

UCD CSN Technical Information #302C

Sample Changes for 8-Position Trays

*Chemical Speciation Network
Air Quality Research Center
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1. PURPOSE AND APPLICABILITY

The purpose of this SOP is to describe the process of loading and unloading samples using standard cups in 8-position trays in the Epsilon 5 EDXRF instruments.

2. SUMMARY OF THE METHOD

XRF queue files are generated for each CSN batch number, the queue files include application name, Barcode ID and Filter ID. The scanning capabilities of the PANalytical software is used to ingest the sample information from the XRF queue file into the sample changer. Filters are transferred from Petri slides into cups immediately after scanning the barcode. The cups are placed into one of six 8-position trays (as assigned during barcode scanning). The instrument name, assigned tray and position number are noted on the chain-of-custody (COC). The trays are placed into the Epsilon 5 sample changer compartment, and the samples are queued in the software. After analysis is complete, trays are removed and filters are transferred back into labeled petri slides.

3. DEFINITIONS

- **Chemical Speciation Network (CSN):** EPA's PM_{2.5} sampling network, with sites located principally in urban areas.

4. HEALTH AND SAFETY WARNINGS

The Epsilon 5 produces X-rays which can be dangerous when appropriate precautions are not followed. For safety precautions refer to the Epsilon 5 EDXRF Spectrometer System User's Guide and UC Davis Environment Health and Safety (EH&S) website (<http://ehs.ucdavis.edu/>).

5. CAUTIONS

Once filters are placed into cups, they can be identified by manufacturer's serial number (the manufacturer serial numbers are manually typed into the electronic files; transcription errors may occur). Each petri slide is labeled with a barcode to be scanned using the sample changer software. Be very attentive when transferring filters into cups, and be sure to place every filter in the correct tray as well as the proper position. Similar caution must be taken when unloading filters from the cups. Ensure the filters return to the proper labeled petri slide.

Check the green "Free to Open" light on the control panel of the Epsilon 5 before opening the sample changer cover. Only open the cover if the button is illuminated.

Any errors or unusual occurrences during a sample change must be reported. If the Epsilon 5 generates an error message or has a software malfunction, record it in both the physical station log book and the Microsoft Access log on the desktop. CSN requires the analysis of all samples including invalid and damaged samples. If a filter is dropped or appears unusual (hole, particles, uneven sampling, etc.) add appropriate comments to the electronic records in the CSN Data Management Site. No notes are required for the electronic or physical log books.

6. INTERFERENCES

Not applicable.

7. PERSONNEL QUALIFICATIONS

Only trained lab personnel designated by the Laboratory Manager may perform sample changes for CSN. Personnel must complete XRF courses, training and certifications as required by UC Davis EH&S, and instrument specific training with the Lab Manager.

6. EQUIPMENT AND SUPPLIES

- a. Sample holder insert
- b. Sample holder cups
- c. 47 mm Filter holders
- d. 8-position sample trays (labeled A-F)
- e. 4-position sample tray (labeled S)
- f. Forceps
- g. XRF Queue files generated from CSN Data Management Site
- h. Barcode scanner
- i. Log books (physical and electronic)
- j. Petri slide holder
- k. Rubber bands

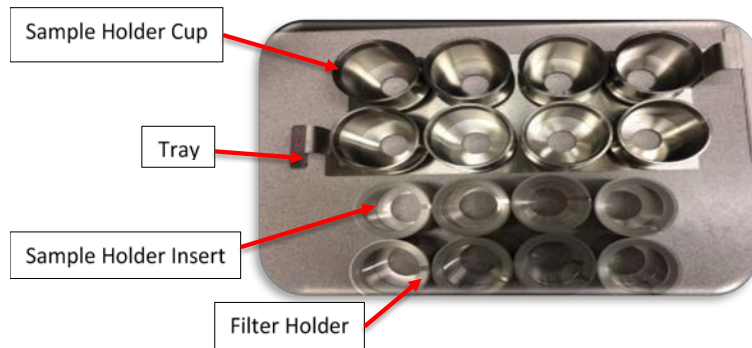
7. PROCEDURAL STEPS

In order to meet the reporting timeline for the CSN network the Epsilon 5 instruments run 24-hours/day, seven days a week. In addition, since the samples have cold storage requirements, it is important to unload sample trays as soon as possible after analysis. Load minimum number of samples each morning and afternoon to minimize the length of time that samples are kept outside of refrigeration.

