DRI STANDARD OPERATING PROCEDURE

Pre-firing and Acceptance Testing of Quartz Fiber Filters For Aerosol and Carbonaceous Material Sampling

DRI SOP #2-106r11
Revised May 20, 2024

Desert Research Institute
Division of Atmospheric Sciences
2215 Raggio Parkway
Reno, NV 89512

(775) 674-7056

Prepared By: _____ **Date:** 5/20/24

Reviewed By: _____ **Date:** 5/20/24

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1. GENERAL DISCUSSION

1.1 Purpose of Procedure

This procedure delineates the process for pre-firing and acceptance testing of quartz fiber filters. Quartz fiber filters absorb organic gases from ambient air and organic artifacts from the manufacturing process. By pre-firing the quartz filters before sampling, these absorbed gases and artifacts are reduced to constant, insignificant levels.

The filters are pre-fired in preparation for thermal/optical reflectance and/or transmittance (TOR/TOT) carbon analysis, which is a thermal desorption process that subjects the filters to temperatures between 25 °C and 920 °C. In preparation for this analysis, the filters are pre-fired at 900° C to remove all possible interferences with the TOR/TOT analysis. Filters that will be used for additional ionic analysis also undergo ionic acceptance testing to ensure that any impurities are minimal.

1.2 Measurement Principle

Not applicable

1.3 Measurement Interferences and Their Minimization

Not applicable

1.4 1.4 Ranges and Typical Values

Not applicable

1.5 Typical Lower Quantifiable Limits, Precision, and Accuracy

As defined by the SOP for TOR carbon analysis, pre-fired quartz filters are acceptance tested after pre-firing. The upper limit for organic carbon levels is 1.5 µg/cm^2, elemental carbon levels is 0.5 µg/cm^2, and total carbon levels is 2.0 µg/cm^2. The upper limit for ions is <1.0 µg/filter. Anions routinely tested for are chloride, nitrate, sulfate, ammonium and soluble sodium and potassium by Ion Chromatography (IC).

1.6 Personnel Responsibilities

All technicians in the laboratory should read and understand this entire standard operating procedure before performing pre-firing and acceptance testing preparation.

The laboratory coordinator is responsible for: 1) ensuring that the procedure is being followed, 2) maintaining the supplies necessary to insure uninterrupted pre-firing, and 3) ensuring that documentation is properly maintained.

The DRI quality assurance officer is responsible for revising the procedure when necessary.

1.7 Definitions

There are no terms in this procedure which require definitions.

1.8 Related Procedures

- DRI SOP #2-226 Model 2015 Multi-Wavelength Thermal/Optical Carbon Analysis (TOR/TOT) of Aerosol Filter Samples.
- DRI SOP #2-228 Anion Analysis of Filter Extracts and Precipitation Samples by Ion Chromatography using the DIONEX ICS-5000^{+} System
- DRI SOP #2-229 Cation Analysis of Filter Extracts and Precipitation Samples by Ion Chromatography using the DIONEX ICS-5000^{+} System

2. APPARATUS, MATERIALS, AND FORMS

2.1 Apparatus and Instrumentation

- Muffle Oven (Model 51894, General Signal Corp., Watertown, WI).
- Quartz fiber filters 2500 QAT-UP (Pall Sciences, Ann Arbor, MI),) in 25, 37, or 47 mm disks, as required by the projects in progress.
- Flat-tipped tweezers (Millipore, South San Francisco, CA).
- Calipers
- Gloves, nitrile (Van Waters & Rogers, #82026).
- Coors Evaporating Dishes, 12 cm, #60234 (Van Waters & Rogers, #60234).
- Household aluminum foil (local grocery store).
- Light table
- PetriSlides, 47mm, #PD1504700 (Van Waters and Rogers, Brisbane, CA).
- Extraction Vials, 15 ml, #188271 (Intermountain Scientific, Kaysville, UT).

2.2 Reagents

Not applicable

2.2 Forms

The only paperwork required for the pre-firing process is the DRI Quartz Fiber Filter Acceptance Log (Figure 2-1).

DRI QUARTZ-FIBER FILTER ACCEPTANCE LOGSHEET

Filter Size	Mfg by	MFG Lot #	DRI Lot #	Prefire Date	Temp C	Start time	Stop time	Initial Filter Diameter A	Initial Filter Diameter B	Prefired Filter Diameter A	Prefired Filter Diameter B	# Filters/box (write on box too) -1	-2	-3	-4	Prefire/LC Tech(s)	

Figure 2-1. DRI Quartz-Fiber Filter Acceptance Log.

3. CALIBRATION PROCEDURES

Not applicable

4. PROCEDURES

4.1 General Flow Diagram

The process of pre-firing is depicted in Figure 4-1.

