TITLE: ANALYSIS BATCH PREPARATION AND SHIPMENT

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Prepared by: Justin Knoll
Program Manager

justin.knoll Digitally signed by justin.knoll Date: 2018.05.30 15:54:41 -04'00'

Reviewed by: Bill Barnard
Database Manager

Bill.Barnard Digitally signed by Bill.Barnard DN: cn=Bill.Barnard Date: 2018.05.30 14:45:30 -04'00'

Reviewed by: Anne Glubis
Quality Assurance Manager

anne.glubis Digitally signed by anne.glubis Date: 2018.05.30 17:00:27 -04'00'

Annual Review			
Reviewed by:	Title:	Date:	Signature:

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1 Procedures

1.1 Scope and Applicability

The Filter Shipping and Handling Unit (FiSH) is responsible for the regular shipment of exposed and blank ambient air sampling filters to analytical laboratories along with the transfer of data corresponding to said filters.

1.2 Summary of Method

Shipments of filters and corresponding data are known as Analysis Batches. Each Analysis Batch includes filters and electronic records for a specific time period, typically between two weeks and a month. The data is sent electronically and filters are shipped in coolers to the respective analytical lab. The procedures below describe the process to prepare and ship an Analysis Batch to the respective lab.

1.3 Definitions

Analysis Batch	A set of exposed filter media and associated electronic records to be shipped to contract laboratories
EPA	United States Environmental Protection Agency
CSN	EPA Chemical Speciation Network
FiSH	Wood Filter Shipping and Handling Unit, Newberry, FL
DRI	Desert Research Institute, Reno, NV
PCOC	Preliminary Chain of Custody form
QA	Quality Assurance

1.4 Health & Safety Warnings

Employ lifting equipment and other handling aids to eliminate the need to move heavy objects manually. Do not strain to lift any object.

1.5 Cautions

Not following procedures in order may result in inaccurate filter records.

1.6 Interferences

Not Applicable

1.7 Personnel Qualifications/Responsibilities

Personnel should be certified for these procedures by a qualified person following standards set forth in GLO3110-001 *Training Chemical Speciation Network Filter Shipping and Handling Personnel*. Certification shall be documented on the FiSH Demonstration of Capability form GLF-3110-001.

1.8 Equipment and Supplies

To perform these procedures, it is necessary to have access to the CSN QA front end of the CSN Database along with a new Analysis Batch Checklist. Necessary equipment also includes exposed

filter media in petri slides, trays for petri slides, 12” x 12” resealable plastic bags for trays, petri boxes, frozen ice packs and hard sided coolers for shipment.

1.9 Procedures

1.9.1 Analysis Batch and Preliminary Chain of Custody (PCOC) Creation

Create a batch based upon the intended sample date range of a group of filters.

a) Log into the computer work station and open the CSN Database QA Front End by double clicking QA Database icon on desktop.
b) Open the Analysis Batch Review and Update tab (Figure 1), Select start and stop date of batch range from drop down menu. Click the Add New Analysis Batch ID button, select the next Analysis Batch number , incremented by one from the previous number (current format is an A followed by a seven digit number) for each Analysis Batch shipment to the contract analytical laboratories. Include information related to the ship date and the person generating the Analysis Batch, but do not add an entry in the ShipLabOutDate field until the day of shipment. Close table.
c) Creation of PCOCs: This form is initiated using the CSN QA Dashboard shown in Figure 4 by clicking Lab COC Report button. Clicking on that button opens the Print Lab COC Report form shown in Figure 5. To print the preliminary Lab COC Report the user:
d) Chooses an Analytical Batch number from the Analysis Request ID combo box.
e) Selects the filter type from the Select Filter Type combo box and,
f) Clicks on the Print COC form button.
g) After the user has clicked on the Print COC form button, the report will open. The user needs to right click with the mouse to open the menu and then chooses Print to print the report.
h) The report can be closed by clicking on the X in the upper right corner of the report window.
i) Upon closing, with the Lab Filter Detail report still highlighted, select view print preview, inspect for correct Analysis Batch and Filter Type, and then print. Close print preview. Repeat the above process to create PCOCs for the other two filter types. These PCOCs (shown in Figure 6) are used during the filter sorting process 1.9.3.

