STANDARD OPERATING PROCEDURE FOR CLEANING NYLON FILTERS USED FOR COLLECTION OF PM2.5 MATERIAL

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1.0 Procedural Section

1.1 Purpose and Applicability

Nylon filters are used for the collection of PM2.5 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 1µg per filter for a particular ion. This has prompted development of a procedure, described in this Standard Operating Procedure (SOP), for cleaning the nylon filters prior to their use for field sampling.

1.2 Summary of Method

Fifty filters are placed in a 2 liter polypropylene jar with about 100 mL of "polished" deionized water (18 Megaohm; water that has been passed through a secondary deionization system). The filters are shaken in the water for about 1 minute and the water is decanted off and discarded. This process is repeated once more. The jar is then filled with polished deionized water and placed on a TCLP apparatus (Toxicity Characteristic Leaching Procedure, 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 filters are collected in a plastic colander. 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, 24 hours. Finally, the filters are dried in a convection oven set at 45°C, checked for residual contamination, and packaged for later use.

1.3 Health and Safety Warnings

The PM2.5 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 and will minimize the potential for laboratory contamination.

2.0 Apparatus and Reagents

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

1. Two-liter polypropylene wide-mouth Mason jars (VWR Catalog no. 16128-660 or equivalent)

2. TCLP apparatus (Toxicity Characteristic Leaching Procedure, EPA SW-846 Method 1311) that hold six (6) 2-liter jars.

3. Programmable timer (VWR Lab Controller or equivalent)

4. Convection drying oven (VWR Model 1320 or equivalent)

5. 11-in. by 11-in. glass drying rack (custom made from 1/4-in. glass rods in parallel rows attached to 3/8-in. glass rods serving as a frame; center to center distance for the 1/4-in. parallel glass rods is ½ in.)

6. Plastic colander about 8 inches in diameter from kitchen appliance store.

Reagents needed are as follows:

1. Polished deionized water (18 Megaohm; water that has been passed through a secondary deionization system)

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 that 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 and are placed in a 2-liter polypropylene jar that contains about 100 mL of polished deionized water. The lid is attached and the jar is shaken gently for about 1 minute. The water is then carefully poured out of the jar without losing any filters. This rinse procedure is repeated once more. This duplicate rinse procedure is then performed with five other 2-liter jars each loaded with 50 filters. Each jar is labeled with a letter, i.e., A, B, C, etc., 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 then run to the end of the day, that is, 7 to 8 hours. The water is carefully poured out of each jar and the jars are again carefully filled to overflowing. The jars are placed on the apparatus, which is then run overnight, or for 14 to 16 hours. The beginning of the next work day, the water is poured out once again, the jars are filled to overflowing, capped, and placed on the apparatus for about 24 hours, or to the beginning of the next work day. Depending on one'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, 24 hours.

NOTE:	The filters tend to stick to the sides of the jars. Therefore, the TCLP apparatus is connected to the power source through a timer. This timer is programed to rotate the jars for 15 minutes and then allow them to be still for 2 minutes. During this rest period, the filters stuck to the sides of the jar slip away and fall to the lower part of the jar of water. 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 taken to a Class 100 clean room for drying of the filters. The lid of a jar is removed and the water along with filters are gently poured into a pre-cleaned plastic colander placed in a sink in the clean room. It may be necessary to add polished deionized water to the jar several times in order to remove all the filters. The excess water is allowed to drain from the filters and colander for several minutes. Any filters that fall into the sink during this process are to be discarded.

4. With gloves and a clean forceps, the filters are removed from the colander one by one and laid 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 for one-half hour. The filters may curl slightly during the drying process. A large amount of curling indicates that the oven temperature is too high.

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 new oven used only for drying filters is recommended.

5. The dried filters are removed from the drying rack using a clean forceps and placed in the same manufacturer's plastic containers that they were taken from for the purpose of cleaning. These containers are washed with deionized water and dried before use. Filters shall be inspected for pin holes and/or tears; any damaged filter shall be discarded. Twenty-five (25) 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, etc.).

3.2 Filter Acceptance Testing

One filter from each jar of cleaned filters is selected at random for analysis. Blank filters are analyzed according to the analytical procedure described elsewhere in the SOPs for Anion[1] and Cation[2] analysis contained in the laboratory Quality Assurance Project Plan (QAPP). For lot acceptance, the ion concentrations of interest (sodium, potassium, ammonium, nitrate, and sulfate) must each be less than 1.0 µg/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 database when it is loaded into a sample module in the SHAL. 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 STN 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 3/28/2000, filters were soaked three times for 30 minutes in deionized water without shaking or ultrasonication. Drying and acceptance procedures were identical to those described above.

Prior to 12/1/2001, 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 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 2/1/02, 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, again rinsed 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 in order to determine that the ions of interest are below their maximum allowable concentration, as presented in Table 1 below.

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

Ion of Interest	MAC, µg/mL
Nitrate	0.01
Sulfate	0.01
Ammonium	0.01
Sodium	0.01
Potassium	0.02

Replace the ion exchange beds in the water deionization system if these limits are exceeded.

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

1. Hardison, Eva. Standard Operating Procedure for PM2.5 Anion Analysis. Quality Assurance Project Plan Chemical Speciation of Particulate Matter, Volume II, Appendix A-5.1, revision 2, September 8, 1999.

2. Hardison, Eva. Standard Operating Procedure for PM2.5 Cation Analysis. Quality Assurance Project Plan Chemical Speciation of Particulate Matter, Volume II, Appendix A-5.2, revision 2, September 8, 1999.

Attachment A

Method for Programming the VWR Lab Controller

The device in 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 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 two seconds, the display automatically returns to the programed 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.