Method 201A and 202
Best Practices to Reduce Blanks

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Draft
December 31, 2013

Final
October 27, 2015

EPA Contract No. EP-D-11-006
Work Assignment 3-07

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PM	Particulate matter
EPA	U.S. Environmental Protection Agency
TSP	Total suspended particulate
$PM_{2.5}$	Particulate matter less than or equal to 2.5 micrometers in diameter
CPM	Condensable particulate matter
ERG	Eastern Research Group, Inc.
%RSD	Percent relative standard deviation
FTRB	Field train recovery blank
g	Grams
QC	Quality control
DQI	Data quality indicators
ND	Non-detect
SD	Standard deviations
UPL	Upper prediction limits
PTFE	Polytetrafluoroethylene

1.0 EXECUTIVE SUMMARY

Particulate matter (PM) is one of the six common air pollutants that the U.S. Environmental Protection Agency (EPA) sets National Ambient Air Quality Standards for as required under the Clean Air Act. Historically, EPA first regulated total suspended particulate (TSP), followed by PM that is less than 10 micrometers in diameter (PM_{10}), which is inhalable and harmful to human health, and then particulate matter less than or equal to 2.5 micrometers in diameter ($PM_{2.5}$). The $PM_{2.5}$ particulate matter consists of both filterable PM and condensable particulate matter (CPM). Method 201A collects filterable PM using a filter and a set of cyclones to separate the PM_{10} and $PM_{2.5}$ from larger sized particles. Method 202 uses a condenser, dry impingers, and a filter to collect CPM. CPM is not captured by a filter at stack conditions, but forms solid or liquid particulate matter immediately after discharge from the stack. Method 202 is combined with Method 201A or other filterable PM methods for source testing.

In December 2010, amendments to Methods 201A and 202 were promulgated. The updated Method 201A includes an additional cyclone to provide a measurement of $PM_{2.5}$. $PM_{2.5}$, also known as fine particulate, is of interest because it is believed to pose the greatest health risk of PM. The updated Method 202 includes revised sample collection and recovery procedures to reduce the formation of reaction artifacts that could lead to inaccurate measurements of CPM. The updates to Method 202 also eliminated most of the hardware and analytical options previously available, which increased the precision of the method and improved the consistency in the measurements obtained between source tests performed under different regulatory authorities. Several performance-based criteria were written into the methods to provide some flexibility to stationary source test teams. After promulgation, states, local agencies, facilities, and source testers provided feedback indicating that clarification of the procedures in these updated methods is necessary to ensure that they are used effectively. In addition, Method 202 field train recovery blank levels greater than the allowable 2.0 milligrams (mg) limit established in the method have been reported. Issues of primary concern for elevated blank concentrations are the contributions to the total field sample results from filters, reagents, and sampling trains, the probe extensions in particular. The blank contribution to sample mass needs to be very low to ensure that results for the CPM measurement from Method 202 source tests are attributable to the source and not to the materials used in the sample collection, recovery, and analysis.

In order to provide direction and greater clarity, Eastern Research Group, Inc. (ERG), under EPA Contract # EP-D-11-006, Work Assignment (WA) 3-07, identified several best practices associated with the implementation of Methods 201A and 202. ERG identified these best practices through a survey of source testing firms and laboratory evaluations of the filters, reagents, and sampling train glassware used in conducting Method 201A and 202 sampling. This document summarizes the best practices determined from the surveys and laboratory evaluations.

The project elements were conducted as four tasks. The tasks were as follows:

- Task 2, Method 201A and 202 Best Practices Surveys
- Task 3, Method 202 CPM Filter Evaluation
- Task 4, Method 202 Reagent Evaluation
- Task 5, Method 202 Sampling Train Glassware Evaluation.

ERG selected the source testing firms surveyed in Task 2 based on the expertise the firms have in these methods, as well as their ability to control blank levels. ERG evaluated filters in Task 3 to determine whether CPM filters can meet the residual mass specification in Method 202 and the potential contribution to the field sample mass concentration. Under Task 4, ERG evaluated whether reagents used in Method 202 sample recovery could meet the residual mass specification and determined the potential contribution of reagents to the sample mass concentration. Task 5 was designed to evaluate the ability to clean Method 202 sampling trains to achieve sufficiently low blank results and evaluate the potential contribution of individual components of the sampling train to the sample mass concentration. The overall technical objective of WA3-07 was to identify best practices for Methods 201A and 202 that minimize residual mass contribution to field samples and reduce blank results to the allowable limit. This effort did not evaluate the potential for contamination in the field or field recovery techniques that contribute to elevated field train recovery blank concentration.

In Task 2, ERG surveyed three source testing firms that have the proven capability of achieving field train recovery blanks below the Method 202 limit. The survey included questions regarding the materials used and the procedures employed to control blank levels in Method 202 source testing. ERG used responses from the survey to determine what materials and procedures result in low field train recovery blanks and make suggestions on the best practices for the implementation of Methods 201A and 202.

In Task 3, ERG evaluated different types of filters used to collect CPM in Method 202 sampling to determine their residual blank levels. ERG focused on the evaluation of a single commercial lot of filters of each type and not on the variation between lots of filters. The three different types of filters were Teflon® membrane filters, Teflon® membrane filters backed with hydrophobic media, and Teflon® membrane filters backed with hydrophilic media. ERG obtained one box of each type of filter and processed the filters as received from the vendor without any additional preparation. Ten filters of each type were processed and analyzed according to the procedures of Method 202. ERG determined the residual mass concentrations, the averages of the residual masses, and the percent relative standard deviations (%RSD) of the residual mass measurements for each filter type.

In Task 4, ERG evaluated different reagent grades of hexane and water used in the recovery and extraction of Method 202 samples to determine their residual blank levels. The three grades of water were ASTM Type II quality ion exchange water, ultrafiltered water, and water distilled in glass. Three grades of hexane based on the manufacturer’s stated residual upon evaporation values were identified. The three grades of hexane represented one that was clearly above the reagent blank limit specification of the method, one that did not meet the specification, but that was close to the limit, and one that was below the limit. Reagents were processed as received from the vendor without any additional preparation. Ten 450 milliliters (mL) aliquots of each grade of each reagent were processed and analyzed according to the procedures of Method 202. The residual mass concentrations, the averages of the residual masses, and the %RSDs of the residual mass measurements were determined for all three grades of each reagent. Reagent aliquots of 450 mL were used because that volume is three times the volume standard 150 mL required for reagent blanks in Method 202.

In Task 5, ERG investigated the capability to achieve sampling train blank results at or below the allowable 2.0 mg field train recovery blank limit in Method 202. ERG evaluated sampling train glassware commonly used in Method 202 source testing to determine residual blank levels and the contribution to the blank from individual components of sampling trains. ERG evaluated three complete Method 202 sampling trains that have been used in the field to test source emissions. The trains were cleaned and prepared as they would be for deployment in the field, including baking at 300°C. ERG recovered the sampling trains according to the proof blank procedures of Method 202, the CPM filters were not included in the evaluations. One train was then recovered two additional times for three blank samples from that train and a total of five blank samples of full Method 202 sampling trains. A different train was separated into its components; the probe extension, the condenser, the impingers, and the CPM filter housing. Each component was recovered four times. ERG then processed and gravimetrically analyzed all the blank samples generated. ERG determined the residual mass concentrations, the residual mass averages, and the %RSDs of the residual mass measurements. The %RSD provided precision, which is a measurement of the variability among measurements for each test.

Method 201A and 202 field sample results are determined by the gravitational analysis of tared weigh pans containing the residual masses from the sample train recovery rinses and the filter extracts, or in the case of Method 201A, the filters themselves. Method 201A collects filterable PM using a filter and a set of cyclones that separate the PM_{10} and $PM_{2.5}$ from larger sized particles. Method 202 uses a condenser, dry impingers, and a filter to collect CPM. Method 201A samples consist of the filterable PM filter and the reagent rinses of the stainless steel nozzle and combined cyclone and filter sampling head. Method 202 samples consist of the CPM filter and the reagent rinses of the sample portion of the train. See Figure 1, the combined M201A/202 sampling train configuration schematic.

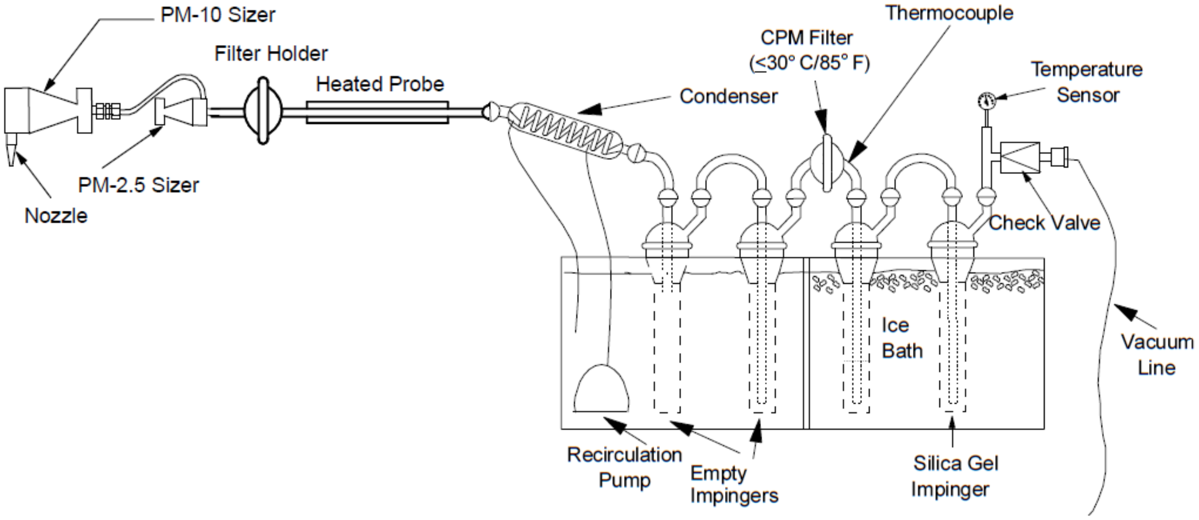

Figure 1. Schematic of Combined Method 201A & 202 Field Sampling Train

Methods 201A and 202 include several different types of blanks, which are referenced throughout this report and need to be differentiated in order to avoid confusion. Of primary concern is the field train recovery blank (FTRB) for Method 202. This blank is generated using the Method 202 sample recovery procedures on a sampling train that has been used for one or two test runs, assembled as it would be for testing, including the CPM filter, with an addition of 100 mL of water in the first impinger, and purged with nitrogen. The Method 202 field sample weight is corrected with the FTRB result or 2.0 mg, whichever is less. Method 202 also includes a field train proof blank (proof blank), which is required if the sampling train glassware is not baked. This field train proof blank is generated from a sampling train prior to sampling that is assembled as it would be for testing, including the CPM filter, without any additional water or a nitrogen purge. Laboratory reagent blanks are evaluations of the reagents as received from the vendor and are not reported by either method, but are suggested as a way to verify that the residual mass of the reagent meets the method performance specifications. Field reagent blanks are taken from the wash bottles used during source testing and verify that the reagents have not been contaminated. An acetone field reagent blank is required for Method 201A, and field reagent blanks are suggested for Method 202.

1.1 Gravimetric Analysis Procedures

ERG used the following gravimetric analysis procedures to determine the residual mass concentrations for all samples generated in Tasks 3, 4, and 5. Samples were evaporated to dryness in numbered aluminum weighing pans that were tared prior to use. ERG completed gravimetric analysis in the temperature and humidity controlled balance room at ERG's laboratory using a Sartorius BP211D five place analytical balance, sensitive to 0.00001 grams (g). The calibration was checked daily using ASTM Class S weights prior to conducting measurements. Each day that gravimetric analysis was performed the temperature, relative humidity, and the balance calibration check were recorded in the balance room lab notebook. All measurements were recorded on the balance room lab notebook page for the day they were taken. All pan tares and sample measurements were weighed to constant weight. Constant weight is defined as a change of less than or equal to 0.1 mg, between two consecutive weighings, with at least six hours between weighings. Nitrile gloves were worn during handling and gravimetric analysis of all samples.

ERG numbered and tared the weighing pans before they were used for samples. Once numbered the pans were desiccated for at least 24 hours, they were weighed to constant weight to determine the tare weight. The pans were kept in their desiccators until they were ready to be weighed and then placed directly on the balance. A timer was used to verify that the reading on the balance was stable and did not change for at least 20 seconds. Once a pan was placed on the balance, it typically took between two and five minutes to reach a stable value. After reaching a stable value the sample pan was removed from the balance and the balance was allowed to return to zero. If the balance did not return to zero, the sample was reweighed. Once a stable value was reached and the balance returned to zero after the sample was removed, it was recorded in the balance room lab notebook. All tare weights and the initial and all subsequent weighings until constant weight was achieved were recorded in the balance room lab notebook along with the conditions and balance calibration check information for that day.

After the sample pans evaporated to dryness, they were desiccated for at least 24 hours prior to gravimetric analysis. The sample pans were kept in their desiccators until they were ready to be weighed and then placed directly on the balance. A timer was used to verify that the reading on the balance was stable for at least 20 seconds and if the balance returned to zero after the sample was removed it was recorded in the balance room lab notebook. Sample pans were weighed to constant weight and then received an additional weighing after constant weight was achieved. This final weighing was taken at least 6 hours after the weighing that established constant weight. Sample weights are reported as:

- The measurement that established constant weight (Constant Weight) and
- The average of the measurement that establishes constant weight and the additional measurement (Average Weight).

All sample weights were recorded in the balance room lab notebook with the conditions and balance calibration check information for that day.

1.2 Quality Control

The data quality objective for the laboratory evaluations conducted in Tasks 3, 4, and 5 was to generate accurate and representative data of the residual mass contributions of filters, reagents, and sampling train glassware of Methods 202 source sampling. The overall objective was attained by measuring the residual masses of filter blanks, solvent blanks, and glassware blanks using the procedures in Method 202. Low blank results confirm that the residual mass contributions of filters, reagents, and sampling train glassware result in low CPM residual mass contributions to field samples. Sample handling was a critical component in maintaining high quality in all tasks for this project and ERG minimized the potential for measurement bias through elevated attention to detail while conducting associated analytical procedures. All sample containers were labeled with the unique sample identification, date, and operator initials. All sample recoveries, extractions, and analyses adhered to the procedures in Method 202 as written.

ERG evaluated reagent blanks for the reagents used in the sample preparation, recovery, and processing for each of the laboratory experiments. For reagent blanks, 200 mL of each reagent was taken directly from their actual wash bottles and collected in sample jars. The reagents were then quantitatively transferred into beakers by pouring the reagent and then rinsing the sample jar into the beaker with the same reagent. The beakers were placed in the fume hood for evaporation. Following the sample processing procedures from Method 202, the water reagent blank (RBW) beaker was placed on a hot plate to expedite evaporation. Once the reagent blanks had evaporated to approximately 10 mL and the water beaker was allowed to cool, they were quantitatively transferred into tared, numbered weighing pans, desiccated, and gravimetrically analyzed according to the same procedures used for samples.

A dust pan was used in all three tasks to verify that any dust that could have settled in the pans during sample evaporation was not a significant source of bias in the samples. Each task had a dedicated dust pan for that task. A tared, numbered weigh pan was set out in the hood with sample pans every time that they were exposed. When samples were not being evaporated in pans, a watch glass was placed over the dust pan, so that it was only exposed when samples were exposed. After all sample and reagent blank pans associated with the dust pan were evaporated to dryness, the dust pan was placed in a desiccator in the balance room for gravimetric analysis.

For the gravimetric analysis, ERG followed the quality control (QC) criteria and procedures specified in ERG's SOP for Gravimetric Determination for Particulate Emissions Measurements (ERG-MOR-002), see Appendix. To ensure that mass measurements from gravimetric analysis did not add excess uncertainty to the test results, gravimetric analysis was performed at ERG's laboratory using a Sartorius BP211D analytical balance, which is sensitive to 0.00001 g. The balance is calibrated annually to manufacturer's specifications with NIST traceable weights. Daily balance calibration checks were measured using ASTM Class S weights. A deviation of more than ±0.0002 grams at any weight level required that the zero be checked or balance maintenance be performed. Acceptable deviation for a five place balance is ≤ 0.00005 g. The daily temperature, relative humidity, and balance calibration check were recorded during gravimetric analysis. Mass measurements were performed in a temperature and humidity controlled balance room that meets the specifications for filter weighing in Method 5, which requires that the temperature be maintained at 20 ± 5.6 °C and the humidity be recorded. The temperature and humidity of the room is controlled using a Data Aire, Inc. Mini Data Alarm Processor II, which is set to maintain a temperature of 70°F (21°C) and a relative humidity of 50%. The actual temperature and humidity was monitored and documented using an Omega OM-CP-PRHTEMP2000 data logger.

The acceptance of mass measurements for all samples was dependent on achieving a constant weight during gravimetric analysis. The constant weight criteria used was a change of less than or equal to 0.1 mg, between two consecutive weighings, with at least 6 hours between weighings. Data quality indicators (DQI) included determining residue mass for 90% of each set of samples. All negative results are reported as non-detects (ND). Averages, standard deviations (SD), detection limits (calculated as three times the SD), %RSD, and upper prediction limits (UPL) of mass measurements of the data sets were calculated. ERG set target of 95% of sample results falling at or below the calculated UPL value for measurements in this project. ERG also used the UPL calculation to evaluate to what degree the different materials contribute to the total Method 202 field sample result. Sample weights were recorded as the measurement that established constant weight (Constant Weight) and also as the average of the measurement that establishes constant weight and one additional measurement (Average Weight). The precision for both methods of sample weighing was evaluated by calculating and comparing the %RSDs.

1.3 Findings

The results were used to evaluate the mass concentration levels of reagent blanks, filter blanks, and sampling train blanks that are achievable using commercially available materials and to suggest media and procedures for use in these methods. The findings from these tasks demonstrate that the method-defined blank limits for the filters, reagents, and sampling trains are achievable, the FTRB limit is achievable, and it is possible to limit the residual mass contributions to the field sample result from the materials used in the sample collection, recovery, and analysis. In order to accomplish these things special attention must be paid to the selection of materials and to the procedures used in these methods. The selections of specific materials or materials with specific properties that limit residual mass contributions are identified as best practices in this report. Likewise, procedures or techniques that aid in the control of FTRB results and residual mass contributions are identified as best practices.

2.0 METHOD 201A AND 202 BEST PRACTICES SURVEYS

Under WA 3-07, Task 2 and with direction from the EPA work assignment manager (WAM), ERG surveyed three source testing firms that have demonstrated acceptable performance controlling blank levels by applying best sampling and analysis practice expertise to Methods 201A and 202. Throughout this document they will be referred to as Firms A, B, and C.

- Firm A reported achieving Method 202 field train recovery blank results that are usually between 0.9 and 1.4 mg, very rarely above 2.0 mg, performing Method 201A and 202 testing primarily on gas turbines, biomass boilers, coal boilers, cement kilns, oil refinery units, gas and diesel internal combustion engines, glass furnaces, and various other manufacturing processes.
- Firm B reported achieving Method 202 field train recovery blank results that are typically 1.5 mg, testing gas turbines primarily, but also some utility boilers and solid fuel boilers.
- Firm C uses subcontractors to perform source testing for gas-fired and other very low PM sources and has extensive knowledge applying these methods.

These firms were provided with a questionnaire to collect information on the materials and procedures they use to control blank levels for these methods. From this information, ERG identified several best practices for conducting accurate source testing with low field train recovery blank results for Methods 201A and 202.

2.1 Materials

The materials used in Methods 201A and 202 can be one of the most significant contributors to the field blank results. These materials include the components of the sampling train itself, the filters used in the sampling train, the reagents used for sample recovery, and the nitrogen used for the post-sampling purge in Method 202. In order to meet the field train recovery blank criteria, the contribution from the materials needs to be as low as possible. The sampling train consists of stainless steel and glass components that are standard for isokinetic stack sampling hardware. The contribution to the blank from the sampling train is a combination of the purity of the recovery reagents, train preparation, and the sample recovery. Materials are addressed in this section and procedures are addressed in the next section. Table 1 summarizes the materials used by the three firms that responded to the questionnaire.

One filter used in each of these test methods and each filter must meet their respective method specifications. Method 201A specifies the use of a nonreactive, nondisintegrating glass, quartz, or polymer filter that does not have an organic binder and has an efficiency of at least 99.95% on 0.3 micrometer (µm) doctyl phthalate particles. Method 202 specifies the use of a nonreactive, nondisintegrating polymer filter that does not have an organic binder, does not contribute more than 0.5 mg of residual mass to the CPM measurements, and has an efficiency of at least 99.95% on 0.3 micrometer doctyl phthalate particles. In addition they must be able to withstand the rigors of the source test and not degrade during the sample recovery. For Method 202, that includes sonication and solvent extraction. Methods 201A and 202 include the use of reagents for the sample recovery from the sampling hardware and require that the residues of evaporations for all reagents used in both methods be 1.0 mg/L or less.

Table 1. Best Practice Survey Materials

Firm	M201A Filter	M202 Filter	Acetone	Water	Hexane
A	47 mm Millipore Glass Fiber filters	90 mm Sartorius PTFE Membrane 1.2 µm pore size [Blank: < 0.5 mg/filter]	Capillary GC/GCMS grade [Blank: < 1.25 mg/L.]	ACS grade ASTM D1193 Type 1 [Blank: < 1.8 mg/L]	Honeywell non-spectrophotometric grade [Blank: < 1.35 mg/L]
B	45 mm Whatman 934-AH Glass Microfiber	47 mm Pall Zeflour 3.0 µm pore size PTFE Membrane [Blank: < 0.2 mg/filter]	GC Resolv grade [Blank: 0.05 mg/L]	Deionized [Blank: 0.05 mg/L]	GC Resolv grade [Blank: 0.05 mg/L]
C	High Purity Glass Fiber for gravimetric analysis High Purity Quarts Fiber for chemical analysis	100% PTFE Membrane 1.2 µm pore size (90 mm typically) [Blank: < 1.5 mg/filter]	Histology or HPLC grade [Blank: < 2.5 mg/L]	HPLC grade or reagent grade water that is double deionized, activated carbon filtered, UV irriated, and membrane filtered [Blank: ~3.66 mg/L]	GC, pesticide non-spectrophotometric grade

Firm A

Firm A reported using 47 mm Millipore Glass Fiber filters for Method 201A source testing and Sartorius 90 mm 1.2 µm pore size PTFE membrane filters with a typical blank result of < 0.5 mg for Method 202. The Method 202 filter housing includes a PTFE frit and backup filters are not used. These filters for are used straight out of the box with no pre-test preparation. Firm A recovers Method 201A samples using Capillary GC/GCMS grade acetone with a reagent blank of < 0.25 mg per 200 mL or < 1.25 mg/L. Firm A uses ACS grade ASTM D1193, Type 1 water, with a reagent blank of < 0.36 mg per 200 mL or <1.8 mg/L, and Honeywell non-spectrophotometric grade hexane, with a reagent blank of < 0.27 mg per 200 mL or <1.35 mg/L, as the sample recovery reagents for Method 202 (Note the Method 202 reagent blank results were reported by Firm A without a volume, so 200 mL, which was the volume given for the acetone blank, was used to calculate mass per volume concentrations). The post-sampling purge is performed using glass fiber thimble filtered AirGas CEMS grade nitrogen. Dedicated PTFE transfer lines are used to deliver the nitrogen for the purge. Viton O-rings are used for the sampling train impingers and are replaced when signs of wear occur or if a pre-test leak check does not pass.

