UCD CSN Technical Information #302C

Sample Changes for 8-Position Trays

Chemical Speciation Network
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Date Modified	Initials	Section/s Modified	Brief Description of Modifications
11/26/2018	MGN	all	Small editing changes; added pictures
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1. PURPOSE AND APPLICABILITY

The purpose of this technical information (TI) document is to describe the process of loading and unloading samples using standard cups in 8-position trays in the Epsilon 5 Energy Dispersive X-ray Fluorescence (EDXRF, referred to as XRF) instruments.

2. SUMMARY OF THE METHOD

XRF queue files are generated for each CSN batch number, which include the application name, Barcode ID and Filter ID. The scanning capabilities of the PANalytical software are 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 form (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

Not applicable.

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 (https://safetyservices.ucdavis.edu/).

5. CAUTIONS

Once filters are placed into cups, identification is possible with the manufacturer's code. The manufacturer codes are manually typed into the electronic files; therefore, transcription errors may occur. Each Petri slide is labeled with a barcode that can 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. Caution must also be taken when unloading filters from the cups. Ensure the filters return to the properly 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 to the Lab Manager. If the Epsilon 5 generates an error message or has a software malfunction, record the error in both the physical station log book and the Excel logbook. CSN

requires the analysis of all samples including invalid and damaged samples. If a filter is dropped or appears unusual (e.g., hole, particles, uneven sampling, etc.), add descriptive comments to the electronic records in the CSN Data Management Site. Filter integrity comments are not required for the electronic or physical logbooks.

6.   INTERFERENCES

Not applicable.

7.   PERSONNEL QUALIFICATIONS, DUTIES, AND TRAINING

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

8.   EQUIPMENT AND SUPPLIES
- Sample holder insert
- Sample holder cups
- 47 mm Filter holders
- 8-position sample trays (labeled A-F)
- 4-position sample tray (labeled S)
- Forceps
- XRF Queue files generated from CSN Data Management Site
- Barcode scanner
- Log books (physical and electronic)
- Petri slide holder
- Rubber bands

9.   PROCEDURAL STEPS

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

9.1. Overview and General Definition

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

Figure 1. 8-position tray with sample assembly components.
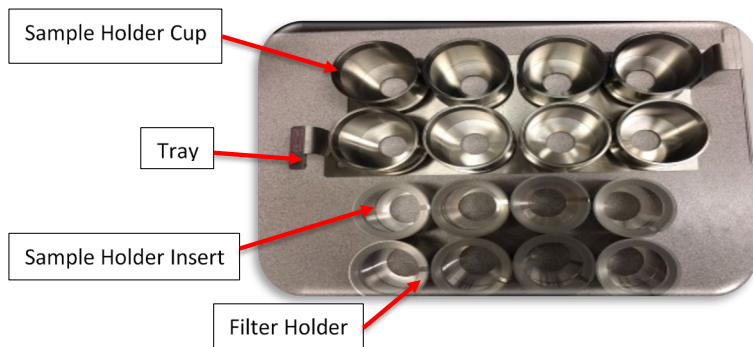
The Epsilon 5 sample changer compartment holds six 8-position sample trays labeled A-F (Figure 2). Positions in the tray are numbered #1-8. There is also one 4- position tray labeled *S*. The *S* tray includes 1 or 2 multi-elemental reference filters, and an MTL Teflon blank. Each Epsilon 5 has an assigned "S" tray that is analyzed daily for monitoring the analyzers' 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 5 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 (e.g., Tray A)
- Yellow/Orange: Queued to be measured (e.g., E6-E8, Tray F,)
- Green/Orange: Measured and okay (e.g., Trays S, D, E1-E 4)
- Gray/Yellow: Currently analyzing (E5)

Figure 4. Status of sample analysis differentiated by color code.
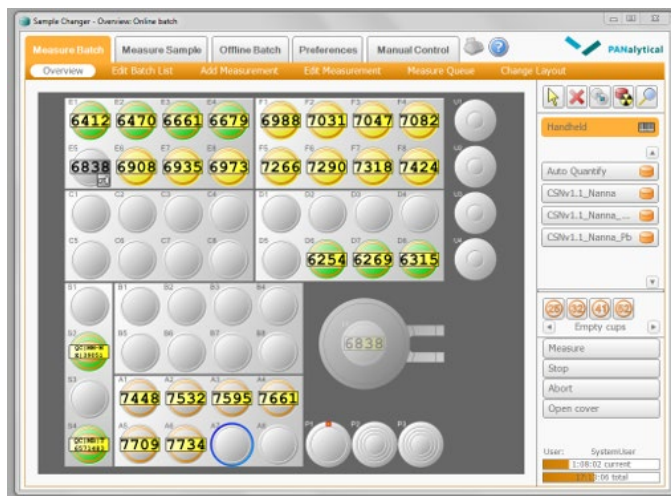
The samples loaded in the sample changer compartment correspond with the empty Petri slides located in the Petri slide holder (Figure 5). Each Petri slide holder is labeled with the instrument name (e.g., Odin or Nanna). Each compartment is labeled with a letter that represents one of the XRF sample 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.

