

White Paper on the Approach for the Development of a New Federal Reference Method (FRM) for Pb in Total Suspended Particulates (TSP)

On November 12, 2008 EPA substantially strengthened the National Ambient Air Quality Standards (NAAQS) for lead (see 73 FR 66934). EPA revised the level of the primary (health-based) standard from 1.5 micrograms per cubic meter ($\mu\text{g m}^{-3}$) of lead (Pb) to $0.15 \mu\text{g m}^{-3}$ of Pb measured in total suspended particles (TSP) and revised the secondary (welfare-based) standard to be identical in all respects to the primary standard. In conjunction with strengthening the Pb NAAQS, EPA identified the need for states to improve existing lead monitoring networks by requiring monitors to be placed in areas with sources that emit one ton per year (tpy) or more of Pb in urban areas. Pb in TSP (Pb-TSP) remains the indicator for the NAAQS. The FRM for Pb-TSP (40 CFR part 50, Appendix G) was promulgated in 1979 and is based on Flame Atomic Absorption Spectroscopy (FAAS). The need for lowered measurement sensitivity (detection limits) in response to the much lowered NAAQS; advances in measurement technology that have occurred since promulgation of the Pb-TSP FRM; and new methods that are now available with improved precision, detection limits, throughput, and extraction efficiency support the need for a new Pb-TSP FRM.

For this peer review, the approach for developing a new FRM for Pb-TSP is described. This new FRM is intended for use by analytical laboratories performing the analysis of Pb in TSP filters to support the NAAQS. The analytical method will be evaluated with multiple matrices for bias, precision, and repeatability. Sampling procedures for NAAQS-related data collection will continue to be performed in accordance with the FRM described in 40 CFR part 50, Appendix B and therefore are not included as part of this new FRM or the peer review. This document has been prepared by staff from the Ambient Air Monitoring Group in the Office of Air Quality Planning and Standards which will be the focus of the review by the CASAC AAMMS.

Approach for Evaluation of Method Performance

The proposed FRM and the approach for evaluating method performance are based on the following guidance documents and references:

- EQL-0510-191, Determination of Lead in TSP by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Heated Ultrasonic Nitric and Hydrochloric Acid Filter Extraction. <http://www.epa.gov/ttn/amtic/files/ambient/pb/EQL-0510-191.pdf>
- EQL-0710-192, Heated Nitric Acid Hot Block Digestion and ICP-MS Analysis for Lead (Pb) on TSP High-Volume Filters. <http://www.epa.gov/ttn/amtic/files/ambient/pb/EQL-0710-192.pdf>
- Validation and Peer Review of U.S. Environmental Protection Agency Chemical Methods of Analysis, prepared for The EPA Forum on Environmental Measurements (FEM); FEM Document Number 2005-01, October 14, 2005. http://www.epa.gov/fem/pdfs/FEM_MV_doc_final_10-14-2005.pdf

- Guidance for Methods Development and Methods Validation for the RCRA Program; Development and Validation of SW-846 Methods Phase 2: Formal Validation, April 6, 1992. <http://www.epa.gov/osw/hazard/testmethods/pdfs/methdev.pdf>
- EPA's Office of Solid Waste (OSW) compendium of sampling and analytical methods for Evaluating Solid Waste, SW-846 Method 6020A¹, "Inductively Coupled Plasma-Mass Spectrometry", Revision 1, February 2007. <http://www.epa.gov/wastes/hazard/testmethods/sw846/pdfs/6020a.pdf>
- Harper et al., (1983). Simplex Optimization of Multielement Ultrasonic Extraction of Atmospheric Particulates; Anal. Chem., 55 (9), 1553-1557.
- Long et al., (1979). Lead Analysis of Ambient Air Particulates: Interlaboratory Evaluation of EPA Lead Reference Method; APCA Journal, 29, 28-31.

Identification of the Regulatory Need

The need for lowered sensitivity (detection limits) in response to the much lowered NAAQS; advances in measurement technology that have occurred since promulgation of the Pb-TSP FRM; and new methods that are now available with improved precision, detection limits, and extraction efficiency support the regulatory need for a new Pb-TSP FRM. This new method is intended for use by analytical laboratories performing the analysis of Pb-TSP and filters to support the NAAQS. Once a draft FRM is developed, it will be published in the Federal Register for review and comment.