Firm B

Firm B reported using 45 mm Whatman 934-AH glass microfiber filters in their Method 201A sampling trains. For Method 202, Firm B uses Pall Zeflour 47 mm 3.0 µm pore size PTFE membrane filters, which have a typical blank result of < 0.2 mg. The Method 202 filter housing includes a PTFE frit and backup filters are not used. Filters for both methods are used straight out of the box with no pre-test preparation. GC Resolv grade acetone with a reagent blank of 0.05 mg/L is used for Method 201A sample recovery. Deionized (DI) water and GC Resolv grade hexane with reagent blanks of 0.05 mg/L for both are used for Method 202 sample recoveries. The DI water is generated by passing purchased distilled water through a Barnstead ion exchange resin column. The post-sampling purge is performed using filtered CEMS grade 5.1 nitrogen. PTFE transfer lines, which are visually inspected for cleanliness, are used to deliver the nitrogen for the purge. Viton O-rings are used for the sampling train impingers and are replaced when signs of wear are observed.

Firm C

Firm C specified the use of high purity glass fiber filters in Method 201A source testing trains when only gravimetric analysis is to be performed. An acid wash is required for these filters if the PM concentration is low and SO_2 is present in the source. High purity quartz fiber filters are specified when chemical analysis is to be performed. A backup glass or quartz filter is sometimes used if sulfur dioxide or sulfuric acid vapor adsorption may be significant. In Method 202 sampling trains, 100% PTFE membrane filters (which are hydrophobic) with a 1.2 µm pore size, typically of the 90 mm diameter size are used. Blank results for Method 202 filters are < 1.5 mg. Filters that are 100% PTFE and free of all non-PTFE components are required by Firm C, which has found that polymethylpentane components are particularly problematic due to their partial solubility in hexane. For Method 201A sample recovery of low level sources, Firm C specifies that Histology or HPLC grade acetone with field recovery reagent blanks ranging from -0.2 to 1.0 mg per 400 mL or < 2.5 mg/L is used (Note the Method 201A reagent blank result was reported by Firm C without a volume, so 400 mL, which was the volume given for the water and hexane blanks, was used to calculate mass per volume concentrations). For Method 202, sample recoveries reagent grade water that is double deionized, activated carbon filtered, UV irradiated, and membrane filtered or HPLC grade water with a reagent blank of 0.9 mg in 246 mL (~3.66 mg/L) is used. For the hexane GC, pesticide non-spectrophotometric grade is used.

2.2 Procedures

In addition to the materials used in Methods 201A and 202, the procedures employed also play a major role in best practices to reduce the field blank results and accurately implement these methods. These procedures include techniques for handling the materials, preparation of the glassware, the recoveries of samples, and preparation of the sample collection and recovery areas.

Firm A

Firm A minimizes the contribution to the sample weight from the filters by performing the filter tares in a dry laboratory with operators using latex gloves and forceps. These filters are baked to ensure complete dryness before taring. In addition after initial tare, the filters are stored in sterile plastic Petri dishes, which are not reused, for storage and transport. Solvent purity is maintained by keeping the solvents in their original bottle until use. The condenser and first knockout impinger of the Method 202 sampling trains are dedicated for use in Method 202 testing only. The glassware of the sampling trains is cleaned in a multi-step process. First it is soaked in hot water with Liquinox laboratory detergent and brushed as necessary. It is rinsed at least three times with hot water until there are no more signs of soap. Finally, the glassware gets three rinses with each of the following, DI water, acetone, and hexane. Sometimes an additional baking step at 250°F is added and the ball joint o-rings are not removed. Firm A reported field train proof blanks, which exclude the probe extension, with < 1.0 mg total, < 0.5 mg in each fraction, aqueous and organic. Typically two trains are reused in rotation for subsequent runs during sampling, but up to three or four trains may be used.

Firm B

Firm B takes great care in providing a clean environment for sample and blank recoveries. Firm B cleans their mobile lab and recovery locations in order to minimize sample contamination. Sampling train glassware is not dedicated for Methods 201A and 202, but is cleaned according to the following procedure before use with these methods. The glassware is soaked in tap water with Liquinox laboratory detergent. It is then rinsed with tap water, DI water, acetone, and hexane. Finally, all components except the probe extensions are baked at 300°C for 6 hours.

In addition to cleaning the glassware, Firm A takes other steps to ensure that the contribution to the samples and blanks from the glassware is as small as possible. The glassware is cleaned in a clean laboratory environment. Operators wear gloves when handling glassware and glassware is capped with foil after cleaning. Immediately prior to use in the field, the glassware is rinsed with DI water, acetone, and hexane and allowed to dry. Field train proof blanks are not required by the method in this case, because the glassware is baked. During sampling, usually two trains are reused in rotation for subsequent runs.

Firm C

In order to prevent sample contamination, Firm C specifies that all areas where samples are exposed, including the sample recovery area, should be enclosed and kept clean and free of dust, dirt, and debris. Trailers or trucks can be used as recovery areas, but only side doors should be used, not roll-up doors. Foot traffic in and out of the recovery area should be controlled at all times and completely restricted while samples are exposed. Outside the recovery area mats and shoe cleaning equipment should be available to avoid introducing outside dirt from shoes. To minimize the contributions to the sample weight from the filters, Firm C suggests using plastic Petri dishes for storage and transport and nitrile gloves for all sample and filter handling. This includes removing filter fragments from the filter holder, which is reported to be more effective than brushing because fewer fragments are lost or introduced.

Firm C requires an acid wash for M201A filters if the PM concentration is low and SO_2 is present in the source. For acetone, PTFE wash bottles are suggested for minimizing sample and blank contributions. A 5-place balance with charge neutralization is specified for sample weighing and a constant weight criterion of 0.1 mg is used. Aluminum pans are preferred over PTFE baggies for acetone sample recovery rinses. For glassware, off-site cleaning is specified, as is a complete recovery rinse in the field before use. Firm C requires that the glassware selected should look brand new, and be completely free of deposits. Sample collection of acetone rinses requires certified contaminant-free amber glass jars with PTFE lid liners. Firm C reported field train proof blanks in the 3 to 5 mg range for the combined organic and inorganic total.

2.3 Conclusions

Many of the procedures reported by these three firms overlap with or are closely related in their approach for reducing possible sources of contamination. Knowing and following the method specifications for materials will limit the contribution to the sample result, especially for the reagents, which have specifications designed to limit their contributions. Table 2 summarizes the best practices from the survey for conducting Method 201A and 202 source testing to generate accurate measurements and low field train recovery blank results.

Table 2. Summary of Best Practices for Method 201A and Method 202

Topic	Best Practice	Comments
Method 201A Filter	High purity binder free glass or quartz fiber	Acid washing filters reduces SO_2 artifact
Method 202 Filter	100% PTFE membrane	Non-PTFE filters found to contain extractable organic mass
Filter Tare	Clean, dry, environmentally controlled lab	A balance with resolution of 0.01 mg may be necessary
Filter Handling	Gloves and forceps	Minimize handling
Filter Storage and Transport	Sterile Petri dish	
Acetone	0.05 mg/L residue on evaporation	Solvent purity can have a significant impact on FTRB results. Keep in original solvent bottle until needed.
Water	0.05 mg/L residue on evaporation	In-house prepared water purity varies greatly. Keep in original solvent bottle until needed.
Hexane	0.05 mg/L residue on evaporation	Solvent purity varies greatly between grades and suppliers. Keep in original solvent bottle until needed.
Aqueous Fraction Recovery and Transport Containers	PTFE wash bottles, polymer sample jars	Certified low residual mass by manufacturer when possible
Organic Fraction Recovery and Transport Containers	Glass sample jars	Certified low residual mass by manufacturer when possible
Sampling Train Glassware Cleaning	• Soaking in hot water and lab detergent, brushing as necessary • Rinsing with hot water until free of soap • Rinsing 3x with DI water, acetone, and hexane • Bake at 300 °C for 6 hours (if possible)	Probe extension cleaning is critical because most are too long to be baked at 300 °C
Sampling Train Storage and Transport	▪ Seal open ends with clean aluminum foil ▪ Store glassware in a clean environment	Aluminum foil may need to be rinsed to avoid roll lubricant contamination
Sampling Train Field Setup	• Wear synthetic gloves • Rinse glass train components with DI water, acetone, and hexane immediately prior to use	
Sampling Train Recovery	Perform sample recovery in an enclosed, controlled access area that is kept clean and free of dust	Keeping areas clean may require a HEPA filtered air supply for ventilation and HEPA filter vacuuming prior to sample recovery. Clean area immediately prior to use.

Although stakeholders have reported that the 2.0 mg limit for Method 202 field train recovery blanks is difficult to achieve, these three firms have shown that with the proper materials and procedures it is possible to meet the limit and to accurately implement both test methods. Because 2.0 mg is the limit for Method 202 field sample correction, any field train recovery blank values above 2.0 mg represent source test results that may be biased high. The field train recovery blank needs to be very low to ensure that results for the CPM measurement from Method 202 source tests are attributable to the source and not to the materials or procedures used in the sample collection, recovery, and analysis. Field train recovery blanks are performed after one or two source tests have been run and the resulting samples collected, so FTRBs are an evaluation of the sample recoveries as well as an evaluation of the contribution to the sample result from the materials and procedures. Low FTRB results, like those reported by these three firms, verify both aspects. Proof train blanks may be necessary to determine if high FTRB results from poor sample recovery or contaminated supplies and glassware. In the case of these methods, measuring particulate matter at or near the method detection limits requires implementation of the best practices that these three firms have shared to generate precise and accurate results often required for particulate matter regulations or standards.

3.0 METHOD 202 CONDENSABLE PARTICULATE MATTER FILTER EVALUATION

ERG completed Method 202 filter evaluations from August through November of 2013 at the ERG laboratory facility, in accordance with WA 3-07 Task 3. Method 202 measures CPM, which is not captured by a filter at stack conditions, but forms solid or liquid particulate matter immediately after discharge from the stack. Method 202 mimics stack conditions using a condenser and dry impingers and collects CPM on a filter. Method 202 is combined with a filterable PM method, such as Method 201A, and follows the filterable PM method. See Figure 1, the combined M201A/202 sampling train configuration schematic.

The Method 202 field sample weight is corrected using the Field Train Residual Blank (FTRB) result or 2.0 mg whichever is less, as specified by the method. Filters have the potential for significant residual mass concentrations, which can cause the FTRB result to exceed the 2.0 mg limit and contribute mass to samples, biasing CPM measurements high. Thus, the contribution to the residual mass field sample results from CPM filters must not exceed the limit in the method of 0.5 mg to ensure that results for the CPM measurements from Method 202 source tests are attributable to, and representative of, the source and not to the filters used in the sample collection.

Task 3 evaluated the ability to achieve sufficiently low residual mass blank results from Method 202 CPM filters and evaluated the potential contribution of the CPM filter to the total field sample mass concentration for Method 202 sampling trains. Method 202 specifies the use of polymer filters that are nonreactive and nondisintegrating, and that have an efficiency of at least 99.95% on 0.3 micrometer (µm) dioctyl phthalate particles. Method 202 requires that the filters do not contribute more than 0.5 mg of residual mass to CPM measurements. This criterion is referred to here as the "CPM filter blank limit". The CPM filter blank is obtained by:

- Folding the filter in quarters and placing into an extraction tube
- Recovering the inorganic fraction of the filter
- Recovering the organic fraction of the filter
- Extracting the inorganic fraction three times with hexane using a separatory funnel, combining the extracts with the organic fraction
- Evaporating the samples to approximately 10 mL in beakers
- Evaporating the samples to dryness in aluminum weigh pans
- Desiccating the samples for at least 24 hours
- Gravimetrically analyzing (weighing) the sample pans.

The objective of Task 3 was to generate representative data of the blank contributions of filters used in Methods 202 source sampling. Task 3 evaluated whether Teflon® membrane filters can pass the CPM filter blank limit for the method and the potential contribution of CPM filters to the Method 202 field sample weight. The evaluation was accomplished by recovering and analyzing three different types of CPM filters used for Method 202 sampling to determine their residual blank levels. This task focused on the evaluation of a single commercial lot of filters of each type and not on the variation between lots of filters. This effort did not evaluate the potential for contamination in the field or poor field sample recovery techniques that contribute to elevated field train recovery blank results. All sample processing and gravimetric analysis was performed at ERG's laboratory facility.

3.1 Procedures

ERG evaluated three different types of filters that are commonly used to collect CPM in Method 202 sampling to determine their residual blank levels. Teflon® filters are typically used as Method 202 CPM filters. Teflon® filters that include a backing or substrate to suit the conditions of the source are also used. In order to represent different types of Teflon® filters the three filter types selected for evaluation were:

- 100% Teflon® membrane filters (Pall Zefluor, 47 mm, 1 µm pore size, cat# P5PL047)
- Teflon® membrane filters backed with hydrophobic media (Tisch, 90 mm, 1 µm pore size, cat# SF13867)
- Teflon® membrane filters backed with hydrophilic media (Tisch, 90 mm, 1 µm pore size, cat# SF13852).

All these filter types meet the Method 202 efficiency requirements and the CPM filter blank results are used to determine if the CPM filter limit of 0.5 mg was met. The Teflon® membrane filters selected are 100% Teflon® and do not contain any other substrate or material. One box of each type of filter was obtained and the filters were processed as received from the vendor without any additional preparation. Additional preparation of CPM filters, such as an acid wash, is sometimes used in source testing depending on the expected source conditions, but is not required by the method. Ten filters of each type were processed and analyzed according to the procedures of Method 202, generating a total of 30 CPM filter samples. All work done in this task was performed by ERG personnel at ERG's laboratory facility. A summary of all samples generated in this task and associated sample identification codes is presented in Table 3.

Table 3. Task 3 Sample Summary

CPM Filter Type	ID Code	Number of Samples
100% Teflon® Membrane (TM)	TM01-10	10
Hydrophobic Teflon® Membrane (TF)	TF01-10	10
Hydrophilic Teflon® Membrane (TL)	TL01-10	10

The sample recovery procedures for each sample listed in Table 3 generated two fractions, an inorganic and an organic. ERG personnel processed and gravimetrically analyzed all the samples at ERG's laboratory. All solvents used for all activities conducted in Task 3, including glassware cleaning, sample recovery, and sample processing, met the specifications required in Method 202 that manufacturer's stated residue on evaporation not exceed 1.0 mg/L.

Glassware & Laboratory Prep

All laboratory glassware used for sample processing, including beakers, separatory funnels, and graduated cylinders, were washed, rinsed with DI water and hexane, and baked in an oven at 350°C for 6 hours before use. All reagents used in the preparation of the glassware met the criteria established in Method 202 of an evaporation residue of < 1.0 mg/L. Tare weights were taken for the beakers before sample recovery and recorded in the lab notebook.

Before sample recovery began, all areas of ERG's laboratory where samples and the associated recovery and processing materials were to be handled, including the laboratory fume hood, were thoroughly cleaned to prevent any contamination. The benchtop in the fume hood where the recoveries took place was wiped down using water and dried using paper towels.

Sample Recovery Procedures

ERG generated the Task 3 Method 202 CPM filter blanks by solvent extraction of the filters using a sonicator according to the procedures Method 202. The filters were extracted as received from the vendor without any additional preparation. The Method 202 CPM filter recovery procedures generate two sample fractions, an inorganic and an organic, each of which is ultimately evaporated to dryness in its own aluminum weighing pan for gravimetric analysis. Before extraction sample beakers were labeled with the unique sample ID, date, and operator initials. The filters were folded into quarters and placed into extraction tubes that came precleaned from the vendor with certificates of analysis from the manufacturer that document the cleanliness specifications. The inorganic fraction is generated by:

- Adding 20 mL deionized, ultra-filtered (DIUF) water, covering the filter
- Placing the extraction tube in a sonication bath for two minutes

- Pouring the resulting extract into the inorganic fraction beaker
- Repeating this step two times for a total of three water extractions.

(Note: the term aqueous, Aq, was used for sample identification codes for inorganic fraction samples in this task). The organic fraction is generated by:

- Adding 20 mL of hexane, covering the filter
- Placing the extraction tube in a sonication bath for 2 minutes
- Collecting the resulting extract in the organic fraction beaker
- Repeating this step two times for a total of three hexane extractions.

High purity Fisher Optima water, with an evaporation residue of < 1.0 mg/L, was used for inorganic fraction recoveries. Hexane, with an evaporation residue of < 0.1 mg/L, was used for organic fraction recoveries. Three extractions of each solvent produced a total sample volume of 60 mL for each fraction. Ten filters of each of the three CPM filter types selected were recovered for a total of 30 CPM filter blank samples.

All filters were wet but unchanged after the three water extraction sonications. The filters appeared translucent upon adding hexane and appeared whole, not broken or dissolved after the organic extractions. Color returned to the filters once they dried. The hexane extracts for the hydrophobic Teflon membrane (TF) samples were slightly cloudy. Sample handling was a critical component in maintaining high quality and preventing contamination for this task and great care was taken to ensure sample integrity and proper identification. After recovery, ERG operators processed the samples for gravimetric analysis and measured the final weights of the sample beakers. Organic sample final beaker weights include the 90 mL generated from the separatory funnel extractions of the inorganic fraction. The sample beaker initial and final weights are presented in Table 4.

Table 4. Task 3 Sample Beaker Weights

Sample ID	Tare Weight (g)	Final Weight (g)	Net Weight (g)	Sample ID	Tare Weight (g)	Final Weight (g)	Net Weight (g)
TF01 Aq	106.8	166.4	59.6	TF01 Org	123.9	215.9	92.0
TF02 Aq	111.8	171.9	60.1	TF02 Org	108.7	201.3	92.6
TF03 Aq	115.6	175.8	60.2	TF03 Org	115.0	207.9	92.9
TF04 Aq	97.4	157.8	60.4	TF04 Org	100.0	190.8	90.8
TF05 Aq	113.6	173.7	60.1	TF05 Org	88.4	178.7	90.3
TF06 Aq	107.3	167.5	60.2	TF06 Org	110.8	201.4	90.6
TF07 Aq	115.2	175.5	60.3	TF07 Org	108.4	200.0	91.6
TF08 Aq	100.9	161.2	60.3	TF08 Org	108.9	201.0	92.1
TF09 Aq	110.3	170.9	60.6	TF09 Org	103.8	195.4	91.6
TF10 Aq	110.1	170.1	60.0	TF10 Org	114.9	205.0	90.1

Note: TF is the hydrophobic Teflon membrane, TL is the hydrophilic Teflon membrane, TM is the 100% Teflon membrane.

Table 4. Task 3 Sample Beaker Weights (Continued)

Sample ID	Tare Weight (g)	Final Weight (g)	Net Weight (g)	Sample ID	Tare Weight (g)	Final Weight (g)	Net Weight (g)
TL01 Aq	110.3	169.1	58.8	TL01 Org	110.7	199.5	88.8
TL02 Aq	88.4	146.5	58.1	TL02 Org	111.8	200.8	89.0
TL03 Aq	106.8	165.0	58.2	TL03 Org	112.8	206.7	93.9
TL04 Aq	95.2	153.5	58.3	TL04 Org	114.9	207.0	92.1
TL05 Aq	113.7	171.7	58.0	TL05 Org	100.9	193.3	92.4
TL06 Aq	108.9	166.8	57.9	TL06 Org	106.8	198.4	91.6
TL07 Aq	108.4	166.2	57.8	TL07 Org	115.6	205.2	89.6
TL08 Aq	110.3	168.8	58.5	TL08 Org	112.8	203.9	91.1
TL09 Aq	123.9	182.2	58.3	TL09 Org	103.8	198.3	94.5
TL10 Aq	95.2	154.2	59.0	TL10 Org	114.7	210.1	95.4
TM01 Aq	88.4	146.2	57.8	TM01 Org	104.6	194.3	89.7
TM02 Aq	110.8	167.1	56.3	TM02 Org	115.2	206.7	91.5
TM03 Aq	115.0	172.1	57.1	TM03 Org	108.8	200.0	91.2
TM04 Aq	97.4	147.2	49.8	TM04 Org	100.0	192.3	92.3
TM05 Aq	123.9	180.6	56.7	TM05 Org	113.6	198.9	85.3
TM06 Aq	108.4	165.5	57.1	TM06 Org	115.6	202.6	87.0
TM07 Aq	110.3	171.1	60.8	TM07 Org	100.9	190.5	89.6
TM08 Aq	108.9	161.4	52.5	TM08 Org	106.7	195.5	88.8
TM09 Aq	112.7	169.2	56.5	TM09 Org	114.9	206.2	91.3
TM10 Aq	103.7	160.8	57.1	TM10 Org	95.2	187.9	92.7

Note: TF is the hydrophobic Teflon membrane, TL is the hydrophilic Teflon membrane, TM is the 100% Teflon membrane.

Sample Processing Procedures

All laboratory glassware used for sample processing, including beakers, separatory funnels, and graduated cylinders, were washed, rinsed with DI water and hexane, and baked in an oven at 350°C for 6 hours before use. The same sample beakers used for the recoveries were used for the sample processing. Aluminum weighing pans were numbered and tared before use.

