# **CHAPTER IV**

# ANALYTICAL TECHNIQUES -PARAMETERS AND METHODOLOGIES

This chapter provides guidance on certain physical, chemical, and engineering tests that are useful for characterizing geologic strata and soils affected by surface mining operations. Additional detail and guidance is available for soils and soil substitutes through separate documents specifically designed to address topsoil and revegetation issues. These sections provide methods currently recognized and accepted by the WVDEP. Other acceptable methods do exist, although it is highly recommended that the WVDEP be consulted prior to any significant deviation from these procedures.

### A. PHYSICAL ANALYSES

The geologic regulations require a description of the nature and physical properties of all strata to be affected by mining. This description is a detailed account of the rock stratigraphy and lithology, and is based on the rock and/or coal materials collected during the geologic sampling program outlined in Chapter III. The use of this information will allow a preliminary assessment of site conditions and assist in identifying both desirable and potentially problem-causing strata. This section will describe methods to analyze rock and coal material in a manner that will adequately determine the physical characteristics of the area strata. This raw information can then be used to develop geologic maps and cross sections (Chapter VI) along with the description of the site specific and regional geology (Chapter VIII).

#### 1. Lithology

Lithology is the physical description of a rock, generally determined megascopically. It considers the gross physical features of the rock, which include color, mineralogical composition, and grain size. The individual lithotypes identified during a drilling and sampling program should be classified according to standard geologic practices found in texts on sedimentary petrology or geology field techniques. For rock cores, a good classification of the lithotypes can be accomplished using standardized comparison charts available for both the southern and northern portion of West Virginia. The recommended classifications for the southern portions of West Virginia are covered in a book call "Cored Rocks of the Southern Appalachian Coal Fields" (Ferm & Weisenfluh, 1991). The northern half of West Virginia more closely approximates the classifications used in "A Guide to Cored Rocks in the Pittsburgh Basin" (Ferm & Smith, undated).

The following lithologies are common in West Virginia and these general descriptions should be consulted when determining gross lithology:

### Conglomerate

A conglomerate consists of gravel and/or pebble size (>2 mm mean diameter) particles in a matrix of fine-grained sand or silt. Depending upon the nature of the matrix and the ratio of coarse to fine grains, a modifier such as conglomeratic mudstone or conglomeratic sandstone may be appropriate.

#### Sandstone

Sandstone contains more than 50 percent sand-size (<2 mm and >1/16 mm in diameter) particles. The particles are predominately quartz and may be cemented with silica, iron oxide, carbonates, or clays. Qualitative modifiers, such as calcareous (noticeable fizz), argillaceous (contains a significant fraction of clay & silt), micaceous, and pyritic, are used when they seem to add useful information.

#### Shale

The term shale is best reserved for fine-grained rocks characterized by a finely laminated structure which imparts a fissility approximately parallel to the bedding. Shales normally contain at least 50% silt-sized (1/256 to 1/16 mm mean diameter) particles and 35% clay-sized (<1/256 mm mean diameter) particles. Descriptive modifiers such as carbonaceous, calcareous, pyritic, or arenaceous (contains a notable fraction of sand) are used when they seem to add useful information.

#### Clay Shale

A clay shale is a consolidated, finely laminated sediment consisting of no more than 10 percent sand and having a silt/clay ratio of less than 1:2. In other words, it is a clay-rich shale. It is also considered a soft shale, comprised primarily of clay-sized material, that when exposed to weathering, will degrade relatively quickly back to clay.

#### <u>Mudstone</u> (siltstone and claystone)

Mudstone may be a useful field name for fine-grained rocks composed of silt-sized (1/126 to 1/16 mm mean diameter) and clay-sized (<1/256 mm mean diameter) particles, which lack the characteristic thin lamination or fissility of shale. Mudstones tend to have block or spheroidal fracture patterns. Mudstone may contain as much as 50% sand-sized particles, if properties are judged to be dominated by silt and/or clay. Mudstones may contain any proportion of carbonates or carbonaceous material, provided the grains are dominantly silt- and/or clay-sized particles.

a. Siltstone -- Siltstone is an indurated rock largely comprised of silt-sized (1/256 to 1/16 mm mean diameter) particles. Well-sorted siltstones can normally be

identified with the use of a hand lens and are gritty to the touch. It is texturally and compositionally similar to a shale but lacks the fine lamination or fissility.

b. Claystone -- Claystone is an indurated rock dominated by clay-sized (<1/256 mm mean diameter) particles. Well-sorted claystones can normally be identified by their aphanitic to waxlike texture. They can be scraped or shaved smoothly with a knife and have a soapy feel when wet. When the claystone is poorly sorted, containing large portions of both clay and silt, identification will be difficult without a microscope.

# <u>Underclays</u> (fireclays)

The use of the terms underclay and fireclay should only be used according to their correct definition. These terms are often incorrectly considered to be synonymous and are often applied to any strata immediately below a coal seam. However, underclays may not have the refractory properties required to be a true fireclay. Nor are all fine-grained materials a true underclay. Shales and mudstones, which are lithified rock materials, are often misidentified as underclays.

In its true form, underclays are a layer of fine-grained detrital material, usually clay, lying immediately beneath a coal bed or forming the floor of a coal seam (Bates and Jackson, 1987). It represents the old soil (paleosol) in which the plants (from which the coal formed) were rooted, and often contains fossilized plant roots. Therefore, these terms should be restricted to their correct usage for paleosols and refractory clays.

## Limestone

Limestone is a sedimentary rock consisting of at least 50% (by weight) calcium carbonate. Although the primary constituent of limestone is calcite, it may contain other minor constituents including quartz, feldspar, clays, pyrite, and siderite. It may also contain a host of clastic grains of various mineralogies, ranging in size from clay- to sand-sized particles, depending upon the depositional environment. When dominated by high proportions of calcite, limestone will have a vigorous fizz when contacted by 10% HC1. Should the calcium be significantly replaced by other elements, such as magnesium (e.g., dolomite) or iron (e.g., siderite), a noticeable reduction in the rocks's fizz will occur.

### **Intercalate**

Intercalate is a term coined by Sobek et al., (1978), to describe rocks which contain at least two different rock types, that are so intimately interlayered or "intercalated" that they cannot conveniently be sampled separately. Intercalates have at least three or more layers within a 5-inch measured section. This rock type can be defined in greater detail by listing in order of abundance some or all of the kinds of rocks included (e.g., intercalate - sandstone/mudstone).

### Carbolith

Carbolith is a name that was coined by Smith et al., (1974) and accepted by Sobek et al., (1978) to cover dark-colored sedimentary rocks that will make a black or very dark streak or powder (Munsell color value of 3 or less), and that contain at least 25% carbonaceous matter. However, its use is not recommended because it serves more as a modifier than a rock descriptor. Correctly used terms such as carbonaceous mudstone, carbonaceous shale, and bone coal are much more descriptive than the word carbolith.

### Chert, Flint, and Jasper

Chert, flint, and jasper are rocks consisting dominantly of amorphous silica or extremely small (cryptocrystalline) quartz grains. They can normally be recognized by their hardness (7 on Mohs scale) and conchoidal fracture. Both flint and jasper are technically considered synonymous with chert. However, for field identification purposes, the term chert can be used for the white to gray variety, jasper can be used for the pink to red variety, and flint can be used for the green to black variety. Although relatively rare in the West Virginia coal field, it does occur in disseminated nodules and concretions along with some bedded deposits (e.g., Kanawha Black Flint).

