

Appendix B***CHESTER LabNet* Standard Operating Procedures**

<u>SOP #</u>	<u>SOP Title</u>
AD-001.05	General Administrative Procedures
AD-002.03	Waste and Sample Disposal
AD-003.04	Refrigerated Storage Monitoring
AD-004.03	Glassware Cleaning for Inorganics Laboratory
AD-005.04	Reagent Procurement and Control
AD-006.06	Laboratory Deionized Water Supply
AD-007.04	Laboratory Information Management System (LIMS)
AD-008.06	Sample Receipt and Log In
GR-001.07	8x10 Quartz & Glass Fiber Filter Inspection and Gravimetry
GR-001a.02	Punching of Exposed 8x10" Quartz or Glass Fiber Filters (renamed ME-008)
GR-002.06	80-125 mm Filter Inspection and Gravimetry **DEACTIVATED 7/14**
GR-003.02	Gravimetric Processing of 25-47mm Quartz Filters **DEACTIVATED 2/02**
GR-004.04	Chemical Impregnation of Cellulose Filters
GR-005.02	Impregnation of Cellulose Filters with Sodium Carbonate **DEACTIVATED 1/02**
GR-006.07	Filter Cassette Loading and Unloading
GR-007.02	Inspection & Preparation of 25-47mm Teflon Filters **MERGED W/GR-010, 5/03**
GR-008.03	Oil Coating of Teflon Filters
GR-009.01	Inspection & Preparation of 82.6-125mm Quartz & Glass Fiber Filters **DEACT 3/02**
GR-010.05	Teflon & Quartz Fiber Filter ... Cahn I - **SUSPENDED 1/2013 **
GR-011.02	Inspection and Preparation of ... Carbon Impregnated Filters **SUSPENDED 6/05**
GR-012.01	Inspection & Preparation of 102mm Teflon Filters **DEACTIVATED 3/02**
GR-013.02	Inspection & Preparation of 8x10" Pallflex Weave Filters **DEACTIVATED 1/02**
GR-014.01	Gross Weighing of 25-47mm Teflon Filters **DEACTIVATED 3/02**
GR-015.03	Quartz Filter Preparation for Carbon Analysis
GR-016.06	Preparation & Use of Control Charts for Gravimetric Analysis
GR-017.02	Acceptance Testing of 47mm Teflon Filters, CFR 50 Part 50 App. L (Drop Test)
GR-018.02	Dickson Temperature and Humidity Data Logger
GR-019.04	Sartorius ME5 Microbalance: Teflon and Quartz fiber filter preparation and gravimetry.
GR-020.02	Use of Balance Run Logbooks and Filter Tracking Logbooks
IC-001.02	Borate Eluant Anions ** DEACTIVATED 4/00 **
IC-002.02	Preparation of Air Filters for Fluoride Analysis ** DEACTIVATED 4/00 **
IC-003.05	Extraction of Media for Ion Chromatographic Analysis
IC-004.02	Clean-Up of Anion Columns **DEACTIVATED 1/02**

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SOP #	SOP Title
IC-005.04	Ion Chromatography: Anions **DEACTIVATED 4/08**
IC-006.04	Ion Chromatography: Cations **DEACTIVATED 4/08**
IC-007.02	Clean-Up of Cation Columns **DEACTIVATED 1/02**
IC-008.04	Hexavalent Chromium by IC-PCD (Air filters) **DEACTIVATED 8/2011**
IC-009.02	Ion Chromatography: Anions & Cations
IC-010.03	Hexavalent Chromium by IC-PCD (Dionex ICS-1100)
ME-001.03	Analysis of Elements by ICP-AES (P40) **DEACTIVATED 3/03**
ME-002.05	Analysis of Elements by Graphite Furnace **DEACTIVATED 10/2014**
ME-003.05	Sample Digestion for Analysis of Elements by ICP (EPA Method 3050)
ME-004.02	Analysis of Mercury in Aqueous Samples **DEACTIVATED 9/10**
ME-005.02	Analysis of Mercury in Solid Samples **DEACTIVATED 9/10**
ME-006.03	Analysis of Mercury in Hopcalite Sorbent Tubes **DEACTIVATED 9/10**
ME-007.02	Analysis of Elements by ICP (Optima 2000) **DEACTIVATED 4/7/14**
ME-008.03	Subsectioning of Exposed 8x10" ... Filters Modified 40CFR50 Appendix G
ME-009.02	Analysis of Mercury in Aqueous and Solid Samples EPA 7470 & 7471 (Nippon 3320A)
ME-010.01	Analysis of Mercury by NIOSH 6009 (Nippon 3320A)
ME-011.01	Analysis of Elements by Inductively-Coupled Plasma Emission (Optima 8300)
ME-012.01	Digestion of Filters for Metals Analysis (Appendix G - Hot Sonication)
OC-001.05	Organic & Elemental Carbon by the Thermal-Optical Method (NIOSH5040 & IMPROVE_A)
XR-001.02	Resuspension of Particulate Matter onto Filter Media
XR-002.05	Analysis of Elements in Air Particulates by X-Ray Fluorescence (Kevex 770 & 772)
XR-003.02	Preparation of Samples for Resuspension
XR-004.02	Kevex XRF Spectrometer Calibration
XR-005.02	Kevex Spectrometer Data Generation, Interpretation and Reporting
XR-006.02	X-Ray Fluorescence (Kevex-771) **SUSPENDED 1/09**
XR-007.02	Analysis of Elements in Air Particulates by X-Ray Fluorescence (Thermo ARL QUANT'X)
QA-001.06	Laboratory Training
QA-002.04	Laboratory Data and Report Validation
QA-003.05	Implementation, Distribution, & Control of Std. Operating Procedures
QA-004.04	Distribution and Control of Laboratory Logbooks
QA-005.02	Control of Laboratory QA/QC Records **DEACTIVATED 9/11/01**
QA-006.04	Determination of Detection Limits, Precision & Bias, & DoC
QA-007.04	Calibration of Laboratory Pipettes
QA-008.04	Assembly and Preparation of Data Reports (Original QA-008 merged with QA-002)
QA-009.02	Internal Auditing
QA-010.01	Laboratory Balance Calibration and Verification
QA-011.01	Control and Handling of Standards and Reference Materials
ST-001.03	Halide & Hydrogen Halide Emissions from Stationary Sources (M26/26a)

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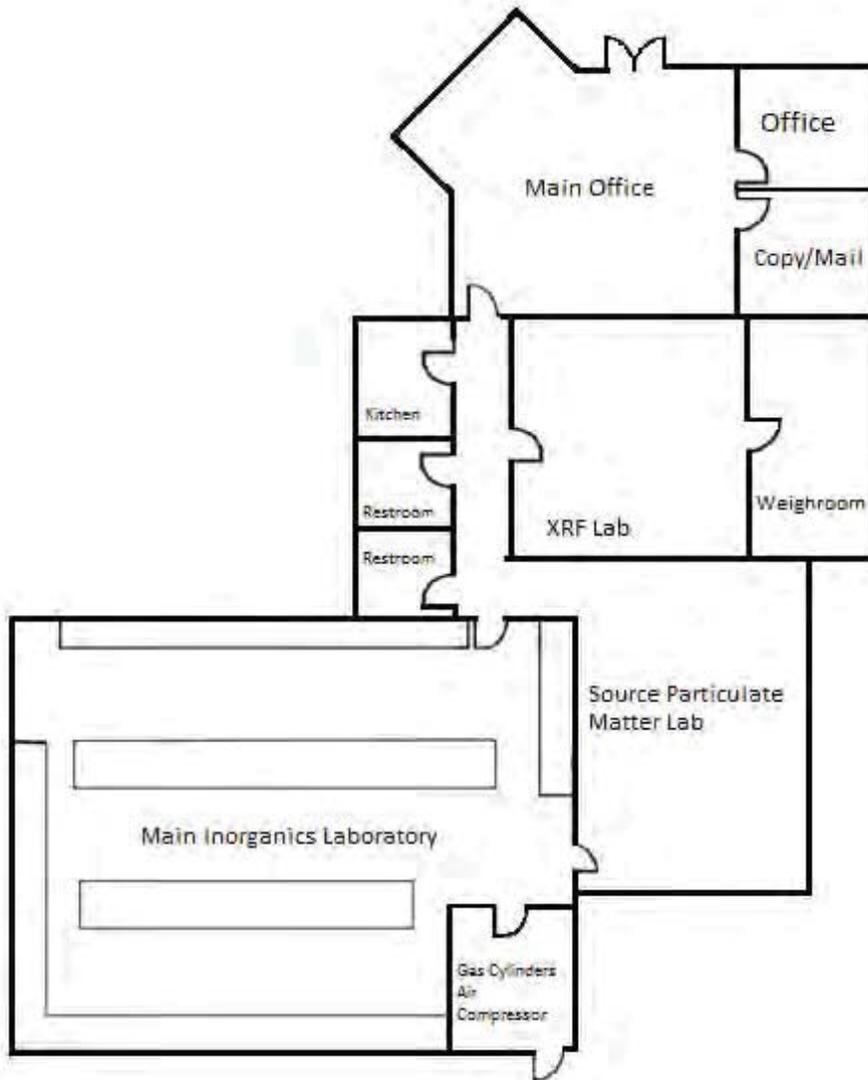
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SOP #	SOP Title
ST-002.02	Particulate Emissions from Stationary Sources (M5-5F, ODEQ M5, "old" M202))
ST-003.03	Sulfur Dioxide Emissions from Stationary Sources (M6)
ST-004.03	Nitrogen Oxide (NOx) Emissions from Stationary Sources (M7D)
ST-005.02	Sulfuric Acid Mist & Sulfur Dioxide Emissions from Stationary Sources (M8)
ST-006.02	Elements by EPA Method 29 or CARB Method 436 (M29)
ST-007.01	Hydrogen Sulfide Content of Fuel Gas Streams **SUSPENDED 3/06**
ST-008.02	Inorganic Lead Emissions from Stationary Sources (M12)
ST-009.03	Total Fluoride Emissions (From Source Samples) (M13B)
ST-010.04	Total Reduced Sulfur Emissions (M15/16) **DEACTIVATED 5/2015**
ST-011.02	Total Reduced Sulfur ** DEACTIVATED** 6/05 (merged w/ ST010 6/14/05)
ST-012.01	Sulfur Dioxide Emissions **DEACTIVATED** 8/05
ST-013.03	Particulate & Gaseous Mercury (M101, 101A, 102)
ST-014.03	Beryllium Screening (M103)
ST-015.02	Beryllium Emissions from Stationary Sources (M104)
ST-016.01	Mercury in Sewage Sludge **SUSPENDED** 6/05
ST-017.01	Particulate & Gaseous Arsenic Emissions **SUSPENDED** 6/05
ST-018.02	Ammonia in Stationary Sources (CTM027)
ST-019.04	Condensable Particulate Matter Method 202 & NCASI modification (M202)
ST-020.03	Determination of PM10 & PM2.5 from Stationary Sources (M201A)
WC-001.01	Chemical Oxygen Demand (COD) **DEACTIVATED** 9/11/09
WC-002.01	Specific Conductance **DEACTIVATED 9/11/09**
WC-003.02	Fluoride by Ion Selective Electrode
WC-004.01	Ammonia-Nitrogen by Ion Selective Electrode **DEACTIVATED** 10/05
WC-005.01	Gravimetric Oil & Grease in Liquids ** DEACTIVATED ** 4/05
WC-006.01	Soil pH **SUSPENDED** 1/05
WC-007.01	Gravimetric TPH. ** DEACTIVATED ** 4/00
WC-008.01	Alkalinity. **SUSPENDED** 1/05
WC-009.01	Cation Exchange Capacity. **SUSPENDED** 1/05
WC-010.01	Redox Potential (eH). **DEACTIVATED** 9/11/09
WC-011.01	Hardness. **SUSPENDED** 1/05
WC-012.01	Nitrite-Nitrogen. **SUSPENDED** 1/05
WC-013.01	Organic Matter. Walkley-Black Method **DEACTIVATED** 9/11/09
WC-014.04	pH in Aqueous Solutions. (Unsuspended 1/11/10)
WC-015.01	Phosphorous/Phosphate, All Species **SUSPENDED** 1/05
WC-016.01	Total Dissolved Solids. **SUSPENDED** 1/05
WC-017.01	Total Kjeldahl Nitrogen. **DEACTIVATED** 10/05
WC-018.01	Total Suspended Solids. **SUSPENDED** 1/05
WC-019.01	Turbidity. **DEACTIVATED** 9/11/09
WC-020.01	Hexavalent Chromium. **SUSPENDED** 1/05
WC-021.02	Alkalinity in Teflon Filters

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Appendix C
Laboratory Floor Plan



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Appendix D**CHESTER LabNet Most Commonly Utilized Methods
(by Issuing Authority and Method Number)****NELAC Accredited Method(s)**

<u>Method Number</u>	<u>Analyte/Element</u>	<u>Instrumentation</u>
CARB MLD 039 (modified)	Hexavalent Chromium	IC-PCD
40 CFR 60 Method 202	Condensable Particulate Matter (source emissions)	Balance (gravimetry)
40 CFR 50 Appendix J	PM10 (ambient air)	Balance (gravimetry)
40 CFR 50 Appendix L	PM2.5 (ambient air)	Balance (gravimetry)
NIOSH 5040	Diesel PM (elemental Carbon)	OC/EC
DRI SOP#2-216r2 (IMPROVE_A)	Organic/Elemental Carbon	OC/EC
40 CFR 60 Method 26A (Method 26 is analytically the same as Method 26A)	HF, HCl, HBr, Cl ₂ , Br ₂	IC - Anions

US EPA IO methods

<u>Method Number</u>	<u>Analyte/Element</u>	<u>Instrumentation</u>
2.1	TSP & PM ₁₀	Balance (gravimetry)
2.2	PM ₁₀	Balance (gravimetry)
2.3	PM ₁₀	Balance (gravimetry)
3.1	metals prep.	wet chemical
3.3	metals	XRF
3.4	metals	ICP

EPA Water/Wastewater methods (by reference from other methods)

<u>Method Number</u>	<u>Analyte/Element</u>	<u>Instrumentation</u>
300.0	Anions	IC
300.7 (revoked) ASTM Method D6919-03	Cations	IC
340.2	F	Ion Selective Electrode

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EPA SW-846 methods (by reference from other methods)

<u>Method Number</u>	<u>Analyte/Element</u>	<u>Instrumentation</u>
3050	metals prep.	wet chemical
6010	metals	ICP
7470/7471	Hg	CVAA

ODEQ methods

<u>Method Number</u>	<u>Analyte/Element</u>	<u>Instrumentation</u>
5	Particulates	Balance (gravimetry)
8	Particulates	Balance (gravimetry)

40 CFR 50, 51 & 60 Source Testing methods

<u>Method Number</u>	<u>Analyte/Element</u>	<u>Instrumentation</u>
5	Particulates	Balance (gravimetry)
6	SO ₂	titrimetric
7 (A & D)	NO _x	IC
8	H ₂ SO ₄ /SO ₂	titrimetric
12	Pb	ICP
13B	F	IC or ISE
14	F	IC or ISE
26	H _x , H _x & X ₂	IC
29	Multi-metals	ICP
101	Hg	CVAA
101A	Hg	CVAA
102	Hg	CVAA
103	Be	ICP
104	Be	ICP
105	Hg	CVAA
108	As	ICP
201A	Particulates	Balance (gravimetry)
202	Particulates	Balance (gravimetry)
306	Cr & CrVI	ICP and IC-PCD
CTM 027	NH ₄	IC
CTM 013 & 013A	H ₂ SO ₄ /SO ₂	IC or titration

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NIOSH methods

<u>Method Number</u>	<u>Analyte/Element</u>	<u>Instrumentation</u>
0500	Particulates	Balance (gravimetry)
0600	Particulates	Balance (gravimetry)
5040	Elemental Carbon	OC/EC
6004	SO ₂	IC
6006	Diborane	ICP
6009	Hg	CVAA
6011	Br ₂ & Cl ₂	IC
6014	NO ₂	IC
6016	NH ₃	IC
7300	metals	ICP
7902	F	IC

CARB methods

<u>Method Number</u>	<u>Analyte/Element</u>	<u>Instrumentation</u>
005	As	ICP
421	HF & HCl	IC
423	As	ICP
425	Total Cr	ICP
436	multiple metals	ICP
SOP MLD039	Hexavalent Cr (CrVI)	IC-PCD

40 CFR 50, Ambient Air Methods

<u>Method Number</u>	<u>Analyte/Element</u>	<u>Instrumentation</u>
Appendix B	TSP	Balance (gravimetry)
Appendix J	PM10	Balance (gravimetry)
Appendix L	PM2.5	Balance (gravimetry)
Appendix Q	Pb	XRF

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Appendix E

Laboratory Accreditation/Certification/Recognition

CHESTER LabNet maintains the following certifications and accreditations:

Organization	Certificate Number	Parameter List
ORELAP	OR100051-010	PM ₁₀ ; PM _{2.5} ; CrVI (ambient air); Carbon (Improve_A); Carbon (NIOSH 5040); Condensable PM (Method 202); Hydrogen Halides and Halides (Method 26/26A).

The certificates and parameter lists (which may differ) for each organization may be found in files maintained by the Laboratory Director.

If accreditation is terminated or suspended, the laboratory will immediately cease to use the certificate number reference in any way and inform clients impacted by the change.

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Appendix F**Data Qualifiers**

List of CHESTER LabNet Data Qualifiers		
<u>Location</u>	<u>Qualifier</u>	<u>Meaning</u>
Standard Reports		
Data Summary (on request)	U	Non-detect
Data Summary (on request)	B	Greater than LOD, less than LOQ
QC Summary	N/C	Duplicate RPD can't be calculated as one or both data points are less than the LOD
QC Summary	#	Duplicate RPD control limits do not apply as one or both of the data points is less than the LOQ
QC Summary	*	Spike recovery limits do not apply where spiked amount is less than ¼ sample result.
Special "CLP-like" Reports		
Data Summary (Form I)	J	Concentration greater than LOD but less than contract required detection limit
Data Summary (Form I)	U	Concentration less than LOD
Data Summary (Form I)	E	Concentration is estimated based upon interferences
Data Summary (Form I)	N	Spike recovery not in control
Data Summary (Form I)	*	Duplicate analysis not in control
Data Summary (Form I)	D	Reported value is from a dilution
Data Summary (Form I)	P	Analyzed by ICP-AES
Data Summary (Form I)	MS	Analyzed by ICP-MS
Data Summary (Form I)	CV	Analyzed by CVAAS
Data Summary (Form I)	C	Analyzed by manual spectrophotometric equipment
Data Summary (Form I)	" "	Where no data has been entered
Data Summary (Form I)	NR	Analyte is not required
QC Summary (Form II – ICV/CCV)	M	Analyzed for analyte
QC Summary (Form II – ICV/CCV)	NR	Analyte is not required
QC Summary (Form II – CRI/CRA)	J	Concentration greater than LOD but less than contract required detection limit
QC Summary (Form II – CRI/CRA)	U	Concentration less than LOD
QC Summary (Form III – blanks)	J	Concentration greater than LOD but less than contract required

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List of CHESTER LabNet Data Qualifiers		
Location	Qualifier	Meaning
		detection limit
QC Summary (Form III – blanks)	U	Concentration less than LOD
QC Summary (Form III – blanks)	P	Analyzed by ICP-AES
QC Summary (Form III – blanks)	MS	Analyzed by ICP-MS
QC Summary (Form III – blanks)	CV	Analyzed by CVAAS
QC Summary (Form III – blanks)	C	Analyzed by manual spectrophotometric equipment
QC Summary (Form V – spikes)	N	Spike amount is less than ¼ sample result
QC Summary (Form V – spikes)	P	Analyzed by ICP-AES
QC Summary (Form V – spikes)	MS	Analyzed by ICP-MS
QC Summary (Form V – spikes)	CV	Analyzed by CVAAS
QC Summary (Form V – spikes)	C	Analyzed by manual spectrophotometric equipment
QC Summary (Form V – spikes)	NR	Analyte is not required
QC Summary (Form V – post-spikes)	P	Analyzed by ICP-AES
QC Summary (Form V – post)	MS	Analyzed by ICP-MS
QC Summary (Form V – post)	CV	Analyzed by CVAAS
QC Summary (Form V – post)	C	Analyzed by manual spectrophotometric equipment
QC Summary (Form V – post)	NR	Analyte is not required
QC Summary (Form VI – duplicates)	*	Both sample results are greater than the LOQ, AND the RPD is out of control
QC Summary (Form VI – duplicates)	P	Analyzed by ICP-AES
QC Summary (Form VI – duplicates)	MS	Analyzed by ICP-MS
QC Summary (Form VI – duplicates)	CV	Analyzed by CVAAS
QC Summary (Form VI – duplicates)	C	Analyzed by manual spectrophotometric equipment
QC Summary (Form VII – LCS)	J	Concentration greater than LOD but less than contract required detection limit
QC Summary (Form VII – LCS)	U	Concentration less than LOD
QC Summary (Form VIII – Serial Dilutions)	E	RPD greater than 10% and original sample concentration is greater than 50 times the LOD.
QC Summary (Form VIII – Serial Dilutions)	P	Analyzed by ICP-AES
QC Summary (Form VIII – Serial Dilutions)	MS	Analyzed by ICP-MS
QC Summary (Form VIII – Serial Dilutions)	CV	Analyzed by CVAAS
QC Summary (Form VIII – Serial Dilutions)	C	Analyzed by manual spectrophotometric equipment

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Note: many of the larger projects will have their own reporting guidelines, Contract Required Quantitation Limits, Action Limits and Data Qualifier Codes. The laboratory will use the guidelines issued by the client in all regards when reporting data, to include reported Quantitation limits and Qualifier Codes.

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Appendix G
Employee Resumés

Quality Assurance Management Plan

Paul D. Duda

President, Laboratory Director, Lead Project Manager, LIMS Administrator

Background:

Hire date: 1989. Experience in air quality filter analysis by X-Ray Fluorescence; experience as project manager; experience with SAS and CLP data package requirements. Experience as Laboratory Information Management System (LIMS) administrator, coordinating all LIMS activities; special expertise in interfacing laboratory data to client-specific databases and end-user data programs.

Career Chronology:

<u>Employment Information</u>	<u>Responsibilities and Duties</u>
President; Laboratory Director; Lead Project Manager; LIMS Administrator; CHESTER LabNet, Tigard, OR 2001 – Present	Corporate affairs for laboratory, including proposal writing, marketing and sales, program and project management, overall profit/loss for company, all accounting/payroll and purchasing; report production for all projects requiring EPA, CLP deliverables. Oversee all procedures, QA/QC and corrective actions associated with sample receipt, log-in, chain-of-custody and storage; project management, and general and specialized report production; client management; oversees operation and maintenance of laboratory information management system (LIMS), including all software and hardware, general data entry, QA/QC, coordination with other project managers and technical staff, training of new users.
Project Manager, LIMS Administrator, Sample Custodian, CHESTER LabNet, Tigard, OR 1992 - 2001.	Project management, all accounting/payroll and purchasing; report production for all projects requiring EPA, CLP deliverables. Oversees all procedures, QA/QC and corrective actions associated with sample receipt, log-in, chain-of-custody and storage; project management, and general and specialized report production; client management; oversees operation of laboratory information management system (LIMS), including all software and hardware, general data entry, QA/QC, coordination with other project managers and technical staff, training of new users.
Gravimetry Laboratory and XRF Analyst, CHESTER LabNet, Tigard, OR 1989 - 1992.	Performed all operations of the filter gravimetry laboratory, including maintaining supplies, filter media acceptance testing, gravimetric analysis of filter media following EPA protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation. Also served as XRF technician, including preparation of samples for analysis, instrument operation, interpretation of spectral results, QA/QC.
1987 - 1988	Miscellaneous employment.

Education:

- Graduate Studies, Business Administration, Portland State University, Portland, OR, 1991-1992.
- B.S., Engineering Management, University of Portland, Portland, OR, 1987.

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Sheri Heldstab
Conventional Chemistry Laboratory Technical Director, QA Officer, Senior Chemist,
Health & Safety Officer

Background:

Hire date: 1992. Experience in inorganic analysis of environmental samples, method development of unusual sample matrices, data interpretation and validation; experience with SAS and CLP data package preparation and requirements; experience in technical writing of Standard Operating Procedures, QA/QC project plans, experience with ORELAP accreditation requirements and documentation (QAMP).

Career Chronology:

<u>Employment Information</u>	<u>Responsibilities and Duties</u>
Conventional Chemistry Technical Director, QA/QC Officer, Senior Chemist, Health & Safety Officer CHESTER LabNet, Tigard, OR 1999 - Present	Oversee all operations of the conventional chemistry laboratory; analyze samples; ensure data meets QA/QC requirements; general technical guidance for clients and staff; manage the flow of samples and data through the laboratory; oversee and train other chemists; oversee day to day operation of the laboratory; manage flow of samples and data through the laboratory; ensure meeting of due dates, and proper maintenance of instruments, and adherence to all QA/QC protocols. Oversight of standard operating procedure and program document production and implementation, oversight of accreditation specific requirements, QC review of data reporting, technical guidance on general QA issues, report production for all projects requiring EPA CLP deliverables.
Account Manager, Lab Support, Portland, OR 1998 - 1999	Performed all duties required to run a one person branch office, including service calls, resolution of client disputes, marketing to new clients, filling of orders and recordkeeping.
Chemist, ChemTrace, Portland, OR 1997 - 1998	Primary operator for IC and GFAA. Performed analysis on high purity water for various nutrients, microbiological testing and silica content.
Senior Chemist, CHESTER LabNet, Tigard, OR 1994 - 1997	Primary operator for IC, ICP, GFAA, CVAA. Analyzed variety of air quality samples using primarily CFR methods. Supervised one Chemist. Generated CLP QC reports. Managed sample throughput and Level I data validation of laboratory.
Associate Chemist, CHESTER LabNet, Tigard, OR 1992 - 1994	Primary operator for IC and performance of bench methods. Analyzed variety of environmental samples using CFR, SW846, DW, SM, NIOSH, OSHA and a variety of other methods.
Laboratory Technician, ASiMI, Washougal, WA 1991 - 1992	Analyzed high purity raw silicon for contaminants utilizing specialized equipment. Generated QC reports to be used in the preparation of Certificates of Lot Analysis.
Chemist, Coffey Laboratories, Portland, OR 1990 - 1991	Analyzed a variety of environmental samples for inorganic constituents using DW, SW846 and SM methods.

Education:

- B.S., Biology (Chemistry minor), University of Oregon, 1989
- Secondary Teaching Certification, University of Oregon School of Education, 1990

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Richard H. Sarver
XRF Technical Director, Senior XRF Analyst

Background:

Hire date: 1986. Experience in analytical chemistry, including biochemical applications and environmental air quality analysis, specializing in the analysis of air particulates by x-ray fluorescence.

Career Chronology:

<u>Employment Information</u>	<u>Responsibilities and Duties</u>
XRF Technical Director, Senior XRF Analyst, CHESTER LabNet, Tigard, OR 1986 - Present.	Coordinate all XRF activities with project managers as needed; train XRF technicians and oversee all XRF operations; market XRF capabilities to outside clients; responsible for maintenance and repair of instruments, supervise sample flow, data interpretation, QA/QC and report generation from XRF analysis; perform highly specialized sample preparation for non-deposit samples, including size fraction and resuspension; technical guidance for clients and in-house staff. Perform XRF analysis and provide technical assistance for state and federal agencies, industrial, consulting and university clients. Ensure adherence to all QA/QC protocols. Awarded EPA equivalency method EQL-0589-072, "Determination of Lead Concentration in Ambient Particulate Matter by EDXRF Spectrometry in May 1989. Principal scientist for the XRF analysis of air particulates for the U.S. EPA national PM2.5 Chemical Speciation Program. Developed XRF method of analysis for the Hazardous Element Sampling Train (HEST), which used activated carbon to trap volatile metals. Continues to participate in the ongoing effort to obtain equivalent method status to EPA Method 29.
Analytical Chemist, Pioneer Hi-Bred International Portland, OR 1980-1986.	Utilized FID/GC analysis to determine metabolic pathways of resident microorganisms in the digestive tract of stressed mice. Handled animals and performed analytical work. Developed SOPs for in house use.

Education:

A.A.S., Chemical Technology, Chemeketa Community College, Salem, OR 1980.

Selected Publications and Presentations:

Sarver, R. H. 1996. Aerosolization as a Means of Sample Preparation of Geological Materials for XRF Analysis and its Validity Compared to EPA Method 3050A Digestion. Journal of the Air & Waste Management Association. 46: 234-240.