After sample recovery, ERG processed the samples in preparation for gravimetric analysis. The sample recovery procedures yielded an inorganic fraction and an organic fraction for each filter: 30 inorganic fraction samples and 30 organic fractions total. The inorganic fraction of each sample was extracted with 30 mL of hexane three times using a separatory funnel. The resulting hexane extracts were combined with the associated organic fraction for that sample. The organic fractions had a total sample volume of 120 mL. The inorganic sample beakers were placed in an oven set to 105°C to expedite evaporation to approximately 10 mL. The organic sample beakers were placed into the laboratory fume hood where they were allowed to evaporate to approximately 10 mL. The organic samples were then quantitatively transferred into tared weighing pans using hexane and the pan numbers were recorded in the lab notebook. The inorganic samples were allowed to cool in the fume hood, and then quantitatively transferred into tared weighing pans using water and the pan numbers were recorded in the lab notebook. The sample pans were allowed to evaporate to dryness in the fume hood and placed in desiccators in the balance room for gravimetric analysis. Samples were handled and processed in a manner that ensured the integrity of samples and minimized the opportunity for contamination. Nitrile gloves were worn during all sample handling and processing.

3.2 Quality Control

All work conducted in this task followed the CPM filter recovery procedures in Method 202 as written. The solvents and glassware used for all activities conducted in Task 3 met the specifications in Method 202, which require the use of water and hexane that have a manufacturer's stated residue on evaporation value of 1.0 mg/L or less. Sample recovery rinses were collected in beakers that were cleaned, solvent rinsed, and baked prior to use. All sample containers were labeled with the unique sample ID, date, and operator initials. Weighing pans were numbered and the corresponding sample IDs were recorded in the lab notebook. Sample handling was a critical component in maintaining high quality for this task and great care was taken to process samples in a manner that ensured the integrity of samples and minimized the opportunity for contamination. All sample weights were recorded in the balance room lab notebook with the conditions and balance calibration check information for that day, see Table 5, the Gravimetric Analysis Daily Conditions table and Table 6, the Gravimetric Analysis Daily Balance Calibration table.

Table 5. Task 3 Gravimetric Analysis Daily Conditions

Pan #	Tare Weights			Constant Weights			Additional Weights		
	Date	Temp (°F)	%RH	Date	Temp (°F)	%RH	Date	Temp (°F)	%RH
44	8/22/2013	75.59	63.9	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
62	8/20/2013	74.92	63.2	9/18/2013	71.54	48.8	10/21/2013	70.87	39.4
63	8/20/2013	74.92	63.2	9/18/2013	71.54	48.8	9/23/2013	73.91	47.4
64	8/20/2013	74.92	63.2	9/16/2013	75.37	52.4	10/17/2013	73.68	59.0
65	8/22/2013	75.59	63.9	9/16/2013	75.37	52.4	10/17/2013	73.68	59.0
66	8/20/2013	74.92	63.2	9/16/2013	75.37	52.4	10/17/2013	73.68	59.0
68	8/20/2013	74.92	63.2	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
69	8/20/2013	74.92	63.2	9/16/2013	75.37	52.4	9/18/2013	71.54	48.8
71	8/22/2013	75.59	63.9	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
72	8/22/2013	75.59	63.9	9/16/2013	75.37	52.4	9/18/2013	71.54	48.8
73	8/20/2013	74.92	63.2	9/16/2013	75.37	52.4	9/18/2013	71.54	48.8
74	8/20/2013	74.92	63.2	9/18/2013	71.54	48.8	10/21/2013	70.87	39.4
75	8/20/2013	74.92	63.2	9/18/2013	71.54	48.8	9/27/2013	75.48	48.1
76	8/20/2013	74.92	63.2	9/18/2013	71.54	48.8	9/27/2013	75.48	48.1
77	8/20/2013	74.92	63.2	11/12/2013	72.56	27.1	11/12/2013	72.56	27.1
78	8/20/2013	74.92	63.2	9/23/2013	73.91	47.4	9/24/2013	71.88	51.4
79	8/22/2013	75.59	63.9	9/18/2013	71.54	48.8	9/23/2013	73.91	47.4
80	8/22/2013	75.59	63.9	9/18/2013	71.54	48.8	9/23/2013	73.91	47.4
81	8/22/2013	75.59	63.9	9/23/2013	73.91	47.4	9/24/2013	71.88	51.4
82	8/22/2013	75.59	63.9	9/23/2013	73.91	47.4	9/24/2013	71.88	51.4
84	8/22/2013	75.59	63.9	9/18/2013	71.54	48.8	9/23/2013	73.91	47.4

Table 5. Task 3 Gravimetric Analysis Daily Conditions (Continued)

Pan #	Tare Weights			Constant Weights			Additional Weights		
	Date	Temp (°F)	%RH	Date	Temp (°F)	%RH	Date	Temp (°F)	%RH
126	9/9/2013	71.43	58.6	9/20/2013	71.21	50.9	9/26/2013	75.03	47.3
128	9/9/2013	71.43	58.6	9/27/2013	75.48	48.1	10/22/2013	70.64	51.4
129	9/9/2013	71.43	58.6	10/18/2013	70.87	48.0	10/21/2013	70.87	39.4
130	9/9/2013	71.43	58.6	10/21/2013	70.87	39.4	10/22/2013	70.64	51.4
131	9/9/2013	71.43	58.6	10/18/2013	70.87	48.0	10/21/2013	70.87	39.4
132	9/9/2013	71.43	58.6	10/21/2013	70.87	39.4	10/22/2013	70.64	51.4
133	9/24/2013	71.88	51.4	10/21/2013	70.87	39.4	10/22/2013	70.64	51.4
134	9/24/2013	71.88	51.4	10/21/2013	70.87	39.4	10/22/2013	70.64	51.4
135	9/24/2013	71.88	51.4	10/29/2013	71.77	50.8	10/30/2013	71.32	55.4
136	9/24/2013	71.88	51.4	10/29/2013	71.77	50.8	10/30/2013	71.32	55.4
137	9/24/2013	71.88	51.4	10/31/2013	72.89	54.0	11/5/2013	72.11	35.3
138	9/24/2013	71.88	51.4	10/31/2013	72.89	54.0	11/5/2013	72.11	35.3
139	9/24/2013	71.88	51.4	10/31/2013	72.89	54.0	11/5/2013	72.11	35.3
140	9/24/2013	71.88	51.4	10/31/2013	72.89	54.0	11/5/2013	72.11	35.3
141	9/24/2013	71.88	51.4	10/31/2013	72.89	54.0	11/5/2013	72.11	35.3
142	9/24/2013	71.88	51.4	10/31/2013	72.89	54.0	11/5/2013	72.11	35.3
143	9/24/2013	71.88	51.4	10/31/2013	72.89	54.0	11/5/2013	72.11	35.3
144	9/24/2013	71.88	51.4	10/31/2013	72.89	54.0	11/5/2013	72.11	35.3
145	9/24/2013	71.88	51.4	10/31/2013	72.89	54.0	11/5/2013	72.11	35.3
146	9/24/2013	71.88	51.4	10/31/2013	72.89	54.0	11/5/2013	72.11	35.3
147	9/24/2013	71.88	51.4	10/31/2013	72.89	54.0	11/5/2013	72.11	35.3
148	9/24/2013	71.88	51.4	10/31/2013	72.89	54.0	11/5/2013	72.11	35.3
149	9/24/2013	71.88	51.4	11/11/2013	72.44	21.9	11/12/2013	72.56	27.1
150	9/24/2013	71.88	51.4	11/11/2013	72.44	21.9	11/12/2013	72.56	27.1
151	9/24/2013	71.88	51.4	11/11/2013	72.44	21.9	11/12/2013	72.56	27.1
152	9/24/2013	71.88	51.4	11/11/2013	72.44	21.9	11/12/2013	72.56	27.1
153	9/24/2013	71.88	51.4	11/11/2013	72.44	21.9	11/12/2013	72.56	27.1
154	9/24/2013	71.88	51.4	11/11/2013	72.44	21.9	11/12/2013	72.56	27.1
155	9/24/2013	71.88	51.4	11/11/2013	72.44	21.9	11/12/2013	72.56	27.1
156	9/24/2013	71.88	51.4	11/11/2013	72.44	21.9	11/12/2013	72.56	27.1
157	11/4/2013	72.89	26.8	11/12/2013	72.56	27.1	11/12/2013	72.56	27.1
158	11/4/2013	72.89	26.8	11/12/2013	72.56	27.1	11/12/2013	72.56	27.1
159	11/4/2013	72.89	26.8	11/12/2013	72.56	27.1	11/12/2013	72.56	27.1
160	11/4/2013	72.89	26.8	11/12/2013	72.56	27.1	11/12/2013	72.56	27.1
161	11/4/2013	72.89	26.8	11/12/2013	72.56	27.1	11/12/2013	72.56	27.1
162	11/4/2013	72.89	26.8	11/12/2013	72.56	27.1	11/12/2013	72.56	27.1
163	10/30/2013	71.32	55.4	11/12/2013	72.56	27.1	11/12/2013	72.56	27.1
164	10/31/2013	72.89	54.0	11/11/2013	72.44	21.9	11/12/2013	72.56	27.1
165	10/31/2013	72.89	54.0	11/11/2013	72.44	21.9	11/12/2013	72.56	27.1
166	10/30/2013	71.32	55.4	11/11/2013	72.44	21.9	11/12/2013	72.56	27.1
167	10/31/2013	72.89	54.0	11/11/2013	72.44	21.9	11/12/2013	72.56	27.1
168	10/31/2013	72.89	54.0	11/11/2013	72.44	21.9	11/12/2013	72.56	27.1

Table 6. Task 3 Gravimetric Analysis Daily Balance Calibrations

Date	Known (g)	Measured (g)	Date	Known (g)	Measured (g)
8/20/2013	6.00000	6.00002	10/17/2013	6.00000	6.00003
	6.00500	6.00502		6.00500	6.00503
	6.50000	6.50002		6.50000	6.50003
8/22/2013	6.00000	6.00005	10/18/2013	6.00000	6.00004
	6.00500	6.00503		6.00500	6.00503
	6.50000	6.50003		6.50000	6.50003
9/9/2013	6.00000	6.00003	10/21/2013	6.00000	6.00001
	6.00500	6.00503		6.00500	6.00500
	6.50000	6.50003		6.50000	6.50002
9/13/2013	6.00000	6.00003	10/22/2013	6.00000	6.00003
	6.00500	6.00504		6.00500	6.00502
	6.50000	6.50004		6.50000	6.50002
9/16/2013	6.00000	6.00003	10/29/2013	6.00000	6.00001
	6.00500	6.00503		6.00500	6.00503
	6.50000	6.50006		6.50000	6.50000
9/18/2013	6.00000	6.00006	10/30/2013	6.00000	6.00004
	6.00500	6.00505		6.00500	6.00504
	6.50000	6.50006		6.50000	6.50004
9/20/2013	6.00000	6.00000	10/31/2013	6.00000	6.00003
	6.00500	6.00500		6.00500	6.00503
	6.50000	6.50002		6.50000	6.50003
9/23/2013	6.00000	6.00002	11/4/2013	6.00000	6.00005
	6.00500	6.00505		6.00500	6.00503
	6.50000	6.50006		6.50000	6.50004
9/24/2013	6.00000	6.00003	11/5/2013	6.00000	6.00005
	6.00500	6.00500		6.00500	6.00501
	6.50000	6.50001		6.50000	6.50003
9/26/2013	6.00000	6.00004	11/11/2013	6.00000	6.00005
	6.00500	6.00501		6.00500	6.00502
	6.50000	6.50003		6.50000	6.50004
9/27/2013	6.00000	6.00004	11/12/2013	6.00000	6.00003
	6.00500	6.00501		6.00500	6.00501
	6.50000	6.50003		6.50000	6.50001

3.3 Analytical Results

The residual mass of every sample generated in this task was measured by gravimetric analysis. The raw results, in grams, are presented in Table 7, the Task 3 Raw Sample Results table. All negative results are reported as non-detects.

Table 7. Task 3 Raw Sample Results

Pan #	Constant Weight (g)	Additional Weight (g)	Average Weight (g)	Tare Weight (g)	Net Constant Weight (g)	Net Average Weight (g)
44	6.35404	6.35409	6.35407	6.35385	0.00019	0.00021
62	6.26143	6.26137	6.26140	6.25982	0.00161	0.00158
63	6.35934	6.35944	6.35939	6.35729	0.00205	0.00210
64	6.35292	6.35290	6.35291	6.35121	0.00171	0.00170
65	6.35724	6.35729	6.35727	6.35715	0.00009	0.00012
66	6.40306	6.40313	6.40310	6.40129	0.00177	0.00181
68	6.34797	6.34788	6.34793	6.34619	0.00178	0.00174
69	6.35431	6.35432	6.35432	6.35254	0.00177	0.00177
71	6.35461	6.35453	6.35457	6.35443	0.00018	0.00014
72	6.35214	6.35210	6.35212	6.35191	0.00023	0.00021
73	6.32984	6.32982	6.32983	6.32823	0.00161	0.00160
74	6.37608	6.37603	6.37606	6.37467	0.00141	0.00138
75	6.30608	6.30609	6.30609	6.30470	0.00138	0.00138
76	6.39041	6.39043	6.39042	6.38890	0.00151	0.00152
77	6.35325	6.35323	6.35324	6.35464	ND	ND
78	6.35573	6.35573	6.35573	6.35546	0.00027	0.00027
79	6.34146	6.34151	6.34149	6.34158	ND	ND
80	6.33975	6.33980	6.33978	6.33898	0.00077	0.00080
81	6.34240	6.34232	6.34236	6.34222	0.00018	0.00014
82	6.37389	6.37384	6.37387	6.37412	ND	ND
84	6.35890	6.35894	6.35892	6.35895	ND	ND
126	6.25186	6.25184	6.25185	6.25170	0.00016	0.00015
128	6.24488	6.24488	6.24488	6.24394	0.00094	0.00094
129	6.24852	6.24855	6.24854	6.24742	0.00110	0.00112
130	6.25483	6.25481	6.25482	6.25373	0.00110	0.00109
131	6.26028	6.26023	6.26026	6.25926	0.00102	0.00099
132	6.26884	6.26880	6.26882	6.26784	0.00100	0.00098
133	6.30550	6.30546	6.30548	6.30543	0.00007	0.00005
134	6.32262	6.32258	6.32260	6.32254	0.00008	0.00006
135	6.27470	6.27467	6.27469	6.27452	0.00018	0.00016
136	6.27855	6.27848	6.27852	6.27854	0.00001	ND
137	6.29084	6.29079	6.29082	6.29095	ND	ND
138	6.28650	6.28652	6.28651	6.28644	0.00006	0.00007
139	6.30700	6.30699	6.30700	6.30676	0.00024	0.00024
140	6.27926	6.27920	6.27923	6.27911	0.00015	0.00012
141	6.27525	6.27528	6.27527	6.27530	ND	ND
142	6.29353	6.29346	6.29350	6.29311	0.00042	0.00038
143	6.30138	6.30141	6.30140	6.29997	0.00141	0.00142
144	6.30784	6.30783	6.30784	6.30650	0.00134	0.00134
145	6.27288	6.27285	6.27287	6.27170	0.00118	0.00116
146	6.31756	6.31759	6.31758	6.31627	0.00129	0.00130
147	6.28535	6.28538	6.28537	6.28413	0.00122	0.00124
148	6.32049	6.32054	6.32052	6.31941	0.00108	0.00110
149	6.33337	6.33339	6.33338	6.33322	0.00015	0.00016
150	6.30653	6.30658	6.30656	6.30668	ND	ND
151	6.26560	6.26563	6.26562	6.26555	0.00005	0.00007
152	6.30988	6.30993	6.30991	6.30985	0.00003	0.00005

Negative results are reported as non-detects (ND)

Table 7. Task 3 Raw Sample Results (Continued)

Pan #	Constant Weight (g)	Additional Weight (g)	Average Weight (g)	Tare Weight (g)	Net Constant Weight (g)	Net Average Weight (g)
153	6.30271	6.30275	6.30273	6.30315	ND	ND
154	6.28734	6.28737	6.28736	6.28785	ND	ND
155	6.33004	6.33003	6.33004	6.33058	ND	ND
156	6.32140	6.32145	6.32143	6.32198	ND	ND
157	6.32923	6.32922	6.32923	6.32905	0.00018	0.00018
158	6.28491	6.28489	6.28490	6.28475	0.00016	0.00015
159	6.29220	6.29219	6.29220	6.29204	0.00016	0.00015
160	6.31450	6.31451	6.31451	6.31429	0.00021	0.00022
161	6.31569	6.31571	6.31570	6.31553	0.00016	0.00017
162	6.29761	6.29756	6.29759	6.29746	0.00015	0.00012
163	6.29280	6.29284	6.29282	6.29303	ND	ND
164	6.28738	6.28732	6.28735	6.28753	ND	ND
165	6.30354	6.30355	6.30355	6.30363	ND	ND
166	6.32132	6.32127	6.32130	6.32156	ND	ND
167	6.29956	6.29960	6.29958	6.29978	ND	ND
168	6.28883	6.28883	6.28883	6.28903	ND	ND

Negative results are reported as non-detects (ND)

Results for the CPM filter blanks for the TF, TL, and TM filters, in mg, are in Tables 8, 9, and 10, the CPM filter results tables.

Table 8. TF Sample Results

Inorganic				Organic				Total		
Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)	Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)	Sample ID	Net Constant Weight (mg)	Net Average Weight (mg)
TF01 Aq	44	0.19	0.21	TF01 Org	62	1.61	1.58	TF01	1.80	1.79
TF02 Aq	65	0.09	0.12	TF02 Org	63	2.05	2.10	TF02	2.14	2.22
TF03 Aq	71	0.18	0.14	TF03 Org	64	1.71	1.70	TF03	1.89	1.84
TF04 Aq	72	0.23	0.21	TF04 Org	66	1.77	1.81	TF04	2.00	2.02
TF05 Aq	78	0.27	0.27	TF05 Org	68	1.78	1.74	TF05	2.05	2.01
TF06 Aq	79	ND	ND	TF06 Org	69	1.77	1.77	TF06	1.77	1.77
TF07 Aq	80	0.77	0.80	TF07 Org	73	1.61	1.60	TF07	2.38	2.39
TF08 Aq	81	0.18	0.14	TF08 Org	74	1.41	1.38	TF08	1.59	1.52
TF09 Aq	82	ND	ND	TF09 Org	75	1.38	1.38	TF09	1.38	1.38
TF10 Aq	84	ND	ND	TF10 Org	76	1.51	1.52	TF10	1.51	1.52

Table 9. TL Sample Results

Inorganic				Organic				Total		
Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)	Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)	Sample ID	Net Constant Weight (mg)	Net Average Weight (mg)
TL01 Aq	133	0.07	0.05	TL01 Org	129	1.10	1.12	TL01	1.17	1.17
TL02 Aq	134	0.08	0.06	TL02 Org	130	1.10	1.09	TL02	1.18	1.15
TL03 Aq	135	0.18	0.16	TL03 Org	131	1.02	0.99	TL03	1.20	1.16
TL04 Aq	136	0.01	ND	TL04 Org	132	1.00	0.98	TL04	1.01	0.98
TL05 Aq	137	ND	ND	TL05 Org	143	1.41	1.42	TL05	1.41	1.42
TL06 Aq	138	0.06	0.07	TL06 Org	144	1.34	1.34	TL06	1.40	1.41
TL07 Aq	139	0.24	0.24	TL07 Org	145	1.18	1.16	TL07	1.42	1.40
TL08 Aq	140	0.15	0.12	TL08 Org	146	1.29	1.30	TL08	1.44	1.42
TL09 Aq	141	ND	ND	TL09 Org	147	1.22	1.24	TL09	1.22	1.24
TL10 Aq	142	0.42	0.38	TL10 Org	148	1.08	1.10	TL10	1.50	1.49

Table 10. TM Sample Results

Inorganic				Organic				Total		
Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)	Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)	Sample ID	Net Constant Weight (mg)	Net Average Weight (mg)
TM01 Aq	149	0.15	0.16	TM01 Org	153	ND	ND	TM01	0.15	0.16
TM02 Aq	150	ND	ND	TM02 Org	154	ND	ND	TM02	ND	ND
TM03 Aq	151	0.05	0.07	TM03 Org	155	ND	ND	TM03	0.05	0.07
TM04 Aq	152	0.03	0.05	TM04 Org	156	ND	ND	TM04	0.03	0.05
TM05 Aq	157	0.18	0.18	TM05 Org	163	ND	ND	TM05	0.18	0.18
TM06 Aq	158	0.16	0.15	TM06 Org	164	ND	ND	TM06	0.16	0.15
TM07 Aq	159	0.16	0.15	TM07 Org	165	ND	ND	TM07	0.16	0.15
TM08 Aq	160	0.21	0.22	TM08 Org	166	ND	ND	TM08	0.21	0.22
TM09 Aq	161	0.16	0.17	TM09 Org	167	ND	ND	TM09	0.16	0.17
TM10 Aq	162	0.15	0.12	TM10 Org	168	ND	ND	TM10	0.15	0.12

Statistical analyses and replicate measurement comparisons of the data sets can be seen in Tables 11 through 13.

Table 11. TF Statistical Analysis

	Aq Net Const Wt (mg)	Aq Net Avg Wt (mg)	Org Net Const Wt (mg)	Org Net Avg Wt (mg)	Total Net Const Wt (mg)	Total Net Avg Wt (mg)
Average	0.19	0.19	1.66	1.66	1.85	1.85
Standard Deviation	0.23	0.23	0.20	0.21	0.31	0.32
Detection Limit	0.68	0.70	0.60	0.64	0.92	0.96
% RSD	118.74	124.08	12.07	12.96	16.53	17.32
UPL (n=10)	0.33	0.34	1.79	1.79	2.04	2.05

Table 12. TL Statistical Analysis

	Aq Net Const Wt (mg)	Aq Net Avg Wt (mg)	Org Net Const Wt (mg)	Org Net Avg Wt (mg)	Total Net Const Wt (mg)	Total Net Avg Wt (mg)
Average	0.12	0.11	1.17	1.17	1.30	1.28
Standard Deviation	0.13	0.12	0.14	0.15	0.16	0.17
Detection Limit	0.40	0.37	0.42	0.44	0.48	0.50
% RSD	109.33	114.27	11.80	12.52	12.28	13.05
UPL (n=10)	0.20	0.19	1.26	1.27	1.40	1.39

Table 13. TM Statistical Analysis

	Aq Net Const Wt (mg)	Aq Net Avg Wt (mg)	Org Net Const Wt (mg)	Org Net Avg Wt (mg)	Total Net Const Wt (mg)	Total Net Avg Wt (mg)
Average	0.13	0.13	0.00	0.00	0.13	0.13
Standard Deviation	0.07	0.07	0.00	0.00	0.07	0.07
Detection Limit	0.21	0.20	0.00	0.00	0.21	0.20
% RSD	56.85	52.14	-	-	56.85	52.14
UPL (n=10)	0.17	0.17	0.00	0.00	0.17	0.17

The target of 95% of sample results falling at or below the calculated UPL value was not achieved for all data sets. This can be attributed to the fact that the number of samples in the data sets is low enough that one result above the UPL caused the percentage of samples at or below the UPL to be less than 95%. For the results of the dust pan and the reagent blanks for the hexane and water used in this task see Table 14, the Reagent Blank and Dust Pan Results table.

