WASTE ISOLATION PILOT PLANT
Sandia National Laboratories

Interim Change Notice (ICN)

Form Number: 419-A **Effective: 2/7/97**

Procedure: QAP 5-1 Revision 3 Page 1 of 1

ICN Number 1 (Reminder: 6 ICNs max)

Document Type and Number TOP-552 Current Revision 0

Title Calibration , Use and Maintenance of Philips XRG 3100 X-ray Generator

Description of change: Minor: x Non-Minor: ☐

8.2 VLR 6/16/97

1.) In section 6.3 ,third sentence, change 0.02 degrees to 0.2 degrees.
2.) In section ~~8.2~~ ,last paragraph, add new next-to-last sentence reading: At the end of each spectrum verify that the instrument is in calibration by comparing the mechanical and electronic degree readouts. Document the comparison by recording the values in the scientific or laboratory notebook.

Rationale for change:
1.) Correct typographic error
2.) This will provide documentation that for each spectrum the instrument was in calibration for that specific test.

(Identify the ICN by number next to the affected paragraph, insert change, if desired, and file ICN with the affected document.)

For minor ICNs:

Author James T. Baumhardt Date 6/11/97

Subject Matter Expert Hans W. Papenguth Date 6/16/97

QA Reviewer Nancy J Lo-Lloyd Date: 6/16/97

For non-minor ICNs:

Department Manager _____ Date _____

SNL WIPP Project Manager _____ Date _____

Note: Signatures on this document indicate that all comments have been resolved and incorporated.

SNL WIPP
CONTROLLED DOCUMENT

Effective Date 6/16/97

(If Numbered in Red Ink)

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SANDIA NATIONAL LABORATORIES
GEOCHEMISTRY DEPARTMENT 6118
WASTE ISOLATION PILOT PLANT PROJECT

TOP-552

CALIBRATION, USE AND MAINTENANCE OF PHILIPS XRG 3100 X-RAY GENERATOR

Revision 0

Effective Date: 3/14/96

Author:	Jim Krumhansl	Signature	Date 2/26/96
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PI Approval:	Hans W. Papenguth	Signature	Date 3/1/96
QA Approval:	DEBORAH COFFEY	Print / Signature	Date 3/13/96
Records: Center Approval	Peggy Warner	Print / Signature	Date 3/14/96

1.0 REVISION HISTORY

None. This is an entirely new document.

2.0. PURPOSE

This procedure provides for the calibration, operation and maintenance of the Philips XRG 3100 X-ray Generator as part of the laboratory geochemistry research activities in support of the Waste Isolation Pilot Plant (WIPP) Project.

3.0 SCOPE

This procedure is applicable only for the Philips XRG 3100 X-ray Generator.

This document is not meant to substitute for the manufacturer's instruction manuals for the Philips XRG 3100 X-ray Generator. The user is responsible for reading and understanding these manuals (see references).

4.0 SAFETY

This document does not address ES&H issues. Laboratory ES&H procedures described in the SOPs of the laboratory in which the equipment is used shall be adhered to.

5.0 RESPONSIBILITIES

The Principal Investigator (PI), or designee, whose activities warrant the use of this procedure is responsible for implementing the requirements of this procedure.

The Project Scientist (PS), or designee, is responsible for performing the calibrations and measurements, following the requirements of this procedure, documenting calibrations, and assuring the use of the latest revisions of this document is followed.

The Quality Assurance Manager (QA Manager) is responsible for monitoring the work to assure proper implementation of the procedure and assuring its continued effectiveness.

6.0 CONTROLS

Controls are established by written procedures or instructions prepared in accordance with QAP 5.3, PREPARING, REVIEWING AND APPROVING TECHNICAL OPERATING PROCEDURES (Revision 1, effective date:,7/31/95) of the Sandia National Laboratories WIPP Quality Assurance Program. Procedures are issued in accordance with QAP 6.1, DOCUMENT CONTROL SYSTEM

(Revision 1, effective date: 7/31/95) of the Sandia National Laboratories WIPP Quality Assurance Program.

6.2 STANDARDS

Calibration will be verified using the quartz standard slide supplied with the machine.

6.2 FREQUENCY

The instrument will be recalibrated upon failure of a performance test (section 7.0), and this can be done by the operator at the time of the experiment. The instrument's calibration shall be verified with a performance test immediately prior to use.