Sarver, R.H. and Lytle, C.R. 2000. Parameter optimization for the analysis of PM2.5 by energy dispersive x-ray fluorescence (EDXRF). Presented at PM2000: Particulate Matter and Health, Air & Waste Management Association Specialty Conference, Charleston, SC, January 24-28, 2000.

Sarver, R.H., Mace, J.C. and Duda, P.D. 2002. XRF: Inter-Excitation Quality Assurance and Deposit Uniformity. Presented at Symposium on Air Quality Measurement Methods & Technology, Air & Waste Management Association Conference, San Francisco, CA, November 13-15, 2002.

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Jennifer Schleis
 Gravimetry Laboratory Technical Director, Senior Gravimetry Laboratory
 Technician, XRF Analyst, Chemist

Background:

Hire date: 2007. General laboratory experience. Prior experience in NELAC accredited laboratory. Experience in filter weighing using CFR methods. Instrument experience including IC-PCR, ICP, IC, OC/EC, CVAA. Experience with inorganic analytical methods including CFR, NIOSH, and OSHA methods.

Career Chronology:

<u>Employment Information</u>	<u>Responsibilities and Duties</u>
Gravimetry Laboratory Technical Director; Chemist; Senior Gravimetry Laboratory Technician; XRF Analyst CHESTER LabNet, Tigard, OR 2010 - Present	Oversees and performs all operations of the filter gravimetry laboratory to include acceptance testing, QA/QC, inventory and archives. Performs all operations of the filter gravimetry laboratory. Ensures data meets QA/QC requirements; provides general technical guidance for clients and staff; manages the flow of samples and data through the laboratory; oversees and trains other technicians; oversees day to day operation of the laboratory; manages flow of samples and data through the laboratory; ensures meeting of due dates, proper maintenance of instruments and adherence to all QA/QC protocols. Performs XRF analyses, ensures adherence to all QA/QC protocols. Performs other analytical duties in the Conventional Chemistry lab for those methods for which she has active DOCs.
Chemist; Gravimetry Laboratory Technician CHESTER LabNet, Tigard, OR 2007 - 2010	Analyzed a variety of air quality samples using primarily CFR methods, performed level I data review in real time. Analyzed samples for metal constituents by ICP, CVAA; analyzed samples by IC, IC-PCR and wet chemical methods. Performed all operations of the filter gravimetry laboratory, including filter media acceptance testing, gravimetric analysis of filter media following CFR protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation.
Laboratory Technician Aquatic Biotech & Environmental Lab, Athens, GA 2005 – 2006	Analyzed mutant strains of transgenic fish via specialized assays, gel electrophoresis, PCR, sequencing, DNA isolation; performed in-vitro fertilization and dissections; maintained aquaria and stocks of fresh and saltwater fish.
Laboratory Technician University of Georgia, Athens, GA 2004 – 2005	Performed high-throughput DNA sequencing; maintained and operated all laboratory equipment; provided weekly presentation of data summaries and quality assurance; provided technical assistance to students and research staff.

Education:

B.S. Environmental Economics and Management, University of Georgia, 2003
 Continuing Education, Portland Community College, 2015 (Chemistry)

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Quality Assurance Management Plan

Lisa Ball
Project Manager, Sample Custodian

Background:

Hire date: 1997. Experience as Project Manager. Experience as environmental analytical chemist.

Career Chronology:

<u>Employment Information</u>	<u>Responsibilities and Duties</u>
Project Manager, Sample Custodian, CHESTER LabNet, Tigard, OR 2003 - Present.	Project management. Performs all procedures, QA/QC and corrective actions associated with sample receipt, log-in and chain-of-custody, project management and general and specialized report production; client management; general data entry; coordination with other project managers and technical staff; training of new users. Provides technical guidance to clients.
Project Manager, Sample Custodian, Gravimetry Laboratory Coordinator, CHESTER LabNet, Tigard, OR 2001 - 2003.	Project management. Performs all procedures, QA/QC and corrective actions associated with sample receipt, log-in and chain-of-custody, project management and general and specialized report production; client management; general data entry; coordination with other project managers and technical staff; training of new users. Provides technical guidance to clients. Oversee and perform all operations of the filter gravimetry laboratory.
Chemist, Gravimetry Laboratory Coordinator, CHESTER LabNet, Tigard, OR 1997 - 2001.	Performed all operations of the filter gravimetry laboratory, including maintaining supplies, filter media acceptance testing, gravimetric analysis of filter media following EPA protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation. Analyzed air quality samples using primarily CFR methods, including: sample preparation, and digestion and analysis of samples. Principal Operator of IC, ICP, CVAA. Responsible for Level I data review and reporting.
Extraction Chemist, Oregon Analytical Laboratory, Beaverton, OR, 1997 (full-time, temporary).	Performed extractions for total petroleum hydrocarbons (TPH and TPHD), hydrocarbon identification (HCID), PAHs and oil and grease. Extractions included separatory agitation as well as distillations. Digested, extracted and analyzed water and soil samples for a variety of inorganic constituents including: CODs, pHs, alkalinity, open-cup flashpoints, Total Kjeldahl Nitrogen analysis, Cyanide distillation and analysis.
Chemist, American Environmental Network, Durham, OR, 1996-1997.	Primary wet chemist. Brought new wet chemistry methods on line and wrote corresponding SOPs for wet chemistry methods.

Education:

B.S., Integrated Science, Portland State University, 1996. OSHA 1910.120: 24-hour, 1996.

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Katie Hawks
Chemist, Gravimetry Laboratory Technician

Background:

Hire date: 2014. Prior experience as an analytical chemist for production facility. General laboratory experience. Instrumental experience including ICP. Experience with inorganic analytical methods including CFR, NIOSH, and OSHA methods.

Career Chronology:

<u>Employment Information</u>	<u>Responsibilities and Duties</u>
Chemist Gravimetry Laboratory Technician <i>CHESTER LabNet,</i> Tigard, OR 2014 - Present	Analyzes a variety of air quality samples using conventional chemistry techniques utilizing primarily CFR methods; performs instrumental analysis using ICP and CVAA; performs level I data review in real time. Performs all operations of the filter gravimetry laboratory, including filter media acceptance testing, gravimetric analysis of filter media following CFR protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation.
Sales Associate Shane Company, Tigard, OR 2012 - 2014	Administered Clientele Liaison Program; trained associates on updated software; tracked clientele data; resolved difficult customer situations.
Beauty Consultant Mary Kay, Beaverton, OR 2010 - 2013	Achieved top 5 in sales for national area; recruited and mentored 25 team members; built client base of over 400 customers; earned multiple bonuses.
Chemist Wah-Chang, Albany, OR 2007 - 2010	Analyzed samples by ICP and ICP-MS; designed experiments; advised and communicated with outside departments, co-workers, and students; constructed and executed streamlining procedures; handled various samples, chemicals, and acids, including HF safely; trained students on instrumentation and lab safety.
Medical Technician OHSU, Portland, OR 2006 - 2007	Assisted doctors during sampling; made slides for specimens; logged in and prepared samples; made reagents and standards.
Volunteer Intern Portland Crime Lab, Portland, OR 2005 - 2006	Validated new equipment and trained users; analyzed samples; performed detection limit studies; created analyte list using FTIR-ATR.

Education:

B.S., Chemistry, Western Oregon University, 2006

Julie Delarue
Chemist, Gravimetry Laboratory Technician, XRF Analyst

Background:

Hire date: 2015. Prior experience as an analytical chemist for production facility. General laboratory experience, instrumental experience including ICP, UV/Vis and IR spectrophotometer, GC, and XRF.

Career Chronology:

<u>Employment Information</u>	<u>Responsibilities and Duties</u>
Chemist Gravimetry Laboratory Technician <i>CHESTER LabNet,</i> Tigard, OR 2015 - Present	Analyzes a variety of air quality samples using conventional chemistry techniques utilizing primarily CFR methods; performs instrumental analysis using IC-PCR; performs level I data review in real time. Performs all operations of the filter gravimetry laboratory, including filter media acceptance testing, gravimetric analysis of filter media following CFR protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation.
Laboratory Technologist, Husky Energy, Lloydminster, SK (CAN) 2010 – 2014	Performed troubleshooting and method development; analyzed solid, liquid and gaseous samples by GC and XRF; prepared diesel fuel certification for sale. Responsible for all analytical documentation.
QA Laboratory Technologist, Nestle Purina Petcare, Innisfail, AB (CAN) 2009 - 2010	Performed Protein, Moisture and Fat analyses on pet food; responsible for formal release of product. Responsible for all analytical documentation.
Analytical Laboratory Technologist, Nova Chemicals, Calgary, AB (CAN) 2008 - 2009	Analyzed solid, liquid and gas samples by GC; performed troubleshooting and method development. Responsible for all analytical documentation.

Education:

- B.S., Environmental Sciences, University of Alberta (CAN), 1999
- A.S., Chemical Technology, SAIT Polytechnic (CAN), 2009

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Theodore (“Ted”) Perry
Chemist, Gravimetry Laboratory Technician

Background:

Hire date: 2015. Prior experience as an analytical chemist for independent testing laboratories. Sample preparation and analysis for N content in Fertilizer using bench chemistry techniques; instrumental experience with GFAA, FAA and FTIR.

Career Chronology:

<u>Employment Information</u>	<u>Responsibilities and Duties</u>
Chemist Gravimetry Laboratory Technician <i>CHESTER LabNet,</i> Tigard, OR 2015 - Present	Analyzes a variety of air quality samples using conventional chemistry techniques utilizing primarily CFR methods; performs instrumental analysis using IC-PCR; performs level I data review in real time. Performs all operations of the filter gravimetry laboratory, including filter media acceptance testing, gravimetric analysis of filter media following CFR protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation.
Chemist, Thornton Laboratory, Tampa, FL 2013 - 2015	Prepared and analyzed fertilizer samples for Nitrogen content, including TKN, Ammonium content, insoluble nitrogen, Nitrate and slow release nitrogen. Responsible for all analytical documentation.
Chemist Mission Mountain Labs Arlee, MT 2012 - 2013	Prepared and analyzed primarily nutraceutical samples for metals analysis by GFAA, FAA and FTIR. Responsible for all analytical documentation.
Work Study, Surplus Dept. Oregon State University Corvallis, OR 2011 -2012	Moved surplus supplies and equipment from buildings or storage into other buildings or storage.
Work Study, Physical Science Dept. Linn-Benton Community College Albany, OR 2008 - 2010	Cleaned and prepared laboratory glassware, inventoried chemical stocks and set up and prepared laboratory space and equipment for students.

Education:

B.S., Chemistry (Environmental Chemistry option), Oregon State University, 2012

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Appendix H

Chemistry

H.1 Method Validation

Reference methods are validated using the following criteria where possible:

- LOD study;
- Precision and Bias study; and
- Evaluation of Selectivity.

The 2009 TNI Standard QAMP template states, *“In addition, the action level (compliance level, project decision level etc.) is used to establish the LOQ and/or LOD.”* The laboratory is rarely aware of “the action level (compliance level, project decision level)...” for any given set of samples. The laboratory is not always aware of who the regulatory agency is governing any given set of samples. It is the client’s responsibility to ensure that the laboratory’s LODs are sufficient to meet their project’s reporting/regulatory limits.

a) Limit of Detection (LOD)

The Limit of Detection (LOD) is the laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. None of the promulgated methods performed at **CHESTER LabNet** require LOD studies.

LODs are not required for any component for which spiking solutions or quality control samples are not available. These include XRF analysis, gravimetric analysis (both Gravimetry Laboratory and Conventional Chemistry), and OC/EC analysis. At this writing, no PE/PT samples are available for any ambient air quality method and very few are available for source emission methods. 40CFR60 Method 26A (and Method 26) and 40CFR60 Method 29 are the two most common Source Emissions methods for which PT samples are available.

The 2009 TNI Standard QAMP template states, *“The laboratory will select methods with LODs that are expected to meet the intended data use.”*

The laboratory has no control over method selection, regardless of the intended use. The laboratory will conduct instrumental LOD studies for the *instruments* where an LOD study is possible.

The 2009 TNI Standard QAMP template states, *“LODs are determined in samples that represent the quality system matrices to be evaluated. All sample processing/preparation steps and all determinative steps are used to validate the method for all targeted analytes. The representative quality system matrix will be free from the target analytes of interest or interfering analytes that impact the LOD.”*

The quality system matrix is “Air”, in the case of all work performed at **CHESTER LabNet**. Due to the fact that sampling is part of the promulgated method, it is not

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possible for the laboratory to perform an LOD study using "All sample processing/preparation steps..." Also, due to the need for sampling media (filters, sorbent tubes, chemical solutions) it is not possible to find a quality system matrix "free from the target analytes of interest or interfering analytes that impact the LOD." The laboratory determines the LOD for each determinative step (e.g. instrument) where an LOD is possible to be determined, using a matrix free from the target analytes of interest or interfering analytes that impact the LOD. Briefly, these matrices are:

- IC (Anion/Cation) – DI Water;
- IC-PCR (Cr6) – dilute bicarbonate solution or DI Water (method dependent);
- CVAA – DI water followed by digestion listed in SW-846 Method 7470; and
- ICP – 7% HNO₃.
- [Note: XRF and OCEC results are reported with uncertainties in lieu of an absolute detection limit.]

The 2009 TNI Standard QAMP template states, "When the method or applicable regulation specifies an LOD study, only the specified method will be used. The laboratory will document the process used to derive the LOD and will retain all the supporting data."

No method performed at **CHESTER LabNet** requires an LOD study. LOD studies are performed annually on the instruments listed above, and raw data is retained. Refer to SOP QA-006 for further details.

In brief, the laboratory uses the following procedure to determine the LOD for the method:

1. Three sets of seven standards set at 2 – 4 times the expected LOD are analyzed on non-consecutive days.
2. The standard deviation of the three sets is determined, averaged, and multiplied by three (yielding a three-sigma value).
3. This is compared to previous LOD studies and historical blank data (ICB's/CCB's) for reasonableness. Analyst experience with the particular instrument is also used when setting LODs. A reported LOD will never be lower than the calculated LOD, but may be made higher than the calculated LOD based on blank data and/or analyst experience.

Once the LOD has been determined, the validity of the LOD is verified by a detection (value above zero) for each target analyte in a quality control sample in the same matrix as the LOD study was performed. This is most commonly performed by examining the results of the LOD study data. As the LOD standard is made at 2 to 4-times the LOD, each analyte of interest should have a detectable response. The concentration of the analytes in the quality control sample will be no more than 3 times the derived LOD unless the test contains multiple analytes. In the latter case, the concentration of the target analytes will

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be no greater than 4 times the LOD. This verification will be performed on each instrument that is used for the test.

LODs are performed/repeated:

- before reporting the LOD for a given analyte;
- any time there is a change that affects how the method is performed; or
- when there is a change in instrumentation that affects the sensitivity of the analysis.

LODs are repeated or verified annually for each quality system matrix/technology/analyte combination.

b) Limit of Quantitation

The Limit of Quantitation (LOQ) is an estimate of the minimum amount of a substance that can be reported with a specified degree of confidence.

LOQs are not required for components or properties for which spiking solutions or QC samples are not available. These include XRF analysis, gravimetric analysis (both Gravimetry Laboratory and Conventional Chemistry), and OC/EC analysis.

Based on CLP guidelines, the LOQ is set at five times the accepted LOD result from the LOD study described above.

The laboratory will verify the LOQ by the analysis of a QC sample containing the analytes of concern at a concentration 1 - 2 times the derived (claimed) LOQ. The LOQ is considered verified if recovery of each analyte is within the laboratory's acceptance limits, or the client's data quality objectives.

The LOQ will be verified annually for each quality system matrix, technology and analyte *unless the LOD was determined or verified*. Since the laboratory performs an LOD study for each instrument annually, verification is rarely performed.

c) Precision and Bias

Precision is the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance, or range, in either absolute or relative terms.

Bias is the systematic error that contributes to the difference between the mean of a significant number of test results and the accepted reference value.

Precision and bias using non-reference, modified reference or laboratory-developed methods are established using the procedure outlined below and compared to the criteria established by the laboratory. The Precision and Bias study is only performed when the method is first brought online or when a change in instrumentation gives cause to believe that precision and bias of the method may have changed.

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The 2009 TNI Standard QAMP template states, "*Precision and bias are determined by processing samples through all phases of the method (e.g., sample preparation, cleanup, analysis, etc.) and are evaluated across the analytical calibration range of the method. This study is performed for all quality system matrices for which the test is to be used.*"

Due to the fact that sampling is part of the promulgated method, it is not possible for the laboratory to perform a Precision and Bias study "*by processing samples through all phases of the method*"

The laboratory uses the same matrices as described in subsection "a" above (LOD's) in performing Precision and Bias studies. Refer to SOP QA-006 for further detail. Briefly, the Precision and Bias study is as follows:

- Prepare the method blank and standards following all preparatory steps contained in the method (if any). In instances where no preparation is performed on the samples (samples are run as received), prepare a standard in the same matrix (e.g., DI water). Following the steps in the appropriate standard operating procedure, measure the method blank and one standard at each concentration level together in one analytical run.
- Calculate the mean recovery for each of the three results.
- On a second, non-consecutive day, repeat above.
- On a third, non-consecutive day, repeat steps above.
- Calculate mean recovery for each level over the three days, and for all nine samples.
- Calculate the relative standard deviation for each of the separate means obtained.
- Compare the standard deviations for the different days and for the different concentrations; compare the overall mean and standard deviation with established criteria.

d) Selectivity

Selectivity is the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances (EPA-QAD).

The laboratory evaluates selectivity through the use of method spikes. With many source emissions samples, the chemistry occurring in the source being sampled can be wildly different from another source. Often, the chemistry occurring in the gas stream of the source includes pollution control device emissions containing non-target substances specifically designed to remove the target substance. Selectivity using any substance other than a sample from the source is moot.

Some methods are incompatible with Selectivity tests. Gravimetric analyses, both on filters and source emission samples, do not lend themselves to selectivity. Either the sample and its container have mass, or they don't exist. By virtue of existence, the target analyte (mass) has been responded to. Non-

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target substances do not exist, however, some physical characteristics of the sample (e.g. static) may have an effect on the reading.

H.2 Demonstration of Capability

Demonstration of Capability (DoC): A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision.

Before reporting any data with a given method, a satisfactory DoC is performed. Thereafter, each analyst demonstrates continuing proficiency through the procedures outlined in Ongoing Demonstration of Capability.

a) Initial Demonstration of Capability (IDoC)

An IDoC is performed:

- before using any method;
- when an analyst learns a method new to the analyst;
- each time there is a change in instrument type, personnel or method; and
- if the laboratory or analyst has not performed the method in a twelve-month period.

The IDoC(s) for each analyst is documented on a DoC form retained in the analyst's DoC folder maintained by the QA Officer. The document identifies the analyst(s) involved in preparation and/or analysis; matrix; analyte(s); the method(s) performed; the laboratory-specific SOP used for analysis (including revision number); the date(s) of analysis; and a summary of the results used to calculate the mean recovery and standard deviations.

All raw data, preparation records and calculations for each IDoC are retained either in hardcopy or electronically and are available for review.

When the method specifies a DoC procedure to be followed, only those procedures will be used. If no procedures are specified, the laboratory uses its own procedure. The laboratory uses the same matrices as described in subsection (a) above (LOD's) in performing IDoC studies. Refer to SOP QA-006 for further detail. Briefly, the IDoC study is as follows:

- Prepare four samples in a clean matrix following the entire procedure described in the associated SOP (including any preparatory steps), spiking the clean matrix at a level 1-4 times the LOQ.
- Following the steps in the appropriate standard operating procedure, measure the method blank and the low level standards.
- Using all the results, calculate the mean recovery and the standard deviation of the samples in the same units as the reporting units for samples.
- Compare the recovery and standard deviation to the corresponding acceptance criteria in the method.

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- Complete the Demonstration of Capability Certification Statement and place in the appropriate employee's QA file.

b) Ongoing Demonstration of Capability

After the demonstration of capability is completed, on-going proficiency is maintained and demonstrated at least annually. Each analyst is expected to consistently meet the QC requirements of the method, the laboratory SOP, client requirements and/or the 2009 TNI Standard. Ongoing DoCs for each analyst are documented on a DoC form retained in the analyst's DoC folder maintained by the QA Officer, and all records related to the demonstration are retained.

The laboratory uses SOP QA-006 to demonstrate ongoing DOC. The same process as IDoC is used for ongoing DoC's with the exception that ongoing DoCs usually utilize the most recent four consecutively-run second source standards (e.g., ICV's, LCS's) in the calculation of the statistics, rather than a low level standard. Note that these standards must only be consecutive, not necessarily on the same day or in the same run.

H.3 Calibration

Section 23.2.2 includes information on calibration of support equipment. This Section covers calibration of analytical equipment.

Initial instrument calibration and continuing instrument calibration verification are an important part of ensuring data of known and documented quality. If more stringent calibration requirements are included in a mandated method or by regulation, those calibration requirements override any requirements outlined here or in laboratory SOPs. Generally, procedures and criteria regarding instrument calibrations are provided in the SOPs governing the instruments. The XRFs have a separate SOP discussing calibration of the instruments due to the complexity of the calibration.

H.3.1 Initial Instrument Calibration

- Records:

Initial instrument calibration includes calculations, integrations, acceptance criteria and associated statistics referenced in the test method SOP. Sufficient raw data records are collected to allow reconstruction of the initial instrument calibration. These include, at a minimum, calibration date, instrument, analysis date, analyte names, analyst's signature or initials, concentration and response, calibration curve or response factor, or unique equation or coefficient used to reduce instrument responses to concentration.

Calibration date and expiration date (when recalibration is due) is documented for equipment requiring calibration, where practicable (see Section 23.1).

- Number of Standards and Concentrations:

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If the reference or mandated method does not specify the number of calibration standards to use, the minimum number is three, not including blanks or a zero standard, except as noted below.

The lowest calibration standard is the lowest concentration for which quantitative results can be reported without qualification. The lowest calibration standard is at or below the Limit of Quantitation (LOQ) and is greater than the Limit of Detection. Results that are less than the LOQ are considered to have increased uncertainty, and are reported with a qualifier code if requested to do so by the client.

The highest calibration standard is the highest concentration for which quantitative results can be reported. Data reported exceeding the highest calibration standard without dilutions is considered to have increased uncertainty and are reported with an explanation of the reason for the reporting of said data in the case narrative (e.g., "one shot" sample where re-analysis is not possible).

For instrumentation where single point calibration is recommended by manufacturer's instructions, such as with ICP (with a zero and single point calibration), the following apply:

- a) For single point plus zero blank calibrations, the zero point and the single point standard are analyzed prior to the analysis of samples and the linear range of the instrument is established by analyzing a series of standards, one of which is at the lowest quantitation level.
- b) Zero blank and single point calibration standards are analyzed with each analytical batch.
- c) A standard corresponding to the limit of quantitation is analyzed with each analytical batch and must meet established acceptance criteria when using single point plus zero blank calibrations.
- d) The linearity of single point plus zero blank calibrations is verified at a frequency established by the method or the manufacturer. Linearity may also be verified with each analytical batch by not reporting any data higher than the calibration standard.

Note that the 2009 TNI Standard does not address either thin-film XRF or OC/EC analyses, both of which are used almost solely for air quality analyses. Both of these instrumentations also retain their calibrations for extended periods of time (over 12 months, sometimes several years), and both do not have detection limits, reporting, rather, to their uncertainties.

- Evaluation, Verification and Corrective Action:

All initial instrument calibrations are verified with a standard obtained from a second source traceable to a national standard when commercially available. If a second source is not available, a standard prepared from a different lot may be used. If no standard is commercially available at all, the laboratory will attempt to make a standard in-house, or find alternate means of ensuring the accuracy of the calibration.

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Criteria for the acceptance of an initial instrument calibration is established (e.g., correlation coefficient or relative percent difference) and defined in the instrumental SOPs. The criteria used are appropriate to the calibration technique.

Where appropriate, the laboratory has manual integration procedures that are adhered to when evaluating calibration data. These procedures are also documented in the instrumental SOPs.

Any samples that are analyzed after an unacceptable initial calibration are re-analyzed where possible, or the data are reported with qualifiers appropriate to the scope of the unacceptable condition (see Section 12, "Control of Non-conforming Environmental Testing").

Quantitation is always determined from the initial calibration unless the test method or applicable regulations require quantitation from the continuing instrument calibration verification, except in the case of OC/EC analysis. OC/EC analysis includes an internal single point standard with every sample analysis which is used to adjust the calibration constant for minor fluctuations in gas flows/furnace temperatures/FID functioning.

Corrective actions are performed when the initial calibration results are outside acceptance criteria. Calibration points are not dropped from the middle of the curve unless the cause is determined and documented. If the cause cannot be determined, the calibration curve is re-prepared. If the low or high calibration point is dropped from the curve, the working curve is adjusted and sample results outside the curve are qualified.

H.3.2 Continuing Instrument Calibration Verification

- Records:

The calculations and associated statistics for continuing instrument calibration are included or referenced in the instrumental SOP.

Sufficient raw data records are retained to allow reconstruction of the continuing instrument calibration verification. Continuing instrument calibration verification records connect the continuing verification date to the initial instrument calibration.

Where appropriate, the laboratory has manual integration procedures that are adhered to when evaluating calibration verification data. These procedures are also documented in the instrumental SOPs.

- Frequency:

Calibration is verified for each compound, element, or other discrete chemical species being reported.

Calibration verifications are performed:

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- at the beginning and end of each analytical batch, except instances when an internal standard is used. For methods employing internal standards, one verification is performed at the beginning of the analytical batch. Many inorganic methods require the CCV to be analyzed after every 10 samples. Some methods have more frequent CCV requirements (see specific SOPs).
 - whenever it is expected that the analytical system may be out of calibration or might not meet verification acceptance criteria.
 - when the time period for calibration or the most recent calibration verification has expired.
 - for all analytical systems that have a calibration verification requirement. Requirements can be found in the instrumental method SOPs. Many inorganic methods require the CCV to be analyzed after every 10 samples.
- Evaluation, Verification and Corrective Actions:

The validity of the initial calibration is verified prior to sample analysis by use of an initial instrument calibration verification (ICV) standard, and throughout the run by use of a continuing calibration verification standard (CCV). The acceptance criteria and corrective actions may be found in the instrumental SOPs.

Corrective action is initiated for ICV/CCV results that are outside of acceptance criteria (see Section 12, "Control of Non-conforming Environmental Testing").

H.3.3 Unacceptable Continuing Instrument Calibration Verifications

If routine corrective action for continuing instrument calibration verification fails to produce a second consecutive (immediate) calibration verification within acceptance criteria, then a new calibration is performed or acceptable performance is demonstrated after corrective action with two consecutive calibration verifications.

For any samples analyzed on a system with an unacceptable calibration, some results may be useable if qualified and under the following conditions:

- a) If the acceptance criteria are exceeded high (high bias) and the associated samples are below detection, then those sample results that are non-detects may be reported as non-detects.
- b) If the acceptance criteria are exceeded low (low bias) and there are samples that exceed the maximum regulatory limit, then those exceeding the regulatory limit may be reported.

CHESTER LabNet will only report data associated with failed ICVs/CCVs/ICBs/CCBs if they have no other option; the data reported under such conditions will be heavily annotated.