Table 14. Task 3 Reagent Blank and Dust Pan Results

Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)
DP	77	ND	ND
RBH	126	0.16	0.15
RBW	128	0.94	0.94

The dust pan residual mass was a non-detect, which shows that samples were not contaminated by dust during sample pan evaporation. Reagent blank results show a very low residual mass which is less than the method criteria for the hexane used and a residual mass of less than 1.0 mg for the water.

3.4 Conclusions

The sample weights for both the inorganic (Aq) and organic (Org) fractions of the TF filters and the TL filters were very consistent. Figures 2 and 3 present the Aq and Org results for each of the TF and TL filter samples using the net average weights.

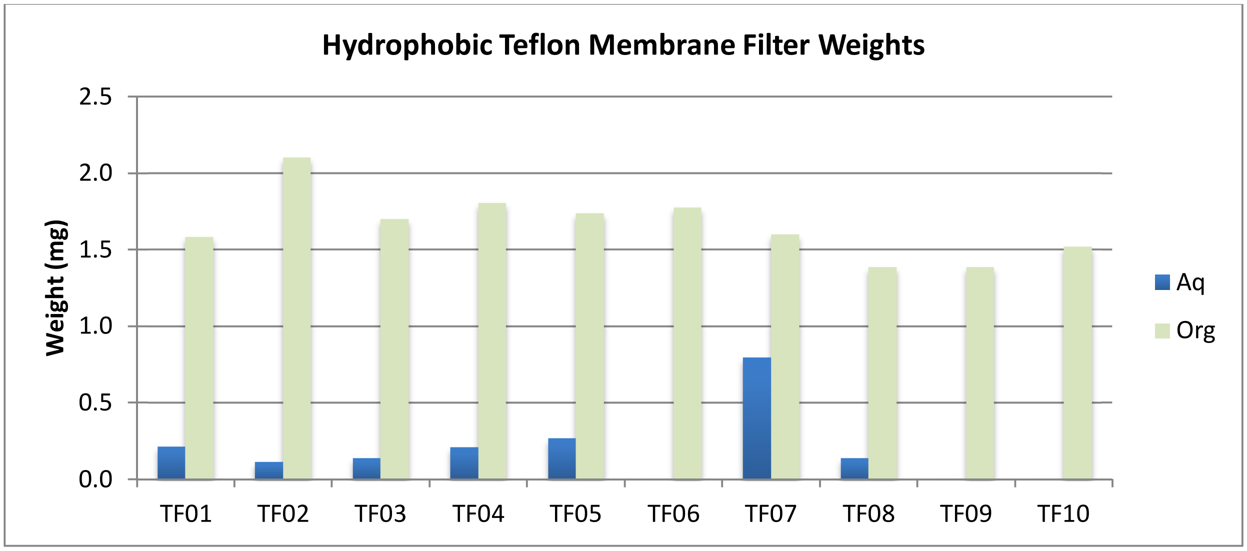

Figure 2. Hydrophobic Teflon Membrane Inorganic and Organic Results (Net Avg Wts)

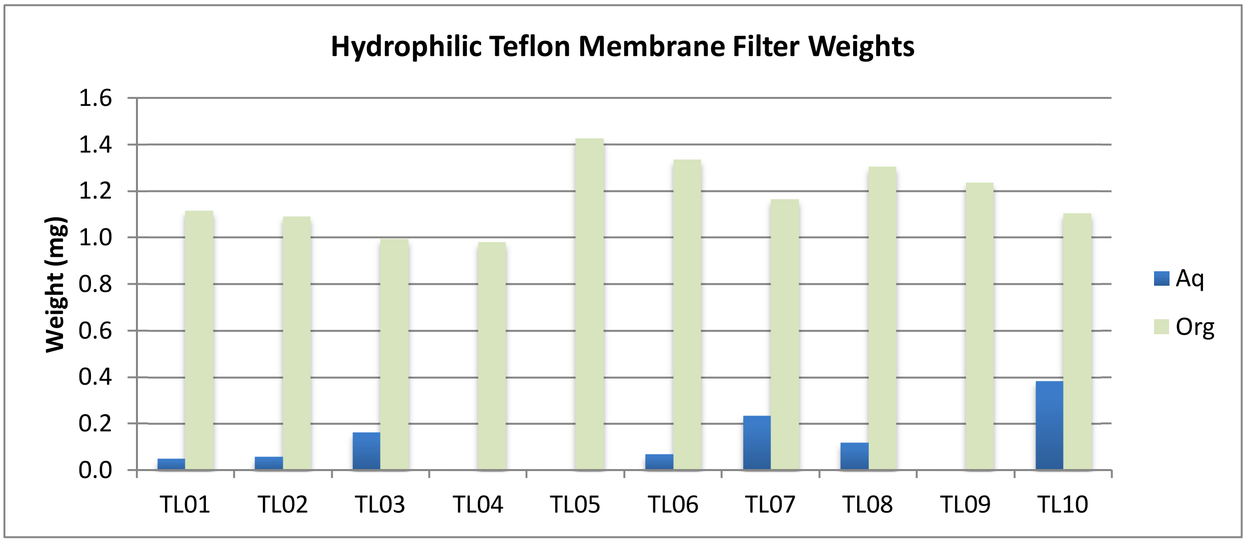

Figure 3. Hydrophilic Teflon Membrane Inorganic and Organic Results (Net Avg Wts)

These graphs show that the organic fractions contribute the majority of the residual mass measured for TF and TL filters. The total residual mass result, the Aq and Org fraction results combined, is the CPM filter blank. The CPM filter blank for both TF and TL filters were also very consistent, as illustrated in Figures 4 and 5, the TF and TL CPM filter blank graphs.

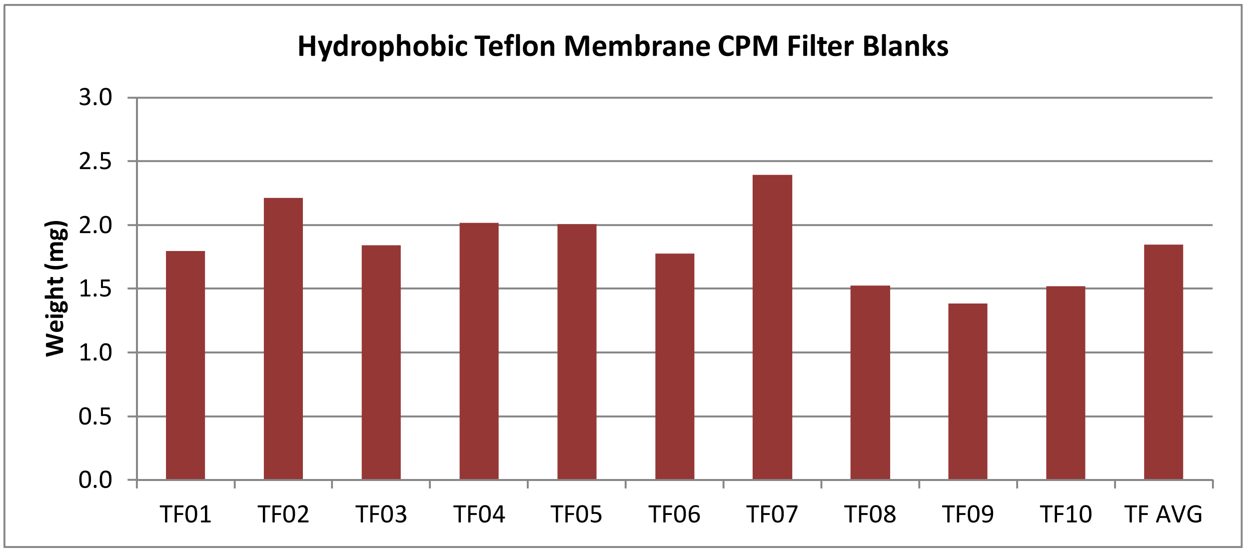

Figure 4. Hydrophobic Teflon Membrane CPM Filter Blank Results (Net Avg Wts)

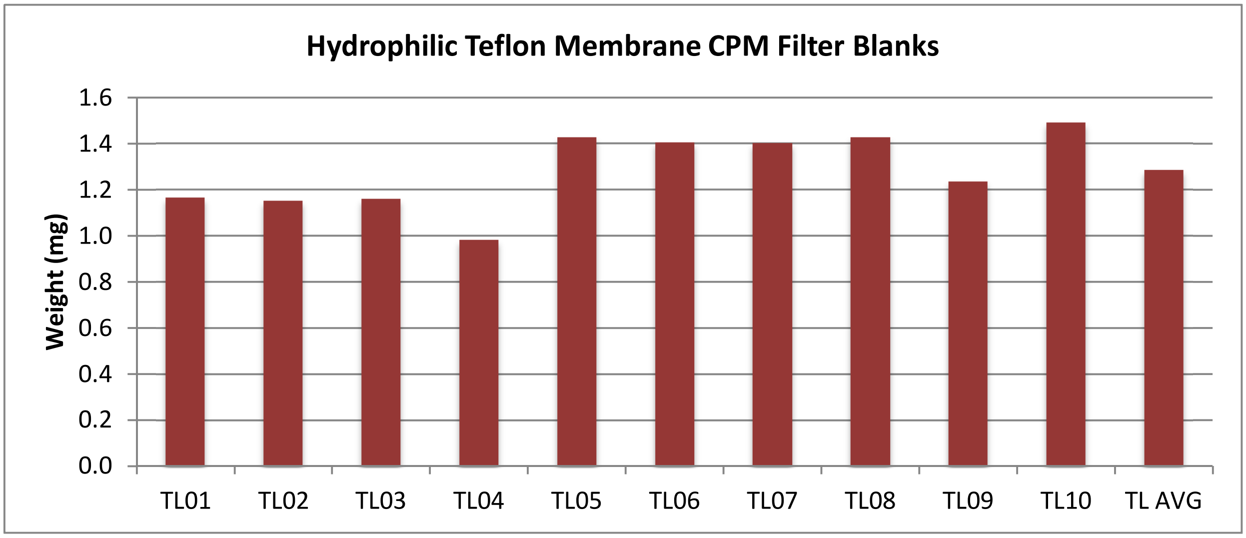

Figure 5. Hydrophilic Teflon Membrane CPM Filter Blank Results (Net Avg Wts)

The TF CPM filter blanks were above the CPM filter blank limit of 0.5 mg and most were very close to the Method 202 FTRB limit of 2.0 mg. The TL CPM filter blanks were above the CPM filter blank limit of 0.5 mg, but they were well below the Method 202 FTRB limit of 2.0 mg. The TM sample weights for the inorganic fractions were very low and consistent. The TM sample weights for the organic fractions were all so low that they were non-detects, so the residual mass results measured for TM CPM filter blanks were the inorganic results alone. The TM CPM filter blank values were all below the CPM filter blank limit of 0.5 mg and are presented in Figure 6, the TM CPM Filter Blank graph.

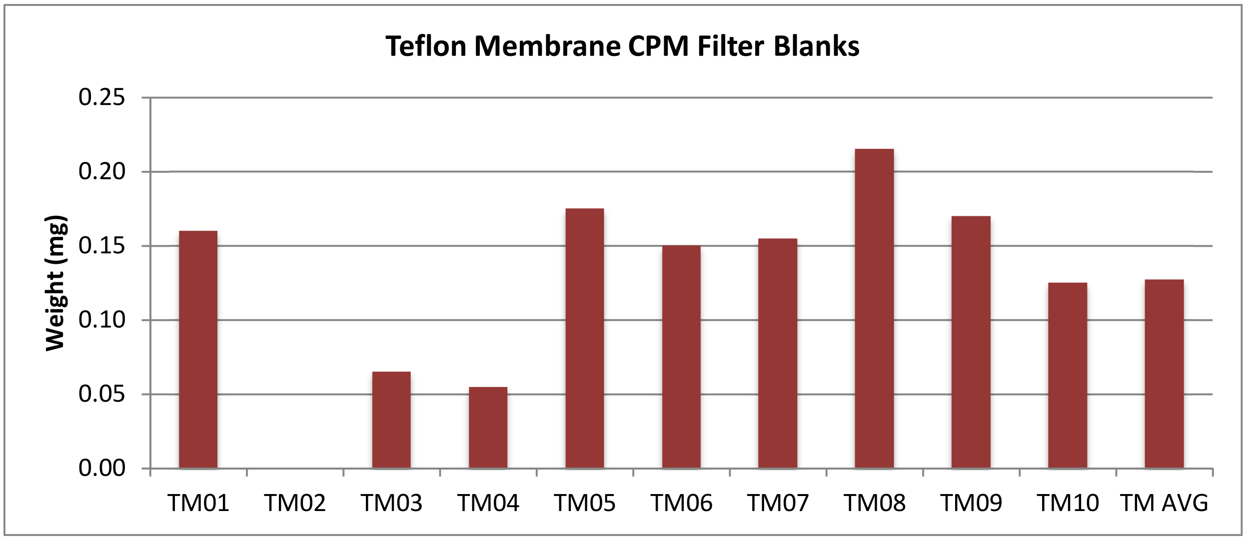

Figure 6. Teflon Membrane CPM Filter Blank Results (Net Avg Wts)

These results show that of the filter types evaluated only the filter made of 100% Teflon® has results low enough to pass the 0.5 mg CPM filter blank limit from the method. In particular the absence of any residual mass for the organic fraction of TM filter samples compared with the elevated levels of organic fraction residual masses in the TF and TL filters represents a striking difference. A comparison of the average inorganic, organic, and total results for all three types of filters is presented in Figure 7, the Average CPM Filter Results graph.

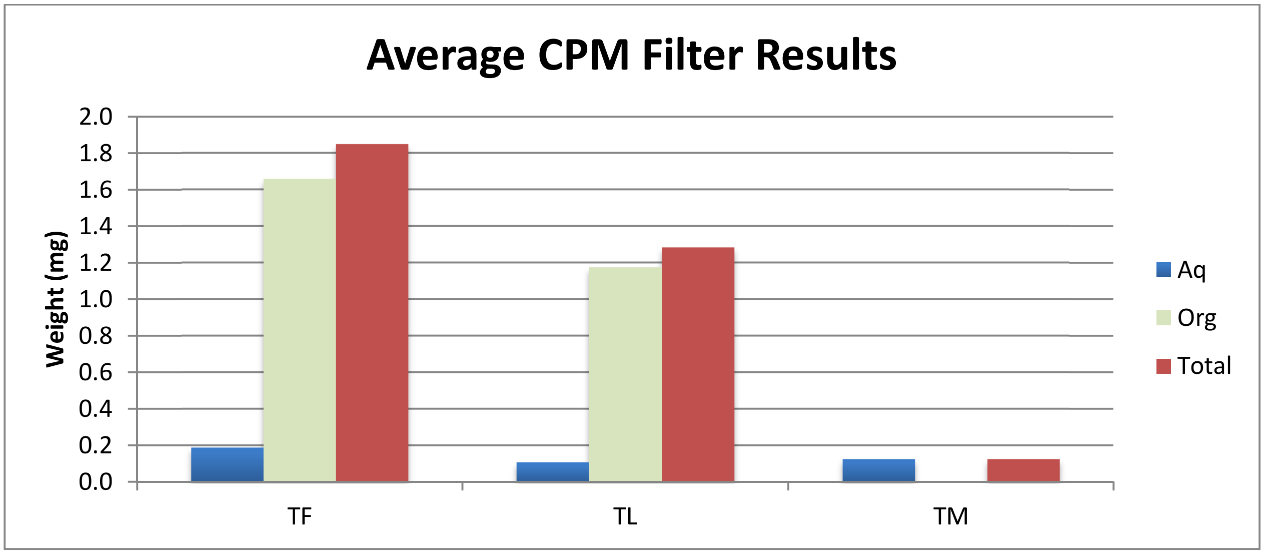

Figure 7. CPM Filter Average Results (Net Average Weights)

The organic results along with the fact that all of the inorganic results for all three filter types were both low and close together points to the likelihood that some component of the substrates used to make the TF and TL filters, hydrophobic and hydrophilic, is soluble in hexane and being recovered during the organic fraction extractions. This finding supports a recommendation that requiring the use of 100% Teflon® filters that do not contain any substrate or support ring other than Teflon® as CPM filters for Method 202 sampling be made a Best Practice. A comparison of the %RSDs calculated using the results of the net constant weights and using the net average weights for all three filter types is presented in Figure 8, the %RSD Comparison graph. This graph shows similar %RSD for the Constant Weights compared to the Average Weights for every set of measurements. The larger %RSDs for the Aq sample fractions are due to the especially small sample weights obtained for the Aq fractions, which include non-detects.

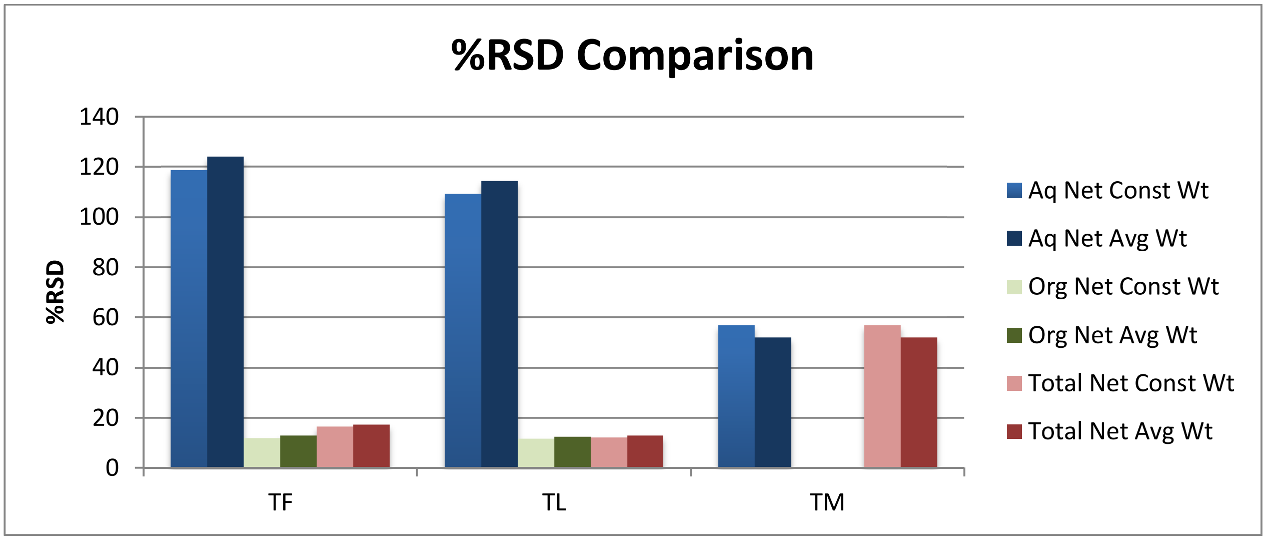

Figure 8. CPM Filter %RSD Comparison

A comparison of the UPLs calculated for each of the three filter types using the net average weights is presented in Figure 9, the CPM Filter UPL Comparison graph. TF filter samples had the highest UPLs for the inorganic fraction, the organic fraction, and the total.

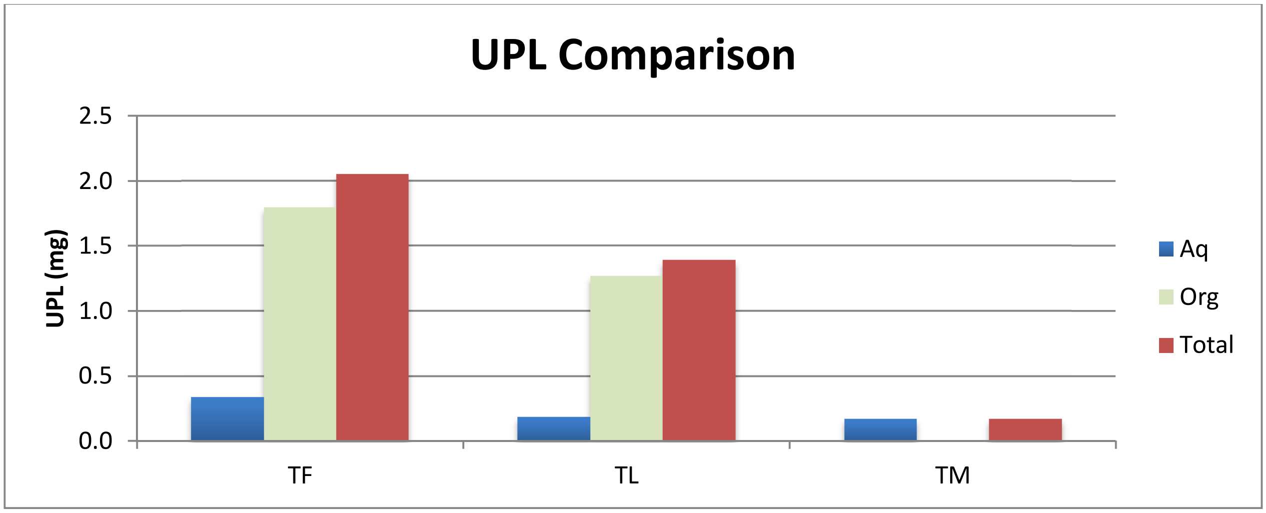

Figure 9. CPM Filter UPL Comparison (Net Average Weights)

The average results and UPL results show that with proper CPM filter selection the contribution to the Method 202 field sample result can be kept at a minimum. Using due diligence and following the requirements of the method would preclude the TF and TL filters evaluated in this task from being used in source testing, but without evaluation that would not be known. Demonstrating that the CPM filter blank for filters proposed for use in Method 202 source testing passes the CPM filter blank limit is recommended as a Best Practice for this method.

