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Shale Analysis for Mud Engineers

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Abstract

In the process of drilling oil and gas wells an interaction occurs between the drilling fluid and the formations drilled. A simple analysis of the formation composition can help understand the problems and successes using a particular fluid in different fields. An understanding of what comprises a formation can lead to better formulation of the drilling fluid.

One of the methods used to identify the basic mineralogy of a formation is X-Ray Diffraction (XRD). XRD determines the crystalline structure of the minerals being tested by determining the angles at which the X-Ray beam is diffracted. This results in a pattern that is unique to a particular mineral. Analysis of the patterns from a formation sample can help identify the minerals present and semi-quantify them.

An oriented clay slide is prepared and a series of chemical and physical manipulations of the slide are conducted. XRD is used to observe the changes at each step to identify the basic clay minerals present and can provide an estimate of their relative concentration.

The cation exchange capacity (CEC) of clays is a method used to determine the reactivity of a clay and thus the shale. Additional information about the reactivity of the shale can be obtained, if the exchangeable cations are identified and quantified.

Introduction

Understanding the minerals that make-up a particular material is vital in many industries. In the oil industry, specifically, understanding the constituents of a geological formation under investigation can have implications as to what site is chosen for drilling. Once a site has been chosen this mineralogical information can be used to anticipate certain expectations about the field due to the minerals revealed. This leads to further implications, such as the customization of drilling fluids for the site. As minerals are crystalline the use of x-ray diffraction (XRD) to identify and determine the approximate amount of each species is used.

The objective of this paper is to help the reader better understand the procedure that OGS Laboratory Inc. (OGS) performs when a shale sample is submitted from the field. This will, in turn, enable the reader to better interpret the XRD analysis reports given out by OGS and other laboratories.

To begin, a simplified explanation of the basic theory of XRD will be given. Hopefully, this will provide the readers with enough information that they may be able to determine if XRD is useful for the applications they are interested in, and suggest other information that may be useful.

XRD - Determining crystal structure

In essence, the diffractogram produced by XRD permits understanding of the arrangement of the atoms in a material. Each mineral has a unique arrangement of atoms, referred to as its crystal structure or lattice, and these atoms are arranged in infinite repeating planes. Depending on the distance between these planes, the x-rays will diffract at a series of specific angles that provides a pattern that is explicit for the mineral in question; see Figure 1. This can be used as a "fingerprint" for that mineral.

As suggested in Figure 2 the sample is placed between a source of x-rays and an x-ray detector. By changing the angle of the sample holder, relative to the source, each crystal plane gets an opportunity to diffract its respective angle of x-rays. The detector identifies which angles produce its respective "reflected" x-ray. For example, quartz's major plane has a "d-spacing" of 3.44 Å. Applying this to an equation called Bragg's Law ($n\lambda = 2d \sin\theta$) the diffraction angle 2θ can be predicted to be about 26.6^{o}. Looking at Figure 3 one can see a large peak at 26.62^{o} with an intensity of 6299 counts per second (cps).

The other peaks in the diffractogram are associated with other crystal planes and are important in verifying that the mineral is actually present (i.e. the rest of its unique fingerprint).

Clays Discussion

It is hard to distinguish the clays from each other in XRD due to the fact that their structure and d-spacings are similar to one another. However, the species can be distinguished by exposing the clays to chemical or physical changes. Chemicals consisting of water and ethylene glycol may be inserted, naturally or artificially, between the lattice planes. This will cause the lattice spacing to alter and thus it will change the position and appearance of the diffraction peak. The application of heat has two effects. Heat expels the aforementioned chemicals therefore altering the crystal planes again. Alternatively, applying heat can result in a variety of instabilities of the crystal within itself. The lattice will have to compensate for these instabilities by changing its d-spacing and, thus, altering the diffraction peaks.

Methods

Sample preparation

The method used is derived from the method described by Schultz.[3] The sample is washed using a variety of organic liquids and solvents. After this the sample is dried and hand ground so that it passes through a fine sieve. The powder is then ground mechanically for 15 minutes to achieve optimum grain size.

Bulk analysis

This entails looking at a broad range of potential angles of diffraction for the sample. Therefore the x-ray diffractometer is set to scan the sample starting at angle 3.5° and ending at 70.0° at a constant speed. This allows the technician to see any possible minerals in the sample. It is vitally important that the powder's orientation be as random as possible so that each mineral gets a fair representation of itself. A further discussion of this topic will be given later.

By using a spreadsheet and a programmed macro the computer mathematically generates the results for the minerals listed in Table 1. See Figure 4 for an example of what is obtained when doing a bulk analysis. If other high intensity peaks appear that are not associated with the minerals listed in Table 1 it is up to the technician doing the analysis to identify those peaks manually. The amounts of these extra minerals may be approximated depending on the mineral. Some of the more common ones are listed in Table 2.

Oriented Clay Slides

About 2 grams of the powdered sample is mixed with 30mL of deionized water. This is stirred and then allowed to stand so that the more dense particles sink and the clay particles remain suspended in the upper layer of water. A pipette is used to extract this clay solution and deposit it on to a frosted glass slide. The water is allowed to evaporate overnight; this is done because, in this case, the particles on the slide need to be oriented.

The individual clays (kaolinite, illite, smectite and chlorite) are distinguished by the clay slide. For each run the range of 2θ of interest is 3.5°-20°. The clay slide is manipulated and then scanned using XRD after each step of the process.

The clay slide will be scanned four times. First, the slide is run in the diffractometer after all the water has evaporated. This initial run is referred to as the "fines" sample, it can be thought of as the "before snap-shot" of the manipulations. The slide is then put in a desiccator with ethylene glycol in it and heated to 70°C. (A spare desiccator is ideal for this because it allows the pool of glycol to sit at the bottom and be partitioned from the slide resting on a screen above the glycol.) After about 2 hours the slide is briefly air dried and analyzed on the diffractometer again. This diffractogram is referred to as the "glycolated" sample. The slide is then put in an oven at 300°C for about an hour and a half. After cooling to room temperature, the diffraction peaks are obtained and this data is labeled "300°C". Similarly, the oven is heated to 550°C and the slide is heated at this temperature for about 30-minutes. On cooling, the slide is run again, and this data is labeled "550°C". The glycolation process, alone, distinguishes the clay smectite. The mixed-layer of illite and smectite is determined by the combined manipulations of glycolation and heating to 300°C. Kaolinite is determined by heating to 300°C. Chlorite is noted by its dramatic lattice collapse after heating to 550°C. Illite is distinguished by its ability to resist all of these manipulations.[3] As with the bulk analysis a macro is performed on the collected data according to some calculations briefly discussed below.