### **Siderite**

Siderite is actually an iron carbonate mineral and commonly occurs in an impure form as both thin beds and nodules in clays, mudstones, and shales. It also may occur as a finely disseminated cement or accessory mineral. The chemical composition is quite variable with magnesium, manganese, calcium, zinc, and cobalt often being present. The maganese rich variety of siderite is called oligonite (Fe, Mn) CO<sub>3</sub>. If manganese supercedes the iron in the mineral, it would be considered mangansiderite (Mn, Fe)CO<sub>3</sub>, which is an iron-rich variety of Rhodochrosite. Siderite is normally yellow-brown, brown-red, or brown-black in color and generally gives a white streak.

#### Coal

Coal is generally defined as a readily combustible rock containing more than 50% by weight, and more than 70% by volume, of carbonaceous material (including inherent moisture) that was formed from compaction and induration of variously altered plant remains (Bates and Jackson,

1987). Coal is further broken down into ranks based on its calorific value (BTUs) and its organic matter content. Coals in West Virginia are restricted to a bituminous rank.

Bituminous coal is dark brown to black in color and can generally be megascopically divided into banded and nonbanded (sapropelic) fractions. The banded variety is the most common coal and consists of alternating bands of varying luster. The different bands are reflective of different macerals (organic lithotypes) in the coal and cannot be easily discerned in the field. The non-banded or sapropelic coals are derived from plant residues such as pollen or algae. These nonbanded coals are more commonly called bone coal, cannel coal, boghead coal, or torbanite. Bone coal is characterized by high ash content and is normally a hard and compact form of coal. The term "bone" is also used for argillaceous partings in the main body of a coal seam. Cannel coal is a dull, waxy coal that breaks in a conchoidal fracture. It has a high percentage of volatile matter and is formed primarily of spore material. Boghead coal is similar to cannel coal but is formed primarily from algae material. Torbanite is normally considered synonymous with boghead coal, although it is often used as a term for a highly carbonaceous oil shale. Additional information on coal descriptions can be gained through ASTM STP661, entitled "Field Description of Coal" (1976). Also, the "Handbook of Practical Coal Geology" by Larry Thomas (1992) and "An Introduction to Coal Technology" by Norbert Berkowitz (1994, provides information on coal descriptions, origin, exploration techniques, and mining geology.

### 2. Color

Color can be an effective tool in mapping and correlations if the causes for the color are understood. Rock color depends largely on the color and grain size of the original sediments and their cementing materials. However, color alterations readily occur as the result of weathering with the result being secondary pigmentation added to the rock.

Color is most accurately determined by using a standardized method of comparison, such as the Munsell System. This system uses predetermined color classification charts to compare with freshly broken surfaces of the rock to be identified. It is very accurate because it eliminates the variance in color perception among loggers, because of habit, lighting conditions, and color differentiation problems. Colors for both dry and wet samples should be recorded.

### 3. Grain Size

Grain size of a rock or soil can be classified by using the particle size classification chart provided in Table IV-1. Grain sizes in rock can normally be estimated using a l0x hand lens and/or a grain size comparison chart. Grain sizes for soils can often be estimated by physically feeling the sample under moist and dry conditions. Sand particles feel gritty when rubbed between the fingers and are not sticky or plastic when dampened. Silt particles feel smooth and powdery when dry but slightly sticky when moist. Silts can not normally be well molded or

Particle Size	Size Range	U.S. Standard Sieve Series
Clay	<0.004 mm	
Silt	0.004 - 0.062 mm	No. 230
Very Fine Sand	0.062 - 0.125 mm	No. 120
Fine Sand	0.125 - 0.25 mm	
Medium Sand	0.25 - 0.5 mm	No. 60
Coarse Sand	0.5 - 1 mm	No. 35
Very Coarse Sand	1 - 2 mm	No. 18
Pebble	2 - 64 mm	No. 10
Cobble	64 - 256 mm	No. 63 mm
Bolder	>256 mm	

Analytical Techniques - Parameters and Methodologies

Table IV-1.--Grain size classifications and relative ranges (Partial listing of Modified Wentworth Scale, after Lane, et al., 1947)

rolled without crumbling. Clays will become sticky and plastic when moist and can be easily molded. Soil materials can also be physically separated through the sieving processes into the various grain-size components. However, standard sieves will only separate down to the medium silt-sized particles, requiring other methods to differentiate between the clay and silt fractions.

### 4. Mineralogy

It is important to identify and describe certain minerals when they are observed, especially those that have a potential effect on the geochemical character of the rock (e.g., calcite, pyrite). Usually this is only possible with core or highwall samples, but careful examination of good rotary cuttings using a l0x hand lens can also reveal certain mineralogies. The most significant minerals that should be noted are the iron disulfides, pyrite and marcasite; carbonates, such as calcite, dolomite, and siderite; iron sulfates, such as melanterite, jarosite, and alunite; silicate minerals, such as muscovite and feldspars; and iron oxides, such as goethite, hematite, and

limonite. It is recommended that a good reference book on mineralogy and/or geology field techniques be used to aid mineral identification.

#### Iron Disulfides

Pyrite and marcasite are very common in coal deposits and will occur on bedding planes, joint planes, and cleats of the coal seam and associated strata as distinct crystals as well as framboids and nodules. These same morphologies can be disseminated throughout the sedimentary rock column, with the framboids more commonly associated with organic matter. Although framboidal pyrite is generally suspected to be one of the most significant contributors to acid production, determination of this form without a microscope is not possible because of its extremely small grain size. Likewise, various studies have subsequently shown that, although important, other forms of pyrite can be just as reactive and capable of generating acid mine drainage as the framboidal forms. However, notation of any visible pyrite and a hand lens description of the morphology should be recorded.

#### Iron Sulfates

Hydrous iron sulfates can be significant acid producers. Melanterite, jarosite, rozenite, copiapite, and others are very similar in appearance and are produced as a white or yellow efflorescence (fluffy or crystalline power or encrustation on a rock or soil surface) from oxidation of iron sulfides. The occurrence of these minerals is confined mainly to areas of weathered overburden and spoil materials, along with coal and coal processing wastes. These mineral salts concentrate in areas prone to repetitive cycles of wetting and drying, which initiate the efflorescence. Some of these sulfates may also form as botryoidal or fibrous masses, along cleats and bedding of exposed coal seams. Such minerals should not be present in fresh, unweathered samples.

#### Carbonates

Calcite and dolomite are the primary sources of useful neutralizing capacity in sedimentary rocks in the coal fields. In northern West Virginia, calcite and dolomite may form relatively thick beds of limestone. Elsewhere, calcite and dolomite are disseminated in thin limestone beds or calcareous shales. These minerals may also act as cementing agents or minor accessory minerals in any sedimentary rock-type. Calcite can be identified by its ability to vigorously effervesce in dilute hydrochloric acid (HCl). Dolomite must be scratched or powdered to significantly effervesce. Siderite will typically have no reaction to HCl unless powdered. Siderite was discussed earlier in this Chapter in the section on lithology. Siderite, where present in any significant quantity, can have a tremendous affect on both the geochemical analyses and the interpretation of results. Therefore, it is important to correctly identify its presence in coal-bearing strata. Likewise, siderite could be a possible source of elevated metals, such as manganese, in backfill material.