4.0 METHOD 202 REAGENT EVALUATION

Method 202 reagent evaluations were performed from July through October of 2013 at the ERG laboratory facility, in accordance with WA 3-07 Task 4. Method 202 measures CPM using a sampling train consisting of a probe extension, a condenser, dry impingers, and a filter. For source testing Method 202 is combined with a filterable PM method, such as Method 201A, which is first in line. See Figure 1, the combined Method 201A and 202 sampling train configuration schematic. Method 202 field samples are generated from the rinsing the sampling train components with reagents and extracting the filters with reagents in sonicators. The contribution to the residual mass field sample results from the reagents needs to be very low to ensure that results for the CPM measurements from Method 202 source tests are attributable to, and representative of, the source and not to the reagents used in the sample collection. Task 4 is designed to evaluate the ability to achieve sufficiently low residual mass blank results from Method 202 reagents and evaluate the potential contribution of the reagents used to the total field sample mass concentration for Method 202 sampling trains.

Task 4 was an evaluation of reagents used in Method 202 sample recoveries. Method 202 specifies the use of DIUF water, acetone, and hexane reagents that have a residue on evaporation value of 1.0 parts per million by weight (ppmw) or less. This criterion is referred to here as the “reagent blank limit.” Method 202 field samples are generated by:

- Recovering the inorganic fraction of the sample by rinsing the sampling train twice with water, collecting the rinses in the inorganic sample jar
- Recovering the organic fraction of the sample by rinsing the sampling train once with acetone and twice with hexane, collecting the rinses in the organic sample jar
- Recovering the inorganic fraction of the filter, by three successive sonications in water, combining the extracts with the inorganic train rinses
- Recovering the organic fraction of the filter, by three successive sonications in hexane, combining the extracts with the organic train rinses
- Extracting the inorganic fraction three times with hexane using a separatory funnel, combining the extracts with the organic fraction
- Evaporating both sample fractions to approximately 10 mL in beakers
- Evaporating the samples to dryness in weigh pans
- Desiccating the samples for at least 24 hours
- Gravimetrically analyzing the sample pans.

The water and hexane reagents are used for two recovery rinses, for the filter extractions, and additional hexane is used for the separatory funnel extraction of the inorganic fraction. Acetone is used for one sampling train rinse, but water and hexane represent the majority of the volume of reagent used in field sample recovery, so only those two reagents were targeted for evaluation in this task. Because it is a reagent used in this method, acetone is a good candidate for further evaluation in determining the best practices for Method 202, given that sufficient volume to generate detectable residual masses is used.

The objective of Task 4 was to generate representative data of the reagent blank residual masses of reagents used in Methods 202 source sampling. Task 4 evaluated whether three grades of water and three grades of hexane passed the reagent blank limit for the method and the potential contribution from water and hexane reagents to the Method 202 field sample weight, field train recovery blank, and field train proof blank. This evaluation was accomplished by recovering and analyzing water and hexane to determine their residual blank levels. Following the reagent specifications from the method should result in reagent blanks that are below the reagent blank limit and that do not contribute a significant amount of residual mass to field samples. To demonstrate that method requirements need to be followed to generate representative Method 202 field samples, three grades of each reagent were evaluated, some that did not have residues on evaporation values below the limit and some that did not follow all of the method specifications. The three grades of water did not have residue on evaporation information from the manufacturer, but were selected based on method of purification. The three grades of hexane evaluated in this task represented one that was above the reagent blank limit, not meeting the specification of the method, one that did not meet the specification, but that was close to the limit, and one that was below the limit. Reagents evaluated in this effort were processed according to the laboratory reagent blank procedures in Method 202. This task focuses on the evaluation of a single lot of each reagent grade and not on the variation between lots of reagents. This effort did not evaluate the potential for contamination in the field or poor field sample recovery techniques that contribute to elevated field train recovery blank results. All sample processing and gravimetric analysis was performed at ERG’s laboratory facility.

4.1 Procedures

Three grades of each Method 202 sample recovery and processing reagent was evaluated to determine their residual blank levels. The three grades of water evaluated were:

- ASTM Type II quality ion exchange water (BDH, cat# BDH1168-4LP)
- Ultrafiltered water (Fisher DIUF, cat# W2-4)
- Water distilled in glass (Teknova PCR Certified, W3350).

The residue on evaporation information was not available from the manufacturer for these three grades of water. At least 5 L from the same lot of each grade of water was obtained and the samples were processed as received from the vendor without any additional preparation. The three grades of hexane evaluated were:

- Hexane with a residual on evaporation value of less than 10 ppmw (GFS, cat# 928)
- Hexane with a residual on evaporation value of 2 ppmw (Sigma-Aldrich Chromasolv, cat# 270504-1L)
- Hexane with a residual on evaporation value of less than 0.1 ppmw (Fisher Optima, cat # H306-4).

All residue on evaporation concentrations were taken from the manufacturer's certifications of analysis for each grade of hexane used. At least 5 L from the same lot of each grade of hexane was obtained and the samples were processed as received from the vendor without any additional preparation. Ten aliquots of 450 mL of each grade of each reagent were processed and analyzed according to the reagent blank procedures of Method 202, generating a total of 30 water and 30 hexane samples. Each aliquot of 450 mL is three times the volume required for reagent blanks in Method 202. ERG personnel processed and gravimetrically analyzed all the samples at ERG's laboratory. A summary of all samples generated in this task and associated sample identification codes is presented in Table 15.

Table 15. Task 4 Sample Summary

Reagent	ID Code	Number of Samples
ASTM Type II Ion Exchange Water	WA01-10	10
Ultrafiltered Water	WB01-10	10
Water Distilled in glass	WC01-10	10
Hexane with Residual on Evaporation of <10 ppmw	HA01-10	10
Hexane with Residual on Evaporation of 2 ppmw	HB01-10	10
Hexane with Residual on Evaporation of <0.1 ppmw	HC01-10	10

Glassware & Laboratory Prep

All laboratory glassware used for sample processing, including beakers and graduated cylinders, were washed, rinsed with DI water and hexane, and baked in an oven at 350°C for six hours before use. All reagents used in the preparation of the glassware met the criteria established in Method 202 of an evaporation residue of < 1.0 mg/L.

Before sample recovery began all areas of ERG's laboratory where samples and the associated recovery and processing materials were to be handled, including the laboratory fume hood, were thoroughly cleaned to prevent any contamination. The benchtop in the fume hood where the recoveries took place was wiped down using water and paper towels.

Sample Recovery Procedures

Task 4 Method 202 reagent blanks were generated according to the procedures in Method 202. The reagent blank samples were the aliquots of the reagents themselves. Ten aliquots of each grade of both reagents were taken, each of which was ultimately evaporated to dryness in its own aluminum weighing pan for gravimetric analysis. Sample beakers were labeled with the unique sample ID, date, and operator initials. Reagent blank samples were generated by:

- Measuring 450 mL of the reagent using a graduated cylinder
- Pouring the reagent into the sample beaker
- Repeating this step nine times for a total of ten samples of each reagent grade.

Ten samples of each of the three selected grades of each reagent were processed for a total of 60 reagent blank samples. Sample handling was a critical component in maintaining high quality and preventing contamination for this task and great care was taken to ensure sample integrity and proper identification. After recovery the samples were processed for gravimetric analysis by ERG personnel.

Sample Processing Procedures

The water sample beakers were placed on a hot plate set below the boiling point of water to expedite evaporation to approximately 50 mL. The water sample beakers were then placed in an oven set to 105°C to evaporate to approximately 10 mL. Once evaporated, the water samples were then allowed to cool in the fume hood and quantitatively transferred into numbered, tared weighing pans using the appropriate grade of water and the pan numbers were recorded in the lab notebook.

The hexane sample beakers were placed into the laboratory fume hood where they were allowed to evaporate to approximately 10 mL. The hexane samples were then quantitatively transferred into numbered, tared weighing pans using the appropriate grade of hexane and the pan numbers were recorded in the lab notebook.

The sample pans were allowed to evaporate to dryness in the fume hood and placed in desiccators in the balance room for gravimetric analysis. Samples were handled and processed in a manner that ensured the integrity of samples and minimized the opportunity for contamination. Nitrile gloves were worn during all sample handling and processing.

4.2 Quality Control

All work conducted in this task followed the reagent blank recovery procedures in Method 202 as written. The solvents used for glassware cleaning in Task 4 met the specifications in Method 202, which requires the use of water and hexane that have a manufacturer's stated residue on evaporation value of 1.0 mg/L or less. Samples were processed in beakers that were cleaned, solvent rinsed, and baked prior to use. All beakers were labeled with the unique sample ID, date, and operator initials. Weighing pans were numbered and the corresponding sample IDs were recorded in the lab notebook. Sample handling was a critical component in maintaining high quality for this task and great care was taken to process samples in a manner that ensured the integrity of samples and minimized the opportunity for contamination. All sample weights were recorded in the balance room lab notebook with the conditions and balance calibration check information for that day, see Table 16, the Gravimetric Analysis Daily Conditions table and Table 17, the Gravimetric Analysis Daily Balance Calibration table.

Table 16. Task 4 Gravimetric Analysis Daily Conditions

Pan #	Tare Weights			Constant Weights			Additional Weights		
	Date	Temp (°F)	%RH	Date	Temp (°F)	%RH	Date	Temp (°F)	%RH
01	7/31/2013	75.37	59.6	8/21/2013	75.37	63.8	8/23/2013	75.26	60.6
02	8/6/2013	75.59	58.1	9/5/2013	76.61	52.8	9/10/2013	76.72	54.9
03	7/31/2013	75.37	59.6	8/23/2013	75.26	60.6	9/3/2013	74.47	65.9
04	7/31/2013	75.37	59.6	8/21/2013	75.37	63.8	8/23/2013	75.26	60.6
05	8/6/2013	75.59	58.1	8/23/2013	75.26	60.6	9/3/2013	74.47	65.9
06	8/6/2013	75.59	58.1	8/23/2013	75.26	60.6	9/3/2013	74.47	65.9
07	8/6/2013	75.59	58.1	9/3/2013	74.47	65.9	9/5/2013	76.61	52.8
08	8/6/2013	75.59	58.1	8/21/2013	75.37	63.8	8/23/2013	75.26	60.6
09	7/31/2013	75.37	59.6	9/5/2013	76.61	52.8	9/10/2013	76.72	54.9
10	7/31/2013	75.37	59.6	9/10/2013	76.72	54.9	9/11/2013	76.27	53.7
11	8/8/2013	74.47	61.3	8/21/2013	75.37	63.8	8/23/2013	75.26	60.6
12	8/14/2013	75.48	58.6	9/3/2013	74.47	65.9	9/5/2013	76.61	52.8
13	8/8/2013	74.47	61.3	8/23/2013	75.26	60.6	9/27/2013	75.48	48.1
14	8/8/2013	74.47	61.3	9/10/2013	76.72	54.9	9/11/2013	76.27	53.7
15	8/8/2013	74.47	61.3	8/22/2013	75.59	63.9	8/23/2013	75.26	60.6
16	8/8/2013	74.47	61.3	8/23/2013	75.26	60.6	9/3/2013	74.47	65.9
17	8/8/2013	74.47	61.3	8/22/2013	75.59	63.9	8/23/2013	75.26	60.6
18	8/9/2013	75.26	66.9	8/23/2013	75.26	60.6	9/3/2013	74.47	65.9
19	8/9/2013	75.26	66.9	9/27/2013	75.48	48.1	10/21/2013	70.87	39.4
20	8/9/2013	75.26	66.9	8/23/2013	75.26	60.6	9/3/2013	74.47	65.9
21	8/9/2013	75.26	66.9	8/23/2013	75.26	60.6	9/3/2013	74.47	65.9
22	8/9/2013	75.26	66.9	8/23/2013	75.26	60.6	9/3/2013	74.47	65.9
23	8/13/2013	74.47	66.6	9/10/2013	76.72	54.9	9/11/2013	76.27	53.7
24	8/15/2013	74.47	52.3	9/3/2013	74.47	65.9	9/5/2013	76.61	52.8
25	8/12/2013	74.69	63.3	8/23/2013	75.26	60.6	9/3/2013	74.47	65.9
26	8/12/2013	74.69	63.3	8/23/2013	75.26	60.6	9/3/2013	74.47	65.9
27	8/12/2013	74.69	63.3	9/10/2013	76.72	54.9	9/27/2013	75.48	48.1

Table 16. Task 4 Gravimetric Analysis Daily Conditions (Continued)

Pan #	Tare Weights			Constant Weights			Additional Weights		
	Date	Temp (°F)	%RH	Date	Temp (°F)	%RH	Date	Temp (°F)	%RH
28	8/9/2013	75.26	66.9	8/23/2013	75.26	60.6	9/3/2013	74.47	65.9
29	8/12/2013	74.69	63.3	9/5/2013	76.61	52.8	9/10/2013	76.72	54.9
30	8/15/2013	74.47	52.3	9/11/2013	76.27	53.7	9/27/2013	75.48	48.1
31	8/12/2013	74.69	63.3	9/10/2013	76.72	54.9	9/27/2013	75.48	48.1
32	8/12/2013	74.69	63.3	9/12/2013	76.83	56.9	9/13/2013	74.24	55.2
33	8/12/2013	74.69	63.3	9/5/2013	76.61	52.8	9/10/2013	76.72	54.9
34	8/14/2013	75.48	58.6	9/10/2013	76.72	54.9	9/11/2013	76.27	53.7
35	8/12/2013	74.69	63.3	9/10/2013	76.72	54.9	9/11/2013	76.27	53.7
36	8/12/2013	74.69	63.3	9/10/2013	76.72	54.9	9/11/2013	76.27	53.7
37	8/15/2013	74.47	52.3	9/12/2013	76.83	56.9	9/13/2013	74.24	55.2
38	8/15/2013	74.47	52.3	9/12/2013	76.83	56.9	9/13/2013	74.24	55.2
39	8/16/2013	75.37	53.5	9/17/2013	75.82	45.7	9/18/2013	71.54	48.8
40	8/14/2013	75.48	58.6	9/12/2013	76.83	56.9	9/13/2013	74.24	55.2
41	8/16/2013	75.37	53.5	9/12/2013	76.83	56.9	9/13/2013	74.24	55.2
42	8/16/2013	75.37	53.5	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
43	8/16/2013	75.37	53.5	9/16/2013	75.37	52.4	10/17/2013	73.68	59.0
45	8/16/2013	75.37	53.5	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
46	8/16/2013	75.37	53.5	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
47	8/15/2013	74.47	52.3	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
48	8/19/2013	75.48	62.1	9/26/2013	75.03	47.3	9/27/2013	75.48	48.1
49	8/16/2013	75.37	53.5	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
50	8/16/2013	75.37	53.5	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
51	8/16/2013	75.37	53.5	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
52	8/16/2013	75.37	53.5	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
53	8/16/2013	75.37	53.5	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
54	8/16/2013	75.37	53.5	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
55	8/19/2013	75.48	62.1	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
56	8/19/2013	75.48	62.1	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
57	8/19/2013	75.48	62.1	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
58	8/19/2013	75.48	62.1	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
59	8/19/2013	75.48	62.1	9/16/2013	75.37	52.4	10/17/2013	73.68	59.0
60	8/19/2013	75.48	62.1	9/13/2013	74.24	55.2	9/16/2013	75.37	52.4
61	8/20/2013	74.92	63.2	9/18/2013	71.54	48.8	9/23/2013	73.91	47.4
70	8/19/2013	75.48	62.1	9/18/2013	71.54	48.8	9/23/2013	73.91	47.4
126	9/9/2013	71.43	58.6	9/20/2013	71.21	50.9	9/26/2013	75.03	47.3
128	9/9/2013	71.43	58.6	9/27/2013	75.48	48.1	10/22/2013	70.64	51.4

Table 17. Task 4 Gravimetric Analysis Daily Balance Calibrations

Date	Known (g)	Measured (g)	Date	Known (g)	Measured (g)
7/31/2013	6.00000	6.00006	9/3/2013	6.00000	6.00005
	6.20000	6.20005		6.00500	6.00503
	6.50000	6.50005		6.50000	6.50005
8/6/2013	6.00000	6.00009	9/5/2013	6.00000	6.00003
	6.20000	6.20006		6.00500	6.00503
	6.50000	6.50008		6.50000	6.50002
8/8/2013	6.00000	6.00008	9/10/2013	6.00000	6.00001
	6.20000	6.20010		6.00500	6.00501
	6.50000	6.50008		6.50000	6.50001
8/9/2013	6.00000	6.00010	9/11/2013	6.00000	6.00002
	6.20000	6.20009		6.00500	6.00500
	6.50000	6.50010		6.50000	6.49998
8/12/2013	6.00000	6.00005	9/12/2013	6.00000	6.00003
	6.20000	6.20003		6.00500	6.00502
	6.50000	6.50007		6.50000	6.50000
8/13/2013	6.00000	6.00002	9/13/2013	6.00000	6.00003
	6.20000	6.20003		6.00500	6.00504
	6.50000	6.50002		6.50000	6.50004
8/14/2013	6.00000	6.00001	9/16/2013	6.00000	6.00003
	6.20000	6.20003		6.00500	6.00503
	6.50000	6.50000		6.50000	6.50006
8/15/2013	6.00000	6.00000	9/17/2013	6.00000	6.00004
	6.20000	6.20004		6.00500	6.00502
	6.50000	6.50004		6.50000	6.50002
8/16/2013	6.00000	6.00003	9/18/2013	6.00000	6.00006
	6.20000	6.20004		6.00500	6.00505
	6.50000	6.50005		6.50000	6.50006
8/19/2013	6.00000	6.00002	9/23/2013	6.00000	6.00002
	6.20000	6.20002		6.00500	6.00505
	6.50000	6.50002		6.50000	6.50006
8/20/2013	6.00000	6.00002	9/26/2013	6.00000	6.00004
	6.00500	6.00502		6.00500	6.00501
	6.50000	6.50002		6.50000	6.50003
8/21/2013	6.00000	6.00004	9/27/2013	6.00000	6.00004
	6.00500	6.00502		6.00500	6.00501
	6.50000	6.50004		6.50000	6.50003
8/22/2013	6.00000	6.00005	10/17/2013	6.00000	6.00003
	6.00500	6.00503		6.00500	6.00503
	6.50000	6.50003		6.50000	6.50003
8/23/2013	6.00000	6.00002	10/21/2013	6.00000	6.00001
	6.00500	6.00500		6.00500	6.00500
	6.50000	6.50001		6.50000	6.50002

4.3 Analytical Results

The residual mass of every sample generated in this task was measured by gravimetric analysis. The raw results, in grams, are presented in Table 18, the Task 4 Raw Sample Results table. All negative results were reported as non-detects.

Table 18. Task 4 Raw Sample Results

Pan #	Constant Weight (g)	Additional Weight (g)	Average Weight (g)	Tare Weight (g)	Net Constant Weight (g)	Net Average Weight (g)
01	6.31627	6.31625	6.31626	6.31511	0.00116	0.00115
02	6.32277	6.32276	6.32277	6.32158	0.00119	0.00119
03	6.34871	6.34872	6.34872	6.34750	0.00121	0.00122
04	6.38325	6.38321	6.38323	6.38205	0.00120	0.00118
05	6.32208	6.32208	6.32208	6.32092	0.00116	0.00116
06	6.28879	6.28884	6.28882	6.28758	0.00121	0.00123
07	6.24101	6.24096	6.24099	6.23999	0.00102	0.00100
08	6.34207	6.34210	6.34209	6.34095	0.00112	0.00113
09	6.30171	6.30170	6.30171	6.30095	0.00076	0.00075
10	6.35562	6.35562	6.35562	6.35471	0.00091	0.00091
11	6.28352	6.28354	6.28353	6.28358	ND	ND
12	6.26693	6.26694	6.26694	6.26707	ND	ND
13	6.29121	6.29124	6.29123	6.29163	ND	ND
14	6.26758	6.26756	6.26757	6.26798	ND	ND
15	6.25430	6.25427	6.25429	6.25451	ND	ND
16	6.25371	6.25374	6.25373	6.25402	ND	ND
17	6.31907	6.31903	6.31905	6.31894	0.00013	0.00011
18	6.28038	6.28039	6.28039	6.28048	ND	ND
19	6.28038	6.28035	6.28037	6.28064	ND	ND
20	6.26252	6.26249	6.26251	6.26266	ND	ND
21	6.27589	6.27589	6.27589	6.27603	ND	ND
22	6.28392	6.28397	6.28395	6.28496	ND	ND
23	6.38514	6.38510	6.38512	6.38503	0.00011	0.00009
24	6.30407	6.30405	6.30406	6.30413	ND	ND
25	6.31173	6.31174	6.31174	6.31193	ND	ND
26	6.29582	6.29580	6.29581	6.29596	ND	ND
27	6.32002	6.31998	6.32000	6.32000	0.00002	0.00000
28	6.38082	6.38078	6.38080	6.38087	ND	ND
29	6.36037	6.36034	6.36036	6.36069	ND	ND
30	6.39350	6.39349	6.39350	6.39373	ND	ND
31	6.33208	6.33209	6.33209	6.33200	0.00008	0.00009
32	6.27526	6.27527	6.27527	6.27527	ND	0.00000
33	6.32049	6.32046	6.32048	6.32068	ND	ND
34	6.29174	6.29176	6.29175	6.29198	ND	ND
35	6.35648	6.35647	6.35648	6.35649	ND	ND
36	6.35951	6.35951	6.35951	6.35976	ND	ND
37	6.34310	6.34305	6.34308	6.34317	ND	ND
38	6.34373	6.34373	6.34373	6.34385	ND	ND
39	6.30671	6.30667	6.30669	6.30697	ND	ND

Negative results are reported as non-detects (ND)

Table 18. Task 4 Raw Sample Results (Continued)

Pan #	Constant Weight (g)	Additional Weight (g)	Average Weight (g)	Tare Weight (g)	Net Constant Weight (g)	Net Average Weight (g)
40	6.36842	6.36841	6.36842	6.36843	ND	ND
41	6.34495	6.34494	6.34495	6.34484	0.00011	0.00011
42	6.36703	6.36704	6.36704	6.36705	ND	ND
43	6.31679	6.31677	6.31678	6.31673	0.00006	0.00005
45	6.29386	6.29387	6.29387	6.29388	ND	ND
46	6.36628	6.36626	6.36627	6.36621	0.00007	0.00006
47	6.33817	6.33823	6.33820	6.33835	ND	ND
48	6.36110	6.36111	6.36111	6.36148	ND	ND
49	6.35749	6.35747	6.35748	6.35743	0.00006	0.00005
50	6.32528	6.32526	6.32527	6.32522	0.00006	0.00005
51	6.35478	6.35476	6.35477	6.35468	0.00010	0.00009
52	6.31663	6.31666	6.31665	6.31651	0.00012	0.00013
53	6.38943	6.38950	6.38947	6.38933	0.00010	0.00013
54	6.39084	6.39086	6.39085	6.39096	ND	ND
55	6.33017	6.33020	6.33019	6.33031	ND	ND
56	6.34846	6.34844	6.34845	6.34859	ND	ND
57	6.36598	6.36593	6.36596	6.36610	ND	ND
58	6.33377	6.33371	6.33374	6.33387	ND	ND
59	6.32796	6.32793	6.32795	6.32818	ND	ND
60	6.35956	6.35949	6.35953	6.35956	0.00000	ND
61	6.33187	6.33184	6.33186	6.33196	ND	ND
70	6.37406	6.37408	6.37407	6.37445	ND	ND
126	6.25186	6.25184	6.25185	6.25170	0.00016	0.00015
128	6.24488	6.24488	6.24488	6.24394	0.00094	0.00094

Negative results are reported as non-detects (ND)

Results for the reagent blanks for the three grades of water and of hexane, in mg, are in Tables 19 and 20, the Reagent Blank Results tables.

