UCD CSN Technical Information #302A

LN2 Fills and Detector Calibrations

Chemical Speciation Network
Air Quality Research Center
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DOCUMENT HISTORY

Revision	Release Date	Initials	Section/s Modified	Brief Description of Modifications
	11/26/2018	KT	all	Small editing changes; added pictures
	06/17/19	MGN	9.1	Update logbook procedures
	4/22/2020	LMK	all	Changed wording for clarification, updated timing procedure and added LN2 audible alarm warning
1.3	5/21/2021	LMK	9.1	Changed wording for clarification, added procedural steps for when LN2 dewar is full.
1.4	11/30/2022	LMK	All	Updated wording and added safety net 58.

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1. PURPOSE AND APPLICABILITY

The purpose of this technical information (TI) document is to describe weekly procedures for 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 E5 analyzer. 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).

6. INTERFERENCES

Not applicable.

7. PERSONNEL QUALIFICATIONS, DUTIES, AND TRAINING

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 (http://safetyservices.ucdavis.edu/tr/cd/suoclcd).

8. EQUIPMENT AND SUPPLIES

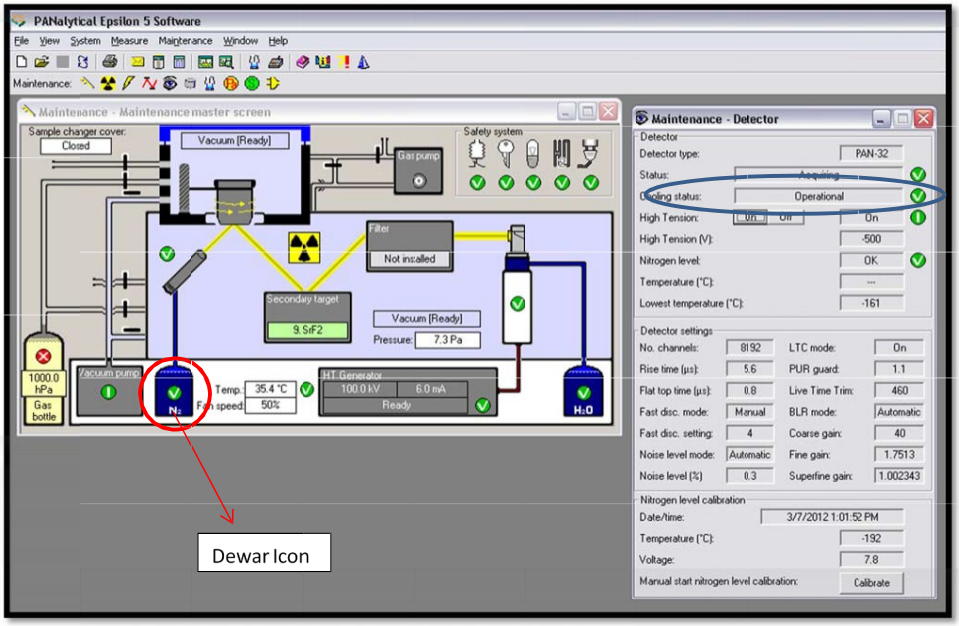
- Liquid nitrogen tank
- Liquid nitrogen tubing
- Adapter
- Safety glasses and face shield
- Cryogenic Safety gloves
- Laboratory coat

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.

Figure 1. Maintenance Master Screen and Detector Maintenance window.



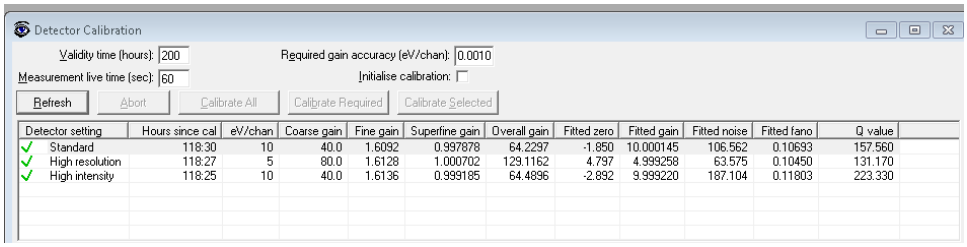
1. The E5 has several potential values for "Cooling status" that are displayed on the Maintenance screen (Figure 1, circled in blue). The statuses include Pre-Operational, Operational, Filling Allowed, Cooling, and Forced Heat-Up. Ensure that the detector state is not in Forced Heat-Up prior to filling the dewar with LN2. Check this by clicking on the picture of the LN2 dewar (Figure 1, circled in red). Refer to the detector states listed in section 9.4 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 tank 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 logbook.