Semi-quantifying using Microsoft Excel

Using a Microsoft Excel macro, the data, obtained by the XRD software, is processed for interpretation. A chart of the minerals commonly found in shales and their respective primary 2θ values are listed in Tables 1 and 2. The pure standards of the minerals in Table 1 are analyzed in the same manner as the bulk samples. Here the highest intensity value is noted for all of the pure standards, see Figures 7-13. This value is used to semi-quantitate the relative amount of each mineral in the mixture. By taking the value at that 2θ reading in the sample and dividing that by the value derived from the pure standard a good estimate of its percentage is given. For example, refer back to Figure 3 where the quartz peak has a maximum at 6299 cps. A sample is run and gives a value of 1417 cps at 26.65°. Therefore the estimated quartz percentage is 1417/6299=22.5%. Quartz, feldspar, calcite, dolomite and siderite are treated in this direct manner. The clays (as a whole) and pyrite are mathematically manipulated more by factoring in an intensity factor.

Using a computer macro that compares the diffractogram of each step the clays are classified and semi-quantified. Standards are utilized but only for identification purposes, see Figures 14-17. An example of the final x-ray analysis process, including bulks and fines, is given in Figure 5. By looking at each graph from bottom to top one can see how the peaks alter after each application of the process. Based on these changing peaks the computer calculates the final volumetric percentages. Both the bulk and fines data is directly interpreted into weight percents because the densities of the minerals are about the same or because an intensity factor is used.

Discussions

General Procedures

When a sample is brought to the facility a series of considerations need to be taken into account. A washing strategy is determined by what needs to be cleaned off of the sample. A sample may be divided into several aliquots, and then washed differently to ensure that if any one of the samples' components gets leeched out in one washing that it will be present in another. Each washing is analyzed by means of the XRD procedure discussed above. Heterogeneous samples maybe compensated for by separating out the unlike particles into the same number of different samples and running them accordingly. The weights of the separated samples are measured so that a percentage of each can be quantitated. The freshly washed samples are then dried at about 250°C to make sure all of the solvents are removed from the sample.

After the grinding process the bulk sample is prepared for analysis by positioning it on a sample holder. The technician uses a specialized tool to assemble this. This tool ensures that the surface of the sample is ideal for analysis in two ways. In order for the samples and standards to be compared correctly their surfaces must be at the same height within the sample holder each time. Conventionally, this is chosen to be level with respect to the sample holder. See Figure 6 for an illustration of this.

The device also allows for ideal packing of the sample. This is vital because the mineral grains vary in size and density. If the sample is packed too loosely the particles will organize themselves in a heterogeneous fashion. This will cause the surface to be erroneous with respect to the sample in question. To extrapolate from this, it is also mentionable that the original powder must be mixed very well before extracting the powder out to be analyzed. Due to the fact that mineral grains can organize themselves almost instantaneously, the technician must also be careful to extract the sample by scooping out the contents by touching the bottom of the container. These techniques, together, will ensure an acceptable randomized bulk sample that is representative of the original cuttings.

Ideally, when the bulk powder is mixed into the water the clays will suspend and the slide will be made accordingly. Often, a suspension is not produced. This can arise due to the presence of a water-soluble mineral, like halite or gypsum. This will dissolve in the water and prohibit the suspension of the clays. The sample must then be washed with water extensively until the clays can be suspended.

In reference to the clay slide itself, it needs to be emphasized that allowing the water to evaporate as slowly as possible renders the most desirable results. When the slide is dried in a heated environment the clay grains do not become as oriented as they do when it is allowed to dry overnight at room temperature.

Ensuring that the clay layer on the slide is not too thick or thin is important as well. If the clay is too thick the sample may peel during heating. If the sample is too thin the clay-peaks are minimized or absent all together. Patience is a key component in retaining desirable results from the clay slides. To add to the making of the slide each step involves heating the sample for a particular length of time. The technician must also allow time for the samples to cool slowly at each step so that the samples will not bubble and peel (this is similar to being too thick). Similar to the bulk analysis, the individual clay slide must be analyzed in the same spot. The technician simply chooses which end of the slide is placed into the diffractometer first and consistently uses that same end.

Limitations

As this is a semi-quantitative computation, emphasis needs to be placed on the fact that this is a general approximation of the contents in the sample. XRD, in general, is ideal for identifying crystalline material. However, the size of the peak is directly proportional to the number of lattice planes presented. Therefore the intensity of the peak is directly related to the percent volume of the species (being related to the number of planes diffracted). A limitation to this results from the shape factor. On grinding the sample into fine particles, the different minerals within the sample adopt different shapes. Quartz takes on a spherical shape during grinding; most other minerals grind to prismatic or rhombic fragments. Clays tend to produce flakes. The spherical quartz grains are randomly orientated and so their peak height is a fair representation of its volumetric abundance. When a sample is put in a sample holder, the non-spherical grains have preferred orientation. Therefore the plane of choice for each of these minerals may either over-represent or under-represent its abundance. These limitations do not seem to be too much of a hindrance due to the needed applications. Also, this phenomenon is likely to also apply to the standards so errors are reduced.

Another limitation to keep in mind is that the standards used in obtaining the "pure" reading may not be interpreted correctly. As shown in Figures 7-17 when applying the pure standards to the macro erroneous minerals can result. This can result from the extra mineral actually being in the standard. Alternatively, a peak from another lattice plane of that standard may be misinterpreted as a peak from another type of mineral. In the case of the clay standards erroneous peaks usually indicate foreign minerals are, indeed, present in the standard. The best example is shown in Figure 17 where the chlorite standard contains a lot of kaolinite. (Scattering Electron Microscopy (SEM) has verified the presence of kaolinite in this standard.) Since the clays are calculated differently this causes very little concern. But, in the case of calculating the total clays in the bulk sample, if a mineral's peak under-represents its amount the “leftover” amount is placed in the “total clay” cell. This, also, can be seen in the pure non-clay standards, Figures 7-13. Again, it is up to the technologist to ensure that the XRD data is interpreted correctly.

Mineral implications in the field

By using the preparation and interpretation techniques discussed above the expectations of that field can be predicted. A brief account is given on the general understanding of how each mineral yields an idea of what to expect and thus leads to modifying drilling fluids for the site.