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Appendix I

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Quality Assurance Management Plan

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Standard Operating Procedure GR-001.07

8X10 QUARTZ AND GLASS FIBER FILTER INSPECTION AND GRAVIMETRY CHESTER LABNET PROPRIETARY METHOD

Approvals:

<u><i>Shirley Hubbard</i></u> Author	<u>2-20-14</u> Date
<u><i>Jim Colvin</i></u> Lead Analyst	<u>1-20-14</u> Date
<u><i>Shirley Hubbard</i></u> QA/QC	<u>2-20-14</u> Date

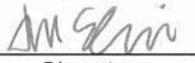
Effective from: 2-20-14
Effective until: present

REVIEW HISTORY

Review date:	Changes made:	Changes made by:
1/10/14	Removed cellophane tape from equipment list; added verification of net weight to Gross QC.	Sheri Heldstab
10/20/13	Updated to 2009 TNI requirements including the addition of Appendix A.	Sheri Heldstab
5/10/11	Changes to reflect new 3g & 5g QC masses/protocol	Sheri Heldstab
1/18/10	Minor changes to text for clarification/grammar	Sheri Heldstab
9/18/08	Minor changes to text for clarification, addition of references to GR-018, and where applicable to operation of Dickson thermometer/hygrometer.	Sheri Heldstab
7/17/07	Additions to text for clarifications.	Sheri Heldstab
5/2/07	Added sections pertaining to new temperature/humidity recorder, updated sections on packing slips to reflect current practices, added information on requirement to pass Cal Out before proceeding to next weigh set.	Sheri Heldstab
3/7/06	Added in sections pertaining to Analyst QC of data, clarified sections pertaining to calibration verification processes.	Sheri Heldstab
11/1/05	Updated to NELAP format.	Sheri Heldstab
11/11/02	Major revision to reflect new balance software. Clarification to text.	Lisa Ball
11/19/01	Updated format. Minor changes and clarifications to text.	Sheri Heldstab
8/14/00	Updated format. Added gross weighing. General additions throughout text.	C.R. Lytle
1/4/95	Header added, typographical errors corrected, references to 94q and 94f changed to 95q and 95f, no other changes to content.	Sheri Heldstab
8/2/94	Coversheet replaced with Chester LabNet coversheet. Review History documentation added. No other changes made.	Sheri Heldstab
12/2/93	No changes. Date of origination.	Sheri Heldstab

ANNUAL REVIEW

The undersigned attests that this standard operating procedure has undergone annual review for adherence to current practices and the latest QA/QC protocols:

<u></u> Signature	<u>Weighroom Tech. Dir.</u> title	<u>3.5.15</u> date
<u></u> Signature	<u>Weighroom Tech Dir.</u> title	<u>6.16.16</u> date
_____ Signature	_____ title	_____ date
_____ Signature	_____ title	_____ date
_____ Signature	_____ title	_____ date

8X10 QUARTZ AND GLASS FIBER FILTER INSPECTION AND GRAVIMETRY CHESTER LABNET PROPRIETARY METHOD

1.0 Introduction

- 1.1 Test Method Reference ID: 40 CFR 50 Appendices B & J; Inorganics Compendium Methods IO 2.1 and IO 3.1; Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II: "Ambient Air Specific Methods, 2.11: PM10 High Volume."
- 1.2 Applicability: This method applies to acceptance testing (inspection) and gravimetric analysis of 8x10" quartz and glass fiber filters.
- 1.3 Detection Limit: The Sartorius balance reads to 0.1 mg. There is no true "detection limit" for this method. All results are reported exactly as obtained from the balance (including negative results), as filter mishandling, either by the laboratory or by the clients, may result in loss of filter material or other spurious results.
- 1.4 Method Performance: Refer to 40 CFR 50 Appendices B and J; EPA Quality Assurance Guidance Handbook for Air Pollution Measurement Systems: Volume 11 Section 2.11; Inorganics Air Compendium Methods IO 2.1 and 3.1. *Sampling is the largest contributor to method performance; Chester Labnet has no control over the actions of the client. See 3-6-15*

2.0 Summary

- 2.1 Scope and Application: The intended use of this method is for the acceptance testing (inspection) and gravimetric analysis of 8x10" quartz and glass fiber filters used for analysis of air particulates. *use 6/7/16 PDD This method meets its intended use. See 6-16-16*
- 2.2 Summary of Method:
- 2.2.1 Filters purchased from commercial vendors are acceptance tested by visual inspection. Filters passing inspection are stamped along the edge with a unique ID number, equilibrated in a temperature and humidity controlled environment, tare weighed, and either stored or shipped immediately to the client. Filters returned from the field are equilibrated in a temperature and humidity controlled

environment and then gross weighed. <sup>PDD
6/7/16</sup> Net weights are electronically transferred to the Laboratory Information Management System (LIMS).

2.2.2 All filters are weighed and data is recorded using a proprietary custom Excel Workbook called FilBERT. ^{SH 6-16-16} FilBERT stands for (FILter Balance Electronic Retrieval Technology). This software automatically records analytical results into two files: the daily log file and the annual data file. The daily log file is a chronological log of analyses performed on a given day on a specific balance and includes: analyst's initials, date/time, filter mass, procedure, net weight and temperature/humidity readings. The annual data file contains all the information pertaining to that filter, organized by the year the filter ID was created and includes the following gravimetric data: Tare, Tare QA, Gross and Gross QA. The software uses the information in the annual data file to calculate the net mass, as well as the difference in QA reweighs.

2.3 Interferences: The most common source of interference is static electricity. The next most common interferent is loss of filter matrix during handling. Positive interferences are less common than negative interferences.

2.4 Sample collection/preservation/shipment/storage: Collection, field preservation and shipment of samples are performed by the client. Chester LabNet has no control over the actions of the client in the field. Upon receipt, new filters and samples are stored in the temperature and humidity controlled Weighroom.

2.4.1 Filters are received from the manufacturer or vendor and are initially kept in their boxes in the Weighroom.

2.4.2 To ensure the ability to supply tare-weighed filters on short notice, a supply of tare weighed filters is stored in a file cabinet drawer in the gravimetry laboratory. These filters are stamped with unique laboratory ID numbers, and are stored individually in glassine envelopes inside third cut filter-folders. ^{file SH 3-6-15} The file folders are identified with the same ID number as is stamped on the filters.

3.0 Safety

- 3.1 Follow the Chester LabNet Chemical Hygiene plan. Always treat samples of unknown origin and/or constitution as hazardous.
- 3.2 This method presents no safety risk beyond typical laboratory safety hazards.
- 3.3 No carcinogenic reagents are used in this method.

4.0 Pollution Prevention and Waste Management

- 4.1 The smallest quantity of chemical feasible is removed from its primary container for use.
- 4.2 Chemicals are used in amounts needed by the method, and excess reagents are not made.
- 4.3 Chester LabNet is a conditionally exempt small quantity generator and as such does not require formal chemical waste processing.
 - 4.3.1 Acidic and Basic wastes are neutralized prior to disposing of them in the sanitary sewer system.
 - 4.3.2 Organic liquids are usually primarily used for cleaning purposes. Organic wastes are generated in very small quantities, and evaporate off with no need for more formal disposal.
- 4.4 Larger quantities of known hazards are returned to the client for disposal.
- 4.5 Expired Chemicals:
 - 4.5.1 Dry chemicals beyond their ^{S46-16-16} ~~real or arbitrary~~ expiration date are lab packed and disposed of by a qualified chemical disposal company.
 - 4.5.2 Acids and Bases beyond their ^{S46-16-16} ~~real or arbitrary~~ expiration date are neutralized prior to being disposed of via the sanitary sewer system.

- 4.5.3 Organic liquids beyond their ^{SH 6-16-16} real or arbitrary expiration date are disposed of by a qualified chemical disposal company if the volume or type of liquid warrants such disposal. Disposal of organic liquids is rare.

5.0 Apparati, Equipment and Supplies

- 5.1 8 x 10" quartz filters (Whatman QMA, Cat. No. 1851-8866)
- 5.2 8 x 10" glass fiber filters (Whatman EPM 2000, Cat. No. 1882-866)
- 5.3 Glassine envelopes
- 5.4 8.5 x 11" third-cut file folders
- 5.5 10 x 13" manila envelopes
- 5.6 Sequential numbering stamp and stamp pad w/ black ink
- 5.7 Disposable gloves
- 5.8 Glue stick (UHU® or equivalent)
- 5.9 Light box
- 5.10 Metal ruler
- 5.11 Kimwipe® brand tissues (or equivalent) ^{PDD 6/7/16}
- 5.12 Bulb for blowing loose particles from filters
- 5.13 Sartorius analytical balance Model B120SFW, pedestal and filter holder
- 5.14 PC running on Windows with Excel 2000
- 5.15 Daily weights: 3.0000g, 5.0000g and 100.0000g ASTM Class 1 weights
- 5.16 Monthly weights: 3.0000g and 5.0000g ASTM Class 1 weights
- 5.17 Anti-static ionizing source (Po Static Eliminator AD1683 by A&D Co., Ltd. or equivalent) ^{StaticMaster SH 2-13-15}
- 5.18 Laminar flow hood with HEPA filter
- 5.19 Dickson Pro Series TP125 temperature/humidity recorder
- 5.20 NIST-traceable temperature/humidity monitor (VWR Cat. No. 35519-044)

6.0 Reagents and Standards

- 6.1 Reagent grade 95% Ethanol

7.0 Preparation, Calibration and Standardization

7.1 Environmental Weighroom Controls

- 7.1.1 All operations involving filters ^{SH 3-6-15} will take place in the Weighroom. Operations not involving the direct weighing of filters ^{SH 3-6-15} will take place in the laminar flow hood in

the weighroom. The analyst must wear gloves to avoid contaminating filters with oil/perspiration from fingers.

7.1.2 The Weighroom is temperature and humidity controlled to satisfy the requirements of four specific reference method and one QA Guidance document, each with its own set of tolerances:

REFERENCE	24 HOUR AVERAGE TEMPERATURE	24 HOUR AVERAGE RELATIVE HUMIDITY
TSP (40 CFR 50, APPENDIX B)	15 - 30 ± 3 °C (59 - 86 ± 5 °F)	< 50 ± 5%
QA Guidance Document 2.11	15 - 30 ± 3 °C (59 - 86 ± 5 °F)	20 - 45% ± 5%
PM10 (40 CRF 50, APPENDIX J)	15 - 30 ± 3 °C (59 - 86 ± 5 °F)	20 - 45 ± 5%
Inorganics Air Compendium Method IO 3.1	15 - 30 ± 2 °C (59 - 86 ± 4 °F)	< 50 ± 5%
PM2.5 (40 CFR 50, APPENDIX L)	20 - 23 ± 2 °C (68 - 73 ± 4 °F)	30- 40 ± 5%
CLN TARGETS	20 - 23 ± 2 °C	30 - 40 ± 5%

The Chester LabNet (CLN) targets for temperature and humidity are set at values that simultaneously satisfy all referenced requirements. Note that the ranges specified for both parameters are maxima allowed within any given 24-hour period.

7.1.3 Each day, prior to weighing filters, check the temperature and humidity. If either of the environmental parameters is out of control, bring it back into the acceptable range, and wait 24 hours before proceeding with any analysis.

7.2 Receipt of new filters

7.2.1 Filters are received from the manufacturer or vendor and are kept in the climate controlled gravimetry laboratory maintained at the above LabNet target values.

7.2.2 For each lot of filters received, at least 2% are set aside in a lot-specific file in the filing cabinet in the gravimetry laboratory. These filters are used in the preparation of method blanks and laboratory control samples for projects in which

chemical analyses are performed on the gross-weighted filters, and may consist of filters which did not pass visual inspection.

7.3 Chemical (metals) analysis of filters

7.3.1 A minimum of two percent of filters from a given lot will be acceptance tested for metals contamination by XRF and ICP prior to inspection or gravimetry.

7.3.2 From each box of filters, set aside 2% or 1 filter per box, whichever is greater, for chemical analysis.

7.3.3 Analyze these filters for metals contamination by ICP (SOP's ME-003, ME-007, ME-008) and XRF (XR-002) ^{SH3-6-15} ^{ME-008 ME-011} ^{or XR-007). ^{PP} 6/7/16}

7.3.4 Compare the data from the above analysis to the manufacturer's specifications, ^{where given,} and to historical data obtained from similar filters. ^{set 3-6-15}

7.3.5 Document the data in the filter lot check spreadsheet. If the laboratory's arbitrary limits for any element should change, document the reason for the change in a line on the spreadsheet.

7.3.6 Filters deemed unacceptable for chemical analysis may still be used for gravimetric analysis.

7.4 Preparation of filter folders for tare-weighted filters ^{SH 3-6-15}

7.4.1 Assemble an equal number of glassine envelopes and third-cut file folders.

7.4.2 Turn the glassine envelope such that the opening is on the right.

7.4.3 Using a metal ruler, precleaned with ethanol and a Kimwipe, slit the top of the glassine envelope such that the top and the right-hand side of the envelope are open and the bottom and left-hand side are still sealed.

7.4.4 Hold a manila folder such that the folded side is at the bottom. Using a glue stick, glue the glassine envelope to the inside back of the file folder such that the open

sides are at the top and right-hand side of the folder.

7.5 Clean workspace and balance:

7.5.1 At the beginning of each workday, clean the countertop of the laminar flow hood and the front of the light box with 95% ethanol and a Kimwipe.

7.5.2 Clean the balance as follows:

7.5.2.1 Remove the filter holder and static guards.

7.5.2.2 Using the bulb, blow all particles off of stage and out of weighing chamber.

7.5.2.3 Wipe the stage, floor, all windows including the top one (inside and outside) with a Kimwipe wetted with ethanol.

7.5.2.4 Thoroughly clean the filter holder with a Kimwipe wetted with ethanol.

7.5.2.5 Allow all parts to dry completely before proceeding with calibrating or using the balance.

7.6 Visual Inspection of filters

7.6.1 **Use gloves when handling filters.**

7.6.2 In the pre-cleaned laminar flow hood, remove the filter from its box, handling only by its edges, and hold above the light box. Inspect the filter for any of the following: deep creases, tears or holes, large dark spots, irremovable foreign particles. Attempt to remove foreign particles by blowing on them with the bulb.

7.6.3 If no flaws are found, the filter is considered "fit for use." Such filters are suitable for both gravimetric and chemical analysis of air particulates.

7.6.4 If holes, tears, or deep creases are found, the filter is rejected, and is either discarded or retained for use as a lot blank for chemical analysis (see section

7.2.2).

7.6.5 After inspection, place filter in the glassine lined file folder, grid-marked side up, and set aside to be given ID numbers.

7.7 Filter ID numbers:

7.7.1 Check the Active Sample Logbook for the next available ID number. If no IDs are available, contact the LIMS administrator or Gravimetry Laboratory Technical Director for a new block of numbers.

7.7.2 Set the sequential stamp to the next ID number and place the stamp in the laminar flow hood.

7.7.3 Using the sequential stamp, stamp the filter ID number on the top right-hand corner of the filter, as close to the edges as possible. Place the number such that no part of the deposit area will overlap the number. Note: the air sampler will leave an approximately one-half-inch margin on all sides of an 8x10" filter. See Analyst's Note 13.4.

7.7.4 Stamp the same ID number onto the filing tab of the manila folder.

7.7.5 Record the ID number, inspection date, analyst's initials and lot number in the Active Sample Logbook.

7.8 Calibrate balance:

7.8.1 Verify the balance is level by examining the leveling indicator on the back right of the balance. The bubble must be within the circle. If it is not, adjust the thumb wheels on either side of the balance until the bubble is contained within the circle.

7.8.2 Ensure that the 24 hour temperature and humidity values are within the control parameters as described in section 7.1.2. Follow SOP GR-018 for Dicksonware operation instructions.

- 7.8.3 Prepare the balance for fixed weight calibration by removing the filter holder.
- 7.8.4 Manually tare the balance by pressing the Tare button on the front of the balance.
- 7.8.5 When the balance stabilizes to 0.0000g, press and hold the CAL button on the front of the balance until it beeps and "+100.0000g" appears on the LCD readout.
- 7.8.6 Place the 100g class 1 weight on the balance stage. This will calibrate the balance at 100.0000g.

Note: Do not handle any class 1 weight without gloves as skin oils can irreversibly damage the weight. The use of forceps for the smaller weights or latex, nitrile, cotton or polyester gloves for the larger weights is required when handling class 1 weights.

- 7.8.7 After the weight has stabilized, the balance will beep to indicate that calibration has occurred. The resulting readout must be 100.0000g. If it is not exactly 100.0000g, remove the weight and repeat the calibration beginning with step 7.8.4.
- 7.8.8 After a successful calibration, proceed by verifying the calibration.

7.9 Verify Calibration:

- 7.9.1 Open FilBERT Sartorius B120S software:

- 7.9.1.1 Open Excel

- 7.9.1.2 In Excel, go to File → Open, then open "B120S FilBERT137" in the desktop directory. Note: older versions of FilBERT are maintained on the computer; the version in the desktop directory is the most current.

- 7.9.1.3 This procedure (opening Excel first, then FilBERT) ensures that another balance may be operated at the same time as the B120 is being used.

7.9.2 Log in to FilBERT program:

7.9.2.1 Click on "Login" and enter the analyst's initials.

7.9.2.2 When prompted for the current temperature and humidity, enter the correct temperature and humidity as displayed by the Dickson software. Refer to SOP GR-018 for operation of the Dickson monitor.

7.9.2.3 Select the most recent Sartorius Calibration log located in C:\MyDocuments\Filbert\Filters\Daily Run Logs.

7.9.3 In FilBERT, click "Tare Weigh Filters."

7.9.3.1 When prompted by the FilBERT, select the file in which to store the daily calibration verification data, located in C:\My Documents\Filbert\Filters\DailyCal.

7.9.3.2 From the pull down menu, select "100.0000g." Click "Tare Weigh Filter."

7.9.4 Cal In.

7.9.4.1 FilBERT will prompt the user to Cal In.

7.9.4.2 As directed by FilBERT, press "OK" to tare the balance.

7.9.4.3 FilBERT will prompt the user to place the 3.0000g weight on the stage. Wait until the reading stabilizes before clicking "OK."

7.9.4.4 The computer will begin taking readings in two second increments. When the computer has taken six consecutive identical readings, the computer will beep and record the data into the selected spreadsheet.

7.9.4.5 FilBERT will ask if the calibration has passed. Click "yes" if the registered value is $\pm 0.0005\text{g}$ of the true value of the weight.

7.9.4.6 FilBERT will repeat steps 7.8.4.3 through 7.8.4.5 for the 5.0000g weight.

7.9.4.7 FilBERT will prompt analyst to remove all weights. Allow the balance to return to 0.0000g. Click "OK" to allow FilBERT to take the reading. The balance must return to and record 0.0000g or else FilBERT will say the calibration has failed and prompt the user to retry the Cal In procedure as in sections 7.9.4.1 through 7.9.4.6.

7.9.4.8 Verify 100.0000g weight

7.9.4.8.1 As prompted by FilBERT, place the 100g class 1 weight on the balance stage and click "Tare Weigh Filter."

7.9.4.8.2 The computer will begin taking readings in two second increments. When the computer has taken six consecutive identical readings, the computer will beep.

7.9.4.8.3 The result must be 100.0000g. If it is not exactly 100.0000g, remove the weight repeat the balance calibration as in section 7.8. After the balance is recalibrated, verify the 100g weight as in 7.9.4.8.

7.9.5 Calibration Verification

7.9.5.1 After the 100g weight has passed, select "3.0000A" from the pull down menu.

7.9.5.2 Wearing a glove or using plastic coated forceps, place the 3.0000g weight, as directed by FilBERT, on the stage. Wait until the reading stabilizes then click "OK".

7.9.5.3 Once the reading stabilizes, FilBERT will ask if the calibration has passed. Click "yes" if the registered value is $\pm 0.0005g$ of the true value of the weight.

7.9.5.4 Repeat steps 7.9.5.1 through 7.9.5.3 five more times, using the following ID sequence: 3.0000g A, 5.0000g A, 3.0000g B, 5.0000g B, 3.0000g C, 5.0000g C.

7.9.5.5 Remove the final 5.0000g weight from the balance and click "Exit" to end weighing session.

7.9.6 Balance Cal Out:

7.9.6.1 The computer will prompt the user to place the 5.0000g standard calibration weight on the stage.

7.9.6.2 The computer will begin taking readings in two second increments. When the computer has taken six consecutive identical readings, the computer will beep and record the data into the selected spreadsheet.

7.9.6.3 FiLBERT will ask if the calibration has passed. Click "yes" if the registered value is $\pm 0.0005g$ of the NIST certified true value of the weight.

7.9.6.4 The computer will prompt the user to place the 3.0000g standard calibration weight on the stage.

7.9.6.5 The computer will begin taking readings in two second increments. When the computer has taken six consecutive identical readings, the computer will beep and record the data into the selected spreadsheet.

7.9.6.6 FiLBERT will ask if the calibration has passed. Click "yes" if the registered value is $\pm 0.0005g$ of the NIST certified true value of the weight.

7.9.6.7 The computer will prompt the user to remove all weights and wait for the balance to stabilize.

7.9.6.8 Wait for the balance to stabilize then press "enter." The computer will begin taking readings in two second increments. When the weight has

registered, it must be 0.0000g. If the Calibrate Out readings are within control, proceed with weighing filters. If it is out of control, recalibrate the balance per sections 7.8 and re-verify the calibration per section 7.9.

7.10 Document Calibration/Verification data:

7.10.1 On a daily basis, fill in the daily balance control chart by writing the three 3g and three 5g weight readings, their average, their moving range and the date and initials of the analyst performing the work, as per SOP GR-016.

7.10.2 If the average mass of the weight is not within control, recalibrate beginning with section 7.8. If unacceptable results are obtained a second time notify the Gravimetry Laboratory Technical Director for corrective actions. Corrective action may include outside service/repair of the balance.

7.10.3 On the last day of the week which the balance is used, open the daily run file in which the current data is stored. Set the print area to include all the calibration data for the respective week and print.

7.10.4 Place the printout in the 3-ring binder labeled "B120S Calibration" in the weighroom.

7.11 Log out of the software to save the data and to close the daily run file. It is important to log out to ensure that the proper data storage file is selected prior to analyzing samples.

7.12 Return the filter holder onto the balance stage.

7.13 Monthly Calibration Check

7.13.1 Perform the Monthly Calibration Check as scheduled by the Gravimetry Laboratory Technical Director.

7.13.2 Follow the Daily Calibration Verification Procedure given in Section 7.9 with the following exceptions:

- 7.13.2.1 Select the Sartorius B120S Monthly Cal log located in C:\MyDocuments\Filbert\Filters\DailyRunLogs. The file is named "SART Monthly Cal" (change of section 7.9.2.3).
- 7.13.2.2 Click on Tare Weigh Filters. When prompted by the software, select a data file in which to store the data. Open the Monthly Calibration file located in C:\My Documents\Filbert\Filters\DailyCal. The file is named "YYYY SART Monthly Cal" where "YYYY" is the current year (change of section 7.9.3.1).
- 7.13.2.3 It is not necessary to verify the 100g weight (change of section 7.9.4.8).
- 7.13.2.4 Use the daily working standard weights for Cal In and Cal Out, however, used the specifically designated standard weights for the Monthly Calibration Check (e.g. one set of standards for Cal In/Out, second discrete set of standards for the Monthly check) **DO NOT ALLOW THESE STANDARDS TO BE ACCIDENTALLY SWITCHED DURING USE.**
- 7.13.3 The passing value of these Monthly Calibration checks is $\pm 0.0005\text{g}$ of the true value of the weight as provided on the annual certification of measurements for that standard weight.
- 7.13.4 Document the Monthly Calibration Check by printing out the raw data and placing it in the Monthly Cal three-ring binder, and filling out the control chart also located in the Monthly Cal binder.

8.0 Procedure

8.1 Tare weigh filters:

- 8.1.1 Check the environmental parameters to ensure they are within control limits (see Section 7.1).
- 8.1.2 Log into the FILBERT Sartorius B120S program:

8.1.2.1 Click on Login and enter the analyst's initials.

8.1.2.2 When prompted for the current temperature and humidity, enter the correct temperature and humidity as monitored and recorded by the Dickson thermometer/hygrometer software. Refer to SOP GR-018 for operation of the Dickson thermometer/hygrometer.

8.1.3 Create or Select a log file:

8.1.3.1 When prompted by the software to select a log file, open C:\My Documents\Filbert\Filters\Daily Run Logs.

8.1.3.2 Create a new log for each day. The files are named in the format YYMMDDNT.

Where: YY is the last two digits of the current year
MM month
DD day
N is the balance used (S for Sartorius)
T type of weighing (T for tare, G for gross)

8.1.4 Create or Select data file:

8.1.4.1 Once the run file is created/selected, click on Tare Weigh Filters.

8.1.4.2 The software prompts the user to select a data file into which FilBERT will store the data. Open the file into which the data is to be stored.

8.1.4.3 Annual data is currently being stored in the form "YYYY Sart"

Where YYYY = the year

Sart = the balance on which the filter is being weighed

These files are located in C:\My Documents\Filbert\Filters.

8.1.5 Balance Control Check (Cal In):

8.1.5.1 Enter the first filter ID into the Filter name box at the top of the screen.
Note that the filter name format must be YY-QNNNN

where:

YY = year

Q = type of filter (in this case, Quartz – F for Glass fiber)

NNNN = four digit number reflecting the chronological filter ID

Failure to keep four digits in the filter ID will cause the filter to not sort properly within the spreadsheet, causing difficulties in retrieving data.

8.1.5.2 FilBERT will prompt the user to place the 3.0000g and the 5.0000g standard weight on the stage.

8.1.5.3 FilBERT will ask if the calibration has passed. Click "yes" if the registered value is ± 0.0005 g of the true value of the weight. If the reading did not pass, perform a manual internal calibration (section 7.8), then begin the Cal In procedure again.

8.1.5.4 FilBERT will prompt the user to remove all the weights and allow the balance to stabilize. Once stable, click "OK." FilBERT will ask if the value is 0.0000g. If it is, click "OK." FilBERT will then tare the balance. If the balance does not return to 0.0000g, FilBERT will prompt the user to recalibrate, then begin the Cal In procedure again.

8.1.6 Tare weigh filters:

8.1.6.1 Place the first filter in the filter holder. Center the filter and ensure that no part of the filter is touching the walls of the balance.

8.1.6.2 FilBERT requires six consecutive identical measurements, taken every 2 seconds, prior to accepting the data as a true measurement. A progress bar at the bottom of the dialog box shows how many identical consecutive readings have been made. Reading may be cancelled at any

time by clicking the "cancel reading" button. The software will then return to the weigh filters dialog.

8.1.6.3 When the reading has been taken and recorded, the computer will beep.

8.1.6.4 Remove the filter from the balance, and place back inside its folder.

8.1.6.5 To weigh the next filter, type in the next filter ID or click the "Increment Filter Number" button if the numbers are consecutive.

8.1.7 Calibration Check:

8.1.7.1 After every 10 filters are weighed, the software prompts the user to remove everything from the balance and perform a Calibration Check.

8.1.7.2 Before another filter can be weighed, the computer will prompt the user to place either the 3.0000g or the 5.0000g standard calibration weight on the stage.

8.1.7.3 FilBERT will ask if the calibration has passed. Click "yes" if the registered value is $\pm 0.0005g$ of the NIST certified true value of the weight. If the calibration check fails, the weigh set preceding the calibration check must be weighed again with passing QC.

8.1.7.4 Remove the weight from the balance stage and wait for the balance to stabilize, then press "enter" to read and record 0g. When the weight has registered, it must be 0.0000g. If the measurement is not 0.0000g, then the filter set is invalid, the balance must be manually tared, and the filter set must be weighed again.

8.1.7.5 After the Cal Check readings have been taken, FilBERT will request the next filter ID to be weighed. Continue to tare weigh.

8.1.8 Final Cal Out:

- 8.1.8.1 After all filters have been weighed, click the exit button. The software prompts the user to perform the Cal Out.
- 8.1.8.2 Remove the last filter from the balance, wait for the balance to read 0.0000g before following the Cal Out prompts in FiLBERT.
- 8.1.8.3 The computer will prompt the user to place the 5.0000g standard calibration weight on the stage.
- 8.1.8.4 Once weighed and recorded, FiLBERT will ask if the calibration has passed. Click "yes" if the registered value is ± 0.0005 g of the NIST certified true value of the weight. If the reading does not pass, then the filter set is invalid and the must be weighed again with passing QC.
- 8.1.8.5 The computer will prompt the user to place the 3.0000g standard calibration weight on the stage.
- 8.1.8.6 Once weighed and recorded, FiLBERT will ask if the calibration has passed. Click "yes" if the registered value is ± 0.0005 g of the NIST certified true value of the weight. If the reading does not pass, then the filter set is invalid and the must be weighed again with passing QC.
- 8.1.8.7 The computer will prompt the user to remove all weights and wait for the balance to stabilize.
- 8.1.8.8 Wait for the balance to stabilize then press "enter." When the weight has registered, it must be 0.0000g. If the measurement is not 0.0000g, then the filter set is invalid, the balance must be manually tared, and the filter set must be weighed again with passing QC.
- 8.1.8.9 Logout of FiLBERT to save data.
- 8.1.8.10 Record the tare weighing date and analyst's initials in the Sartorius B120S Logbook and the Active Sample Logbook.

8.2 Tare QA Weigh Filters

- 8.2.1 Wait a minimum of 20 hours before performing QA reweight.
- 8.2.2 After the 20 hour holding period, 20% of the filters, chosen at random, must be reweighed to pass QA.
- 8.2.3 Follow the weighing procedure for tare weighing (section 8.1). Click "QA Tare Weighed Filters" instead of "Tare Weigh Filters."
- 8.2.4 Filters must fall within $\pm 0.0010\text{g}$ of the initial tare weight to pass QC. If any filter fails this check, the entire set of 10 filters must be re-tare weighed.