Table 19. Water Sample Results

WA				WB				WC			
Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)	Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)	Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)
WA01	11	ND	ND	WB01	23	0.11	0.09	WC01	41	0.11	0.11
WA02	13	ND	ND	WB02	25	ND	ND	WC02	42	ND	ND
WA03	14	ND	ND	WB03	26	ND	ND	WC03	43	0.06	0.05
WA04	15	ND	ND	WB04	27	0.02	0.00	WC04	45	ND	ND
WA05	16	ND	ND	WB05	28	ND	ND	WC05	46	0.07	0.06
WA06	17	0.13	0.11	WB06	29	ND	ND	WC06	49	0.06	0.05
WA07	18	ND	ND	WB07	31	0.08	0.09	WC07	50	0.06	0.05
WA08	19	ND	ND	WB08	32	ND	ND	WC08	51	0.10	0.09
WA09	20	ND	ND	WB09	33	ND	ND	WC09	52	0.12	0.13
WA10	21	ND	ND	WB10	35	ND	ND	WC10	53	0.10	0.13

Table 20. Hexane Sample Results

HA				HB				HC			
Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)	Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)	Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)
HA01	01	1.16	1.15	HB01	12	ND	ND	HC01	48	ND	ND
HA02	02	1.19	1.19	HB02	24	ND	ND	HC02	54	ND	ND
HA03	03	1.21	1.22	HB03	30	ND	ND	HC03	55	ND	ND
HA04	04	1.20	1.18	HB04	34	ND	ND	HC04	56	ND	ND
HA05	05	1.16	1.16	HB05	36	ND	ND	HC05	57	ND	ND
HA06	06	1.21	1.23	HB06	37	ND	ND	HC06	58	ND	ND
HA07	07	1.02	1.00	HB07	38	ND	ND	HC07	59	ND	ND
HA08	08	1.12	1.13	HB08	39	ND	ND	HC08	60	ND	ND
HA09	09	0.76	0.75	HB09	40	ND	ND	HC09	61	ND	ND
HA10	10	0.91	0.91	HB10	47	ND	ND	HC10	70	ND	ND

Results for the two highest grades of hexane revealed the residual masses were not detectable by gravimetric analysis at the volume processed in this task. The sample volume of 450 mL (3 times the Method 202 reagent blank volume) was selected for this reagent evaluation based on the calculation that the manufacturer's residue on evaporation specification value for the highest purity hexane selected would produce a residual mass of approximately 0.45 mg and be detectable on the 5-place analytical balance used in this task. In fact, the actual manufacturer's stated residual on evaporation value for the highest purity hexane was 10 times lower than the specification, resulting in much lower residual masses. Statistical analyses and replicate measurement comparisons of the data sets can be seen in Tables 21 and 22.

Table 21. Water Statistical Analysis

	WA Net Const Wt (mg)	WA Net Avg Wt (mg)	WB Net Const Wt (mg)	WB Net Avg Wt (mg)	WC Net Const Wt (mg)	WC Net Avg Wt (mg)
Average	0.01	0.01	0.02	0.02	0.07	0.07
Standard Deviation	0.04	0.03	0.04	0.04	0.04	0.05
Detection Limit	0.12	0.10	0.12	0.11	0.13	0.15
% RSD	316.23	316.23	191.07	210.93	61.93	71.75
UPL (n=10)	0.04	0.03	0.05	0.04	0.09	0.10

Table 22. Hexane Statistical Analysis

	HA Net Const Wt (mg)	HA Net Avg Wt (mg)	HB Net Const Wt (mg)	HB Net Avg Wt (mg)	HC Net Const Wt (mg)	HC Net Avg Wt (mg)
Average	1.09	1.09	0.00	0.00	0.00	0.00
Standard Deviation	0.15	0.16	0.00	0.00	0.00	0.00
Detection Limit	0.46	0.47	0.00	0.00	0.00	0.00
% RSD	13.88	14.25	NA	NA	NA	NA
UPL (n=10)	1.19	1.19	0.00	0.00	0.00	0.00

NA is Not Applicable

The target of 95% of sample results falling at or below the calculated UPL value was not achieved for all data sets. This can be attributed to the fact that the number of samples in the data sets is low enough that one result above the UPL caused the percentage of samples at or below the UPL to be less than 95%. The results of the dust pan and the reagent blanks for the hexane and water used in for glassware cleaning and preparation are displayed in Table 23, the Reagent Blank and Dust Pan Results table.

Table 23. Task 4 Reagent Blank and Dust Pan Results

Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)
DP	22	ND	ND
RBH	126	0.16	0.15
RBW	128	0.94	0.94

The dust pan residual mass was a non-detect, which shows that samples were not contaminated by dust during sample pan evaporation. Reagent blank results show a very low residual mass which is less than the method criteria for the hexane used and a residual mass of less than 1.0 mg for the water. These hexane and water reagent blanks were taken from the reagents used in the glassware cleaning and preparation.

4.4 Conclusions

Results for most of the water reagent blank samples evaluated for WA and WB were non-detects, as seen in Table 19. The detected results were very low. This indicates that the ion exchange water that meets ASTM Type II specifications and the ultrafiltered water both meet the Method 202 reagent blank limit. WC had measurable results for 8 of the 10 samples analyzed, which were all very low and shows that WC meets the Method 202 reagent blank limit as well. The WC reagent blank results are presented in Figure 10, the Water C residual mass results using the net average weights graph.

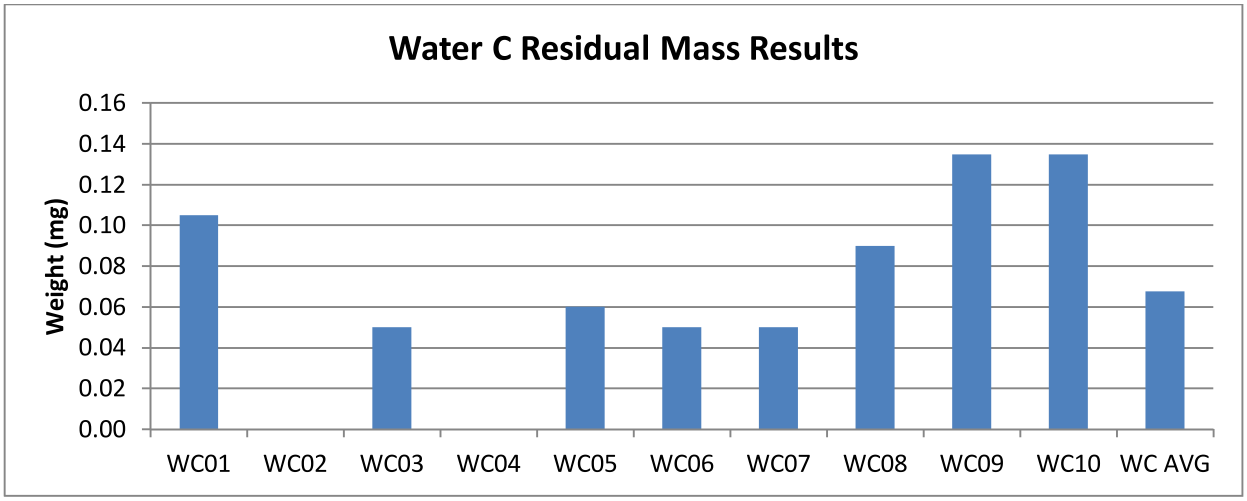

Figure 10. Water C Reagent Blank Residual Mass Results (Net Average Weights)

Results for the hexane reagent blank samples evaluated for HB and HC were all non-detects, as seen in Table 20. This indicates that the hexane with a residue on evaporation value of 2 ppmw and the hexane with a residue on evaporation value of less than 0.1 ppmw both meet the Method 202 reagent blank limit. Measurable results were obtained for all 10 HA samples analyzed, which were all very consistent and show that HA does not meet the Method 202 reagent blank limit. The HA reagent blank results are presented in Figure 11, the Hexane A residual mass results using the net average weights graph. This result was expected as the manufacturer's stated residual on evaporation was above the Method 202 reagent blank limit.

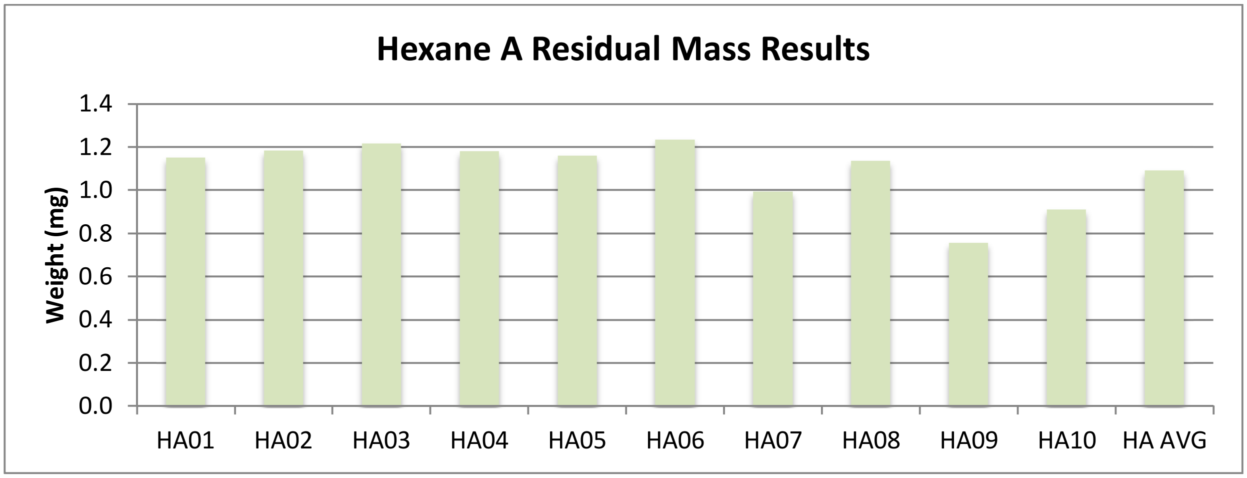

Figure 11. Hexane A Reagent Blank Residual Mass Results (Net Average Weights)

These results show that of all the reagents evaluated, only the hexane with the highest stated residue on evaporation, HA, had results high enough to exceed the reagent blank limit of Method 202. The amount of volume evaluated, 450 mL, three times the volume specified in the method, did not result in enough residual mass to be detected on the balance for most of the measurements for two of the waters and two of the hexanes. This finding highlights the fact that the volume required for reagent blanks in Method 202, 150 mL, is low enough that the residual mass of reagents that meet the reagent blank limit of the method will be difficult to detect gravimetrically. A comparison of the average reagent blank residual mass results for all six reagents is presented in Figure 12, the Average Reagent Blank Results using net average weights graph.

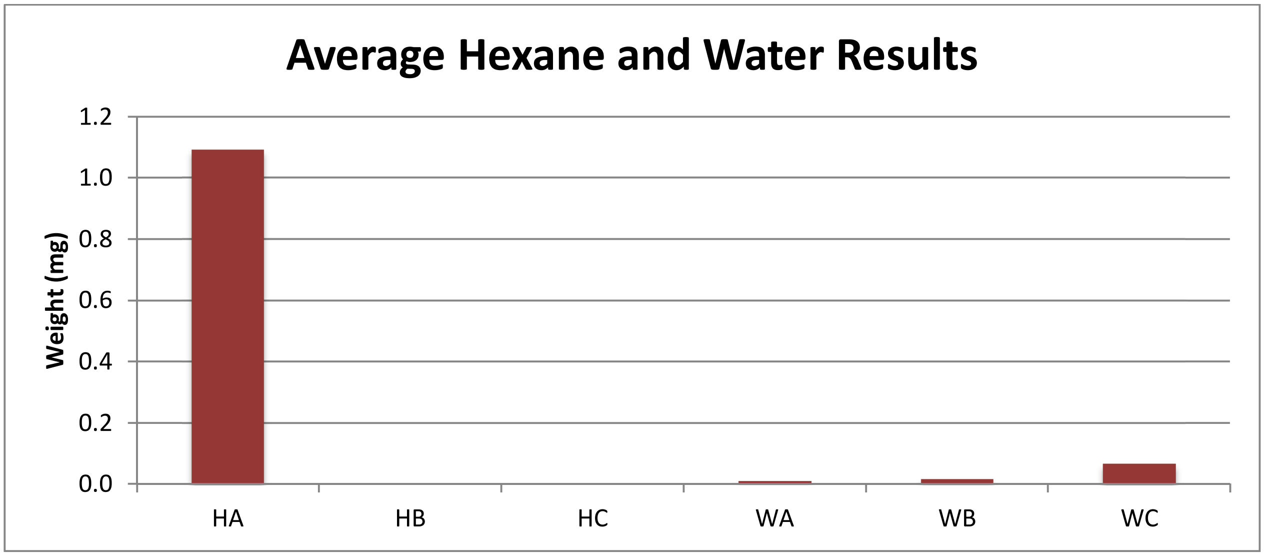

Figure 12. Average Hexane and Water Results (Net Average Weights)

A comparison of the %RSDs calculated using the results of the net constant weights and using the net average weights for all three six reagents is presented in Figure 13, the %RSD Comparison graph. This graph shows similar, but slightly lower %RSDs for the Constant Weights compared to the Average Weights for every set of measurements except for those associated with the WA samples. The HB and HC samples were non-detects. The water %RSDs are high due to the especially low values and non-detects for those sample sets.

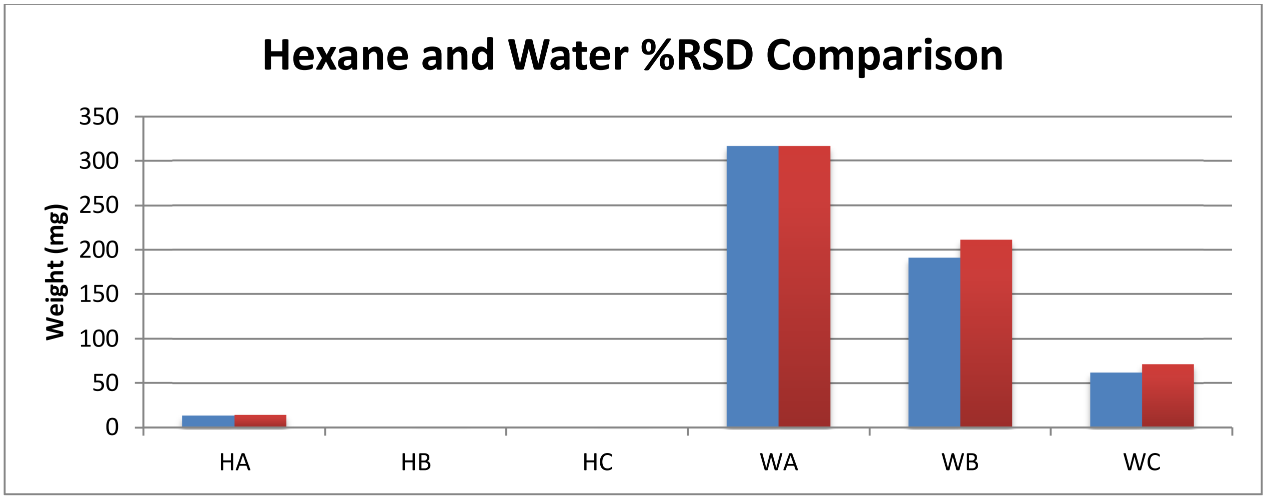

Figure 13. Hexane and Water %RSD Comparison

A comparison of the UPLs calculated for each of the six reagents using the net average weights is presented in Figure 14, the Reagent Blank UPL Comparison graph. The HA reagent blank samples had the highest UPL and were the only reagent to exceed the reagent blank limit.

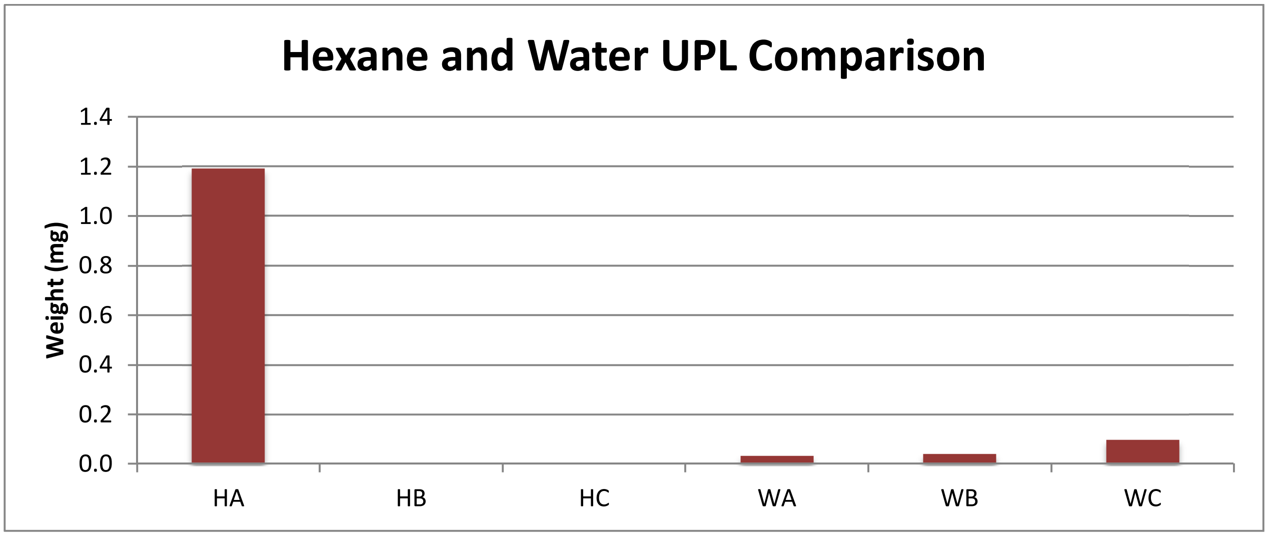

Figure 14. Reagent Blank UPL Comparison (Net Average Weights)

Even though most of the reagents evaluated in this task did happen to pass the reagent blank limit for the residue on evaporation, these results do not indicate that evaluation of reagents to be used can be chosen without regard for the manufacturer's stated specification or without reagent blank evaluation. Hexane with a stated residue on evaporation above, but near the reagent blank limit did not pass on evaluation.

The average results and UPL results show that with proper reagent selection the contribution to the Method 202 field sample result can be kept at a minimum. Using due diligence and following the requirements of the method would preclude the HA hexane reagent evaluated in this task from being used in source testing, but without evaluation that would not be known. Requiring an evaluation to prove the reagent blanks for reagents proposed for use in Method 202 source testing pass the reagent blank limit is suggested as a Best Practice for this method.

5.0 METHOD 202 SAMPLING TRAIN EVALUATION

Method 202 sampling train glassware evaluations were performed from August through November of 2013 at Air-Tech Environmental and the ERG laboratory facility, in accordance with WA 3-07 Task 5. Method 202 field train recovery blank levels greater than the allowable 2.0 mg limit established in the method have been reported by source testers. Field train recovery blank concentrations need to be very low to ensure that results for the CPM measurements are attributable to, and representative of, the source and not to the sampling trains used in the sample collection. Task 5 was designed to evaluate the ability to clean Method 202 sampling train glassware to achieve sufficiently low blank results and evaluate the potential contribution of individual components of the sampling train to the total blank concentration.

Method 202 sampling trains consist of a probe extension that is glass or fluoropolymer-lined, a condenser, a water dropout impinger, a modified Greenburg-Smith impinger with an open tube tip, a CPM filter with filter holder, a modified Greenburg-Smith impinger containing 100 mL water, and an impinger containing silica gel. In addition to the CPM filter, Method 202 field samples are generated from the recovery of sampling train components using water, acetone, and hexane. When operating in conjunction with Method 201A, which is first in line, Method 202 samples consist of the CPM filter and the recoveries of:

- the back half of the Method 201A filterable PM filter holder
- the probe extension
- the condenser
- the first two impingers
- the front half of the CPM filter holder.

See Figure 1, the combined M201A/202 sampling train configuration schematic.

In this task the residual masses of the Method 202 sampling trains were determined using the field train proof blank procedures, with the exception that CPM filters were not included in this evaluation. The Method 202 sampling train blanks were obtained by:

- Assembling the sampling train as it would be for testing, without the CPM filter
- Recovering the inorganic fraction of the sample by rinsing the sampling train twice with water, collecting the rinses in the inorganic sample container
- Recovering the organic fraction of the sample by rinsing the sampling train once with acetone and twice with hexane, collecting the rinses in the organic sample container
- Extracting the inorganic fraction three times with hexane using a separatory funnel, combining the extracts with the organic fraction
- Evaporating the samples to approximately 10 mL in beakers

- Evaporating the samples to dryness in aluminum weigh pans
- Desiccating the samples for at least 24 hours
- Gravimetrically analyzing the sample pans.

The sample recovery was conducted following the procedures in Method 202 on a sampling train without any additional water, a nitrogen purge, or a CPM filter.