Quartz and the feldspars are crucial to understand because, together, they are the most abundant minerals encountered during drilling. Quartz is very hard (hardness of 7) and abrasive. Its crystals can increase the porosity of the shale, allowing fluid to penetrate farther and faster than in similar low quartz shales. This circumstance can destabilize the shale, leading to difficulties in drilling. Likewise, feldspars are hard (6 – 6.5) and are the primary matrix of shales. In other words, the shale will be more easily dispersed if the feldspar content is low. Furthermore, the carbonates (calcite, dolomite and siderite) are of interest because like quartz they may cause additional porosity to the shale, but are significantly softer (3 - 4). Some of the carbonates are also a potential source of low-level carbonate build-up in the mud. Similarly, pyrite is a hard mineral (6 – 6.5) that may impart some increased porosity to the shale.

Beidellite and montmorillonite are known to be the most common and most well known smectites, respectively. It is generally accepted that vermiculites belong in a separate class from smectite due to differing CEC results (discussed below). As a group, vermiculites are much more reactive than the smectites. The smectite concentration in shale is important. Being that smectites swell when hydrated, they can make the formation unstable. Smectite clays are most often controlled these days with inhibitive water-based, oil, or synthetic-based fluids. Furthermore, kaolinite is a dispersive, non-swelling clay. It is commonly found in drilling wells in the North Sea and off the West Coast of Africa. Kaolinite can be controlled most effectively with the addition of a source of potassium. Which leads to illite, which is a dispersive, non-swelling clay that can also be controlled by the addition of a source of potassium or some of the amine compounds, currently in use today. Similarly, chlorite clays are dispersive, non-swelling materials that are generally not found in sufficient quantities to need special attention. Typically, methods to control the kaolinitic and illitic clays will generally control the chlorite clays. Mixed layer clays can be of several types: kaolin/smectite, chlorite/smectite, chlorite/vermiculite and illite/smectite. The most frequently found and therefore most important of these is the illite/smectite mixed layer. When water interacts with this mixed layer the consequences rendered are much more damaging than if it were pure smectite or illite. The illite allows the smectite to swell more easily due to increased exposed surface area. Also, the mixed layer sets up a situation where the less reactive clays act like ball bearings for the swelling clays to roll upon. Macroscopically, this results in disintegrating shale.

Cation Exchange Capacity (CEC)

CEC values are important due to the fact that clays vary in their reactivity. Some of the cations (positively charged atoms) of the clay are easily replaced with other positively charged species. This phenomenon is driven by the fact that the original cations are not as compatible to the negatively charged site as the newly introduced positively charged species. Thus, it is rationalized that the exchangeability of the cations are directly related to the reactivity of the shale. However, the CEC within itself does not provide this information alone. The individual cations of the clays that are replaced and left in solution indicate reactivity of the clay as well. These cations, also called exchangeable bases, primarily are calcium, potassium, magnesium, and sodium. For example, shale with a high CEC value along with a high concentration of calcium may not be as reactive as shale with a lower CEC value and a high sodium concentration.

To summarize the reaction, the Mg^{2+}, Ca^{2+}, K^+, and/or Na^+ initially present in the clay are loosely bonded to negatively charged sites within the clay. The CEC reagent is a positively charged species in solution. The clay and the CEC reagent are allowed to mix with one another for an extended period of time. During this time, the CEC reagent is strongly attracted to the negatively charged sites. It is, therefore, drawn out of solution and bonded to these sites. This in turn, drives the Mg^{2+}, Ca^{2+}, K^+, and/or Na^+ into solution. Either Ion chromatography (IC) or Atomic Absorption Spectroscopy (AA) can quantify these cations.

OGS has used several CEC procedures. Methylenblue (MBT) is desirable for the field due to its fast results. However, it is known that MBT is not very reliable when compared to the other methods.

Ammonium acetate is commonly used in deriving soil CEC values for agricultural applications. This procedure is indeed useful but has limitations. It consists of a lengthy amount of time to complete. This method is regarded as an indirect method, which involves a distillation and titration to derive the CEC value. These can be easily mishandled and/or misinterpreted which will lead to inaccurate CEC values. The ammonium left over from the process is not easily taken out of the solution and therefore interferes with the calculation of the cations, if IC is used to identify the cations. This particular reagent replaces salt and carbonate cations as well. This can increase the CEC value and render inaccurate cation counts.

OGS has found the cobalt(III)hexamine procedure to be very useful.[4] It does not demand as much time as the ammonium acetate procedure. The CEC value is obtained directly by measuring the cobalt(III)hexamine concentration by either AA or by UV/Visible spectrophotometery. See Figure 18 for the complete XRD and CEC spreadsheet.

In the limited time OGS has been using the cobalt procedure, we have found that it gives a higher CEC than does the ammonium procedure. The corresponding cations also increase an equal amount.

CEC data can indicate whether a clay mineral, particularly smectite, has a predisposition to cation retention or whether or not diffusion of charged or uncharged molecules can occur within the clay.[5] In other words, if a shale has a high CEC it might be able to be altered either by nature or by the application of a drilling fluid. This may be detrimental or useful to the drilling process depending on the alteration.

Conclusions

With good organization skills an accurate account as to what is included in a sample can be rendered. Being able to wash and grind a sample effectively is of up-most importance. Preparing the powdered bulk sample to be even and as random as possible is vital to the success of semi-quantitating the minerals. Patience is a necessity in preparing and manipulating the clay slides to obtain accurate results. Utilizing CEC to better understand these minerals is of utmost importance as well. Having experienced and knowledgeable personnel executing the procedures is important to ensure that the best possible analytes are created and that the XRD and CEC data is interpreted logically. Understanding the essential theories and applications behind XRD and CEC will enable the reader to better recognize and understand these very useful applications. It also enables the individuals from the field to better interpret and appreciate the reports submitted by OGS Laboratory Inc. These factors in combination will, hopefully, provide the field personnel a better understanding of the site that they are considering or drilling.

Acknowledgments

A special word of thanks to Dr. James K. Meen, Task Leader of the Materials Characterization Facility at the Texas Center of Superconductivity and Applied Materials of the University of Houston, for his advise.

Nomenclature

AA = Atomic Absorption Spectroscopy
CEC = Cation Exchange Capacity
cps = counts per second
IC = Ion Chromatography
MBT = Methylenblue Test
SEM = Scattering Electron Microscope
XRD = X-Ray Diffraction

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Additional Reading
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2. Cullity, B. D. *Elements of X-Ray Diffraction*; Addison-Wesley Publishing Co., Inc.: Reading, 1956; 514.

