Station No.- | | Date - |

Specimen	1	Id	2	Id	3	Id
Original Wt.						
Wt. after 1st. cycle						
Wt. after 2nd. cycle						
Wt. after 3rd. cycle						
Wt. after 4th. cycle						
Wt. after 5th. cycle						
Total Diff. in Wt.						

Durability index average

Comments -

MODIFIED SODIUM SULFATE SOUNDNESS TEST

Comment: Shale that is greatly distressed by the slake and slake durability tests should not be subjected to this test.

a. Prepare a 50% solution of sodium sulfate by making a saturated solution; mix equal quantities of saturated solution and distilled water.

b. If possible, use noncorrosive containers for test.

c. To prepare test specimens, sieve approximately 2,050 grams of 3/4 to 1/2 inch* rock and 1,020 grams of 1/2 to 3/8 inch rock (enough for 3 specimens).

d. Wash all dust and loose material from rock.

e. Thoroughly dry the rock at 110 ± 5°C. Have specimen photographed, each fraction.

f. Weigh approximately 670 grams of 3/4 to 1/2 inch rock and approximately 330 grams of 1/2 to 3/8 inch rock. Mix portions for 1 specimen; prepare 3 specimens. Record total weight of each specimen and weight of each size fraction ($1/2" < \chi < 3/4"$ and $3/8" < \chi' < 1/2"$).

g. Consult ASTM, part 14, C88-73, page 50, paragraph 7, for immersion procedures.

  1. Immerse specimen for 16-18 hours by at least 1/2 inch at room temperature.

     Note: It is a good idea to keep solution at planned concentration. Before start of immersion, check solution with hydrometer. Use hydrometer reading to keep solution close to proper concentration.

  2. After soaking, drain for 15-20 minutes; place in oven at 110 ± 5°C until dry.

  3. Observe condition of dry specimen and note. Repeat cycle. Depending on condition of specimen, run at least 5 cycles; if relatively unaffected, run 5 additional cycles, or try a saturated solution of sodium sulfate for 5 cycles. Photograph specimen when advantageous.

h. Consult ASTM, part 14, C88-73, page 51, paragraphs 8 and 9, for examination procedures.

*1 inch equals 25.4 mm.

A-5

1. After specimen has cooled, wash it free of sodium sulfate by flushing with hot (43 $\pm$ 6°C) tap water. Check for SO_4 in the rinse water with barium chloride ($BaCl_2$) solution. If SO_4 still present, white precipitate will form.

2. When free of SO_4, dry the specimen at 110 $\pm$ 5°C.

3. Weigh and record total weight of remaining specimen.

4. Seive through a 1/2-inch screen; and what passes the 1/2, through a 5/16-inch screen.

5. Weigh the 2 fractions from the 2 screens and record.

6. Have the 2 fractions photographed.

7. Calculate the soundness index, I_s, as follows:

$$I_s = \frac{W_f \times 100}{W_o} \quad \left(\frac{\text{Final Wt.} \times 100}{\text{Original Wt.}} \right)$$

Sulfate Soundness Test

Rock -	Route -	Station No.		Date -
Sulfate used -	Solution Concentration -			
Specimen	1	2		3
Before - Wt. 1/2"< χ <3/4"				
After - Wt. 1/2"< χ <3/4"				
Difference -				
Before - Wt. 3/8"< χ'<1/2"				
After - Wt. 3/8"< χ'<1/2"				
Difference -				
Before - Total Wt.				
After - Total Wt.				
Difference -				
No. of cycles run				
Comments:				

SULFURIC ACID DETERIORATION TEST

a. Choose pieces of shale that weigh 10-15 grams each. Accumulate as many pieces as needed for number of concentrations of acid (4), times number of time intervals (4) at which leachate is to be analyzed for dissolved cations.

b. Rinse particles of shale with distilled water to remove all dust.

c. Dry in oven at 105°C to a constant weight — approximately 2 to 4 hours or overnight — weigh and record the weight of each particle.

d. Take 75 ml of solutions (0%, 25%, 50%, 75%) of concentrated sulfuric acid (18 M); place in appropriate container (100 ml beaker) and submerge one piece of shale per container and concentration of solution.

e. Cover container to prevent evaporation of H_2O or contamination from without.

f. Observe condition of shale; judgement must be used in scheduling observations. Observe for first hour in case of rapid reaction such as dissolution of calcium carbonate.