### Iron Oxides

Limonite is a mixture of hydrous iron oxides of indefinite composition. It is yellow to brown, and forms as an amorphous earthy or fibrous substance. Limonite is the end result of alteration of iron-bearing minerals such as pyrite, marcasite, and siderite. It is often responsible for the characteristic rusty-brown color on the weathered surface of rocks. Goethite is a yellow to brown/black iron oxide, that often forms as a secondary weathering product.

### Silicate Minerals

The dominant silicate minerals, which are readily identifiable in the field, are quartz (including chert and flint), feldspars, and mica (predominately muscovite). The identification of these minerals may assist in the identification of zones, which when analyzed, might generate potentially misleading values for neutralization potential. For example, two percent feldspar in a sample could give a neutralization potential of 20 tons of CaCO<sub>3</sub>, equivalents/1000 tons of material. Unfortunately, such neutralization is too slow to offer much useful neutralization in a surface mining environment. Also, such identification may provide good markers for stratigraphic correlation. However, to determine if a certain mineral or mineral assemblage is useful for correlation purposes, the entire stratigraphic column must be reviewed to determine if this assemblage is laterally continuous and is unique to the individual stratum.

Quartz is probably the most common mineral in sedimentary rocks. However, any abnormal occurrence (e.g., quartz pebble conglomerate) should be identified and recorded in the geologic log. It can normally be identified by its hardness (7 on Mohs scale) and conchoidal fracture.

Visible feldspars are somewhat rare in sedimentary rocks because of their propensity for weathering and alteration to clay minerals. However, their occurrence and identification in sandstones and conglomerates may be useful as marker beds. Feldspars can normally be identified through the prominent cleavage and hardness (6 on Mohs scale). Color normally ranges from white to pink.

Although muscovite is common in sedimentary rocks, its occurrence in abnormally large quantities (e.g., micaceous sandstone) may make it useful for correlation. Muscovite is normally recognizable by its platy cleavage and its highly vitreous luster. The reflection of light off these cleavage planes is often visible even in fine\_grained rocks (e.g., shales and mudstones).

#### **5.** Cementing Agents

For surface mining applications, the cementing agent of a sedimentary rock is important for both geochemical and engineering applications. A carbonate cement may be beneficial in that it can provide a valuable neutralizing component to the overburden while it may be detrimental to the rocks durability by being more prone to dissolution under acidic conditions. The principal cementing agents are clays, carbonates (primarily calcite), silica (primarily quartz), and iron oxides (primarily hematite or limonite). Although a precise determination of the cementing agents in a rock would require petrographic analysis, the use of a l0x hand lens will normally provide a good indication for most coarser grained rocks (i.e. sandstones).

### 6. Fossils

Fossils can be used for correlation and to provide information on the age and depositional environment of geologic strata. However, these techniques are well beyond the scope and need of both the WVDEP and the coal industry. Therefore, a general notation of macrofossils encountered in drilling or sampling is all that is necessary. Common fossil names recorded down to at least the phylum (e.g., brachiopods, mollusks) level will usually suffice.

# 7. Streak

Streak is a widely used diagnostic tool for mineral identification. However, in overburden rock-sample identification, it can be used to determine if a sample is carbonaceous. A carbonaceous black shale will leave a sooty, black streak, whereas a non-carbonaceous black shale will leave a gray streak.

Streak is noted by scraping a freshly broken face of the sample on a white surface such as unglazed porcelain Streak plates can be purchased, but any surface that is light and is hard enough to powder the sample face when scraped will suffice.

### 8. Sorting

The degree of sorting measures the similarity or uniformity in particle sizes in a sediment or sedimentary rock. It is used primarily for sandstones, as finer-grained materials cannot be precisely classified in the field. Because of the small sample size, a determination of sorting may not be available from rotary cuttings. If the degree of sorting is to be determined, it should be remembered that one of the greatest errors results from misidentification of dark constituents as matrix. Figure IV-1 provides a generalized chart, which can be used to determine sorting. For the purposes of this handbook and normal surface mining applications, bedding can be classified using the following generalized nomenclature:

Analytical Techniques - Parameters and Methodologies



Very Well Sorted



Well Sorted



Moderately Sorted



Poorly Sorted

Very Poorly Sorted

Figure IV – 1 Sorting Table

### 9. Bedding

Bedding is generally considered as the arrangement of rock in layers, strata, or beds. Bedding is caused by differences in texture, composition, or color of the original sediment. These differences result from a change in conditions at a sediment source or a change of sediment source. Observation of bedding character will normally be restricted to highwalls and outcrops where the overall geometry can be seen. Rock cores will provide small-scale details of bedding characteristics, but these may be difficult to relate to the overall bedding geometry. Rotary cuttings will provide little or no information on bedding.

Graded Bedding:	Bedding that displays a gradual and progressive change in particle size, usually from coarse at the base to fine at the top.
Cross Bedding:	Bedding where the minor beds or laminae are deposited oblique or at an angle to the main beds of stratification.
Massive Bedding:	Normally used for relatively thick-bedded strata that are relatively homogeneous and lacks any significant internal structures.

### **10. Fracturing and Weathering Patterns**

The degree of fracturing and weathering of geologic strata can have a tremendous affect on the hydrology of a mine site. In well-cemented, low-permeability rocks, fractures are the primary reservoirs for ground-water storage. They also can be the primary control for ground-water movement and pollutant transport. Weathering directly affects the geochemical characteristics of the rock strata, thus, affecting the ability to predict mine drainage quality.

### Weathering

Weathering is generally considered the in-situ degradation of rock materials into a loosened or altered form such as soil. Once chemically or physically degraded, it is subject to erosion and/or dissolution by surface and ground water. In a limestone-bearing overburden of Pennsylvania, Brady and others (1988) found that this weathering process effectively removed all carbonates to a depth of 20 feet. They also found that while the carbonates had been essentially weathered out of the system, high sulfur zones weathered slower and remained persistant throughout the overburden column. Therefore, an understanding of the depth of weathering is essential in determining the ability of overburden materials to produce or neutralize acids. Figure IV-2 illustrates the potential effects of weathering on geologic strata.

A highly weathered sample will generally be soft and crumbly and will exhibit effects of alteration such as staining caused by the oxidation of iron or other metals, while the less

#### Analytical Techniques - Parameters and Methodologies

weathered samples will remain hard. All such weathering characteristics should be recorded on the geologic logs, including the depth of weathering. Weathered rock samples are often misidentified by loggers having little or no professional training. It is relatively common to see logs that have identified a weathered sandstone as "shale" or "soil" simply because the sandstone had decomposed to the point that it was soft.



Note: In this example, 40 feet or more cover must be mined in order to encounter alkaline material.

Figure IV –2 Cross Section showing the effects of weathering

### Fractures and Joints

Fractures can most easily be described from highwall and outcrop sampling locations where their orientation can be mapped. Fractures may also be identified in rock cores, although their orientation, vertical extent, and horizontal extent will be unknown unless expensive, oriented cores are taken. Rotary cuttings will provide no information on fracture or joints. When fracture or joints are encountered, their orientation and any mineralization (e.g., calcite, siderite, etc.) or filling (e.g., clay or sediment) of the joint should be recorded.

### **B. CHEMICAL ANALYSES**

Chemical analyses should be conducted to identify coal and coal-bearing strata in the proposed permit area that may contain acid- or toxic-forming or alkalinity-producing materials. The purpose of these analyses is twofold: (1) to identify the strata that may pose potential problems for water quality and revegetation during mining and reclamation; and (2) to identify strata that may be used to neutralize potentially acidic materials and to substitute for, or supplement, topsoil. The results of these analyses affect many areas of the application and should be reflected in the appropriate sections (e.g., PHC, hydrologic reclamation plan, backfilling and grading plan, toxic material handling plan, etc.).