4.2 Preparation

4.2.1 Each DRI lot consists of 100 filters of the same manufacturer lot and size; this generally corresponds to four boxes of 25 filters (37mm and 47mm) or one box of 100 filters (25mm). When filters are to be pre-fired, assign a DRI lot number to each lot; refer to previous entries in the Quartz-Fiber Filter Acceptance Log binder for the correct lot number.

4.2.2 Record the DRI lot number on each box using a marker or gummed label. If more than one box is required to make a lot of 100, use a suffix of -1, -2, etc. to distinguish between the boxes.

4.2.3 Record the manufacturer, manufacturer's lot number, and filter size in the Quartz-Fiber Filter Acceptance Log binder.

4.3 Pre-firing of Filters

4.3.1 Obtain two ceramic dishes, one for a base and one for a lid. Clean with a dry Kimwipe.

4.3.2 Obtain 100 filters of the required size from the stocking shelf in the Shipping and Receiving room, and assign the next consecutive lot number as determined from the Quartz-Fiber Filter Acceptance Log binder.

4.3.3 Wearing gloves, use flat-tipped tweezers to grab one filter at random. Measure diameter of filter with calipers and record in Initial Filter Diameter column A. Set filter rough side up on the side of the bowl. Repeat with a second random filter and record in Initial Filter Diameter column B. If both initial diameters are under 24.4mm, do not proceed and do not use box of filters. Set filter smooth side up on other side of the bowl.

4.3.4 Place additional filters in the dish in a half circle on either side of the bowl, resting on the side of the dish. The completed dish will have the appearance of two half rosettes. Place 50 in each dish if pre-firing 47mm or 37 mm filters or 100 filters per dish if pre-firing 25mm filters. CAUTION: too much overlap of filters will not allow carbonaceous vapors to escape.

4.3.5 Invert the second dish and place it as a cap on top of the first dish.

4.3.6 Repeat the previous steps until sufficient lots are prepared to fill the oven.

4.3.7 Place the dishes in the oven. The dishes are placed three in a stack. Close and latch the oven door.

4.3.8 Turn on the oven. Set the oven temperature to 900 °C by pressing the small black push button and turning the set point adjustment knob until "900" appears in the display. When the push button is released the display will return to the current oven temperature.

4.3.9 Record the pre-fire date, temperature, and pre-fire times in the Quartz-Fiber Filter Acceptance Log binder and the acceptance data base. Line the original boxes with aluminum foil, because the pre-fired filters are returned to them for storage. Also, prepare 2 slides with corresponding lot numbers with added A or B.

4.3.10 Turn the oven off after a minimum of 4 hours have elapsed. Allow the oven to cool without opening the door. Generally the oven is left overnight to cool.