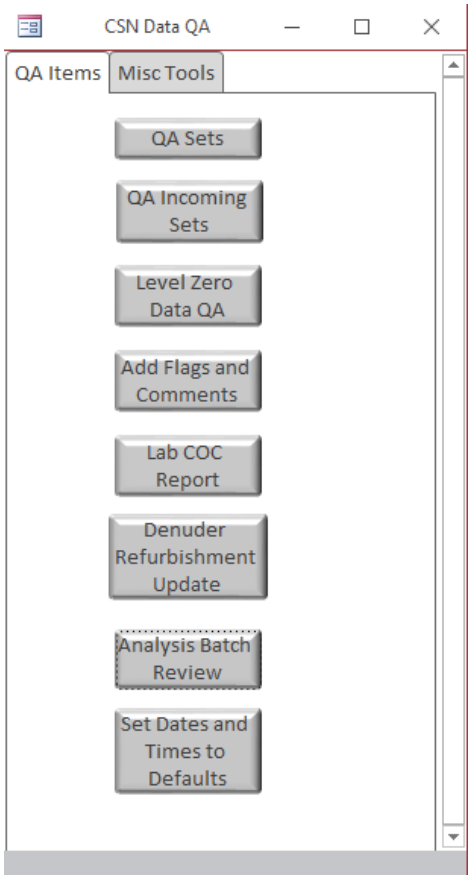

Figure 1. Analysis Request Table

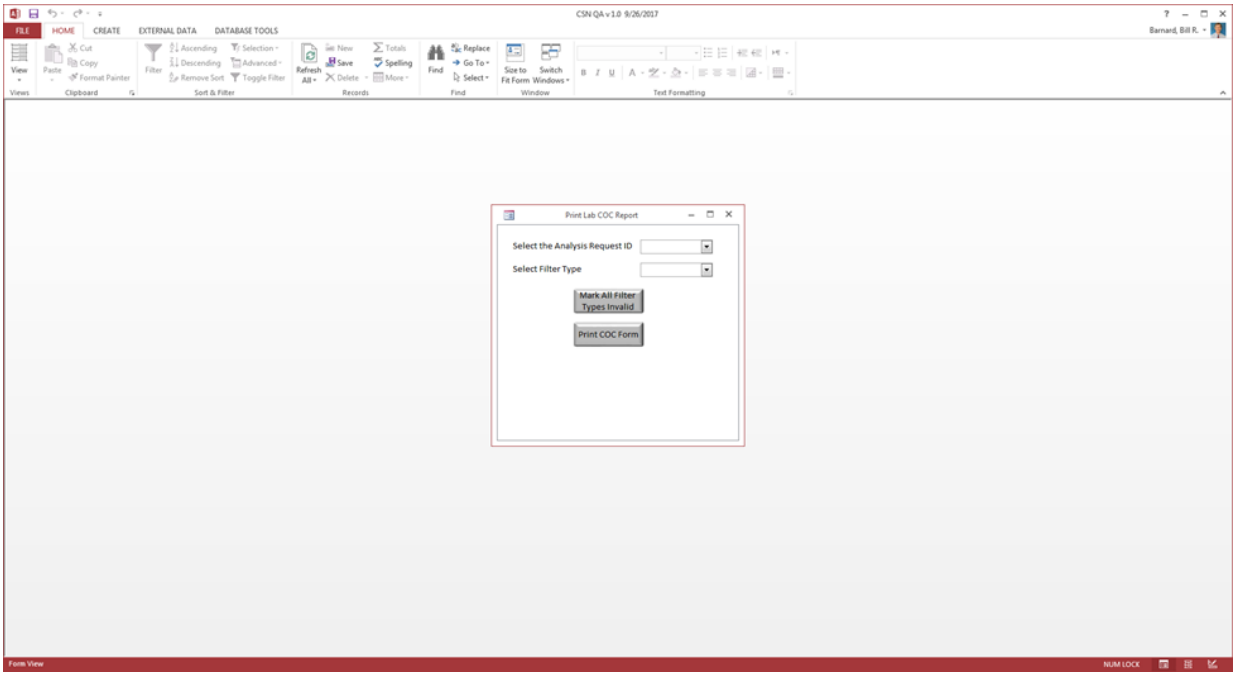

Figure 2. CSN QA Dashboard

CSN Laboratory Chain of Custody Form

Ship Date and Name: 8/21/2017 Knoll
Receive Date and Name:

Analysis Request ID: A0000031
Intended Sample Date: 6/24/2017
Set #: 1

Barcode/Filter Analysis ID	Filter Type	Analysis Requested	Invalid?
Filter Analysis ID F065522	Nylon	Ions	☐
Filter Analysis ID F066000	Nylon	Ions	☐
Filter Analysis ID F066003	Nylon	Ions	☐
Filter Analysis ID F066914	Nylon	Ions	☐
Filter Analysis ID F066917	Nylon	Ions	☐
Filter Analysis ID F066920	Nylon	Ions	☐
Filter Analysis ID F067382	Nylon	Ions	☐
Filter Analysis ID F068072	Nylon	Ions	☐
Filter Analysis ID F068075	Nylon	Ions	☐
Filter Analysis ID F068078	Nylon	Ions	☐
Filter Analysis ID F068525	Nylon	Ions	☐

