Title: Extraction of Filters		Copy No: ##
SOP No.: 6.06/1.8/S	Effective Date: April 9, 2013	Location: ###

QSM Approval: _____

Extraction of Filters

1. Introduction and scope

1.1 This SOP describes procedures used to extract inorganic ions, organic acids, carbohydrates and water soluble metals from particulate matter collected on filters in support of National Air Pollution Surveillance (NAPS) Program and other projects.

2. Sample Requirement

2.1 For the storage conditions and holding time of samples refer to the extraction procedures in Section 4.

3. Apparatus and Materials

3.1 Millipore Super-Q water purifying system (SOP 19.04/*.*/S) that provides high-purity deionized water (DW) (resistance > 18 MOhm cm)

3.2 Barnstead NANOpure Diamond water purifying system that is used to produce ultra-high purity, double deionized water (DDW).

3.3 Mettler PR 1200 technical balance.

3.4 Ultrasonic bath or mechanical shaker.

3.5 Polypropylene test tubes (e.g. 10 or 12 mL, and 50 mL), scintillation vials (e.g. 20 mL), and wide-mouth bottles (e.g. 50 mL).

3.6 Bottles with bottle-top dispenser, and Micropipettes.

NOTE: Other equivalent, commercially available apparatus can also be used.

4. Procedures

4.1 **Teflon filters for multiple analyses of: (i) inorganic anions, cations, and organic acids by IC (6.03/*.*/M), (ii) analysis of carbohydrates by IC-PAD (6.12/*.*/M) and (iii) water-soluble metals by ICP-MS (6.10/*.*/M)**

4.1.1 Exposed filters are stored at room temperature in the laboratory cabinet (filters in the original perti-dishes) or in the refrigerator (filters in

scintillation vials) and should be analyzed within 6 months of reception by the Ion Analysis Laboratory (SOP 6.1/*.*/S).

4.1.2 Extraction solution: degassed DDW (for preparation instructions see 6.03/*.*/M).

4.1.3 Check the bottle-top dispenser by weighing 15 mL of DDW on a top loading balance. Record results in the control chart.

4.1.4 Place filters in labeled 20 mL-HDPE scintillation vials for extraction. Wet filters with 100 µL of isopropanol and add 15 mL of DDW.

4.1.5 Prepare the quality control samples:

4.1.5.1 Prepare **Vial Blank** (VialBlank) – add 15 mL of extraction solution and 100 µL of isopropanol to a labeled 20 mL-HDPE scintillation vial, and carry through processing identical to that carried out for the samples.

4.1.5.2 Prepare **Method Blank** (MetBlank) – place a blank Teflon filter in a labeled 20 mL HDPE scintillation vial, wet with 100 µL of isopropanol, add 15 mL of the extraction solution and carry through processing identical to that carried out for the samples.

4.1.5.3 Prepare **Spikes** (SP-MDL and SP-VS) – place blank Teflon filters in a labeled 20 mL - HDPE scintillation vials (20mL), wet with 100 µL of isopropanol, add 15 mL of the lowest calibration standard solution or verification standard, and carry through processing identical to that carried out for the samples. For instructions on preparation of lowest calibration standard and verification standard consult 6.03/*.*/M and 6.12/*.*/M

4.1.6 Ultrasonicate all samples for 30 min at room temperature (place 2 frozen cold-packs in the sonication bath to maintain room temperature during extraction); mechanical shaking may also be used.

4.1.7 If the samples will be analyzed for carbohydrates by IC-PAD (6.12/*.*/M), remove and discard the filters immediately following the extraction.

4.1.8 If the samples will be analyzed for water-soluble metals by ICP-MS (6.10/*.*/M), make sure that the ICP-MS analyst is consulted and has received the sample list prior to extraction of samples.

4.1.9 Analyze the samples as soon as possible (within 24 hours).

4.1.10 Store the residue of extracts in a freezer up to 6 months (SOP 2.6/*.*/S).

4.2 **Teflon Filters for multiple analyses of inorganic anions, cations, and organic acids (Method: 6.03/*.*/M)** Note: Teflon filters from projects other than NAPS Program.