7.1 Overview and General Definition

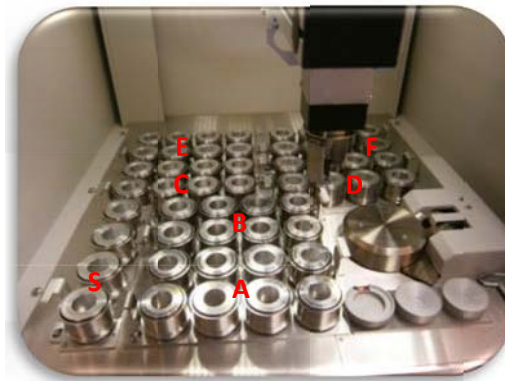
The sample assembly components are shown in Figure 1. For CSN, 47 mm filters are used with a custom made filter holder.

Figure 1: 8-position tray with sample assembly components.



The Epsilon 5 sample changer compartment holds six 8-position sample trays labeled A-F (Figure 2). Positions in the tray are numbered #1-8. There is also one 4-position tray labeled S. The S tray includes 1 or 2 multi-elemental reference filters, and a MTL Teflon® blank. Each Epsilon 5 has an assigned “S” tray that is analyzed daily for monitoring sampling performance.

Figure 2: Sample changer compartment with trays.



The letter on the handle of each sample tray matches an etched letter on the surface of the sample changer (Figure 3). The trays are keyed to prevent them from being loaded backwards in the sample changer; this ensures samples #1-8 are in the proper orientation.

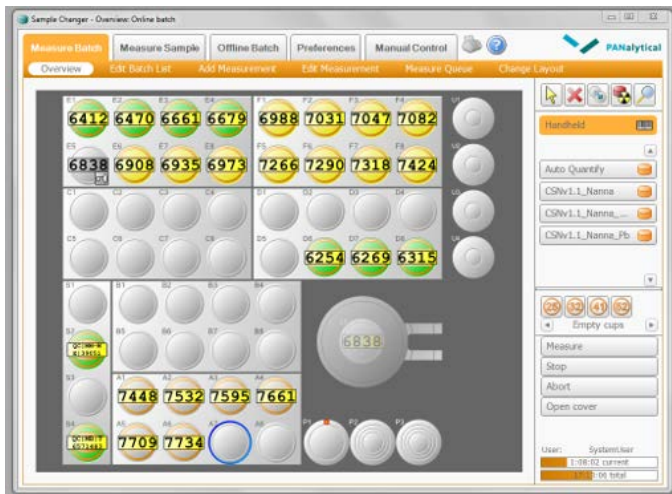
Figure 3: Letter on Tray handle matches etched letter on the instrument table



The Epsilon software displays the configuration of the trays in the compartment. The individual samples are color-coded based on the analysis status (Figure 4):

- Orange/Gray: Not queued to be measured (Ex. Tray A)
- Yellow/Orange: Queued to be measured (Ex. E6-E8, Tray F,)
- Green/Orange: Measured and okay (Ex. Trays S, D, E1-E4)
- Gray/Yellow: Currently analyzing (E5)

Figure 4: Status of sample analysis differentiated by color-code.



The samples loaded in the sample changer compartment correspond with the empty petri slides located in the petri slide holder (Figure 5). Each petri slide holder is labeled with the instrument name (Odin, or Nanna). Each compartment is labeled with a letter that represents one of the XRF sample trays (A-F, S). Petri slides do not stack perfectly into the holders; wrap the petri slides with rubber bands and place in the correct compartment (the barcode labels may be placed on the top or bottom of the petri slide).

Figure 5: Petri slide holder



7.2 Procedure for Unloading Analyzed Samples

- 1) Sample changes can be made while the instrument is analyzing as long as the green *Free to Open* light is illuminated (Figure 6).

Figure 6: Green “Free to Open” light



- 2) CSN samples are assigned an XRF tray and position during the loading process. The procedure is unique to CSN and allows flexibility for prioritizing sample analysis. At the end of the week a laboratory technician may load a partial tray in order to maximize the number of samples analyzed over the weekend. Before loading, check the COC for notes, and never remove a tray if the instrument is analyzing a sample from the tray. Removing a tray may result in alignment issues, errors, or a stop in analysis.
- 3) Note which trays have completed analysis. The trays are unloaded in alphabetical order.
- 4) After making sure all 8 samples in the tray have been analyzed, open the sample changer cover and remove the first tray of filters. As a precautionary measure, perform sample changes for one instrument at a time.
- 5) Set the tray on the desk or sample handling table and close the sample changer cover (Figure 7).