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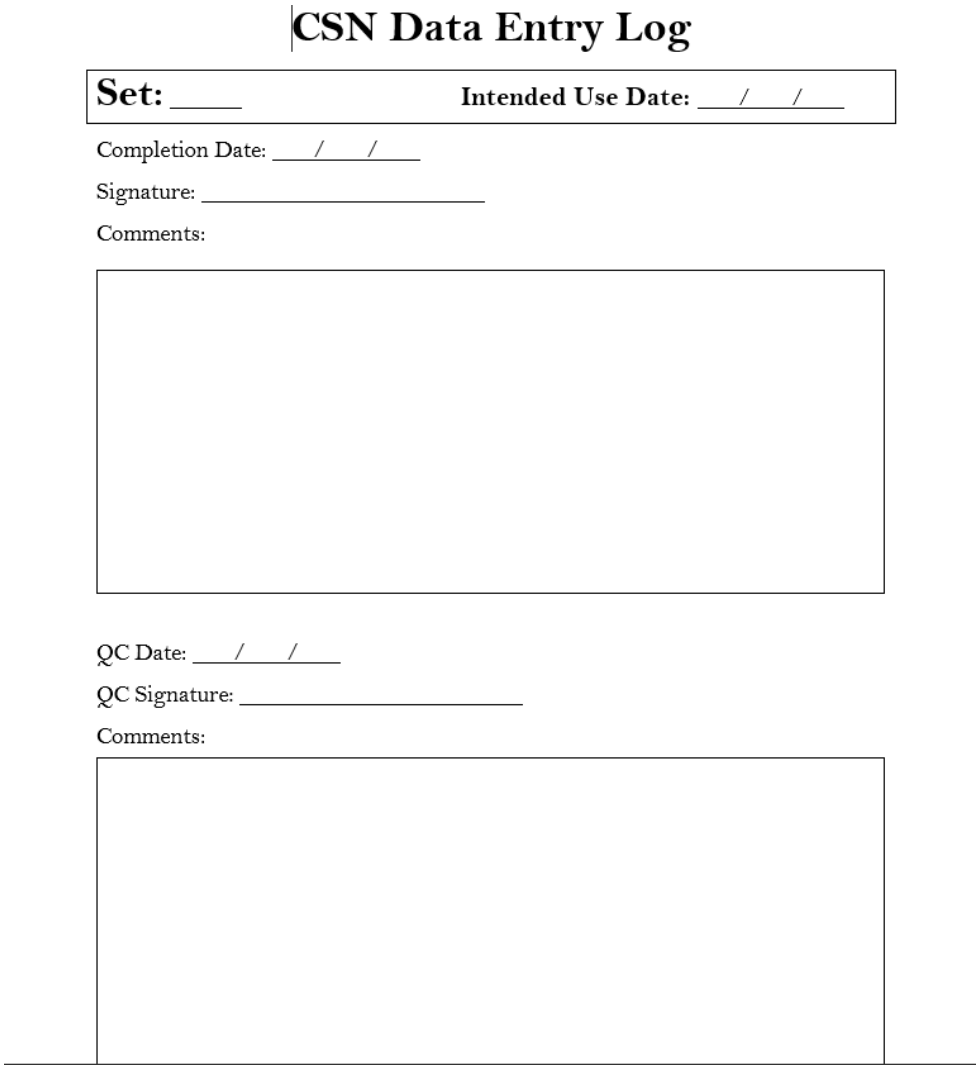
Figure 3. Preliminary Chain of Custody

1.9.2 Data Entry and Review

Confirm that data entry for all data sets included in the Analysis Batch has been completed, quality checks have been performed, and any data entry errors detected during the quality check process have been corrected. Confirmation can be found on the CSN Data Entry Log (Figure 7) which is kept in a file folder with each sample set's Field Site Chain of Custody (FSCOC) forms, Field Sampling Null and Validity Coding Form and the Level 0 Sample Check in Form and then filed in FiSH filing area by intended sample date and set number. Document these actions on the first page of the Analysis Batch Checklist (see Figure 8).



CSN Data Entry Log

Set: _____ **Intended Use Date:** ___/___/___

Completion Date: ___/___/___

Signature: _____

Comments:

QC Date: ___/___/___

QC Signature: _____

Comments:

Figure 4. CSN Data Entry Log

Analysis Batch Checklist

Analysis Batch #_____A0000040_____

Date Range_____2/1/2018-2/28/2018____

Set #	Intended Sample Date	Data Entered By/Date	QA Performed By/Date
3	2/1/18	CG 2/12	MAE 2/12
5A	2/1/18	NP 2/27	MAE 2/28
5Q6Q	2/1-2/4/18	AD 2/26, KL 2/20	MAE 2/28, 2/26
4	2/4/18	RW 2/20	MAE 3/1
5	2/7/18	RW 2/20	MAE 3/1
6A	2/7/18	CG 3/14	MAE 3/15
7Q	2/7/18	NP 2/27	MAE 3/1
1Q2Q	2/10-2/13/18	KL 3/8, 3/16	MAE 3/8, 3/21
6	2/10/18	NP 3/5	MAE 3/7
1	2/13/18	NP 3/6	MAE 3/7
1A	2/13/18	CG 3/20	MAE 3/27
2	2/16/18	NP 3/6	MAE 3/7
3Q4Q	2/16-2/19/18	NP 3/27, AD 3/21	MAE 3/28, 3/21
2A	2/19/18	NP 3/1	MAE 3/1
3	2/19/18	NP 3/6	MAE 3/7
7A	2/19/18	NP 3/1	MAE 3/1
4	2/22/18	HC 3/8	MAE 3/27
5Q6Q	2/22-2/25/18	NP 3/13, 3/14	MAE 3/16
3A	2/25/18	NP 3/26	MAE 3/26
5	2/25/18	HC 3/27 AD corrected	MAE 3/27
6, 7FB	2/28/18	HC 3/22 AD corrected	MAE 3/27
7Q8QFB	2/28/18	NP 3/28	MAE 3/29