4.2.1 Exposed filters are stored in the refrigerator and should be analyzed within 6 months of reception by the Ion Analysis Laboratory.

4.2.2 Extraction solution: degassed, deionized water

4.2.3 Check a bottle-top dispenser by weighing extraction volume of extraction solution on a top loading balance (see 4.2.4). Record results in the control chart.

4.2.4 Place filters in clean, labeled wide-mouth polypropylene bottles (50 mL size) for extraction (putting particle deposition side down). Wet filters with 120 µL isopropanol and add the extraction solution as follows:

- 8 mL for extraction of 1 filter
- 10 mL for extraction of 2 filters
- 12 mL for extraction of 3 filters

4.2.5 Prepare the quality control samples:

4.2.5.1 Prepare **Vial Blank** (VialBlank) – add the extraction solution and 120 µL of isopropanol to a labeled wide-mouth polypropylene bottle (50 mL size), and carry through processing identical to that carried out for the samples.

4.2.5.2 Prepare **Method Blank** (MetBlank) – Place a blank Teflon filter in a labeled wide-mouth polypropylene bottle (50 mL size), wet with 120 µL of isopropanol, add extraction solution, and carry through processing identical to that carried out for the samples.

4.2.5.3 Prepare **Spikes** (SP-MDL and SP-VS) – Place blank Teflon filters in a labeled wide-mouth polypropylene bottles (50 mL size), wet with 120 µL of isopropanol, add extraction volume of lowest calibration standard solution or verification standard, and carry through processing identical to that carried out for the samples. For instructions on preparation of lowest calibration standard and verification standard consult 6.03/*.*/M.

4.2.6 Ultrasonicate all samples for 30 min at room temperature (mechanical shaking may also be used); place 2 frozen cold-packs in the sonication bath to keep it at room temperature during extraction.

4.2.7 After extraction add chloroform to stabilize organic acids:

- 16 µL for 8 mL extract
- 20 µL for 10 mL extract
- 24 µL for 12 mL extract

4.2.8 Analyze the samples within 24 hours from extraction of filters.

4.2.9 Store remaining extracts in a freezer up to 6 months.

4.3 **Teflon Filters for analyses of inorganic anions** (Method: 6.05/*.*/M)

4.3.1 Exposed filters are stored in the refrigerator and should be analyzed within 6 months of reception by the Ion Analysis Laboratory.

4.3.2 Extract filters by following procedures: from 4.2.2 to 4.2.6

4.3.3 Analyze samples within 24 hours from extraction of filters.

4.3.4 Store remaining extracts in the refrigerator up to 6 months.

4.4 Nylon Filters: for analyses of inorganic anions (Method: 6.05/*.*/M)

4.4.1 Exposed filters are stored in the refrigerator and should be analyzed within 6 months of reception by the Ion Analysis Laboratory.

4.4.2 Extraction solution: carbonate eluent (1.0 mM $NaHCO_3$ – 3.5 mM Na_2CO_3). For instructions on preparation of carbonate eluent see 6.05/*.*/M.

4.4.3 Check bottle-top dispenser by weighing 8 mL of deionized water on a top loading balance. Record results in the control chart

4.4.4 Place filters in clean, labeled 12-mL polypropylene test tubes for extraction

4.4.5 Prepare the quality control samples:

4.4.5.1 Prepare **Vial Blank** (VialBlank) – add 8 mL of carbonate eluent to a labeled 12-mL polypropylene test tube and carry through processing identical to that carried out for the samples.

4.4.5.2 Prepare **Method Blank** (MetBlank) – Place a blank Nylon filter in a labeled 12-mL polypropylene test tube, add 8 mL of carbonate eluent, and carry through processing identical to that carried out for the samples.

4.4.5.3 Prepare **Spikes** (SP-MDL and SP-VS) – Place blank Nylon filters in labeled 12-mL polypropylene test tubes, add 8 mL of lowest calibration standard solution or verification standard, and carry through processing identical to that carried out for the samples. For instructions on preparation of lowest calibration standard and verification standard consult 6.05/*.*/M.