g. Thereafter observe daily, noting significant changes in condition of shale or leachate, such as cracks, decrepitation, or change in color.

h. Begin elemental analysis of leachate when it appears that reaction between acid and shale has started, or such analyses shall be made at arbitrarily chosen times of 1, 14, 28, and 56 days. Tests shall be made using standard flame atomic absorption techniques for K^+, Ca^{+2}, Mg^{+2}, Fe, and Al^{+3}. Avoid disturbing particle of shale as much as possible and remove leachate so as to avoid including any flakes of clay with leachate. If particulate matter abounds and is difficult to avoid, agitate material to remove as much fine particulate matter as possible with the leachate and separate two by filtration. Save particulate matter for X-ray diffraction analysis.

i. After removal of leachate for chemical analysis, carefully wash sulfate from remaining rock or sediment.

j. Dry, powder, and prepare rock for X-ray diffraction so as to determine what changes in mineralogy have occurred.

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Appendix B

Results of Chemical Analyses

TABLE A

Cations Leached From Shale #1 By Sulfuric Acid

Cations	Days	Percent Acid			
		0	25	50	75
K	1	5.60×10^{-8}	2.51×10^{-7}	1.67×10^{-7}	1.89×10^{-7}
	14	3.38×10^{-7}	4.71×10^{-6}	4.02×10^{-7}	4.91×10^{-8}
	28	3.35×10^{-7}	1.10×10^{-5}	8.65×10^{-7}	7.55×10^{-8}
	56	6.27×10^{-7}	1.21×10^{-5}	2.02×10^{-6}	1.42×10^{-7}
Ca	1	2.41×10^{-6}	3.56×10^{-6}	1.54×10^{-6}	7.19×10^{-6}
	14	2.52×10^{-6}	1.03×10^{-6}	1.18×10^{-6}	1.48×10^{-5}
	28	3.33×10^{-6}	9.68×10^{-7}	1.25×10^{-6}	2.65×10^{-5}
	56	5.02×10^{-6}	5.22×10^{-7}	5.89×10^{-7}	2.99×10^{-5}
Mg	1	3.96×10^{-7}	1.56×10^{-5}	4.56×10^{-6}	1.61×10^{-6}
	14	8.84×10^{-7}	2.08×10^{-4}	1.77×10^{-5}	5.06×10^{-5}
	28	1.25×10^{-6}	3.99×10^{-4}	2.83×10^{-5}	8.03×10^{-5}
	56	2.01×10^{-6}	4.52×10^{-4}	4.36×10^{-5}	9.87×10^{-6}
Fe	1	7.83×10^{-9}	1.91×10^{-5}	5.37×10^{-6}	1.59×10^{-6}
	14	–	2.54×10^{-4}	1.19×10^{-5}	4.66×10^{-6}
	28	–	4.78×10^{-4}	1.83×10^{-5}	6.55×10^{-6}
	56	–	4.98×10^{-4}	2.04×10^{-5}	5.96×10^{-6}
Al	1	–	1.11×10^{-5}	4.82×10^{-6}	1.37×10^{-6}
	14	–	2.04×10^{-4}	2.47×10^{-5}	5.12×10^{-6}
	28	–	4.41×10^{-4}	3.86×10^{-5}	8.75×10^{-6}
	56	–	4.08×10^{-4}	5.56×10^{-5}	1.20×10^{-5}

Note: Data expressed in moles of cation normalized by dividing percent cation (by weight of shale) by atomic weight of element so values can be compared one specimen to another.