Comments:

4/3/18 4/4/18 William R. Barnard 4/3/18
Program Manager Date Quality Reviewer Date Technical Reviewer Date

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Figure 5. Analysis Batch Checklist Page 1

1.9.3 Filter Sorting

1. Following the PCOC sequence (shown in Figure 5), sort the filters for shipment, working from top to bottom and starting on page 1 proceeding to end of final page of PCOC (see Figure 9 for filter sorting guidance).
2. The exposed filters in petri slides are arranged in petri trays in groups of 50 (two rows of 25), with 2 trays to a box. After locating and loading each filter/petri slide into a tray, a checkmark in ink is added next to the Filter Analysis ID to certify that the filter/petri slide has been accounted for.
3. Boxes are identified using permanent marker labeled by Filter Type and order of Filter Analysis IDs within PCOC (e.g., the first 100 filters on the Quartz PCOC would be in Quartz Box 1).
4. If filters are missing during this process, a space is left in the tray to accommodate the filter when it is located.
5. If there are additional filters not listed on PCOC, they will be inserted in sequence, and investigated.
6. Record missing or additional filters, on both the PCOC and petri tray and report them to the Program Manager, who will investigate and attempt to locate any missing filters.

Orientation of the Petri Slides

Use the AMEC set number as the starting point for the orientation of the Petri slide tray. The AMEC set numbers will be on the "front left hand corner" of the Petri slide tray.

When viewed from the top, the first (1st) sample will be in the upper left-hand corner of the petri tray, the 25th sample will be in the bottom left-hand side of the petri tray. The 26th sample will be located on the upper right side of the petri tray and the last (50th) will be in the bottom right side of the petri tray (See picture 1 below). Fill the tray with the petri slides with the long side of the slide pointing to the right (See picture 2 below).

Filling the Petri trays

The Petri trays are filled as listed in the CSN Chain of Custody (COC) forms. The 1st sample in the first tray will also be the first sample in page one of the COC. Continue following COC until all 50 positions have been filled. The 51st sample on the COC will become the 1st sample in the next tray until all samples have been placed in Petri trays.

Barcode ID labels

Barcode ID labels: in the most recent batch some of the barcodes were ran outside the label. We need the entire barcode because we scan the barcode ID into the software of our instruments (see picture 2 below). Please ensure the entire barcode is contained on the petri slide labels.

Picture 1: Top view- petri slide tray with CSN samples in preferred orientation.
Please note all the Petri slides are oriented so filters are on the right.

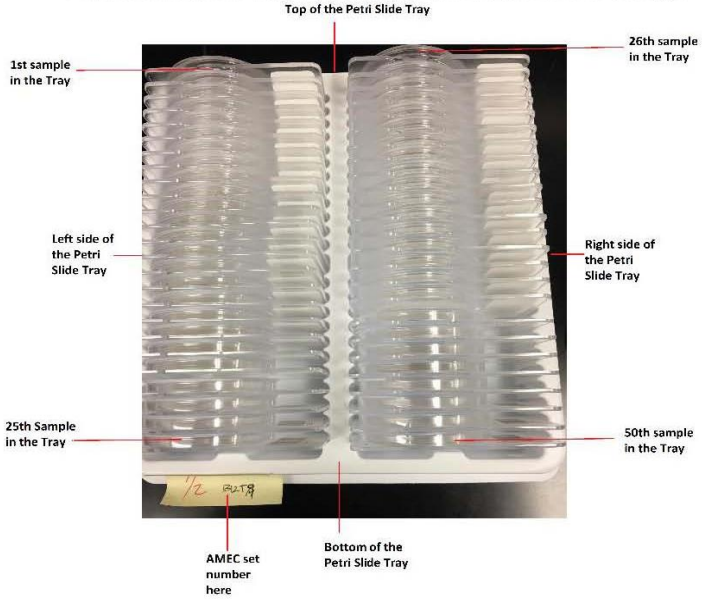

Figure 6. Petri Slide Orientation

1.9.4 Auto flagging Queries

When all data has been entered and filters sorted, a series of established queries are run to create flags for filters that have issues and to update the comments section of the data submittal to the analytical laboratory to clarify why the filters were assigned the flag, if the flag itself is not self-explanatory. In order to create the flags and add the comments, run the following "auto flagging" queries in the listed order. The process for adding either validity flags (e.g. the data may be suspect) or null flags (invalid data) is initiated by clicking the Add Flags and Comments button in CSN QA dashboard. Clicking on the button opens the Add Null and Validity Flags and Comments form shown in Figure 10.