4.4.6 Ultrasonicate the samples for 30 minutes.

4.4.7 Analyze samples within 24 hours from extraction of filters.

4.4.8 Store remaining extracts in a refrigerator up to 6 months.

4.5 **Citric Acid-Coated Filters for analyses of ammonium** (Method: 6.05/*.*/M)

4.5.1 Exposed filters are stored in the refrigerator and should be analyzed within 6 months of reception by the Ion Analysis Laboratory.

4.5.2 Extraction solution: degassed DDW.

4.5.3 Check a bottle-top dispenser by weighing extraction volume of DDW on a top loading balance (see 4.5.4). Record results in the control chart

4.5.4 Place filters in clean, labeled polypropylene test tubes for extraction. Use 12-mL tubes for extraction of 47-mm filters and 50-mL tubes for the 70-mm filters

4.5.5 Add 10 mL of the extraction solution to the 47-mm filters and 20 mL to the 70-mm filters

4.5.6 Prepare the quality control samples:

4.5.6.1 Prepare **Vial Blank** (VialBlank) – add extraction volume of DDW to a labeled polypropylene test tube and carry through processing identical to that carried out for the samples.

4.5.6.2 Prepare **Method Blank** (MetBlank) – Place a blank filter in a labeled polypropylene test tube, add extraction volume of DDW, and carry through processing identical to that carried out for the samples.

4.5.6.3 Prepare **Spikes** (SP-MDL and SP-VS) – Place blank filters in labeled polypropylene test tubes, add extraction volume of lowest calibration standard solution or verification standard, and carry through processing identical to that carried out for the samples. For instructions on preparation of lowest calibration standard and verification standard consult 6.05/*.*/M.

4.5.7 Ultrasonicate the samples for 30 min.

4.5.8 Analyze samples within 24 hours from extraction of filters.

4.5.9 Store remaining extracts in a refrigerator up to 6 months.

4.6 Carbonate-Coated Filters for analyses of inorganic anions (Method: 6.05/*.*/M)

4.6.1 Exposed filters are stored in the refrigerator and should be analyzed within 6 months of reception by the Ion Analysis Laboratory.

4.6.2 Extraction solution: 0.09% (v/v) H_2O_2 (hydrogen peroxide): dilute 3 mL of 30% H_2O_2 to 1L with degassed, deionized water.

4.6.3 Check a bottle-top dispenser by weighing extraction volume of DDW (see 4.6.4) on a top loading balance. Record results in the control chart

4.6.4 Place filters in clean, labeled polypropylene test tubes. Use 12- mL tubes for extraction of 15 mm filters or 50- mL tubes for the 47 mm filter.

4.6.5 Add the extraction solution as follows:

- 8 mL for extraction of 15 mm filter
- 20 mL for extraction of 1 47 mm filter
- 40 mL for extraction of 2 filters 47 mm filters

4.6.6 Prepare the quality control samples:

4.6.6.1 Prepare **Vial Blank** (VialBlank) – add extraction volume of 0.09% (v/v) H_2O_2 to a labeled polypropylene test tube, and carry through processing identical to that carried out for the samples.

4.6.6.2 Prepare **Method Blank** (MetBlank) – Place a blank filter in a labeled polypropylene test tube, add extraction volume of 0.09%

(v/v) H_2O_2, and carry through processing identical to that carried out for the samples.

4.6.6.3 Prepare **Spikes** (SP-MDL and SP-VS) – Place blank filters in labeled polypropylene test tubes, add extraction volume of lowest calibration standard solution or verification standard, and carry through processing identical to that carried out for the samples. For instructions on preparation of lowest calibration standard and verification standard consult 6.05/*.*/M.

4.6.7 Ultrasonicate samples for 30 min followed by a 24 h rest period (sulphite is converted to sulphate in the process).

4.6.8 Analyze the samples the day after extraction of filters is performed.

4.6.9 Store remaining extracts in a refrigerator up to 6 months.

4.7 Potassium Hydroxide-Coated Filters: for analyses of organic acids (6.03/*.*/M)

4.7.1 Exposed filters are stored in the refrigerator and should be analyzed within 6 months of reception by the Ion Analysis Laboratory.

