# Standard Operating Procedure for Cleaning Nylon Filters Used for the Collection of $PM_{2.5}$ Material

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## 1.0 Procedural Section

### 1.1 Purpose and Applicability

Nylon filters are used for the collection of $PM_{2.5}$ material in the chemical speciation particulate samplers. These filters are analyzed for the following ions: nitrate, sulfate, ammonium, sodium, and potassium. The filters, as purchased and received from different manufacturers, show unacceptable levels of these ions, often exceeding the maximum level of 1 $\mu$g per filter for a particular ion. This has prompted the development of a procedure for cleaning the nylon filters prior to their use for field sampling; this procedure is described in this standard operating procedure (SOP).

### 1.2 Summary of Method

Fifty nylon filters are placed in a 2 L polypropylene jar with approximately 1000 mL of polished deionized water (18.2 MΩ-cm; water that has been passed through a secondary deionization system). The filters are shaken in the water for approximately 2 minutes, and the water is decanted and discarded. This process is repeated. The jar is then filled with polished deionized water and placed on a Toxicity Characteristic Leaching Procedure (TCLP) apparatus (TLCP, EPA SW-846 Method 1311). The jar is rotated for 7 to 8 hours, and the water is replaced with fresh polished deionized water. The jar is then rotated overnight for 14 to 16 hours before the water is replaced again. After another 24 hours of washing, the water is drained from the filters and the filters are dried. (The order of extended washing may vary; that is the sequence may be 24 hours, 7 to 8 hours, and then 12 to 14 hours rather than 7 to 8 hours, 12 to 14 hours, and 24 hours.) The filters are dried on glass racks in a convection oven set at 45°C. One filter out of 50 is desorbed and analyzed by IC to test for residual contamination, prior to being approved for later use.

### 1.3 Health and Safety Warnings

The $PM_{2.5}$ filter-preparation operations do not involve unusual risks from electrical equipment or chemical exposures. Standard RTI laboratory health and safety precautions will be followed.

### 1.4 Cautions

Laboratory personnel should always wear clean clothes and wash hands thoroughly before performing filter handling and analysis procedures. The use of gloves rinsed with deionized water is required for all steps of the filter cleaning process because this will minimize the potential for laboratory contamination.

## 2.0 Apparatus and Reagents

The nylon filters used are Whatman 47 mm nylon membrane filters, 1.0 μm pore size (Whatman catalog number 7410-004).

The only reagent needed is polished deionized water (18.2 MΩ-cm; water that has been passed through a secondary deionization system).

Several pieces of equipment are used for cleaning the nylon filters. Included are:

1. 2-L polypropylene wide-mouth Mason jars (VWR Catalog Number 16128-660 or equivalent)
2. TCLP apparatus (TLCP, EPA SW-846 Method 1311) that holds six 2-L jars.
3. Programmable timer (VWR Lab Controller or equivalent)
4. Convection drying oven (VWR Model 1320 or equivalent)
5. 11" x 11" glass drying rack (custom made from 1/4" glass rods in parallel rows attached to 3/8" glass rods serving as a frame; center-to-center distance for the 1/4" parallel glass rods is ½")
6. Plastic colander approximately 8" in diameter from a kitchen appliance store.

## 3.0 Filter Cleaning

### 3.1 Cleaning Procedure

The nylon filters are cleaned using the following procedure, which should be started at the beginning of a work day. The date when the cleaning is started is entered into the log book and the batch is identified by this date.

1. Fifty 47-mm nylon filters are carefully removed from the manufacturer's filter container using either gloves or forceps. Each filter is separated with a blue tissue which is removed prior to placing the 50 filters into a 2-L polypropylene jar that contains approximately 1000 mL of polished deionized water. The lid is attached, and the jar is shaken gently for approximately 2 minutes. The water is then carefully poured out of the jar without losing any filters. This rinse step is repeated. The two-step rinse procedure is then duplicated with five additional 2-L jars each loaded with 50 filters. Each jar is labeled with a letter (i.e., A, B, C), using a marker.

2. Each jar is carefully filled with polished deionized water until it is overflowing; it is then capped tightly and placed on the TCLP apparatus. The apparatus is turned on and mixes the filters and polished water by rotating the jars end-over-end. It cycles until the end of the day (i.e., 7 to 8 hours). The water is carefully poured out of each jar, and the jars are again filled to overflowing. The jars are placed on the TCLP apparatus, turned on, and allowed to run overnight, or for 14 to 16

hours. At the beginning of the next work day, the water is poured out again and replaced, and the jar is placed back on the apparatus for approximately 24 hours, or until the beginning of the next work day. Depending on a person's work schedule, the order of the extended washing may be varied; that is, the sequence may be 24 hours, 7 to 8 hours, and then 12 to 14 hours rather than 7 to 8 hours, 12 to 14 hours, and 24 hours.

**Note:** Because the filters tend to stick to the sides of the jars during rotation, the TCLP apparatus is connected to a timer that is programmed to rotate the jars for 15 minutes, and then allow them to sit at rest for 2 minutes. During this rest period, the filters that were stuck to the sides of the jar slip away and fall to the lower part of the jar of water. This timed cycle of rotation, followed by resting, continues through each cleaning period. The procedure for programming the time is given in Attachment A.

3. The jars are removed from the TCLP apparatus after the final wash and are taken to a Class 100 clean room for drying of the filters. The lid of a jar is removed and the water and filters are gently poured into a pre-rinsed plastic colander placed in a sink in the clean room. It may be necessary to add polished deionized water to the jar several times to remove all of the filters. The excess water is allowed to drain from the filters and the colander for several minutes. Any filters that fall into the sink during this process will be discarded.

