



TENNESSEE BUREAU OF INVESTIGATION

Forensic Services Division

Microanalysis Quality Assurance Manual

SEM/EDS Quality Assurance Policy

Quality Assurance for Zeiss Electron Microscopes with Oxford EDS Detectors

1. Optimization

This optimization will be performed before analyzing each batch of samples. Optimization will be performed using the cobalt sample on the MAC Reference Standard for X-ray Microanalysis and the same SEM conditions as those used in casework. Using the Oxford software, open a project to store data. Once the cobalt is displayed and in proper focus on the SEM, the acquisition can begin on the Oxford software. The beam spot size should be adjusted to obtain an EDS detector dead time of approximately 25-40%. Using the Oxford software, compare two successive beam measurements. The value should be 100% +/- 3%. If not within the 3%, repeat the optimization. Generate a spectrum and record the measured value on the spectrum. This optimization should also be performed monthly for maintenance purposes and stored in the maintenance records for each SEM/EDS instrument.

2. System Check

Collect the following data and record it in the maintenance records for each SEM/EDS:

- Filament changes within the month
- Filament age
- Amperage at saturation of the filament
- Aperture changes within the month
- Sample chamber vacuum value
- Whether the column supports are correctly inflated

If an image is not attainable, the following shall be performed:

- Check the filament and determine whether it is operational. If it is not, replace the filament.
- Center the filament and examine image.
- Check instrument settings.

Full calibration of EDS detector shall be done yearly.
Six months after this calibration, the discriminators will be calibrated

For the systems using Oxford INCA software, this is an INCA Monitor X-ray calibration software function.



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For the systems using Oxford Aztec software, this is performed through the “Calibration” icon in the top left of the Aztec Assist software window.

Record this calibration in the maintenance records for each SEM/EDS.

3. *Detector Check*

This check is to be performed on each SEM/EDS monthly and should be performed with the SEM beam on.

For INCA

Open X-max Support from the Oxford desktop screen. Enter the password: WYE, select “OK,” then “Monitor.” Generate a report to retain in maintenance records.

If the detector is functioning correctly there will be NO entries in red. Entries in red indicate corrective action is necessary. Corrective action: Open INCA software on Oxford EDS system, select “options” in top left of screen, then “detector control,” then “thermal,” then “standby.” Wait a few seconds then select “operate.” If the LED entry does not switch from red, then take the instrument out of service and consult with the Unit Supervisor about next steps.

For Aztec

Open Aztec Assist. From the “Tools” menu choose to “Enable Health Check” and enter the password: energy. Choose the Ultimex Detector button and then the Detector Health Check icon, then press “Start”. Take a screenshot of the data shown and save it in the maintenance records.

If the detector is functioning correctly, there will be no errors displayed and all voltages will be present. If this is not the case, take the instrument out of service and consult with the Unit Supervisor about next steps.

4. *Pump Oil Check*

Rough pump oil should be changed every 6 months.

Record this in the maintenance records for each SEM/EDS.

Preventative maintenance, repairs, and recalibration by service engineers may be performed upon approval and recorded in the SEM/EDS maintenance records.