6.3 PERFORMANCE TEST CRITERIA
The performance test for the diffractometer Involves measuring the actual position of the main quartz peak using the quartz standard slide. This peak should appear at 26.65 degrees. If this measurement is off by more than 0.02 degrees then the instrument must be recalibrated. A performance test will be done immediately after calibration.

7.0 CALIBRATION

General guidelines for instrument calibration are contained in QAP 12-1. The instrument shall be recalibrated if it fails a performance test. To calibrate the instrument position the goniometer at the actual position of the maximum reading from the quartz slide and then modify the THETA software to reflect that the goniometer is at 26.65 degrees.

7.1 CORRECTIVE ACTION

A performance test will be done immediately after calibration. If the instrument fails its performance test a second time then the instrument shall be tagged and placed out of service and the manufacturer (Dapple Systems, Inc.) shall be consulted regarding appropriate repair strategy. It is acceptable for the operator to make repairs on this instrument while receiving guidance over the phone by a qualified service person.

8.0 PROCEDURE: OBTAINING AN X-RAY DIFFRACTION PATTERN

8.1 SAMPLE PREPARATION

Glass or quartz slides may be used for samples but in either

case they should be labeled using black magic marker (red,green, blue rub off too easily).

Create a slurry of the clay (or other mineral) in question. This may be done by placing a few drops water or alcohol on the slide and then stirring in a small amount of clay or, alternatively, the clay-fluid mix may be prepared in a small beaker or pestle and transferred to the slide. If the clays are not free of soluble salts (gypsum, halite) diffraction patterns for these materials will dominate in the scan results. These salts must be washed out and the pattern rerun.

Allow the slide to dry at room temperature. Be sure that the clay covers the area where the X-rays will impinge (see the quartz standard slide to determine where this area is).

If the sample is to be run in the presence of ethylene glycol carefully place two or three drops of this liquid on the sample surface and allow it to stand for at least half an hour (overnight is preferable, but it is necessary to place the slide in a closed container since considerable evaporation will occur in that time period). At the end of this time the sample surface should appear damp, but not wet. If excess ethylene glycol is added it can be dried by placing the slide in the 60° C oven for a short time.

When non-routine processes are carried out the details will be noted in the experimenter's laboratory notebook.

8.2 OPERATING THE PHILIPS 3100 X-RAY MACHINE

Precautions:

a. Know the location of, and be familiar with the Safe Operating Procedure (Room 823, room 2490). Note: a list of operating instructions is also kept on top of the machine just to the left of the tube tower.

b. All operators must have the appropriate Sandia radiation workers training.

c. Users shall never bypass any safety circuits, nor attempt to service the electrical components without authorization and guidance from trained repair personnel. High voltages, up to 60 kV are produced by this machine.

d. A TLD (thermal luminescence detector) badge must be worn at all times.

e. The maximum setting for the copper target is 35 kV and 30 mA. To prevent burning out the (very expensive) X-ray tube never exceed these levels.

g. In the event of an emergency push the "X-rays off" button immediately.

Turn on the cooling water for the water chiller by rotating the valve on the wall counter-clockwise so that the handle points horizontally. Then, turn on the water chiller.

Prior to turning on the power to the generator check to make sure that the last operator turned the kV thumb wheel to 25 and the mA thumb wheel to 10. If not, adjust the thumb wheels downward to reflect these settings.

Turn on the two power strips (back-left top of the generator) so that the computers can boot up.

When the main computer has finished booting type "THETA' and hit return. This activates the software that allows one to operate the diffractometer and collect a pattern.

Turn the key on the front of the generator to the right to get power to the unit.

Before energizing the X-ray On button set the monitor to the MA reading setting. A few seconds after the X-ray On button has been pushed the reading should stabilize at about 10 mA. Then switch to the kV output reading and verify that the output reads 25 kV.

First turn up the kV thumb wheel to 35, and then turn up the mA thumb wheel to 30. The X-ray generation is now fully operational.

Remove the camera cap and check to make sure that no powder has collected on the bottom of the slide holder. Then, place the slide in the camera so that the sample covered part of the surface faces up, extends to the end of the holder, and is centered on the scribe mark on the end of the holder.

