NYE COUNTY NUCLEAR WASTE REPOSITORY PROJECT OFFICE

TECHNICAL PROCEDURE

TITLE: **X-RAY DIFFRACTION PROCEDURE**	**Revision: 0** **Date: 10-15-00** **Page: 1 of 7**
PROCEDURE No.: **TP-8.6**	**SUPERSEDES:** **DRAFT, 02-25-95**
APPROVAL 12.11.00 **Project Manager Date**	**CONCURRENCE** **Principal Investigator Date** 15 DEC 2000 **Project Quality Assurance Officer Date**

1.0 PURPOSE

The purpose of this procedure is to provide for the collection of x-ray diffraction data of minerals from volcanic rocks and sediments from Yucca Mountain, or other samples from other locations. The methods, procedures and documentation are described.

2.0 SCOPE

This procedure is used to produce x-ray diffraction data using the services of subcontracted chemical and/or materials science laboratories. Samples that are sent out for x-ray diffraction analysis will be prepared for analysis by GMI laboratory personnel or other personnel trained to this technical procedure. This procedure provides for sample tracking, calibration, sample preparation, and x-ray diffraction data reduction.

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2.1 APPLICABILITY

This procedure applies to NWRPO principal investigators (PI) performing x-ray diffraction analyses for the NWRPO.

2.2 TRAINING

GMI personnel trained in this procedure by the PI shall use this procedure. Outside subcontracting laboratories will use those personnel that they have trained to run their equipment. Internal standards shall act as quality control items to determine acceptance or rejection of data

3.0 DEFINITIONS

N/A

4.0 RESPONSIBILITIES

4.1 The Principal Investigator is responsible for the preparation of this procedure.

4.2 The Principle Investigator is responsible for the oversight of the performance of this procedure. The PI is also responsible for tracking of samples by use of a Transfer of Custody Form. He/she is also responsible for the sample preparation and the reduction of x-ray diffraction data.

4.3 The subcontracting laboratory is responsible for alignment and calibration of the diffractometers that it uses for the analysis of materials. That laboratory is responsible for tracking the samples and records in its possession

5.0 PROCESS

Calibration - The subcontracting laboratories will calibrate their own equipment using their own procedures. Intensity calibration is not necessary since internal standards are in use. The internal standards will also provide accuracy of the 2θ angle spacing.

Handling, Shipping and Storage Requirements -A unique identifier number shall be attached to each and every sample. When samples are shipped a list of the sample numbers for the samples in the shipment shall be checked. The receiving laboratory will fill out a sample transfer of custody form, a copy of which will be returned by FAX upon receipt of the samples. Samples returned with laboratory data shall also be recorded.

After laboratory analysis is complete all sample material will remain in storage. The X-ray Analysis Form shall also track the location of each sample.

Background – Bragg, (1937)[1] discovered the internal atomic arrangement in crystals by the use of x-ray diffraction analysis. Diffraction analysis is based upon the reflection of x-rays by the atomic planes in the crystal lattice through an angle, which is related quantitatively to the distance of separation of the atomic planes giving rise to Bragg's law:

$$n\lambda = 2d \sin \theta$$

Where n is an integer, λ (Lambda) is a collimated beam of parallel monochromatic x-rays with a specific wave length, d is the spacing of the (hkl) plane of the crystal lattice, and the angle θ (theta) is the striking angle of the radiation to the (hkl) plane. The diffracted x-rays are emitted from the crystal (diffracted) at the same angle θ with respect to the spacing of the planes of that crystal lattice as the incoming beam of x-ray radiation. Thus:

$$\theta = \sin^{-1} (n\lambda/2d(hkl))$$

The magnitude of the theta angle is determined by d, the distance between the successive crystal planes of the crystal, which cause the diffraction. Intensity of the diffracted beam is dependent upon the distribution of atoms with in the lattice structure. The magnitude of the angle theta and its intensity are specifically characteristic of that crystal structure. Obtaining this information gives rise to a mineral identification.

There are various methods of x-ray diffraction and even electron diffraction. The method commonly used during this procedure will be x-ray diffraction of random powder packed slides. Relative intensity data obtained by x-ray diffraction varies with the randomness or the degree of orientation of individual crystals in the sample. Samples with preferred orientation (such as sedimented clay slides that create a preferred orientation along the c-axis are good to use for clay identifications, but not for combined or mixed mineral species due to the differences that one mineral has from another with respect to aligning itself in a preferred orientation. Electron diffraction may be used on a transmission electron microscope (TP-8.7). Clay identifications may use oriented slides specially made for that purpose that have mineral size characteristics of less than 2 microns.

5.1 PROCEDURE FOR RANDOM POWDER PACKED SLIDES

5.1.1 Equipment

The laboratory will determine the type of x-ray generator used during this procedure. A goniometer paper trace record will be made for each sample. The type of instrument and its settings will be recorded by the laboratory and provided with the x-ray traces. Normally the equipment will be a Philips or Siemens generator, with nickel filtered

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[1]. Bragg, W.L., 1937. Atomic Structure of Minerals. Cornell Univ. Press, Ithaca, New York.

copper K- alpha radiation, set at 20 MA and 40 kV, run from about 5 degrees 2 theta to 60 degrees 2 theta at 2 degrees per minute.

5.1.2 Sample Preparation

Cuttings samples will be processed by a milling procedure in TP-8.2 where the sample will be run down to 0.2 mm grain size using a Webber pulverizing mill. An appropriate amount of this sample will be hand ground in a ceramic mortar and sieved in a 62.5 micron US standard sieve. Two (2) grams of the coarse silt and smaller fraction will be mixed with 0.2 grams of powdered fluorite which will serve as an internal standard for all powder pack runs. The fluorite will be ground to the same grain size prior to mixing.

