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UCD IMPROVE Technical Information #301A

LN2 Fills and Detector Calibration

Interagency Monitoring of Protected Visual Environments
Air Quality Research Center
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Prepared By: DocuSigned by: Lindsay Kline B7A73C50DF23499... Date: 10/3/2022

Reviewed By: DocuSigned by: Jason Giacomo B62B01F81613421... Date: 10/4/2022

Approved By: DocuSigned by: Marcus Langston 0A10CFCF79B0452... Date: 10/4/2022

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DOCUMENT HISTORY

Date Modified	Initials	Section/s Modified	Brief Description of Modifications
06/14/21	SRS	All	Separated TI: A-C into individual TIs.
7/29/2022	LMK	All	Updated wording.
8/19/2022	JG	9.1 and 9.4	Removed “Short Grace” and “Long Grace” period from detector states, as these were made obsolete when the manufacturer upgraded all the LN2 level sensors.

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1. PURPOSE AND APPLICABILITY

The purpose of this technical information (TI) document is to describe the procedure used for the liquid nitrogen (LN2) filling of the PANalytical Epsilon 5 EDXRF (E5) analyzers and detector (energy) calibration of the aforementioned analyzer.

2. SUMMARY OF THE METHOD

The LN2 is utilized to cool the PAN-32 Ge X-Ray Detector in the E5analyzer. LN2 fills for each E5 are performed on a weekly basis. Approximately two hours after the LN2 fill is completed (or after the temperature is stable), a full detector calibration is performed for each instrument.

3. DEFINITIONS

- LN2: liquid nitrogen
- PPE: Personal protective equipment
- E5: PANalytical Epsilon 5 EDXRF system

4. HEALTH AND SAFETY WARNINGS

Cryogenic liquids, such as liquid nitrogen, are extremely cold. Contact between cryogenic liquids and exposed skin can produce a painful burn or loss of vision through contact with the eye. Always wear proper personal protective equipment including a buttoned lab coat, cryogenic apron, long pants or a long skirt, heavy leather gloves (or cryogen handling gloves), safety goggles, and a face shield (when the risk of splash is high) whenever handling cryogenic liquids.

Asphyxiation is another hazard when working with cryogenic liquids. If vented into a closed space, a cryogenic liquid will vaporize, displacing oxygen and possibly causing asphyxia. For this reason, never store a container of cryogenic liquid in an enclosed space.

5. CAUTIONS

LN2 dewar must be handled with care in ventilated rooms. Proper personal protective equipment (PPE) must be worn, including a face shield, safety glasses, safety gloves, and a laboratory coat when performing liquid nitrogen fills. For more information, see section 3.3.3, “Liquid Nitrogen Handling,” in the Epsilon 5 EDXRF Spectrometer System User’s Guide and the UC Davis SafetyNet 58: Safety Precautions for Cryogenic Liquids (https://safetyservices.ucdavis.edu/safetynet/safety-precautions-for-cryogenic-liquids).

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6. INTERFERENCES

Not applicable.

7. PERSONNEL QUALIFICATIONS

Only trained lab personnel designated by the laboratory manager may perform LN2 fills. A course offered on the UC Davis campus, "Safe Use of Cryogenic Liquids," is highly recommended (https://safetyservices.ucdavis.edu/training/cryogen-safety).

8. EQUIPMENT AND SUPPLIES

- Liquid nitrogen tank
- Liquid nitrogen tubing
- Adapter
- Safety glasses/face shield
- Cryogenic safety gloves
- Laboratory coat

9. PROCEDURAL STEPS

9.1 Epsilon 5 LN2 Fill

The detector in the Epsilon 5 (E5) must be cooled with LN2. To keep the level of LN2 consistent, the dewar is filled on a weekly basis. Under special circumstances such as holidays, the dewar can be filled on a different schedule with prior Laboratory Manager approval.

