

UCD CSN Technical Instruction #302A

LN2 Fills and Detector Calibrations

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*July 31, 2021
Version 1.3*

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Date: _____

DOCUMENT HISTORY

Date Modified	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
5/21/2021	LMK	9.1	Changed wording for clarification, added procedural steps for when LN2 dewar is full.

TABLE OF CONTENTS

1. PURPOSE AND APPLICABILITY	4
2. SUMMARY OF THE METHOD.....	4
3. DEFINITIONS	4
4. HEALTH AND SAFETY WARNINGS.....	4
5. CAUTIONS.....	4
6. INTERFERENCES	4
7. PERSONNEL QUALIFICATIONS, DUTIES, AND TRAINING.....	4
8. EQUIPMENT AND SUPPLIES	5
9. PROCEDURAL STEPS	5
9.1 Epsilon 5 LN2 Fill.....	5
9.2 Epsilon 5 Detector Calibration.....	6
9.3 Resolution Test.....	7
9.4 Epsilon 5 Detector Cooling Status	8
10. QUALITY ASSURANCE AND QUALITY CONTROL.....	9
11. REFERENCES	9

LIST OF FIGURES

Figure 1. Maintenance Master Screen and Detector Maintenance window.	5
Figure 2. Detector Calibration sub-window.....	6
Figure 3. Detector Calibrations Graph, q value over Time.....	7
Figure 4. Resolution test information.	8

1. PURPOSE AND APPLICABILITY

The purpose of this technical instruction (TI) document is to describe weekly procedures for liquid nitrogen (LN2) fill of the detectors in PANalytical Epsilon 5 EDXRF analyzers and detector (energy) calibration for mentioned analyzer.

2. SUMMARY OF THE METHOD

The LN2 is utilized to cool the PAN-32 Ge X-Ray Detector in the PANalytical Epsilon 5 EDXRF analyzer. LN2 fills for each Epsilon 5 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 XRF system.

4. HEALTH AND SAFETY WARNINGS

Refer to safety sheets for liquid nitrogen available in the XRF lab.

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.

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

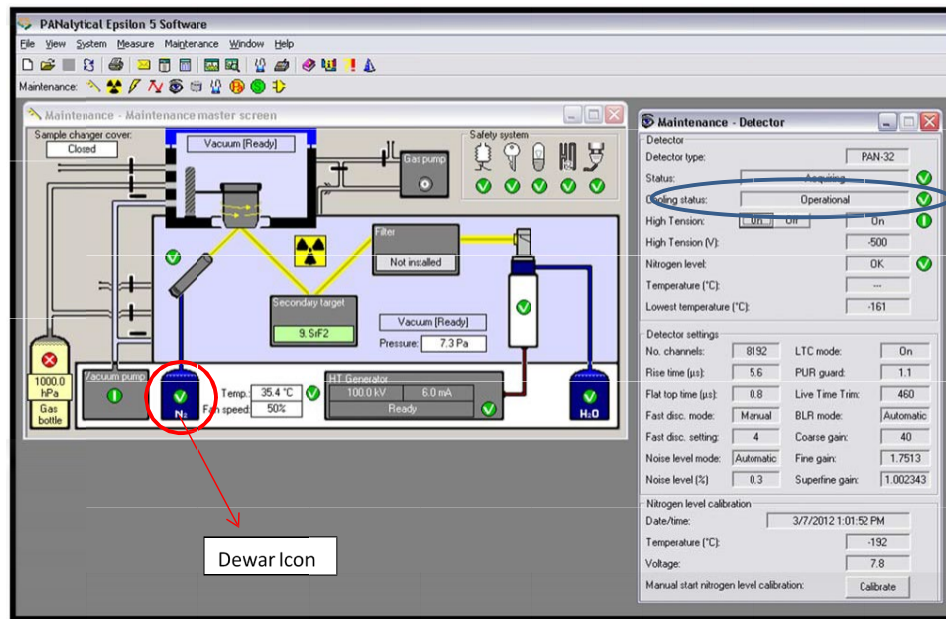
- Liquid nitrogen tank
- Liquid nitrogen tubing
- Adapter
- Safety glasses and face shield
- Cryogenic Safety gloves
- Laboratory coat

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, Long Grace, Short Grace, 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 Epsilon 5.
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) beamstop is utilized as the target to calibrate the analyzer. The software automatically performs the energy calibration calculations.

