# UCD CSN Technical Information #302C Sample Changes for 8-Position Trays

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

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# **DOCUMENT HISTORY**

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|          |                 |             |                       |  |

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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 XRF sample 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 or designated trainer.

### 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

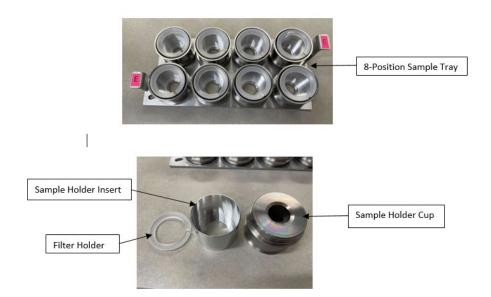
### 9. PROCEDURAL STEPS

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, the number denoting each position is etched on the tray. There is also one 4-position tray labeled *S*. The *S* tray houses the daily quality control filters consisting of a Teflon® blank and a multi-elemental reference filter. Each E5 has an assigned "S" tray that is analyzed daily for monitoring the analyzers' performance.

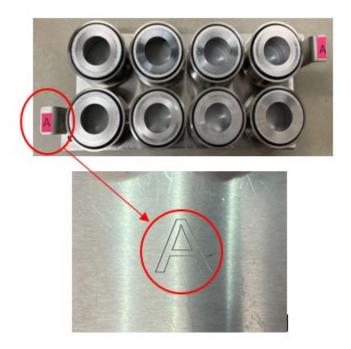
Figure 2. Sample changer compartment with trays.



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.

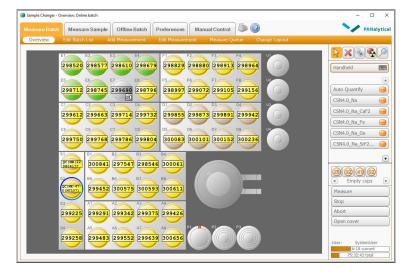
Figure 3. Letter on Tray handle matches etched letter on the instrument table.



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 D)
- Yellow/Orange: Queued to be measured (e.g., Trays A-C, E8, F, S)
- Green/Orange: Measured and okay (e.g., positions E1-E6)
- Gray/Yellow: Currently analyzing (position E7)

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). Each compartment is labeled with a letter that represents one of the E5 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

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.



- 1) CSN samples are assigned an XRF tray and position during the loading process. The procedure 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 unloading/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.
- 2) Visually check which trays have completed analysis, this is shown on the sample changer software. The trays are unloaded in alphabetical order. After unloading the "F" tray continue with the "A" tray.
- 3) 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 tray and one instrument at a time.
- 4) Set the tray on the sample handling bench and close the sample changer cover (Figure 7).

Figure 7. Tray with analyzed samples.



- Should be stack of Petri slides in the corresponding compartment of the Petri slide holder. Place the stack of Petri slides on the sample handling bench.
- The Petri slide on the top of stack corresponds to the sample in the first position of the tray. Confirm the information on the Petri slide sticker matches the filter barcode by hovering the mouse cursor over the filter identity in the sample changer screen. Place this Petri slide on the sample handling bench.
- 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

place it on the sample handling bench (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. Place the sample holder cup back in the tray position it was removed from. 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. Start a new stack of Petri slides with the filter that was just unloaded. Remember to keep this stack separate from the empty Petri slides.
- 9) Place the empty sample holder insert back inside the empty sample holder cup. Position the empty sample holder insert with the filter holder visible (this is opposite to how the sample holder insert is positioned when there is a filter loaded.
- 10) Repeat steps 6-9 for positions 2-8. Once all 8 filters have been unloaded, the filters need to be placed back in the Petri tray. The stack of Petri slides for the unloaded filters is in the opposite order of how they need to be stored. Reverse the order of the stack so the filter unloaded first is now on top and the filter unloaded last is now on bottom. Be careful to not mix up the order of the filters
- 11) Verify the Barcode ID and manufacturer number with the COC before placing the Petri slide in the Petri tray. The COC is also used to confirm the filter is being placed in the correct Petri tray and position. Place the Petri slide with the long side to the left in the Petri tray, this signifies the filter has been analyzed at XRF.
- Repeat steps 5-11 for the rest of the completed trays except for the *S* tray. The *S* tray should not be unloaded during routine sample changes unless there are samples loaded to S3 and S4. Empty trays may be placed back into the sample changer compartment to keep them out of the way until loading occurs.
- 13) Teflon filters have the manufacturer's serial number imprinted on them; however, it may be difficult to identify the sample side for lightly sampled

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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.