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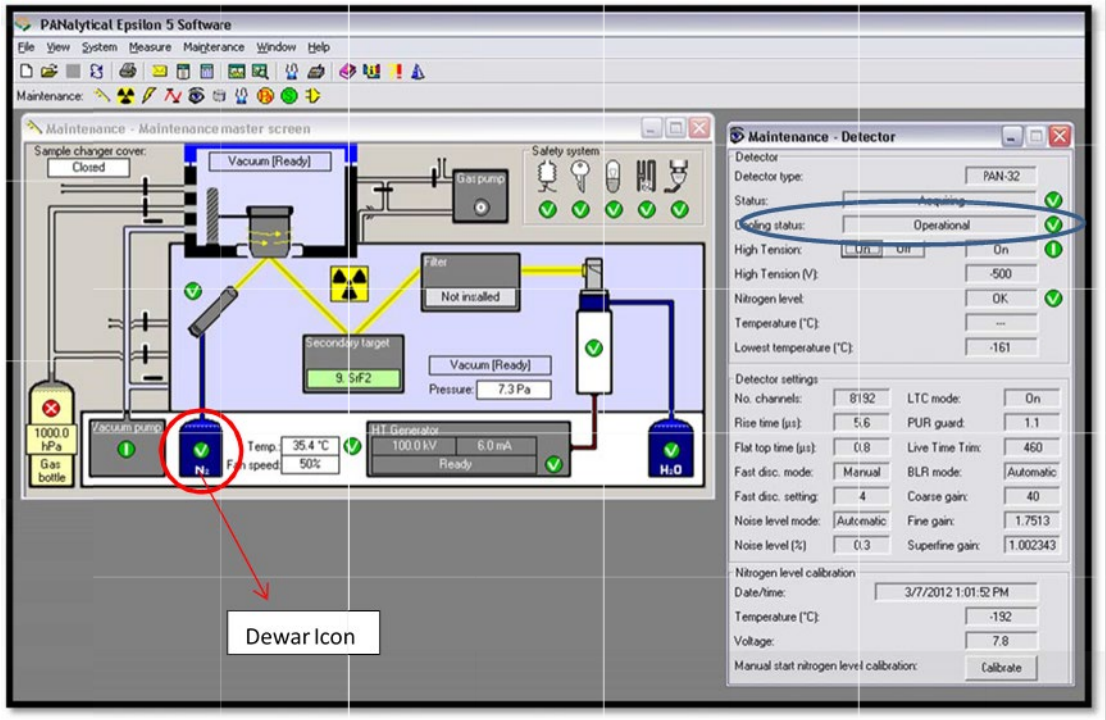
Figure 1. Maintenance Master Screen and Detector Maintenance Window.



1. The E5 has several potential values for “Cooling status” that are displayed on the Maintenance screen (Figure 1, circled in blue). The statuses include Pre-Operational, Operational, Filling Allowed, Cooling, and Forced Heat-Up. Ensure that the detector state is not in Forced Heat-Up prior to filling the dewar with LN2. Check this by clicking on the picture of the LN2 dewar (Figure 1, circled in red). Refer to Section 9.4: “Detector States” for additional information.
2. Open the LN2 fill access door on the right-hand side of the E5.
3. Connect the LN2 tubing to the adapter if it is not already attached.
4. Connect the tubing to the LN2 tank and then carefully insert the adapter into the E5 dewar.
5. Slowly open the valve on the LN2 tank while ensuring that the line from the LN2 tank into the dewar fill tube does not come apart. In addition, note the time the valve to the LN2 is opened (this will be used to record the length of time required to fill the dewar).
6. During filling, nitrogen gas will exhaust from the dewar vent at the back of the E5.
7. The dewar is full when liquid nitrogen begins to steadily discharge from the rear vent of the E5. Close the valve on the LN2 tank.
8. Record the time required to fill the dewar and the temperature of the cabinet in the corresponding log book.