Analytical Approach

Two candidate methods have been identified for extracting lead from filters for subsequent analysis by inductively coupled plasma mass spectrometry (ICP-MS). The candidate methods are 1) EQL-0510-191 which uses a heated ($80 \pm 5^\circ\text{C}$) ultrasonic water bath with 1.02M nitric/2.23M hydrochloric acids and 2) EQL-0710-192 which uses a heated ($95 \pm 5^\circ\text{C}$) graphite hot block with 3.5%(v/v) nitric acid. These methods use equipment that is commonly used by many laboratories, is more practical (use of a single vessel for the entire extraction process and storage), and have improved throughput that make them more efficient and cost effective than the current FRM. ICP-MS has been chosen as the analytical technique because it has much improved sensitivity, selectivity, linear range, and is much more readily available than FAAS in laboratories today.

The FRM is designed to be used for the analysis of Pb in TSP filters. The heated ultrasonic bath and graphite hot block digestion candidate methods are both capable of extracting Pb from the TSP filter media. Pb is generally readily soluble and does not have refractory forms that require a hard digestion such as microwave. The heated ultrasonic bath method uses ultrasound waves to provide mechanical agitation of the sample to help facilitate the extraction of Pb. The heated block digestion method uses sub-boiling heating to extract Pb from the filter strips.

The FRM is currently only intended for the analysis of Pb in TSP filters. Nitric acid alone is sufficient for the extraction of Pb from TSP filters. The ultrasonic extraction method includes hydrochloric acid to allow monitoring agencies to have flexibility for future needs that may

include the extraction of other metals. Hydrochloric acid is needed to aid the extraction of other metals that are not easily brought into solution with nitric acid alone. The FRM will have a caveat to note that the ultrasonic method was not evaluated for other metals and must be evaluated for metals other than Pb before use. The heated block method uses only nitric acid and may not be suitable for metals other than Pb. The same caveat for the ultrasonic method will apply to the heated block method.

The size of the filter strip to be analyzed in the FRM testing will be ¾" X 8" and has an effective sampled area of 5.25 in². A 1" X 8" strip has an effective sampled area of 7 in², but limits the number of strips available for multiple analyses and intra- and inter-laboratory comparisons. Since previously analyzed archived filters will be used for testing, the ¾" X 8" strip size was chosen. The use of ¾" X 8" strips for FRM testing does not preclude the use of 1" X 8" strips as ICP-MS had abundant sensitivity and the evaluation of SRM materials at weights far in excess of sampled filters will be evaluated. Monitoring agencies may use 1" X 8" strips if preferred. Calculations for both filter strip sizes will be included in the FRM.

Pursuant to 40 CFR Part 50, Appendix B, Section 7.1.3, glass fiber (GF) or other relatively inert, nonhygroscopic material is acceptable for use as a filter collection substrate. Therefore, the same analytical approach will be used to evaluate quartz filters. Quartz filters will be sectioned and evaluated in the same manner as GF filters. Because monitoring agencies need to analyze for elements other than lead, quartz filters are included in the FRM evaluation as they have lower backgrounds for several elements other than lead. Filter strips will be used in all FRM testing to assess the impact on the measurement of Pb. Unexposed glass fiber or quartz filter strips will be used for all detection limit determinations, spiking of reference materials, and extraction blanks.

The current FRM in Appendix G provides guidance on the testing of blank filters for Pb prior to sample collection. Appendix G specifies that the blank values be used to correct all lead analysis data unless the values are below the current FRM method LDL of 0.07 µg/m³. EPA currently provides GF filters to monitoring agencies as requested annually. As part of the procurement process, these filters are tested for acceptance by EPA's contractor. The current acceptance criterion for GF filters is 25 µg per filter or 0.01 µg/m³ using a nominal sample volume of 2000 m³; however, the average Pb content of the 50 blank GF filters tested for the filter lot by EPA for calendar years 2008 – 2009 was 2.7 µg/filter or 0.001 µg/m³ using a nominal sample volume of 2000 m³ and the highest result was 4.6 µg/filter or 0.0023 µg/m³. All results were well below the MDL criterion specified in the recently revised Pb method performance test requirements of 0.0075 µg/m³. Laboratories that receive GF filters from EPA and use them for Pb NAAQS analysis will not have to perform blank subtraction. Laboratories that use GF filters not provided by EPA will have to perform lot blank analysis to confirm that blank filters are below the 0.0075 µg/m³ level. In a separate effort, EPA will be revising the GF filter acceptance criteria for Pb to be more in line with the new Pb NAAQS.

