UCD CSN Technical Information #302A

LN2 Fills and Detector Calibrations

Chemical Speciation Network
Air Quality Research Center
University of California, Davis

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Prepared By: M J Navarro Date: 10/16/17

Reviewed By: [signature] Date: 10/16/2017

Approved By: Nicole Hyer Date: 10/16/17

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DOCUMENT HISTORY

Date Modified	Initials	Section/s Modified	Brief Description of Modifications

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1. PURPOSE AND APPLICABILITY

The purpose of this SOP is to describe the procedure used for the liquid nitrogen (LN2) fill, liquid nitrogen calibration, and detector calibration for the Epsilon 5 instruments.

2. SUMMARY OF THE METHOD

The Epsilon 5 instruments use liquid nitrogen to cool the PAN-32 Ge X-Ray Detector.

Liquid nitrogen fills for each Epsilon are performed on a weekly basis. The liquid nitrogen level is calibrated automatically. Several hours after the LN2 fill is completed, detector calibration is performed for each instrument.

3. DEFINITIONS

Not Applicable.

4. HEALTH AND SAFETY WARNINGS

Not applicable.

5. CAUTIONS

Liquid nitrogen should be handled with care in ventilated rooms. Wear a face shield and 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 liquid nitrogen fills. A course offered on the UC Davis campus, "Safe Use of Cryogenic Liquids," is recommended (http://safetyservices.ucdavis.edu/tr/cd/suoclcd).

8. EQUIPMENT AND SUPPLIES

a. Liquid nitrogen tank

b. Safety glasses and face shield
c. Safety gloves
d. Laboratory coat

9. PROCEDURAL STEPS

9.1 Epsilon 5 LN2 Fill

The detector in the Epsilon 5 should be filled on a weekly basis.

1. The Epsilon 5 has several potential "detector states" that are displayed on the Maintenance screen (Figure 1, circled in blue). These detector states 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 detector with liquid nitrogen. Check this by clicking on the picture of the liquid nitrogen dewar (Figure 1, circled in red).
2. Open the LN2 fill access door on the right-hand side of the Epsilon 5.
3. Connect the fill tube to the detector fill line.
4. Insert the tube from the LN tank into the detector fill tube using the adapter.
5. Slowly open the valve on the LN tank while ensuring that the line from the LN2 tank into the detector fill tube does not come apart.
6. Note the time required to fill the detector and the temperature of the cabinet in the corresponding log book. Also create a new entry with the same information in the Microsoft Access log on the desktop.

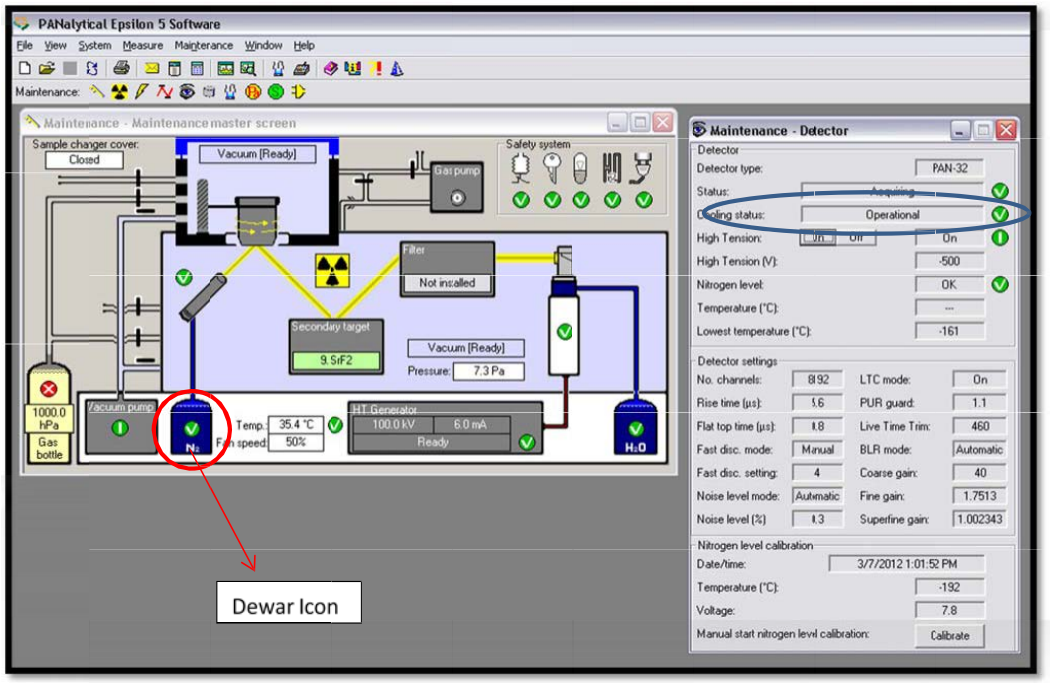
9.2 Epsilon 5 Detector Calibration

Before starting the detector calibration, abort analysis. No sample can be running during detector calibration, as the Tungsten (W) underside of the vacuum seal is utilized to perform the measurements.

1. Click on the "System" drop-down menu, then "Detector Calibration."
2. Select, "Calibrate All."
3. When both the detector calibration and the liquid nitrogen calibration are complete, copy the screen by pressing "ALT+Print Scrn."
4. On the desktop, there is a folder named "Detector and LN Calibrations." Inside the folder is a document called Detector and LN Calibrations. Open it. Type the date and press "CNTRL+V" to paste the screen shot. Save and close the file.
5. On the Epsilon software, click on File, then Print. After making sure that the resulting data would be copied to the clipboard in a delimited format, click on OK. On the same folder, open an excel sheet called EpsilonName_ Detector and LN Calibrations. Paste the corresponding numbers at the bottom of the data set based on their respective setting. Review the graphs to make sure that visually there are no significant jumps or discrepancy with the values that the detector

was calibrated from last week. Save and close the file.

Figure 1. Maintenance Master Screen.



9.3 Epsilon 5 Detector States

The different detector states which may be encountered by the user of Epsilon 5 during operation, are briefly described. The user software displays the state in the detector status screen.

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 liquid nitrogen.

Cooling: After the N2 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 liquid nitrogen 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 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 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

Not applicable.