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CONF-830882 - 5

UCRL--89414

DE83 016574

QUANTITATIVE DETERMINATION OF MINERALS IN NEVADA TEST SITE SAMPLES BY X-RAY DIFFRACTION

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This paper was prepared for submittal to
Second Symposium on Containment of Underground
Nuclear Explosions, Kirtland AFB, Albuquerque,
New Mexico, August 2-4, 1983.

July, 1983

Lawrence
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Abstract

The external standard intensity ratio technique has been developed into a routine procedure for quantitatively determining mineralogic compositions of Nevada Test Site (NTS) samples by X-ray diffraction. This technique uses ratios of X-ray intensity peaks from the same run which eliminates many possible errors. Constants have been determined for each of thirteen minerals commonly found in NTS samples -- quartz, montmorillonite, illite, clinoptilolite, cristobalite, feldspars, calcite, dolomite, hornblende, kaolinite, muscovite, biotite, and amorphous glass. Ratios of the highest intensity peak of each mineral to be quantified in the sample and the highest intensity peak of quartz are used to calculate sample composition. The technique has been tested on samples with three to eleven components representative of geologic environments at NTS, and is accurate to 7.0 wt% of the total sample. The minimum amount of each of these minerals detectable by X-ray diffraction has also been determined. QUANTS is a computer code that calculates mineral contents and produces a report sheet. Constants for minerals in NTS samples other than those listed above can easily be determined, and added to QUANTS at any time.

Introduction

X-ray diffraction (XRD) is a quick and easy technique to identify and quantify mineral content of Nevada Test Site (NTS) samples. Sample preparation is quick, analysis equipment is automated, and run time for each sample is on the order of one hour. Rocks from NTS have a small range of mineralogic composition; there are probably no more than twenty minerals that have been found at NTS. These minerals include quartz, montmorillonite, illite, clinoptilolite, cristobalite, feldspars, calcite, dolomite, hornblende, kaolinite, muscovite, and biotite. Amorphous glass, which is commonly found at NTS, is not a mineral. However, for ease of discussion, it will be included in the list of minerals in this paper. Each sample typically contains five to ten minerals total. Because most of the minerals are found repeatedly in NTS samples, qualitative interpretation of results is straightforward and quickly accomplished. Quantification of the minerals is another story. For the past number of years, Lawrence Livermore National Laboratory has been reporting semi-quantitative results from X-ray diffraction analysis. Three minerals (montmorillonite, a clay; clinoptilolite, a zeolite; and calcite, a carbonate) have been quantified by reporting that the mineral content of a sample falls at or within ranges of tested values by comparing the X-ray intensities of the unknowns to previously run known compositions. All other minerals were reported as occuring weakly, moderately, or highly. These adjectives could not indicate quantity, since intensities from minerals widely differ, and no known values had been tested. For example, 5 wt% quartz may give a "high" intensity peak, while it may require 50 wt% cristobalite to form such a "high" peak. It became obvious that there was a need to solve this confusing problem. Although containment concerns center on determination of clay and carbonate content, determination of total mineral content of a sample is helpful in characterizing the geologic setting. It was necessary to develop a new powder X-ray diffraction procedure to routinely determine mineralogic composition of NTS samples.

X-ray Diffraction Principles

This is a very brief review on XRD principles; for more thorough information the reader should check Klug and Alexander (1954). A monochromatic beam of radiation strikes the sample and the reflections collected by a counter as the scan progresses give information on d spacings (separation of parallel planes in the crystal lattice) and intensities (counts of photons) from single crystals or powdered samples. As the beam strikes the sample, some of it is absorbed and some is reflected. Some reflected beams reinforce each other (the path difference equals a whole number of wavelengths), and Bragg's Law describes the situation, as shown in Figure 1. At other angles θ the beam is not reflected due to interference. Because all minerals have characteristic atomic structures, they can be identified by a unique X-ray diffraction pattern. For example, Figure 2A shows the XRD pattern of pure quartz. Reflections will always occur at certain 2θ angles (or d spacings) and the relative intensities of the reflections will always be in certain ratios to each other. Figure 2B shows a sample that contains quartz with other minerals. Quartz can be identified because reflections from its atomic structure will always occur at the same 2θ angles (or d spacings) and the relative intensities of the reflections are again at the same ratios to each other, as in Figure 2A. No matter how many components are in a sample, if quartz is present it can be recognized by its characteristic XRD pattern. The same is true for all minerals.

The intensities of X-ray diffraction lines may differ due to several reasons -- polarization, Lorentz, temperature, atomic scattering, structure, multiplicity, absorption, and machine factors (Klug and Alexander, 1954). These differences in intensities can have a large effect on quantitative work. But because our X-ray scan is of short length, many of these factors can be assumed to be small and thus require no correction. Choice of an efficient analysis method can reduce other factors.

A theta compensating slit has been added to our X-ray unit. It functions as an automatic divergence slit and parallel (Soller) slit. The purpose of this slit is to keep a constant area of the sample irradiated throughout the scan, as opposed to a fixed divergence slit which allows different areas of irradiation as the scan progresses. There were two reasons for installing the theta compensating slit on our equipment. Since it is difficult to directly obtain comparable intensities due to a number of factors listed above, the next best step is to keep the area of the sample irradiated constant throughout the scan. The same X-ray intensity is always on the same amount of sample and comparison of intensities is easier. The second and more important reason was to give us better data at low angles. We need to identify and quantify clays, which occur at low angles. Figure 3 shows how difficult this could be before the theta compensating slit was installed. The same sample was run twice, once in a fixed divergence slit mode, and the second time in a compensating slit mode. At low angles in the fixed mode (Figure 3A) the X-ray source radiates almost directly into the detector. Background is high at the beginning of the scan, and progressively drops off as the scan continues. Because of the X-ray source peak and the progressively different background values, intensity corrections may need to be done before interpretations can begin. A peak at about 5°2θ is due to 15 wt% montmorillonite. It is difficult to identify this peak in Figure 3A. But in 3B, a scan utilizing the compensating slit, background is relatively constant throughout the scan, and the montmorillonite peak is easier to identify. Installation of the theta compensating slit has made XRD analysis at low angles much easier to accomplish.