The following guidelines suggest several analyses that can be useful to identify strata that may have potential acid/toxic-forming or neutralizing properties. While these are not the only methods available for determining the geochemical character of overburden materials, they are procedures that WVDEP recognizes as acceptable methods for analyses. A more complete description of these tests is provided in the EPA publication EPA-600/2-78-054, entitled "Field and Laboratory Methods Applicable to Overburdens and Minesoils" (Sobek, et al., 1978). All test results should include a description of, or reference to, the analytical methods used, dates of analyses, and person(s) responsible for the analyses.

### 1. Net Acid-Base Accounting

The most commonly used type of overburden analysis in West Virginia is net acid-base accounting (NAB). Because of the length of time this method has been in use and the frequency of its use, the methods for analysis have become fairly standard. However, NAB will often overestimate the potential of a geologic stratum to generate acidity and/or its capacity to effectively neutralize acidity. Another problem has to do with NAB's inability to predict the generation of undesirable reaction products such as iron. Each of these potential problems is addressed more fully in the following sections. NAB consists of the following items:

### Paste pH

A measurement of pH indicates the activity of hydrogen ions in solution, and hence the degree of acidity. Usually, pH has little direct effect on plants unless it drops to some extreme value, such as below 3.0. Instead, the effect of pH is to control the solubility of spoil minerals, thus influencing the availability of their constituent elements for plant uptake, possibly in concentrations detrimental to plant growth. Other elements, such as molybdenum and selenium, although not toxic to plant growth, can be taken up and concentrated by plants in levels toxic to grazing animals. Raising the soil or spoil pH by the addition of a soil amendment, such as agricultural lime, reduces the solubility of many toxic elements so that they are no longer available for uptake by plants. Therefore, the toxic effect of these elements is essentially eliminated. However, some elements, notably molybdenum and selenium, are more soluble under alkaline conditions and are not tied up by liming.

With regard to water quality, elements that are mobilized in the acidic spoil environment have the potential to be carried to streams by acid mine drainage, causing both chemical and physical degradation of the receiving waters. This can reduce the quality of the water both as an aquatic habitat and for domestic and agricultural uses.

In addition, paste pH can assist in evaluating the results of the NAB analyses and the acceptability of submitted samples. For example, it would be very unlikely for a sample to have both a low paste pH and also a high neutralization potential. Also, a low paste pH indicates that a sample is derived from weathered material because fresh, unweathered rock typically associated with coal-bearing strata will normally have a neutral to alkaline pH.

The value for paste pH is determined electrometrically, directly on a sample ground to pass a minus 60 mesh sieve and mixed with distilled water to form a paste. A stratum is defined as acid/toxic if the resultant paste pH value is 4.0 or less.

### "Fizz" Rate

This qualitative test provides a relative indication of the amount, if any, of the calcareous minerals (calcite and dolomite) present in the tested sample. These minerals, if present, will "Fizz" when dilute HCl is added to the sample, indicating the availability of effective neutralization potential (NP) in the rock material tested. Note, however, that dolomite must be finely powdered before it will "Fizz." Conversely, lack of "Fizz" indicates a deficiency of calcareous minerals, and thus, ineffective neutralizing capacity for preventing the formation of acid/mine drainage. Table IV-2 provides a recommended "Fizz" rating scale.

#### Analytical Techniques - Parameters and Methodologies

"Fizz" Rate	Description
0 = None	No reaction or an occasional bubble caused by trapped air
1 = Slight	Minimal reaction; the reaction ranges from a few small bubbles per second to many fine bubbles that may cover the entire sample/acid interface
2 = Moderate	Active bubbling with only a small amount of splashing
3 = Strong	Very active bubbling, including substantial splashing of acid

Table IV-2.--"Fizz" Rating Scale (after Noll et al., 1988)

"Fizz" tests are normally conducted by the geologist in the field by applying cold 10% HCl directly to a sample. Laboratories will also conduct a "Fizz" test by placing approximately 0.5 gram of sample (ground to 60 mesh) on aluminum foil or a watchglass and adding cold 25% HCl. This is used as a precursor to the neutralization potential titration to ensure the addition of adequate acid. Because of the lack of standardization in "Fizz" ratings, a key to the rating scale used by both the geologist and laboratory should be provided with the test results.

Although the "Fizz" test may appear to only be of marginal importance, the "Fizz" test can have a large impact on the reliability and reproducibility of neutralization potential data (Kania, 1998). This is primarily because the "Fizz" rating determines the volume and the strength of the acid that is used to digest the prepared sample, which in turn can affect the NP determination results (Evans and Skousen, 1995; Skousen et al., 1997). The NP result is then somewhat dependent on the "Fizz" test results, and the "Fizz" test results are a matter of human judgement. Studies by Evans and Skousen (1995) tended to show that the values for NP would generally be higher where larger volumes of acid were used to digest the samples. If a sample was determined to have a higher "Fizz" rate by one laboratory, then additional acid may be added for the digestion process (i.e., 40 ml of HCl rather than 20 ml of HCl). Therefore, a higher NP calculation may result.

"Fizz" Rating	HCl Volume	HCl Normality
None (0)	20 ml	0.1 N
Slight (1)	40 ml	0.1 N
Moderate (2)	40 ml	0.5 N
Strong (3)	80 ml	0.5 N

Table IV-3.–Volume and Normality of HCl used to do NP digestion based on the sample "Fizz" ratio [From Kania (1998), after Sobek and other (1978) and Noll and others (1988)].

Given the difficulties that the current "Fizz" rating system introduces into the NP determinations, a reproducible, objective carbonate-rating test could significantly improve the reproducibility of NP data. However, until such a test is refined, individuals who generate and interpret data from acid/base accounts need to be more aware of the influence of the "Fizz" rates on the neutralization potential calculations. Where "Fizz" tests and NP seems to be in conflict, further testing or analysis should be provided. Additional detail and examples of the effect of this procedure are included in the paper by Kania (1998), which is included in the Pennsylvania Department of Environmental Protection's "Coal Mine Drainage Prediction and Pollution Prevention in Pennsylvania" (1998).

### Neutralization Potential (NP)

The minerals that account for the NP of coal overburden strata, as determined in the net acid-base account, fall into three general categories: (1) effective carbonate minerals, such as calcite (CaCO<sub>3</sub>) and dolomite [CaMg(CO<sub>3</sub>)<sub>2</sub>,]; (2) ineffective carbonate minerals, such as siderite (FeCO<sub>3</sub>); and (3) non-carbonate minerals, such as the feldspars, micas, and some clay minerals, all of which are complex silicates. Siderite, although a carbonate mineral, will not provide any effective neutralization to the system. Under field conditions, any neutralization or alkalinity provided by the siderite is supposedly compensated by acidity generated during the hydrolysis of the resultant Fe<sup>++</sup> compounds. Also, while providing neutralization under laboratory conditions, the non-carbonate materials under field conditions will react so slow as to be considered inert. The important difference between these three categories, in terms of effective neutralization of acid mine drainage, is that the effective carbonates react very rapidly; whereas the ineffective carbonates react very slowly or have no effect. Likewise, as stated in the previous section on "Fizz" tests, the resultant NP calculations can vary significantly based on the amount and normality of the acid used to conduct the test.

