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PNL TEST PROCEDURE

TITLE: HTA-3-1, SOLIDS ANALYSIS: SCANNING ELECTRON MICROSCOPY

1.0 APPLICABILITY

This procedure provides guidelines for analysis of solids by scanning electron microscopy (SEM) and energy-dispersive x-ray spectrometry (EDS).

This procedure applies to solids analysis for waste repository programs. It also applies to work conducted for other waste repository supporting organizations, such as the Materials Characterization Center (MCC).

This procedure is not limited to a particular type or form of solid. Solid samples for analysis may include metals and nonmetals, organic or inorganic materials, and may be examined as powders, fragments, monoliths, sheets, wires, or any other form that will physically fit into the microscope's sample chamber.

The form of this procedure may not comply with all the requirements of PAP-501 due to the conversion from Westinghouse Hanford Co. format to PNL format. Exceptions to PAP-501 will be addressed during the next revision of this procedure.

2.0 DEFINITIONS

None.

3.0 EQUIPMENT AND MATERIALS

- Scanning electron microscope.
- X-ray multichannel analyzer.
- Si(Li) solid-state x-ray detector.
- Vacuum evaporator/sputter coater.

Operation of the above equipment is described in the appropriate manufacturer's operating instructions. Applicable manufacturer's instructions shall be referenced in the appropriate SEM operator's laboratory notebook.

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4.0 PROCEDURE

4.1 Description of Method

Scanning electron microscopes are used to observe the surface topography and local elemental compositions of solid materials. For topographic imaging, a focused beam of 0.1 to 50 keV electrons is scanned over the specimen and a detected secondary electron signal is used to form a facimile image of the specimen surface. Scanning electron micrographs, produced by photography of images on a cathode ray tube, reveal surface topography with a large depth of field at usable magnifications from about 10X to over 50000X. Micrographs taken at different sample tilt positions can be used for steroscopic analysis. The primary beam energy may be varied to reduce charging of nonconducting samples or to enhance production of characteristic x-rays for elemental analysis.

Compositional analyses of local areas in the scanning electron microscope can be performed by detecting characteristic x-rays generated with a stationary or scanned electron probe. The x-rays are detected with a solid-state Si(Li) detector and analyzed with a multichannel analyzer (MCA). Compositional images (x-ray maps) can be obtained by using signals from the multichannel analyzer. Backscattered electrons can also be used to produce images in which the contrast varies with average atomic number.

4.2 Sample Preparation

Sample preparation for SEM involves mounting the sample on an electrically conducting holder and, in the case of poorly conducting samples, providing an electrically conducting surface coating on the sample to inhibit charging under the electron beam of the microscope. Samples may be clamped to the mounting stub or affixed with conducting silver paint, or affixed with nonconducting adhesives (double-stick acetate tape, Eastman 910 glue, or epoxy) and then grounded to the mounting stub with silver paint. An anticharge coating is applied to nonconducting samples by evaporating carbon in a vacuum evaporator or by sputter deposition of carbon or gold.

During sample preparation it is essential that no contamination be introduced to the sample and that the sample identification be maintained at all times. If grinding is required to reduce particle sizes of particulates, the grinding operations shall be performed in a cleaned boron carbide mortar and pestle using spectroscopic-grade solvents or distilled, deionized water. Particulate samples may be mounted on acetate tape on a SEM mounting stub and coated with a thin layer of carbon or gold to prevent charging. Preparation of radioactive samples shall be done in a hood or glovebox designated for radioactive materials.

4.3 Calibration

The calibration of scanning electron microscopes shall be in accordance with PAP-1201.

The SEM performance shall be checked periodically to assure optimum results. This is done during routine maintenance at six-month intervals by a microscope field service engineer under service contract. Calibration results, including date and any adjustments made, shall be recorded in the SEM laboratory notebook. Calibration standards employed by field service engineers shall be noted in the SEM laboratory notebook. Image magnifications shall be accurate within ±10% of the indicated readout value.

The energy calibration of the Energy Dispersive X-Ray (EDX) spectrometer shall be checked during each day's use by observing the position (peak channel number) of x-ray peaks from the specimen stub or from the suitable specimen standard. The microscope operator shall adjust the EDX spectrometer energy calibration to ±10 eV of known x-ray peak energy. The operator shall indicate completion of the calibration check by a notation in the SEM laboratory notebook.

4.4 Sample Analysis

For particle identification by EDX microanalysis in the SEM, scanning electron micrographs showing the particles to be analyzed are taken at suitable magnifications to reveal the particle morphology (high magnification) and its relation to the whole assemblage of particles (low magnification). The actual magnifications used will depend on particle size and the nature of the sample. Bulk (monolithic) samples are qualitatively analyzed by the same procedures described above for powder samples. The micrographs are used as keys to the analysis locations and are kept with the operator's notebook. Key micrographs must indicate the location of each EDX analysis on the sample, the identifying number of the analysis, the sample identification and image magnification.

The acquisition time for EDX analysis varies with the x-ray count rate but is typically 40 to 100 seconds with count rates from 200 to 2000 counts per second. Each x-ray spectrum is to be recorded on a floppy disk using the MCA computer system and is identified by the floppy disk number and data file number recorded on the disk. A notation must be made in a notebook referencing disk and data file numbers to the key micrographs.

During or after x-ray data acquisition, the microscope operator may identify characteristic x-ray peaks (perform qualitative analysis using peak identification aids in the MCA).

After examination, the specimen shall be removed from the microscope and stored in an approved repository.

Sample identity shall be maintained at all times (from preparation and transportation through analysis and storage). Prepared samples shall be kept in storage at least one year.

4.5 Records

Records will be maintained and controlled so as to conform with requirements of PAP-1701. Laboratory notebooks provide a mechanism for control of most records. Records of SEM analyses are kept in laboratory notebooks by hydrothermal run number. The records include key micrographs and cross-reference information pertaining to EDX analyses. EDX spectral records are stored on magnetic media with backup copies. The notebook will include all necessary sample identification and notes on analyses.