Although the theta compensating slit has made examination of NTS samples more direct and less time-consuming, the slit has placed a limitation on us. Widely used relative intensities published by the Joint Committee on Powder Diffraction Standards (JCPDS) cannot be utilized, since this data was obtained under fixed slit conditions. Fixed slit intensities are not directly comparable to those obtained under compensating slit conditions. Also, constants which will be described later in this paper cannot be directly used by others operating under fixed slit conditions. A compensating slit can also be run in a fixed slit mode -- we are not limited to one method of operation. It is important, though, to realize that the intensities collected under these two different operating conditions are two different types of data, and are not directly comparable.

Figure 4 shows a diagramatic equatorial view of an XRD unit. The sample is tangential to the focusing circle. The detector pivots around the sample during a scan. For parafocusing, the sample-detector distance must remain equal throughout the scan. To accomplish this, the sample rotates with one-half the angular velocity as the detector (the sample will be at angle θ and the detector at angle 2θ to the X-ray source). The compensating slit rotates with the sample in order to maintain the constant area of irradiation of the sample throughout the scan.

XRD Quantification Methods

Why are intensities important? Qualitative determinations of minerals are accomplished by identifying peak locations in an XRD pattern, but minerals can be quantified by knowing integrated intensities, densities, and absorption properties of the sample:

$$I_{ij} = \frac{K_j \, x_j}{\rho_j[x_j\,(\mu_j^* - \mu_m^*) + \mu_m^*]} \qquad \text{(Eq. 1)}$$

I_{ij} = integrated intensity of the i^{th} line from component j

K_j = constant dependent on the geometry of the diffractometer and the nature of component j

x_j = weight fraction of component j

ρ_j = density of component j

μ_j^* = mass absorption coefficient of component j

μ_m^* = mass absorption coefficient of the matrix (material that would remain if component j were removed)

Chemical variation and changes in the number of components can change densities and mass absorption coefficients, making this equation difficult to solve. Frequently, μ_m^* is not known, and Equation 2 is used:

$$I_{ij} = \frac{K_j \, x_j}{\rho_j \, \mu_T^*} \qquad \text{(Eq. 2)}$$

μ_T^* = total mass absorption including component j

This equation may require empirical corrections because of microabsorption effects.

If another component is added to the sample μ_T^* drops out of the equation because the intensities are measured on the same sample, and the mass absorption coefficients are characteristic of the sample. Small errors resulting from microabsorption effects can be eliminated using this equation:

$$\frac{I_{ij}}{I_{k1}} = \frac{K_j}{K_1} \frac{\rho_j}{\rho_1} \frac{x_j}{x_1} = \text{constant} \frac{x_j}{x_1} \qquad \text{(Eq. 3)}$$

I_{k1} = integrated intensity of the kth line from component 1

K_1 = constant dependent on the geometry of the diffractometer and the nature of component 1

ρ_1 = density of component 1

x_1 = weight fraction of component 1

These equations were developed by Klug and Alexander (1954), and have been used and modified by many researchers.

There are a number of techniques which utilize these equations for quantification of components in a sample. The first measures the intensity of the unknown and compares it to a pure standard. This requires a pure standard to be run also. The mass absorption coefficients of the standard and the unknown must be known. The second technique involves spiking or diluting the sample after its initial run. The spiking method adds fixed amounts of the component of interest and records the intensity of a XRD line from this component at each amount. The amount of the original sample is extrapolated from this data. The dilution method follows these same steps, except an amorphous dilutent is added. This method requires repeated scans and collection of data for each component of interest. It is also necessary to obtain exactly the same component as the one to be quantified for the spiking method. The third technique is the internal standard method. A fixed amount of a standard material not found in the sample is added. The intensity of a XRD line from the standard is ratioed against the intensity of a line from the unknown, and compared to a calibration curve. The calibration curve is a graph of I_{ij}/I_{kl} plotted against x_j/x_l for samples containing known mixtures of components j and l. At least three points are needed to define the linear calibration curve. The fourth technique is the standardless method. The sample is chemically or mechanically treated to reduce the amount of a component. This method can be used for crystalline components only. A quantitative determination of a component is made by comparing the intensities of XRD lines of the component before and after treatment. This method requires repeated scans for collecting data, and chemical or mechanical treatment of each component in the sample. The external standard is the fifth technique. Ratios of XRD lines of components in the sample are compared to ratios of lines from previously mixed and run standards. This is the method we use for semi-quantitative analysis of montmorillonite, clinoptilolite, and calcite. Standards of known compositions must be made for each component in the sample. The sixth technique, matrix flushing (Chung, 1974) utilizes Reference Intensities ($I_{mineral}/I_{corundum}$) published by the JCPDS, instead of an external standard. The concept is that mass absorption coefficients, which are difficult to measure, are flushed out of the equation, and a simple relationship then exists between intensity and concentration. Corundum is usually added as the flushing agent, but a component found in the sample may be used. This method has proven successful for quantifying amorphous components.

All of these quantitative techniques have been rigorously tested and work well. Some of the techniques require mass absorption coefficients, which can be difficult to accurately determine. They must be determined for each mineral to be quantified. A slight change in chemical composition requires a new coefficient to be determined. It is known that NTS minerals have variable chemical compositions -- for example, both Ca and Na clays exist, and there are various feldspars from the solid solution series. This would require knowing many mass adsorption coefficients, and using the correct one in the equation. Other quantitative techniques require multiple runs for each mineral. This is time-consuming considering the number of samples we may be expected to handle. We would like to quantify glass, an amorphous material. Most of these techniques have only been tested on crystalline materials. Another problem is that approximately twenty minerals have been found at NTS, although only five to ten minerals are commonly found in any one sample. We would like to accomplish the quantification of all minerals in one step. Some of the techniques have been applied to binary systems, while others have successfully handled multi-component systems. But -- the maximum number of components accurately measured by other workers has been four. We need to choose a technique that allows chemical variation of the samples (requires no mass absorption coefficients), can be used routinely (does not require multiple runs for each mineral), and can handle five to ten (or more) minerals at one time. All of the equations included in this paper require that constants be determined. These must not be difficult to measure for the quantification technique we choose.