Siderite (FeCO<sub>3</sub>) has long been suspected of interfering with the accuracy of NP determinations and of complicating the interpretation of the data (Meek, 1981; Morrison et al., 1990; Wiram, 1992; Leavitt et al., 1995). Unless the iron fraction of the siderite molecule is completely oxidized, the calculated NP may be overestimated, along with creating problems with reproducibility. This results primarily from additional acidity being generated from the iron as it oxidizes, making it difficult to hold a titration end point during the laboratory procedure.

Although a variety of methods have been identified, which are intended to oxidize the siderite prior to titration, a method described by Skousen et al., (1997) seems to be a relatively effective and reproducible technique. If the hydrogen peroxide step performs according to its intent, it should generally decrease the NP of strata with a significant siderite content, but should not appreciably affect the NP values of strata that do not include significant amounts of siderite (Kania, 1998).

#### Analytical Techniques - Parameters and Methodologies

The laboratory conditions used to determine NP force the rapid and complete reaction of both effective carbonates and non-carbonate minerals using hot hydrochloric acid. (*Siderite should not be reflected in the NP calculations if the laboratory correctly reacted and precipitated it out of solution during the laboratory digestion process.*) This is the reason for the use of the term NP, which essentially means the quantity of acid that can be neutralized under idealized laboratory conditions. Thus, this value represents a maximum, "best case" situation, not what may actually take place in the field.

NP is expressed as x tons CaCO<sub>3</sub> equivalents/1000 tons sampled material. It is important to recognize the meaning of the term "CaCO<sub>3</sub> equivalents," because this is crucial to an interpretation of the net acid-base account. This term simply indicates that if a sample is determined to have a neutralization potential of x tons of CaCO<sub>3</sub> equivalents/1000 tons of material, then, regardless of its actual mineralogy, it has reacted with acid as though it contained x tons of CaCO<sub>3</sub>. The measured NP may, in fact, be due entirely to non-carbonate minerals and contain no CaCO<sub>3</sub> at all. Therefore, where a "Fizz" test fails to identify calcareous materials in a sample, the effectiveness of the NP must be determined through leaching tests or a determination of sample mineralogy (e.g., X-ray diffraction analysis).

#### **Forms of Sulfur**

*Total Sulfur:* An analysis for total sulfur, at least on a routine basis, is preferred over an analysis of other sulfur fractions, for the following reasons: (1) in the specific cases of unweathered rocks, pyritic sulfur is normally the dominant sulfur form, and so total sulfur does not significantly overestimate potential acidity; (2) the method of analysis for other sulfur forms (especially pyrite) is more difficult, time consuming, and requires close attention by an experienced analyst to give reliable results; (3) even in the most skilled hands, the pyritic sulfur analysis is subject to variable interference related to variations in rock mineralogy; and (4) the furnace combustion method for total sulfur is relatively straightforward and fast. If total sulfur does not indicate a problem with potential acidity, then neither will pyritic sulfur because pyritic sulfur must be less than total sulfur. Likewise, some sulfate forms (especially jarosite) can generate acidity when it hydrolyzes. As a result, a two-step sulfate procedure may be necessary that first leaches out the water-soluble fraction using hot water, followed by an HCl extraction, which is then multiplied by a different conversion factor of 23.44 [See Report of the Soil Working Group, Texas Mineland Reclamation Monitoring Program Issues Report (Askenasy, et al., 1998), for more information]. Such an increase in complexity and lack of reproducibility makes the total sulfur calculation preferable over sulfur fractionation methods. This is especially true for initial screening and analysis of samples.

The preferred method for the determination of total sulfur is to measure the sulfur dioxide produced when a sample is heated to approximately  $1600^{\circ}$  C in a stream of oxygen. The

method for measuring the sulfur dioxide and subsequent calculation of sulfur is determined by the type of equipment available.

<u>Sulfide Sulfur</u>: Although there are many sulfide minerals that may be present, pyrite and marcasite are the two most common sulfide minerals in coal and coal-bearing sedimentary rocks and are normally the principal contributors to acid production. Both are iron disulfide (FeS<sub>2</sub>) minerals with pyrite crystallizing in the isometric system and marcasite crystallizing in the orthorhombic system. Pyrite is generally considered the more abundant mineral and marcasite the more reactive. Because the testing procedure for sulfide sulfur does not differentiate between these two minerals, or any other sulfide that may be present, the remainder of this text will use the term pyrite (pyritic) for all sulfide minerals.

Even though analysis of the pyritic sulfur form is a more specific method for determining the acid-forming potential, the tests are more difficult and time-consuming. There also remains some uncertainty regarding the actual amount of acid that will be formed during weathering of the overburden. The possible occurrence of several varieties of pyrite in the same sample, which exhibit a wide range of acid-forming reactivity under normal weathering conditions, accounts for that uncertainty. However, the laboratory conditions for oxidizing pyrite to determine sulfide sulfur are so extreme, and the reaction is so complete, that it does not represent the natural overburden differences in pyrite reactivity. Thus, the determination of acidity from pyrite is termed potential acidity, i.e., that amount of acid that would be formed if all the pyrite present were to be oxidized.

This method selectively extracts the sample, using various acids to separate the different sulfur forms. Sulfates are first extracted with hydrochloric acid then pyrite is extracted with nitric acid. This oxidizes the pyritic sulfur to sulfate, which is soluble and goes into the leachate. The organic sulfur is unaffected and remains in the sample. This procedure avoids gross overestimation of potential acidity by the exclusion of non-acid forming sulfur.

<u>Sulfate Sulfur:</u> Although not significant in unweathered (usually less then 1%) sedimentary rocks of the West Virginia coal field, sulfate sulfur is common in weathered spoils and refuse materials. As a result, weathering products, especially iron and aluminum sulfate salts derived from the oxidation of pyrite and reactions between acidic sulfate-bearing waters on aluminum minerals (e.g., feldspars), can be significant acid producers (Sobek, et al., 1978; Ott, 1986). Although sulfate salts (e.g., melanterite, rozenite, jarosite, etc.) were once thought to be basically inert reaction products, it is now recognized that the hydrolysis of the Fe<sup>++</sup>, from either ferric sulfates or the oxidation of ferrous sulfates, can result in acidity by freeing H<sup>+</sup> ions. Therefore sulfate sulfur cannot be discounted as an unreactive component in the NAB calculations and should be analyzed for whenever the pyritic sulfur content is required.

**Organic Sulfur:** The role of organic sulfur in water quality impacts is not well understood, although its effects are generally considered to be negligible.

### **Potential Acidity (PA)**

This is a calculated value determined by multiplying the percent sulfur of the sampled material by 31.25 and expressing the result, (PA), as tons  $CaCO_3$  equivalents/1000 tons of sampled material. The factor of 31.25 is derived from a stochiometric calculation expressing the equations for the oxidation of pyrite to sulfuric acid and subsequent exsolution of  $CO_2$ . The resultant sulfur percentage multiplied by this factor is expressed as calcium carbonate equivalents in tons/1000 tons. However, based on studies by Cravotta et al., (1990), a factor of 62.5 may be necessary where  $CO_2$  is not exsolved. It is doubtful that either factor is completely representative of field conditions since some  $CO_2$  is both exsolved and trapped in the mine backfill.