TABLE B

Cations Leached From Shale #2 By Sulfuric Acid

Cations	Days	Percent Acid			
		0	25	50	75
K	1	$4.12X10^{-8}$	$6.96X10^{-7}$	$1.96X10^{-7}$	$8.80X10^{-8}$
	14	$8.36X10^{-8}$	$9.54X10^{-6}$	$1.66X10^{-6}$	$2.99X10^{-8}$
	28	$1.33X10^{-7}$	$1.05X10^{-5}$	$2.38X10^{-6}$	$6.57X10^{-8}$
	56	$1.05X10^{-7}$	$1.43X10^{-5}$	$4.83X10^{-6}$	$1.34X10^{-7}$
Ca	1	$5.02X10^{-8}$	$6.26X10^{-7}$	$3.57X10^{-7}$	-
	14	$3.99X10^{-9}$	$4.02X10^{-7}$	$5.61X10^{-7}$	$2.79X10^{-7}$
	28	$4.32X10^{-8}$	$2.87X10^{-7}$	$5.76X10^{-7}$	$1.92X10^{-7}$
	56	$1.23X10^{-7}$	$2.32X10^{-7}$	$1.79X10^{-7}$	$2.50X10^{-7}$
Mg	1	$8.27X10^{-8}$	$3.44X10^{-5}$	$5.26X10^{-6}$	$3.67X10^{-7}$
	14	$1.57X10^{-7}$	$2.06X10^{-4}$	$3.55X10^{-5}$	$3.32X10^{-5}$
	28	$1.42X10^{-7}$	$3.49X10^{-4}$	$4.08X10^{-5}$	$5.06X10^{-5}$
	56	$2.86X10^{-7}$	$3.06X10^{-4}$	$5.14X10^{-5}$	$3.80X10^{-5}$
Fe	1	$7.20X10^{-9}$	$8.99X10^{-5}$	$1.10X10^{-5}$	$5.53X10^{-6}$
	14	$9.67X10^{-9}$	$6.64X10^{-4}$	$1.59X10^{-5}$	$4.64X10^{-6}$
	28	-	$6.61X10^{-4}$	$2.33X10^{-5}$	$6.63X10^{-6}$
	56	$3.58X10^{-9}$	$8.74X10^{-4}$	$2.60X10^{-5}$	$3.46X10^{-6}$
Al	1	-	$5.60X10^{-5}$	$1.04X10^{-5}$	$1.78X10^{-6}$
	14	-	$4.49X10^{-4}$	$6.08X10^{-5}$	$9.08X10^{-6}$
	28	-	$6.45X10^{-4}$	$6.75X10^{-5}$	$1.14X10^{-5}$
	56	-	$7.60X10^{-4}$	$1.01X10^{-4}$	$1.07X10^{-5}$

Note: Data expressed in moles of cation normalized by dividing percent cation (by weight of shale) by atomic weight of element so values can be compared one specimen to another.

TABLE C

Cations Leached From Shale #3 By Sulfuric Acid

Cations	Days	Percent Acid			
		0	25	50	75
K	1	7.88×10^{-8}	3.84×10^{-6}	7.32×10^{-7}	1.86×10^{-7}
	14	1.52×10^{-7}	1.34×10^{-5}	4.71×10^{-6}	1.46×10^{-7}
	28	2.00×10^{-7}	1.78×10^{-5}	1.29×10^{-5}	3.94×10^{-7}
	56	2.09×10^{-7}	1.99×10^{-5}	1.45×10^{-5}	4.09×10^{-7}
Ca	1	9.48×10^{-9}	8.51×10^{-7}	5.24×10^{-7}	7.26×10^{-8}
	14	$-$	2.17×10^{-7}	4.59×10^{-7}	1.71×10^{-7}
	28	9.73×10^{-9}	2.57×10^{-7}	3.92×10^{-7}	3.07×10^{-7}
	56	7.24×10^{-9}	1.94×10^{-7}	2.02×10^{-7}	2.67×10^{-7}
Mg	1	1.56×10^{-8}	5.96×10^{-5}	1.83×10^{-5}	2.59×10^{-6}
	14	9.87×10^{-9}	2.06×10^{-4}	5.22×10^{-5}	4.81×10^{-5}
	28	1.77×10^{-8}	2.21×10^{-4}	7.40×10^{-5}	9.50×10^{-5}
	56	1.19×10^{-8}	1.92×10^{-4}	6.33×10^{-5}	7.98×10^{-6}
Fe	1	6.80×10^{-9}	1.39×10^{-4}	2.45×10^{-5}	4.60×10^{-6}
	14	1.07×10^{-8}	5.34×10^{-4}	3.01×10^{-5}	7.48×10^{-6}
	28	$-$	5.73×10^{-4}	3.22×10^{-5}	1.56×10^{-5}
	56	$-$	7.18×10^{-4}	3.01×10^{-5}	7.36×10^{-6}
Al	1	$-$	9.67×10^{-5}	3.06×10^{-5}	8.08×10^{-6}
	14	$-$	4.52×10^{-4}	1.06×10^{-4}	1.38×10^{-5}
	28	$-$	5.63×10^{-4}	1.47×10^{-4}	2.48×10^{-5}
	56	$-$	6.08×10^{-4}	1.41×10^{-4}	2.05×10^{-5}

Note: Data expressed in moles of cation normalized by dividing percent cation (by weight of shale) by atomic weight of element so values can be compared one specimen to another.