8.3 Document Tare weights:

- 8.3.1 After Tare QA weighings have been performed, select the daily run file tab at the bottom of the window.
- 8.3.2 Set the print area to include both the tare and the reweight data, then print.
- 8.3.3 *physically 5/13/15*
Count the number of filters weighed and write the total number of filters weighed in that weigh set in the right-hand margin of the printout.
- 8.3.4 Do not close this file manually, the FilBERT program will close this window automatically upon logging out.
- 8.3.5 By client request on a project specific basis, this printout may be affixed into a bound logbook using tape. Authenticity is attested by the analyst signing the printout across its edge underneath the taped portion of the printout's edge.
- 8.3.6 Transfer the data from the balance computer to the LIMS system and release the data:
 - 8.3.6.1 Open the Tare Daily Run Log file that contains the data to be put on the LIMS.

8.3.6.2 Delete the columns and rows that are not needed. The necessary columns must be in the following order: LIMS ID, Tare Weight, Temperature, Humidity, Analyst Initials, Tare Date, Time and the lot number. Save the file as BULK TARE.csv in the worklist directory.

8.3.6.3 Import and save data to the LIMS per SOP AD-007.

8.3.6.4 Write the worklist number at the top of the printout and request another analyst to perform a QC peer review.

8.4 QC peer review: This section applies to both Tare and Gross weight QC peer review

8.4.1 QC-peer review must be performed by someone other than the analyst who weighed or QA reweighed the filters.

8.4.2 Review the printouts for accuracy, including the following items:

- No filters numbers were skipped. For tare weights, filter ID numbers should be in sequential order.
- All weigh sets are bracketed by a Cal in and Cal out, and that all Cal ins and Cal outs pass QC.
- Reweights were performed no sooner than 20 hours after the initial weighing.
- Temperature and Humidity were in control for all weighings.
- An appropriate number of reweights were completed on each set of filters weighed.
- Number of filters weighed is the same as the number written in the right-hand margin of the printout.
- No two consecutive measurements are identical or nearly identical (one filter weighed twice).
- For Net Weight calculations only (after gross weighing is completed): verify the net mass on the printed worklist against the net mass on the LIMS (LIMS calculates net mass independently from FILBert) at a frequency of at least 20%, where possible.

- 8.4.3 After review, date and initial the upper right corner of each log to indicate the person performing the QC review and the date on which the review took place.
- 8.4.4 Once the review is complete, distribute the worklist on the LIMS, and place the printed logs in the appropriate 3-ring binders in the weighroom.
- 8.5 Filter Storage: If filters are not to be immediately shipped, store the tared filters in the appropriate drawer of the filter storage file cabinet in the gravimetry laboratory.
- 8.6 Shipping filters:
 - 8.6.1 Upon client request, the project manager notifies the Gravimetry Laboratory Technical Director, who then schedules the work based upon current workload, inventory, and client delivery requirements.
 - 8.6.2 Filter ID labeling: The LIMS Administrator or Project Manager will prepare sample ID label pairs to be affixed to the filter file folder tabs and the manila envelopes. Prepare filters as follows:
 - 8.6.2.1 Checking the ID number of each filter, place the appropriate ID label onto the corresponding tab of each file folder, covering the previously stamped filter ID.
 - 8.6.2.2 Place a corresponding label onto the upper right-hand corner of a manila envelope.
 - 8.6.2.3 Place the file folder containing the filter into the corresponding envelope.
 - 8.6.3 Generate an electronic packing slip and analysis request sheet in Excel format on the LIMS hard drive. Print Packing slips and Filter/analysis request sheets. Save each sheet in a new tab in the Excel file, labeling the new tab by ship date.
 - 8.6.4 Fill in the client information, the analyst's initials and the logout date in the Active Sample Logbook.

8.6.5 The project manager ships the filters as requested by the client.

8.7 Filter receipt and log-in:

8.7.1 Filters returned from the field are logged-in by the project manager or sample custodian, who completes the chain-of-custody (if present) and enters the appropriate filter and field information into the LIMS. See SOP AD-008, Sample Receipt and Log-In.

8.7.2 The appropriate lines in the filter ID Tracking Logbook are marked with the date and time the filters were placed in the weighroom, and the initials of the person who logged the filters into the weighroom.

8.7.3 The login date and time is written on the bottom right corner of the envelope of the first filter and then is placed on the "To Be Gross Weighed" shelf in the filter receiving area of the gravimetry laboratory. Any corresponding worklists or run sheets are retained with the filters until all gravimetric analysis is complete and has passed QC.

8.7.4 Allow the filters to equilibrate for a minimum of 24 hours in the temperature and humidity controlled weighroom.

8.8 Gross weigh filters:

8.8.1 Verify that the filters have equilibrated for a minimum of 24 hours after entering the gravimetry laboratory.

8.8.2 Check the environmental parameters to ensure they are within control limits (see Section 7.1).

8.8.3 Log into the FilBERT program:

8.8.3.1 Click on "Login" and enter the analyst's initials.

8.8.3.2 When prompted for the current temperature and humidity, enter the correct temperature and humidity as displayed by the Dickson TP125

software program. Refer to SOP GR-018 for operation of the Dickson thermometer/hygrometer.

8.8.4 Create or select a log file:

8.8.4.1 When prompted by the software to select a log file, open C:\My Documents\Filbert\Filters\Daily Run Logs.

8.8.4.2 Create a new log for each day. The files are named in the format YYMMDDNT.

Where: YY is the last two digits of the current year
MM month
DD day
N is the balance used (S for Sartorius)
T type of weighing (T for tare, G for gross)

8.8.5 Create or Select data file:

8.8.5.1 Once the run file is created/selected, click on "Gross Weigh Filters."

8.8.5.2 The software prompts the user to select a data file into which FilBERT will store the data. Open the file into which the data is to be stored.

8.8.5.3 Data is currently being stored in the form "YYYY Sart"

Where YYYY = the year

Sart = the balance on which the filter is being weighed

These files are located in C:\My Documents\Filbert\Filters.

8.8.6 Balance Control Check (Cal In):

8.8.6.1 Enter the first filter ID into the Filter name box at the top of the screen.

8.8.6.2 FilBERT will prompt the user to place the 3.0000g or the 5.0000g standard weight on the stage.

8.8.6.3 FilBERT will ask if the calibration has passed. Click "yes" if the registered value is ± 0.0005 g of the true value of the weight. If the reading did not pass, begin the Cal In procedure again starting with 8.8.6.2.

8.8.6.4 FilBERT will prompt the user to remove all the weights and allow the balance to stabilize. Once stable, click "OK." FilBERT will ask if the value is 0.0000g. If it is, click "OK." FilBERT will then tare the balance. If the balance does not return to 0.0000g, FilBERT will prompt the user to begin the Cal In procedure again starting at 8.8.6.2.

8.8.7 Gross Weigh Filters:

8.8.7.1 Inspect filter: carefully unfold the filter if it was folded, note any irregularities, anomalies, missing parts of filter, etc. in the LIMS. If any unusual foreign objects are present, such as large insects, footprints, hair, pine needles/leaves, bird droppings or other obviously improper materials, contact the client for instructions on how to proceed (remove the particle or leave as part of the mass). Refold the filter prior to placing it on the balance.

8.8.7.2 Place the first filter in the filter holder. Center the filter and ensure that no part of the filter is touching the walls of the balance.

8.8.7.3 FilBERT requires six consecutive identical measurements, taken every 2 seconds, prior to accepting the data as a true measurement. A progress bar at the bottom of the dialog box shows how many identical consecutive readings have been made. Reading may be cancelled at any time by clicking the "cancel reading" button. The software will then return to the weigh filters dialog.

8.8.7.4 When the reading has been taken and recorded, the computer will beep.

8.8.7.5 Remove the filter from the balance, and place back inside its folder and envelope.

8.8.7.6 To weigh the next filter, type in the next filter ID or click the "Increment Filter Number" button if the numbers are consecutive.

8.8.8 Calibration Check:

8.8.8.1 After every 10 filters, FilBERT prompts the user to remove everything from the balance and perform a Calibration Check. Perform as in section 8.1.7.

8.8.9 Final Cal Out:

8.8.9.1 After all filters have been weighed, click the "exit" button. FilBERT will prompt the user to remove everything from the balance and calibrate out. Perform as in Section 8.1.8.

8.8.10 Record the gross weighing date and analyst's initials in the Sartorius B120S Logbook and the Active Sample Logbook.

8.9 Gross QA Weigh Filters

8.9.1 Wait a minimum of 20 hours before performing QA reweight.

8.9.2 After the 20 hour holding period, 20% of the filters, chosen at random, must be reweighed to pass QA:

8.9.3 Follow the weighing procedure for gross weighing filters (section 8.8). Click "QA Gross Weighed Filters" instead of "Gross Weigh Filters."

8.9.4 The filters must fall within +/- 0.0020g of the initial weighing to pass QC. If either filter fails this check, the entire set of 10 filters must be re-gross weighed.

8.9.5 Document Gross weights:

8.9.5.1 After Gross QA weighings have been performed, select the daily run file tab at the bottom of the window entitled Log.

8.9.5.2 Set the print area to print both the gross and the reweight data, then print.

8.9.5.3 ^{Physically gross to IS} Count the number of filters weighed and write the total number of filters weighed in that weigh set in the right-hand margin of the printout.

8.9.5.4 Do not close this file manually, the FilBERT program will close this window automatically upon logging out.

8.9.5.5 By client request on a project specific basis, this printout may be affixed into a bound logbook using tape. Authenticity is attested by the analyst signing the printout across its edge underneath the taped portion of the printout's edge.

8.9.6 Transfer the data from the balance computers to the LIMS and release the data:

8.9.6.1 Open the Gross Daily Run file that contains the data that needs to be put on the LIMS

8.9.6.2 Delete the columns and rows that are not needed. The necessary columns must be in the following order: LIMS ID, Gross Weight, Temperature, Humidity, Analyst Initials, Gross Date and Gross Time. Save the file as gross810.csv in the worklist directory.

8.9.6.3 Import and save the data to the LIMS per SOP AD-007.

8.9.6.4 Write the worklist number at the top of the printout and request another analyst perform a QC peer review.

8.9.7 ^{QC} Peer review data as per section 8.4.

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8.10 Chemical analysis of filters: If the filters are to be further analyzed for chemical composition, transfer them to the appropriate sample staging area and notify the pertinent Technical

Director.

8.11 Storage of filters:

- 8.11.1 When the filters are completely analyzed per the client's request, CLN will store the filters for an amount of time to be determined by the client in the archive room.

9.0 QA/QC

9.1 Weighroom temperature:

- 9.1.1 Frequency: monitored continuously by the Dickson ProSeries model TP125 temperature/humidity monitor. This monitor continuously records real time data to a computer file through Dickson proprietary software, taking a reading every 5 minutes.
- 9.1.2 QC statistic: temperature in °C.
- 9.1.3 Control limits: 24 hour average temperature within 20 - 23 °C, with no more than a ± 2 °C change from the average of the previous 24-hour period.
- 9.1.4 Corrective action: adjust the thermostat in the gravimetry laboratory. Continue adjusting until the temperature comes within the control limits. If control limits are unattainable, notify the laboratory director for an HVAC system service call. See Section 7.1 for a detailed discussion of this QC parameter.
- 9.1.5 Note: The Dickson TP125 monitor is calibrated monthly or as needed by Chester LabNet against NIST-traceable monitor. For calibration and operation of the Dickson temperature/humidity logger, see SOP GR-018.
- 9.1.6 Note: If abnormal spikes occur during a 24 hour period prior to weighing, consult the Gravimetry Laboratory Technical Director. Some outliers may be excluded at the discretion of the Technical director.

9.2 Weighroom humidity:

- 9.2.1 Frequency: monitored continuously by the Dickson ProSeries model TP125 temperature/humidity monitor. This monitor continuously records real time data to a computer file through Dickson proprietary software, taking a reading every 5 minutes.
- 9.2.2 QC statistic: relative humidity in percent.
- 9.2.3 Control limits: 24-hour average within 30 – 40%, with no more than a $\pm 5\%$ from the average of the previous 24-hour period
- 9.2.4 Corrective action: for exceedances of the upper control limit, turn on one, two or three of the de-humidifiers in the gravimetry laboratory. For exceedances of the lower control limit, fill and turn on the humidifier in the gravimetry laboratory. Do not attempt to adjust the wall-mounted humidistat, which is pre-set by the HVAC service company. Continue the use of the humidifiers/dehumidifiers until the humidity is within control limits. Periodically check that the humidifiers have water and that the water reservoirs in the dehumidifiers still have capacity. The latter have automatic cutoff sensors that will turn the units off when the reservoirs are full. See Section 7.1 for a detailed discussion of this QC parameter.
- 9.2.5 Note: If abnormal spikes occur during a 24 hour period prior to weighing, consult the Gravimetry Laboratory Technical Director. Some outliers may be excluded at the discretion of the Technical director.

9.3 Daily Balance Calibration:

- 9.3.1 Frequency: daily at the beginning of the workday.
- 9.3.2 QC statistic: mass of an ASTM Class 1 calibration verification weight (100.0000 grams).
- 9.3.3 Control limits: the weight must register 100.0000g
- 9.3.4 Corrective Action: re-tare and recalibrate the balance as follows: clean the balance stage by blowing air across it with a squeeze bulb. Wipe down the floor and inside walls of the balance with a Kimwipe moistened with 95% ethanol. Re-tare the balance. If the balance is still out of control limits, first check the bubble level at the back of the balance. If the balance is not level, level it using the thumb wheels attached to the feet. Recalibrate. If the balance fails calibration again, notify the Gravimetry Laboratory Technical Director for further action.

9.4 Monthly Balance Calibration

- 9.4.1 Frequency: once per month
- 9.4.2 QC statistic: masses of NIST traceable 3.0000g and 5.0000g weights.
- 9.4.3 Control limits: $3.0000g \pm 0.0005g$, $5.0000g \pm 0.0005g$
- 9.4.4 Corrective Action: re-tare and recalibrate the balance as follows: clean the balance stage by blowing air across it with a squeeze bulb. Wipe down the floor and inside walls of the balance with a Kimwipe moistened with 95% ethanol. Re-tare the balance. If the balance is still out of control limits, first check the bubble level at the back of the balance. If the balance is not level, level it using the thumb wheels attached to the feet. Recalibrate. If the balance fails calibration again, notify the Gravimetry Laboratory Technical Director for further action.

9.5 Balance Calibration verification:

- 9.5.1 Frequency: daily, immediately after balance calibration (Section 9.3, above)
- 9.5.2 QC statistic: masses of NIST traceable weights (3.0000g and 5.0000g).
- 9.5.3 Control limits: the average of three weighings must be within $\pm 0.5mg$ of the certified mass (per QA Guidance document 2.11).
- 9.5.4 Corrective action: re-tare and recalibrate the balance as follows: clean the balance stage by blowing air across it with a squeeze bulb. Wipe down the floor and inside walls of the balance with a Kimwipe moistened with 95% ethanol. Re-tare the balance. If the balance is still out of control limits, first check the bubble level at the back of the balance. If the balance is not level, level it using the thumb wheels attached to the feet. Recalibrate. If the balance fails calibration again, notify the Gravimetry Laboratory Technical Director for further action.

9.6 Demonstration of constant weight:

- 9.6.1 Frequency: 20% of the filters in the weigh set are reweighed at least 20 hours after the initial weighing. Filters must be held in the temperature and humidity controlled gravimetry laboratory.
- 9.6.2 QC statistic: change in filter mass.
- 9.6.3 Control limits: the tare reweights must be within $\pm 0.0010\text{g}$ of the initial tare masses, and the gross reweights must be within $\pm 0.0020\text{g}$ of the initial gross masses (per QA guidance document 2.11).
- 9.6.4 Corrective action: all of the filters in the batch must be reweighed and held for an additional 20 hours in the gravimetry laboratory. 20% of the filter batch is weighed again. If control limits are exceeded a second time, all of the filters in the batch are reweighed for a third time and held for an additional 20 hours in the gravimetry laboratory. 20% of the filter batch is weighed again. If control limits are exceeded, the project manager notifies the client that constant weight cannot be achieved using normal equilibration methods. Corrective action at this point depends upon instructions from the client and can include: averaging of weights; taking the most recent weights as final; extended equilibration period with a reweighing occurring only at the end of the period (i.e., no daily weighings during the period); etc.

9.7 QC Peer-review: ^{5/13-6-15}

- 9.7.1 Frequency: every weigh set printout.
- 9.7.2 QC statistic: data accuracy
- 9.7.3 Control limits: all data present and accurate on printouts
- 9.7.4 Corrective action: locate and correct errors found, reweighing filters if necessary.

9.8 Computer controlled weighing process

- 9.8.1 The computer receives data from the balance via a general-purpose, RS232 port on a two-second cycle. The program accumulates readings and calculates the differences between successive readings.
- 9.8.2 The computer continues to take readings until six consecutive readings agree exactly to a tenth of a milligram. This weight is then displayed as the result. See Analyst Note 13.1.

9.9 Balance servicing and third party balance and weight verification:

- 9.9.1 The balance is serviced and the calibration is verified by an outside service company on an annual basis (currently during the month September). Upon satisfactory calibration verification, the laboratory is issued a certificate, which is placed in the Balance & Weights Certificates binder in the gravimetry laboratory. Previous certificates are archived in a three-ring binder kept in the gravimetry laboratory.
- 9.9.2 The ASTM Class 1 weights are verified for mass on an annual basis by the same outside service company. Upon satisfactory verification, the laboratory is issued a certificate for each weight, which is placed in the balance and weights certificates ring binder kept in the gravimetry laboratory.

9.10 Records of environmental conditions:

- 9.10.1 The temperature and humidity in the gravimetry are continuously monitored, seven days a week, by the Dickson TP125 monitor. The data is downloaded once per week, and the monitor memory is cleared to store the following week's data.
- 9.10.2 On the last day of the weighing week, after all weighings are complete, download the current week's data to the gravimetry computer (/Documents/Filbert/Dickson) and save the file by the year/month/dates.
- 9.10.3 The chart is printed out after saving and stored in the Temperature and Humidity Graphs three-ring binder located in the Weighroom.
- 9.10.4 For operation and calibration of the Dickson monitor, refer to SOP GR-018.

9.11 Anti-static ionizing units:

- 9.11.1 Four Polonium anti-static units are placed on the floor of the Sartorius balance, equidistant around the weighing stage.

10.0 Calculations

10.1 Net deposit mass is reported in µg and is calculated by the LIMS using the following algorithm:

$$\text{net deposit mass} = \text{gross weight} - \text{tare weight}$$

where both the gross weight and tare weight data is imported into the LIMS from the files generated by the balance software.

11.0 References

- 11.1 Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II: "Ambient Air Specific Methods," Section 2.11: "PM10 High Volume"
- 11.2 Inorganics Air Compendium Method IO-2.1: "Sampling of Ambient Air for Total Suspended Particulate Matter (SPM) and PM10 using High Volume (HV) Sampler"
- 11.3 Inorganics Air Compendium Method IO-3.1: "Selection, Preparation, and Extraction of Filter Material"
- 11.4 Sartorius Balance operators manual
- 11.5 40 CFR 50, Appendix B: "Reference Method for the Determination of Suspended Particulate Matter in the Atmosphere (High Volume Method)"
- 11.6 40 CFR 50, Appendix J: "Reference Method for the Determination of Particulate Matter as PM₁₀ in the Atmosphere"
- 11.7 FiLBERT Operators manual version 1.23

12.0 Definitions

- 12.1 Analyst: the designated individual who performs the “hands-on” method and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.
- 12.2 Analysts' Notes: non-essential aspects of a method, which may help the analyst during some phase of the method. Notes may include, but not be limited to, historical aspects of the method, “tricks” of the method, unexpected issues to be aware of, or other facts or opinions related to the method, but not directly part of the procedure.
- 12.3 Blank: a clean aliquot of the same matrix as the digested samples. A blank is subjected to the usual analytical and measurement processes.
- 12.3.1 Calibration Blank: for gravimetric purposes, the calibration blank consists of taring the balance with nothing on the stage.
- 12.3.2 Field Blank: a blank prepared by the client in the field. This blank is treated as a sample by the laboratory.
- 12.4 Calculations (Data Reduction): the mathematical process of transforming raw data into a more useable form.
- 12.5 Calibrate: to determine, by measurement or comparison with a standard, the correct value of each reading of the instrument.
- 12.6 Calibration Standard: a substance or reference material used to calibrate and instrument.
- 12.7 Control Limit: a mathematical representation of acceptable limits for a given Quality Control Metric such as percent recovery or percent difference. Limits may be in the form of an absolute number or represented as a percentage.
- 12.8 Corrective Action: the action taken to address and/or eliminate where possible the causes of a nonconformity, such as exceeding a control limit. Actions may include reanalyzing a sample, or noting the non-conformance in the data report.
- 12.9 Frequency: the number of occurrences of a specified event within a given interval. The number of samples or analytical runs with which a given QC sample or metric must be analyzed or verified.
- 12.10 Laboratory Information Management System (LIMS): a comprehensive computerized database system that a laboratory uses for sample tracking and data management, from sample receipt to reporting and archiving.
- 12.11 Matrix/Matrices: the component or substrate that contains the analyte of interest.
- 12.12 QA/QC: Quality Assurance/Quality Control. A series of samples or metrics designed to show precision, accuracy and bias of the procedure are within acceptable limits.

- 12.13 QC Statistic: any of a number of statistical permutations performed on raw data to generate a metric capable of being subjected to control limits and corrective actions.
- 12.14 Reagent: a single chemical or combination of chemicals or a chemical solution used in the preparation or analysis of samples.
- 12.15 Standard: a solution or matrix of a known amount of analyte(s).
 - 12.15.1 Primary standard: a standard received from a vendor with NIST or equivalent traceability. For the purposes of gravimetric analysis, this will consist of a weight of known mass.

13.0 Analysts' Notes

- 13.1 Aside from problems due to analyst inattention, such as the balance side door or top door remaining open or a filter touching the inside of the balance chamber, the weighing of filters proceeds rapidly. For tare weighing, the laboratory has experienced slow weighing only for 8x10" cellulose filters, which are used very rarely.
- 13.2 While Tare weighing filters, keep the aspiration bulb near the balance. Prior to weighing, reexamine the filter and blow off any particles which may have remained or been redeposited on the filter during the inspection process.
- 13.3 When checking the temperature/humidity on a daily basis, also check the humidifiers/dehumidifiers. Empty the dehumidifiers, and fill the humidifiers, each morning and evening while in use.
- 13.4 If an incorrect filter number is unintentionally stamped onto a filter, it is acceptable to line out the incorrect number with a sharpie and restamp the filter with the correct number.

Appendix A: Deviations from Promulgated Methods

A.1: 40 CFR Part 50, Appendix B

Item	Promulgated requirement	SOP	Justification
1	7.9 Filter conditioning environment 7.9.1 Controlled temperature: between 15° and 30°C with less than ±3 °C variation during equilibration period.	The 24 hour average temperature must be within 20-23 ± 2°C	More stringent QC than promulgated method.
2	7.9 Filter conditioning environment 7.9.2 Controlled humidity: Less than 50 percent relative humidity, constant within ±5 percent	The 24 hour average relative humidity must be within 30-40 ± 5%	More stringent QC than promulgated method.
3	Not in promulgated method.	20 hours after tare weight is done, a QA reweight is done on 20% of the filters weighed. Filters must fall within ± 0.0010g of the initial tare weight to pass QC. If any filter fails this check, the entire set of 10 filters in between Calibration checks must be re-tare weighed.	More stringent QC than promulgated method.
4	Not in promulgated method.	20 hours after gross weight is done, a QA reweight is done on 20% of the filters weighed. Filters must fall within ±0.0020 g of the initial gross weight to pass QC. If any filter fails this check, the entire set of 10 filters in between Calibration checks must be re-gross weighed.	More stringent QC than promulgated method.

<u>Item</u>	<u>Promulgated requirement</u>	<u>SOP</u>	<u>Justification</u>
5	Not in promulgated method.	Filters are stored in third-cut manila folder that is lined with a glassine envelope.	<p>Glassine is not prone to static and is a smooth surface which is less likely to cause filter fiber or sample deposit loss during shipping.</p> <p>Manila folders are rigid enough to prevent filter bending which could also lead to filter fiber or sample deposit loss during shipping.</p>
6	Not in promulgated method.	<p>The balance used is calibrated at the beginning of every day, and the calibration is immediately checked by weighing two different weights three times each. These weights are recorded and monitored in a control chart to track long term changes.</p> <p>Before weighing a set of filters the calibration is checked again as well as after every 10 filters and at the end of a weighing session.</p>	More stringent QC than promulgated method.

A.2: Appendix J to 40 CFR, Part 50

Item	Promulgated requirement	SOP	Justification
1	7.4 Filter conditioning environment 7.4.1 Temperature range: 15 to 30C 7.4.2 Temperature control: ± 3 C	The 24 hour average temperature must be within $20-23 \pm 2^{\circ}\text{C}$	More stringent QC than promulgated method.
2	7.4 Filter conditioning environment 7.4.3 Humidity range: 20% to 45% RH 7.4.4 Humidity control: $\pm 5\%$ RH.	The 24 hour average relative humidity must be within $30-40 \pm 5\%$	More stringent QC than promulgated method.
3	Not in promulgated method.	20 hours after tare weight is done, a QA reweight is done on 20% of the filters weighed. Filters must fall within $\pm 0.0010\text{g}$ of the initial tare weight to pass QC. If any filter fails this check, the entire set of 10 filters in between Calibration checks must be re-tare weighed.	More stringent QC than promulgated method.
4	Not in promulgated method.	20 hours after gross weight is done, a QA reweight is done on 20% of the filters weighed. Filters must fall within $\pm 0.002\text{g}$ of the initial gross weight to pass QC. If any filter fails this check, the entire set of 10 filters in between Calibration checks must be re-gross weighed.	More stringent QC than promulgated method.

<u>Item</u>	<u>Promulgated requirement</u>	<u>SOP</u>	<u>Justification</u>
5	Not in promulgated method.	Filters are stored in third-cut manila folder that is lined with a glassine envelope.	<p>Glassine is not prone to static and is a smooth surface which is less likely to cause filter fiber or sample deposit loss during shipping.</p> <p>Manila folders are rigid enough to prevent filter bending which could also lead to filter fiber or sample deposit loss during shipping.</p>
6	Not in promulgated method.	<p>The balance used is calibrated at the beginning of every day, and the calibration is immediately checked by weighing two different weights three times each. These weights are recorded and monitored in a control chart to track long term changes.</p> <p>Before weighing a set of filters the calibration is checked again as well as every 10 filters and at the end of a weighing session.</p>	More stringent QC than promulgated method.

A.3: Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II:
 “Ambient Air Specific Methods,” 2.11: “PM₁₀ High Volume”

Item	Promulgated requirement	SOP	Justification
1	1.2.3 Laboratory Equipment Temperature and RH readings must be recorded daily, either manually or by hygrothermograph. Among the options available to ensure compliance with the reference method specifications are a sling psychrometer and a calibrated precision thermometer (capable of measuring temperatures over a range of 10 to 30C [283 to 303 K] to the nearest 0.1 C). This thermometer should be traceable with an accuracy of 0.1C to a NIST-certified thermometer or an ASTM thermometer.	Temperature and RH readings are recorded at five minute intervals 24 hours per day. Data points are recorded using a data logger that has been calibrated against a NIST-traceable thermometer/hygrometer.	More stringent QC than promulgated method.
2	1.2.4 Mass Reference Standards Mass reference standards must be certified as being traceable to NIST mass standards. Additionally, they must have an individual tolerance of no more than 0.025 mg.	Reference standards are NIST traceable and meet the tolerance specifications of ASTM Class 1. Individual tolerance of current reference standards is greater than 0.025mg (0.034mg).	ANSI/ASTM & NIST Metric Weight Tolerances of 3g and 5g weights are ±0.034mg.
3	1.2.4 Mass Reference Standards Laboratory primary standards should be handled very carefully and should be kept in a locked compartment.	Laboratory primary standards are stored on a shelf in the Weighroom, not in a locked compartment.	The laboratory's facilities are locked 24 hours/day. Visitors are not allowed unsupervised access to the laboratory, therefore, only authorized users have access to the standard weights.