Figure 7: Tray with analyzed samples



- 6) Remove the petri slides in the corresponding compartment of the petri slide holder. Typically, the top petri slide will be flagged with a sticky tab to indicate it is the first tray for unloading. Place the stack of petri slides on the table. The petri slide on top of stack corresponds to the first position in the tray. Confirm that information on the petri slide sticker matches the filter barcode by mousing over the filter identity in the sample charger screen.
- 7) Pick up the sample holder cup from position #1. Place one finger on the inner sample holder insert for support, then turn the sampling cup upside down and line it above the first petri slide (Figure 8). Only handle one filter at a time.

Figure 8: Sample holder cup handling and positioning




- 8) Remove the sample holder cup by lifting it up and off of the sample holder insert. Pick up the filter by its outer support ring using forceps and place it in the petri slide. Make sure the sample side remains face-up.
- 9) Place the Petri slide with the long side to the left in the petri tray. Confirm that it is the correct tray by examining the filter contents and checking the COC.
- 10) Place the sample holder insert back into the sample holder cup.
- 11) Repeat steps 5-8 for positions 2-8, and then for the rest of the completed trays except for the *S* tray. The *S* tray should not be unloaded during routine sample changes (see Section 7.8 for instructions regarding analysis of the *S* tray). Empty trays may be placed back into the sample changer compartment if necessary to keep them out of the way until loading occurs.
- 12) The Teflon® filters have the manufacturer's serial number; however it may be difficult to identify the sample side for lightly sampled filters. Therefore, it is extremely important to keep everything in the proper order while the filters are

separated from their corresponding petri slides. It is very important to handle only one filter at a time.

13) Place analyzed samples into the corresponding tray, as noted in the COC.

7.3 Removing the Analyzed Filters from the Queue

After removing the analyzed filters, they need to be deleted from the Epsilon Sample Changer queue. Towards the right side of the sample changer window, there is a symbol . Click on the symbol to activate the *Delete* function. Then, move the mouse over the middle of the picture of the completed tray and click once to delete the entire tray. It is also possible to delete one sample at a time by clicking on each sample individually. Do not delete the *S* tray.

7.4 Sample Integrity

Prior to loading samples, verify the integrity of the samples. All samples are analyzed regardless of flag status; however, it is important to check the sample for damage such as holes, uniformity, scratches, insects etc. Any damage is immediately reported in the CSN Data Management Site under *Filter Details*. Add the AQS code FX (filter integrity issue) and state the issue in the comment section. Refer to *UCD CSN TI 302#B: Receiving and Inventorying of CSN Samples* further instructions.

7.5 Adding New Samples with XRF Queue file

An XRF queue file is generated based on the samples received within a batch. The sample changer software uses the data within the queue file to link the Lab Analysis ID Barcode with the Sample identity and the instrument specific application. The Lab Analysis ID Barcode (Barcode ID) can be located on the top or bottom of the petri slides. The Barcode ID is scanned into the sample changer software, the tray and position number are assigned.

Review the notes on the COC to find the barcode ID and the tray number for the next sample to be loaded. Retrieve the sample trays from the refrigerator. Minimize the length of time the samples spend outside the refrigerator. Generally, load two XRF trays in the morning, three XRF trays for the night, and full instrumental load for the weekend.

Using the XRF queue file and sample changer software allows sample loading flexibility because the XRF tray and position number are assigned at scanning (ny filter can be loaded at any time to any position or tray). However, it is important to keep good notes of the analyzed samples to prevent duplicate analysis. Make all notes in the COC (Figure 9).

Figure 9: Chain of Custody form includes the samples' assigned instrument name, tray, and position number.

The image shows a 'CSN Laboratory Chain of Custody Form' for the date 4/26/2016 and location BARNARD. It includes fields for 'Ship Date and Name', 'Receive Date and Name', 'Analysis Request ID', 'Intended Sample Date', and 'Set #'. A table lists 12 'Filter Analysis ID's, each with a barcode, 'Filter Type' (Teflon), and 'Analysis Requested' (XRF). The 'Invalid?' column has handwritten 'Nanna (E1)' and 'Nanna (E1)' next to the first and last rows, respectively. Red boxes and arrows highlight these entries with labels: 'Tray Number', 'First sample: Instrument name, Tray, Position', and 'Last sample: Instrument name, Tray, Position'. A 'Batch number' is also indicated. The form is labeled 'Page 1 of 90'.

Select the tray and position where the first samples will be loaded. For example: Tray B position 1 (B1). Highlight the *Handheld* button. The button will turn orange and the *Scan barcode for position: B1* will open (Figure 10).

Figure 10: Scan barcode window.


The image shows a 'Scan barcode for position: B1' window. It features a text input field labeled 'Barcode ID:' and two buttons at the bottom: 'Next' and 'Close'.