TABLE D

Cations Leached From Shale #4 By Sulfuric Acid

Cations	Days	Percent Acid			
		0	25	50	75
K	1	3.33×10^{-8}	7.95×10^{-7}	2.74×10^{-7}	1.25×10^{-7}
	14	2.52×10^{-7}	8.49×10^{-6}	1.04×10^{-6}	5.37×10^{-8}
	28	4.07×10^{-7}	1.34×10^{-5}	1.21×10^{-6}	6.09×10^{-8}
	56	4.81×10^{-7}	2.01×10^{-5}	5.63×10^{-6}	1.78×10^{-7}
Ca	1	6.66×10^{-7}	1.65×10^{-6}	8.31×10^{-7}	2.20×10^{-6}
	14	4.14×10^{-6}	9.96×10^{-7}	9.13×10^{-7}	1.26×10^{-6}
	28	5.69×10^{-6}	7.04×10^{-7}	9.28×10^{-7}	6.04×10^{-7}
	56	9.03×10^{-6}	6.29×10^{-7}	9.61×10^{-7}	1.40×10^{-6}
Mg	1	1.07×10^{-6}	1.68×10^{-5}	3.53×10^{-6}	4.44×10^{-7}
	14	9.01×10^{-7}	1.58×10^{-4}	1.36×10^{-5}	1.99×10^{-5}
	28	1.55×10^{-6}	2.53×10^{-4}	1.70×10^{-5}	2.44×10^{-5}
	56	2.33×10^{-6}	1.96×10^{-4}	5.18×10^{-5}	2.87×10^{-6}
Fe	1	1.17×10^{-8}	1.78×10^{-5}	3.56×10^{-6}	1.05×10^{-6}
	14	6.80×10^{-9}	2.20×10^{-4}	1.18×10^{-5}	4.37×10^{-6}
	28	9.49×10^{-9}	3.21×10^{-4}	9.71×10^{-6}	3.31×10^{-6}
	56	-	3.26×10^{-4}	3.69×10^{-5}	4.23×10^{-6}
Al	1	-	1.04×10^{-5}	3.97×10^{-6}	9.08×10^{-7}
	14	-	1.99×10^{-4}	1.70×10^{-5}	4.04×10^{-6}
	28	-	2.69×10^{-4}	1.92×10^{-5}	4.41×10^{-6}
	56	-	3.11×10^{-4}	9.79×10^{-5}	4.60×10^{-6}

Note: Data expressed in moles of cation normalized by dividing percent cation (by weight of shale) by atomic weight of element so values can be compared one specimen to another.

TABLE E

Cations Leached from Shale #5 By Sulfuric Acid

Cations	Days	Percent Acid			
		0	25	50	75
K	1	7.55×10^{-8}	1.55×10^{-6}	2.89×10^{-7}	2.19×10^{-7}
	14	4.25×10^{-7}	1.20×10^{-5}	4.48×10^{-6}	1.62×10^{-7}
	28	2.49×10^{-7}	1.33×10^{-5}	5.19×10^{-6}	2.39×10^{-7}
	56	5.47×10^{-7}	1.53×10^{-5}	1.58×10^{-5}	4.60×10^{-7}
Ca	1	1.47×10^{-6}	9.21×10^{-7}	3.62×10^{-7}	2.99×10^{-7}
	14	8.28×10^{-6}	5.36×10^{-7}	4.92×10^{-7}	1.11×10^{-6}
	28	6.31×10^{-6}	3.62×10^{-7}	5.76×10^{-7}	8.68×10^{-7}
	56	9.28×10^{-6}	3.44×10^{-7}	3.07×10^{-7}	2.15×10^{-6}
Mg	1	8.88×10^{-7}	2.48×10^{-5}	3.48×10^{-6}	8.64×10^{-7}
	14	6.37×10^{-6}	2.00×10^{-4}	3.36×10^{-5}	4.56×10^{-5}
	28	5.43×10^{-6}	1.86×10^{-4}	3.83×10^{-5}	5.02×10^{-5}
	56	7.11×10^{-6}	1.89×10^{-4}	7.61×10^{-5}	9.62×10^{-6}
Fe	1	1.10×10^{-6}	4.83×10^{-5}	5.41×10^{-6}	1.38×10^{-6}
	14	1.09×10^{-5}	3.06×10^{-4}	1.77×10^{-5}	5.91×10^{-6}
	28	8.72×10^{-6}	3.01×10^{-4}	2.02×10^{-5}	7.38×10^{-6}
	56	1.05×10^{-5}	3.10×10^{-4}	3.20×10^{-5}	9.85×10^{-6}
Al	1	2.37×10^{-7}	3.78×10^{-5}	5.97×10^{-6}	2.38×10^{-6}
	14	1.87×10^{-6}	3.13×10^{-4}	5.63×10^{-5}	9.41×10^{-6}
	28	1.45×10^{-6}	2.85×10^{-4}	7.23×10^{-5}	9.97×10^{-6}
	56	1.74×10^{-6}	3.24×10^{-4}	1.70×10^{-4}	1.58×10^{-5}