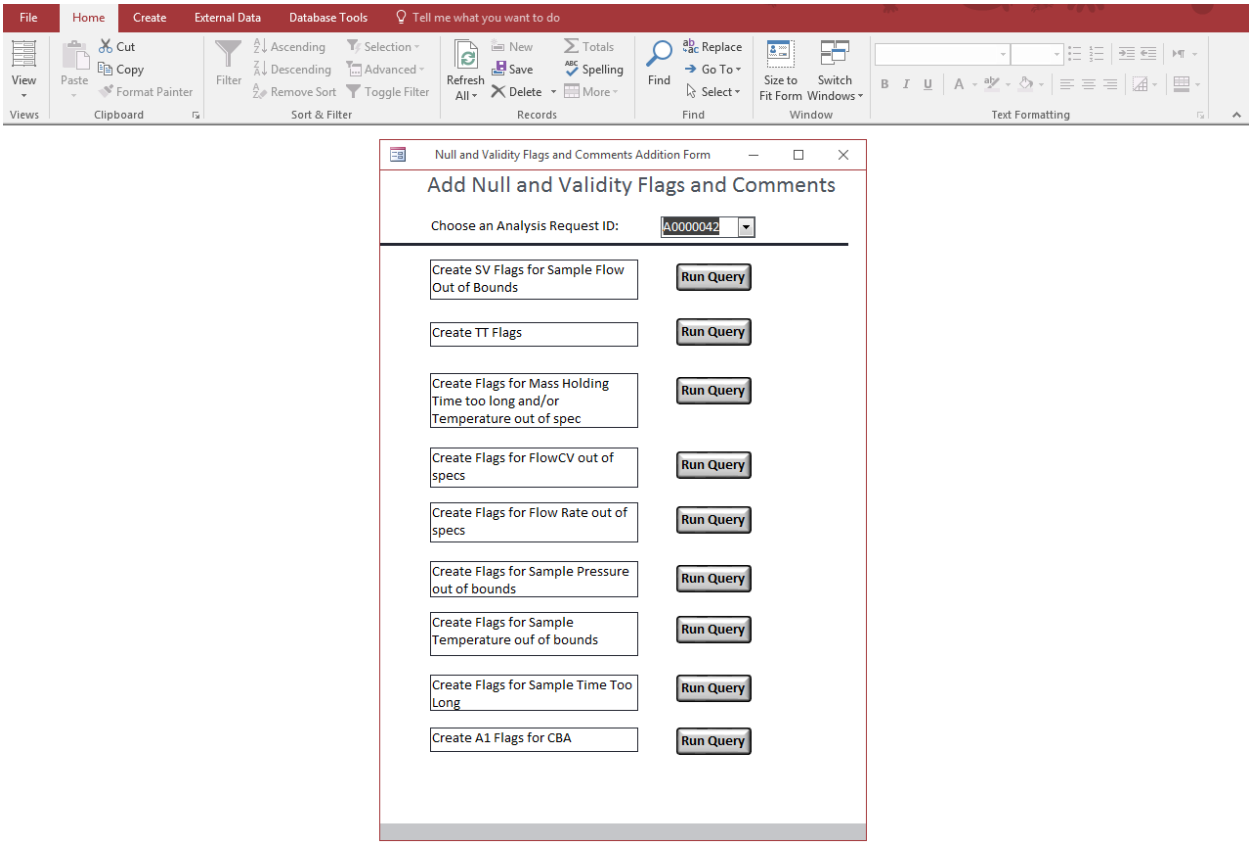

Figure 7. Add Null and Validity Flags and Comments Form

To begin adding the null or validity flags, the user first selects the analytical batch to add flags to from the combo box at the top of the form. Selecting an analytical batch ID number from the list in the combo box then makes all of the buttons for each null or validity flag QA process active. Addition of null or validity flags (and comments if the flag is not self-explanatory) is then performed in the following steps:

a) Press the button next to the corresponding null or validity flag operation that you want to perform (e.g., Create SV Flags for Sample Flow Out of Bounds). Pressing the button will run a query that will append records to the table in the CSN database that contains either the null or validity flag being added. A dialog box will display indicating the number of records that will be appended to the table and asking whether or not to make the changes. The user should confirm the changes to add records to the corresponding table. Once the user confirms that records should be added, they may receive a notification that not all records could be added. This message is displayed if the corresponding flag has already been added to the data table through review of the field flag form that the site operators return with exposed filters. The user should confirm that they want to proceed with the query and only the additional records that don't already exist will be added to the table. Finally, a second dialog box may display indicating additional records will be added. This dialog box is for adding comments to the

Sample Events table to explain why a flag was added if the description of the flag is not self-explanatory.

b) Step a) should be completed for each button until all of the flagging queries have been run.

c) Once all of the queries have been run and record additions have been confirmed, then the form can be closed by clicking on the X in the upper right hand corner of the form which will close the form and return the user to the QA dashboard.

d) All operations shall be documented on page 2 of the Analysis Batch Checklist (Figure 11), which is filed with PCOC for Analysis Batch for further reference.