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.

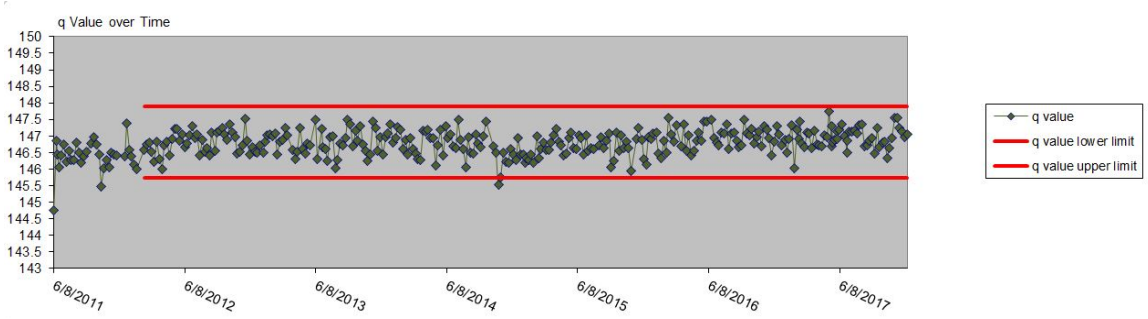
1. Click on the **System** drop-down menu, then **Detector Calibration**.
2. Select, **Calibrate All**.

Figure 2. Detector Calibration sub-window



3. When the detector calibration is completed, click the **Detector Calibration window** to activate it. Then press **Ctrl +P** and verify the data is set to "copy to the clipboard" in a "delimited" format. Click on **OK**. Open the desktop folder named "Detector and LN Calibrations." Then open the Excel sheet named "Epsilon*Name*_ 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 graphs and verify all the values are within acceptance limits, as shown in Figure 3 below. If the values exceed the acceptance 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 acceptance limits more than once, notify the spectroscopist or lab manager.

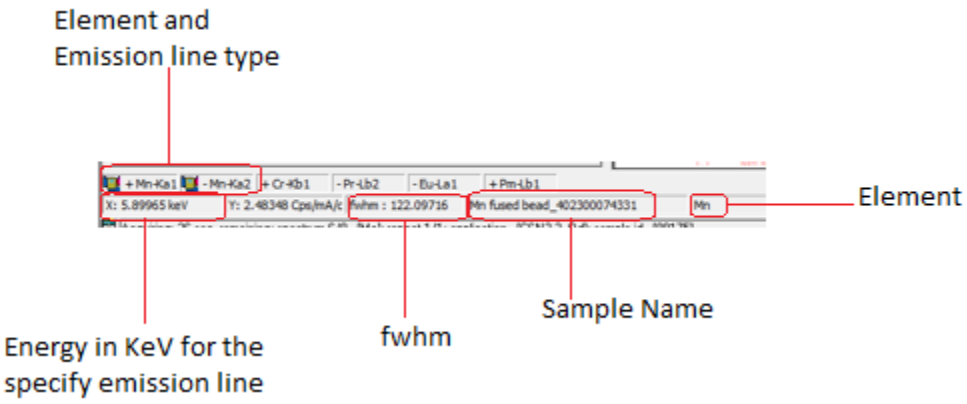
6. In the E5 software, copy the screen using the "Snipping Tool," then open the folder "Detector and LN Calibrations." Type the date and press **CTRL+V** to paste the screen grab. Save and close the file.
7. In the E5 Software, close the sub-windows for the detector and the detector calibrations.

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" then type "Mn fused bead 402300074331" in SampleID.
5. Place the Mn pellet in a stainless steel cup. Next 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.

Figure 4. Resolution test information.



10. Verify the energy for Mn Kα has not shifted. As mentioned above 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 which may be encountered by the user of Epsilon 5 during operation, are briefly described. The user software displays the cooling status in the detector maintenance window, see 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 the detector/dewar is refilled.

Filling allowed: The detector is 'warm'. Both the LN2 level sensor, and 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, one has to wait for 6 hours before switching on the detector high tension in order to allow the crystal and the FET to cool down to -178ºC.

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 to be brought to room temperature. This can be done just waiting for the state 'filling allowed', which can be rather time consuming. 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.

11. REFERENCES

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