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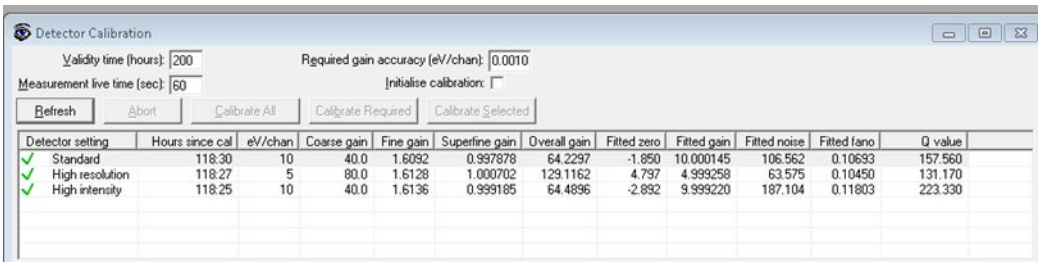
9.2 Epsilon 5 Detector Calibration

Before starting the detector calibration, abort analysis. No sample can be running during detector calibration, as the tungsten (W) underside of the beamstop is utilized as the target to calibrate the detector energy. The software automatically performs the energy calibration calculations.

1. Click on the **System** drop-down menu, then **Detector Calibration**.
2. Select **Calibrate All**.

Figure 2. Detector Calibration sub-window.

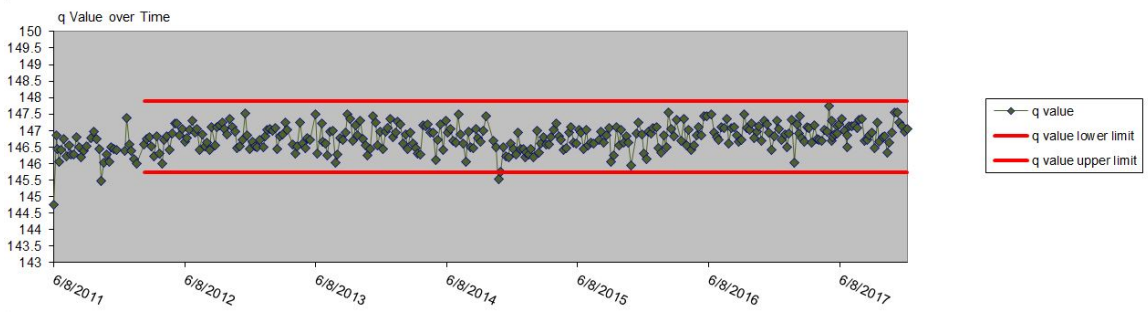


Detector setting	Hours since cal	eV/chan	Coarse gain	Fine gain	Superfine gain	Overall gain	Fitted zero	Fitted gain	Fitted noise	Fitted fano	Q value
✓ Standard	118:30	10	40.0	1.6092	0.997878	64.2297	-1.850	10.000145	106.562	0.10693	157.560
✓ High resolution	118:27	5	80.0	1.6128	1.000702	129.1162	4.797	4.999258	63.575	0.10450	131.170
✓ High intensity	118:25	10	40.0	1.6136	0.999185	64.4896	-2.892	9.999220	187.104	0.11803	223.330

3. When detector calibration is completed, click the Detector Calibration window to activate window. Press Ctrl +P, verify the data is set to “copy to the clipboard” in a “delimited” format, and click OK. Open the desktop folder named “Detector and LN Calibrations”. Then open the excel sheet “EpsilonName_ Detector and LN Calibrations”, where Name is the name of the instrument being calibrated. Paste the corresponding numbers at the bottom of the data set based on their respective setting.

4. Review the q value and verify all the values are within the acceptable limits, as shown in Figure 3 below. If the values exceed the acceptable limits, repeat detector calibrations starting with step 2 above.

Figure 3. Detector Calibrations Graph, q value over Time.



5. If the values are within the bounds, continue to step 6. If the values exceed the acceptable limits a second time, notify the spectroscopist or lab manager.

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6. In the E5 software, copy the screen using the "Snipping Tool", then open the folder "Detector and LN Calibrations". Type the date and press CTRL+V to paste the screen grab. Save and close the file.