If, after using the applicable factor, the resultant potential acidity value is less than 5 tons of CaCO<sub>3</sub> equivalents/1000 tons of material, then the strata is generally considered to be non-acid forming. For materials that have a potential acidity value greater than 5 tons of CaCO<sub>3</sub> equivalents/1000 tons of material, it is suggested that the PA be verified by determining the pyritic sulfur content. Also, because sulfate sulfur is generally considered a weathering product of pyrite and a potential source of acidity, the PA should also include sulfate sulfur if the paste pH indicated a pH less than 7.0. Once the pyritic and sulfate sulfur values are known, they should be added together and multiplied by the 31.25 conversion described above. This will give a more realistic potential acidity value based on the acid-producing sulfur forms.

### Calculation and Interpretation of Net Acid-Base Account

The net acid-base account (NAB) is calculated by subtracting the potential acidity (PA) from the neutralization potential (NP):

$$NAB = NP - PA.$$

The NAB should be calculated for each sample analyzed. Where overburden mixing is proposed, a total weighted NAB may be required to determine the overall geochemical character of the overburden column. This weighted NAB must be calculated using the volume of each individually analyzed strata, **not the thickness** of each strata. Failure to perform the weighted NAB on a volumetric basis would be to overestimate the spoils ability to generate or neutralize acid mine drainage.

A negative value will indicate excess acidity. Because some natural soils have acidity values approaching minus 5 (-5), Sobek et al., (1978) established this value as the "threshold" value for determining whether the sampled material is "acid" or "non-acid." Thus, the material is "acid" if the NAB has a value of minus 5, or numerically more negative. DiPretoro and Rauch (1988)

found a NAB value of +5 to be a better threshold. (It should be kept in mind that if a stratum has a paste pH of 4.0 or less, it is defined as acidic, regardless of the NAB value.) The NAB value is expressed as tons CaCO<sub>3</sub> equivalents/1000 tons of material. Negative NAB values can also be expressed in terms of a deficiency of CaCO<sub>3</sub> and a positive value as an excess of CaCO<sub>3</sub>. In other words, material with an NAB of -8 would have a deficiency of 8 tons of CaCO<sub>3</sub> equivalents/1000 tons of material, meaning that every 1000 tons of tested material would require 8 tons of lime to neutralize the PA.

The primary advantages and disadvantages of the acid-base accounting method are:

- Advantages
  - a. Short turn-around time for sample processing;
  - b. Relatively low cost;
  - c. Relatively simple analytical procedures (except for pyritic sulfur); and
  - d. Results do not require sophisticated interpretation.
  - Disadvantages
    - a. The method assumes that pyrite or sulfur oxidation and carbonate dissolution rates are the same. It also assumes a 1:1 acid to base reaction. Actual acid production and neutralization release rates cannot be predicted with this technique.
    - b. The method assumes that the neutralization potential and potential acidity is totally available for reaction.
    - c. The method assumes that the neutralization potential and potential acidity are the only two variables that impact mine drainage quality.
    - d. The method does not make any predictions on the nature of resultant water quality.
    - e. It is a non ASTM method.

## 2. "Simulated Weathering," or Leaching Tests

Where the results of the NAB account are inconclusive or additional testing is still needed based on site-specific conditions, leaching tests have been utilized in an attempt to simulate what occurs in nature after mining is completed. Leaching tests are generally considered as "kinetic" tests because they include the effects of physical, chemical, and biological processes in an attempt to more closely represent the reaction rates and mechanics that earth materials may experience in the field. Leaching or "kinetic" tests are normally performed in laboratories under relatively controlled environments, although some field-scale testing may be warranted under certain conditions. Because of the wide variety of leaching apparatuses (leaching columns, humidity cells, and Soxhlet reactors) and methods that are available, coordination with WVDEP is a necessity to ensure that a valid and reproducible test is performed. This will prevent the possibility of an erroneous or poorly designed leach test. No leaching test should be performed until there is a clear understanding of the goals, testing methods, apparatuses to be used, and the way samples are prepared and combined (if applicable). Generally, leaching tests should be restricted to an individual stratum of interest rather than a composite of multiple strata. Reproduction of an entire mine backfill in a leaching column, humidity cell, or Soxhlet reactor is difficult at best, and totally meaningless at worst. If such tests are deemed necessary, consideration of a field-scale leaching experiment should be considered. In all instances, special attention should be paid to the physical, chemical, and biological processes involved in the natural systems and incorporate them into the design, operation, and interpretation of these tests. The section on kinetic (leaching) tests by R.J. Hornberger and K. Brady, which is included in the Pennsylvania Department of Environmental Protection's "Coal Mine Drainage Prediction and Pollution Prevention in Pennsylvania" (PDEP, 1998), provides an excellent overview of the history and considerations involved in performing these types of tests.

The ASTM test D3987 "Shake extraction with water" is a short-term test that only measures readily soluble components of the sample. This test is unacceptable for predicting the ability of a stratum to generate acid drainage and should not be used. The Weathering Cell Method described by Sobek, et al., (1978), has several variations and is of a longer term (usually 8 weeks). The test involves a multiple extraction technique that adds the dimensions of time and oxidation plus the option of "seeding" with bacteria. Soxhlet extractions are elevated temperature tests that reduce the time needed to estimate leachate quality. There are several other tests that may be occasionally applicable to overburden, but that are more prone to use with waste rock or coal combustion products. These include the EPA Toxicity Characteristic Leaching Procedure (TCLP) and the Synthetic Precipitation Leaching Procedure (SPLP). The WVDEP should be consulted prior to the use of these latter methods as a substitute for leaching or "kinetic" tests. The applicability of these tests to overburden materials is questionable since they were designed primarily for use with hazardous materials associated with the controlled conditions of sanitary landfills.

Hornberger and Brady (1998) recommended several major principles that should be considered during the design of a simulated weathering test. These can be generally summarized as follows:

- a. The size, shape, and structure of the kinetics test apparatus should be as simple as is practical. However, some complexity may be necessary to allow fluids and gases (i.e., oxygen and carbon dioxide) to enter, circulate through, and exit the apparatus in a manner that is representative of the weathering conditions of the mine environment.
- b. The dimensions of the test apparatus should be proportional to the particle size distribution and volume of the rock sample to be tested, so that there are no adverse interactions between the sample and the container. For example, with a columnar shaped apparatus, the inside diameter of the column should be at least several times greater than the largest particle diameter within the volume of rock or waste sample. This should help

minimize "wall effect" and airlocks that will interfere or bias the movement of gases or water through the sample.