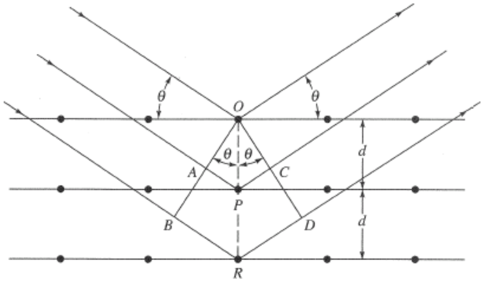
Table 1
Primary shale minerals and their 2θ values

Mineral	2θ (degrees)
Quartz	26.6
Feldspar	27.5
Calcite	29.6
Dolomite	31.0
Siderite	31.8
Pyrite	33.1
Clays	19.9
	34.6
	61.9

Table 2
Additional minerals common in drilling and their 2θ Values

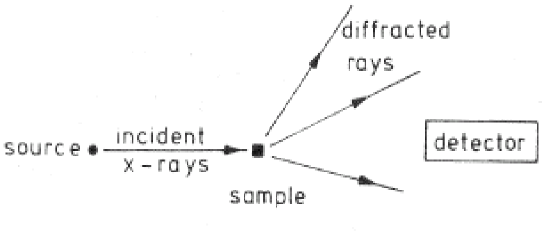
Mineral	2θ (degrees)
Halite	31.7
Barite	28.9
Portlandite	34.0
Gypsum	11.7
Magnetite	35.5

Figure 1
Simplified Diagram of the diffraction of x-rays by a crystal
(Adapted from ref 1)



Diffraction of X-rays by a crystal.

Figure 2
Symbolic Schematic of XRD Experiment
(Adapted from ref 2)



The X-ray diffraction experiment

Figure 3
XRD of pure quartz

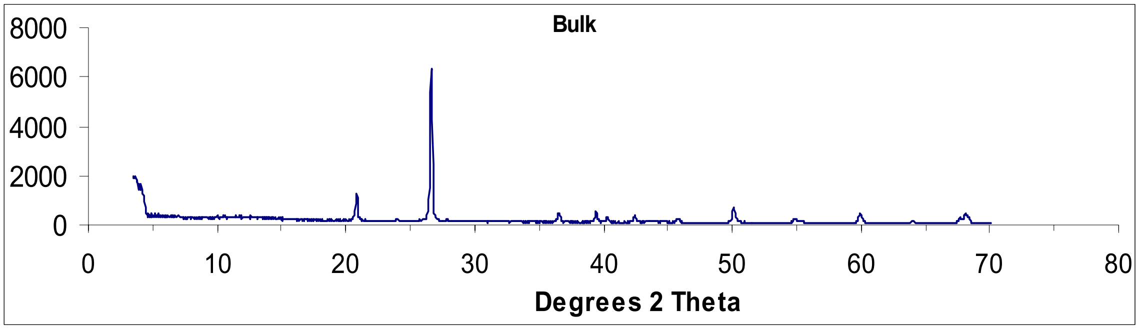
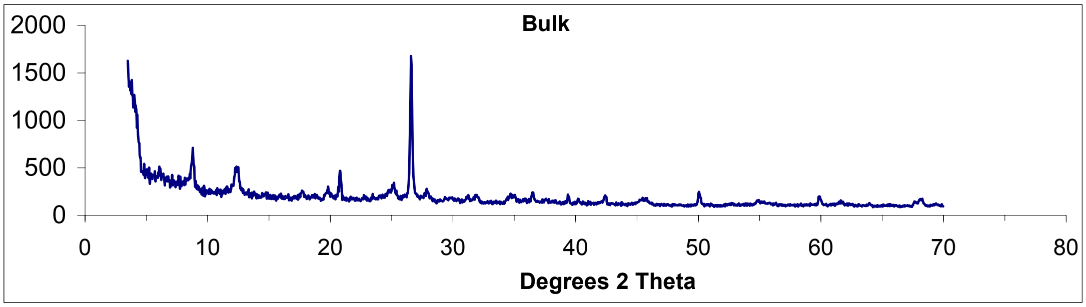


Figure 4
Results after a bulk analysis

X-Ray Diffraction Interpretation and Data

Project: **NDFH 1978** **Sample:** **4/11/2003**

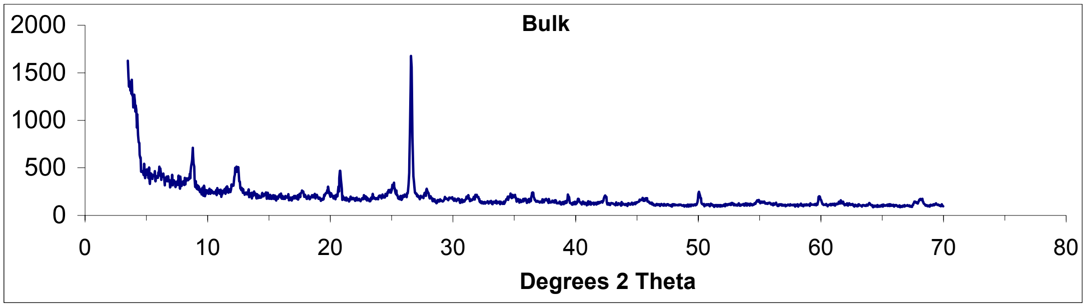


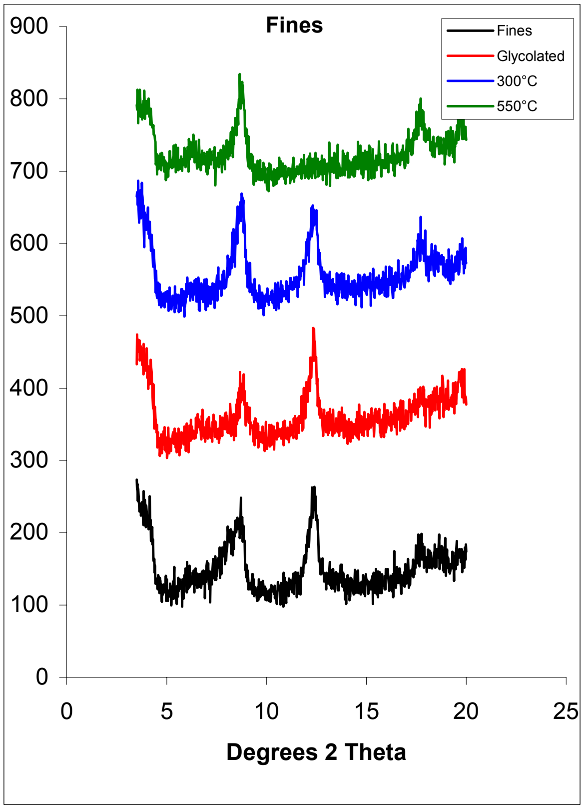
Bulk Composition -	**wt%**
Quartz	24
Feldspar	3
Calcite	1
Dolomite	2
Siderite	3
Pyrite	0
Total Clay	67