4.7.2 Extraction solution: degassed DW

4.7.3 Check a bottle-top dispenser by weighing extraction volume of degassed deionized water (10 or 20 mL) on a top loading balance. Record results in the control chart.

4.7.4 Place filters in clean, labeled, 50-mL wide mouth polypropylene bottles for extraction.

4.7.5 Add 10 mL of degassed deionized water when analyses need to be performed by Capillary Electrophoresis and Ion Chromatography (CE/IC) or 20 mL for IC analyses only

4.7.5.1 Prepare **Vial Blank** (VialBlank) – add extraction volume of degassed deionized water to a labeled 50-mL wide mouth polypropylene bottle and carry through processing identical to that carried out for the samples.

4.7.5.2 Prepare **Method Blank** (MetBlank) – Place a blank filter in a labeled 50-mL wide mouth polypropylene bottle, add extraction

volume of degassed deionized water, and carry through processing identical to that carried out for the samples.

4.7.5.3 Prepare **Spikes** (SP-MDL and SP-VS) – Place blank filters in a labeled 50-mL wide mouth polypropylene bottle, add extraction volume of lowest calibration standard solution or verification standard, and carry through processing identical to that carried out for the samples. For instructions on preparation of lowest calibration standard and verification standard consult 6.03/*.*/M.

4.7.6 Ultrasonicate all samples for 30 min at room temperature (place 2 frozen cold-packs in the sonication bath to maintain room temperature during extraction); mechanical shaking may also be used.

4.7.7 When necessary, eliminate high concentration of potassium hydroxide (e.g. before CE analysis) by passing extract through cation exchange cartridge (Consult SOP 6.07/*.*/S).

4.7.8 Analyze samples within 24 hours from extraction of filters.

4.7.9 Store remaining extracts in a freezer up to 6 months.

4.8 Nitrite-Coated Filters for ozone measurement (Method: 6.03/*.*/M)

4.8.1 Exposed filters are stored in cool, dark place, but not refrigerated, and should be analyzed within 4 weeks of the coating date.

4.8.2 Extraction solution: degassed deionized water.

4.8.3 Place the 15 mm filters in 12-mL polypropylene extraction tubes. Using a verified 5-mL or 10-mL pipette, add 5 mL of extraction solution

4.8.4 Prepare the quality control samples:

4.8.4.1 Prepare **Vial Blank** (VialBlank) – add 5 mL of extraction solution to a labeled 12-mL polypropylene test tube and carry through processing identical to that carried out for the samples.

4.8.4.2 Prepare **Method Blank** (MetBlank) – Place a blank filter in a labeled 12-mL polypropylene test tube, add 5 mL of extraction solution, and carry through processing identical to that carried out for the samples.

4.8.4.3 Prepare **Spikes** (SP-MDL and SP-VS) – Place blank filters in labeled 12-mL polypropylene test tubes, add 5 mL of the lowest calibration

standard solution or verification standard, and carry through processing identical to that carried out for the samples. For instructions on preparation of lowest calibration standard and verification standard consult 6.03/*.*/M.

4.8.5 Ultrasonicate samples for 30 min.

4.8.6 Analyze samples within 24 hours from extraction of filters.

4.8.7 Store remaining extracts in the refrigerator for up to 6 months.

5. Applicable Methods and SOPs

6.03/*.*/M "Determination of Anions and Cations on Multi (2) – Ion Chromatography System"

6.05/*.*/M "Determination of Gaseous and Particulate Inorganic Air Pollutants by Ion Chromatography"

6.10/*.*/M "Determination of Trace Elements in Aqueous Extracts of Airborne Particulate Matter and Other Aqueous Solutions by (ICP-MS)"

6.12/*.*/M "Determination of Levoglucosan and other Carbohydrates in Atmospheric Aerosols by IC-PAD"

2.01/*.*/S "Gravimetric Measurement"

19.02/*.*/S "Volumetric Measurement – MicroPipettes and Bottle-Top Dispensers"

19.04/*.*/S "AAQS water purification system"