9.2.   **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. Never remove a tray if the instrument is analyzing a sample from the tray. Removing the active tray may result in alignment issues, errors, or a halt in analysis.

3) Note which trays have completed analysis. The trays are unloaded in alphabetical order. After unloading the "F" tray continue with the "A" tray.

4) After checking all eight 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 hovering the mouse cursor over the filter identity in the sample changer 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 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. Empty trays may be placed back into the sample changer compartment to keep them out of the way until loading occurs.

12) Teflon filters have the manufacturer's serial number imprinted on them; 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.

9.3. Removing the Analyzed Filters from the Queue

After removing the analyzed filters, delete the sample information from the Epsilon 5 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.
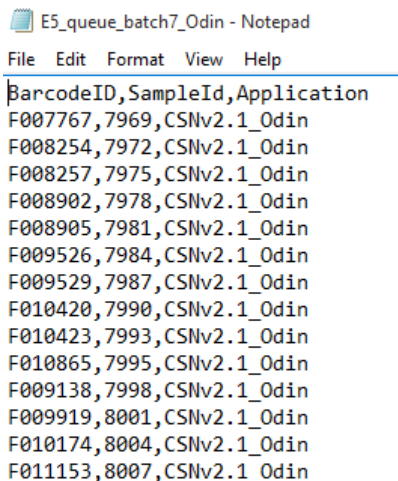
9.4. XRF Queue File

An XRF queue file is generated for all samples regardless of status. In addition, the file may also include laboratory blanks when available.

To generate the XRF queue file, access the CSN Data Management Site and select *Batches* from the top menu. Scroll to select *View Record Details* next to the batch number and select *Generate XRF Queue*. Choose the application associated with the XRF instrument. If lab blanks are received with the current batch, leave selected, otherwise exclude the lab blanks. Then click **Go**.

Save the queue file to *U:\IMPROVE_Lab\XRF_Epsilon5\CSN\Queue files\Queue files\Year\instrument name*. Change the file name to *E5_queue_batch#¬_instrument name* (Figure 9).

Figure 9. XRF queue file.

Upload the queue file to the instrument in the directory

C:\PANalytical\Epsilon5\Programs\Changer\Barcode Scripts and save the file as *E5_queue*. Replace the current file in the folder. Follow the same procedure to generate the queue for each instrument.

PANalytical software will only recognize the queue file in *txt* format. The operator can generate the script file directly from the instrument's computer. Use caution when saving the file to the instrument.

9.5. 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 reported in the CSN Data Management Site under *Filter Details* by approved personnel. 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* for further instructions.

9.6. Adding New Samples with the 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. After the Barcode ID is scanned into the sample changer software, the tray and position numbers 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 the sample changer software allows sample loading flexibility since the XRF tray and position number are assigned at scanning (any filter can be loaded at any time to any position or tray). However, it is important to keep descriptive notes of the analyzed samples to prevent duplicate analyses. Make all notes on the COC (Figure 10).

Figure 10. Chain of Custody form includes the samples' assigned instrument name, tray, and position number.

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 window* will open (Figure 11).

Figure 11. Scan barcode window.

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 9.7).
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 5 name, and tray/position number for the first and last samples in the tray (Figure 9). For example: Nanna B1.

9.7. Loading New Samples into the Sample Chamber

1) Remove the designated 8-position tray from the Epsilon 5 and place it on the sample handling table.

2) Remove the sample holder cups from the tray and place them on the counter.

3) Scan the Petri barcode into the sample changer 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 work with one filter and one Petri slide at a time to prevent sample swaps.

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 from positions two through eight. Double check that the physically loaded filters match the filters displayed on the *Overview* screen of the sample changer software.

6) Place empty Petri slides face down. After all samples in the tray have been loaded, flip the Petri slide stack so that the first position will be at the top of the stack and the eighth position will be at the bottom of the stack. Wrap the Petri slides with rubber bands and place them in the appropriate compartment (A-F) of the Petri slide holder.

7) Make sure the green "Free to Open" light is illuminated. 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 on the sample chamber table (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 9.10 for Recording Sample Changes.

12) Filters not included in analysis must be kept in the refrigerator until ready for analysis.

9.8. Adding QC Samples utilizing "Add Measurement" function

1) To add Quality Control (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 5.

4) Place the cursor in the *Sample Identification* box.

5) Type or scan the QC code for each QC sample.

6) Press F8 to save the entry and the program will automatically move on to the next position.

9.9. 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 reordered for the *S* tray to be analyzed, see Section 10.1.

9.10. 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 your 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.

9.11. Analyzing Samples from Archive

Review *UCD CSN SOP #901: Long-term Archiving of Filters* and follow the procedures for retrieving samples from archive. Follow section 9.4 to generate a new XRF queue file.

To load samples to the XRF queue follow the steps from section 9.6.

10. ADDITIONAL CHECKS/PROCEDURES

10.1. 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 whichever 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.

10.2. Aborting Sample Analysis

1. To abort a sample during analysis, go to the *Measure Batch* tab, then *Overview*.
2. Click the *Abort* button towards the bottom right.
3. The instrument will first depressurize. 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.

11. QUALITY ASSURANCE AND QUALITY CONTROL

Not Applicable.

12. REFERENCES

Not Applicable.