Figure 5
Example of Bulks and Fines

X-Ray Diffraction Interpretation and Data

Project: **NDFH 1978** **Sample:** **4/11/2003**





Bulk Composition -	**wt%**
Quartz	24
Feldspar	3
Calcite	1
Dolomite	2
Siderite	3
Pyrite	0
Total Clay	67

Clay Composition -	**wt%**
Kaolinite	23
Chlorite	7
Illite	31
Smectite	12
Mixed-layer	28
Illite/smectite	25 / 75

Figure 6

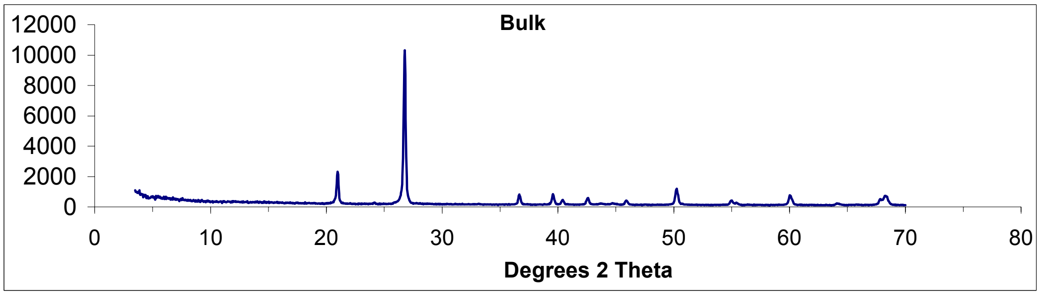
Left to right: Correct and Incorrect way bulk sample should be in sample holder

Figure 7

Quartz Standard

OGS Laboratory, Inc. - X-Ray Diffraction Interpretation and Data

Project: Quartz **Sample:** Quartz

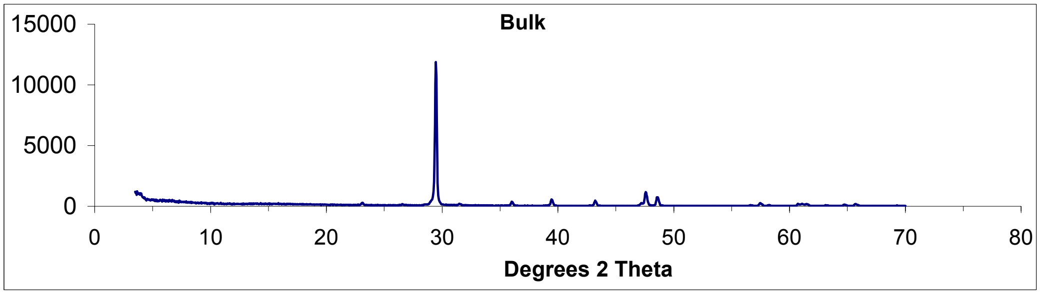


Bulk Composition -	**wt%**
Quartz	96
Feldspar	0
Calcite	0
Dolomite	0
Siderite	0
Pyrite	0
Total Clay	4

Figure 8
Calcite Standard

OGS Laboratory, Inc. - X-Ray Diffraction Interpretation and Data

Project: Calcite **Sample:** Calcite Standard

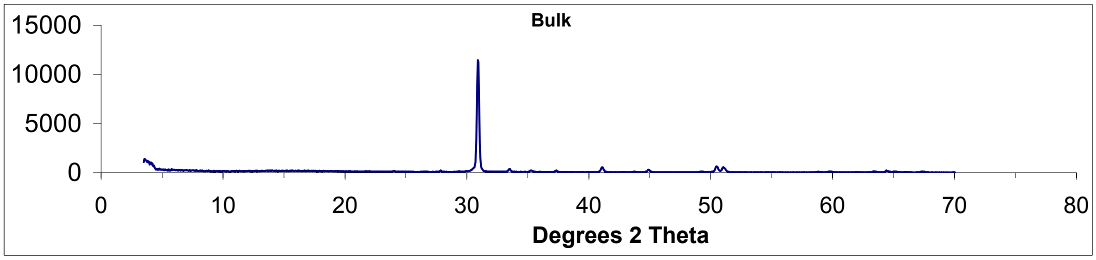


Bulk Composition -	**wt%**
Quartz	0
Feldspar	0
Calcite	92
Dolomite	0
Siderite	1
Pyrite	0
Total Clay	8

Figure 9
Standard of Dolomite

X-Ray Diffraction Interpretation and Data

Project: **STANDARD** **Sample:** **DOLOMITE**
Description:

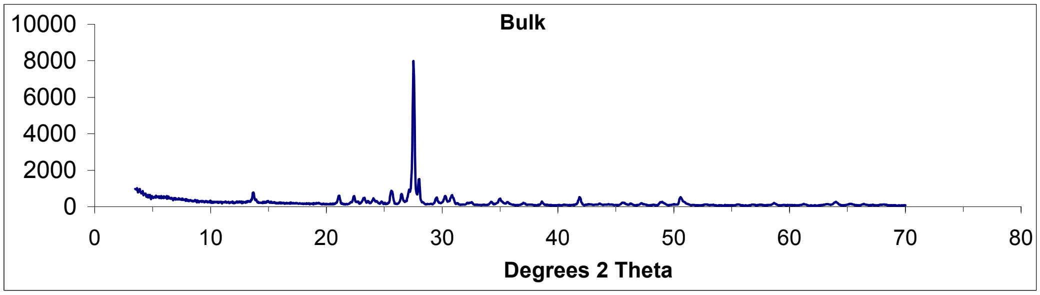


Bulk Composition -	**wt%**
Quartz	0
Feldspar	1
Calcite	1
Dolomite	87
Siderite	2
Pyrite	1
Total Clay	8