Note: Data expressed in moles of cation normalized by dividing percent cation (by weight of shale) by atomic weight of element so values can be compared one specimen to another.

TABLE F

Cations Leached From Shale #6 By Sulfuric Acid

Cations	Days	Percent Acid			
		0	25	50	75
K	1	2.66×10^{-8}	1.14×10^{-6}	2.89×10^{-7}	1.52×10^{-7}
	14	6.42×10^{-8}	9.39×10^{-6}	1.98×10^{-6}	5.93×10^{-8}
	28	8.26×10^{-8}	1.12×10^{-5}	3.43×10^{-6}	1.25×10^{-7}
	56	8.72×10^{-8}	1.12×10^{-5}	3.76×10^{-6}	1.90×10^{-7}
Ca	1	2.60×10^{-8}	9.58×10^{-7}	3.82×10^{-7}	1.92×10^{-7}
	14	2.50×10^{-8}	4.59×10^{-7}	6.96×10^{-7}	2.89×10^{-7}
	28	2.02×10^{-8}	2.57×10^{-7}	9.83×10^{-7}	3.04×10^{-7}
	56	2.12×10^{-8}	1.31×10^{-7}	5.12×10^{-7}	1.96×10^{-7}
Mg	1	4.28×10^{-8}	3.21×10^{-5}	6.09×10^{-6}	5.84×10^{-7}
	14	6.42×10^{-8}	1.79×10^{-4}	3.30×10^{-5}	2.68×10^{-5}
	28	5.96×10^{-8}	1.85×10^{-4}	4.81×10^{-5}	2.82×10^{-5}
	56	4.69×10^{-8}	1.51×10^{-4}	6.50×10^{-5}	3.05×10^{-6}
Fe	1	9.31×10^{-9}	8.79×10^{-5}	1.46×10^{-5}	3.81×10^{-6}
	14	8.95×10^{-9}	6.14×10^{-4}	2.13×10^{-5}	3.99×10^{-6}
	28	–	5.73×10^{-4}	2.69×10^{-5}	3.94×10^{-6}
	56	–	7.11×10^{-4}	3.56×10^{-5}	3.31×10^{-6}
Al	1	–	6.45×10^{-5}	1.10×10^{-5}	3.51×10^{-6}
	14	–	5.00×10^{-4}	6.60×10^{-5}	8.60×10^{-6}
	28	–	5.19×10^{-4}	9.41×10^{-5}	9.08×10^{-6}
	56	–	5.52×10^{-4}	1.26×10^{-4}	1.29×10^{-5}

Note: Data expressed in moles of cation normalized by dividing percent cation (by weight of shale) by atomic weight of element so values can be compared one specimen to another.