The EPA acceptance criterion for Pb in quartz filters is the same as GF filters. Laboratories that use GF or quartz filters (not provided by EPA) will have to perform lot blank analysis to confirm that blank filters are below the 0.0075 µg/m³ level prior to use for sampling.

Quality Assurance (QA)/Quality Control (QC) Requirements

EPA's Office of Solid Waste (OSW) compendium of sampling and analytical methods for Evaluating Solid Waste, SW-846 Method 6020A¹, "Inductively Coupled Plasma-Mass

Spectrometry”, Revision 1, February 2007, serves as the basis for development of the QA/QC procedures for the FRM. The SW-846 compendium of analytical and sampling methods have been evaluated and approved for use in complying with RCRA (Resource Conservation and Recovery Act) regulations. Method 6020A served as a guide for the development of the candidate methods and the combined QC parameters from both methods will be used for the FRM.

The key QC components of the candidate methods are listed below. Specific QC information can be found in the attached copies of the candidate methods.

- calibration coefficient
- calibration curve read back error
- initial calibration verification, ICV
- low level calibration verification, LLCV/QLS
- continuing calibration verification, CCV
- spiked sample extraction
- duplicate sample extraction
- serial dilution of extracted sample
- laboratory blank and fortified laboratory blank
- certified reference material, CRM, in solid form
- internal standard recovery
- reporting limit/quantitation limit (RL/QL)
- method detection limit, MDL

Additional requirements, not included in method 6020A, that are also recommended are:

- A %RSD requirement of 3% for the three sequential replicate readings taken on each sample.
- Setting the extraction sample spike level at 30% of the NAAQS

Intra-Laboratory Method Performance

Both of the candidate methods have demonstrated acceptable analytical performance as FEMs, however not all intra-laboratory performance tests have been conducted by both methods on both glass and quartz filter media. Both methods will be assessed using identical QC criterion to ensure they meet the goals of the FRM testing.

The candidate methods will be assessed by several factors including the analysis of filter strips extracted with solid Standard Reference Materials (SRMs) and analysis of filter strips spiked with NIST-traceable Pb-salt solutions (NIST SRM 3128). Several solid NIST SRMs (Table 1) have been identified that can be used to assess Pb recovery at varying percentages of the NAAQS level of $0.15 \mu\text{g}/\text{m}^3$ based on a nominal volume of 2000m^3 . The SRMs are expected to be recovered within 80 to 120% of the certified value per method 6020A. It is noted that the weights of the SRMs are several times greater than a normal filter loading however the SRMs represent the best available certified materials to assess method performance. The minimum weights as specified by NIST to meet certified values are used. The weights used represent an

extreme challenge to the method. The successful recovery of lead from these SRMs demonstrates method performance at loading levels beyond what would ever be expected on a sampled filter.

Table 1. Candidate SRMs for Performance Testing			
NIST SRM	Minimum Weight (g)	% of NAAQS Level*	$\mu\text{g}/\text{m}^3$
1547 –Peach Leaves	0.150	0.5	0.00078
2709 – San Joaquin Soil	0.250	17	0.026
2583 – Indoor Dust	0.250	86	0.129
2582 – Powdered Paint	0.200	166	0.250

*Based on a ¾”X8” filter strip and a nominal volume of 2000m³

Intra-laboratory performance will be tested before the inter-laboratory testing takes place. If procedural changes are identified during the inter-laboratory study, it may be necessary to repeat some aspects of the intra-laboratory study. The intra-laboratory analysis will consist of the analysis of solution spiked filter strips, solid SRMs combined with filter strips, and the analysis of field sampled filters. Filter strips spiked with NIST Pb solutions will be used for MDL determination and to demonstrate method accuracy at selected percentages of the NAAQS limit. The intra-laboratory method performance testing will be applied to both glass fiber and quartz fiber filters.

This project will use archived GF filters that have already had at least one strip removed prior to testing. This leaves a maximum of 11 strips per filter for analysis and more likely only 9 depending on where the first filter was cut. The size of the filter strip to be analyzed will be ¾” X 8” in order to generate 8 strips for inter-laboratory testing. Four laboratories analyzing two strips each yields n=8.