Analysis Batch Checklist

Analysis Batch #_____

Date Range_____

QA Queries performed

Query	Run By	Date	COMMENTS
Create SV flags for Sample Flow Out of Bounds			
Create TT Flags for Validity			
Create Flags for Mass over 10 days			
Create Flags for Flow CV			
Create Flags for Flow Rate			
Create Flags for Sample Pressure			
Create Flags for Trip Blanks			
Create Flags for Sample Time Too Long			
Update Comments for Flow CV Flag			
Update Comments for Sample Pressure			
Update Comments for Sample Temp.			
Update Comments for Trip Blanks			
QAQry Check End Date Before Start Date			
QAQryIntended Date not equal to start			
QAQryUCDL0FAIDMissingnoNullnoComment			
QAQryUCDL0InvalidSmplNoComments			
QAQryUCDL0NullValidNoFilterRec			
QAQryUCDL0StartEnd>24hrsnoComment			
QAQryUCDL0StartEndDateonFBnoComments			
QAQryUCDL0StartEndSamenoComments			

Data Export

Export Query	Run By	Date	Comments
FilterDataNullFlags			
FilterDataTransfer			
FilterDataValidFlags			
Teflon COC			
Nylon COC			
Quartz COC			

Shipments

Lab	# of packages	Date	Data Export Emailed/Date
UC Davis			
DRI			

2

Figure 8. Analysis Batch Checklist Pg. 2

1.9.5 Mark All Samples with Null Flags as Invalid

The Print Lab COC Report form can also be used to mark filters invalid once all of the null flags have been added using the Add Null and Validity Flags and Comments form as outlined in the previous section. The filters that have null flags can be marked as being invalid prior to shipment to the contract analytical laboratory but using the following steps:

a) Select the Analytical Batch number from the Analysis Request ID combo box
b) Click on the Mark All Filter Types Invalid button. No selection in the Filter Types combo box is necessary to mark filters invalid.
c) Document on Analysis Batch Checklist.

1.9.6 Level Zero Data QA

Level Zero data QA of all operational data entered for a specific analytical batch is accomplished by clicking on the Level Zero Data QA button in the CSN QA dashboard (Figure 4). Clicking on the button opens the Level Zero Data QA Review form shown in Figure 12.

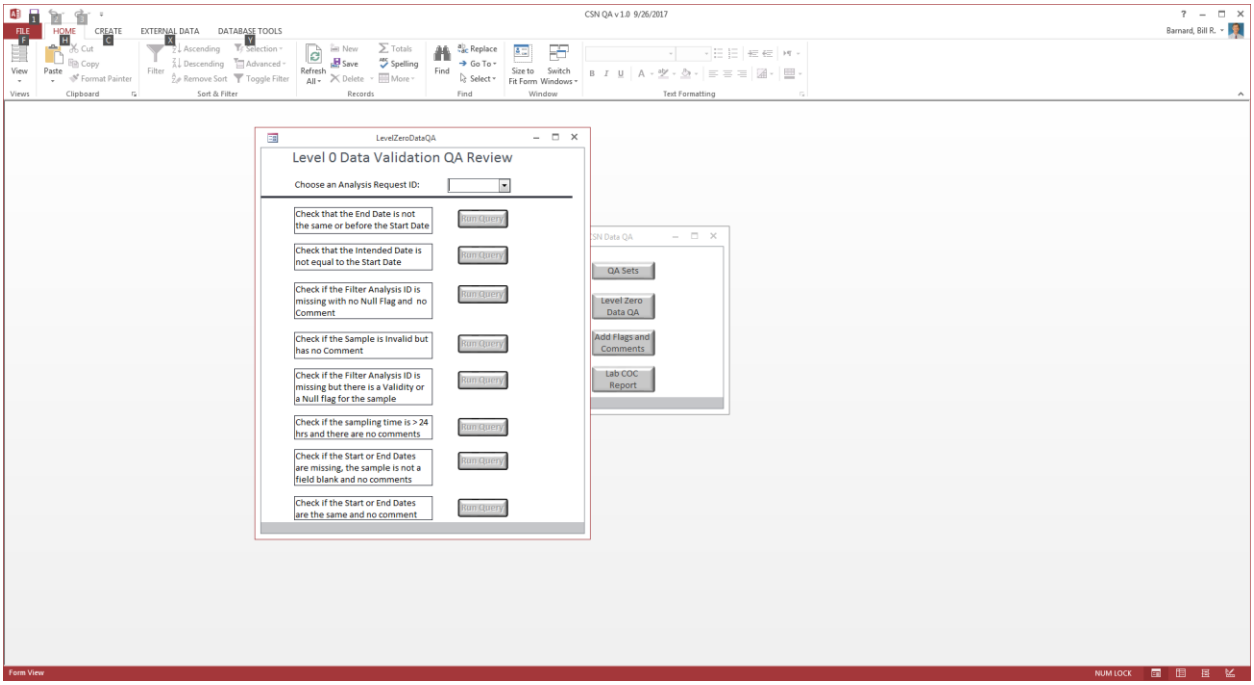

Figure 9. Level 0 Data QA

To begin Level Zero Data Validation Review, the user first selects the analytical batch to review from the combo box at the top of the form. Selecting an analytical batch ID number from the list in the combo box then makes all of the buttons for each Level Zero Data Validation review process active. Level Zero Data Validation is then performed in the following steps:

- Press the button next to the corresponding Level Zero Data Validation Review operation that you want to perform (e.g., Check that the End Date is not the same or before the Start Date). Pressing the button will run a query that will display the results for the analytical batch selected in the combo box.
- Each query that is run from a button selection will open on-screen with the results. If the query returns no records, then there are no records with data that meet that criteria.
- If there are records that are displayed, then the personnel that are reviewing the data should either correct the data that resulted in records being returned if the data are in error or ensure that all of the records have information in the comments field that indicate that the data have been checked and a reason given for why the records are being maintained and have not been corrected.
- Steps a) through c) should be completed for each button until all of the Level Zero Data Review queries have been evaluated.
- Once all of the queries have been run and evaluated, then the form can be closed by clicking on the X in the upper right-hand corner of the form which will close the form and return the user to the QA dashboard.
- Document steps completed and counts on page 2 of the Analysis Batch Checklist (Figure 11).
- Investigate any records that show up in the report for each query. They may or may not be legitimate concerns, and must be certified prior to shipment.

1.9.7 Finalizing the Analysis Batch for Shipment

- Match the auto flagging counts with counts from comment queries.
- Match physical number of filters sorted into boxes with the number of filters expected for each filter type in the Analysis Batch.
- For any discrepancies, repeat procedural steps as needed until discrepancies are resolved.

1.9.8 Electronic Data Submittal to Analytical Laboratories

Once data validation is complete, update the ShipLabOutDate in the Analysis Request table previously described in section 1.9.1.b. The final COC report is then generated following steps previously described in sections 1.9.1.d-j. Compare the updated COC forms with the PCOC. Investigate and reconcile any observed discrepancies. The final COC forms for each filter type are printed and bagged for inclusion in the shipment. An electronic copy of each filter type final COC (Figure 15) is emailed along with the data files.

To create and export data files,

a) Modify the query named FilterDataTransfer (see Figure 14) in the QA front-end of the database to reflect the Analysis Batch being sent to the contract analytical laboratories.

b) Save the query and then right click Filter Data Transfer, select export, text file, then select the file name and destination.

c) Make sure that each file generated is named with the Analysis Request ID in the name and save them into the Filter Transfer Files directory on the file server.

d) In the Export Text Wizard tab, select delimited, click next, the delimiter that separates fields should be “Comma”, click the box to “Include Field Names on First Row” then click finish to run the file export. (The format of the query is such that it produces the correct format for import by the contract analytical laboratory when received).

e) Make similar modifications to the two queries providing null and validity flags, (named FilterDataNullFlags and FilterDataValidFlags respectively) to reflect the criteria necessary to select the data for the filters provided to the contract analytical laboratories.

f) Save each query with the revised criteria. Export the FilterDataNullFlags, and FilterDataValidFlags queries as CSV files using steps c and d for each file.

g) Investigate files to ensure proper data was exported, fix queries and repeat if necessary.

h) Email the three files generated along with the COCs to the contract analytical laboratory distribution list.

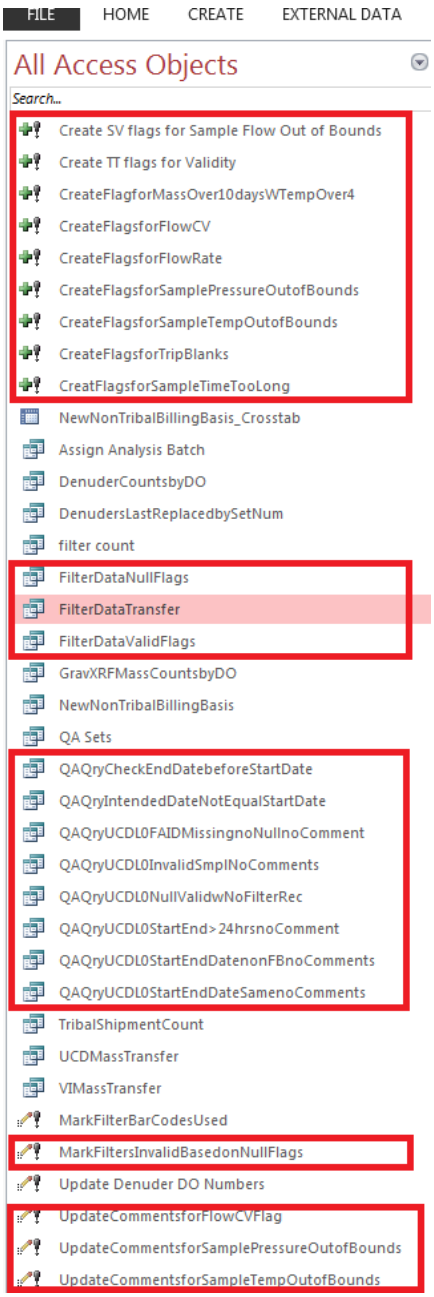

Figure 10. Screen capture highlighting queries locations from sections 1.9.4-1.9.7.