Item	Promulgated requirement	SOP	Justification
4	<p>1.2.4 Mass Reference Standards Always use smooth, nonmetallic forceps for handling mass reference standards. The standards are handled only with these forceps, which are not used for any other purpose. Mark these forceps to distinguish them from the forceps used to handle permeation devices.</p>	<p>Forceps are not marked to distinguish for specific use.</p>	<p>Forceps used for standards are Teflon tipped while forceps used to handle filters are stainless steel. These two very different styles of forceps are easily distinguishable.</p> <p>All Weighroom employees are trained and aware that the Teflon tipped forceps are for standard weight handling purposes only.</p>
5	<p>3.4.2 Laboratory Validation Criteria The sample custodian at the analytical laboratory is responsible for conducting a secondary check of a sample's validity. Do not discard a sample that fails to meet these criteria; instead, forward it to the laboratory supervisor, who will make the final decision of a sample's validity.</p>	<p>In determining the validity of a sample, the Project Manager will contact the client for instruction. Samples are never discarded without the client's permission</p>	<p>Clarification of promulgated method.</p>
6	<p>3.4.2 Laboratory Validation Criteria The custodian should be able to decide well in advance (by the increased fuzziness of the sample outline) when to change the gasket before total gasket failure results. If signs of leakage are observed, void the sample, determine the cause, and instruct the operator to take corrective actions before starting another sampling period.</p>	<p>In determining the validity of a sample, the Project Manager will contact the client for instruction. Samples are never discarded without the client's permission</p>	<p>The laboratory is not responsible for the actions or inactions of the client in the field (e.g. changing gaskets).</p> <p>Clarification of promulgated method.</p>

Item	Promulgated requirement	SOP	Justification
7	<p>3.4.2 Laboratory Validation Criteria Check the exposed filter for physical damage that may have occurred during or after sampling. Physical damage after sampling would not invalidate the sample if all pieces of the filter were put in the folder; however, complete losses of loose particulate after sampling (e.g., loss when folding the filter) would void the sample. Mark such samples as "void" on the HV PM10 data sheet.</p>	<p>In determining the validity of a sample, the Project Manager will contact the client for instruction. Samples are never discarded without the client's permission</p>	<p>The laboratory is not responsible for the actions or inactions of the client in the field.</p> <p>Clarification of promulgated method.</p>
8	<p>3.4.2 Laboratory Validation Criteria Check the appearance of the particles. Any changes from normal color may indicate new emission sources or construction activity in the area. Note any change on the data sheet.</p>	<p>Filter anomalies are noted in LIMS, however, sample deposit composition is not the laboratory's responsibility.</p>	<p>The laboratory is not responsible for the actions or inactions of the client in the field.</p> <p>Clarification of promulgated method.</p>
9	<p>4.1 Filter Handling The filters should be separated by a sheet of 8-1/2- x 11-in. tracing paper.</p>	<p>Filters are stored in third-cut manila folder that is lined with a glassine envelope.</p>	<p>Glassine is not prone to static and is a smooth surface which is less likely to cause filter fiber or sample deposit loss during shipping.</p> <p>Manila folders are rigid enough to prevent filter bending which could also lead to filter fiber or sample deposit loss during shipping.</p>

Item	Promulgated requirement	SOP	Justification
10	Table 4-2 Visual Check: Visually Inspect each filter. Make sure there are no defects. Discard filter if requirements are not met.	Filters that do not pass inspection are reserved for extraction/digestion lot specific QC.	<p>The laboratory uses filters that failed inspection for purposes of Method Blank and Laboratory Control Sample (Fortified Blank) analysis. These filters are considered imperfect but not contaminated.</p> <p>Filters that failed inspection due to discoloration or other possible reasons that might indicate contamination are discarded.</p>
11	Table 4-2 Equilibrate in controlled environment for 24hr. RH between 20 and 45%, with ± 5 prevariation; and temperature between 15-30°C with ± 3 °C variation.	The 24 hour average temperature must be within 20-23 ± 2 °C and relative humidity within 30-40 ± 5 %	More stringent QC than promulgated method.
12	Table 4-2 Make sure there are no pinholes, tears, etc.; complete documentation; make sure there is no evidence of malfunction or sample loss; Void the affected samples; report to supervisor.	In determining the validity of a sample, the Project Manager will contact the client for instruction. Samples are never discarded without the client's permission	<p>The laboratory is not responsible for the actions or inactions of the client in the field.</p> <p>Clarification of promulgated method.</p>
13	Table 4-2 Tare weighing procedure: Begin weighing the filter within 30s after removal from the equilibration chamber. (Referenced in table 4-2)	The entire gravimetry laboratory is maintained within 20-23 ± 2 °C and 30-40% ± 5 % relative humidity. This is the environment in which the filters are stored, equilibrated, tare weighed, and gross weighed.	<p>More stringent QC than promulgated method.</p> <p>Maintaining the entire room at constant temperature/humidity, rather than having an "equilibration chamber" ensures that the filters are never handled outside of the temperature/humidity control limits and decreases the likelihood of errors due to differences between equilibration conditions and conditions at the balance.</p>

Item	Promulgated requirement	SOP	Justification
14	Table 4-2 Gross weighing procedure: Begin weighing the filter within 30s after removal from the equilibration chamber. (Referenced in table 4-2)	The entire gravimetry laboratory is maintained within $20-23 \pm 2^{\circ}\text{C}$ and $30-40 \pm 5\%$ relative humidity. This is the environment in which the filters are stored, equilibrated, tare weighed, and gross weighed.	<p>More stringent QC than promulgated method.</p> <p>Maintaining the entire room at constant temperature/humidity, rather than having an "equilibration chamber" ensures that the filters are never handled outside of the temperature/humidity control limits and decreases the likelihood of errors due to differences between equilibration conditions and conditions at the balance.</p>
15	4.3 Filter Equilibration: Temperature should be held constant with a mean value between 15 and 30 C, with a variability of not more than ± 3 C. (Referenced in table 4-2)	The 24 hour average temperature must be within $20-23 \pm 2^{\circ}\text{C}$ and relative humidity within $30-40\% \pm 5\%$	More stringent QC than promulgated method.
16	4.3 Filter Equilibration: Relative humidity (RH) should be held constant at a mean value between 20 and 45 percent, with a variability of not more than ± 5 percent. (Referenced in table 4-2)	The 24 hour average temperature must be within $20-23 \pm 2^{\circ}\text{C}$ and relative humidity within $30-40\% \pm 5\%$	More stringent QC than promulgated method.
17	4.4 Initial Weighing Procedures (Tare Weight) Enough filters to last for at least a 1-month sampling period should be numbered and weighed at one time.	Filters are inspected and weighed according to work load and inventory. Often filters are weighed over several days to build stock reserves.	<p>The laboratory is not responsible for the actions or inactions of the client in the field. Some clients may request weighed filters the day prior to the beginning of sampling, thus, the laboratory must have enough Tare weighed filters available to handle last minute requests.</p> <p>Clarification of promulgated method.</p>

Item	Promulgated requirement	SOP	Justification
18	<p>4.4 Initial Weighing Procedures (Tare Weight)</p> <p>5. Place the tared filter, with the filter ID number facing upward, in its original container or a comparably sized box. Place a sheet of 8-1/2- x 11-in. tracing paper between each filter.</p>	<p>Filters are stored in third-cut manila folder that is lined with a glassine envelope.</p>	<p>Glassine is not prone to static and is a smooth surface which is less likely to cause filter fiber or sample deposit loss during shipping.</p> <p>Manila folders are rigid enough to prevent filter bending which could also lead to filter fiber or sample deposit loss during shipping.</p>
19	<p>4.5.2 Zero and Calibration Checks After every 5 to 10 weighings, the operator should recheck the balance zero and record these check values on the Internal Quality Control Log Sheet. Zero QC checks within ± 0.5 mg of true zero are acceptable. The calibration of the balance must be checked at least daily and possibly after every 15 filters unless laboratory records indicate that the balance is stable over longer periods of time.</p>	<p>After every 10 weighings the operator checks and records that the balance returns to zero (0.0000g).</p> <p>A 3g and 5g standard weight is measured and must read within $\pm 0.0005g$ of true value.</p> <p>The balance is calibrated once per day of use and checked after every 10 weighings.</p>	<p>More stringent QC than promulgated method.</p>
20	<p>4.5.4 QC Supervisor Duties The QC supervisor must certify the acceptability of all filter weights and recorded QC data daily.</p>	<p>The QA Officer or designated alternate does a QC check/review of data before any data is reported/released on to LIMS.</p>	<p>Clarification of promulgated method.</p>

Item	Promulgated requirement	SOP	Justification
21	<p>4.5.3 Tare and Gross Weight Checks: On each day of operation, the operator should reweigh five to seven exposed and unexposed filters per balance. ... Because of the loss of volatile components, no definitive limits are set for exposed filters; however, if the difference exceeds ± 5.0 mg, the laboratory QC supervisor should investigate immediately.</p>	<p>20 hours after gross weight is done, a QA reweight is done on 20% of the filters weighed. Filters must fall within ± 0.0020g of the initial gross weight to pass QC. If any filter fails this check, the entire set of 10 filters in between Calibration checks must be re-gross weighed.</p>	<p>More stringent QC than promulgated method.</p>
22	<p>4.6 Postsampling Documentation and Inspection 2. If the exposed filter was packaged for shipment, remove the filter from its protective envelope and examine the shipping envelope. If sample material has been dislodged from a filter, recover as much as possible by brushing it from the envelope onto the deposit on the filter with a soft camel-hair brush.</p>	<p>Brushes are not used on loose deposit. Sample may be carefully poured/shaken onto deposit. Loose deposit is annotated in the LIMS and notations are reported to the client.</p>	<p>Brushes may cause positive or negative contamination for further analyses of filter.</p>
23	<p>4.6 Postsampling Documentation and Inspection 4. If insects are embedded in the sample deposit, remove them with Teflon-tipped tweezers and disturb as little of the sample deposit as possible. If more than 10 insects are observed, refer the sample to the supervisor for a decision on acceptance or rejection of the filter for analysis.</p>	<p>Insects on deposit are noted in notes to client. Unless large enough to easily remove without disrupting the deposit, insects are left to be weighed with the filter. The client is contacted. Clients make the ultimate decision regarding rejection of filter for analysis.</p>	<p>Clarification of promulgated method. The decision to accept or reject filters is up to the client.</p>

Item	Promulgated requirement	SOP	Justification
24	<p>4.6 Postsampling Documentation and Inspection 6. Place defective filters, with the type of defect(s) listed, in separate clean envelopes, label the envelopes, and submit them to the laboratory supervisor for final approval of filter validity.</p>	<p>In determining the validity of a sample, the Project Manager will contact the client for instruction. All post-sampled filters that are shipped to CLN are weighed. Defects are documented in notes to the client.</p>	<p>Clarification of promulgated method.</p> <p>The decision to accept or reject filters is up to the client.</p>
25	<p>4.7 Final Weighing Procedure (Gross Weight 5. If the filter is to receive further analysis, place it in a protective covering and note on the envelope or folder what additional analyses are required. Place an asterisk after the gross weight column on the Laboratory Data Form to indicate that the filter requires additional analysis. Carefully transport each packaged filter to the sample custodian, who will forward it to the laboratory responsible for the additional analyses.</p>	<p>Due to the small staffing levels, it is not necessary to return filters to the sample custodian. Filters that are to receive further analysis are kept with QC paperwork until the QC process has been completed. Post-QC the filters will be placed in the staging area for the next analysis.</p>	<p>Clarification of promulgated method.</p>
26	<p>4.8 Calculation of PM10 Net Filter Loading The gross weight minus the tare weight of an HV PM10 filter is the net weight of PM10 for that filter. Each calculation of this process must be independently validated. Refer to Section 5.0 for information regarding the calculation of PM10 mass concentration.</p>	<p>Each calculation is performed by computer software.</p>	<p>More stringent QC than promulgated method.</p> <p>Automating this calculation is more accurate than human calculation.</p>

<u>Item</u>	<u>Promulgated requirement</u>	<u>SOP</u>	<u>Justification</u>
27	Not in promulgated method.	<p>The balance used is calibrated at the beginning of every day, and the calibration is immediately checked by weighing two different weights three times each. These weights are recorded and monitored in a control chart to track long term changes.</p> <p>Before weighing a set of filters the calibration is checked again as well as every 10 filters and at the end of a weighing session.</p>	<p>More stringent QC than promulgated method.</p> <p>This helps ensure the balance is maintaining its calibration and that there are no changes during the weighing session.</p>

A.4: Compendium Method IO-2.1: "Sampling of Ambient Air for Total Suspended Particulate Matter (SPM)

Note: Compendium Method IO-2.1 references Compendium Method IO-3.1

Item	Promulgated requirement	SOP	Justification
1	<p>8.2.2 Filter Handling For convenience, filters can be packed in groups of 50 or less in their original containers or in a box of comparable size. The filters should be separated by a sheet of 8 ½ x 11" tracing paper. Filter inventory can be controlled by stacking the filters in numerical order so that the operator will use the proper filter first. One side of the shipping box can be cut away to allow the operator to remove the filter easily without damaging the corners.</p>	<p>Filters are stored in third-cut manila folder that is lined with a glassine envelope.</p> <p>Filter inventory is kept in order in a filing cabinet with filter ID stamped on envelope and filter.</p> <p>Filters are sent to client in labeled folders packed in appropriate sized boxes.</p>	<p>Glassine is not prone to static and is a smooth surface which is less likely to cause filter fiber or sample deposit loss during shipping.</p> <p>Manila folders are rigid enough to prevent filter bending which could also lead to filter fiber or sample deposit loss during shipping.</p> <p>The laboratory is not responsible for the actions or inactions of the client in the field. Filter inventory in the field is the responsibility of the client.</p> <p>Clarification of promulgated method.</p>

Item	Promulgated requirement	SOP	Justification
2	<p>9.5.2.1 If signs of leakage are observed, void the sample, determine the cause, and instruct the operator to take corrective actions before starting another sampling period.</p> <p>9.5.2.2 Check the exposed filter for physical damage that may have occurred during or after sampling. Physical damage after sampling would not invalidate the sample if all pieces of the filter were put in the folder; however, complete losses of loose particulate after sampling (e.g., loss when folding the filter) would void the sample. Mark such samples as "void" on the HV data sheet</p>	<p>In determining the validity of a sample, the Project Manager will contact the client for instruction. Samples are never discarded without the client's permission</p>	<p>The laboratory is not responsible for the actions or inactions of the client in the field (e.g. changing gaskets).</p> <p>Clarification of promulgated method.</p>
3	<p>9.5.3.2.2 If the exposed filter was packaged for shipment, remove the filter from its protective envelope and examine the shipping envelope. If sample material has been dislodged from a filter, recover as much as possible by brushing it from the envelope onto the deposit on the filter with a soft camel's-hair brush.</p>	<p>Brushes are not used on loose deposit. Sample may be carefully poured/shaken onto deposit. Loose deposit is annotated in the LIMS and notations are reported to the client.</p>	<p>Brushes may cause positive or negative contamination for further analyses of filter.</p>

Item	Promulgated requirement	SOP	Justification
4	<p>9.5.3.4.2 If insects are embedded in the sample deposit, remove them with Teflon®-tipped tweezers and disturb as little of the sample deposit as possible. If more than 10 insects are observed, refer the sample to the supervisor for a decision on acceptance or rejection of the filter for analysis.</p>	<p>Insects on deposit are noted in notes to client. Unless large enough to easily remove without disrupting the deposit, insects are left to be weighed with the filter. The client is contacted. Clients make the ultimate decision regarding rejection of filter for analysis.</p>	<p>Clarification of promulgated method.</p> <p>The decision to accept or reject filters is up to the client.</p>
5	<p>9.5.3.2.6 Place defective filters, with the type of defect(s) listed, in separate clean envelopes. Label the envelopes and submit them to the laboratory supervisor for final approval of filter validity.</p>	<p>Filters that do not pass inspection are reserved for extraction/digestion lot specific QC.</p>	<p>The laboratory uses filters that failed inspection for purposes of Method Blank and Laboratory Control Sample (Fortified Blank) analysis. These filters are considered imperfect but not contaminated.</p> <p>Filters that failed inspection due to discoloration or other possible reasons that might indicate contamination are discarded.</p>

A.5: PM₁₀ using High Volume (HV) Sampler” & Compendium Method IO-3.1:
 “Selection, Preparation, and Extraction of Filter Material”

Item	Promulgated requirement	SOP	Justification
1	3.1.1 Controlled Temperature. Temperature between 15 to 30°C with less than ± 2°C variation during equilibration period.	The 24 hour average temperature must be within 20-23 ± 2 °C	More stringent QC than promulgated method.
2	3.1.2 Controlled Humidity. Less than 50% relative humidity, consistent within ±5 %.	The 24 hour average relative humidity must be within 30-40% ± 5%	More stringent QC than promulgated method.
3	5.1.2 Verify that the weighing room conditions are within the limits. Filter equilibrium and weighing Should be performed under controlled atmospheric conditions-- a temperature of 25±10°C and a relative humidity <50% (normally 50±5% humidity).	The entire gravimetry laboratory is maintained within 20-23 ± 2 °C and 30-40% ± 5% relative humidity This is the environment in which the filters are stored, equilibrated, tare weighed, and gross weighed.	More stringent QC than promulgated method.
4	5.1.3 Use the results from the motorized psychrometer to verify the temperature and relative humidity indicated by the hygrothermograph. Record the psychrometer values on the strip chart, along with the date, time, and your initials.	Temperature and RH readings are recorded at five minute intervals 24 hours per day. Data points are recorded using a computer controlled data logger that has been calibrated against a NIST-traceable thermometer/hygrometer. Electronic data files are backed up daily.	More stringent QC than promulgated method. Updated technology.

5	5.1.4 Record the room equilibration data on the Weighing Room Atmospheric Condition Form (see Table 4).	Electronic records of Weighroom atmospheric condition data are kept in files on the Weighroom computer and weekly printout of graph is stored in appropriate binder. Additionally, this information is tracked in a daily logbook.	More stringent QC than promulgated method. Updated technology.
6	5.2.1.1 Filters should only be handled with finger cots or vinyl (nonpowdered) gloves. This procedure applies to filter handling in the field as well as in the weigh room.	Filters are handled with Nitrile (nonpowdered) gloves.	Clarification of promulgated method.
7	5.2.2.1 Upon receipt of new high volume filters (8" x 10" quartz fiber), take them to the climate controlled room, remove the paper and plastic envelope (wearing clean plastic gloves), place each on edge in a clean metal file rack, and cover with clean white paper towels.	Upon receipt, all filters are stored in the climate controlled gravimetry laboratory. Each filter is inspected (wearing nitrile gloves) and placed in third-cut manila folder that is lined with a glassine envelope. Both envelope and filter are given matching laboratory ID numbers. Filters are stacked on a shelf dedicated for new filters to be equilibrated before tare weighing.	Glassine is not prone to static and is a smooth surface which is less likely to cause filter fiber or sample deposit loss during shipping. Manila folders are rigid enough to prevent filter bending which could also lead to filter fiber or sample deposit loss during shipping. CLN has found that putting the filters in a glassine lined manila folder allows for the filters to stay protected and clean during the equilibration process.

<p>8</p>	<p>5.2.2.1 Upon receipt of new high volume filters (8" x 10" quartz fiber), take them to the climate controlled room, remove the paper and plastic envelope (wearing clean plastic gloves), place each on edge in a clean metal file rack, and cover with clean white paper towels.</p>	<p>Upon receipt, all filters are stored in the climate controlled gravimetry laboratory. Each filter is inspected (wearing nitrile gloves) and placed in third-cut manila folder that is lined with a glassine envelope. Both envelope and filter are given matching laboratory ID numbers. Filters are stacked on a shelf dedicated for new filters to be equilibrated before tare weighing.</p>	<p>Equilibration of filters is confirmed by tare reweight QC.</p>
<p>9</p>	<p>5.2.2.2 Allow the filters to equilibrate in the metal file rack in the weighing room atmosphere for at Least 24 h. Humidity and temperature must be within Federal Reference method specification, (i.e., <50% and 15-35E C, respectively).</p>	<p>Filters are equilibrated in boxes provided by manufacture for at least 24 hrs.</p> <p>CLN keeps the 24 hour average temperature at 20-23 ± 2°C and the 24 hour average relative humidity at 30-40% ± 5%.</p> <p>Upon receipt, all filters are stored in the climate controlled gravimetry laboratory. Each filter is inspected (wearing nitrile gloves) and placed in third-cut manila folder that is lined with a glassine envelope. Both envelope and filter are given matching laboratory ID numbers. Filters are stacked on a shelf dedicated for new filters to be equilibrated before tare weighing.</p>	<p>Equilibration is confirmed with passing tare reweight QC.</p> <p>Humidity and temperature controls are more stringent than method.</p>

10	<p>5.2.2.4 Manually calibrate the balance. However, checks against two working NIST traceable weights (Class S) standards should be conducted before the daily weighing. If the difference between the traceable weights is more than 0.5 mg, do not use the balance until it has been repaired.</p>	<p>The balance used is calibrated at the beginning of every day, and the calibration is immediately checked by weighing two different weights three times each. These weights are recorded and monitored in a control chart to track long term changes.</p> <p>Before weighing a set of filters the calibration is checked again as well as every 10 filters and at the end of a weighing session.</p>	<p>More stringent QC than promulgated method.</p> <p>This helps ensure the balance is maintaining its calibration and that there are no changes during the weighing session.</p>
11	<p>5.2.2.5 Record the results on the Weighing Balance Check Form (see Table 5). 5.2.2.6 Weigh each filter and record filter numbers and tare weights on the Filter Weighing Form (see Table 6).</p>	<p>Each recording of data from the balance and subsequent calculation is performed by computer software.</p>	<p>More stringent QC than promulgated method.</p> <p>Automating this calculation is more accurate than human calculation.</p>
12	<p>5.2.2.8 After every tenth weighing, recheck the zero of the balance. The balance response should be ± 1 mg from 0.</p>	<p>After every tenth weighing, the balance must be exactly 0.0000g ± 0.0mg.</p>	<p>More stringent QC than promulgated method.</p>
13	<p>5.2.2.8 Record the measurements on the Weighing Balance Check Form</p>	<p>Each recording of data from the balance and subsequent calculation is performed by computer software.</p>	<p>More stringent QC than promulgated method.</p> <p>Automating this calculation is more accurate than human calculation.</p>

<p>14</p>	<p>5.2.2.9 A second analyst should [tare] reweigh 10% of the filters. If the difference between the weights is less than 1.0 mg, the results are acceptable</p>	<p>The laboratory does not have a second analyst reweigh the filters, however, a second analyst does QC the data. Additionally, reweights are preformed at a ≥ 20 hrs after original tare weighing.</p> <p>CLN requires 20% reweight with a difference less than 0.0010g for tare.</p>	<p>CLN uses proprietary software to record the mass of the filters to avoid analyst error or bias. Reweights are performed 20 hours after original weighing of filters. Additionally, a second analyst QC the data to verify the masses and QA. Automating calculations is more accurate than human calculation.</p> <p>It is the laboratories opinion that CLN system is more stringent than the promulgated method.</p>
<p>15</p>	<p>5.2.3.4 For filters not to be analyzed, put an asterisk in the space preceding the four-letter code. Leave this space blank for samples to be analyzed. Sign and date the forms. 5.2.3.5 Archive asterisked high volume filters.</p>	<p>Due to the small staffing levels, it is not necessary to return filters to the sample custodian. Filters that are to receive further analysis are kept with QC paperwork until the QC process has been completed. Post-QC the filters will be placed in the staging area for the next analysis.</p> <p>After all requested analyses have been completed, filters are placed in the filter archive area to be stored by Weighroom personel.</p>	<p>CLN has staging areas for certain analyses and worklists for each analysis are kept with filters at all times to ensure that all analyses requested are completed.</p> <p>Clarification of promulgated method.</p>
<p>16</p>	<p>5.2.3.6 Have a second analyst [gross] reweigh 10% of the filters and verify that the weights have not changed. If the difference between the weights is less than 2.0 mg, the results are acceptable. Use the results from the first weighing. If the difference is greater than this limit, reweigh 100% of that lot and use the last reweigh weight</p>	<p>The laboratory does not have a second analyst reweigh the filters, however, a second analyst does QC the data. Additionally, reweights are preformed at a ≥ 20 hrs after original tare weighing.</p> <p>CLN requires 20% reweight with a difference less than 0.0010g for tare.</p>	<p>CLN uses proprietary software to record the mass of the filters to avoid analyst error or bias. Reweights are performed 20 hours after original weighing of filters. Additionally, a second analyst QC the data to verify the masses and QA. Automating calculations is more accurate than human calculation.</p> <p>It is the laboratories opinion that CLN system is more stringent than the promulgated method.</p>

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Standard Operating Procedure
GR-006.07

FILTER CASSETTE LOADING AND UNLOADING
CHESTER LABNET PROPRIETARY METHOD

Approvals:

<u>Shirley Hubbard</u> Author	<u>1-14-13</u> Date
<u>Shirley</u> Lead Analyst	<u>1-10-13</u> Date
<u>Shirley Hubbard</u> QA/QC	<u>1-14-13</u> Date

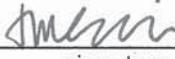
Effective from: 1-14-13
Effective until: present

REVIEW HISTORY

Review date:	Changes made:	Changes made by:
1/7/13	Updated to meet 2011 TNI Standard requirements. Minor changes to text.	Sheri Heldstab
5/20/09	Added section on washing white 47mm cassettes with Citranox. Minor clarification to text.	Sheri Heldstab
9/23/08	Clarification to text, no change to methodology	Sheri Heldstab
7/25/06	Removed section on 47mm Teflon cassettes, as water rinsing was shown to not work as intended.	Sheri Heldstab
6/10/05	Updated formatting to NELAP standards. Added in sections concerning 47mm Teflon cassettes used in Hexavalent Chromium analysis.	Sheri Heldstab
3/20/03	Removed parts referring to filter request forms as this process is now the responsibility of the project manager. Updated some formatting.	Sheri Heldstab
2/27/02	Some content added for clarification. Updated formatting.	Sheri Heldstab
1/22/96	Typographical errors corrected.	C.R. Lytle
8/2/94	New coversheet; review history page added.	Sheri Heldstab
12/2/93	No changes. Date of origination.	Sheri Heldstab

ANNUAL REVIEW

The undersigned attests that this standard operating procedure has undergone annual review for adherence to current practices and the latest QA/QC protocols:

<u></u> signature	<u>Grav. Lab. Tech. Dir.</u> title	<u>01.21.14</u> date
<u></u> signature	<u>WEIGHROOM Tech. Dir.</u> title	<u>01.28.15</u> date
<u></u> signature	<u>weighroom tech. Dir.</u> title	<u>02.09.16</u> date
_____ signature	_____ title	_____ date

FILTER CASSETTE LOADING AND UNLOADING CHESTER LABNET PROPRIETARY METHOD

1.0 Introduction

- 1.1 Test Method Reference ID: Chester LabNet proprietary method.
- 1.2 Applicability: This method is applicable to all filters loaded into sampling cassettes utilized for air quality monitoring (indoor or outdoor; ambient air and very rarely source sampling)
- 1.3 Detection Limit: not applicable.
- 1.4 Method Performance: not applicable.

2.0 Summary

- 2.1 Scope and Application: This intended use of this method is for the loading and unloading of various filter types into their respective air monitoring devices (filter cassettes). This method meets the intended use.
- 2.2 Summary of Method: The cassettes are cleaned appropriately and loaded with pre-inspected and pre-prepared filters.
- 2.3 Interferences: not applicable.
- 2.4 Sample collection/preservation/shipment/storage: Filters are received from the manufacturer or vendor and stored in the climate controlled weighroom. Assembled cassettes will also be stored in the weighroom, or under other controlled conditions based on the required methodology, until shipped to client.

2.4.1 Environmental Weighroom Controls

- 2.4.1.1 All operations involving filters will take place in the weighroom.
Operations not involving the direct weighing of filters will take place in the laminar flow hood in the weighroom. The analyst must wear gloves or

use forceps to avoid contaminating filters with oil/perspiration from fingers.

2.4.1.2 The weighroom is temperature and humidity controlled to satisfy the requirements of three specific reference methods, each with its own set of tolerances. Refer to pertinent filter inspection and gravimetry SOP for specific details.

3.0 Safety

3.1 Follow the Chester LabNet Chemical Hygiene plan. Always treat samples of unknown origin and/or constitution as hazardous.

3.2 This method presents no safety risk beyond typical laboratory safety hazards.

3.3 No carcinogenic reagents are used in this method.

4.0 Pollution Prevention and Waste Management

4.1 The smallest quantity of chemical feasible is removed from its primary container for use.

4.2 Chester LabNet is a conditionally exempt small quantity generator and as such does not require formal chemical waste processing.

4.2.1 Acidic and Basic wastes are neutralized prior to disposing of them in the sanitary sewer system.

4.2.2 Organic liquids are usually primarily used for cleaning purposes. Organic wastes are generated in very small quantities, and evaporate off with no need for more formal disposal.