The stability of lead on the sampled filters will not be evaluated. The GF and quartz filters are stored folded over on the exposed area in an envelope in climate controlled space. Lead is not a volatile element and once the particle is embedded on the surface of the filter there is no reason to believe that there will be any loss of the element. Only the stability of the sample extracts will be evaluated. A set of extracts consisting of spiked filter strips and sampled field filters will be extracted using both methods and both filter matrices. The initial results following extraction will serve as T=0. The same extracts will be stored at ambient room temperature (~23°C) for a period of two months and then re-measured to assess the extract stability. This procedure will be repeated two additional times to assess the extract stability over a six month period.

Analytical Bias, Precision, and Repeatability

Analytical bias will be assessed using spiked filter strips at 30, 100, and 250% of the NAAQS limit; 0.045, 0.15, and 0.375 $\mu\text{g}/\text{m}^3$, respectively. The average Pb concentration for each sample is determined by averaging the concentrations calculated from the three nonconsecutive analyses. The bias between the measured Pb concentration for each spiked filter strip and the true Pb concentration is determined by subtracting the known concentration from the average measured

concentration, dividing by the known concentration, and multiplying by 100. The bias between the measured concentration and the known concentration for each sample is targeted at 10%. If exceeded, corrective action must be taken to determine the source(s) of imprecision, and the determinations must be repeated.

The test for precision is performed on field samples. Seven field sample filters of each filter type will be used for precision. The precision of the analysis (in percent) for each filter and for each method is defined as the maximum minus the minimum divided by the average of the three concentration values and multiplying by 100. The method precision value is targeted at 15 percent. If exceeded, corrective action must be taken to determine the source(s) of imprecision, and the determinations must be repeated. Precision testing was done for both methods using GF filters as part of the FEM process; however quartz filters will need to be evaluated.

Effects of Interferences

Lead, in general, does not suffer from polyatomic interferences presented by hydrochloric acid during ICP-MS analysis because of its high mass (M). Pb is measured as the sum of M206, M207, and M208 because of naturally occurring differences in isotopic abundance. Low mass elements like arsenic (^{75}As) suffer from polyatomic interferences that form in the plasma like arsenic chloride ($^{40}\text{Ar}^{35}\text{Cl}$). These polyatomic interferences can be mitigated by the use of collision/reaction cells. For the analysis of Pb, the use of a collision/reaction is not required because there are no polyatomic interferences for Pb. Some elements have multiple isotopes that overlap each other like cadmium (^{114}Cd) and tin (^{114}Sn). Since both have the same mass to charge ratio, a quadrupole ICP-MS would not be able to distinguish between these elements. Pb has no isobaric interferences at any of the measured masses. The interference of greatest concern would be physical interferences from undissolved particulate matter or extremely high concentrations of dissolved solids. Internal standards are capable of correcting for some change in viscosity but cannot be used to compensate for drastic changes in nebulization efficiency. Internal standard recovery is addressed in Method 6020A and the same guidelines will be employed in the FRM development. Any sample that has an internal standard response below 70% must be diluted fivefold and reanalyzed. If the first fivefold dilution does not yield acceptable internal recovery, the procedure must be repeated.

Method Detection Limit (MDL), Reporting Limits, and Range

The approach follows the recommendation of the EPA Forum on Environmental Measurements method validation guidance where sensitivity can be evaluated from the data and information provided by the MDL, Reporting Limit (RL), precision and other performance metrics evaluated as part of the method development process.

On November 12, 2008 when EPA revised the NAAQS for lead, revisions were also made to the performance testing requirements to add an MDL requirement of 5% of the NAAQS level or $0.0075 \mu\text{g m}^{-3}$. Method sensitivity is also influenced by the sample collection volume and instrument detection limit. ICP-MS instrumentation can easily achieve part per trillion (ppt) level detection limits in clean solutions. The candidate extraction methods in combination with the ICP-MS analysis method have been evaluated to determine what MDL can be achieved using GF filter strips or Teflon filters spiked with NIST-traceable solutions. Quartz filter MDL determinations will be conducted following the same procedure used for glass fiber filters.

The method detection limit (MDL) have been determined for GF filters by spiking TSP strips with the appropriate amount of NIST-traceable lead solution so that the resulting extraction will be between 2-5 times the expected detection limit for the method. The MDL samples were taken through the entire preparation procedure prior to analysis. The standard deviation of spiked strips was used to calculate the MDL per 40 CFR, Part 136, Appendix B. MDL results for both extraction methods are shown in Table 2. The MDL for quartz filters will be determined by the same method used for GF filters.