Figure 10
Microcline Feldspar Standard

OGS Laboratory, Inc. - X-Ray Diffraction Interpretation and Data

Project: Microcline **Sample:** Microcline Feldspar

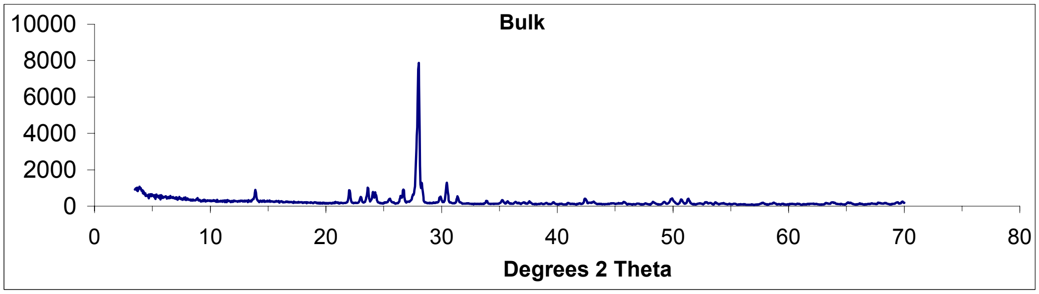


Bulk Composition -	**wt%**
Quartz	4
Feldspar	66
Calcite	4
Dolomite	4
Siderite	0
Pyrite	0
Total Clay	21

Figure 11
Oligoclase Feldspar

OGS Laboratory, Inc. - X-Ray Diffraction Interpretation and Data

Project: Oligoclase **Sample:** Oligoclase Feldspar

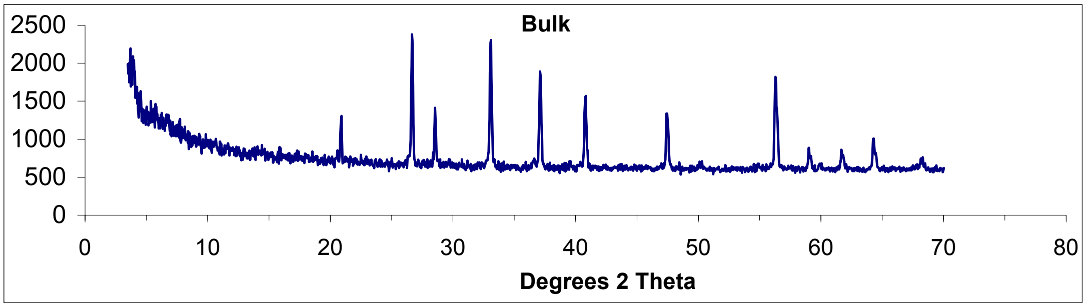


Bulk Composition -	**wt%**
Quartz	4
Feldspar	71
Calcite	4
Dolomite	2
Siderite	6
Pyrite	0
Total Clay	14

Figure 12
Standard of Pyrite

OGS Laboratory, Inc. - X-Ray Diffraction Interpretation and Data

Project: Pyrite Standard **Sample:** Pyrite

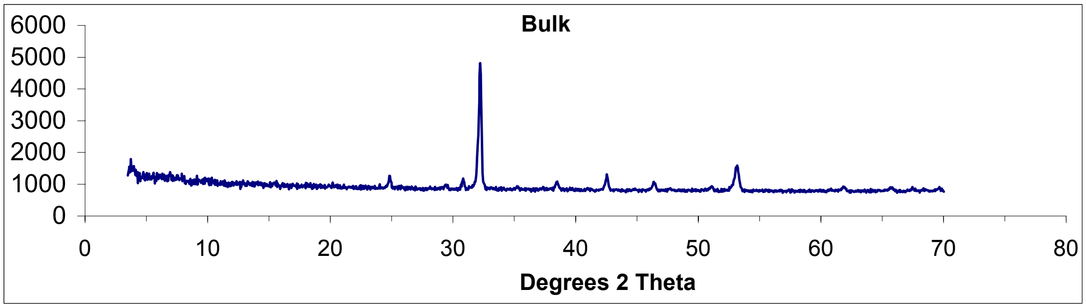


Bulk Composition -	**wt%**
Quartz	17
Feldspar	1
Calcite	1
Dolomite	1
Siderite	0
Pyrite	77
Total Clay	3

Figure 13
Standard of Siderite

OGS Laboratory, Inc. - X-Ray Diffraction Interpretation and Data

Project: Siderite Standard **Sample:** Siderite

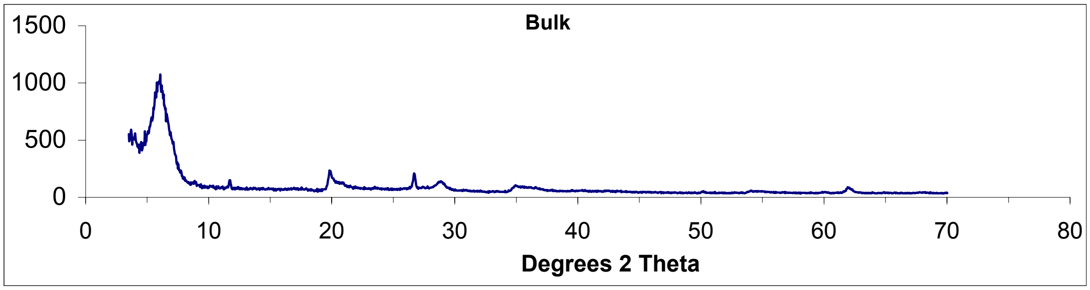


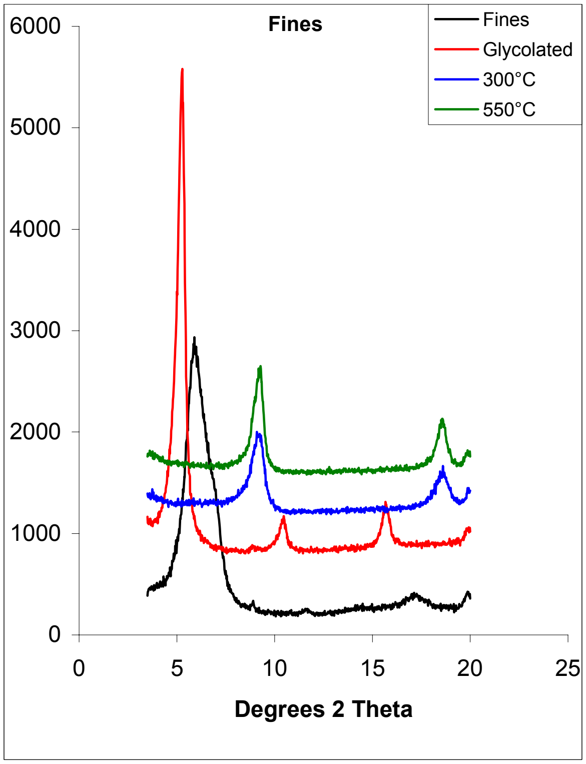
Bulk Composition -	**wt%**
Quartz	2
Feldspar	4
Calcite	9
Dolomite	14
Siderite	65
Pyrite	1
Total Clay	6