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

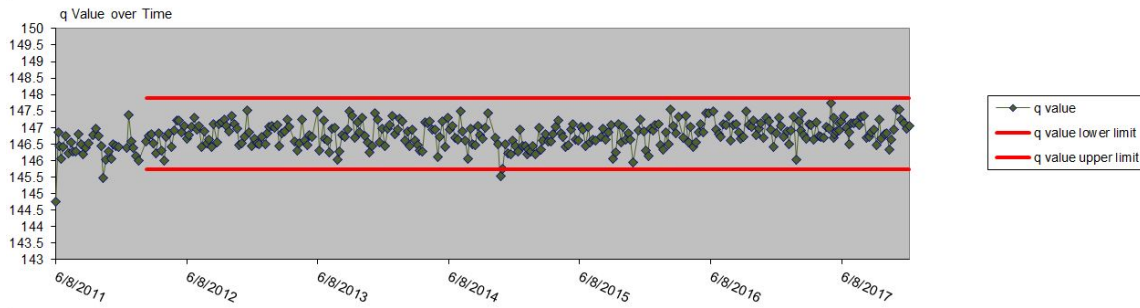
Figure 2. Detector Calibration sub-window

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 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 “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.

- 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



- If the values are within the bounds, continue to Step 6. If the values exceed the acceptance limits more than once, proceed to section 9.3 Resolution Test.
- In the E5 software, copy the screen using the "Snipping Tool," then open the folder "Detector and LN Calibrations." Type the date and press **CNTRL+V** to paste the screen grab. Save and close the file.
- In the E5 Software, close the sub-windows for the detector and the detector calibrations.

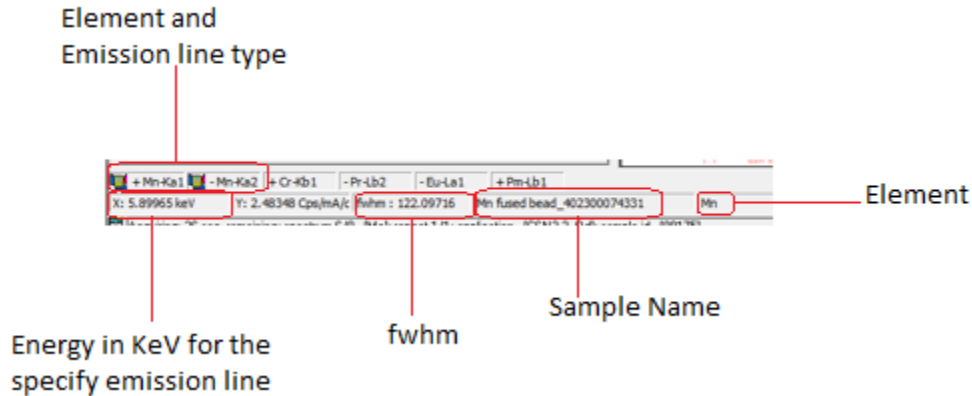
9.3 Resolution Test

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 $K\alpha$ 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.

- Verify the Resolution application has been installed on the instrument. Notify the Spectroscopist if the application needs to be created.
- Locate Petri slide containing the Mn pellet.
- Select the Resolution test application
- Go to "Add Measurement" then type "Mn fused bead 402300074331" in SampleID.
- Place the Mn pellet in a stainless steel cup. Next load to the instrument.
- Queue the sample for analysis.
- 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 K α 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 -178°C.

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.

Short grace period: The system is available for normal use. Please refill LN2 as soon as possible. There are 75 hours (3 days) left to refill the dewar.

Long grace: The system is available for normal use. Please refill LN2 as soon as possible. There are 100 hours (4 days) left to refill the dewar.

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. Safety data sheets for Liquid Nitrogen