The reporting limit (RL) will be determined by the lowest standard in the calibration curve or the lower level calibration verification (LLCV) per 6020A, Section 10.4.3. All values measured or detected will be reported; however, values below the RL will be flagged as estimates. The upper working range for the methods will be determined by the highest standard in the calibration curve. Any samples that exceed the highest standard concentration must be diluted so that the diluted concentration falls within the standard curve range.

The working range of both methods has been demonstrated from 0.00120 to 0.480 $\mu\text{g}/\text{m}^3$ based on a nominal sample volume of 2000 m^3 . The working range is based on the concentrations of the calibration standards, typically 0.005 $\mu\text{g}/\text{mL}$ to 2.0 $\mu\text{g}/\text{mL}$. The working range may be increased to bracket higher ambient sample concentrations if expected by including higher concentration calibration standards. The lower limit of quantification is well below the MDL criterion of 0.0075 $\mu\text{g}/\text{m}^3$. The method range will be evaluated and adjusted if needed to allow the analysis of field samples without additional dilution.

Heated Ultrasonic Bath ¹		Graphite Block Digestion ³	
MDL-1	0.0000702	MDL-1	0.000677
MDL-2	0.0000715	MDL-2	0.000732
MDL-3	0.0000611	MDL-3	0.000715
MDL-4	0.0000587	MDL-4	0.000875
MDL-5	0.0000608	MDL-5	0.000889
MDL-6	0.0000607	MDL-6	0.000863
MDL-7	0.0000616	MDL-7	0.000858
		MDL-8	0.000797
		MDL-9	0.000788
Average	0.0000635	Average	0.000799
Standard Deviation	0.0000051	Standard Deviation	0.0000775
MDL²	0.000016	MDL⁴	0.000224

¹ Assumes 2000 m^3 air sample.

² MDL is 3.143 times the standard deviation of the results for seven consecutive sample replicates analyzed.

³ Assumes 2400 m^3 air sample.

⁴ MDL is 2.896 times the standard deviation of nine replicates.

Inter-laboratory Testing

The inter-laboratory study is designed to test the reproducibility of the method at multiple laboratories and not to evaluate the performance of an individual laboratory. The inter-laboratory

tests are designed to assess the performance of the methods when carried out at multiple laboratories. The data generated will be used to perform statistical calculations and therefore no set acceptance criterion for results has been listed. The inter-laboratory study will generally follow the approach described in Long et al., (1979)⁵. At least four laboratories will need to participate in the study. All laboratories will receive a copy of the standard operating procedure (SOP) for the FRM and will be asked to analyze strips (non-sequentially in triplicate) from ambient air filters and spiked filter strips for Pb using all extraction options and the ICP-MS analysis method in the SOP. The data will be summarized and compared statistically to estimate the expected performance of the method when applied subsequently by other laboratories. Precision and, for the spiked filters, bias will be computed according to the approach described by Long et al., (1979).

Spiked Filters

Each laboratory will receive two sets of filter strips spiked at three different levels to assess bias and precision for lead across laboratories. One set of filter strips will be processed by the heated graphite block method and the other by the heated ultrasonic method. It is expected that each lab will achieve bias $\leq 10\%$ and precision $\leq 15\%$ for the spiked filter strips. Each laboratory will report the average lead concentrations ($\mu\text{g}/\text{m}^3$) for three replicate analyses of each sample. Intra- and inter-laboratory precision and bias will be computed for each filter matrix and processing method.

Sampled Filters

From archived filters with concentrations ranging from 0.05 to 0.4 $\mu\text{g}/\text{m}^3$, fourteen sampled filters of each matrix will be selected and split into two groups of seven and sectioned to produce 8 strips, 4 pairs, per filter. Each pair will consist of adjacent strips from the same filter. The pairs will be randomly distributed to the participating laboratories for extraction and analysis. Each laboratory will receive 7 pairs for the heated block method and 7 pairs for the heated ultrasonic method. All laboratories will analyze pairs from the same filters for each method. Each laboratory will analyze the method extracts in triplicate and report and the individual results, as well as the average and standard deviation for each sample.