This sample will be packed in a small plastic locking bag and a full sample and split sample number will be written on this bag along with the date. These data will be recorded in the laboratory notebook, main logbook, and on *the X-Ray Diffraction Analysis Form.*

5.1.3 Internal Standard

If fluorite may occur naturally in the sample to an extent of about 5% or greater, then corundum powder (1.0 micron size) will be used as the internal standard. The amount of mixing should be about 0.4 grams of Corundum to a 2.0-gram sample. This information needs to be recorded. All powder pack samples will have an internal standard. This will also act as a quality control for the consulting laboratory.

5.1.4 Powder Packed Slides

The laboratory running the x-ray diffraction analysis will make a powder packed slide using an aluminum plate with a shallow well. The well mount will be the standard configuration for powder x-ray diffraction.

5.1.5 Data Reduction and Interpretation

X-ray trace records will be picked for peaks and the following data will be recorded: degrees 2θ, the d spacing obtained* from the $^{o}2\theta$ by use of $d = \lambda / 2\sin(\theta)$. The intensity of the known internal standard will provide an intensity calibration since the quantity of the total amount of standard added to the sample is known. The analysis will be constrained to a total of 100%. Intensity data will be collected from each peak. The d spacing and the intensity data will be compared to similar data in the JCDPS files **.

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* The wavelengths (λ) for copper radiation are:

$K_{\alpha} = 1/3(K_{\alpha 2} + K_{\alpha 1}) = 1.5418\ ^{o}A$

$K_{\alpha 1} = 1.54050\ ^{o}A$

$K_{\alpha 2} = 1.54434\ ^{o}A$

** Mineral Powder Diffraction File, Data Book (Joint Committee on Powder Diffraction Standards, Swarthmore, Pennsylvania1983) 1005 pages or later versions.

5.2 PROCEDURE FOR ORIENTED SLIDE X-RAY DIFFRACTION

5.2.1 This procedure will be used for the identification and determination of relative percentages of clay minerals if additional data beyond 5.1 is required. The procedure used for sample preparation and x-ray diffraction analysis is described in:

Biscaye, P.E. 1964. Mineralogy and sedimentation of the deep-sea sediment fine fraction in the Atlantic Ocean and adjacent seas and oceans, in: Geochem. Tech. Rept. 8: Ph.D. dissertation, Yale Univ.

Two-grain size fractions will be made by sample grinding and sieving: 2 - 20 μ and < 2 μ. Two oriented slides of each size fraction will be made by pipetting (deionized water) dispersed slurry onto biological microscope glass slides. Slides will be dried at room temperature or with the use of infra-red lamps.

Low-resolution scans will be made at $2^{\circ}2\theta$/ min. scanning speed.

High-resolution scans will be made at $1^{\circ}2\theta$/ min on the chart.

Each glass slide will be x-rayed using three conditions:

- 2° to $38^{\circ}2\theta$ at about 35 % relative humidity under fast scan conditions.
- The same slide is then glycolated by the [2]Brunton (1955) vapor method and x-ray again under fast scan conditions. If mixed-layer material data is not identified further then this scan will run from 2° to $14^{\circ}2\theta$. If mixed-layer material is going to be identified then the scan can go to $38^{\circ}2\theta$.
- Finally, the slide is run by slow scan through several limited angles. These are run under room atmospheric conditions on the unglycolated slide except for the case where there are considerable mixed-layer peaks in the 3.5°A region. All 2-20μ slides are to be slow scanned from 17° to $26^{\circ}\,2\theta$, all < 2μ slides are to be slow scanned from 24° to $26^{\circ}2\theta$.

Data reduction is made on the basis of section 5.1.5. All slides will have internal standards.

6.0 REFERENCES

NWRPO Quality Assurance Program Plan

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[2] Brunton, G., 1955. Vapor Pressure Glycolation of Oriented Clay Minerals, American Mineralogist, V. 40, p. 124-126.

7.0 RECORDS

Laboratory notebook for X-ray Diffraction.
X-Ray Analysis Form
    Sample Transfer of Custody Form
    Main logbook sample tracking
    X-ray diffraction records for each sample

8.0 ATTACHMENTS

A: X-Ray Analysis Form

X-RAY ANALYSIS FORM

Sample number:________________________ **Organization:**____________________

Name:____________________**Date:**___________ **Date sent to lab.:**______________

X-Ray Laboratory:_________________________**Date Data Returned:**___________

Equipment and its settings: ____

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___ ______________________________________**Date Samples Returned:** _________

Sample Transfer of Custody Form:____

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Sample Preparation

1. Webber Mill (0.2 mm) grains Y _______ N ________ Other ____________________
2. Hand mortar Ceramic (other ____________) ___________________________________
3. Sample Split Number: ____
4. Weight of sample at start of hand mill: _________________________________
5. Weight of sample at end of hand mill to be sent to lab.: 2 grams ______ Other _________
6. Grain size of hand mill after sieving: < 62.5µ ___ 20-2µ___<2µ___ other ___________
7. Internal Standard: 0.2 or______ grams of Fluorite; or 0.4 or______ grams of corundum
8. Final material bag label: ____
9. Number of slides to be made form this material and type of slides and run data:____

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Data Reduction

Number of traces received from lab. and types: _______________________________

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List results from each trace (use additional paper as required):

Slide sample ID Degrees 2θ d space (I/I^{O}) Slide sample ID Degrees 2θ d space (I/I^{O})

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