Goehner (1982) has modified and tested, on a three component system, an external standard intensity ratio technique based on an external standard method formulated by Copeland and Bragg (1958). The weight fractions of each component to be quantified are obtained:

$$\frac{x_j}{x_1} = K \frac{I_j}{I_1} \qquad \text{(Eq. 4)}$$

K is a constant which can be obtained from a single standard of a one-to-one mixture of components i and j (Chung, 1974).

$$\frac{x_j}{x_1} = K \frac{I_j}{I_1} \rightarrow 1{:}1 \text{ mixture} \rightarrow K = \frac{I_1}{I_j} \qquad \text{(Eq. 5)}$$

K values are known for all components in the sample (Equation 5), the sample to be quantified is run once, intensities of specific diffraction lines are ratioed, and Equation 6 is used to calculate the weight fractions of all components in the sample, totaling 100 wt%.

$$\sum_{k=1}^{n} x_k = 1 \qquad \text{(Eq. 6)}$$

n = number of components in sample

x_k = weight fraction of components

Because intensities that are ratioed are obtained from one sample run of short duration (∿ 1hr.), many XRD problems are minimized (Goehner, 1982). These include changes in barometric pressure, machine factors (KeV and Ma drift), long term drift of the X-ray tube (aging), matching mass absorption coefficients, sample problems such as compactness of the powder and sample transparency, and alignment problems like sample positioning in the focusing circle and slight goniometer alignment changes. Several problems not minimized that the worker needs to be concerned with are particle statistics, preferred orientation, microabsorption, extinction, and obtaining a standard similar to the unknown. The first four of these remaining problems can be dealt with by careful experimental planning and sample preparation. The final one, obtaining a standard similar to the unknown, remains a problem. NTS samples vary in composition. Samples with chemical variation would require more than one standard and proper selection of the correct K constant at the appropriate occasion. It is hard to obtain a representative sample from NTS of most minerals in pure form to use as standards. There were initial hopes of obtaining "average" NTS samples for use as standards, but this was impossible. We will have to make an assumption that all samples will react in XRD analysis like the standards used -- the montmorillonite in a sample tested today and one tested in the future will give comparable XRD results as the montmorillonite used as a standard. Experimental work completed after this assumption was made confirms that this is a valid assumption.

The external standard intensity ratio technique was chosen to quantify minerals in NTS samples. It requires no mass absorption coefficients, can be used routinely, has been tested on three component samples, and K constants can be identified by making one standard for each mineral. Many XRD problems are also minimized because we are using ratios of data gathered over relatively short scans. The standards have to be made with one common mineral that will be found in every NTS sample. Quartz was chosen as the common mineral -- in the several hundred samples I have analyzed by XRD, quartz has always been present in some amount. If quartz is not present in a NTS sample, which seems unlikely, this technique will not work, and there are two alternatives -- use another technique, or infer a nominal amount of quartz is present, calculate mineral composition using Equation 6, and then recalculate these values knowing that quartz is not present in the sample.

Experimental Process

Our X-ray unit utilizes a Cu X-ray tube, and machine operating conditions are 45 KeV and 30 Ma. A theta compensating slit is the diverging slit, and there is a 1° receiving slit. The XRD scans cover 2-45°2θ, automatically stepping 0.04°2θ every four seconds. Good counting statistics are obtained from the step size and counting time. Total scan duration is 73 minutes. The scan length obtains much useful data for the mineral suite in a reasonable amount of time. The highest intensity peak of each mineral in question falls within the scan length. Other peaks of these minerals also fall within this length, ensuring positive identification of all minerals present.

There are three experimental parts to obtaining a working XRD procedure for quantifying minerals in NTS samples. K constants have to be determined for each mineral expected in a NTS sample, the external standard intensity ratio technique must be tested and its accuracy determined, and the minimum amount of each mineral that can be detected by XRD must be determined, to place a lower limit on the quantification procedure.

It was necessary to determine optimal particle size of the samples. For good XRD data, particle size must be small, within the range of 5-60 microns. Grinding and sieving samples can require a large amount of time. Also, several minerals tend to form smaller particles (clays and zeolites) while others are difficult to grind (quartz and calcite). It was imperative that the sample preparation process not selectively choose or eliminate any mineral. Samples of known compositions within various size fractions were analyzed by XRD to determine that all minerals were present. The data showed mineral contents as expected. A decision was made to constrain particle size to >35<45 microns. This size fraction would not require excessive sample preparation, and would yield good XRD data.

Thirteen minerals are common in NTS samples. These are quartz, montmorillonite, illite, clinoptilolite, cristobalite, feldspars, calcite, dolomite, hornblende, kaolinite, muscovite, biotite, and amorphous glass. Because it was difficult to obtain pure "typical" or "average" minerals from NTS samples to use in determining K constants, the assumption was made that all samples will react in XRD analyses like the standards used, and pure minerals were obtained from Wards Natural Scientific Establishment, Inc. to use as standards. Standards were made of each mineral in a one-to-one weight ratio with quartz. A K constant was calculated for each mineral using the highest intensity peak of quartz (100 peak = d spacing of 3.34) and the highest intensity peak of each mineral, as in Equation 5. Each standard was analyzed nine times, and the K values were then averaged. These are shown in Table I. The weight fraction of each mineral was then calculated to determine the accuracy of the K constants, using Equation 4. There was a surprisingly large error in calculating weight fractions of all minerals except muscovite. It was obvious that these K constants were not accurate.

These K constants are simply the slope of the linear calibration curve where the standard is composed of equal amounts of quartz and mineral and the weight fraction is equal to 1.0. It was decided to prepare and test more standards of different compositions to accurately determine the calibration curve for each mineral. The K constant (slope) could then be calculated by a least squares fit of the data. Standards were made at 89:11, 80:20, and 33:66 ratios of the mineral to quartz. Standards were also made at 25:75 and 20:80 of illite and muscovite, which required better control where data points were slightly erratic. Each calibration curve was determined by a minimum of eighteen points. The K constants from the slope of the calibration curves are also shown in Table I. These new K constants proved to be much more accurate when calculating weight ratios of standards, with the exception of muscovite. Muscovite contents are most accurately calculated using the mean of the intensity ratio of the 1:1 standards, and this mean will be the K constant for muscovite. For all other minerals, K constants are the slope of the calibration curve.