### 9.3. Removing the Analyzed Filters from the Queue

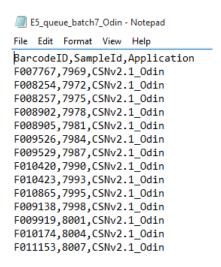
After removing the analyzed filters, delete the sample information from the E5 Sample Changer screen. Towards the right side of the sample changer window, there is a symbol, Elick on the symbol to activate the *Delete* function. Then, move the mouse over the middle of the picture of the completed tray 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 daily QC samples from the *S* tray.

### 9.4. XRF Queue File

An XRF queue file (figure 9) is generated for all samples regardless of status. In addition, the file may also include laboratory blanks. XRF queue files are generated when CSN batches are received, for additional information on creating queue files refer to, UCD CSN TI 904B Receiving and Inventorying CSN Teflon Samples.

XRF queue files saved on the U drive will need to be copied over to the instrument prior to starting analysis for a batch. Go to U:\IMPROVE\_Lab\XRF\_Epsilon5\CSN\Queue files and click on the relevant year, then click on the instrument needed. The batch number is saved as part of the file name, click on the file with the correct batch number and select copy.

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.

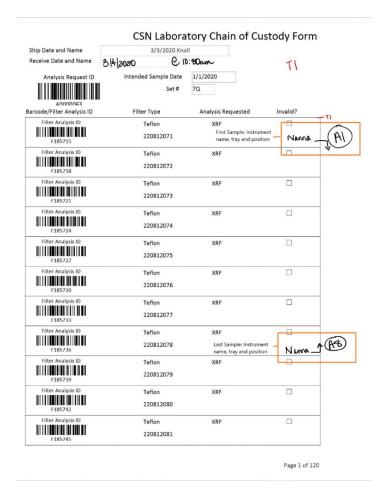
### 9.6. Adding New Samples with the XRF Queue File

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 Filter Barcode ID with the Sample identity and the instrument specific application. The 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, three or four XRF trays for overnight analyzing, 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.



# 9.7. Loading New Samples Into the Sample Changer

- 1) Remove the designated 8-position tray from the E5 and place it on the sample handling bench.
- 2) Open the binder containing the COC for the instrument being loaded, the binder is stored on a shelf above the sample handling bench.
- 3) Remove the tray of samples from the refrigerator that corresponds to the pages of the COC being loaded to the given instrument.
- 4) Select the next 8-samples available for analysis per the COC. Filters ready for analysis will not have any notes recorded in the COC and the Petri slides will have the long side to the right. Write down the E5 name, and tray/position number for the first and last samples in the tray as seen in Figure 10.
- 5) Place the stack of 8 filters on the sample handling bench, remember to keep the Petri slides in the same order as the COC.

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- 6) Remove the sample holder inserts from the sample holder cups and place them on the sample handling bench.
- 7) Click on **handheld scanner** on the sample changer software screen. Then click on the first empty position being loaded, typically position 1 of the tray you will be loading. This will open the "scan barcode for position" popup as seen in figure 11.

Figure 11. Scan barcode window.



- 8) Using the handheld scanner, scan the barcode label of the first Petri slide into the sample changer software. When successfully scanned the filter ID will populate on the sample changer software. To see the barcode ID, use the mouse to hover over the filter ID, a pop-up will appear with additional information.
- 9) Then open the first Petri slide and use forceps to lift the filter by the outer support ring. Filters should be sample side (deposit side) up in the Petri slide, visually inspect both sides of the membrane to ensure the filter is loaded correctly. If neither side has visible deposit then always assume the deposit side is how the filter was removed from the Petri slide. Place the filter on the filter holder, deposit side face up. Remember to work with one filter and one Petri slide at a time to prevent sample swaps.
- 10) 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 position of the tray the filter was scanned into on the screen, typically position 1 is loaded first.
- 11) Place the lid back on the empty Petri slide and place the empty Petri slide face down (starting a new stack, the next empty Petri slide will be added to this stack in the same manner until all 8 empty Petri slides have been added).
- 12) Repeat steps 8-11 for positions 2-8 in the tray.
- 13) 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. Double check the order of the empty Petri slides matches the physically loaded filters on the

- "overview" screen. Wrap the Petri slides with rubber bands and place them in the appropriate compartment (A-F) of the Petri slide holder.
- 14) Make sure the green "Free to Open" light is illuminated. Then, open the sample chamber cover.
- 15) 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).
- 16) Close the sample chamber cover and repeat steps 3-16 for the remaining empty trays.
- 17) 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.
- 18) Log the sample change, refer to Section 9.10 for Recording Sample Changes.
- 19) Filters not being loaded for analysis are placed back in the refrigerator.

### 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, it can be manually changed if needed.
- 3) For *Application*, select the current version of the application being run on the particular E5.
- 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 daily QC samples are loaded to the *S* tray and are analyzed once per day.. 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 first initial and full last 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 in 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.