TABLE G

Cations Leached From Shale #7 By Sulfuric Acid

Cations	Days	Percent Acid			
		0	25	50	75
K	1	$1.59X10^{-8}$	$6.62X10^{-7}$	$4.25X10^{-7}$	$1.78X10^{-7}$
	14	$2.53X10^{-8}$	$1.31X10^{-6}$	$5.96X10^{-7}$	$7.06X10^{-8}$
	28	$1.43X10^{-8}$	$1.12X10^{-5}$	$2.03X10^{-6}$	$6.22X10^{-8}$
	56	$4.09X10^{-8}$	$1.23X10^{-5}$	$1.85X10^{-6}$	$1.26X10^{-7}$
Ca	1	$4.62X10^{-8}$	$1.18X10^{-6}$	$4.44X10^{-7}$	$2.94X10^{-7}$
	14	$3.69X10^{-8}$	$8.06X10^{-7}$	$6.99X10^{-7}$	$3.17X10^{-7}$
	28	$2.82X10^{-8}$	$3.64X10^{-7}$	$1.16X10^{-6}$	$3.04X10^{-7}$
	56	$4.99X10^{-8}$	$4.04X10^{-7}$	$8.63X10^{-7}$	$2.46X10^{-7}$
Mg	1	$1.02X10^{-7}$	$1.82X10^{-5}$	$3.79X10^{-6}$	$7.69X10^{-7}$
	14	$7.73X10^{-8}$	$4.19X10^{-5}$	$1.45X10^{-5}$	$1.43X10^{-5}$
	28	$9.05X10^{-8}$	$1.80X10^{-4}$	$3.27X10^{-5}$	$1.88X10^{-5}$
	56	$1.48X10^{-7}$	$1.97X10^{-4}$	$2.53X10^{-5}$	$1.52X10^{-6}$
Fe	1	$1.11X10^{-8}$	$3.80X10^{-5}$	$1.71X10^{-5}$	$2.11X10^{-6}$
	14	$8.95X10^{-9}$	$1.51X10^{-4}$	$1.72X10^{-5}$	$3.74X10^{-6}$
	28	-	$5.59X10^{-4}$	$2.54X10^{-5}$	$3.40X10^{-6}$
	56	-	$7.04X10^{-4}$	$2.01X10^{-5}$	$2.51X10^{-6}$
Al	1	-	$3.52X10^{-5}$	$7.97X10^{-6}$	$2.57X10^{-6}$
	14	-	$1.18X10^{-4}$	$3.07X10^{-5}$	$4.08X10^{-6}$
	28	-	$4.74X10^{-4}$	$5.89X10^{-5}$	$5.04X10^{-6}$
	56	-	$5.34X10^{-4}$	$6.60X10^{-5}$	$5.78X10^{-6}$

Note: Data expressed in moles of cation normalized by dividing percent cation (by weight of shale) by atomic weight of element so values can be compared one specimen to another.

TABLE H

Cations Leached From Shale #8 By Sulfuric Acid

Cations	Days	Percent Acid			
		0	25	50	75
K	1	2.46×10^{-8}	1.16×10^{-6}	2.06×10^{-6}	8.54×10^{-8}
	14	2.49×10^{-7}	5.01×10^{-6}	1.24×10^{-6}	6.96×10^{-8}
	28	1.10×10^{-7}	9.85×10^{-6}	2.63×10^{-6}	1.18×10^{-7}
	56	1.49×10^{-7}	1.08×10^{-5}	4.35×10^{-6}	2.40×10^{-7}
Ca	1	1.20×10^{-8}	8.88×10^{-7}	3.02×10^{-7}	1.67×10^{-7}
	14	3.04×10^{-8}	2.08×10^{-7}	8.28×10^{-7}	2.04×10^{-7}
	28	1.20×10^{-8}	2.40×10^{-7}	9.91×10^{-7}	1.72×10^{-7}
	56	7.24×10^{-9}	2.12×10^{-7}	4.24×10^{-7}	2.99×10^{-7}
Mg	1	1.97×10^{-8}	2.51×10^{-5}	4.32×10^{-6}	3.85×10^{-7}
	14	9.25×10^{-8}	1.41×10^{-4}	2.28×10^{-5}	2.35×10^{-5}
	28	4.89×10^{-8}	1.78×10^{-4}	3.78×10^{-5}	3.22×10^{-5}
	56	4.81×10^{-8}	1.67×10^{-4}	6.29×10^{-5}	4.07×10^{-6}
Fe	1	8.59×10^{-9}	6.46×10^{-5}	1.48×10^{-5}	6.11×10^{-6}
	14	1.09×10^{-8}	4.96×10^{-4}	2.42×10^{-5}	6.14×10^{-6}
	28	–	6.77×10^{-4}	2.51×10^{-5}	4.94×10^{-6}
	56	–	7.04×10^{-4}	3.71×10^{-5}	3.64×10^{-6}
Al	1	–	4.89×10^{-5}	1.34×10^{-5}	3.71×10^{-6}
	14	–	3.82×10^{-4}	5.71×10^{-5}	1.11×10^{-5}
	28	–	5.52×10^{-4}	8.19×10^{-5}	1.08×10^{-5}
	56	–	5.41×10^{-4}	1.47×10^{-4}	1.61×10^{-5}

Note: Data expressed in moles of cation normalized by dividing
percent cation (by weight of shale) by atomic weight of
element so values can be compared one specimen to another.