Sixteen samples of various contents from the list of thirteen minerals were prepared and analyzed by XRD to test the external standard intensity ratio technique and determine its accuracy for quantifying minerals in NTS samples. Sample content ranged from three to eleven mineral components. With the exception of biotite and cristobalite, which were in two and three samples respectively, each mineral was present in at least four different samples. Quartz was present in all. The samples were prepared to be representative of geologic environments found at the NTS. Altered samples were composed of predominately clays and zeolites, fresh samples were composed entirely of quartz, cristobalite, and glass, and there were various intermediate compositions. Equation 6 was used to quantify each sample. The error in quantifying each mineral in a sample and the maximum error for each sample were tabulated and are shown in Table II. The distribution of error is shown in Figure 5. The average error is 0.0 wt%, which was expected knowing that calculated quantities could deviate above or below the known value. The standard deviation of this error is 2.89 wt%. The maximum error for this technique is ± 7.0 wt%. This maximum error occurs approximately one third of the time (5 out of 16) and is the most conservative method of reporting error in the technique. For this reason, the error in the external standard intensity ratio technique for quantifying minerals in NTS samples is ± 7.0 wt%.

The final experimental phase was to determine the minimum amount of each mineral that could be detected by X-ray diffraction. This would place a lower limit on the quantification procedure based on what could actually be observed in the XRD data. Each XRD pattern contains a small amount of background. The analyst must be able to detect at least one peak (preferably two or three) from a minerals pattern above this background level to positively identify the presence of that mineral. The minimum amount of each of the minerals detectable by XRD is listed in Table III. With the exception of glass, all minerals can be accurately identified below 10 wt%, and some as low as 0.5 wt%. The minimum amount of glass detectable is 40 wt%. This is a much higher amount than the other minerals. Glass is an amorphous material, and is identified by XRD as a wide hump extending from approximately 19 to 36°2θ. Small amounts of glass (less than 40 wt%) are difficult to distinguish from normal or abnormal background levels. Lithologic information and/or knowledge of the entire mineral composition of the sample will help indicate if much glass is present in a sample. For example, knowing that the sample came from a vitrophyre would give a high probability that the sample composition included glass, and a highly altered sample would most likely contain clay and zeolite, with small amounts of glass, if any at all.

The minimum amounts of minerals detectable shown in Table III are the true minimum that can be observed in the XRD data. It is possible that a content lower than these values may be calculated using Equation 6. It must be remembered that the calculated values are based on what is actually observed in the XRD pattern with an error of ± 7.0 wt%.

QUANTS

QUANTS is a computer code written to calculate mineral contents based on Equation 6 and produce a report sheet. K constants for each mineral are stored in the code. An input file consisting of drill hole identification, sample date, XRD date, and sample information must be made. The sample information includes depth in feet (depth in meters is calculated), sample type (cuttings, sidewall, or percussion gun), and ratios of the highest intensity peak of each mineral present and the highest intensity peak of quartz -- the same two peaks of the sample that were used for calculating the K constant of the standard. If a mineral is not present in a sample, a value of 0.0 must be entered. These values must occur in the input file in the same order they occur in on the report sheets. The input file and the XRD data file are stored for easy computer access. A report sheet is shown in Figure 6.

Routine XRD Procedure

NTS samples submitted for X-ray diffraction are crushed and sieved, the size fraction from >35<45 microns is X-rayed under the machine conditions previously specified, and the raw data is reduced by an analysis code and displayed in a readable manner (Goehner, 1982). The analyst then identifies the minerals present. Glass is identified by subtracting out the background of the scan, and determining if the subtracted data represents glass or background. Occasionally some peaks will be composed of more than one mineral and will need to be deconvoluted (namely illite, muscovite, and biotite). The highest intensity peak of each mineral present and the highest intensity peak of quartz (the same two peaks of the sample that were used to calculate the K constant of the standard) are ratioed, and an input file is made for QUANTS. The code is run, and a report sheet is issued. The input file and the XRD data file are stored for future use.

Conclusions

The external standard intensity ratio technique has been successfully tested to quantify minerals in NTS samples. K constants have been determined for thirteen minerals commonly found in NTS samples -- quartz, montmorillonite, illite, clinoptilolite, cristobalite, feldspars, calcite, dolomite, glass, hornblende, kaolinite, muscovite, and biotite. The quantification of these minerals is accurate to ± 7.0 wt%. The minimum amount of each mineral that can be detected by X-ray diffraction has also been determined. These are listed in Table III.

Discussion

It is possible that minerals other than those listed above may be found in NTS samples. In order to accurately quantify all minerals in a sample, a K constant for each mineral must be stored in QUANTS. New K constants can easily be determined, and then added to the code at any time.

The establishment of this technique as a routine procedure for quantifying minerals is an accomplishment in several ways. First, it eliminates other methods that produced semi-quantiative results for selected minerals. We can now accurately quantify all minerals in NTS samples. Secondly, most analysts who utilize X-ray diffraction are attempting to identify and quantify chemical compounds. We have switched this emphasis to commonly found minerals. Finally, we have stretched the bounds of quantitative techniques. Most of these techniques have been developed, tested, and utilized on systems containing two to three components. The external standard intensity ratio technique has now been tested and will routinely be used on systems containing up to eleven components from a list of thirteen commonly found minerals. There is a possibility that this technique can be expanded even further.

Acknowledgments

I would like to thank Bill Beiriger and Bern Qualheim for the large amount of time they spend helping with sample preparation. Norm Burkhard was instrumental in writing the code QUANTS. I also appreciate the suggestions and editorial comments made by Larry McKague.