Task 5 evaluated whether blanks of Method 202 sampling trains can pass the 2.0 mg field train recovery blank limit from the method. The evaluation was accomplished by recovering and analyzing blanks from Method 202 sampling trains that have been thoroughly cleaned, solvent rinsed, and baked as required by the method. In addition to the thorough DI water rinse that the method requires prior to sampling, the sampling trains were also rinsed with acetone and hexane. This effort did not evaluate the potential for contamination in the field or poor field sample recovery techniques that contribute to elevated field train recovery blank results. Task 5 also evaluated the potential contribution of individual components of the sampling train to the total blank. This objective was accomplished by recovering and analyzing individual glassware components from a Method 202 sampling train separately. In order to simulate some aspects of real world Method 202 source testing all the sample recoveries were performed in a mobile laboratory. All further sample processing and gravimetric analysis was performed at ERG's laboratory facility.

5.1 Procedures

Three Method 202 sampling trains that have previously been used to conduct Method 202 source emissions testing were supplied by Air-Tech Environmental. The trains, identified as A, B, and C, were cleaned and prepared as they would be for deployment in the field, following the Method 202 requirements and without a CPM filter. On August 27, 2013 Air-Tech Environmental personnel, under supervision of the ERG Project Director, recovered the sampling trains according to the proof blank procedures of Method 202. The recoveries were conducted in the mobile lab at Air-Tech Environmental. Train C was then recovered two additional times for a total of five full Method 202 sampling train samples as follows:

- Train A Recovery (A)
- Train B Recovery (B)
- Train C Three Recoveries (C1, C2, and C3).

Train A was separated into its components; the probe extension including the back half of the Method 201A filterable PM filter holder, the condenser, the impingers with connecting glassware, and the front half of the Method 202 CPM filter housing. Each individual component was then recovered independently four times generating 16 samples:

- Probe Extension 4 Recoveries (PE1, PE2, PE3, and PE4)
- Condenser 4 Recoveries (CND1, CND2, CND3, and CND4)
- Impingers 4 Recoveries (IMP1, IMP2, IMP3, and IMP4)
- Filter Housing 4 Recoveries (FH1, FH2, FH3, and FH4).

See Table 24, the Task 5 Sample Summary, for a breakdown of all the samples generated in this task. Each sample listed in the table consists of two fractions, an inorganic and an organic. ERG processed and gravimetrically analyzed all the samples at ERG's laboratory. All solvents used for all activities conducted in Task 5, including cleaning, preparation, sample recovery, and sample processing, met the specifications required in Method 202 that manufacturer's stated residue on evaporation not exceed 1.0 mg/L.

Table 24. Task 5 Sample Summary

Sample	ID Code	Number of Samples
Method 202 Train A	A	1
Method 202 Train B	B	1
Method 202 Train C	C1-3	3
Probe extension with M201A filter housing (back half)	PE1-4	4
Condenser	CND1-4	4
Impingers with connecting glassware	IMP1-4	4
CPM filter housing (front half)	FH1-4	4

Sampling Train Glassware Prep

All components from each of these trains were cleaned by Air-Tech Environmental personnel. The probe extensions for Trains A, B, and C were approximately 6 feet in length. The trains were cleaned by:

1. Washing with soap and water
2. Scrubbing with a brush
3. Rinsing with DI water, acetone, and hexane
4. Baking at 300°C for six hours at the ERG laboratory (the back halves of the M201A filterable PM filter holders and the probe extensions were not baked).

On August 27, 2013, immediately prior to the sample recoveries, all components of all three trains were rinsed with DI water, acetone, and hexane. All reagents used in the preparation of the sampling trains met the criteria established in Method 202 of an evaporation residue of < 1.0 mg/L.

Sample Recovery Procedures

Before sample recovery began the mobile laboratory at Air-Tech Environmental was thoroughly cleaned to prevent any contamination. The benchtop and surrounding areas where the recoveries took place were wiped down using water and paper towels. In addition, the benchtop was covered with aluminum foil. See Figure 15 for a picture of the Air-Tech Environmental Mobile Lab sample recovery area used for all sample recoveries performed for this task.

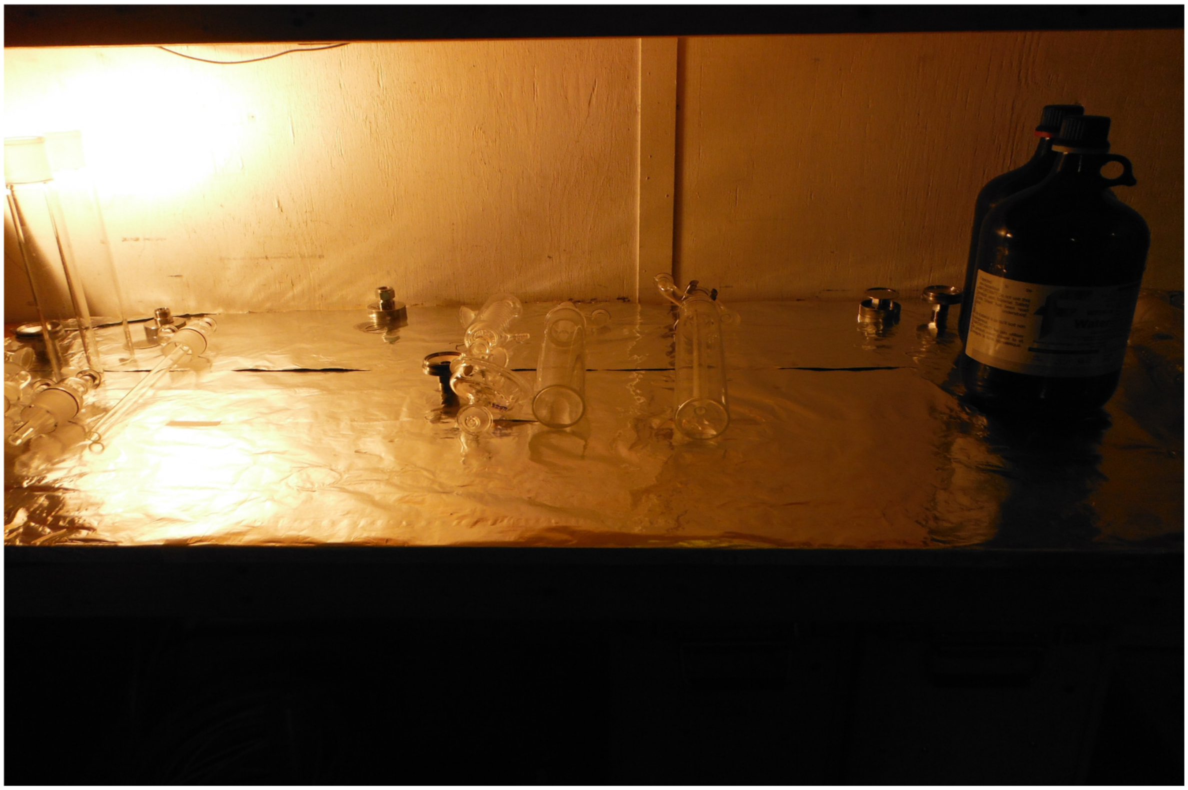

Figure 15. Air-Tech Environmental Mobile Lab Sample Recovery Area

Task 5 Method 202 full train samples were generated by recovering the sampling trains according to the field train proof blank procedures Method 202, omitting the CPM filter, using trains A, B, and C. The sampling trains were prepared as they would be for source testing in the field, including washing, solvent rinsing, and baking. Following the field train proof blank procedures, the trains were not purged with nitrogen and water was not added to any of the impingers. For this task the CPM filter was not included. The Method 202 field train proof blank recovery procedures generate two sample fractions, an inorganic and an organic, each of which is ultimately evaporated to dryness in its own aluminum weighing pan for gravimetric analysis. The inorganic fraction (Note: the term aqueous, Aq, was used for sample identification codes for inorganic fraction samples in this task) is generated by rinsing sample portion of the train twice with water and collecting the rinses in the inorganic fraction sample bottle. The components of the sample portion of the train include:

- the back half of the Method 201A filterable PM filter holder
- the probe extension
- the condenser
- the first two impingers with connecting glassware
- the front half of the CPM filter holder.

High purity Fisher Optima water, with an evaporation residue of < 1.0 mg/L, was used for inorganic fraction recoveries. The organic fraction is generated by rinsing the sample portion of the train once with acetone then twice with hexane and collecting the rinses in the organic fraction sample bottle. Acetone, with an evaporation residue of < 0.5 mg/L, and hexane, with an evaporation residue of < 0.1 mg/L, were used for organic fraction recoveries. Train C was recovered two additional times after the initial sample recovery for three samples from Train C and a total of five Method 202 full sampling train samples.

Task 5 Method 202 sampling train component samples were generated following the same procedures as for full trains with the exception that the rinses of each component were collected separately in their own sample jars. Train A was used for the individual component recoveries after the full train samples were collected without any further preparation. The sampling train was divided into four separate components as follows:

- the probe extension including the back half of the Method 201A filterable PM filter holder (PE)
- the condenser (CND)
- the impingers with connecting glassware (IMP)
- the front half of the Method 202 CPM filter housing (FH).

Inorganic and organic fractions were recovered for each component by collecting the reagent rinses in the appropriate sample bottle. Each of the four individual components received four sequential recoveries for a total of 16 Method 202 sampling train component samples.

All samples, inorganic and organic fractions for both full M202 trains and individual components, were recovered in pre-cleaned 500 mL amber glass jars labeled with the unique sample identification (ID) and date. The sample jars have certificates of analysis from the manufacturer that document the cleanliness specifications. The tare weights were taken for the sample jars before sample recovery and recorded in the lab notebook. Chains of Custody (COC) were used for all samples. Information on the COCs included:

- the unique sample ID
- the glassware train or component
- the date collected

- the operator initials
- whether it is the inorganic or organic fraction.

Sample handling was a critical component in maintaining high quality and preventing contamination for this task and great care was taken to ensure sample integrity and proper identification. After recovery the samples and associated COCs were taken to ERG's laboratory for sample processing and gravimetric analysis by ERG personnel and final weights were measured for the sample jar samples. The sample jar initial and final weights can be seen in Table 25, the Sample Jar Weights table.

Table 25. Task 5 Sample Jar Weights

Sample ID	Jar #	Tare Weight (g)	Final Weight (g)	Net Weight (g)
A Aq	1	275.6	393.8	118.2
A Org	2	277.2	448.9	171.7
B Aq	3	279.4	396.4	117.0
B Org	4	276.0	424.4	148.4
C1 Aq	5	279.5	394.4	114.9
C1 Org	6	279.5	420.1	140.6
C2 Aq	7	275.4	398.6	123.2
C2 Org	8	279.7	445.8	166.1
C3 Aq	9	276.6	397.8	121.2
C3 Org	10	278.3	435.3	157.0
PE1 Aq	11	279.6	306.2	26.6
PE1 Org	12	276.9	310.8	33.9
PE2 Aq	13	279.7	299.2	19.5
PE2 Org	14	277.8	301.5	23.7
PE3 Aq	15	277.0	297.0	20.0
PE3 Org	16	278.9	315.6	36.7
PE4 Aq	17	274.4	298.5	24.1
PE4 Org	18	276.6	294.0	17.4
CND1 Aq	19	278.4	301.0	22.6
CND1 Org	20	279.6	304.0	24.4
CND2 Aq	21	274.3	299.7	25.4
CND2 Org	22	279.7	300.6	20.9
CND3 Aq	23	279.7	305.2	25.5
CND3 Org	24	278.8	300.5	21.7
CND4 Aq	25	276.0	296.9	20.9
CND4 Org	26	277.9	300.8	22.9
IMP1 Aq	27	279.6	325.8	46.2
IMP1 Org	28	279.7	314.9	35.2
IMP2 Aq	29	276.6	323.5	46.9
IMP2 Org	30	277.7	343.4	65.7

Table 25. Task 5 Sample Jar Weights (Continued)

Sample ID	Jar #	Tare Weight (g)	Final Weight (g)	Net Weight (g)
IMP3 Aq	31	274.2	324.0	49.8
IMP3 Org	32	279.7	315.1	35.4
IMP4 Aq	33	279.0	332.3	53.3
IMP4 Org	34	278.4	333.8	55.4
FH1 Aq	35	279.9	300.6	20.7
FH1 Org	36	278.2	293.3	15.1
FH2 Aq	37	280.5	301.0	20.5
FH2 Org	38	277.9	294.8	16.9
FH3 Aq	39	278.8	297.8	19.0
FH3 Org	40	279.6	298.3	18.7
FH4 Aq	41	279.7	298.6	18.9
FH4 Org	42	279.9	299.1	19.2
RBW	43	279.5	484.6	205.1
RBA	44	279.8	433.5	153.7
RBH	45	279.1	409.8	130.7

Sample Processing Procedures

All laboratory glassware used for sample processing, including beakers, separatory funnels, and graduated cylinders, were washed, rinsed with DI water and hexane, and baked in an oven at 350°C for six hours before use. Sample beakers were labeled with the unique sample ID, date, and operator initials. Aluminum weighing pans were numbered and tared before use.

After sample recovery the samples were processed in preparation for gravimetric analysis. The sample recovery procedures yielded an inorganic fraction and an organic fraction for each full train and component, 21 inorganic fraction samples and 21 organic fractions total. The inorganic fraction of each sample was extracted with 30 mL of hexane three times using a separatory funnel and beakers. The resulting hexane extracts were combined with the organic fraction for that sample. The sample beakers were placed into the laboratory fume hood where they were allowed to evaporate to approximately 10 mL. The organic samples were then quantitatively transferred into tared weighing pans using hexane and the pan numbers were recorded in the lab notebook. The inorganic samples were then quantitatively transferred into tared weighing pans using water and the pan numbers were recorded in the lab notebook. The sample pans were allowed to evaporate to dryness in the fume hood and placed in desiccators in the balance room for gravimetric analysis. Samples were handled and processed in a manner that ensured the integrity of samples and minimized the opportunity for contamination. Nitrile gloves were worn during all sample handling and processing.

5.2 Quality Control

All work conducted in this task followed the proof blank procedures in Method 202, with the exception that CPM filters were omitted. The solvents and glassware used for all activities conducted in Task 5 met the specifications in Method 202 which requires the use of water, acetone, and hexane that have a manufacturer's stated residue on evaporation value of 1.0 mg/L or less. Air-Tech Environmental, a firm experienced in Method 202 source testing and that conforms to ASTM D7036-04, provided the Method 202 sampling trains and performed the sample recoveries. Sample recovery rinses were collected in 500 mL amber glass sample jars that came cleaned by the manufacturer with certificates of analysis. All sample containers were labeled with the unique sample ID, date, and operator initials. COCs were used for samples recovered by Air-Tech Environmental personnel. The chains of custody contained the unique sample ID, the full train or component, the date collected, the operator, and whether it is the inorganic or organic fraction. Weighing pans were numbered and the corresponding sample IDs were recorded in the lab notebook. Sample handling was a critical component in maintaining high quality for this task and great care was taken to process samples in a manner that ensured the integrity of samples and minimized the opportunity for contamination. All sample weights were recorded in the balance room lab notebook with the conditions and balance calibration check information for that day, see Table 26, the Gravimetric Analysis Daily Conditions table and Table 27, the Gravimetric Analysis Daily Balance Calibration table.

Table 26. Task 5 Gravimetric Analysis Daily Conditions

Pan #	Tare Weights			Constant Weights			Additional Weights		
	Date	Temp (°F)	%RH	Date	Temp (°F)	%RH	Date	Temp (°F)	%RH
67	9/4/2013	75.03	57.4	9/23/2013	73.91	47.4	9/26/2013	75.03	47.3
83	9/4/2013	75.03	57.4	9/26/2013	75.03	47.3	9/27/2013	75.48	48.1
85	9/4/2013	75.03	57.4	9/26/2013	75.03	47.3	9/27/2013	75.48	48.1
86	9/6/2013	75.71	49.5	9/26/2013	75.03	47.3	9/27/2013	75.48	48.1
87	9/4/2013	75.03	57.4	9/23/2013	73.91	47.4	9/26/2013	75.03	47.3
88	9/4/2013	75.03	57.4	9/18/2013	71.54	48.8	9/23/2013	73.91	47.4
89	9/4/2013	75.03	57.4	9/23/2013	73.91	47.4	9/26/2013	75.03	47.3
90	9/6/2013	75.71	49.5	9/23/2013	73.91	47.4	9/26/2013	75.03	47.3
91	9/4/2013	75.03	57.4	9/18/2013	71.54	48.8	9/23/2013	73.91	47.4
92	9/4/2013	75.03	57.4	9/23/2013	73.91	47.4	9/26/2013	75.03	47.3
93	9/4/2013	75.03	57.4	9/18/2013	71.54	48.8	9/23/2013	73.91	47.4
94	9/6/2013	75.71	49.5	9/23/2013	73.91	47.4	9/26/2013	75.03	47.3
95	9/4/2013	75.03	57.4	9/19/2013	71.88	50.5	9/23/2013	73.91	47.4
96	9/4/2013	75.03	57.4	9/23/2013	73.91	47.4	9/26/2013	75.03	47.3
97	9/4/2013	75.03	57.4	11/12/2013	72.56	27.1	11/12/2013	72.56	27.1
98	9/4/2013	75.03	57.4	9/18/2013	71.54	48.8	9/25/2013	75.37	50.1
99	9/4/2013	75.03	57.4	9/25/2013	75.37	50.1	9/26/2013	75.03	47.3

Table 26. Task 5 Gravimetric Analysis Daily Conditions (Continued)

Pan #	Tare Weights			Constant Weights			Additional Weights		
	Date	Temp (°F)	%RH	Date	Temp (°F)	%RH	Date	Temp (°F)	%RH
100	9/4/2013	75.03	57.4	9/25/2013	75.37	50.1	9/26/2013	75.03	47.3
101	9/4/2013	75.03	57.4	9/18/2013	71.54	48.8	9/25/2013	75.37	50.1
102	9/4/2013	75.03	57.4	9/18/2013	71.54	48.8	9/25/2013	75.37	50.1
103	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
104	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
105	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
106	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
107	9/6/2013	75.71	49.5	9/25/2013	75.37	50.1	9/26/2013	75.03	47.3
108	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
109	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
110	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
111	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
112	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
113	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
114	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
115	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
116	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
117	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
118	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
119	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
120	9/6/2013	75.71	49.5	9/19/2013	71.88	50.5	9/25/2013	75.37	50.1
121	9/9/2013	71.43	58.6	9/20/2013	71.21	50.9	9/25/2013	75.37	50.1
122	9/9/2013	71.43	58.6	9/20/2013	71.21	50.9	9/25/2013	75.37	50.1
123	9/9/2013	71.43	58.6	9/20/2013	71.21	50.9	9/25/2013	75.37	50.1
124	9/9/2013	71.43	58.6	9/20/2013	71.21	50.9	9/25/2013	75.37	50.1
125	9/9/2013	71.43	58.6	9/27/2013	75.48	48.1	10/22/2013	70.64	51.4
126	9/9/2013	71.43	58.6	9/20/2013	71.21	50.9	9/26/2013	75.03	47.3
127	9/9/2013	71.43	58.6	9/26/2013	75.03	47.3	9/27/2013	75.48	48.1
128	9/9/2013	71.43	58.6	9/27/2013	75.48	48.1	10/22/2013	70.64	51.4

Table 27. Task 5 Gravimetric Analysis Daily Balance Calibrations

Date	Known (g)	Measured (g)	Date	Known (g)	Measured (g)
9/4/2013	6.00000	6.00002	9/23/2013	6.00000	6.00002
	6.00500	6.00503		6.00500	6.00505
	6.50000	6.50003		6.50000	6.50006
9/6/2013	6.00000	6.00001	9/25/2013	6.00000	6.00006
	6.00500	6.00501		6.00500	6.00504
	6.50000	6.50003		6.50000	6.50005
9/9/2013	6.00000	6.00003	9/26/2013	6.00000	6.00004
	6.00500	6.00503		6.00500	6.00501
	6.50000	6.50003		6.50000	6.50003
9/18/2013	6.00000	6.00006	9/27/2013	6.00000	6.00004
	6.00500	6.00505		6.00500	6.00501
	6.50000	6.50006		6.50000	6.50003
9/19/2013	6.00000	6.00002	10/22/2013	6.00000	6.00003
	6.00500	6.00503		6.00500	6.00502
	6.50000	6.50006		6.50000	6.50002
9/20/2013	6.00000	6.00000	11/12/2013	6.00000	6.00003
	6.00500	6.00500		6.00500	6.00501
	6.50000	6.50002		6.50000	6.50001

5.3 Analytical Results

The residual mass of every sample generated in this task was measured by gravimetric analysis. The raw results, in grams, are presented in Table 28, the Task 5 Raw Sample Results table.

Table 28. Task 5 Raw Sample Results

Pan #	Constant Weight (g)	Additional Weight (g)	Average Weight (g)	Tare Weight (g)	Net Constant Weight (g)	Net Average Weight (g)
67	6.35735	6.35738	6.35737	6.35659	0.00076	0.00077
83	6.38177	6.38175	6.38176	6.38187	ND	ND
85	6.32505	6.32507	6.32506	6.32445	0.00060	0.00061
86	6.36159	6.36157	6.36158	6.36115	0.00044	0.00043
87	6.35331	6.35334	6.35333	6.35341	ND	ND
88	6.33890	6.33888	6.33889	6.33823	0.00067	0.00066
89	6.37749	6.37752	6.37751	6.37720	0.00029	0.00030
90	6.34179	6.34180	6.34180	6.34133	0.00046	0.00047
91	6.35237	6.35239	6.35238	6.35199	0.00038	0.00039
92	6.35324	6.35324	6.35324	6.35259	0.00065	0.00065
93	6.35805	6.35801	6.35803	6.35759	0.00046	0.00044

*Pan 97 required many more weighings then other samples to achieve constant weight.