5.0 Apparati, Equipment and Supplies

5.1 Gloves

5.2 Appropriate filter cassettes:

- 5.2.1 25 mm cassettes (Gelman #4376, black plastic)
- 5.2.2 37 mm cassettes (Gelman #4339, clear plastic, 3 parts)
- 5.2.3 37 mm cassettes (SKC #225-5, 2 parts)
- 5.2.4 47 mm aerosol holder (Nucleopore #430400, clear plastic, 3 parts)
- 5.2.5 47 mm PM2.5 filter rings with stainless steel support screen (white or blue rings) and secondary containers (clear plastic mailers or metal canisters)
- 5.2.6 37 mm dichot rings (yellow or white plastic rings, available from ThermoAndersen # SAFH240P)
- 5.2.7 47 mm Teflon filter cassettes with black coupling ring (Savillex #4-147-4T)

5.3 Appropriate filter cassette accessories:

- 5.3.1 37 mm, 42 mm, or 47 mm drain discs (Whatman #'s 230800, 230900, or 231100)
- 5.3.2 25 mm cellulose support pads (Gelman #66238)
- 5.3.3 37mm sealing bands (SKC #225-25)
- 5.3.4 25mm sealing bands (SKC #225-2503)
- 5.3.5 electrical tape
- 5.3.6 green 47mm Teflon cassette wrenches
- 5.3.7 end caps appropriate to each cassette type, *where applicable. 2-8-16 SH*

5.4 Cassette opener tool (SKC #225-13-5)

5.5 Appropriate-sized inspected and conditioned filters

5.6 Metal or plastic forceps

5.7 Kimwipes

~~5.8 Flathead screwdriver 1.6.14 PX~~

5.9 Cassette Separator (Airmetrics part# 600-007)

5.10 1L plastic beakers for 47mm cassette ^{1.8.14} (white ring) cleaning

5.11 Ultrasonicator capable of holding 1L plastic beakers

6.0 Reagents and Standards

6.1 Reagent grade 95% ethanol.

6.2 Citranox brand laboratory detergent.

6.3 De-Ionized Water

7.0 Preparation, Calibration and Standardization

7.1 Using ethanol and Kimwipe, clean the forceps and all work surfaces of the laminar flow hood.

7.2 Clean filter cassettes:

7.2.1 For most cassette types: Using a Kimwipe wetted with ethanol, clean any parts of the cassette which will contact either the air sample (insides of chamber etc) or any parts which will contact the filter, drain disc or support pad (filter support grating, o-rings, or other seals). Allow cassettes to dry thoroughly prior to loading.

7.2.2 For 25 mm filter cassettes:

7.2.2.1 Disassemble filter cassettes and place in large vessel such as a 1L beaker.

7.2.2.2 Fill vessel with DI water, add a small amount of Citranox detergent and sonicate for a minimum of 15 minutes.

*30
J.S. 1.8.14*

7.2.2.3 When sonication is complete, remove filter cassettes from the vessel and rinse thoroughly with DI water.

7.2.2.4 Place rinsed cassettes on a clean absorbent surface to dry. Dry at room temperature. Cassettes must be completely dry prior to loading.

7.2.3 For 47mm blue or white plastic ring cassettes:

7.2.3.1 After removal of filters from cassettes, place cassettes, including screens, in 1L plastic beakers.

7.2.3.2 Add DI water until cassettes are submerged.

7.2.3.3 Add a small amount of Citranox brand laboratory detergent to water

7.2.3.4 Sonicate for ³⁰60 minutes.
JS1.8.14

7.2.3.5 Remove from sonicator.

7.2.3.6 Individually rinse each cassette part with fresh DI water and allow to air dry until fully dry prior to reloading.

7.2.3.7 Secondary containers:

7.2.3.7.1 Mailers (clear plastic containers): clean in the same manner as the cassettes.

7.2.3.7.2 Metal canisters: wipe canister with ethanol wetted Kimwipe until Kimwipe shows no signs of dirt being removed.

7.2.3.8 Storage of cleaned cassettes/secondary containers:

7.2.3.8.1 Store cleaned cassettes in sealed Ziploc bags.

7.2.3.8.2 Store cleaned plastic mailers in Ziploc bags.

7.2.3.8.3 Store metal canisters fully assembled (e.g. with their "lids" on) in an enclosed cardboard box.

7.3 Ensure a sufficient supply of pre-inspected and, if necessary, pre-weighed filters available to be loaded into the cassettes. Refer to the appropriate SOP for filter inspection and preparation.

8.0 Procedure

8.1 Loading cassettes:

8.1.1 The project manager will inform the analyst of the details concerning the cassette type, filter type, and necessity of support rings, drain discs or other special requests.

Running #2-8-16

- 8.1.2 Perform all work in the clean laminar flow hood.
- 8.1.3 Disassemble the filter cassette.
- 8.1.4 Using clean forceps only to handle the pre-prepared filter, place the support pad, drain disc, metal screen, and/or filter onto the filter mount.
- 8.1.5 Reassemble the filter cassette. For 47mm Teflon cassettes, tighten the black coupling ring using the associated green wrenches.
- 8.1.6 For 25 mm and 37 mm cassettes, wrap each joint of the cassette with a sealing band or black electrical tape, and insert the red/blue plastic end caps into the openings of the cassette. If using sealing bands, allow bands to shrink until fully dry prior to shipping.

Note: sealing bands will loosen in the presence of moisture and may slide off the cassette. Ensure that the client understands this prior to using sealing bands. For wet conditions, electrical tape should be used.

- 8.1.7 For each cassette, affix the appropriate filter label as required by client to the outside of the cassette and secondary cassette container after the cassette has been fully assembled. Alternatively, the laboratory ID may be written directly onto the cassette using a fine tip permanent marker such as a Sharpie.

8.2 Responding to a client request for filters:

- 8.2.1 Upon client request, the project manager notifies the weighroom Technical Director, who then schedules the work based upon current workload and client delivery requirements.
- 8.2.2 After the cassettes are prepared, notify the project manager that the filter cassettes are ready for shipment.

8.3 Unloading Cassettes: Upon receipt from the field, unload the used cassettes as follows:

- 8.3.1 If necessary, log received samples in to the LIMS ID tracking log (see SOP GR-020)
- 8.3.2 Perform all filter handling inside the laminar flow hood in the weighroom.
- 8.3.3 Open the cassettes very carefully. The use of the cassette opener or cassette wrenches may be needed due to the tight seal formed by the cassette holders.
- 8.3.4 Open the cassette as slowly as possible to avoid damaging or dropping the filter.
- 8.3.5 Place the filter in a clean, labeled Petri slide, Petri dish, or extraction vessel. For dichot samples, the filter may be placed in the same Petri dish that the dichot ring was received in.
- 8.3.6 If the filters are to be gross weighed, place the filters, in their containers, on the baker's rack ^{JSI-8-14} next to the balance. The filters must be stored sedentary and exposed to the weighroom environment, either in their cassettes or after they have been unloaded from their cassettes into Petri slides, a minimum of 24 hours before gross weighing may be performed. This is to allow the filter to reach temperature and humidity equilibrium with the controlled environment of the weighroom. Refer to pertinent filter inspection and gravimetry SOP for specific details

8.4 Clean used empty cassettes prior to storing.

9.0 QA/QC

9.1 None applicable.

10.0 Calculations

10.1 None applicable

11.0 References

11.1 Various manufacturers' instruction manuals for various types of cassettes.

12.0 Definitions

12.1 Analyst: the designated individual who performs the "hands-on" method and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

12.2 Analysts' Notes: Non-essential aspects of a method, which may help the analyst during some phase of the method. Notes may include, but not be limited to, historical aspects of the method, "tricks" of the method, unexpected issues to be aware of, or other facts or opinions related to the method, but not directly part of the procedure.

12.3 Reagent: a single chemical or combination of chemicals or a chemical solution used in the preparation or analysis of samples.

13.0 Analysts' Notes

13.1 Be extremely careful when disassembling cassettes with exposed filters. It is very easy to damage exposed filters during the unloading process, either by direct physical damage or by having the filter pop out of the cassette and fall to the ground.

13.2 When loading filters that arrive from the manufacturer with a stamped ID on the support ring (e.g. 47m Teflon), double check the stamped ID against the laboratory ID for accuracy by referencing the appropriate logbook.

APPENDIX A: Differences from Promulgated methods

There is no promulgated method for ^{Cassette loading & unloading} ~~data and report validation~~. This method is a *CHESTER LabNet* proprietary method for internal use. ^{SM 1-21-14}

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Standard Operating Procedure GR-018.02

DICKSON TEMPERATURE AND HUMIDITY DATA LOGGER CHESTER LABNET PROPRIETARY METHOD

Approvals:

 _____ Author	<u>5.28.12</u> Date
 _____ Lead Analyst	<u>5.31.12</u> Date
 _____ QA/QC	<u>5.31.12</u> Date

Effective from: 5.31.12
Effective until: present

DICKSON TEMPERATURE AND HUMIDITY DATA LOGGER CHESTER LABNET PROPRIETARY METHOD

1.0 Introduction

- 1.1 Test Method Reference ID: Chester LabNet proprietary method
- 1.2 Applicability: This method is applicable to the tracking and documenting of temperature and humidity in the weighroom.
- 1.3 Detection Limit: N/A
- 1.4 Method Performance: Refer to Dickson Pro Series User's Manual (located in the XRF room, with the other manuals).

2.0 Summary

- 2.1 Scope and Application: The intended use of this method is for tracking and documenting temperature and humidity in the weighroom.
- 2.2 Summary of Method: The Dicksonware Datalogger software, in conjunction with the Dickson Pro Series TP125 thermometer/hygrometer, monitors, logs and tracks trends in the temperature and humidity of the weighroom. The Dicksonware software is calibrated monthly or as needed against an NIST traceable second source thermometer/hygrometer. A weekly graph of the recorded temperature/humidity of the weighroom is retained in both electronic and hardcopy format.
- 2.3 Interferences: Spikes (both positive and negative) in temperature or humidity may result from the weighroom door being opened or closed, or when humidifiers/dehumidifiers are emptied, or refilled. *Set 11-6-15*
- 2.4 Sample collection/preservation/shipment/storage: N/A

3.0 Safety

3.1 No safety hazards exist with this SOP beyond standard office safety hazards.

4.0 Pollution Prevention and Waste Management

4.1 Water is the only chemical utilized in this method.

5.0 Apparati, Equipment and Supplies

5.1 Dickson Pro Series PT125 thermometer/hygrometer

5.2 Dickson Datalogger software

5.3 NIST traceable thermometer/hygrometer

5.4 PC running on Windows with Excel 2000

5.5 Weighroom dedicated HVAC system with wall mounted thermostat

5.6 Humidifiers with filters

5.7 Dehumidifiers with filters *and sump pumps set 11.6.15*

6.0 Reagents and Standards

6.1 N/A

7.0 Preparation, Calibration and Standardization

7.1 The weighroom is temperature and humidity controlled to satisfy the requirements of four specific reference methods, each with its own set of tolerances:

Reference Method	Average Temperature	Average Relative Humidity
TSP (40 CFR 50, Appendix B)	15 – 30 ± 3 °C (59 – 86 ± 5 °F)	<50% ± 5%
TSP (IO 3.1)	15 – 30 ± 2 °C (59 – 86 ± 4 °F)	<50% ± 5%
PM ₁₀ (40 CFR 50, Appendix J)	15 – 30 ± 3 °C (59 – 86 ± 5 °F)	20 - 45% ± 5%
PM _{2.5} (40 CFR 50, Appendix L)	20 – 23 ± 2 °C (68 – 73 ± 4 °F)	30 - 40% ± 5%
CLN Targets	20 – 23 ± 2 °C	30 - 40% ± 5%

The Chester LabNet (CLN) targets for temperature and humidity are set at values that simultaneously satisfy all three reference methods. ^{four SH 11-12-13} Note that the ranges specified for both parameters are the maxima allowed within any given 24-hour period.

7.2 Monthly Dicksonware calibration:

- 7.2.1 Note that this calibration may also be performed if the analyst, during the daily NIST visual check, notices that the Dickson thermometer/hygrometer is out of control.
- 7.2.2 Close the Dicksonware program
- 7.2.3 Click the LoggerCal icon on the computer desktop.
- 7.2.4 Click the F/C button to change the reader to Celsius format.
- 7.2.5 Click the "read unit" button, the reading will appear in the CH#1 and CH#2 boxes, where CH#1 is the temperature and CH#2 is the humidity.
- 7.2.6 Compare the values given by the Dickson unit to the NIST traceable unit mounted on the wall. If the values are not in agreement, change the target values in the Dicksonware software to those obtained from the NIST traceable wall mounted unit.
- 7.2.7 Click the "adjust both channels" button. This will take several minutes to complete. If only one channel needs adjusting, the other channel may be left unaltered during this process.
- 7.2.8 When the unit is finished adjusting, click "exit" to close the program.
- 7.2.9 Re-open the Dicksonware software to resume operations in the weighroom.

- 7.2.10 Record the calibration ^{L information} in the electronic file "Dicksonware Log." The date, time and type (monthly or 'analyst judged') ^{is 12-2-14} is recorded. Print the file after every calibration and place it in the "Monthly Control Charts" binder.

8.0 Procedure

8.1 Daily Weighroom Temperature and Humidity Control Check

- 8.1.1 The Dickson realtime logger should be open at all times.

- 8.1.1.1 If for some reason, it has been closed, ^{select "download" from the menu bar;} follows steps 8.2.1 and 8.2.2. _(to obtain data for temp & P/H control check)

- 8.1.1.2 Save the data ^{yes/no} as "now" as in step 8.1.2 below. The software will display a dialog box asking "clear the data and restart?"; click "no". Open the real-time logger again. _{is test}

- 8.1.2 Save the realtime data to "My documents/Dickson/Now". When prompted to overwrite the previous file, click "yes".

- 8.1.3 Open the "Now" file created in step 8.1.2 in Excel, using the "all files" and "comma" parameters.

- 8.1.4 In Excel, open the Weekly Temperature file (My documents/Dickson/Weekly Temp RH).

- 8.1.4.1 If it is the first day of the recording week, open the file named "weekly temp RH" and enter the date for the first day. The remaining dates will fill in automagically. If it is not the first day of the recording week, open the file created at the beginning of that week.

8.1.4.2 Save this file in the format YYMMDD, where:

YY = year

MM = month

DD = day

April 1, 2010 would be saved as "100401".

8.1.5 Copy the pertinent information from the "Now" file for the previous 24 hours (Date, Time, Temperature and Humidity)

8.1.6 Paste this data into the Weekly Temperature file. Note: verify that the correct file is being pasted into, and that the data is being pasted into the correct date within that file.

8.1.7 Record the Averages, Maximums and Minimums in the Temperature and Humidity logbook. Verify that the room passes the 24 hour control limits as given in section 7.1.

8.1.8 If the environment is in control, weighroom operations involving balances may commence. If the room is out of control, determine at what point weighing may commence. The room must be in control for at least 24 hours before weighing can commence. If there are outliers that create an "out of control" situation, contact the weighroom technical director prior to weighing.

8.2 Weekly Dicksonware downloading and logging:

8.2.1 Close the realtime Dicksonware window. A dialog box will appear asking if the user would like to discard the data. Click "yes".

8.2.2 Select "download" from the menu bar (note: this may take a moment to open).

8.2.3 Print the graphed data on a color printer.

- 8.2.4 Save the graphed data by selecting "save" from the menu. Save the file in the subdirectory "My Documents/Dickson", using the file naming format of "Downloaded Data-YYYY-MM-D1-D2", where:

YYYY = year

MM = month

D1 = date of last download

D2 = date of current download

If the data is for more than one full month, save the file in the subdirectory "My Documents/Dickson", using the file naming format of "Downloaded Data-YYYY-M1-D1-M2-D2", where:

YYYY = year

MM = month

D1 = date of last download

D2 = date of current download

M1 = month 1

M2 = month 2

- 8.2.5 After the file has been saved, Dicksonware will prompt the user to restart and clear the datalogger. Do so if the file has been saved as described in section 8.2.4.
- 8.2.6 Immediately reopen the realtime datalogger.
- 8.2.7 Place the printout in the most current Dickson Temperature/Humidity 3-ring binder in the weighroom.

9.0 QA/QC

- 9.1 The Dickson thermometer/hygrometer has no specific QC associated with it, aside from the monthly calibration as described in section 7.2. The following QC relate to the weighroom itself.

9.2 Weighroom temperature:

- 9.2.1 Frequency: monitored continuously by a Dickson combination thermometer/hygrometer.
- 9.2.2 QC statistic: temperature in °C.
- 9.2.3 Control limits: 24-hour average of 20 – 23 °C, with no more than a ± 2 degree change from the average occurring over any 24-hour period.
- 9.2.4 Corrective action: Contact the Weighroom Coordinator. Adjust the thermostat on the south wall of the weighroom. Wait at least two hours for temperature to stabilize. Continue adjusting until the temperature comes within the control limits. If control limits are unattainable, notify the laboratory director for an HVAC system service call.
- 9.2.5 Note: The thermometer/hygrometer is calibrated against an NIST-traceable instrument. The current certificate is stored in a 3-ring notebook with weight certifications in the weighroom.

9.3 Weighroom humidity:

- 9.3.1 Frequency: monitored continuously by a Dickson combination thermometer/hygrometer.
- 9.3.2 QC statistic: relative humidity in percent.
- 9.3.3 Control limits: 24-hour average of 30 – 40 %, with no more than a ± 5 percent change from the average occurring over any 24-hour period
- 9.3.4 Corrective action: for exceedances of the upper control limit, turn on one or more of the de-humidifiers in the gravimetry laboratory. For exceedances of the lower control limit, fill and turn on the humidifier. Do not attempt to adjust the wall-mounted humidistat. Wait at least two hours for the humidity to stabilize. Continue the use of the humidifiers/dehumidifiers until the humidity is within control limits. Periodically check that the humidifiers have water and that the water reservoirs in the dehumidifiers still have capacity. The latter have automatic cutoff sensors that will turn the units off when the reservoirs are full.
- 9.3.5 Note: The thermometer/hygrometer is calibrated against NIST-traceable instruments. The current certificate is stored in a 3-ring notebook with weight certifications in the weighroom.

9.4 NIST verification of Dickson

- 9.4.1 Frequency: daily
- 9.4.2 QC statistic: temperature and humidity
- 9.4.3 Control Limits: ± 0.5 °C and $\pm 2\%$ relative humidity off of the readings of the NIST traceable thermometer/hygrometer located in the weighroom.
- 9.4.4 Corrective Action: calibrate the Dicksonware software as described in section 7.2.
- 9.4.5 Note: the Dicksonware software is calibrated once per month, even if it is still within the control limits stated above. Documentation of this monthly calibration is as per section 7.2.

10.0 Calculations

10.1 N/A

11.0 References

11.1 Dickson Datalogger and Dicksonware operators manual
+ App B, 103.1, App J, App L SA 11-23-13

12.0 Definitions

- 12.1 Analyst: the designated individual who performs the "hands-on" method and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.
- 12.2 Analysts' Notes: Non-essential aspects of a method, which may help the analyst during some phase of the method. Notes may include, but not be limited to, historical aspects of the method, "tricks" of the method, unexpected issues to be aware of, or other facts or opinions related to the method, but not directly part of the procedure.
- 12.3 Calculations (Data Reduction): the mathematical process of transforming raw data into a more useable form.
- 12.4 Calibrate: to determine, by measurement or comparison with a standard, the correct value of each reading of the instrument.
- 12.5 Control Limit: A mathematical representation of acceptable limits for a given Quality Control Metric such as percent recovery or percent difference. Limits may be in the form of an absolute number or represented as a percentage.

12.6 Corrective Action: the action taken to address and/or eliminate where possible the causes of a nonconformity, such as exceeding a control limit. Actions may include reanalyzing a sample, or noting the non-conformance in the data report.

12.7 Frequency: The number of occurrences of a specified event within a given interval. The number of samples or analytical runs with which a given QC sample or metric must be analyzed or verified.

12.8 QA/QC: Quality Assurance/Quality Control. A series of samples or metrics designed to show precision, accuracy and bias of the procedure are within acceptable limits.

12.9 QC Statistic: any of a number of statistical permutations performed on raw data to generate a metric capable of being subjected to control limits and corrective actions.

12.10 Reagent: a single chemical or combination of chemicals or a chemical solution used in the preparation or analysis of samples.

13.0 Analysts' Notes

13.1 N/A

APPENDIX A: Differences from Promulgated methods

There is no promulgated method; this method was developed in-house.

The QC controls in this method are those used in the referenced methods above.

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Standard Operating Procedure GR-019.04

SARTORIUS ME5 MICROBALANCE: TEFLON AND QUARTZ FIBER
FILTER PREPARATION AND GRAVIMETRY
(25 mm, 37 mm and 47 mm)
TSP, PM₁₀ and PM_{2.5}
CHESTER LABNET PROPRIETARY METHOD

Approvals:

<u>Shirley H. Hester</u> Author	<u>11-20-15</u> Date
<u>Jim Ellis</u> Lead Analyst	<u>9.20.15</u> Date
<u>Shirley H. Hester</u> QA/QC	<u>11-20-15</u> Date

Effective from: 11-20-15
Effective until: present

SARTORIUS ME5 MICROBALANCE: TEFLON AND QUARTZ FIBER FILTER
PREPARATION AND GRAVIMETRY
(25 mm, 37 mm and 47 mm)
TSP, PM10 and PM2.5
CHESTER LABNET PROPRIETARY METHOD

1.0 Introduction

- 1.1 Test Method Reference ID: 40 CFR 50 Appendices B (TSP), J (PM₁₀) and L (PM_{2.5}).
- 1.2 Applicability: This method is applicable to the inspection, packaging, and tare and gross weighing of 25 mm, 37 mm, and 47mm filters of Teflon or Quartz Fiber composition on the Sartorius ME5 microbalance. This method could, in theory, be utilized for other matrices of filters of the same size range; however, it is not common to find other matrices in use. Particulate mass may be speciated by TSP, PM₁₀ or PM_{2.5} depending on the sampling method employed by the client.
- 1.3 Detection Limit: The balance reads to 0.001 mg. Variance of up to 0.003 mg is allowed on weights of a known mass. A true detection limit does not apply to this method.
- 1.4 Method Performance: Refer to promulgated methods listed in section 1.1. Field sampling is a major component of Method Performance. Chester LabNet has no control over the actions of the client in the field.

2.0 Summary

- 2.1 Scope and Application: The intended use of this method is for the inspection, packaging and weighing of 25 – 47 mm Teflon and Quartz fiber filters for analysis of air samples. This method could, in theory, be utilized for other matrices of filters of the same size range; however, it is not common to find other matrices in use. This method meets its intended use.
- 2.2 Summary of Method: Filters purchased from commercial vendors are acceptance tested by visual inspection. Filters passing inspection are equilibrated in a temperature and humidity controlled environment, tare weighed, and either stored or shipped immediately to the client. Filters returned from the field are equilibrated in a temperature and humidity controlled environment for 24 hours, then gross weighed. Tare and gross weights are electronically

transferred to the Laboratory Information Management System (LIMS).

All filters are weighed and data is recorded using a proprietary custom software program called FilBERT. FilBERT stands for FILter Balance Electronic Retrieval Technology. This software automatically records analytical results into two files: the daily log file and the data file. The daily log file is a chronological log of all analyses performed on a given day, including analyst's initials, date/time and temperature/humidity readings. The data file contains all the information pertaining to that filter, such as Tare, Tare QA, Gross and Gross QA. The software uses the information in the data file to calculate the net mass, as well as the difference in QA reweights.

- 2.3 Interferences: There are no traditional interferences involved with this method. Anything which would change the final balance reading during weighing (such as temperature/humidity being out of control, static buildup, or fingerprints on the filters etc.) would behave as an interferent.
- 2.4 Sample collection/preservation/shipment/storage: New filters are received from the manufacturer or vendor, each box is stamped with the received date, and then stored in the climate controlled weighroom. Temperature of the filters and room should be kept at an average temperature of 20 to 23° C ± 2°C off the average, and at 30% - 40% relative humidity ± 5% off the average relative humidity over a 24-hour period.

Collection and shipment of samples is performed by the client. Chester LabNet has no control over the actions of the client in the field. Upon receipt, samples are stored in the weighroom for at least 24 hours prior to weighing to allow for equilibration with the weighroom environmental conditions.

3.0 Safety

- 3.1 Follow the Chester LabNet Chemical Hygiene plan. Always treat samples of unknown origin and/or constitution as hazardous.
- 3.2 This method presents no safety risk beyond typical laboratory safety hazards.
- 3.3 No carcinogenic reagents are used in this method.

4.0 Pollution Prevention and Waste Management

4.1 The smallest quantity of chemical feasible is removed from its primary container for use.

4.2 Chemicals are used in amounts needed by the method.

5.0 Apparati, Equipment and Supplies

5.1 25 mm, 37 mm, or 47 mm Teflon or Quartz fiber filters

5.2 Petrislides or dishes

5.3 Fine point Sharpie permanent marker

5.4 Disposable gloves

5.5 Back lit light box

5.6 Kimwipes brand wipers

5.7 Aspirator bulb for blowing loose particles from filters

5.8 Sartorius ME5 microbalance

5.9 100 mg and 500 mg NIST-traceable standard weights (two sets)

5.10 Metal forceps

5.11 Plastic tipped metal forceps

5.12 PC running on Windows with Excel 2000

5.13 Anti-static ionizing source (Polonium Strips)

5.14 Laminar flow hood with HEPA filter

5.15 Dickson ProSeries temperature/humidity recorder with USB connector

5.16 NIST-traceable thermometer/hygrometer (VWR Cat. No. 35519-044)

6.0 Reagents and Standards

6.1 Reagent grade ethanol

7.0 Preparation, Calibration and Standardization

7.1 Environmental Weighroom Controls:

7.1.1 All operations involving filters will take place in the weighroom. Operations not involving the direct weighing of filters will take place in the laminar flow hood in the weighroom. Handle all filters with metal or plastic forceps only. Take care to

avoid contaminating filter with finger grease or particulate matter.

- 7.1.2 The weighroom is temperature and humidity controlled to satisfy the requirements of three specific reference methods, each with its own set of tolerances:

REFERENCE METHOD	AVERAGE TEMPERATURE	AVERAGE RELATIVE HUMIDITY
TSP (40 CFR 50, APPENDIX B and IO 3.1)	15 - 30 ± 3 °C (59 - 86 ± 5 °F)	< 50 ± 5%
PM10 (40 CRF 50, APPENDIX J)	15 - 30 ± 3 °C (59 - 86 ± 5 °F)	20 - 45 ± 5%
PM2.5 (40 CFR 50, APPENDIX L)	20 - 23 ± 2 °C (68 - 73 ± 4 °F)	30 - 40 ± 5%
CLN TARGETS	20 - 23 ± 2 °C	30 - 40 ± 5%

The Chester LabNet (CLN) targets for temperature and humidity are set at values that simultaneously satisfy all three reference methods. Note that the ranges specified for both parameters are the maxima allowed within any given 24-hour period.

7.2 Receipt of new filters:

- 7.2.1 Filters are received from the manufacturer or vendor, the received date is written on the package, and the filters are stored in the climate controlled gravimetry laboratory maintained at the above target values.

7.2.2 Acceptance test filters:

- 7.2.2.1 This step only applies to filters for PM2.5 use only. These filters will always be Teflon, assuming the client is following 40 CFR 50, Appendix L.

- 7.2.2.2 Out of each delivery batch of filters, randomly select three boxes from each manufacturing lot. If the manufacturing lot consists of three or fewer boxes of filters, use all boxes from that manufacturing lot. Note that this may mean that more than three boxes will be chosen if the

delivery lot consists of more than one manufacturer's lot.

7.2.2.3 Randomly select two filters from each of the selected boxes from the delivery lot.

7.2.2.4 Place the filters in a Petrislide and label them according to the manufacturer's lot and box from which they were selected.

7.2.2.5 Assign each filter a number in sequence, starting with the number 1 and continuing chronologically throughout the year. For example, the first set of delivery batch acceptance filters may be assigned numbers 1 – 6, then the second set of delivery lot acceptance filters will be assigned numbers 7 – 12.

7.2.2.6 Allow these filters to equilibrate in the temperature and humidity controlled weighroom for at least 24 hours.

7.2.3 Tare weigh the filters as per section 8.3, with the following exceptions:

7.2.3.1 Name the log file for these filters "MLATMMDD", where:

M=Sartorius ME5 balance

LAT=Lot Acceptance Test

MM=month testing was performed

DD=day testing was performed

7.2.3.2 Assign an ID for these filters using the nomenclature "YY-MLATXXX", where:

YY = two digit year abbreviation (e.g. "08")

M= Sartorius ME5 balance

LAT=Lot Acceptance Test

XXX=number of filter being tested, ranging from 001 - 999 (see section 7.2.2.5)

7.2.4 After Tare weighing, store the filters for 24 hours in the temperature and humidity controlled weighroom.

7.2.5 After a minimum of 24 hours has elapsed, Tare QA weigh 100% of the filters as per section 8.4.

7.2.6 After a minimum of 24 hours has elapsed, Gross weigh the filters as per section 8.10.

7.2.7 The Tare weight, Tare QA weight and Gross weight must all be within $\pm 15 \mu\text{g}$ of each other.

7.2.7.1 If the three weighings are not within $\pm 15 \mu\text{g}$ of each other, allow the filters to equilibrate for 24 hours and reweigh again. In this situation, the Tare QA weight will be considered the Tare weight, the Gross weight will be considered the Tare QA weight and the reweighing will be considered the Gross weight.