*This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

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List of figures and Tables

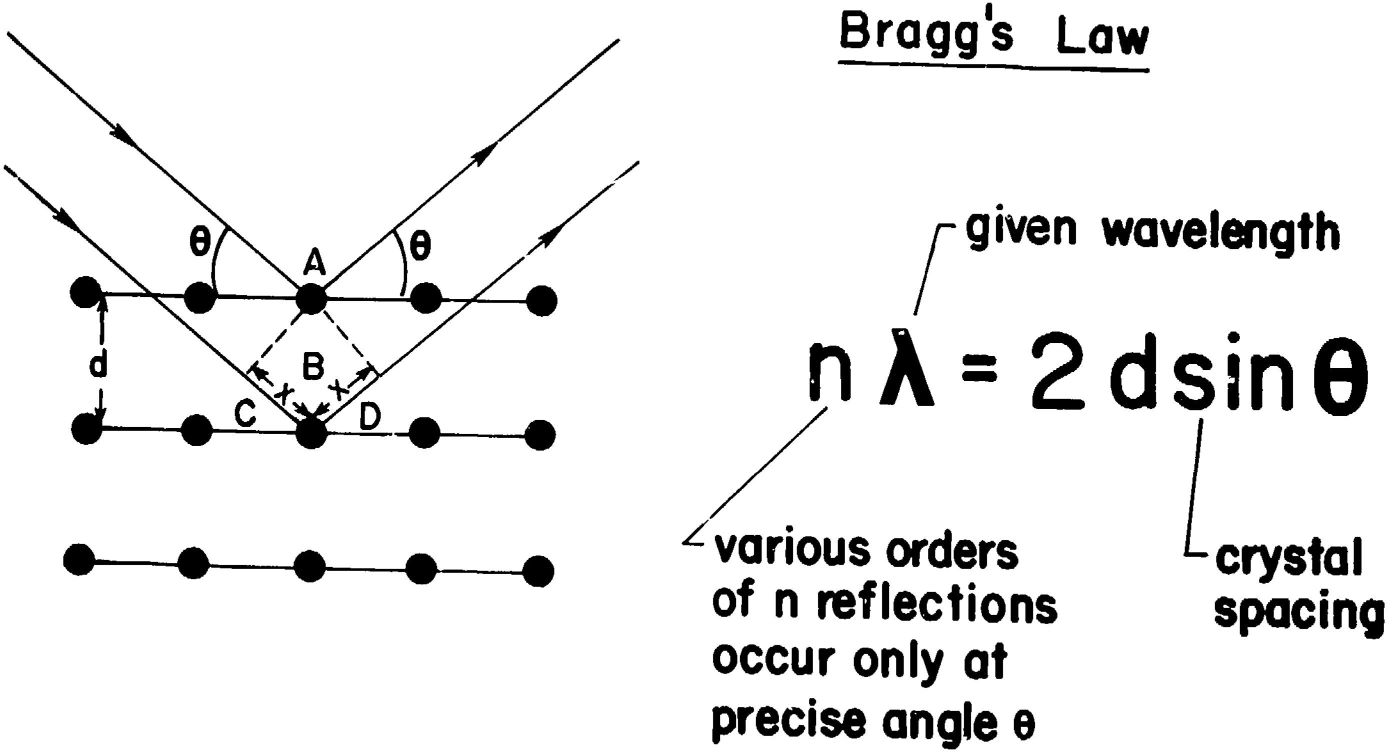

Figure 1. For a given d spacing and a given wavelength λ, the various orders n of reflection occur only at precise values of angle θ, which satisfy Bragg's Law.

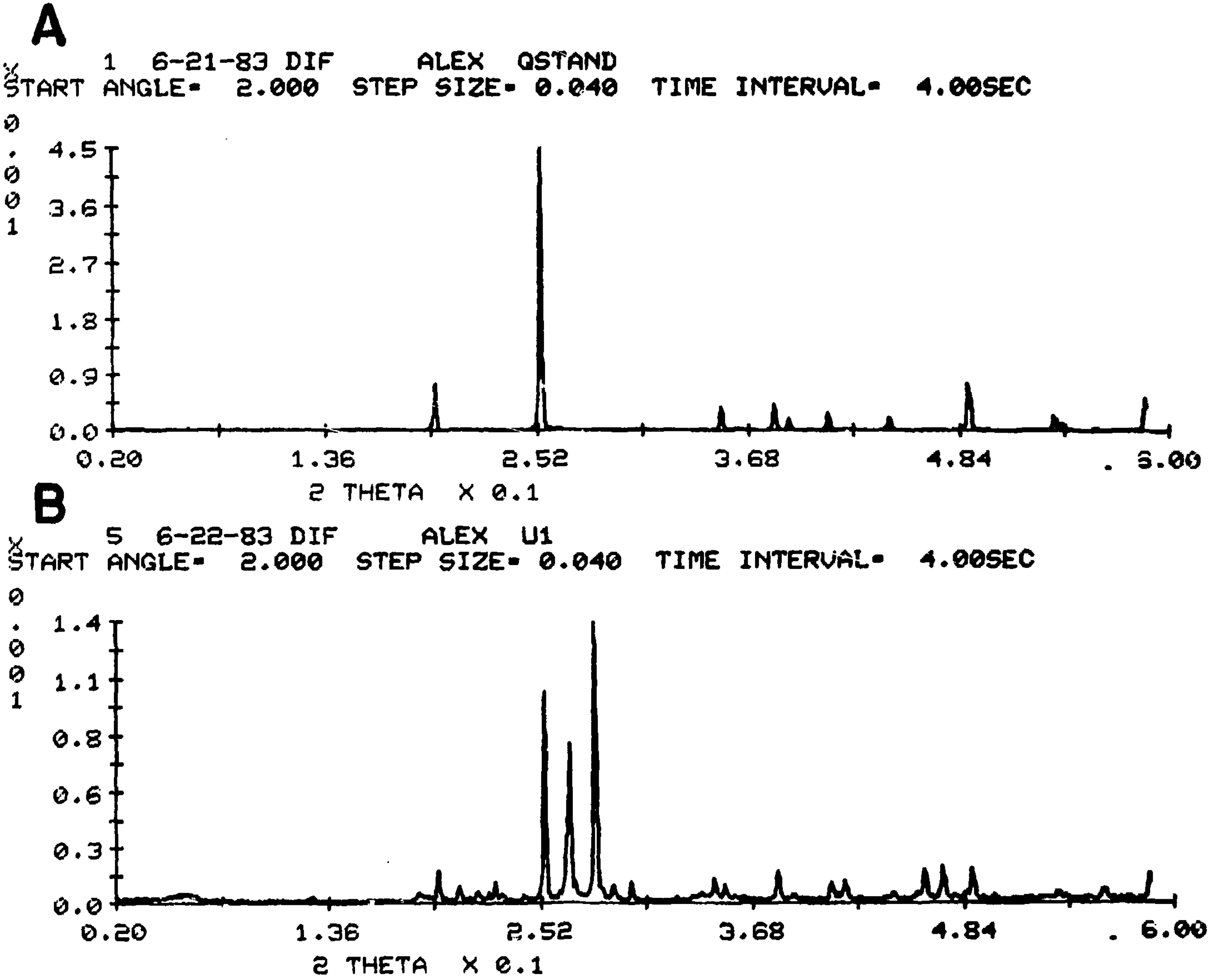

Figure 2. Quartz has a unique XRD pattern (A) due to its characteristic atomic structure. This unique pattern allows identification of quartz in samples composed of quartz and other minerals (B).