Standard deviations and 95% confidence intervals will be computed at each filter concentration for each method (across labs) and overall (across labs and methods) to evaluate the expected inter-laboratory and inter-method variability. The 95% confidence interval represents the range of values between which one would expect to find the means of measurements derived from a group of samples 95% of the time. In this particular application, it is only an estimate, albeit a reasonable estimate, of the range of means within which one might expect to find the results obtained by other laboratories. Method precision and confidence interval assumes that the laboratories participating in the inter-laboratory testing represent the typical skill level for all laboratories that would perform testing under the FRM.

References

1. Method 6020A – Inductively Coupled Plasma Mass Spectrometry. U.S. Environmental Protection Agency. Revision 1, February 2007
2. Validation and Peer Review of U.S. Environmental Protection Agency Chemical Methods of Analysis, prepared for The EPA Forum on Environmental Measurements (FEM); FEM Document Number 2005-01, October 14, 2005.

3. Guidance for Methods Development and Methods Validation for the RCRA Program; Development and Validation of SW-846 Methods Phase 2: Formal Validation, April 6, 1992.
4. Harper et al., (1983). Simplex Optimization of Multielement Ultrasonic Extraction of Atmospheric Particulates; *Anal. Chem.*, 55 (9), 1553-1557.
5. Long et al., (1979). Lead Analysis of Ambient Air Particulates: Interlaboratory Evaluation of EPA Lead Reference Method; *APCA Journal*, 29, 28-31.

Test Matrix for the Development of a Pb FRM

Test Description	Criteria
ICP-MS Calibration and Quality Control	
Calibration Range	Demonstrate working range of the method. 1ppb to 2000ppb as measured (0.00023– 0.480 $\mu\text{g}/\text{m}^3$)
Correlation Coefficient	0.998 or greater
Calibration Standard Readback	$\pm 10\%$
ICV,CCV	90-110% Recovery
LLCV	70-130% Recovery
ICB, CCB	$<$ Lower limit of quantitation
%RSD of replicate readings	Demonstrate the %RSD of the three consecutive replicate reads per sample analysis are $\leq 3\%$.
Intra-laboratory Method Performance	
MDL: n=7 replicates per 40CFR, Part 136, Appendix B	Unexposed filter strips spiked with NIST traceable solutions to determine the MDL. Must be below 5% of the NAAQS limit, 0.0075 $\mu\text{g}/\text{m}^3$.
Low Level Quality Control Sample (LLQC)	Recovery: 70-130% Filter strip spiked at 30% of the NAAQS level based on a nominal flow rate of 2000 m^3 for n=7
NIST 1547 @ 0.87 $\mu\text{g}/\text{g}$ (~0.5% NAAQS) 150mg combined with unexposed filter strip.	Recovery : 80 - 120%
NIST 2709 @ 17.3 $\mu\text{g}/\text{g}$ (~17% NAAQS) 250mg combined with unexposed filter strip.	Recovery : 80 - 120%
NIST 2583 @ 85.9 $\mu\text{g}/\text{g}$ (~86% NAAQS) 250mg combined with unexposed filter strip.	Recovery : 80 - 120%
NIST 2582 @ 208 $\mu\text{g}/\text{g}$ (~166% NAAQS) 200mg combined with unexposed filter strip.	Recovery : 80 - 120%
Analytical Bias and Precision NIST SRM 3128	n=3 spiked filter strips (low, medium, high) GFF and Quartz. Triplicate, non-sequential analysis. Bias: $\leq 10\%$ Precision: $\leq 15\%$
Method Comparison – field filters, n=7	Section and extract 3 strips for each method from each filter. Triplicate, non-sequential analysis. Intra-Method Precision: $\leq 15\%$ Inter-Method Precision: $\leq 15\%$
Extract Storage Stability	3 unexposed filter strips spiked with NIST traceable solution and 3 sampled filters. Analyze at T=0 Store capped at ambient temperature. Analyze at T=2 months Analyze at T=4 months Analyze at T=6 months T=2, T=4, and T=6 time points $\pm 10\%$ of T=0
Inter-laboratory Method Performance	
Spiked filter strips, Glass and Quartz filters	n=3 spiked filter strips (low, medium, high) for GFF and Quartz. Triplicate, non-sequential analysis.
Sampled filter strips, Glass and Quartz filters	n=14 (7 pairs) per matrix per method. Triplicate, non-sequential analysis