1.9.9 Shipment of Filters

a) Group sorted and labeled boxes with petri slides by filter type in chronological order.
b) Load boxes into respective coolers.
c) Pack sides and top with ice packs to ensure temperature control and prevent shifting during overnight shipping process, taking care to not intermingle filter types as they are shipped to different contract analytical labs.
d) Once all three filter types are packed in coolers and iced down, shipping information will be generated on the CSN FiSH Shipping Computer, located in the Shipping room.
e) To create shipping labels, open UPS Worldship application, click on ship button, find destination under company name or enter manually if new destination. Check for correct address.
f) Enter weight and description for package #1, click add packages and repeat for necessary number of packages in shipment.
g) Click “Process Shipment” this will print airbills to be applied to coolers.
h) Click “Return Shipment” and enter weight, description for same number of shipments, then click “Process Shipment” this will print return airbills to be discussed in next step.
i) Add bagged hard copy of finalized COCs, along with return shipping labels for respective labs into the first cooler of each filter type (containing Box 1-4 of each filter type).
j) After the cooler has been looked over to ensure all items are present, securely tape the cooler and attach the outgoing shipping air bill, repeat for all coolers.
k) Place the box in the designated area for outgoing shipments.

Analysis Request ID
A0000031

Intended Sample Date 6/24/2017
Set # 1Q

Barcode/Filter Analysis ID	Filter Type	Analysis Requested	Invalid?
Filter Analysis ID F066958	Teflon 220452939	XRF	☑
Filter Analysis ID F066964	Teflon 220453950	XRF	☐
Filter Analysis ID F066970	Teflon 220453952	XRF	☐
Filter Analysis ID F066976	Teflon 220453974	XRF	☐
Filter Analysis ID F066982	Teflon 220453954	XRF	☐
Filter Analysis ID F066988	Teflon 220453956	XRF	☐
Filter Analysis ID F066994	Teflon 220453958	XRF	☐
Filter Analysis ID F067000	Teflon 220453960	XRF	☐
Filter Analysis ID F067006	Teflon 220453962	XRF	☐
Filter Analysis ID F067012	Teflon 220453964	XRF	☐
Filter Analysis ID F067018	Teflon 220453966	XRF	☐
Filter Analysis ID F067024	Teflon 220453968	XRF	☐

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Figure 11. Finalized Chain of Custody

1.10 Data and Records Management

- PCOCs for each filter type, along with completed Analysis Batch Checklist will be retained by Program Manager.
- Filter Data File, Null Flag File, Validity Flag File and final COCs for each type will be stored on network drive.
- UPS shipment information is retained in UPS Shipping Computer for tracking purposes.

2 Quality Control and Quality Assurance

2.1 Quality Specialist Procedures for validating each Sample Set

a) The “QA Sets” query from the CSN Database QA Front End is run to bring up the data associated with a given batch including information regarding sample request ID, start date, start time, end date, end time, sample volume, average flow, average flow CV, average ambient temperature, average BP, filter type, filter ID, set number, intended use date, comments, and check-in temperature. Additionally, if flags were assigned to the data by the operator on the field sampling null value and validity coding form, those flags are checked in the database to ensure that they were entered or that the correct flags were assigned.
b) All of this information is checked against the FSCOC, the Field Sampling Null Value and Validity Coding Form, and the Level 0 Sample Event Check In Form for each individual record to determine if data entry errors exist.
c) Should a data entry error be found, the error is recorded in the comments section of the CSN Data Entry Log.
d) The batch of data is returned to the person who entered it to make any corrections necessary.
e) Corrections noted by the Quality Specialist on the data entry log are initialed once complete, providing documentation that the corrections were made.
f) Once the corrections are complete, the batch of data is returned to the Program Manager for a final assessment.

2.2 Procedures for Analysis Batch Checklist

During the preparation of a new Analysis Batch, the Program Manager reviews Analysis Batch Checklist (Figures 8 and 10) to ensure that the data entry and review has been performed for all sets included in the Analysis Batch, along with documenting the results of each query described in section 1.9.4, 1.9.5, and 1.9.6.

2.3 Analysis Batch Checklist Validation

The Quality Specialist will then review the Analysis Batch Checklist items and use the queries to verify query statements and flag counts, indicate their verification status on the checklist, and initiate corrective actions as needed to resolve discrepancies.

3 References

Wood Environment & Infrastructure Solutions, Inc. (Wood). 2017. *Chemical Speciation Network (CSN) Standard Operating Procedures (SOP) GLO3110-001, Revision 0, Training Chemical Speciation Network Filter Shipping and Handling Personnel*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-D-15-001. Gainesville, FL.

Wood Environment & Infrastructure Solutions, Inc. (Wood). 2017. *Chemical Speciation Network (CSN) Standard Operating Procedures (SOP) GLO3110-002, Revision 0, Field Shipping and Handling*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-D-15-001. Gainesville, FL.

Wood Environment & Infrastructure Solutions, Inc. (Wood). 2017*. Chemical Speciation Network (CSN) Standard Operating Procedures (SOP) GLM3180-009, Revision 0, Determination of Particulate Matter (PM) Gravimetric Mass for the Chemical Speciation Network.* Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-D-15-001. Gainesville, FL.