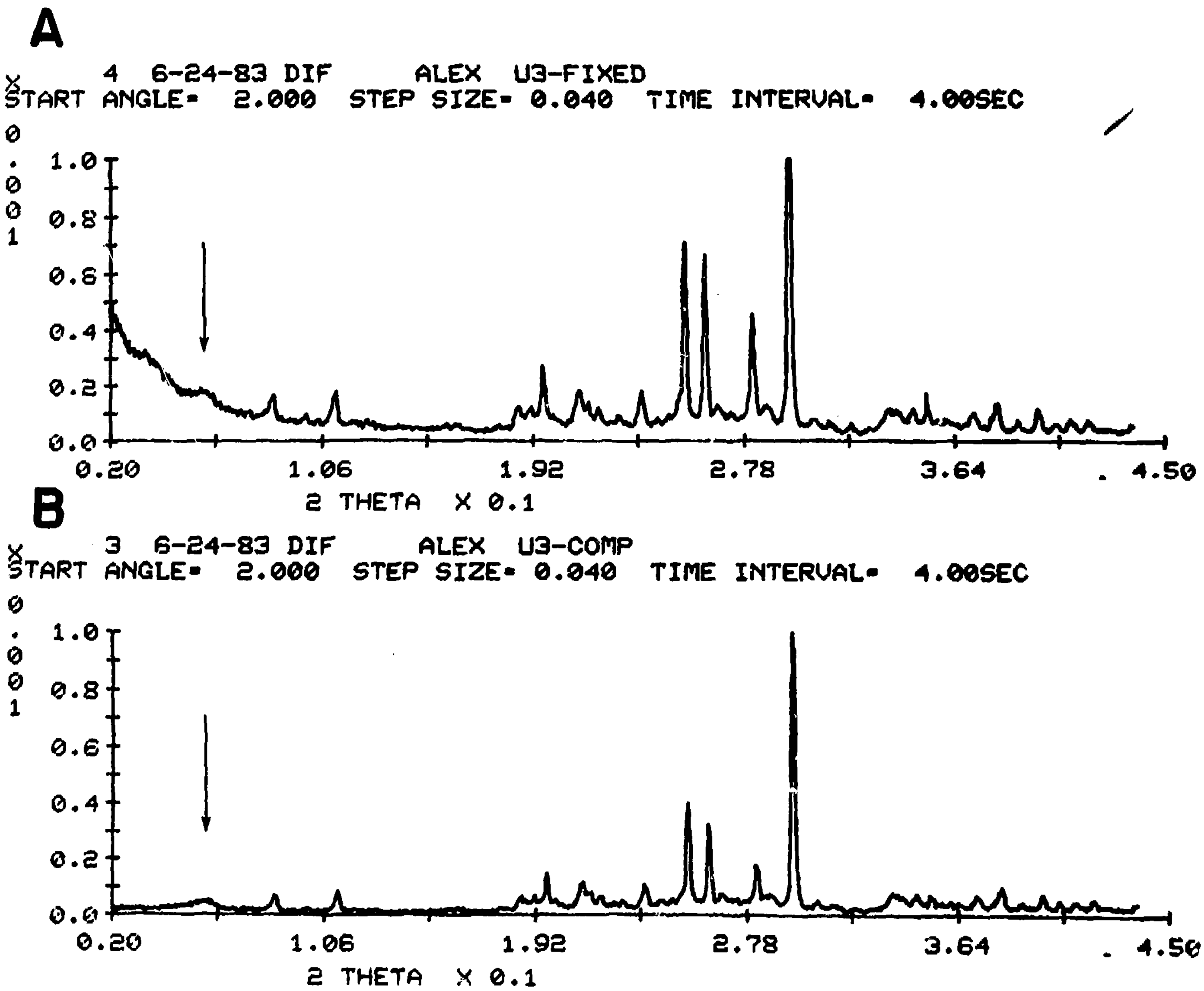

Figure 3. A fixed slit scan (A) shows progressively decreasing background. A montmorillonite peak (~5°2θ) is difficult to distinguish. A theta compensating slit scan (B) shows relatively constant background, and the montmorillonite peak is easier to identify.