Figure 14
Standard of Smectite

OGS Laboratory, Inc. - X-Ray Diffraction Interpretation and Data

Project: Montmorillonite **Sample:** Sodium Montmorillonite





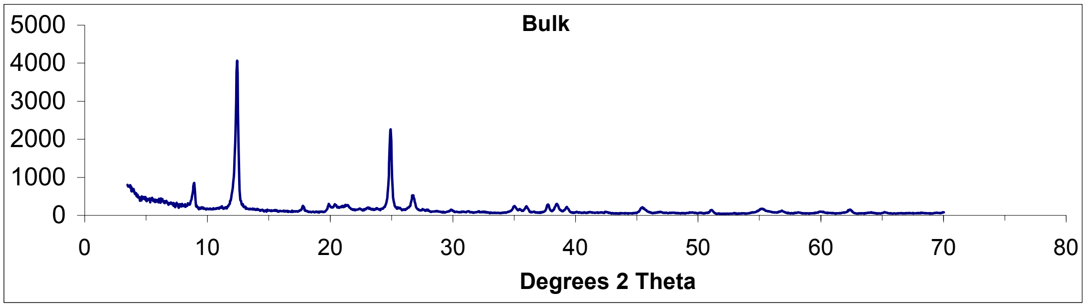
Bulk Composition -	**wt%**
Quartz	3
Feldspar	1
Calcite	2
Dolomite	0
Siderite	0
Pyrite	0
Total Clay	94

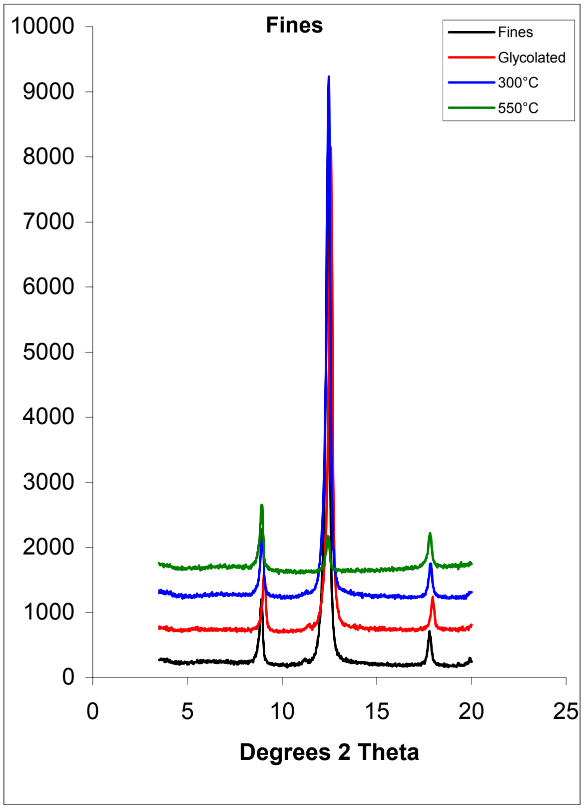
Clay Composition -	**wt%**
Kaolinite	2
Chlorite	4
Illite	7
Smectite	85
Mixed-layer	2
Illite/smectite	5 / 95

Figure 15
Standard of Kaolinite

OGS Laboratory, Inc. - X-Ray Diffraction Interpretation and Data

Project: Kaolin **Sample:** Fisher Kaolin Standard





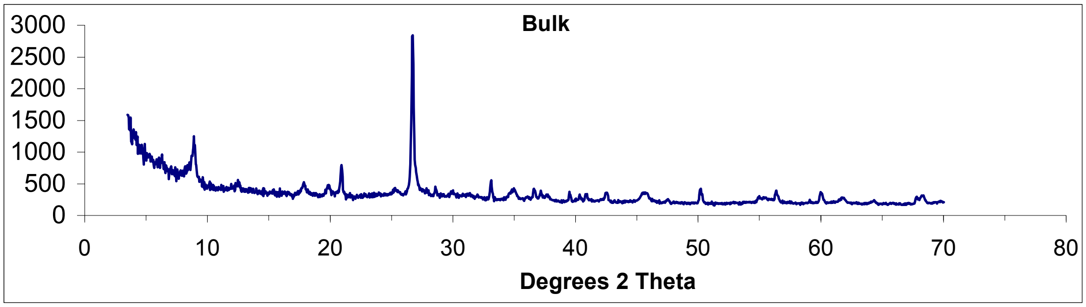
Bulk Composition -	**wt%**
Quartz	5
Feldspar	2
Calcite	1
Dolomite	0
Siderite	1
Pyrite	0
Total Clay	91

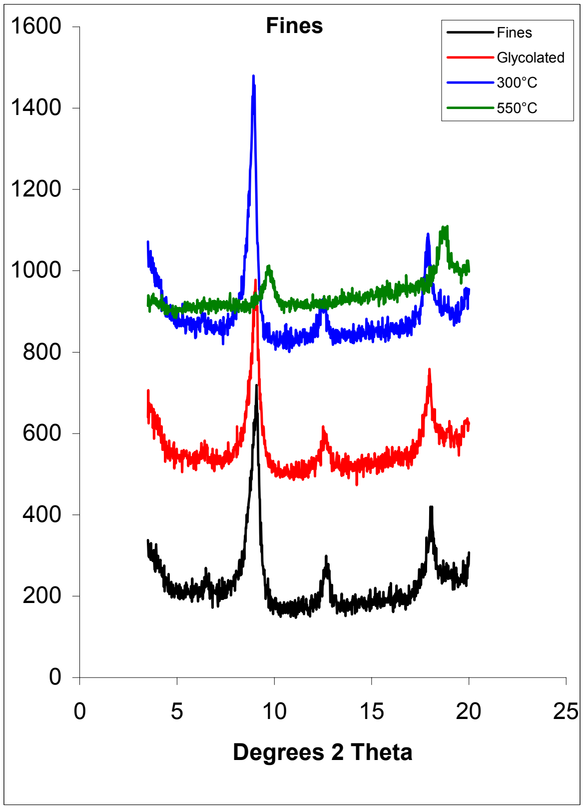
Clay Composition -	**wt%**
Kaolinite	79
Chlorite	0
Illite	12
Smectite	0
Mixed-layer	8
Illite/smectite	40 / 60