7.2.7.2 If the weighings from 7.2.7.1 are not within $\pm 15 \mu\text{g}$ of each other, repeat section 7.2.7.1 until the weights are within $\pm 15 \mu\text{g}$ of each other.

7.2.8 Document the acceptance weighing.

7.2.8.1 Record the date the filters started equilibration in the weighroom, the manufacturer's lot number, the quantity of boxes received for a given manufacturer's lot, the type of filter, the date the manufacturer's lot was accepted and the filter ID range (as prestamped on the filter by the manufacturer) in the filter receiving logbook.

7.2.8.2 If the filters required more than 3 consecutive weighings to pass the $\pm 15 \mu\text{g}$ control limits, also record the number of days required prior to that manufacturer's lot passing the control limits in the filter receiving logbook. This is the number of days all filters of that lot will be required to equilibrate in the weighroom prior to tare or gross weighing for sampling usage.

7.2.8.3 Print the daily run log associated with all acceptance weighings (Tare, Tare QA and Gross) and place in the Lot Acceptance Test 3-ring binder.

7.3 Clean workspace and balance:

- 7.3.1 At the beginning of each workday, clean the countertop of the laminar flow hood and the front of the light box with 95% ethanol and a Kimwipe.
- 7.3.2 Using the bulb, blow particles from the balance stage, weighing chamber and surrounding area.
- 7.3.3 Wipe the area surrounding the balance with a Kimwipe wetted with ethanol.
- 7.3.4 Allow all parts to dry completely before proceeding with QA program.
- 7.3.5 Do not clean the inside of the chamber with anything other than the air from the bulb.

7.4 Internal Calibration:

- 7.4.1 Verify the balance is level by examining the leveling indicator on the back right corner of the instrument. The bubble must be within the circle. If it is not, adjust the thumb wheels on the bottom of the balance until the bubble is contained within the circle.
- 7.4.2 Sartorius ME5 Balance Internal Calibration: manually perform an internal calibration of the balance by pressing the "Cal" button then the "start" button on the ME5 balance.

7.5 Balance Calibration and Verification:

- 7.5.1 Log in to "FiIBERT Sartorius ME5" program by double clicking the shortcut icon on the balance computer.
 - 7.5.1.1 Click on "Login" and enter the analyst's initials.

- 7.5.1.2 When prompted for the current temperature and humidity, enter the correct temperature and humidity as displayed by the Dickson software. Refer to SOP GR-018 for operation of the Dickson monitor.
- 7.5.1.3 Select the most recent ME5 Calibration log located in C:\MyDocuments\Filbert\Filters\DailyRunLogs. The file is named "YYYYME5CAL" where "YYYY" is the current year.
- 7.5.1.4 Click on Tare Weigh Filters. When prompted by the software, select a data file in which to store the data. Open the Daily Calibration file located in C:\My Documents\Filbert\Filters\DailyCal.
- 7.5.2 After the manual internal calibration is complete, select "100 mg A" from the pull down menu. Click "Tare Weigh Filter."
- 7.5.3 FilBERT will prompt the analyst to Cal In.
 - 7.5.3.1 As directed by FilBERT, press "OK" to tare the balance.
 - 7.5.3.2 FilBERT will prompt the user to place the 500 mg weight on the stage. Wait until the reading stabilizes, then click "OK."
 - 7.5.3.3 The computer will begin taking readings in two second increments. When the computer has taken six consecutive identical readings, the computer will beep and record the data into the selected spreadsheet.
 - 7.5.3.4 FilBERT will ask if the calibration has passed. Click "yes" if the registered value is ± 0.003 mg of the NIST certified true value of the weight.
 - 7.5.3.5 FilBERT will repeat steps 7.5.3.2 through 7.5.3.4 for the 100 mg weight.
 - 7.5.3.6 FilBERT will prompt analyst to remove all weights. Allow the balance to return to 0.000 mg. Click "OK" to allow FilBERT to take the reading. The balance must return to and record 0.000 mg or else FilBERT will note that the calibration has failed and prompt the user to retry the Cal In

procedure. (Sections 7.5.3.1 through 7.5.3.6.)

7.5.3.7 After the calibration passes, FilBERT will prompt user to place the "100 mg A" weight on the stage.

7.5.3.8 FilBERT will take readings in two second increments. When the software has taken six consecutive identical readings the computer will beep and record the data into the selected spreadsheet.

7.5.3.9 Repeat steps 7.5.3.7 – 7.5.3.8 five more times, using the following ID sequence: 100mg A, 500mg A, 100mg B, 500mg B, 100mg C, 500mg C.

7.5.3.10 Remove the 500mg weight from the balance and click "Exit" to end weighing session and proceed to the Balance Cal Out.

7.5.4 Balance Cal Out:

7.5.4.1 FilBERT will prompt the user to place the 500mg standard calibration weight on the stage.

7.5.4.2 FilBERT will take readings in two second increments. When the software has taken six consecutive identical readings the computer will beep and record the data into the selected spreadsheet.

7.5.4.3 FilBERT will ask if the calibration has passed. Click "yes" if the registered value is ± 0.003 mg of the NIST certified true value of the weight.

7.5.4.4 FilBERT will repeat steps 7.5.3.2 through 7.5.3.4 for the 100mg weight.

7.5.4.5 FilBERT will prompt the analyst to remove all weights. Allow the balance to return to 0.000mg. Click "OK" to allow FilBERT to take the reading. The balance must return to and record 0.000mg or the calibration failed and must be repeated.

7.5.5 Document Calibration/Verification data:

7.5.5.1 On a daily basis, fill in the daily balance control chart by writing in the three 100 mg and 500 mg weight readings, their average, their moving range and the date and initials of the analyst performing the work, as per SOP GR-016.

7.5.5.2 If the average mass of the weight is not within control (see section 9.5), recalibrate beginning with section 7.4. If unacceptable results are obtained a second time notify the Weighroom Technical Director for corrective actions. Corrective action may include outside service/repair of the balance.

7.5.5.3 On the last day of the week which the balance is used, open the daily run file in which the current data is stored. Set the print area to include all the calibration data for the respective week and print.

7.5.5.4 Place the printout in the 3-ring binder labeled "ME5 Calibration" in the weighroom.

7.5.6 Log out of the software to save the data and to close the daily run file. A new daily run file will need to be selected prior to analyzing samples.

7.6 Monthly Calibration Check

7.6.1 Perform the Monthly Calibration Check as scheduled by the Weighroom Technical Director.

7.6.2 Follow the Daily Calibration Verification Procedure given in Section 7.5 with the following exceptions:

7.6.2.1 Select the ME5 Monthly Cal log located in C:\MyDocuments\Filbert\Filters\DailyRunLogs. The file is named "ME5 Monthly Cal" (change of section 7.5.1.3).

7.6.2.2 Click on Tare Weigh Filters. When prompted by the software, select a data file in which to store the data. Open the Monthly Calibration file located in C:\My Documents\Filbert\Filters\DailyCal. The file is named "YYYY ME5 Monthly Cal" where "YYYY" is the current year (change of section 7.5.1.4).

7.6.2.3 Use the daily working standard weights for Cal In and Cal Out, however, used the specifically designated standard weights for the Monthly Calibration Check (e.g. one set of standards for Cal In/Out, second discrete set of standards for the Monthly check) **DO NOT ALLOW THESE STANDARDS TO BE ACCIDENTALLY SWITCHED DURING USE.**

7.6.2.4 The passing value of these Monthly Calibration checks is ± 0.005 mg of the true value of the weight as provided on the annual certification of measurements for that standard weight.

7.6.2.5 Document the Monthly Calibration Check by printing out the raw data and placing it in the Monthly Cal three-ring binder, and filling out the control chart also located in the Monthly Cal binder.

8.0 Procedure

8.1 Visual inspection and labeling of filters:

8.1.1 Open 5 to 10 Petrislides and place them at the top of the lightbox

8.1.2 Using the bulb, blow any loose particles out of the inside of each Petrislide.

- 8.1.3 Using the forceps, remove the filter from its box, handling only by its edges, and hold in front of the light box. Inspect both sides of the filter for any of the following:

- deep creases
- tears or holes
- discoloration
- non-uniformity
- foreign particles
- pinholes (Teflon only)
- separation of membrane from support ring (Teflon only)
- other indications of poor manufacture (e.g. filter ID errors)

Attempt to remove foreign particles by blowing on them with the aspirator bulb. If particles can't be removed, the filter must be discarded.

- 8.1.4 If no flaws are found, the filters are considered "fit for use". Place the filter inside the Petrislide and replace the lid of the slide. Flawed filters are retained for use as filter lot blanks or discarded, depending on individual project needs.

8.2 Assign ID numbers to filters:

- 8.2.1 For 25mm or 37mm Teflon filters, use a fine point sharpie to carefully write the appropriate filter ID on the PMP support ring of the filters. Write the filter ID on the Petrislide.
- 8.2.2 For 47mm Teflon filters, write the prestamped filter ID on the Petrislide of every 10th filter inspected and when any change in the chronological order of the prestamped IDs occurs.
- 8.2.3 Sample ID labels may be placed on the Petrislide at this point if the project manager has already generated the labels.
- 8.2.4 Store on the shelf in the weighroom.

- 8.2.5 Record the inspection date, manufacturer's lot number and analyst's initials in the appropriate active sample logbook. For 47mm Teflon filters, also record the prestamped filter ID.

8.3 Tare weigh filters:

- 8.3.1 Verify that the filters have equilibrated for the appropriate amount of time needed for that manufacturer's and delivery lot number (see section 7.2)

- 8.3.2 Check the environmental parameters to ensure they are within control limits (see Section 7.1).

- 8.3.3 Log into the FilBERT program by double clicking the shortcut located on the desktop of the balance computer:

- 8.3.3.1 Click on "Login" and enter the analyst's initials.

- 8.3.3.2 When prompted for the current temperature and humidity, enter the correct temperature and humidity as monitored and recorded by the Dickson thermometer/hygrometer software. Refer to SOP GR-018 for operation of the Dickson thermometer/hygrometer.

- 8.3.4 Create or Select a log file:

- 8.3.4.1 When prompted by the software to select a log file, open C:\My Documents\Filbert\Filters\Daily Run Logs.

- 8.3.4.2 Create a new log for each day. The files are named in the format YYMMDBW, where:

- YY is the year
 - MM month
 - DD day
 - B balance (M for ME5)
 - W "T" for tare weights, "G" for gross weights

8.3.5 Create or Select data file:

8.3.5.1 Once the run file is created/selected, click on Tare Weigh Filters.

8.3.5.2 The software prompts the user to select a data file into which FilBERT will store the data. Open the file into which the annual data is to be stored.

8.3.5.3 Annual data is currently being stored in the form "YYYYME5"

Where YYYY is the year

These files are located in C:\My Documents\Filbert\Filters.

8.3.6 Balance Control Check (Cal In):

8.3.6.1 Enter the first filter ID into the Filter name box at the top of the screen.
Note that the filter name format must be YY-TNNNN

where:

YY = year

T = type of filter (in this case, Teflon)

NNNN = four digit number reflecting the chronological filter ID

Failure to keep four digits in the filter ID will cause the filter to not sort properly within the spreadsheet, causing difficulties in retrieving data.

8.3.6.2 Follow the Cal In procedure in section 7.5.3.1 – 7.5.3.6.

8.3.7 Tare Weigh Filters:

8.3.7.1 Once the Cal In procedure is complete, FilBERT will prompt the user to place the filter on the balance.

8.3.7.2 Using the forceps, wave the filter between the static dischargers for approximately 30 seconds. Alternatively, set the filter on the bottom static discharging unit for approximately 30 seconds. Next, place the

filter on the stage and close the door on the balance. Wait until the reading stabilizes and "mg" appears on the balance LCD screen, then click "OK".

8.3.7.3 The computer will begin taking readings in two second increments. When the computer has taken six consecutive identical readings, the computer will beep and record the data into the daily run log and the year file.

8.3.7.4 Remove the filter from the stage and place it back in its Petrislide.

8.3.7.5 Repeat steps 8.3.7.2 through 8.3.7.4 up to nine more times changing the filter ID accordingly. The program will prompt the user to perform a calibration check after the tenth filter. If the weigh set is less than ten filters, click "Exit" after the last filter has been weighed to perform a Cal Out.

8.3.8 Calibration Check:

8.3.8.1 After every 10 filters are weighed, the software prompts the user to remove everything from the balance and perform a Calibration Check.

8.3.8.2 Before another filter can be weighed, FilBERT will prompt the user to place either the 100 mg or 500 mg standard calibration weight on the stage.

8.3.8.3 The computer will begin taking readings in two second increments. When the computer has taken six consecutive identical readings, the computer will beep and record the data into the daily run log and year file.

8.3.8.4 FilBERT will ask if the calibration has passed. Click "yes" if the registered value is ± 0.003 mg of the NIST certified true value of the weight. If the calibration check fails, the weigh set preceding the calibration check must be weighed again with passing QC.

8.3.8.5 Remove the weight from the balance stage and wait for the balance to stabilize, then press "enter" to read and record 0.000 mg. When the weight has registered, it must be 0.000 mg. If the measurement is not 0.000 mg, then the filter set is invalid, the balance must be manually tared, and the filter set must be weighed again.

8.3.8.6 After the Cal Check readings have been taken, FilBERT will request the next filter ID to be weighed. Continue to tare weigh.

8.3.9 Final Cal Out:

8.3.9.1 Remove the last filter and click "Exit" to end the weighing session. Follow the Cal Out procedure as in section 7.5.4.

8.3.9.2 If the Final Cal Out readings are out of control, the entire weigh set must be tared again.

8.3.9.3 After all Tare weighing procedures are completed, log out of FilBERT to save the data file.

8.4 Tare QA Weigh filters:

8.4.1.1 Wait a minimum of 20 hours before performing QA reweight.

8.4.1.2 After the 20 hour holding period, one filter from each set of ten weighed, chosen at random, must be reweighed to pass QA. This correlates to 10% of the filters being reweighed.

8.4.1.3 Follow the Tare Weigh procedure (Section 8.3), choosing "QA Tare Weighed Filters" instead of "Tare Weigh Filters" in FilBERT.

8.4.1.4 Filters must fall within ± 0.01 mg of their initial weighing for Teflon filters, and within ± 0.04 mg of their initial weighing for quartz filters to pass QC. If the filter fails this check, log out of FilBERT and repeat the tare and tare QA process for the entire set of 10 filters associated with the failed QA filter.

8.5 Document Tare weights:

8.5.1 After Tare QA weighings have been performed, open the daily run log.

8.5.2 Set the print area to print both the tare and the reweight data, then print. In the right-hand margin of the printout, record the total number of filters weighed in that batch. Note that the filters should be physically counted to obtain the total number of filters weighed; this verifies that no filters were accidentally omitted from a weighing batch.

8.5.3 Do not close this file manually, FilBERT will close this window and save the file automatically upon logging out.

8.5.4 Transfer the data from the balance computer to the LIMS system and release the data:

8.5.4.1 Open the Tare Daily Run file that contains the data that needs to be put on the LIMS.

8.5.4.2 Delete the columns and rows that are not needed. Ensure that the columns are in the following order: LIMS ID, Tare Weight, Temperature, Humidity, Analyst Initials, Tare Date, Time, filter ID, and lot number (note: manufacturer's filter ID and lot number must be added to the last columns of the TTARE.csv worklist. The TARE.csv worklist only requires the lot number. This information is to be found in the active sample logbook). Save the appropriate .csv file in the computer's worklist directory.

8.5.4.3 Import and save the data to the LIMS per SOP AD-007.

8.5.4.4 Write the worklist number and the quantitative number of filters imported on the top of the printout and submit for QC review.

8.5.4.5 Record the appropriate information in the ME5 Daily Logbook and the ME5 Active Sample Logbook.

8.5.5 QC review: This section applies to both Tare and Gross weight QC review

8.5.5.1 QC review must be performed by someone other than the analyst who weighed the filters.

8.5.5.2 Review the printouts for accuracy, including the following items:

- The correct number of filters were weighed (see section 8.5.2)
- No filters numbers were skipped. For tare weights, filter ID numbers should be in sequential order.
- All weigh sets are bracketed by a Cal in and Cal out, and that all Cal in's and Cal out's pass QC.
- Reweights were performed no sooner than 20 hours after the initial weighing.
- Temperature and Humidity were in control for all weighings
- An appropriate number of reweights were completed on each set of filters weighed.
- Reweights were within control limits (see 8.4.4)
- The number of filters weighed matches the number written in the right hand margin of the printout.
- No two consecutive measurements are identical or nearly identical (one filter weighed twice).
- Tare/Net masses on raw data match the data in the LIMS.

8.5.5.3 After review, date and initial the upper right corner of each log to indicate the person performing the QC review and the date on which the review took place.

- 8.5.5.4 Once the review is complete, distribute the worklist on the LIMS, and place the printed logs in the appropriate 3-ring binders in the weighroom.
- 8.6 Filter Storage: If filters are not to be immediately shipped, store the tared filters on the appropriate shelf in the gravimetry laboratory.
- 8.7 Shipping filters: Ship the filters as requested by the client or the project manager. When filters are removed from the weighroom, record the date and initials of the person removing the filters in the "out" column of the appropriate Active Sample Logbook. Likewise, record the client name and client number in the "client" column.
- 8.8 Filter ID labeling: The LIMS Administrator or Project Manager will prepare sample ID labels to be affixed to the slides, or the analyst loading the filters into cassettes will handwrite the laboratory's filter ID on the cassette using a sharpie marker. When labeling filters in slides or cassettes for 47mm Teflon filters, it is critical to cross reference the Filter ID printed on the filter and the assigned laboratory filter ID before shipping to clients.
- 8.9 Filter receipt and log-in:
- 8.9.1 Filters returned from the field are logged-in by the sample custodian, who completes the chain-of-custody (if present) and enters the appropriate filter and field information into the LIMS. See SOP AD-008, Sample Receipt and Log-In.
 - 8.9.2 The appropriate lines in the filter ID Tracking Logbook are marked with the date, time the filters were placed in the weighroom, and the initials of the person who logged the filters into the weighroom.
 - 8.9.3 The sample custodian or weighroom technician then transfers the filters to an empty tray on the filter rack in the weighroom.
 - 8.9.3.1 Place the filters in numerical order on a tray, left to right, and back to front. The first filter will be the back left filter, the last filter in the set will be the front right filter.

8.9.3.2 Remove the slide cover partially, such that 75% of the filter remains covered, but the filter is exposed to the controlled temperature and humidity of the weighroom environmental conditions.

8.9.3.3 On the tray itself, using a dry erase marker, write the date and time the filters were placed on the tray.

8.9.4 If the filters are received in filter cassettes, remove the filter from the cassette, place in a labeled Petrislide, and placed on the filter rack as per section 8.9.3 to allow for 24 hours of equilibration. See SOP GR-006 for guidance on unloading filter cassettes.

8.9.5 Equilibrate the filters for a minimum of 24 hours at the temperature and humidity specified in Section 7.1.2.

8.10 Gross weigh filters:

8.10.1 Verify that the filters have equilibrated for the appropriate amount of time needed for that manufacturer's and delivery lot number (see section 7.2.).

8.10.2 Check the environmental parameters to ensure they are within control limits (see Section 7.1).

8.10.3 Filters must equilibrate for a minimum of 24 hours in the weighroom under controlled temperature and humidity prior to gross weighing.

8.10.4 Gross weigh the filters following the same procedure as described in Section 8.3, substituting "Gross" for "Tare" where appropriate.

8.11 Gross QA:

8.11.1 After the 20 hour holding period, 10% of the filters from each weigh set, chosen at random, must be reweighed to pass QA, following the same procedure as in section 8.4, substituting "Gross" for "Tare" where appropriate.

8.12 Document Gross weights:

- 8.12.1 After Gross QA weighings have been performed, open the daily run log.
- 8.12.2 Set the print area to print both the gross and the reweight data, then print. In the right-hand margin of the printout, record the total number of filters weighed in that batch. Note that the filters should be physically counted to obtain the total number of filters weighed; this verifies that no filters were accidentally omitted from a weighing batch.
- 8.12.3 Do not close this file manually, FilBERT will close this window and save the file automatically upon logging out.
- 8.12.4 Transfer the data from the balance computer to the LIMS system and release the data.
 - 8.12.4.1 Open the Gross Daily Run file that contains the data that needs to be put on the LIMS.
 - 8.12.4.2 Delete the columns and rows that are not needed. The columns should be in the following order: LIMS ID, Gross Weight, Temperature, Humidity, Analyst Initials, Gross Date and then Time. For Teflon filters, save the file as GROSS10.CSV in the computer's worklist directory. For quartz filters, name the file GROSS40.CSV.
 - 8.12.4.3 Import and save data to the LIMS per SOP AD-007.
 - 8.12.4.4 Write the worklist number at the top of the printout.
 - 8.12.4.5 Record the appropriate information in the ME5 Daily Logbook and the ME5 Active Sample Logbook.

8.13 QC review: See section 8.5.5

8.14 Chemical analysis of filters:

8.14.1 If the filters are to be further analyzed by conventional chemistry, notify the conventional chemistry technical director that the gravimetry is complete. Transfer to the sample staging area in the main laboratory.

8.14.2 If the filters are to be further analyzed by XRF, notify the XRF technical director that the gravimetry is complete. Transfer the filters to the XRF staging area.

8.15 Storage of filters:

8.15.1 After the filters have been completely analyzed, store the filters in the archive room for an amount of time to be determined by the client or for a minimum of six months.

9.0 QA/QC

9.1 Weighroom temperature:

9.1.1 Frequency: monitored continuously by a Dickson Pro Series TP125 combination thermometer/hygrometer (see Section 9.9, below).

9.1.2 QC statistic: temperature in °C.

9.1.3 Control limits: 24-hour average within 20 – 23 °C, with no more than a ± 2 °C change from the average occurring over any 24-hour period.

9.1.4 Corrective action: contact the Weighroom Technical Director. Adjust the thermostat in the weighroom. Wait at least two hours for temperature to stabilize. Continue adjusting until the temperature comes within the control limits. If control limits are unattainable, notify the laboratory director for an HVAC system service call. See Section 7.1 for a detailed discussion of this QC parameter.

9.1.5 Note: The thermometer/hygrometer is calibrated against NIST-traceable instruments. The current certificate is stored in a 3-ring binder with weight certifications in the weighroom.

9.2 Weighroom humidity:

- 9.2.1 Frequency: monitored continuously by a Dickson Pro Series thermometer/hygrometer displayed on the computer by the Dicksonware software (see SOP GR-018).
- 9.2.2 QC statistic: relative humidity in percent.
- 9.2.3 Control limits: 24-hour average within 30 – 40 %, with no more than a $\pm 5\%$ change from the average occurring over any 24-hour period
- 9.2.4 Corrective action: for exceedances of the upper control limit, turn on and/or adjust one or more of the de-humidifiers in the gravimetry laboratory. For exceedances of the lower control limit, fill and turn on the humidifier. Wait at least two hours for the humidity to stabilize. Continue the use of the humidifiers/dehumidifiers until the humidity is within control limits. Periodically check that the humidifiers have water. See Section 7.1 for a detailed discussion of this QC parameter. See Analyst's note 13.2.
- 9.2.5 Note: The Dicksonware is calibrated monthly or as needed against NIST-traceable instruments. Refer to SOP GR-018 for details.

9.3 Balance Calibration:

- 9.3.1 Frequency: at the beginning of the day, and, if necessary, during the day.
- 9.3.2 QC statistic: the ME5 has an internal calibration weight. Calibration is performed manually each day before weighing and automatically as needed.

9.4 Initial Calibration Zero:

- 9.4.1 Frequency: once, immediately after calibration
- 9.4.2 QC statistic: analysis result
- 9.4.3 Control limits: 0.000 mg
- 9.4.4 Corrective action: recalibrate balance. If recalibration fails, check to see if balance is level. Check for dirt or misalignment of balance stage components. For initial calibration, clean NIST weight. If nothing is evident upon visual inspection, call the balance repair/calibration service and schedule a maintenance visit.

9.5 Initial Calibration Verification:

- 9.5.1 Frequency: once, immediately after Initial Calibration Zero
- 9.5.2 QC statistic: average of 3 analyses results.
- 9.5.3 Control limits: certified value of weights ± 0.003 mg (two weights are used - one ~100 mg and one ~500 mg weight. The certified value may not be exactly 100.000 or 500.000mg)
- 9.5.4 Corrective action: weigh verification weight a second time. If verification weight is still outside control limits, recalibrate balance and reweigh verification weight. If verification weight fails after recalibration, check to see if balance is level. Check for dirt or misalignment of balance stage components. For initial calibration, clean NIST weight. If nothing is evident upon visual inspection, call the balance repair/calibration service and schedule a maintenance visit.

9.6 Continuing Calibration Zero:

- 9.6.1 Frequency: every 10 weighings
- 9.6.2 QC statistic: analysis result
- 9.6.3 Control limits: 0.000 mg
- 9.6.4 Corrective action: recalibrate balance, if recalibration resolves the problem, reweigh all filters since the last acceptable Continuing Calibration Zero. If recalibration fails, check to see if balance is level. Check for dirt or misalignment of balance stage components. For initial calibration, clean NIST weight. If nothing is evident upon visual inspection, call the balance repair/calibration service and schedule a maintenance visit.

9.7 Continuing Calibration verification (note: the mass used for this check alternates every set of 10 filters between a 100 mg weight and a 500 mg weight):

- 9.7.1 Frequency: after each set of 10 filters has been weighed
- 9.7.2 QC statistic: analysis result
- 9.7.3 Control limits: certified value of weights ± 0.003 mg (two weights are used - one ~100 mg and one ~500 mg weight. The certified value may not be exactly 100.000 or 500.000mg)
- 9.7.4 Corrective Action: recalibrate balance, if recalibration resolves the problem, reweigh all filters since the last acceptable Continuing Calibration verification. If

recalibration fails, check to see if balance is level. Check for dirt or misalignment of balance stage components. For initial calibration, clean NIST weight. If nothing is evident upon visual inspection, call the balance repair/calibration service and schedule a maintenance visit.

9.8 Monthly Calibration Check

- 9.8.1 Frequency: once per month
- 9.8.2 QC statistic: analysis result
- 9.8.3 Control limits: certified value of weights ± 0.005 mg (two weights are used - one ~100 mg and one ~500 mg weight. The certified value may not be exactly 100.000 or 500.000mg)
- 9.8.4 Corrective Action: recalibrate balance. If recalibration fails to correct the issue, check to see if balance is level. Check for dirt or misalignment of balance stage components. If nothing is evident upon visual inspection, call the balance repair/calibration service and schedule a maintenance visit.

9.9 Demonstration of constant weight:

- 9.9.1 Frequency: 10% or 1 from each filter batch is reweighed a minimum of 20 hours after the initial weighing. Filters must be held in the temperature and humidity controlled gravimetry laboratory during this time.
- 9.9.2 QC statistic: change in filter mass.
- 9.9.3 Control limits: For tare weighings, the second weights must be within ± 0.01 mg for Teflon filters, and ± 0.04 mg for quartz filters, of their initial weights. For gross weighing, the second weights must be within ± 0.01 mg for Teflon filters, and ± 0.04 mg for quartz filters, of the initial gross weight. For 47mm Teflon filters, the second gross weights must be within ± 10 μ g or within $\pm 2\%$ of the net weight, whichever is greater.
- 9.9.4 Corrective action: all of the filters in the batch must be reweighed and held for an additional 24 hours in the gravimetry laboratory. 10% of the filter batch is weighed again. If control limits are exceeded a second time, all of the filters in the batch are reweighed for a third time and held for an additional 24 hours in the gravimetry laboratory. 10% of the filter batch is weighed again. If control limits are exceeded, the project manager notifies the client that constant weight cannot be achieved using normal equilibration methods. Corrective action at this point

depends upon instructions from the Weighroom Technical Director and can include scheduling a maintenance visit by the balance service technician.

9.10 Computer controlled weighing process

9.10.1 The computer receives data from the balance via a general-purpose, RS232 port on a two-second cycle. The program accumulates readings and calculates the differences between successive readings.