Scan or type the Barcode ID, and the Sample ID will show up in the sample changer window. Mouse over the Sample ID to see the Barcode ID, the application, position, and sample status.

- 1) Using forceps, transfer the sample into the sample cup (Section 7.7).
- 2) The sample changer software will automatically move to the next available position. Repeat steps 2-3 until all eight positions are filled.
- 3) Verify the set of samples are the next 8-samples in the COC. Write down the Epsilon name, and tray/position number for the first and last samples in the tray (Figure 9). For example: Nanna B1 and Nanna B8.

7.6 Loading New Samples into the Sample Chamber

- 1) Remove the designated 8-position tray from the Epsilon and place it on the sample handling table.

- 2) Remove the sample holder cups from the tray and place them directly behind the samples.
- 3) Scan the petri barcode into the Sample Charger Software. Then open the first petri slide and use forceps to lift the filter by the outer support ring and place it on the filter holder. The sample side should be facing up. It is important to handled only one filter at a time.
- 4) Place the sample holder cup over the top of the sample holder insert. Tip the cup slightly and support the filter mount cup by applying gentle pressure on the bottom of the cup. Then, turn the assembly upside down and place it in the first position of the tray.
- 5) Continue with the rest of the samples, moving to position 2, 3, etc., until all eight have been loaded. Double-check that the physically-loaded filters match the filters displayed on the *Overview* screen of the Sample Charger software.
- 6) Stack the empty petri slide with the far-left petri (for position 1) at the top of the stack. Wrap the petri slides with rubber bands and place the petri slides in the appropriate compartment (A-F) of the petri slide holder.
- 7) Make sure the green “Free to Open” light is illuminate. Then, open the sample chamber cover.
- 8) Load the filled sample tray into the sample chamber compartment with the letter written on the sample tray matching the etched letter in the sample chamber (Figure 3).
- 9) Close the sample chamber cover and repeat steps 2-10 for the remaining empty trays.
- 10) Add the newly populated filters to the analysis queue by clicking the  symbol (in the *Overview* screen of the *Measure Batch* tab. Then, click on the newly-loaded trays. The samples will change from gray to yellow.
- 11) Next refer to Section 7.9 for Recoding Sample Changes.
- 12) Filters not included in analysis must be placed in the refrigerator until ready for analysis.

7.7 Adding QC Samples utilizing “Add Measurement” function

- 1) To add QC samples to the queue, double-click on the first position that will be loaded in the sample changer software. This will open the *Add Measurement* screen.
- 2) The *location* should already be the correct position.
- 3) For *Application*, select the current version of the application being run on the particular Epsilon.
- 4) Place the cursor in the *Sample Identification* box.
- 5) Type the SampleId for the QC sample. Review acceptable QC code name convention in.
- 6) Press F9 to save the entry and the program will automatically move on to the next

position.

7.8 Analyzing the QC Samples

The QC samples are loaded to the *S* tray and are analyzed once per day. The current method is to add the *S* tray to run directly after the current tray being analyzed, and then continue with the A-F cycle. If the queue needs to be re-ordered for the *S* tray to be analyzed, see Section 8.1.

7.9 Recording Sample Changes


- 1) Each instrument has its own physical logbook as well as an electronic log in Microsoft Access. The physical logbook is labeled with the instrument name and is located on the shelf above the respective computer. The Access log is located on the computer desktop.
- 2) In the physical logbook, on the first available row, record the date, time, first and last samples loaded, and the letters of the trays loaded. Sign the entry with full name. Make sure to follow the same format as previous entries.
- 3) Open the Access logbook by clicking on the icon entitled *Enter_LogBooks.accdb*. Click on *Add New Record*. The date and time will automatically fill in. The default Code Action is *LD* (loading). Fill in the first and last filter information and the *Initials* box. Then click, *Save This New Record* before closing.

8. ADDITIONAL CHECKS/PROCEDURES

8.1 Changing the Order of the Queue (Usually for S Tray)

- 1) In the *Measure Batch* tab, click on *Measure Queue*.
- 2) Highlight the samples of the *S* tray (or whatever samples need to be moved).
- 3) Use the *Move Up* or *Move Down* buttons on the right as many times as needed to change the order.

8.2 Aborting Sample Analysis

- 4) To abort a sample while in mid-analysis, go to the *Measure Batch* tab, then *Overview*.
- 5) Click the *Abort* button towards the bottom right.
- 6) The instrument will first come to air; then, the sample will be removed and put back to its original tray position. No new analysis will begin until the *Measure* button is clicked or the  button is used to re-queue.

9. QUALITY ASSURANCE AND QUALITY CONTROL

Not Applicable.

10. REFERENCES

Not Applicable.