Figure 16
Standard of Illite

OGS Laboratory, Inc. - X-Ray Diffraction Interpretation and Data

Project: Illite **Sample:** Illite





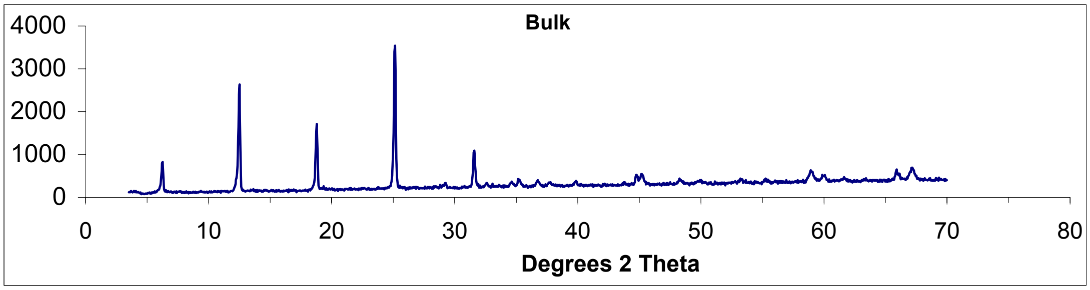
Bulk Composition -	**wt%**
Quartz	20
Feldspar	2
Calcite	2
Dolomite	1
Siderite	2
Pyrite	10
Total Clay	63

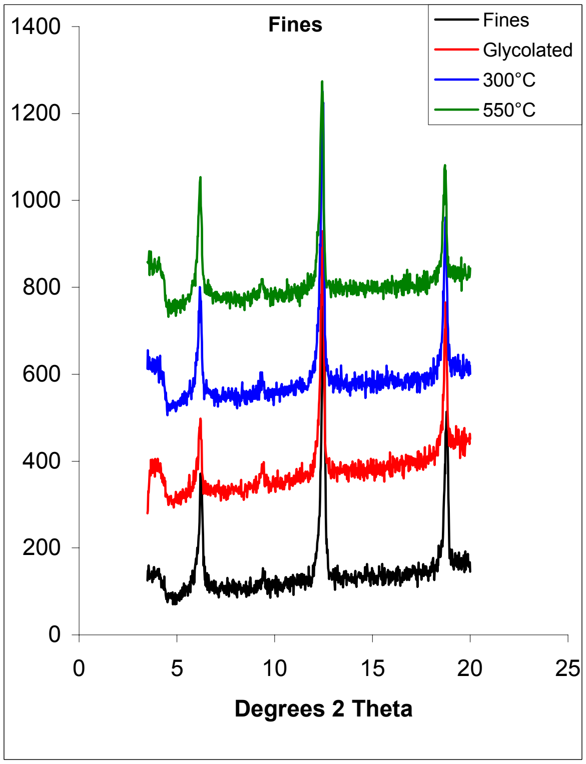
Clay Composition -	**wt%**
Kaolinite	8
Chlorite	3
Illite	56
Smectite	4
Mixed-layer	29
Illite/smectite	35 / 65

Figure 17
Standard of Chlorite

X-Ray Diffraction Interpretation and Data

Project: **Chlorite Std** **Sample:** **1/16/04**
Description: **Chlinochlore**





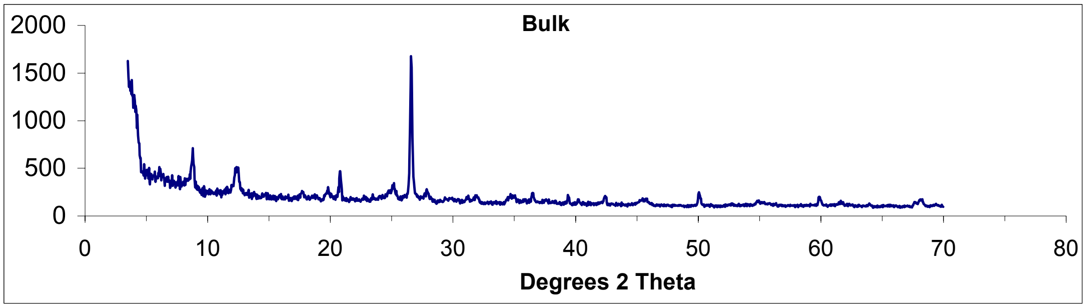
Bulk Composition -	**wt%**
Quartz	2
Feldspar	1
Calcite	2
Dolomite	1
Siderite	0
Pyrite	0
Total Clay	93

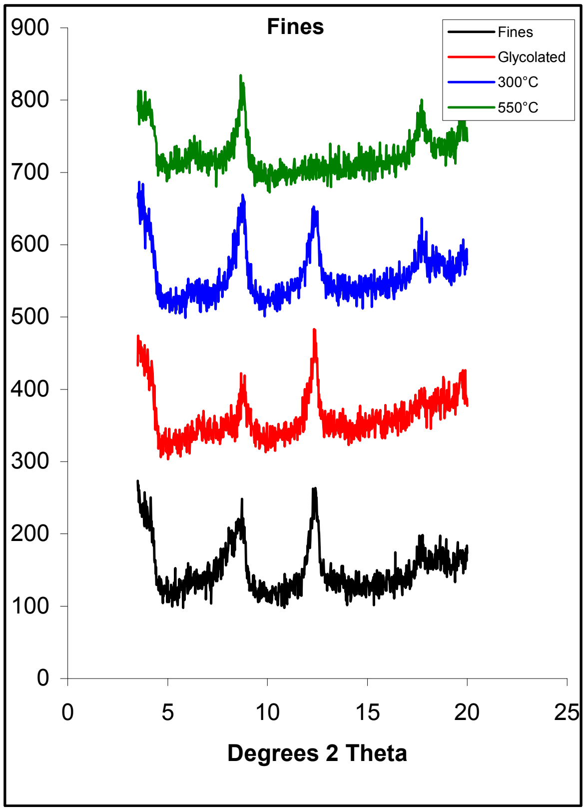
Clay Composition -	**wt%**
Kaolinite	45
Chlorite	17
Illite	19
Smectite	18
Mixed-layer	0
Illite/smectite	20 / 80

Figure 18
Complete Report: XRD & CEC

X-Ray Diffraction Interpretation and Data

Project: **NDFH 1978** **Sample:** **4/11/2003**





Bulk Composition -	**wt%**
Quartz	24
Feldspar	3
Calcite	1
Dolomite	2
Siderite	3
Pyrite	0
Total Clay	67

Clay Composition -	**wt%**
Kaolinite	23
Chlorite	7
Illite	31
Smectite	12
Mixed-layer	28
Illite/smectite	25 / 75

CEC -	**meq/100 g**
	13.1

Exchangeable Bases -	**meq/100 g**
Potassium	2.6
Magnesium	0.5
Sodium	2.2
Calcium	10.8