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Appendix C

Contrasting Degrees of Deterioration of Specimens

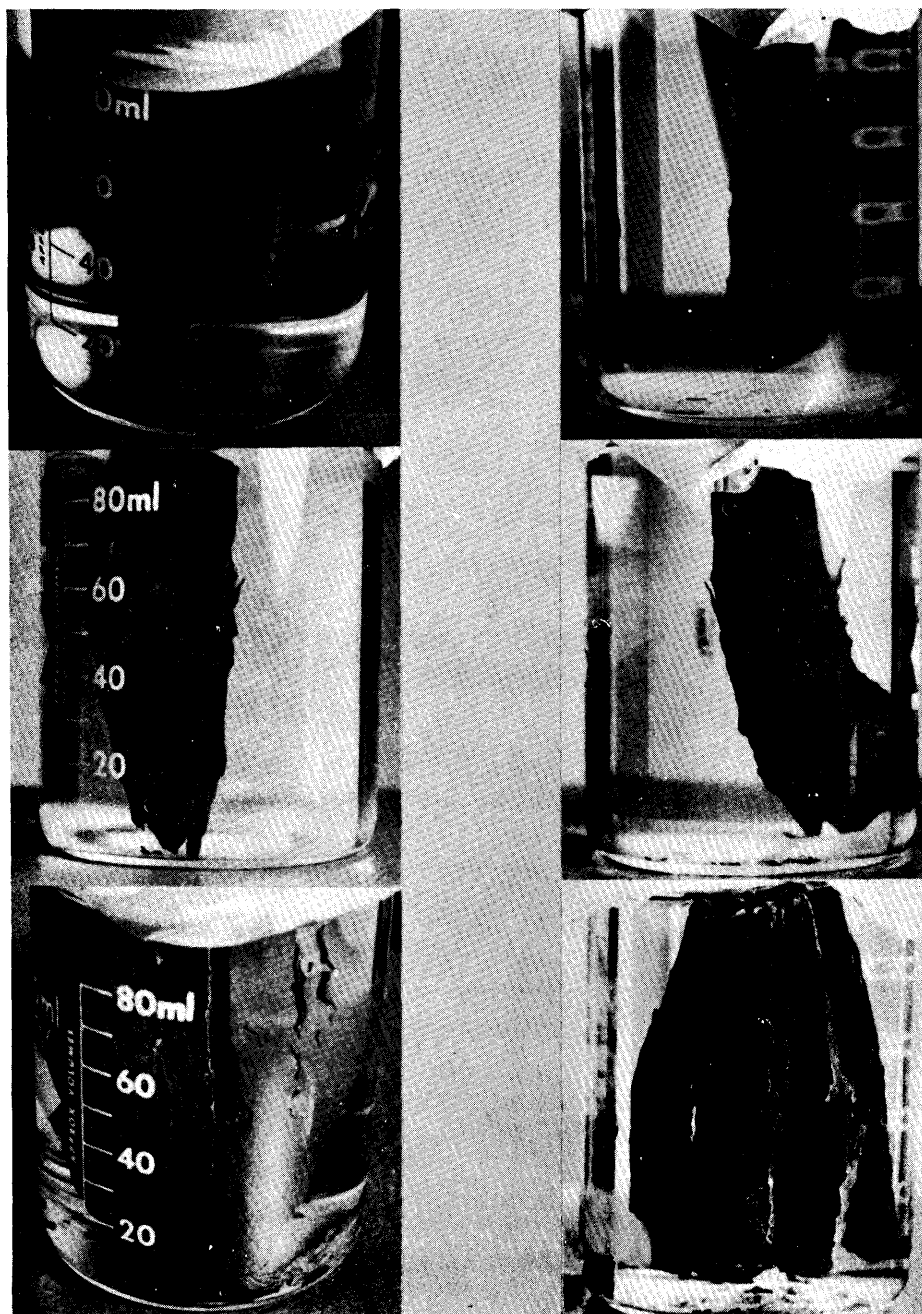Subjected to Sulfuric Acid Test

Figure A. A severely distressed, acid leached particle,
1-P-25, moving down on the left 2, 14, and 55