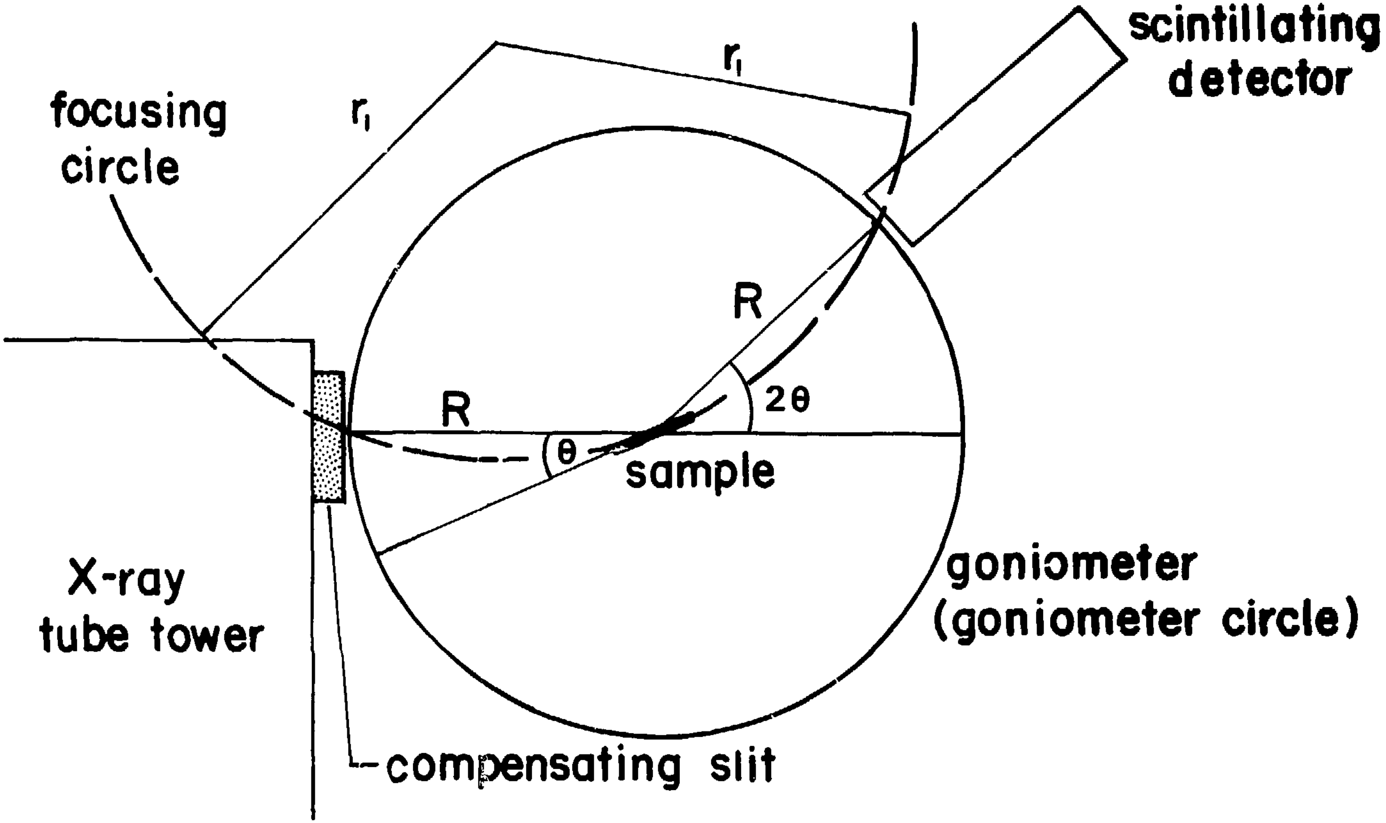

Figure 4. Diagrammatic equatorial view of an XRD unit.

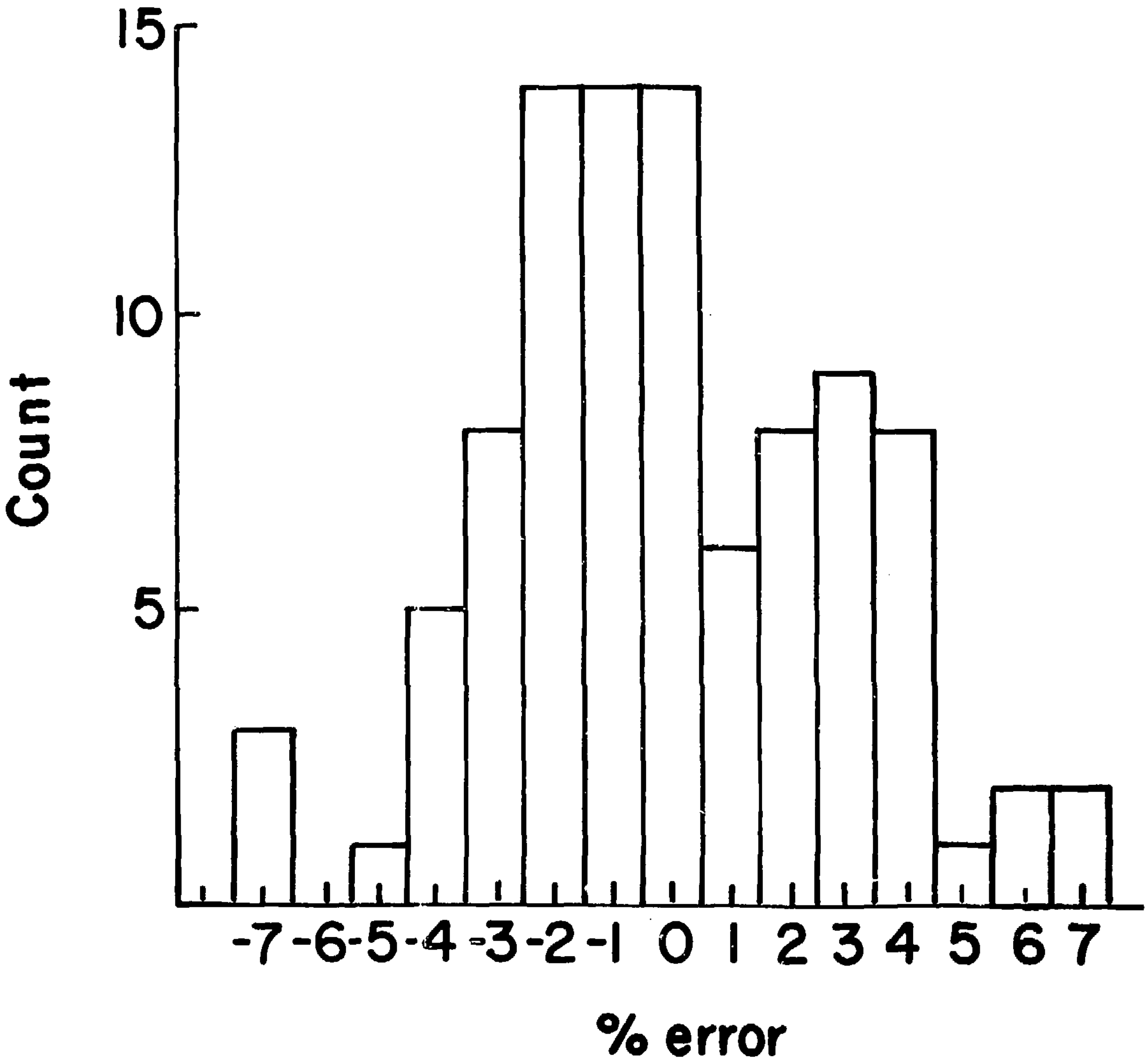

Figure 5. Distribution of error in quantifying known sample contents by the external standard intensity ratio technique.