4. With gloves and a clean forceps, the filters are removed from the colander one by one and placed separately on the drying rack, which has been thoroughly pre-rinsed with polished deionized water shortly before use. The loaded rack is carefully placed in the oven, which is set at 45°C. The filters are allowed to dry completely. The filters sometimes curl slightly during the drying process. A large amount of curling indicates that the oven temperature is too high. If so, slightly reduce the temperature so the filters dry without curling.

   **Note:** The drying oven must be kept free of any dust or particulate material and should only be operated in a clean environment. The oven should be visually inspected for any contamination prior to each use. A dedicated oven used only for drying $PM_{2.5}$ nylon filters is used.

5. The dried filters are removed from the drying rack using clean forceps and are placed back into the original manufacturer's plastic containers. These containers are washed with deionized water and are dried before reuse. Filters will be inspected for pinholes and tears; any damaged filter will be discarded. Twenty-five filters are placed in each container. Each container is labeled with the batch number (i.e., start date for cleaning) and the jar identifier (i.e., A, B, C).

### 3.2 Filter Acceptance Testing

One filter from each set of dried filters is selected at random for analysis. Blank filters are analyzed according to the analytical procedure described elsewhere in the SOPs for Anion (Hardison, 2008) and Cation (Hardison, 2008) analysis contained in the laboratory Quality Assurance Project Plan. For lot acceptance, the filter loadings of the ions of interest (i.e., sodium, potassium, ammonium, nitrate, and sulfate) must each be less than 1.0 $\mu$g per filter. If any ion exceeds the limit, the entire lot must be rejected. Rejected lots may be re-cleaned using the same procedure.

Each accepted batch of filters is assigned a unique number. Each filter's batch number is recorded in the $PM_{2.5}$ database when it is loaded into a sample module in the Sample Handling and Archiving Laboratory. The lot number can be used to trace the acceptance test results in case there is a question about any filter.

> **Note:** Several different cleaning procedures were used during the course of the $PM_{2.5}$ Speciation Trends Network contract, which began in early 2000. This note summarizes the procedures used for cleaning nylon filters prior to finalization of the method described in this SOP.
>
> Prior to March 28, 2000, filters were soaked three times for 30 minutes in deionized water without shaking or ultrasonication. Drying and acceptance procedures were identical to those previously described.
>
> Prior to December 1, 2001, filters were cleaned using a shaker for the final 24-hour wash in deionized water. In fall 2001, some batches of filters received from the supplier were noted to be partially disintegrating in the shaker. It was concluded that the filter's durability was somewhat variable, and that shaking for 24 hours was too forceful for the less durable filters; therefore, the more gently rolling method was adopted.
>
> Prior to December 1, 2002, filters were placed in a polypropylene jar of sodium carbonate/sodium bicarbonate solution (the eluent used for anion analysis). The jar containing the filters was placed in an ultrasonic bath for 1 hour. The filters were then rinsed three times with deionized water, rinsed gently using a jar roller mill in deionized water for about 1 hour, rinsed again manually three or four times, and then rinsed gently in fresh deionized water for 24 hours using the jar roller mill. This procedure was abandoned for the following reasons: the ultrasonic bath sometimes caused partial disintegration of the filters, sodium from the eluent solution was sometimes still present on the filters, and the TCLP apparatus was better than a roller because it provides end-over-end mixing. The method described in this SOP was subsequently adopted.

## 4.0 Quality Control

The quality control activities include the following:

1. Perform ion analyses of the polished deionized water whenever the deionizer beds are changed to determine that the ions of interest are below their maximum allowable concentration, as presented in Table 1. Replace the ion exchange beds in the water deionization system if these limits are exceeded.

**Table 1. Maximum Allowable Concentration (MAC) for Ions of Interest**

| Ion of Interest | MAC, $\mu$g/mL |
|---|---|
| Nitrate | 0.01 |
| Sulfate | 0.01 |
| Ammonium | 0.01 |
| Sodium | 0.01 |
| Potassium | 0.02 |

2. Keep all jars closed and stored in a clean environment when not in use.
3. Periodically wipe down the inside of the drying oven with wet, lint-free tissues.

## 5.0 References

Hardison, E. 2008. *Standard Operating Procedure for $PM_{2.5}$ Anion Analysis*. Quality Assurance Project Plan Chemical Speciation of Particulate Matter, Volume II, Appendix A-5.1, revision 6.

Hardison, E. 2008. *Standard Operating Procedure for $PM_{2.5}$ Cation Analysis*. Quality Assurance Project Plan Chemical Speciation of Particulate Matter, Volume II, Appendix A-5.2, revision 6.

# Attachment A
# Method for Programming the VWR Lab Controller

The device is programmed for the repeat mode, which permits repeatedly turning equipment on or off at one or two unique time intervals.

1. Press the CHANNEL SELECT key until the OUTLET channel is selected.

2. Delete all time-of-day program times by pressing the C key, and then the REPEAT key.

3. Press the OUTLET ON/OFF key to ON.

4. Press the 1, 5, 0, and 0 keys to program 15 minutes (15.00) power on.

5. Press the REPEAT key.

6. Press the 2, 0, and 0 keys to program 2 minutes power off.

7. Turn on the toggle switch on the TCLP apparatus (if it is not already on) and press the START/STOP key to begin counting down.

8. At zero, the outlet switches to OFF, the alarm sounds for 2 seconds, the display automatically returns to the programmed 2 minutes, and the timer begins counting down. At the next zero, it switches, alarms, displays 15.00, and begins counting down. This process will repeat until the C key is pressed, or the toggle switch is turned off.