Replace the cap on the camera and then open the shutter ("Window 2" red button top-middle of the gray box on top of the X-ray generator). If the shutter will not open check to make sure that the camera cap is properly positioned and that the relay lever is properly positioned on the rim of the camera.

Acquire the X-ray spectrum in the manner described in the operating instructions supplied by DAPPLE, Inc. To do this you will need to adjust step size and count rate. Normally, a step size of 0.025 degrees and a count time of 2 seconds per step (1 minute, 20 seconds per degree) is a good place to start. However, these parameters may be adjusted depending on the diffracting power of the various samples. What matters is the quality of the output pattern not the parameters used to acquire the spectrum. A log of the file name for each sample will be kept with the output patterns and crossed referenced with the applicable data in the laboratory notebook.

8.3 X-RAY SHUTDOWN

First, turn down the mA thumb wheel to 10.

Next, turn the kV thumb wheel to 25.

Push the X-ray Off button and the turn the key off.

Turn off the two power strips controlling the computer.

After 5 minutes, turn off the water cooler and turn the valve on the wall clockwise to the vertical (off) position.

Remove your sample from the camera and clean off the underside of the sample holder.

8.4 DATA RETRIEVAL AND INTERPRETATION

When a scan has been completed the spectrum is automatically loaded to the DAPPLE software and stored in the pre-assigned file on the main computer.

To locate the two-theta readings on the pattern select the Peaks option from the main menu bar at the top of the screen and then select the Mark Peaks option in the selection box. You may then position the mouse cursor at the top of the peak and click with the left mouse button. To erase an incorrect selection place the cursor over that selection and click the right mouse button; then hit the delete key on the key board.

Although output files will normally not become corrupted or lost it will be the practice of this program to obtain a hard copy of each pattern run. To do this select Etc. from the main menu and Print from the selection box. Click on both Data and Current Window in the next selection box and then hit Print. The printed output for the project will be

kept in a loose-leaf notebook along with reference to what the pattern is. Each sample will be uniquely identified and traceable to the printed output.

Identification of the phases will be carried out by matching the peaks on the diffraction patterns from various libraries. The PC-PDF is the main source for such information but other matches may be obtained by running "knowns" directly. When a "known" is used to identify a pattern the information on the source of the "known" may be entered in the lab book or kept with the pattern for that material.

9.0 MAINTENANCE

Maintenance and trouble shooting will be performed on the instrument when indicated by the performance test criteria, according to manufacturer's specifications, and shall be documented in the appropriate scientific notebook.

10.0 QA RECORDS

Calibration verification results (accuracy) precision data, performance tests and data printouts will be submitted to SWCF or the results will be recorded in the laboratory notebooks in accordance with Sandia National Laboratories WIPP Quality Assurance Procedure 20-2, "REPAIRING, REVIEWING, AND APPROVING SCIENTIFIC NOTEBOOKS" (Revision 1, effective date:7/31/95)

11.0 REFERENCES

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X-ray Diffraction Procedures for Polycrystalline and Amorphous Materials, 2nd Ed., Klug, H.P. and Alexander, L.E., 1979, J. Wiley & Sons, New York, N.Y., pp. 966

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Operation of the X-ray diffractometer, Sandia ES&H SOP# SP472650, 12/7/95.

Theta.XRD Operators Manual, Dapple Systems, Inc.

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Soil Chemical Analysis - Advanced Course, M.L. Jackson, 2nd Ed., 1979, 895 pp.

QAP 5.3, PREPARING, REVIEWING AND APPROVING TECHNICAL OPERATING PROCEDURES (Revision 1, effective date: 7/31/95).

QAP 6.1, DOCUMENT CONTROL SYSTEM (Revision 1, effective date: 7/31/95)

QAP 20.2, PREPARING, REVIEWING AND APPROVING SCIENTIFIC NOTEBOOKS (Revision 1, effective date 7/31/95)

QAP 12-1, WIPP CALIBRATION LABORATORY QUALITY ASSURANCE PROGRAM, (Revision 1, effective date 7/31/95)

Soil Chemical Analysis - Advanced Course, M.L. Jackson, 2nd Ed., 1979, 895 pp.

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QAP 6.1, DOCUMENT CONTROL SYSTEM (Revision 1, effective date: 7/31/95)

QAP 20.2, PREPARING, REVIEWING AND APPROVING SCIENTIFIC NOTEBOOKS (Revision 1, effective date 7/31/95)

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