7. In the E5 Software, close the sub-windows for the detector and the detector calibrations.

9.3 Resolution Test

This test is performed when there is a question about recognition of specific peaks in the spectra. The purpose of the resolution test is to gauge the method's ability to differentiate detected peaks from individual X-rays. This is especially important in regions of the X-ray spectra where characteristic X-ray peaks overlap. The test measures the full width half max (FWHM) of the Mn Kα peak, which is the standard peak for this purpose. The measured FWHM should be less than the manufacturer's specification of 140 eV. Only approved personnel may perform the resolution test, check with the Lab Manager and/or Spectroscopist.

1. Verify the Resolution application has been installed on the instrument. Notify the Spectroscopist if the application needs to be created
2. Locate Petri slide containing the Mn pellet.
3. Select the Resolution test application
4. Go to "Add Measurement". Type "Mn fused bead 402300074331" in SampleID and insure the resolution test application is selected.
5. Place the Mn pellet in a stainless steel cup and load to the instrument.
6. Queue the sample for analysis.
7. To review the results, open the resolution test application.
8. Highlight the result file and open the spectra window. To view the spectra, select the spectra button on the lower right-hand corner.
9. Check the FWHM value in the lower left corner, see Figure 4.

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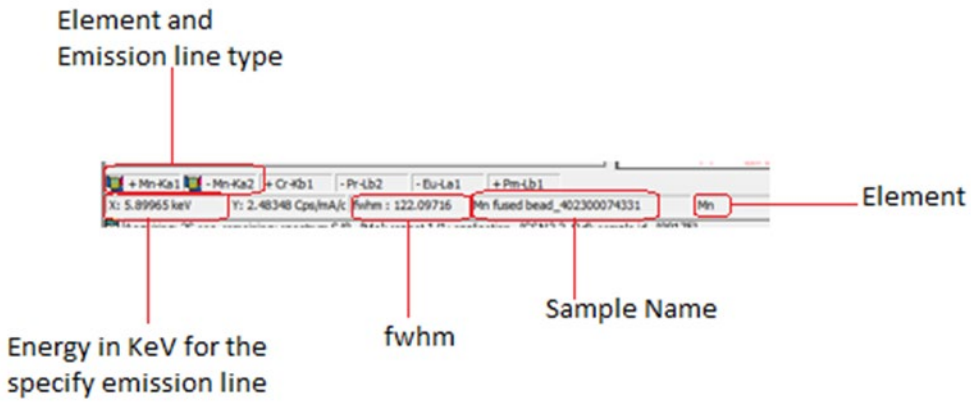
Figure 4. Resolution Test Information.



10. Verify the energy for Mn Kα has not shifted. The energy calibration is automatic, a shift is not expected.
11. Notify the Laboratory Manager and spectroscopist of the resolution test results.

9.4 Epsilon 5 Detector Cooling Status

The different detector cooling statuses that may be encountered by the user of the E5 during operation are briefly described. The user software displays the cooling status in the detector maintenance window (Figure 1). In addition to the statuses described below each E5 is equipped with an external LN2 alarm. In the event the LN2 is near empty an audible alarm will sound until refilled.

***Filling allowed*:** The detector is 'warm'. Both the LN2 level sensor as well as the detector temperature are at room temperature for at least one hour. It is allowed to start filling with LN2.

***Cooling*:** After the LN2 level sensor has detected more than 20 degrees temperature decrease due to filling the dewar, the user must wait 6 hours before switching on the detector high tension in order to allow the crystal and the FET to cool down to -178 ºC.

***Pre-operational*:** The system is available for normal use. As the LN2 consumption is not yet constant it can warm up a little quicker than expected.

***Operational*:** The system is available for normal use.

***Forced heat-up*:** The detector crystal has warmed above LN2 temperatures and *must be* brought to room temperature. This can be done by waiting for the status to reach 'filling allowed'. Additionally, acceleration of this procedure can be achieved by blowing with *dry* air into the liquid nitrogen fill opening.

10. QUALITY ASSURANCE AND QUALITY CONTROL

Not applicable.

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11. REFERENCES

Epsilon 5 EDXRF Spectrometer System User's Guide.

UC Davis SafetyNet 58: Safety Precautions for Cryogenic Liquids (https://safetyservices.ucdavis.edu/safetynet/safety-precautions-for-cryogenic-liquids).