9.10.2 The computer continues to take readings until six consecutive identical readings agree exactly to one microgram. This weight is then displayed as the result.

9.11 Balance servicing and third party balance and weight verification:

9.11.1 The balance is serviced and the calibration is verified by an outside service company on an annual basis (currently during the month September). Upon satisfactory calibration verification, the laboratory is issued a certificate, which is stored in a 3-ring binder with weight certifications in the weighroom. Previous certificates are archived in the back of the three-ring binder.

9.12 Records of environmental conditions:

9.12.1 The temperature and humidity in the weighroom are continuously monitored, seven days a week, by the Dicksonware software and Pro Series TP125 monitor located on the wall behind the balances.

9.12.2 On Fridays, download and store the temperature and relative humidity data as described in SOP GR-019.

9.13 Anti-static ionizing units:

9.13.1 Two antistatic units are suspended in a holder, facing each other. The filter is passed between these two units.

9.13.2 Each unit has a date of manufacturer stamped on the underside. The recommended expiration time given by the manufacturer is one year from the date of manufacture.

10.0 Calculations

10.1 Net deposit mass is reported in μg and is calculated by the LIMS using the following algorithm:

$$\text{net deposit mass} = (\text{gross weight} - \text{tare weight}) \times 1000 \mu\text{g}/\text{mg}$$

where both the gross weight and tare weight data is imported into the LIMS from the files generated by the balance software.

11.0 References

- 11.1 Sartorius ME Series operators' manual
- 11.2 40 CFR 50, Appendix L: "Reference Method for the Determination of Fine Particulate Matter as $\text{PM}_{2.5}$ in the Atmosphere."
- 11.3 40 CFR 50, Appendix J: "Reference Method for the Determination of Particulate Matter as PM_{10} in the Atmosphere."
- 11.4 EPA Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, Compendium Method IO-3.1: "Selection, Preparation and Extraction of Filter Material."
- 11.5 EPA Quality Assurance Guidance Document 2.12; Monitoring $\text{PM}_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods. USEPA, November 1998.
- 11.6 Standard Operating Procedure for $\text{PM}_{2.5}$ Gravimetric Analysis. Environmental and Industrial Services Division, RTI International.
- 11.7 Standard Operating Procedure for Gravimetric Analysis of Particulate Collected with R&P Partisol Samplers and MetOne SASS Samplers. State of Oregon Department of Environmental Quality.

12.0 Definitions

- 12.1 Analyst: the designated individual who performs the "hands-on" method and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.
- 12.2 Analysts' Notes: non-essential aspects of a method, which may help the analyst during some phase of the method. Notes may include, but not be limited to, historical aspects of the method, "tricks" of the method, unexpected issues to be aware of, or other facts or opinions related to the method, but not directly part of the procedure.

- 12.3 Batch: environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents.
- 12.3.1 Analytical Batch: a group of samples that are analyzed together as a group.
- 12.4 Blank: a clean aliquot of the same matrix as the digested samples. A blank is subjected to the usual analytical and measurement processes.
- 12.4.1 Calibration Blank: nothing sitting on the balance stage.
- 12.4.2 Field Blank: a blank prepared by the client in the field. This blank is treated as a sample by the laboratory.
- 12.4.3 Lab Blank: a blank filter retained by the laboratory, and reweighed when the sampled filters are gross weighed.
- 12.5 Calculations (Data Reduction): the mathematical process of transforming raw data into a more useable form.
- 12.6 Calibrate: to determine, by measurement or comparison with a standard, the correct value of each reading of the instrument.
- 12.7 Calibration Standard: a substance or reference material used to calibrate an instrument.
- 12.8 Cal Out (otherwise known as a Continuing Calibration Blank (CCB)): a blank "standard" analyzed at the end of an analytical batch and at least every 10 samples during an analytical batch to verify that the lower end of the calibration curve remains valid during the course of the analytical run.
- 12.9 Cal Out (otherwise known as a Continuing Calibration Verification Standard (CCV)): a standard weight analyzed at the end of an analytical batch and at least every 10 samples during an analytical batch to verify that the calibration remains valid during the course of the analytical run. The concentration of the CCVs should vary over the course of the analytical run.
- 12.10 Control Limit: a mathematical representation of acceptable limits for a given Quality Control Metric such as percent recovery or percent difference. Limits may be in the form of an absolute number or represented as a percentage.
- 12.11 Corrective Action: the action taken to address and/or eliminate where possible the causes of a nonconformity, such as exceeding a control limit. Actions may include reanalyzing a sample, or noting the non-conformance in the data report.
- 12.12 Delivery batch: "Filter lot" as defined by 40CFR50 Appendix L: a batch of filters which all arrive in one shipment. See "Filter Lot".
- 12.13 Reweighings (also known as Duplicates): a second weighing of a sample, performed once every 10 samples.

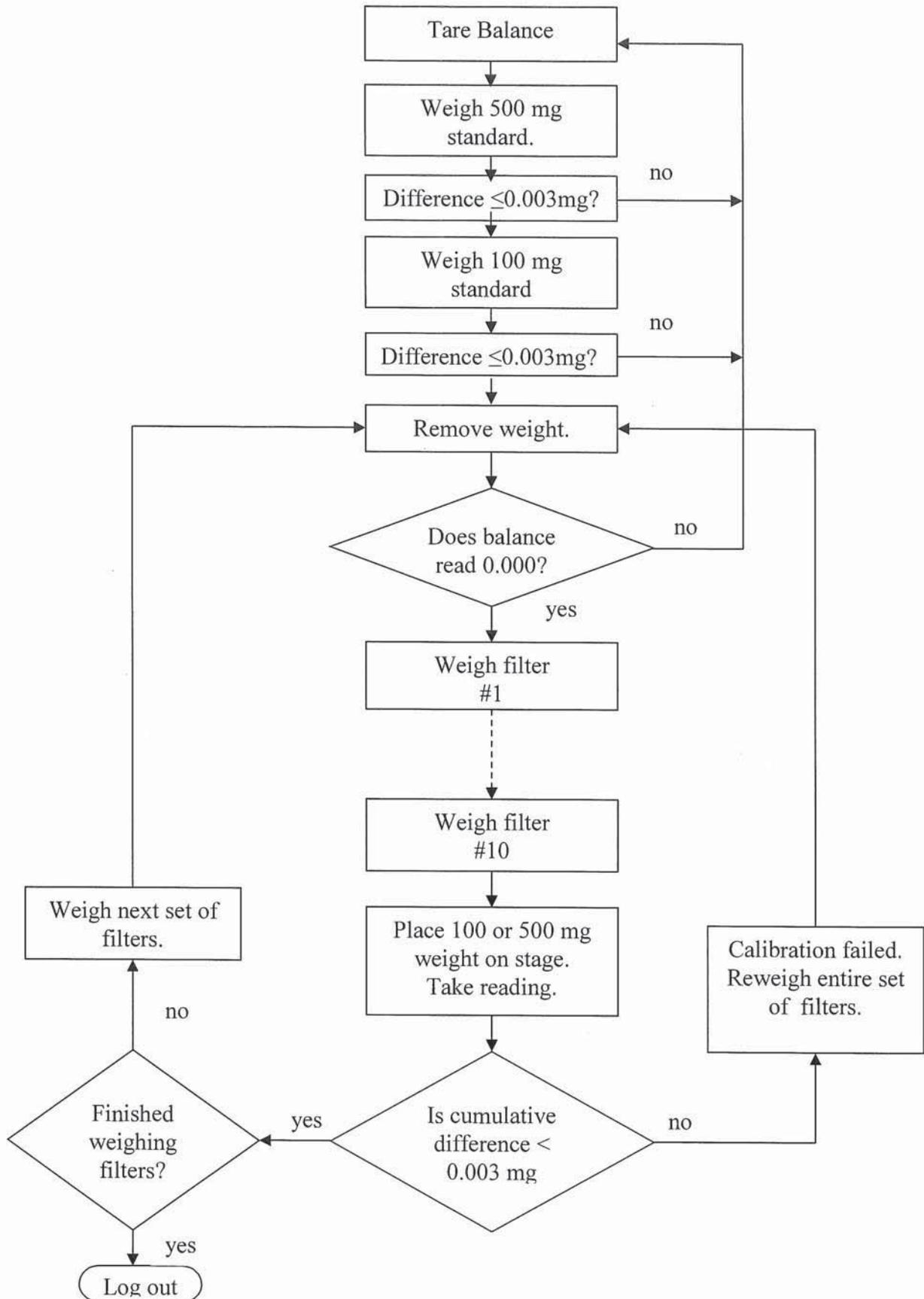
- 12.14 Filter Lot:
 - 12.14.1 Filter lot as defined by 40CFR50 Appendix L: a batch of filters which all arrive in one shipment. See "Delivery batch".
 - 12.14.2 Filter lot as defined by manufacturer: a unique number assigned to a group of filters all manufactured at the same time. For the purposes of this document, this is the definition that applies to the term "manufacturer's lot".
- 12.15 Filter rack: a series of shelves with multiple removable trays used for storage of filters during their equilibration periods.
- 12.16 Frequency: the number of occurrences of a specified event within a given interval. The number of samples or analytical runs with which a given QC sample or metric must be analyzed or verified.
- 12.17 Laboratory Information Management System (LIMS): a comprehensive computerized database system that a laboratory uses for sample tracking and data management, from sample receipt to reporting and archiving.
- 12.18 Matrix/Matrices: the component or substrate that contains the analyte of interest.
- 12.19 QA/QC: Quality Assurance/Quality Control. A series of samples or metrics designed to show precision, accuracy and bias of the procedure are within acceptable limits.
- 12.20 QC Statistic: any of a number of statistical permutations performed on raw data to generate a metric capable of being subjected to control limits and corrective actions.
- 12.21 Reagent: a single chemical or combination of chemicals or a chemical solution used in the analysis of samples.

13.0 Analysts' Notes

- 13.1 While Tare weighing filters, keep the aspiration bulb near the balance. Prior to weighing, reexamine the filter and blow off any particles which may have remained or been redeposited on the filter during the inspection process.
- 13.2 When checking the temperature/humidity on a daily basis, also check the humidifier. Fill the humidifier each morning and evening while in use.
- 13.3 Place filters and standard weights on the balance stage by holding them as close above the pan as possible then allowing the filter to drop onto the stage. Do not allow forceps to touch the balance stage.

- 13.4 Always wait for the balance LCD screen to display "mg" before pressing "enter".
- 13.5 Always allow the balance to return to 0.000 mg between filters.
- 13.6 If an accidental EXIT of FilBERT occurs, it is acceptable to manually type in the Cal Out information into the Excel spreadsheet, however, this action must be documented in the columns to the right of the data manually entered.

13.7 Brief summary of tasks in order:



APPENDIX A: Differences from Promulgated methods

A.1 Deviations from EPA 40 CFR 50 APPENDIX L

<u>Item</u>	<u>40 CFR 50 Appendix L requirement</u>	<u>SOP</u>	<u>Justification</u>
1	8.3.2 If possible, both weighings should be carried out by the same analyst.	There are several trained analysts who perform weighing duties.	Reference standard calibration checks, and 10% reweight QC verify accuracy of data.
2	8.3.6 The post-sampling conditioning and weighing shall be completed within 240 hours (10 days) after the end of the sample period, unless the filter sample is maintained at temperatures below the average ambient temperature during sampling (or 4 °C or below for average sampling temperatures less than 4 °C) during the time between retrieval from the sampler and the start of the conditioning.	<p>The client is responsible for keeping the filters at an appropriate temperature and/or sending the filters from the field <i>to arrive at</i> Chester LabNet within 168 hours (7 days) from the time of sample collection (to allow for weighing to be completed within the 10 day "hold time").</p> <p>The laboratory shall maintain the filters either in the temperature/humidity controlled weighroom (during equilibration and weighing) or stored at <4°C until equilibration and weighing are commenced. Gross weighing shall be completed by the laboratory within 240 hours after the end of the sampled period assuming the client has delivered the filters in a timely manner.</p>	The laboratory has no control over the actions or inactions of the client prior to, during, or after sampling.

<u>Item</u>	<u>40 CFR 50 Appendix L requirement</u>	<u>SOP</u>	<u>Justification</u>
3	10.2 Unacceptable filters should be discarded.	Filters that do not pass inspection are reserved for extraction/digestion lot specific Quality Control samples.	<p>The laboratory uses filters that failed inspection for purposes of Method Blank and Laboratory Control Sample (Fortified Blank) analysis. These filters are considered imperfect but not contaminated.</p> <p>Filters that failed inspection due to discoloration or other possible reasons that might indicate contamination are discarded.</p>

A.2 Deviations from EPA 40 CFR 50 Appendix J

<u>Item</u>	<u>40 CFR 50 Appendix J requirement</u>	<u>SOP</u>	<u>Justification</u>
4	7.2.1 The user's goals in sampling determine the relative importance of various filter characteristics (e.g., cost, ease of handling, physical and chemical characteristics, etc.) and, consequently, determine the choice among acceptable filters.	Filter type is chosen by the client.	The laboratory has no control over the actions or inactions of the client prior to, during, or after sampling.

<u>Item</u>	<u>40 CFR 50 Appendix J requirement</u>	<u>SOP</u>	<u>Justification</u>
5	<p>7.5 <i>Analytical Balance.</i> The analytical balance must be suitable for weighing the type and size of filters required by the sampler. The range and sensitivity required will depend on the filter tare weights and mass loadings. Typically, an analytical balance with a sensitivity of 0.1 mg is required for high volume samplers (flow rates >0.5 m³ /min). Lower volume samplers (flow rates <0.5 m³ /min) will require a more sensitive balance.</p>	<p>The laboratory uses a Sartorius ME5 microbalance capable of reading to 0.001mg.</p>	<p>Clarification of option within promulgated method.</p>
6	<p>9.4 Following equilibration, weigh each filter and record the presampling weight with the filter identification number.</p> <p>9.5 Install a preweighed filter in the sampler following the instructions provided in the sampler manufacturer's instruction manual.</p>	<p>Filters are Tare weighed, then held for a minimum of 20 hours prior to 10% of the filters being reweighed. Filters must pass reweight QC criteria before being used in sampling.</p>	<p>More stringent than promulgated method.</p>

<u>Item</u>	<u>40 CFR 50 Appendix J requirement</u>	<u>SOP</u>	<u>Justification</u>
7	<p>9.16 Equilibrate the exposed filter in the conditioning environment for at least 24 hours under the same temperature and humidity conditions used for presampling filter equilibration (see 9.3).</p> <p>9.17 Immediately after equilibration, reweigh the filter and record the postsampling weight with the filter identification number.</p>	<p>Filters are equilibrated for a minimum of 24 hours.</p> <p>Filters are gross weighed after the 24 hour period has elapsed, but may not be immediately gross weighed, depending on filter receipt time/day and other conditions.</p> <p>After the initial gross weighing, filters are held for a minimum of 20 hours prior to 10% of the filters being reweighed. Filters must pass reweight QC criteria before net weight is calculated.</p>	<p>Immediate gross weight after 24 hour equilibration time may be impracticable.</p> <p>Reweighting 10% of the filters is more stringent than the promulgated method.</p>

A.3 Deviations from EPA Quality Assurance Guidance Document 2.12

<u>Item</u>	<u>QA Guidance Document 2.12 requirement</u>	<u>SOP</u>	<u>Justification</u>
8	<p>4.3.3 & 7.9 - 12 Filter Cassette Protective Containers should be marked for identification purposes.</p>	<p>Protective containers are not labeled or marked.</p> <p>The cassette, rather than the mailer, is labeled with the filter ID.</p>	<p>Identification of filter can be seen on the cassette though the protective container.</p>
9	<p>4.3.3 If reuse of the containers is desired, a cleaning procedure that does not cause further contamination or degradation of the container should be developed.</p>	<p>Containers are either hand cleaned using Kimwipes and Ethanol or sonicated for 30minutes with Citranox then rinsed with DI water.</p>	<p>Clarification of option within promulgated method.</p>

<u>Item</u>	<u>QA Guidance Document</u> 2.12 requirement	<u>SOP</u>	<u>Justification</u>
10	7.2 The microbalance environment should maintain a slightly positive pressure.	The Weighroom has a slightly negative pressure.	The pressure does not seem to pose issues with the weighing process as demonstrated by years of consistently passing QC.
11	7.2 Dust contamination can be minimized by taking clean room measures such as cleaning the weighing area daily, installing sticky floor covering on the entrance(s) to the weighing area, and wearing clean laboratory clothing over anything exposed to uncontrolled environments.	The weighroom is cleaned often/as needed to minimize dust. Working surfaces are cleaned once per day of use, at a minimum.	The weighroom is used exclusively for weighing and storing filters; maintaining a tidy weigh area has proven that clean room measures are not necessary.
12	7.2 Ideally, the microbalance should be situated within a laminar flow fume hood to minimize contamination by the analyst.	Microbalance is kept in environmentally controlled room. Analysts are trained in filter/balance handling to prevent/minimize contamination.	Microbalances may not read properly when located in an area with constant airflow passing the balance stage.
13	7.3 Laboratory primary standards should be handled very carefully and should be kept in a locked compartment.	The primary standard is internal to the microbalance. Laboratory working standards are stored on a shelf in the weighroom, not in a locked compartment.	The laboratory maintains a locked entryway. Only authorized personnel are allowed access to laboratory/standards.
14	7.3 The working standards' masses should be verified against the laboratory primary standards every 3 to 6 months to check for mass shifts associated with handling or contamination.	Working standards are verified monthly against a second set of working standards. Primary standard is internal to the balance.	More stringent than promulgated method.

<u>Item</u>	<u>QA Guidance Document</u> 2.12 requirement	<u>SOP</u>	<u>Justification</u>
15	7.3 The verified values of the working standards as measured relative to the laboratory primary standards should be recorded in a laboratory QC notebook and used to check the calibration of the microbalance.	Primary standard is internal to the balance. Working standards are verified and documented with each weighing session against this internal primary standard. Control charts of both daily and monthly working standards show moving range to note trends	More stringent than promulgated method.
16	7.3/7.4 Mark the standards' specific nonmetallic forceps to distinguish them from the forceps used to handle filters.	Forceps are not marked to distinguish for specific use.	Forceps used for standards are Teflon tipped while forceps used to handle filters are stainless steel. These two very different styles of forceps are easily distinguishable.
17	7.4 The analyst should wear antistatic, powder-free gloves; these gloves act as an effective contamination barrier.	Analysts do not wear gloves, nor do they handle filters with their hands. Forceps are used in all filter handling operations.	Analysts are trained in filter/balance handling to prevent/minimize contamination.
18	7.5 If any defects are found during the visual inspection, discard the filter.	Filters that do not pass inspection are reserved for extraction/digestion lot specific QC.	The laboratory uses filters that failed inspection for purposes of Method Blank and Laboratory Control Sample (Fortified Blank) analysis. These filters are considered imperfect but not contaminated. Filters that failed inspection due to discoloration or other possible reasons that might indicate contamination are discarded.
19	7.6 RH and temperature should be measured and recorded on a continuous basis during filter conditioning.	Relative humidity and temperature are measured and recorded at 5 minute intervals.	Clarification of ambiguity in promulgated method.

<u>Item</u>	<u>QA Guidance Document</u> 2.12 requirement	<u>SOP</u>	<u>Justification</u>
20	7.6 If spikes of temperature or especially, RH occur during the condition period, the appropriate local decision maker should evaluate all relevant data and decide if the spikes are significant enough to compromise the condition period.	Generally, 6 outliers are allowed during the conditioning period. More than 6 outlying points, grouped outliers, or large spikes are referred to the Weighroom Technical Director to decide if the environment is "in control."	Clarification of option within promulgated method.
21	7.6 Use of standard deviation of averages during the period will be more statistically representative of the condition and will probably minimize spikes more than the rolling average method.	The rolling average method is used, with data points taken at 5 minute intervals.	Clarification of option within promulgated method.
22	7.6 Filters should be placed on a covered rack	Filters are placed on an open baker's rack with multiple shelves. The top and bottom shelves are not used as these act as a cover for the filters.	Clarification of option within promulgated method.
23	7.7 After an initial 24-hour conditioning ... lot blanks are reweighed ... These measurements should be recorded in the QC notebook. ... These weighings should continue until the 24-hour weight change is less than 15 µg.	The data is recorded and stored in the lot acceptance logbook and binder as well as electronically. Lot blanks are weighed until the weight change is less than 15 µg.	Clarification of documentation process.

<u>Item</u>	<u>QA Guidance Document</u> 2.12 requirement	<u>SOP</u>	<u>Justification</u>
24	7.7 Laboratory blank and replicate measurements should agree within 15 µg [of their initial masses]	Replicate measurements shall agree to within ± 10 µg of initial weight. Laboratory blank Gross weight shall agree to within ± 15 µg of its Tare weight.	More stringent than promulgated method.
25	7.7 A filter lot is defined as a single shipment of filters from a filter manufacturer or from another source.	For the purposes of defining lot blanks, the laboratory defines a lot as a single shipment of filters of the same manufacturer's lot. If more than one lot is shipped in a single shipment, each manufacturing lot is considered a separate lot.	More stringent than promulgated method.
26	7.8, 7.9.5, 7.14 Weigh enough laboratory blanks during a presampling weighing session to provide at least one single-use laboratory blank during each subsequent postsampling weighing session.	Laboratory Blanks are provided per client request and are weighed as needed with the appropriate filters.	Lab Blanks are weighed per client request. The laboratory may not have any information pertaining to the number of filters the client will ship back at any given time. Thus, it may not be possible for the laboratory to determine how many filters are needed to "provide at least one single-use laboratory blank during each subsequent postsampling weighing session."
27	7.8 Change the antistatic strips every 6 months.	Per manufacturer's guidelines, antistatic strips are changed 1 year from the manufacturing date.	No adverse electrostatic effects have been encountered as a result of following the manufacturer's guidelines.

<u>Item</u>	<u>QA Guidance Document</u> 2.12 requirement	<u>SOP</u>	<u>Justification</u>
28	7.8 Earth-grounded conductive mats may also be placed on the weighing table surface and beneath the analyst's shoe surfaces to reduce electrostatic charge buildup.	Antistatic floors have been installed and an antistatic chair is used at balances.	More stringent than promulgated method.
29	7.9 The pre-sample filter weighing is required to be conducted within 30 days of the sampling period.	The laboratory shall weigh filters in close temporal approximation to the estimated date of shipment.	The laboratory has no control over the actions or inactions of the client prior to, during, or after sampling.
30	7.9 - 2 [For both pre- and post-sampling weighings] Clean the microbalance's weighing chamber with a fine brush, if necessary	A brush is never used to clean the microbalance due to the possibility of both contamination and static buildup/discharge.	Per the direction of the company who services and certifies the microbalance, the chamber is blown out using an aspirator bulb.
31	7.9 - 2 Clean the surfaces around the microbalances and the forceps with antistatic solution or methyl alcohol-moistened wipes.	Ethanol and Kimwipes are used to clean the area around the microbalance and forceps.	No differences in weights have been observed whether antistatic solution is used or not. Similarly, no differences in weights have been observed in the use of ethanol versus methanol.
32	7.9 - 5 Verify the working standards' masses every 3 to 6 months or after any incident of rough handling against the laboratory's primary standard weights.	Primary standard is internal to the balance. Working standards are verified monthly against a second set of working standards. Constant repeatability of working standards is additional verification.	More stringent than promulgated method.

<u>Item</u>	<u>QA Guidance Document</u> 2.12 requirement	<u>SOP</u>	<u>Justification</u>
33	7.9 – 9, 7.11 – 4, 7.14 One routine filter should be reweighed at the end of the weighing session. Record the replicate measurement on the laboratory data form. If the replicate disagrees from the original by more than 15µg, reweigh the filter.	10% of filters from each weigh set are reweighed after a second 20 hour equilibration period from the initial gross weighing. The replicate(s) must agree with the original weight by ≤ 10µg to pass. If >10µg, the filter weigh set is considered 'failed,' and will be re-gross weighed, followed by another reweight attempt. This process continues until the filters pass the QC requirements or the project manager/client specifies otherwise.	More stringent than promulgated method.
34	7.9 – 12 Prepare several extra filters in case a filter is invalidated during the installation process.	Extra filters are not prepared for "in case" situations in the field unless requested.	The client is responsible for requesting a suitable number of filters to meet their needs.
35	7.10- 1 Upon receipt of the sample from the field, examine the field data sheet. Determine whether all data needed to verify sample validity and to calculate mass concentration ... are provided. If data are missing or unobtainable from a field operator or if a sampler malfunction is evident, save the filter for inspection and record on the laboratory data form that the sample has been voided and the reason. Notify the appropriate personnel.	The laboratory examines all field data sheets and Chains of Custody for accuracy and completeness prior to weighing filters. Any discrepancies shall be addressed with the client as soon as feasibly possible. If a malfunction is evident, a notation shall be made in the comments field of the LIMS.	The laboratory has no control over the actions or inactions of the client prior to, during or after sampling. Not all clients include field data sheets with the documentation sent to the laboratory.

<u>Item</u>	<u>QA Guidance Document</u> 2.12 requirement	<u>SOP</u>	<u>Justification</u>
36	7.10 - 5 Transfer the filter in its filter-handling container to the conditioning chamber.	<p>Upon receipt, filters are placed in the weighroom in such a manner as to allow them to equilibrate with weighroom temperature and humidity.</p> <p>Filters may be transferred from their cassettes/mailers to a different container (Petrislide) at any point between receipt and the first gross weighing.</p>	Clarification of unclear verbiage of method requirement.
37	7.10 – 5 If the filter will receive further analysis, return it to the filter-handling container and note on the container and the laboratory data form that additional analyses are required. Transfer the filter to the laboratory responsible for performing the additional analyses.	<p>If the filter is to receive further analysis, analysts are informed and receive worklist information. Analysts either seek out these filters for further analysis or the weighroom analyst will hand deliver them to the appropriate sample staging area.</p>	<p>Filter data must pass QC requirements prior to the performance of additional analyses. Only after all gravimetric QC has passed and been formally verified by a second individual may filters be transferred to the appropriate lab/staging area for further analyses.</p>
38	7.11 The post-sampling conditioning and weighing shall be completed within 240 hours (10 days) after the end of the sampling period, unless the filter is maintained at 4 °C or below during the entire time between retrieval from the sampler and start of the conditioning, in which case the period shall not exceed 30 days	<p>The client is responsible for ensuring that samples are stored properly until such time as they are shipped to the laboratory. The laboratory shall use all due diligence possible to ensure that samples are conditioned and weighed within 30 days from the end of sampling; in addition, the laboratory shall store filters at or below 4 °C if conditioning cannot be commenced upon receipt.</p>	The laboratory has no control over the actions or inactions of the client prior to, during, or after sampling.

<u>Item</u>	<u>QA Guidance Document</u> 2.12 requirement	<u>SOP</u>	<u>Justification</u>
39	7.13b Air Pollution control agencies shall archive PM2.5 filters for a minimum of 1 year after collection.	Filters are archived for 5 years unless otherwise directed by client.	More stringent than promulgated method.
40	7.12 A filter's post-sampling mass minus its pre-sampling mass is the net mass loading for that filter. Record this value on the laboratory data form.	The proprietary software governing the balance performs this calculation automatically, and records the data in an electronic file, which is then imported into the LIMS.	More stringent record keeping than promulgated method.
41	12.3.2 Figure 4-1 Procurement log. Notes on acceptance/rejection tests.	Filter Tracking logbook tracks all filters that are received by the laboratory in addition to analytical microbalances, and reference standards, but does not include filter cassettes, mailers or Petrislides.	Receipt of cassettes, mailers and Petrislides are organized in the ordering/receiving department.
42	12.3.4 Figure 8-1 Data records from the completed PM2.5 sampler run data sheet as weighing laboratory operation records.	Weighing laboratory tracks filter inspection before shipping, sample validation and post-sample inspection in separate logging systems. Leak, flow rate and field blank checks are not tracked in the weighing laboratory as shown in Table 8-1.	The laboratory has no control over the actions or inactions of the client prior to, during, or after sampling.

<u>Item</u>	<u>QA Guidance Document</u> 2.12 requirement	<u>SOP</u>	<u>Justification</u>
43	4.3.7 ... The mass reference standards should be selected so as to bracket the maximum and minimum expected filter weights ... 7.3 Mass reference standards should be in the range of 100 to 200 mg, given that the mass range [typically is] from 110 to 160 mg.	100 mg and 500 mg weights used.	When this QA Guidance Document was first written, there were a very limited number of manufacturers and tare weights were more consistent between manufacturers. Current manufacturers' filter tare masses may range between 180 – 310 mg depending on manufacturer. Clarification of ambiguity in promulgated method.