days, on the right 8, 14, and 55 days.
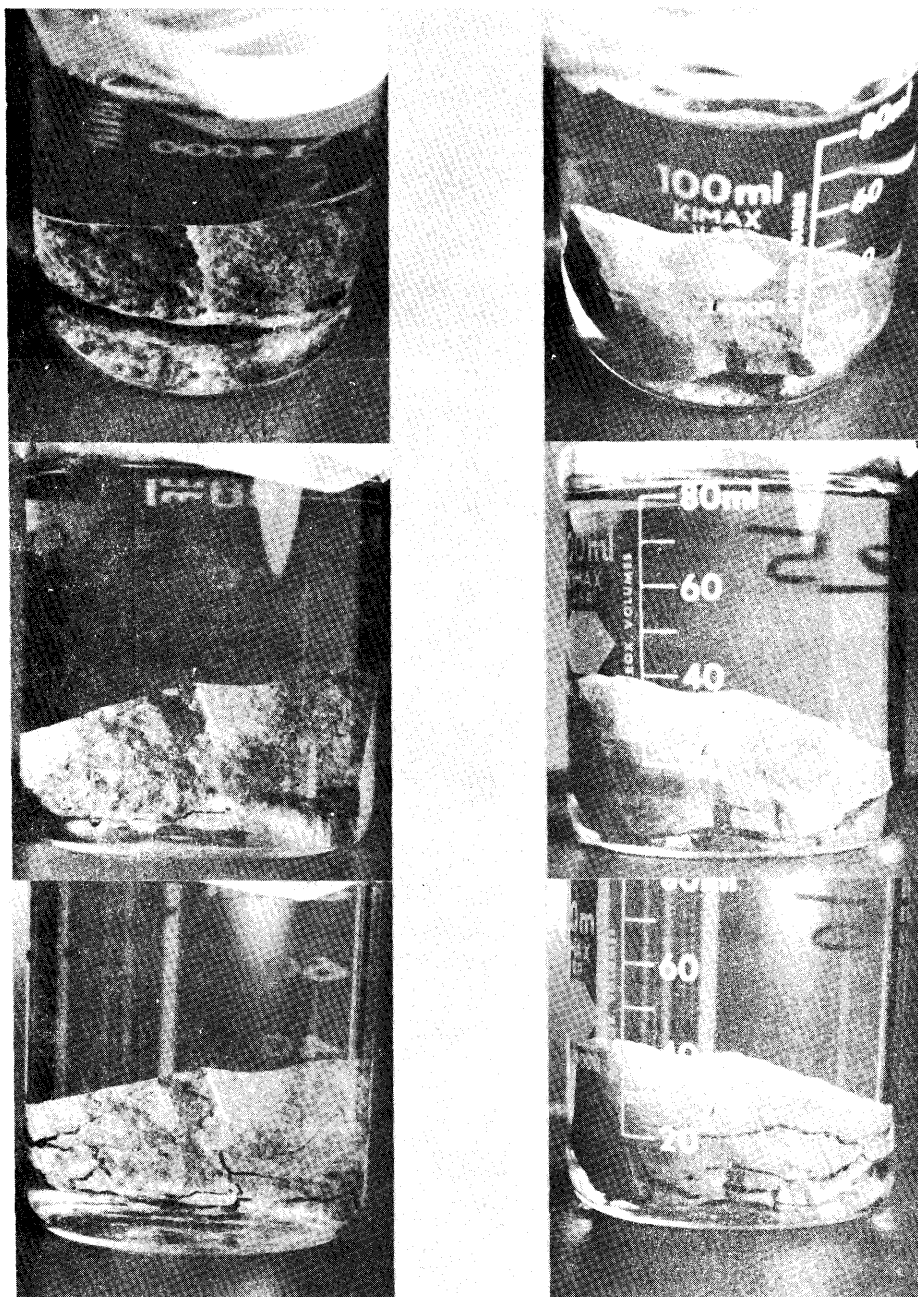
Front   Back

Figure B. A slightly distressed, acid leached particle,
           7-P-25, moving down both columns 1, 14, and
           55 days.

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