2.06/*.*/S "Laboratory Refrigerators and Freezers"

6.01/*.*/S "Sample Management"

6.02/*.*/S "Labware Cleaning"

6.07/*.*/S "Solid Phase Extraction"

6. References

6.1 US EPA, Compendium Method IO-3.1, Preparation and Extraction of Filter Material, June 1999

6.2 Ogawa & Co., Protocol for Ozone Measurement Using the Ozone Passive Sampler Badge, January 2001

7. Revisions

July 2001: Authors: Ewa Dabek and Maria Piechowski; New Document

April 2003: Reviewers: Maria Piechowski, Michal Suski

Changes through text: "below 10°C" changed to 4°C; "<-10°C" changed to -20°C; numbering changed due to addition of paragraph 4.2; title changed in paragraphs: 4.1-4.6; word "clean" added in paragraphs: 4.1.2, 4.3.2, 4.4.2, 4.5.2, 4.6.2; … (60 mL size)… changed to (50 mL size); tube size "12 mL" added; In paragraph 4.4.3: "(47 mm filter size) or 20 mL (70 mm filter size)" added; In paragraph 4.6.3: "mechanical shaking" added; Paragraph added: 4.2. Teflon Filters: for analyses of sulphate; Section added: 5. Applicable SOPs.

September 2004: Reviewers: Maria Piechowski, Michal Suski

Title changed in paragraphs: 4.1, 4.2, and 4.5.

August 2005: Reviewers: Maria Piechowski, Michal Suski

In paragraph 3.3: "10 or" added; In paragraph 3.4: "and Micropipette-10mL" added; Title in paragraph 4.5: "sulphate" changed to "inorganic anions"; In paragraph 4.5.2: "(12 mL for 15 mm filter size or 50 mL for 47 mm)" added; In paragraph 4.5.3: "8 mL (15 mm filter), 20 mL (1 filter 47 mm) or 40 mL (2 filters 47 mm each)" added; Paragraphs: 4.6.3 and 4.6.4 reedited; 4.6.5 added; New section: 4.7. Nitrite-Coated Filters: for ozone measurement.

July 2007: Reviewers: Maria Piechowski, Alicia Berthiaume, Michal Suski

"(4°C)" and "-20°C" removed through text.

May 2009: Reviewer: Maria Piechowski

Section 5 - "2.02/*.*/S" changed to "19.02/*.*/S".

December 2009: Reviewers: Maria Piechowski, Nicole Houle, Susannah Krack

Section 2.1 – new text; Section 3.3 – "scintillation vials (e.g. 20 mL)" – added; Section 4 – Methods added though section; Section 4.1 - new; Sections: 4.2 – 4.8 - preparation of Vial Blank (VialBlank), Method Blank (MetBlank), and spiked Method Blank (MetBlank-SP) included; Section 4 - numbering within section changed, holding time of filters and extracts added; Section 5 – Methods: 6.03/*.*/M, 6.05/*.*/M, 6.10/*.*/M, 6.12/*.*/M, and SOP 6.01/*.*/ - added; Section 6 – new.

May 2011: Reviewer: Michal Suski

Section 4.1.5 – new; sections 4.1.5, 4.1.6 and 4.1.7 re-numbered to 4.1.6, 4.1.7 and 4.1.8 respectively; section 4.8.1 changed to: "Exposed filters are stored in cool, dark place, but

not refrigerated, and should be analyzed within 4 weeks of the coating date."; Section 6.2 – new.

April 2013 Reviewer: Michal Suski, Valbona Celo

Section 3 – source of deionized and ultra-pure water added, technical balance added; Section 4 – added requirement to use double-deionized water for extraction of samples which will be analysed for water-soluble metals by ICP-MS; instructions for preparation of blanks and spikes re-written for clarity and consistence with current practices; Section 5 – added SOP 19.04 (AAQS water purification system). All sections reviewed and edited for clarity.

Lead Reviewer: Michal Suski
Title: Chemist

Lead Reviewer: Valbona Celo
Title: Supervisor, ICP-MS Laboratory

Approved by: Ewa Dabek
Title: Head, Particulate Characterization Unit