- Rock samples should be as representative of the physical (particle size distribution) and c. chemical (i.e., mineralogic composition) characteristics of the consolidated overburden strata, or backfilled mine spoil, or waste dump to be simulated in the test. According to Hornberger and Brady (1998, p. 7-12), "The mineralogical composition and size distribution of rock materials within a backfilled surface mine are important factors in determining whether the mine spoils produce concentrations of acidity or alkalinity." Particle size in the field is affected by blasting practices, effects and types of equipment used in handling, geologic properties of the rocks themselves (hardness, durability, bedding, cements, etc), and tectonic features such as naturally occurring fractures and faults. Unfortunately, since most samples will come from a test borehole, the particle size distribution will not be representative of the backfill. However, despite this, careful attention must be paid to the percentages of fine versus coarse sample because the finer grain may have a larger surface area. According to Brady (1974), a grain of fine colloidal clay has about 10,000 times as much surface area as the same weight of medium-sized sand. Several studies have shown a disproportionate percentage of total sulfur in the finer grained portion of a sorted sample. Therefore, the distribution of particles sizes and subsequent surface areas available for reaction must be considered in the testing procedure.
- d. The ratio of the surface area of rock sample to the volume of leachate may be the most important parameter in kinetic test design, performance, and data interpretation. The volume of the water added to the apparatus (influent) minus the water retained or consumed by the sample, should equal the volume of leachate exiting the leaching apparatus. The volume of water added to the apparatus should be proportioned with the sample material to represent the volume of water, which would be representative of the water encountered in the mine backfill conditions (rainfall, ground water, etc.). Too little or too much water could bias the sample by diluting or giving significantly more concentrated results than would be expected under natural field conditions. According to Bradham and Caruccio (1995), for optimum test performance, the volume/weight of influent water in a single leaching episode should not be greater than 1 times the rock sample weight (and normally not greater than 0.5 times the rock sample weight) regardless of the type of testing apparatus. However, the influent must be sufficient to generate enough effluent water to run the appropriate analysis.
- e. Samples should not be completely submerged or saturated throughout the duration of the test because pyrite oxidation rates will be typically reduced and cause the resultant effluent to be lower in AMD reaction products such as sulfates and metal concentrations. To simulate the weathering conditions in the backfill, samples should normally be exposed to either partial saturation or cycles of saturated and unsaturated conditions. The ultimate decision of how the sample is subjected to saturated conditions may be partially

determined by where the materials will be placed in the mine backfill (high or low). In the unsaturated zones, the weathering of the sample will be enhanced by alternating wetting and drying cycles, and the effects of humidity, water flow, and air flow through the voids or pore spaces.

- f. Leach cycles are generally considered as the procedures and time required for water to be introduced, circulated through the test medium and apparatus, and subsequently removed as a leachate. To help determine the overall testing period, along with the number and length of individual leach cycles, it must be remembered that the cycles and overall test period should be of sufficient duration to simulate weathering and natural hydrologic conditions, which would occur in the mine environment, and to ensure reproducibility and repeatability of the tests. From an economic and practical standpoint, the tests should include enough cycles to be within the cost and time constraints necessary for the pursuit of a mine permit.
- g. Typically, the pore gas composition of the sample in the leaching apparatus should be similar to the anticipated gas composition in the reclaimed, mine backfill. Without sufficient oxygen, the pyrite oxidation process will be retarded and the full weathering potential may not be reached. Likewise, without sufficient carbon dioxide, the dissolution and solubility of carbonate rocks will be significantly reduced. Oxygen content is not typically a problem in most laboratory environments. However, carbon dioxide may have to be added to adequately represent field conditions. A mixture of 10% oxygen, 10% carbon dioxide, and 80% nitrogen in a pressurized gas cylinder would supply adequate and representative amounts of both gases to allow for both pyrite oxidation and carbonate mineral dissolution.
- h. Iron-oxidizing bacteria can catalyze the pyrite oxidation process by six orders of magnitude over what would occur by abiotic pyrite oxidation processes. Therefore, any leaching or kinetic test should innoculate the sample with Thiobacillus bacteria at the beginning of the test unless it can be demonstrated that sufficiently high pH or temperature will exist at the mine site, which would affect their survival.

Based on the aforementioned considerations, a leaching test can be developed that should help determine the alkaline- or acid-forming nature of a geologic stratum. Although one recommended methodology for conducting leaching tests is outlined in Sobek, et al. (1978), there are numerous methods available which can and have been used by WVDEP and other state regulatory agencies. However, these tests are far from standardized. Therefore, the interpretation of any leaching test is dependent on the method used. As a result, a detailed description of the method must be provided. This description must include:

1) the stratigraphic interval tested

- 2) the exact volume of water added at any time
- 3) the exact volume of water withdrawn at any time
- 4) the exact weight of sample being leached
- 5) the size range of the sample
- 6) chemical analysis and description of the source of water used in leaching and/or any acid mine drainage which might be used as a source of bacteria (e.g., distilled deionized water of pH 5.7)
- 7) frequency and duration of leaching, including flushing cycles and contact times
- 8) the water level(s) relative to surface of rock samples
- 9) description of contact with air; such as open to atmosphere, circulated humidified air, circulated dry air, air with  $pCO_2$  of  $10^{-2}$ , etc.
- 10) the apparatus used should be sketched or photographed (with reference scale) in order to show procedures (diameters, heights, etc)
- 11) temperature (if other than normal room temperature)
- 12) how strata samples were composited (if applicable)
- 13) any other relevant factors.

To conduct a leaching test, geologic samples are generally leached with water at weekly intervals. It is recommended that at the onset of this procedure, the sample column be inoculated with pyrite-oxidizing bacteria (Thiobacillus ferrooxidans) within the first week of the test. An analysis of the inoculate should be included along with all leachate results. Each week, the sample column is flushed with distilled water and subsequently analyzed to determine water quality trends. Once the leaching is complete, moist air should be passed over the sample for a minimum of three days. At the end of the week, the sample column is again flushed with distilled water. This cycle continues for a minimum of eight weeks, although ten weeks is preferable.

By leaching at weekly intervals, the acid-forming reactions are allowed to proceed and any generated acids are given time to react with whatever neutralizing minerals are present. In addition to testing individual strata, it is theoretically possible to test various treatment scenarios, or blends of strata can be tested in order to model the effectiveness of overburden treatment or

#### Analytical Techniques - Parameters and Methodologies

mixing, as these are commonly used methods of handling acidic material. However, as stated above, this procedure is much more difficult since the recreation of all of the microenvironments at a mine site are difficult to reproduce in the laboratory environment. As a result, any such proposals for leaching materials in a layered, mixed, or lime-amended sequence, must be designed with the approval of WVDEP after specific design provisions have been met.

With regards to the resulting leachates; the leachates from these weathering tests should typically be analyzed for at least the following constituents:

- 1) pH
- 2) total hot acidity
- 3) total alkalinity
- 4) total manganese
- 5) total iron
- 6) total aluminum
- 7) total dissolved solids
- 8) sulfate
- 9) other constituents as needed (i.e., major anions and cations; other metals)

The weekly analyses will record the changes or trends in these constituents can then be used to predict the potential of the mine site to generate acid drainage.

### Extended leaching tests

Where the standard 8 week leaching test is inconclusive, or a trend suggests potential water-quality problems if the test was allowed to continue, an extended leaching test may be required. The following are criteria that may indicate a need to extend the duration of the leach test:

- (1) The pH declines throughout the duration of the test or exhibits a sharp drop at the end. If the pH approaches or drops below 6.0 only at the end of the 8 or 10 week test, then the test needs to be continued to determine if the trend continues.
- (2) Alkalinity declines throughout the test, or at the end of the test, to suggest that it may decrease below the level of acidity if the test is conducted for a longer period of time.
- (3) Acidity increases throughout the test, or at the end of the test, to suggest that it may exceed alkalinity if the test is conducted for a longer period of time.
- (4) Sulfate maintains a substantial value, or increases, throughout the test. A relatively steady level suggests that ongoing acid formation is being neutralized, while an increase suggests that, in addition, bacterial growth is progressing with an increased rate of pyrite

oxidation. If available neutralization capacity is exceeded before all of the pyrite is oxidized, the leachates will likely become acidic.