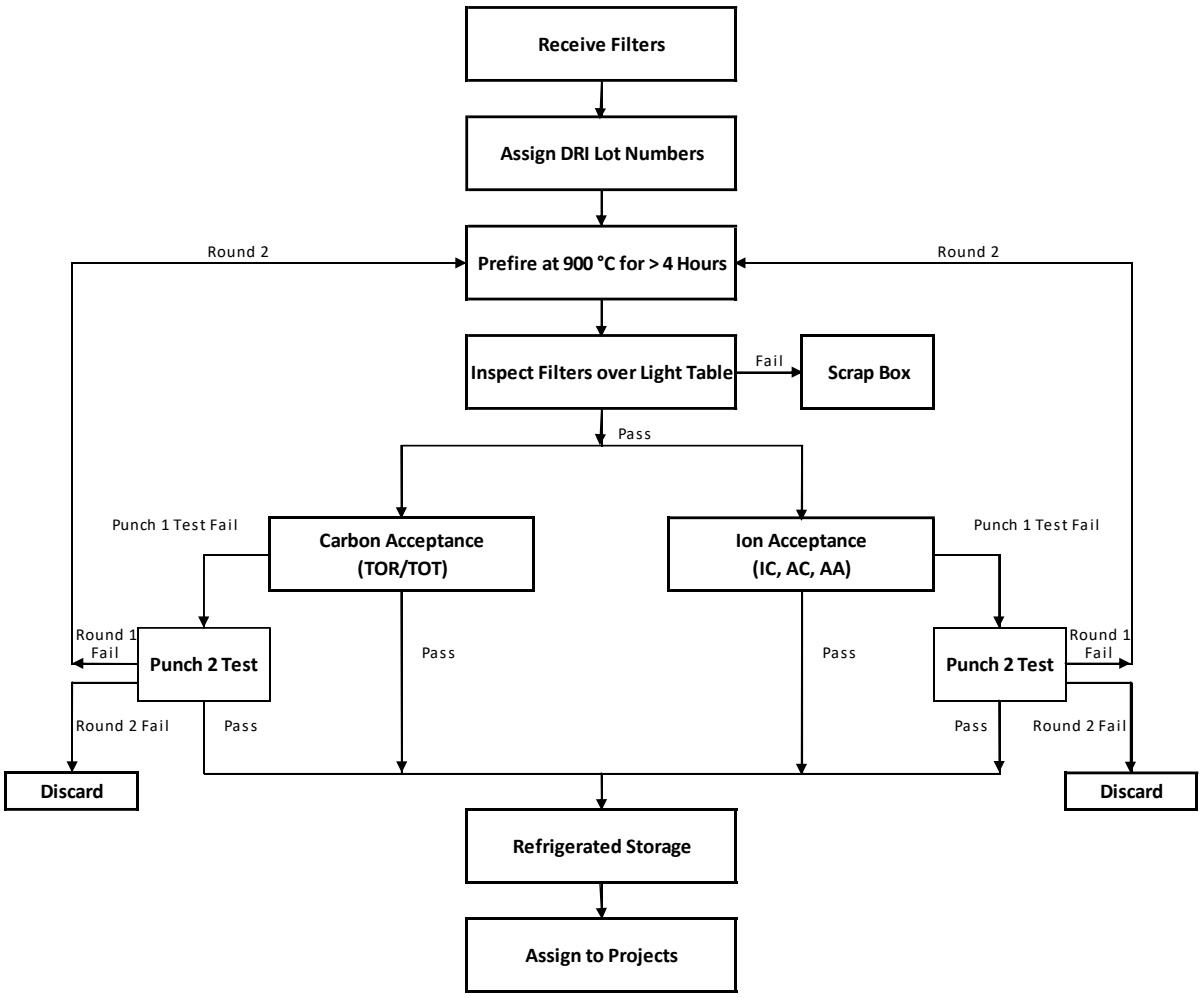

Figure 4-1. DRI Quartz Pre-firing Flow Diagram.

4.4 Acceptance Testing

4.4.1.1 When the oven has cooled, remove the dishes from the oven..

4.4.1.2 Using flat-tipped tweezers, grab filter A with rough side up and measure diameter again with calipers. Record in prefired filter diameter column A on acceptance logsheet. Repeat with filter B with smooth side up, and record in prefired filter diameter column B on acceptance logsheet. If filter diameters are under 24mm, the box of filters cannot be used.

4.4.1.3 Using flat tipped tweezers, hold each filter to the light table and inspect it for holes or uneven texture. Place any rejects in a separate unlined box for use in test packs. Place the accepted filters in the prepared aluminum lined boxes. NOTE: inspect the filters carefully; for most air sampling projects the equivalent value of each filter may reach several hundreds of dollars; make sure that only clean, unblemished filters are accepted.

4.4.1.4 When all filters have been light checked, place two of the filters in PetriSlides for carbon acceptance testing. Label the PetriSlides with the code "Q"+lot pre-made barodemnumber+"A" or “B” (e.g., "Q160A"). For 37mm and 47mm filters, also fold and place two filters in extraction tubes for wet chemistry acceptance testing. Store the boxes of filters, extraction tubes, and PetriSlides in the designated freezer. Inform the laboratory coordinator that there are pre-fired filters that will need an analysis list for acceptance testing.

4.4.1.5 Carbon acceptance testing is performed as described in SOP #2-226

4.4.1.6 Ion acceptance testing is performed as described in SOPs #2-203r9, and #2-208r4.

4.4.1.7 Two additional filters from lots that fail acceptance testing are subjected to further testing. The process outlined above is followed except the filters are identified with a “C” and “D” suffix. If the filters fail again, the lot is discarded and a note is made in the Acceptance Binder.

4.4.1.8 Boxes containing filters which pass acceptance testing are placed in zip-lock bags, and stored in the freezer until they are assigned to a project.

5. QUANTIFICATION

Not applicable

6. QUALITY CONTROL

Not applicable

7. QUALITY ASSURANCE

All work within the DRI EAF will adhere to the DRI EAF Quality Manual Revision 3, 03/2014.

8. REFERENCES

Refer to the oven's owner's manual for additional information concerning its operation.

9. DOCUMENT CHANGES

07/30/07: r6 – minor formatting changes. Added title page with signature block.
07/12/17: r7 – Updated all references to DRI carbon analyzer model, changed from Model 2001 to Model 2015. Updated SOP versions in related procedures.
09/27/17: r8 – Updated Figures 2-1 and 4-1, removed revision numbers from SOP reference, further updated SOP references, and made minor editorial changes.

08/20/2018: r9- SDK - Made figures 508 compliant
09/26/2023: r10 SDK Revised Prefire form , Fig 2-1
05/20/2024: r11 JG – Added new measuring procedures to protocol