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                              XRD ANALYSES OF SAMPLES FROM UE4AF

  DEPTH
 FT    M     *   QUAR   MONT   ILLI   CLIN   CRIS   FELD   CALC   DOLO   GLAS   HORN   KAOL   MUSC   BIOT
----------------------------------------------------------------------------------------------------------

  200.  61.  S    71.     0.     0.    11.     0.     9.    9.7   0.2     0.     0.     0.     0.     0.
  300.  91.  S    71.     0.     0.     6.     0.    10.    7.6   0.      0.     0.     5.     0.     0.
  475. 145.  S    47.    19.     0.    12.     0.     9.   11.4   0.4     0.     0.     0.     0.     0.
  550. 168.  S    43.    34.     0.     0.     1.     8.    6.4   0.      0.     0.     7.     0.     0.
  650. 198.  S    13.    19.     0.     4.     0.    58.    5.1   0.      0.     0.     0.     0.     0.
  700. 213.  S    25.     4.     0.     0.     0.    45.   22.3   2.1     0.     0.     0.     1.     0.
  825. 251.  S     5.    82.     0.     0.     2.    10.    1.3   0.      0.     0.     0.     0.     0.
  850. 259.  S     4.    45.     0.     0.     2.    21.   28.3   0.2     0.     0.     0.     0.     0.
  925. 282.  S     5.    42.     0.     7.     2.    44.   0.     0.      0.     0.     0.     0.     0.
  930. 290.  S     3.    86.     0.     0.     1.    10.   0.2    0.      0.     0.     0.     0.     0.
 1000. 305.  S     1.    81.    13.     0.     1.     4.   0.     0.      0.     0.     0.     0.     0.
 1025. 312.  S     4.    80.     0.     0.     1.    12.   2.5    0.1     0.     0.     0.     0.     0.
 1050. 320.  S     2.    82.     0.     0.     2.    14.   0.     0.      0.     0.     0.     0.     0.
 1100. 335.  S     6.    78.     0.     0.     3.    11.   2.0    0.      0.     0.     0.     0.     0.
 1175. 358.  S     2.    87.     0.     0.     2.     9.   0.     0.      0.     0.     0.     0.     0.
 1220. 372.  S     8.    51.    13.     5.     2.    20.   0.     0.      0.     1.     0.     0.     0.
 1240. 378.  S     2.    75.     0.     0.     2.    20.   0.4    0.      0.     0.     0.     0.     0.
 1300. 396.  S     2.    67      0.     0.    23.     8.   0.     0.      0.     0.     0.     0.     0.
 1320. 402.  S     1.    18.    10.     0.     1.    15.   0.     0.     56.     0.     0.     0.     0.
 1340. 408.  S    12.    54.    24.     0.     2.     8.   0.     0.      0.     1.     0.     0.     0.
 1360. 415.  S     3.    22.    32.     0.     4.    26.   11.5   0.2     0.     1.     0.     0.     0.
 1400. 427.  S     4.    41.    15.     0.     1.    39.   0.     0.      0.     0.     0.     0.     0.
 1440. 439.  S    82.     0.     0.     0.     0.     4.   0.     0.5     0.     0.    13.     0.     0.
 1460. 445.  S    63.     0.     0.     0.     0.    16.    1.9   0.5     0.     0.    18.     0.     0.

TOTAL NUMBER OF SAMPLES:  24                MINERALS                                  ANALYST:   GAYLE PAWLOSKI
                               QUAR = QUARTZ                 DOLO = DOLOMITE           SAMPLE DATE:   JUNE 2,1974
                               MONT = MONTMORILLONITE        GLAS = GLASS              XRD DATE:   APRIL 9,1983
                               ILLI = ILLITE                 HORN = HORNBLENDE
  * INDICATES SAMPLE TYPE      CLIN = CLINOPTILOLITE         KAOL = KAOLINITE
      C = CUTTINGS             CRIS = CRISTOBALITE           MUSC = MUSCOVITE
      G = PERCUSSION GUN       FELD = FELDSPARS              BIOT = BIOTITE
      S = SIDEWALL             CALC = CALCITE
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Figure 6. The computer code QUANTS quantifies the mineralogic composition and produces a report sheet.

Mineral	K constant 1:1 ($\bar{x}$)	 slope of calibration curve
Quartz	1.0000	1.0000
Montmorillonite	23.8202	22.0412
Illite	50.6306	30.2904
Clinoptilolite	12.2582	9.7432
Cristobalite	1.4695	1.2940
Feldspars	1.3267	1.2774
Calcite	0.6790	0.6544
Dolomite	0.4901	0.3528
Glass	52.7867	36.8405
Hornblende	3.2370	2.7698
Kaolinite	10.5109	10.5970
Muscovite	2.5758	1.9180
Biotite	0.3694	0.4304

Table I. K constants for minerals commonly found in NTS samples, determined by two different methods.

Sample	Q	MO	IL	CC	CR	FS	CA	DO	GL	HO	KA	MU	BO	Maximum Error
1	+4	-2				+1	-3							+4
2	+4	+2				-4	-1							±4
3	+2	-3	-1	-7		+5	+3	+3			-1			-7
4	+4	-2		-1		+2		0		-1		-2		+4
5	+4	-4	+7	-3		+2		0		-2	-2	+2	-4	+7
6	+4	-3	0	0		+3		+1		-2	-3			+4
7	+6			-7						-1	+1	+1		-7
8	+7		-2			+3	-2	-4			0	-3		+7
9	-1	0		+1		+4	+1	0		-2	0		-3	+4
10	+6	-7	+3	-5		+3								-7
11	0					+4			-4					±4
12	+2	-1				-1		-1						+2
13	0			-1		+4			-2					+4
14	0	+3		-2	+2	-2								+3
15	+3		-3		-2	0	0	-1		-1	+3			±3
16	+2				0	-2			-1					±2

$\bar{x}$ = 0.0 wt% s = 2.89 wt% n = 95

Q = Quartz
MO = Montmorillonite
IL = Illite
CC = Clinoptilolite
CR = Cristobalite
FS = Feldspars
CA = Calcite
DO = Dolomite
GL = Glass
HO = Hornblende
KA = Kaolinite
MU = Muscovite
BO = Biotite

Table II. Error in quantifying known sample contents using the external standard intensity ratio technique.

Quartz	0.5 wt%	Dolomite	0.5 wt%
Montmorillonite	5.0	Glass	40.0
Illite	7.0	Hornblende	2.0
Clinoptilolite	5.0	Kaolinite	5.0
Cristobalite	1.0	Muscovite	3.0
Feldspars	2.0	Biotite	5.0
Calcite	0.5		

Table III. Minimum amount of mineral detectable using X-ray diffraction.