After the WVDEP has approved the recommended procedures, the laboratory is to be notified that an extended test duration may be required. Based on the progression of the leachate quality during the normal test time (8 weeks), a decision must be made as to whether to continue or suspend the leach test. The results are to be immediately evaluated by the responsible laboratory personnel, in consultation with the applicant and the WVDEP, to either continue or terminate the test. The results and the laboratory evaluation used in reaching this decision will be submitted to the WVDEP as a part of the final leaching test results. If the test is continued, and a clear trend is not established by the end of 16 weeks, the test is to be continued to 20 or 24 weeks, but leachate should only be collected at the 20th and 24th week, not weekly. After a review of the technical literature relevant to leaching tests, and consultation with researchers actively engaged in overburden evaluation, it is concluded that the test shall be terminated at no later than the 24th week.

The main advantages and disadvantages of leaching tests are summarized as follows:

- Advantages:
  - a. Test methods are designed to more closely simulate field conditions;
  - b. Reaction rates (kinetics) can be evaluated;
  - c. Leaching of overburden constituents other than acidity and alkalinity can be evaluated (iron, manganese, aluminum, etc.); and
  - d. Allows for some prediction of actual effluent water quality.
- Disadvantages:
  - a. Test time (since at least 2 months are required to conduct an analysis);
  - b. Cost of analysis;
  - c. Long-term predictive capability of leaching tests are uncertain;
  - d. Disparity in the particle size and subsequent reactive surfaces between bench-scale and field conditions;
  - e. No consensus on the test procedures and methods of interpretation; and
  - f. Data interpretation requires more sophisticated review than the acid/base accounting method.

# C. ENGINEERING ANALYSES

Depending upon the type of surface mining operation proposed, an assessment of the engineering or mechanical properties of geologic strata may be required. The type and amount of information required will depend upon the site-specific conditions and the type of mining operation proposed. The following sections address the types of information most commonly required by the WVDEP.

### **1. Rock Durability**

Durable rock as defined in the regulations ia a rock that does not slake in water and will not degrade to form soil material. In surface mining applications, durable rock is often required for underdrains and rock chimney drains in valley fills/head-of-hollow fills. It is also required to be the predominate constituent of a single lift durable rock fill, making up 80 percent of the total volume of the fill. The successful performance of excess spoil structures is directly related to the durability of the rock in the fill mass and underdrains. Nondurable rock will degrade into soil-sized particles as a result of overburden pressure and moisture absorption, often resulting in the clogging of the void spaces between the rocks. This clogging may cause excess pore water pressure to develop, resulting in a decrease in the shear strength of the fill material. This decrease could cause the structure to fail.

Geologic materials removed from their in-situ environment, during the surface mining of coal, exhibit changes in physical integrity. Such changes are caused by physical and chemical mechanisms induced by variations in moisture and stress regimes. The rock used in fills or drains is typically subjected to blasting, handling, compaction, and weathering. Therefore, a combination of tests are generally needed to best simulate surface mining conditions.

Testing procedures recognized and recommended by WVDEP and OSM may vary depending upon the site-specific conditions and design of a proposed mining area. However, WVDEP primarily recognizes the use of the slake durability test using procedures defined by Chandra (1970) and Franklin and Chandra (1972) (See the following section regarding slake durability testing methods.). For each stratigraphic unit to be disturbed or at least every 5 feet a rock sample or core should first be subjected to:

1. Slake Durability Test -- The slake durability test determines minor abrasion effects and saturation/desiccation stresses. As defined by Chandra (1970) and Franklin and Chandra (1972), oven-dried samples of rock are placed in a wire mesh drum partially immersed in water. The drum is rotated at 20 rpm for approximately 10 minutes; the sample is then removed, dried, and run through a second cycle. If the sample is found to have a Slake Durability Index rating of greater then 90%, then the sample is considered durable.

Slake Durability testing should be conducted on proposed overburden at a frequency of every 2000 feet, the same as the frequency of acid/base accounting. Samples should also be taken at the same vertical frequency as recommended for acid/base accounting. As previously recommended for acid/base accounting, attempts should be made to sample and account for both weathered and unweathered sections of overburden. Also, when calculating the total amount of durable material in an area, volumetric corrections for the local topography and weathering patterns need to be included. These testing and calculations should used to develop a site specific handling and/or mixing plan as needed.

Some alternatives to these testing procedures may be allowed or required based on the operation plan or the site-specific conditions. However, any such deviations or requirement should be authorized by the WVDEP prior to initiation. All such test results should include a description of, or reference to, the analytical methods used, dates of analyses, and person(s) responsible for the analyses.

# 2. Engineering Properties (Underground Operations)

All underground mining and auger operations must provide adequate geologic and hydrologic information to determine the potential for subsidence and subsidence-related damage. To help predict subsidence effects, certain information on the engineering properties of geologic strata is needed. This section will briefly describe some of the testing procedures that may be required for these types of operations. However, this section was written only as a brief introduction to testing methods that may be applicable to underground operations.

For standard room-and-pillar mining operations, the regulations require additional information on the engineering properties (basically strength and durability) of clays or soft rock in the stratum immediately above and below the coal seam to be mined. This information is needed for the development of a subsidence control plan and would be used to determine the capacity of the weaker strata to withstand the increased static load to be imposed in the area of remaining pillars. Samples of soft rock or clay may be taken by core drilling or from freshly exposed test pits. In either case, care should be exercised in wrapping and sealing the samples to retain as much of the natural moisture as possible. Depending on test procedures and requirements, the size, shape, and number of samples may vary. Site-specific requirements for laboratory testing should be determined, by a qualified technical specialist, based on the type and thickness of clay or soft rock, thickness of overburden, dip of the strata, and probability for saturation. If a subsidence control plan is not required, or there are no clays or soft rocks present3., a waiver of the requirements for engineering properties should be requested. (See Chapter VIII.) Planned subsidence caused by longwall mining or by removal of pillars in standard room-andpillar mining requires additional information, beyond just the immediate roof and floor rock. Such tests will depend upon the amount of subsidence planned (partial versus complete), the overlying lithology (amount of competent rock), depth of the coal seam, and the thickness of the coal seam.

Engineering properties tests may include rock durability as determined in the previous section or any of the following:

Unconfined uniaxial compressive strength -- This is a compressive test performed by taking a cylindrical specimen material, having a length to diameter ratio of at least 3:1, and loading it axially to failure without the use of any lateral restraint,

- Triaxial testing—The primary recognized test for coal core analysis is the Triaxial method. The test apparatus has a thick cylindrical cell that has the ability to apply lateral pressure as an axial load to the rock specimen.
- Direct shear -- This is a shear test in which a cylindrical specimen of the clay or soft rock material is subjected to an applied normal load and is stressed to failure by lateral movement of one end or section of the shear box. This test is also used to determine the shear strength of competent rock units (e.g., massive sandstones) above the underground workings and their ability to support the overlying material without a shear failure.
- Atterberg Limits -- This is an indicator of the type of clay minerals in the rock and their plasticity. This involves measuring the liquid limit, plastic limit, and plasticity index of the fine-grained fraction (minus No. 40 sieve size) of the rock material.
- Clay Content -- This is a simple grain-size analysis performed on a disaggreagated sample of the clay or soft rock. The disaggreagated sample is passed through a series of progressively smaller, standardized sieves, with the clay fraction being collected in the bottom pan. The clay fraction is then compared with the original weight of the sample to determine the percent clay. This test will not work for well indurated rock materials.
- Moisture Content -- This test compares the weight of the raw sample to that of a dried sample to determine the amount of inherent water in a particular stratum. Water content along with the Atterberg Limits, will help determine the clay strength.

All test results should include a description of, or reference to, the analytical methods used, dates of analyses, and person(s) responsible for the analyses.