**Pan 98 sample weights are approximate due to failure of sample pan to stabilize during gravimetric analysis.

Table 28. Task 5 Raw Sample Results (Continued)

Pan #	Constant Weight (g)	Additional Weight (g)	Average Weight (g)	Tare Weight (g)	Net Constant Weight (g)	Net Average Weight (g)
94	6.37651	6.37648	6.37650	6.37605	0.00046	0.00045
95	6.31717	6.31713	6.31715	6.31781	ND	ND
96	6.33861	6.33857	6.33859	6.33832	0.00029	0.00027
97*	6.26945	6.26940	6.26943	6.26238	0.00707	0.00704
98**	6.47676	6.47517	6.47597	6.27459	0.20217	0.20138
99	6.24800	6.24795	6.24798	6.24608	0.00192	0.00190
100	6.30181	6.30180	6.30181	6.30092	0.00089	0.00089
101	6.25243	6.25244	6.25244	6.25216	0.00027	0.00028
102	6.22581	6.22583	6.22582	6.22526	0.00055	0.00056
103	6.24508	6.24505	6.24507	6.24446	0.00062	0.00061
104	6.24482	6.24482	6.24482	6.24427	0.00055	0.00055
105	6.25273	6.25277	6.25275	6.25198	0.00075	0.00077
106	6.26031	6.26027	6.26029	6.25950	0.00081	0.00079
107	6.25683	6.25680	6.25682	6.25609	0.00074	0.00072
108	6.25449	6.25453	6.25451	6.25388	0.00061	0.00063
109	6.23637	6.23634	6.23636	6.23566	0.00071	0.00069
110	6.23379	6.23377	6.23378	6.23353	0.00026	0.00025
111	6.25077	6.25077	6.25077	6.25003	0.00074	0.00074
112	6.28599	6.28595	6.28597	6.28529	0.00070	0.00068
113	6.23101	6.23104	6.23103	6.23041	0.00060	0.00062
114	6.24313	6.24308	6.24311	6.24304	0.00009	0.00007
115	6.26728	6.26726	6.26727	6.26715	0.00013	0.00012
116	6.26699	6.26698	6.26699	6.26691	0.00008	0.00007
117	6.23702	6.23701	6.23702	6.23697	0.00005	0.00004
118	6.28030	6.28028	6.28029	6.28022	0.00008	0.00007
119	6.24469	6.24467	6.24468	6.24462	0.00007	0.00006
120	6.24443	6.24444	6.24444	6.24413	0.00030	0.00031
121	6.23017	6.23013	6.23015	6.22969	0.00048	0.00046
122	6.29788	6.29788	6.29788	6.29770	0.00018	0.00018
123	6.28017	6.28016	6.28017	6.27998	0.00019	0.00019
124	6.24309	6.24304	6.24307	6.24286	0.00023	0.00020
125	6.25991	6.25993	6.25992	6.25970	0.00021	0.00022
126	6.25186	6.25184	6.25185	6.25170	0.00016	0.00015
127	6.26890	6.26892	6.26891	6.26873	0.00017	0.00018
128	6.24488	6.24488	6.24488	6.24394	0.00094	0.00094

*Pan 97 required many more weighings then other samples to achieve constant weight.

**Pan 98 sample weights are approximate due to failure of sample pan to stabilize during gravimetric analysis.

All negative results are reported as non-detects. On sample evaporation, pans 97 and 98 were observed to have a large amount of residue compared to all other samples. Pan 97 required many more measurements then other samples to achieve constant weight. Pan 98 did not stabilize during gravimetric analysis and the sample weights associated with it should be considered approximate. Pans 97 and 98 were used for the gravimetric analysis of C1 samples.

Results for the full train samples, in mg, are in Table 29, the Full Train Sample Results table. The train component sample results, in milligrams, are presented in Table 30, the Component Sample Results table.

Table 29. Full Train Sample Results

Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)
A Aq	67	0.76	0.77
A Org	83	ND	ND
B Aq	85	0.60	0.61
B Org	87	ND	ND
C1 Aq*	98	202.17	201.38
C1 Org**	97	7.07	7.04
C2 Aq	88	0.67	0.66
C2 Org	99	1.92	1.90
C3 Aq	89	0.29	0.30
C3 Org	100	0.89	0.89

*C1 Aq sample weights are approximate due to failure of sample pan to stabilize during gravimetric analysis.

**C1 Org required many more weighings then other samples to achieve constant weight.

Table 30. Component Sample Results

Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)
PE1 Aq	91	0.38	0.39
PE1 Org	102	0.55	0.56
PE2 Aq	92	0.65	0.65
PE2 Org	96	0.29	0.27
PE3 Aq	93	0.46	0.44
PE3 Org	101	0.27	0.28
PE4 Aq	86	0.44	0.43
PE4 Org	110	0.26	0.25
CND1 Aq	90	0.46	0.46
CND1 Org	114	0.09	0.07
CND2 Aq	94	0.46	0.45
CND2 Org	115	0.13	0.12
CND3 Aq	103	0.62	0.61
CND3 Org	116	0.08	0.07

Table 30. Component Sample Results (Continued)

Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)
CND4 Aq	104	0.55	0.55
CND4 Org	117	0.05	0.04
IMP1 Aq	105	0.75	0.77
IMP1 Org	120	0.30	0.31
IMP2 Aq	106	0.81	0.79
IMP2 Org	121	0.48	0.46
IMP3 Aq	111	0.74	0.74
IMP3 Org	124	0.23	0.20
IMP4 Aq	112	0.70	0.68
IMP4 Org	125	0.21	0.22
FH1 Aq	107	0.74	0.72
FH1 Org	118	0.08	0.07
FH2 Aq	108	0.61	0.63
FH2 Org	119	0.07	0.06
FH3 Aq	109	0.71	0.69
FH3 Org	122	0.18	0.18
FH4 Aq	113	0.60	0.62
FH4 Org	123	0.19	0.19

Statistical analyses and replicate measurement comparisons of the data sets can be seen in Tables 31 through 36.

Table 31. Full Train Statistical Analysis

	Aq Net Const Wt (mg)	Aq Net Avg Wt (mg)	Org Net Const Wt (mg)	Org Net Avg Wt (mg)	Total Net Const Wt (mg)	Total Net Avg Wt (mg)
A	0.76	0.77	0.00	0.00	0.76	0.77
B	0.60	0.61	0.00	0.00	0.60	0.61
C1	202.17	201.38	7.07	7.04	209.24	208.42
C2	0.67	0.66	1.92	1.90	2.59	2.55
C3	0.29	0.30	0.89	0.89	1.18	1.19
Average	40.90	40.74	1.98	1.97	42.87	42.71
Standard Deviation	90.15	89.80	2.96	2.95	93.00	92.64
Detection Limit	270.46	269.39	8.87	8.84	279.01	277.91
% RSD	220.44	220.38	149.57	149.89	216.93	216.90
UPL (n=5)	121.53	121.06	4.62	4.60	126.06	125.57

Table 32. Full Train C Replicate Statistical Analysis

	Aq Net Const Wt (mg)	Aq Net Avg Wt (mg)	Org Net Const Wt (mg)	Org Net Avg Wt (mg)	Total Net Const Wt (mg)	Total Net Avg Wt (mg)
C1	202.17	201.38	7.07	7.04	209.24	208.42
C2	0.67	0.66	1.92	1.90	2.59	2.55
C3	0.29	0.30	0.89	0.89	1.18	1.19
Average	67.71	67.45	3.29	3.28	71.00	70.72
Standard Deviation	116.45	115.99	3.31	3.30	119.72	119.25
Detection Limit	349.34	347.96	9.93	9.91	359.16	357.76
% RSD	171.98	171.97	100.54	100.88	168.61	168.62
UPL (n=3)	202.17	201.38	7.12	7.09	209.24	208.42

Table 33. Probe Extension Replicate Statistical Analysis

	Aq Net Const Wt (mg)	Aq Net Avg Wt (mg)	Org Net Const Wt (mg)	Org Net Avg Wt (mg)	Total Net Const Wt (mg)	Total Net Avg Wt (mg)
PE1	0.38	0.39	0.55	0.56	0.93	0.95
PE2	0.65	0.65	0.29	0.27	0.94	0.92
PE3	0.46	0.44	0.27	0.28	0.73	0.72
PE4	0.44	0.43	0.26	0.25	0.70	0.68
Average	0.48	0.48	0.34	0.34	0.83	0.82
Standard Deviation	0.12	0.12	0.14	0.15	0.13	0.14
Detection Limit	0.35	0.35	0.42	0.44	0.38	0.42
% RSD	24.19	24.51	40.55	43.66	15.48	16.96
UPL (n=4)	0.60	0.59	0.48	0.49	0.95	0.95

Table 34. Condenser Replicate Statistical Analysis

	Aq Net Const Wt (mg)	Aq Net Avg Wt (mg)	Org Net Const Wt (mg)	Org Net Avg Wt (mg)	Total Net Const Wt (mg)	Total Net Avg Wt (mg)
CND1	0.46	0.46	0.09	0.07	0.55	0.53
CND2	0.46	0.45	0.13	0.12	0.59	0.56
CND3	0.62	0.61	0.08	0.07	0.70	0.68
CND4	0.55	0.55	0.05	0.04	0.60	0.59
Average	0.52	0.52	0.09	0.08	0.61	0.59
Standard Deviation	0.08	0.07	0.03	0.03	0.06	0.06
Detection Limit	0.23	0.22	0.10	0.10	0.19	0.19
% RSD	14.86	14.46	37.76	41.60	10.45	10.82
UPL (n=4)	0.60	0.59	0.12	0.11	0.67	0.66

Table 35. Impinger Replicate Statistical Analysis

	Aq Net Const Wt (mg)	Aq Net Avg Wt (mg)	Org Net Const Wt (mg)	Org Net Avg Wt (mg)	Total Net Const Wt (mg)	Total Net Avg Wt (mg)
IMP1	0.75	0.77	0.30	0.31	1.05	1.08
IMP2	0.81	0.79	0.48	0.46	1.29	1.25
IMP3	0.74	0.74	0.23	0.20	0.97	0.94
IMP4	0.70	0.68	0.21	0.22	0.91	0.90
Average	0.75	0.74	0.31	0.30	1.06	1.04
Standard Deviation	0.05	0.05	0.12	0.12	0.17	0.16
Detection Limit	0.14	0.14	0.37	0.35	0.50	0.47
% RSD	6.06	6.44	40.29	39.31	15.81	15.06
UPL (n=4)	0.80	0.79	0.43	0.41	1.22	1.20

Table 36. Filter Housing Replicate Statistical Analysis

	Aq Net Const Wt (mg)	Aq Net Avg Wt (mg)	Org Net Const Wt (mg)	Org Net Avg Wt (mg)	Total Net Const Wt (mg)	Total Net Avg Wt (mg)
FH1	0.74	0.72	0.08	0.07	0.82	0.80
FH2	0.61	0.63	0.07	0.06	0.68	0.69
FH3	0.71	0.69	0.18	0.18	0.89	0.87
FH4	0.60	0.62	0.19	0.19	0.79	0.80
Average	0.66	0.67	0.13	0.12	0.80	0.79
Standard Deviation	0.07	0.05	0.06	0.07	0.09	0.08
Detection Limit	0.21	0.16	0.19	0.20	0.26	0.23
% RSD	10.60	7.86	49.05	54.94	10.99	9.63
UPL (n=4)	0.74	0.72	0.19	0.19	0.88	0.87

The target of 95% of sample results falling at or below the calculated UPL value was not achieved for all data sets. This can be attributed to the fact that the number of samples in the data sets is so low that one result above the UPL will cause the percentage of samples at or below the UPL to be less than 95%. For the results of the dust pan and the reagent blanks for the hexane, acetone, and water used in this task see Table 37, the Reagent Blank and Dust Pan Results table. The dust pan residual mass was a non-detect, which shows that samples were not contaminated by dust during sample pan evaporation. Reagent blank results show very low residual masses which are less than the method criteria for the hexane and acetone used and a residual mass of less than 1.0 mg for the water.

Table 37. Task 5 Reagent Blank and Dust Pan Results

Sample ID	Pan #	Net Constant Weight (mg)	Net Average Weight (mg)
DP	95	ND	ND
RBH	126	0.16	0.15
RBA	127	0.17	0.18
RBW	128	0.94	0.94

5.4 Conclusions

The sample weights for both the inorganic (Aq) and organic (Org) fractions of Trains A and B were very close to each other. The total results for both Train A and Train B (the Aq and Org fractions combined for each) are well below the FTRB limit of 2.0 mg. On Sample evaporation of the first recovery of Train C a very large amount of residue was observed in pan 98 which was used for the C1 Aq sample, and in pan 97 which was used for the C1 Org, but not as much as pan 98. Not only does the total C1 result (C1 Aq plus C1 Org) exceed the FTRB limit by over 200 mg, both fractions are above the FTRB limit independently. Figure 16, the Full Train Inorganic and Organic Results graph shows a comparison of the inorganic and organic results of each full sampling train using the net average weights. The total train blank results for each full Method 202 sample, using the net average weights, are shown in Figure 17, the Full Train Total Combined Inorganic and Organic Results graph. The high results for the C1 samples are immediately apparent, as is the reduction of those results on successive recoveries.

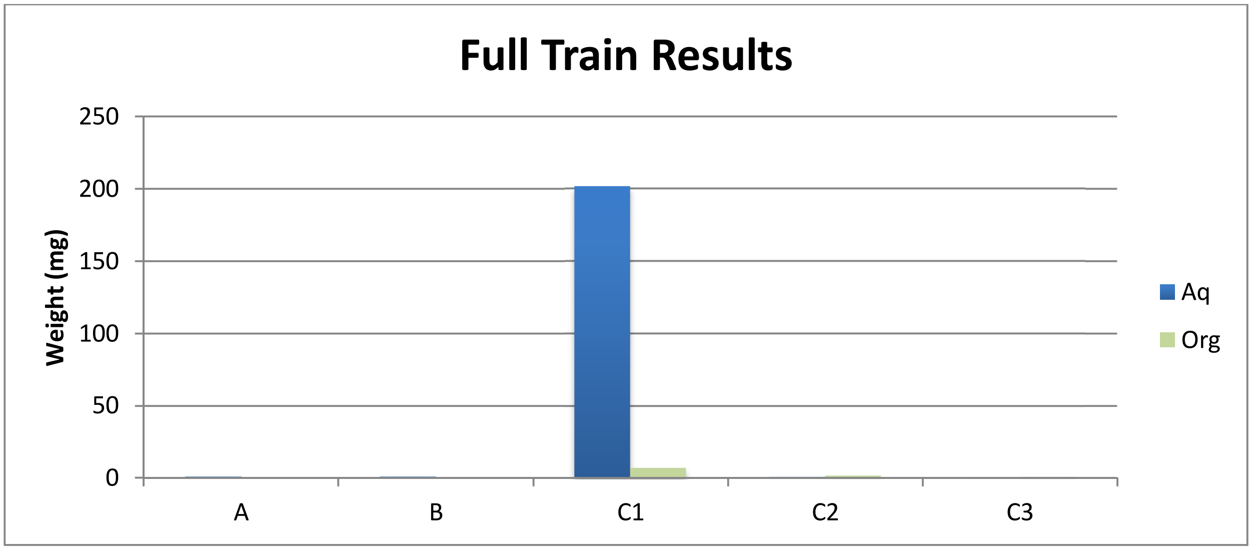

Figure 16. Full Train Inorganic and Organic Results (Net Average Weights)

Figure 17. Full Train Total Combined Inorganic and Organic Results (Net Average Weights)

The second recovery of Train C, samples C2 Aq and C2 Org, shows large reduction in the net sample weights for both fractions compared to the first recovery of Train C (C1 samples). The C2 Aq result is very close to the results for the inorganic fractions of Trains A and B. The C2 Org result is just under 2.0 mg, which is the field train recovery blank limit in Method 202. The total C2 result (C2 Aq plus C2 Org) fails the FTRB limit, but only by approximately 0.5 mg. The third recovery of Train C, samples C3 Aq and C3 Org, show an even further reduction. C3 Aq results are about half of the results for the inorganic fractions of Trains A, B, and C2. The C3 Org result is below 1.0 mg, which is still higher than A Org or B Org, but it is low enough that the total C3 result (C3 Org plus C3 Aq) is below the FTRB limit. The downward trend for the Train C Org results on successive recoveries can be seen in Figure 18, the Full Train C Organic Results comparison using the net average weights. The cause of the elevated Train C results is not known. It is possible that the probe extension was a source of contamination. The probe extensions were cleaned and rinsed prior to recovery but not baked because they were too long to fit into the oven at ERG's laboratory. This is a typical constraint due to the length of the probe extensions. The probe extensions for Trains A and B were prepared in the same manner as for Train C, but the results for Trains A and B did not show the same elevated concentrations. The large reduction from the C1 Aq result to the C2 Aq result shows that the inorganic portion of the Train C sample was readily recoverable. The total results for the recoveries of Train C show a significant drop in the amount of residual mass measured and that field train proof blank residual mass results above 200 mg can be brought down below the FTRB limit. The full Method 202 train total results in this task demonstrate that it is possible to achieve field train blank results that are well below the FTRB limit of 2.0 mg and in the case of Trains A and B are less than half of the limit.

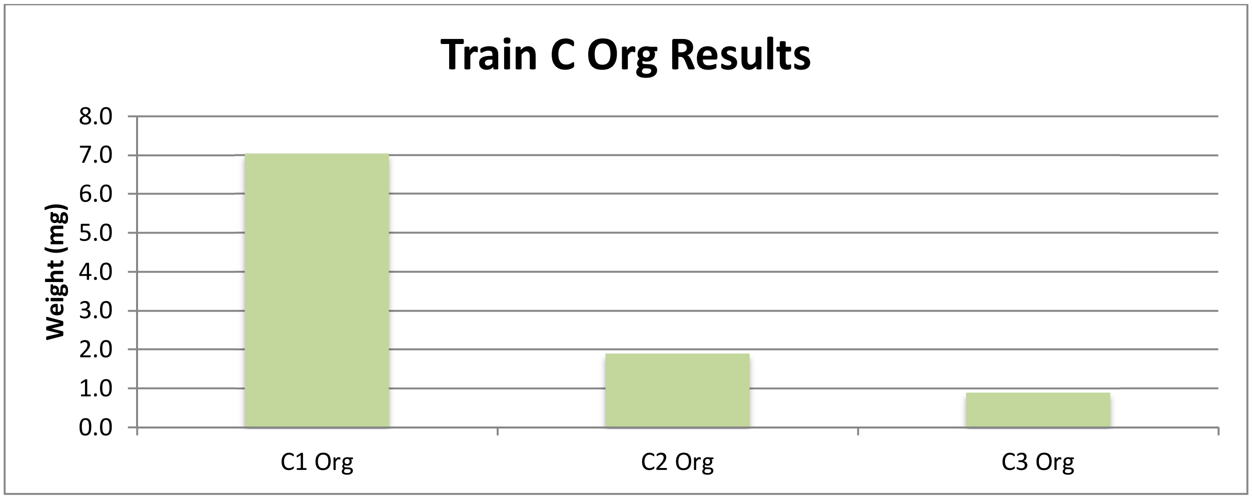

Figure 18. Full Train C Organic Results (Net Average Weights)

For the sampling train components a comparison of the inorganic and organic results of each sample recovered using net average weights can be seen in Figure 19, the Component Inorganic and Organic Results graph. The successive recoveries of all four components did not show a clear trend of decreasing results for the inorganic or organic fractions. This is not necessarily unexpected as the full train results for the set used in the component evaluation, Train A, were already very low. Figure 20, the Component Average Inorganic and Organic Results graph shows the average of the replicate measurements for the inorganic and organic results of each component, using the net average weights. When the total combined inorganic and organic results of the four successive recoveries of each component are averaged, as seen in Figure 21, the Component Average Total Combined Inorganic and Organic Results graph, using net average weights, it becomes clear that the component with the highest results is the impingers and connecting glassware.

Comparing the calculated %RSDs for the total (Aq plus Org) results of the component samples using the net average weights shows the probe extensions to have the highest value at 16.78% and the impingers close behind at 14.96%. The %RSD is a measure of the variability and the component that has the highest %RSD may indicate the possibility of a greater contribution to the total residual mass of a full train. Figure 22 displays the %RSD comparison for components using the net constant weights and the net average weights. The calculated UPLs for the replicate measurements of the impinger samples had the highest of all components. The impingers having both the highest UPL result and the highest total averaged result indicate that the impingers have the highest potential for contribution to the total full train residual mass concentration. A comparison of the calculated UPLs of the components, using the net average weights, can be seen in Figure 23. The two impingers with connecting glassware represent, depending on the length of the probe extension, the largest sample surface area of all component sections evaluated.

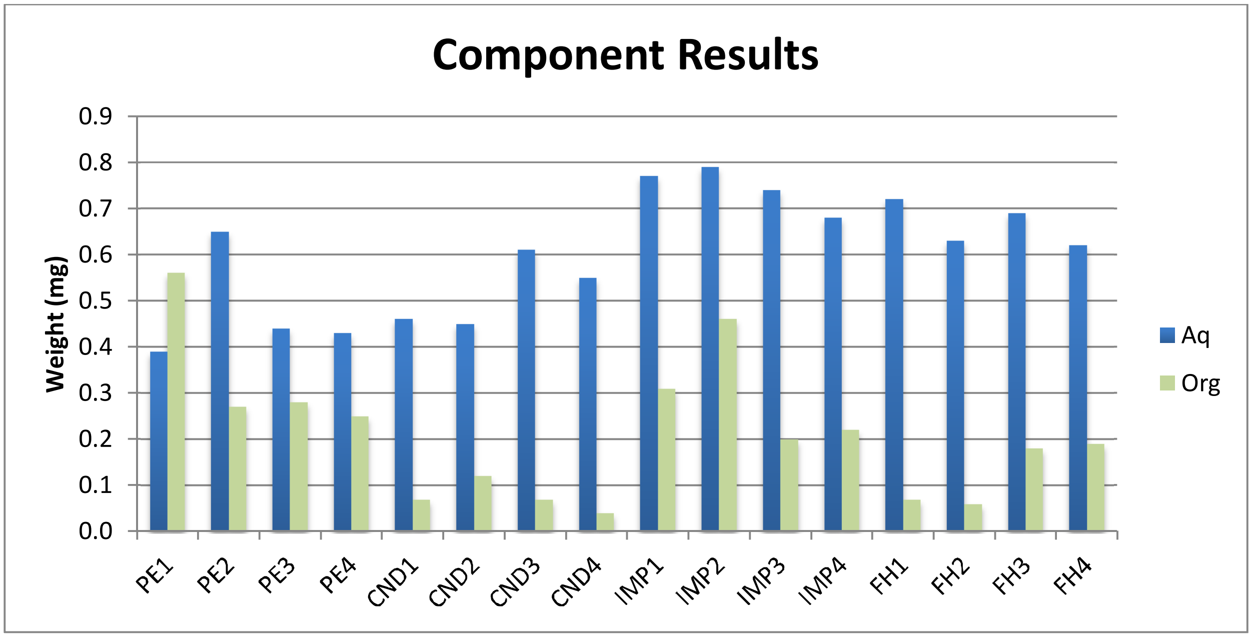

Figure 19. Component Inorganic and Organic Results (Net Average Weights)

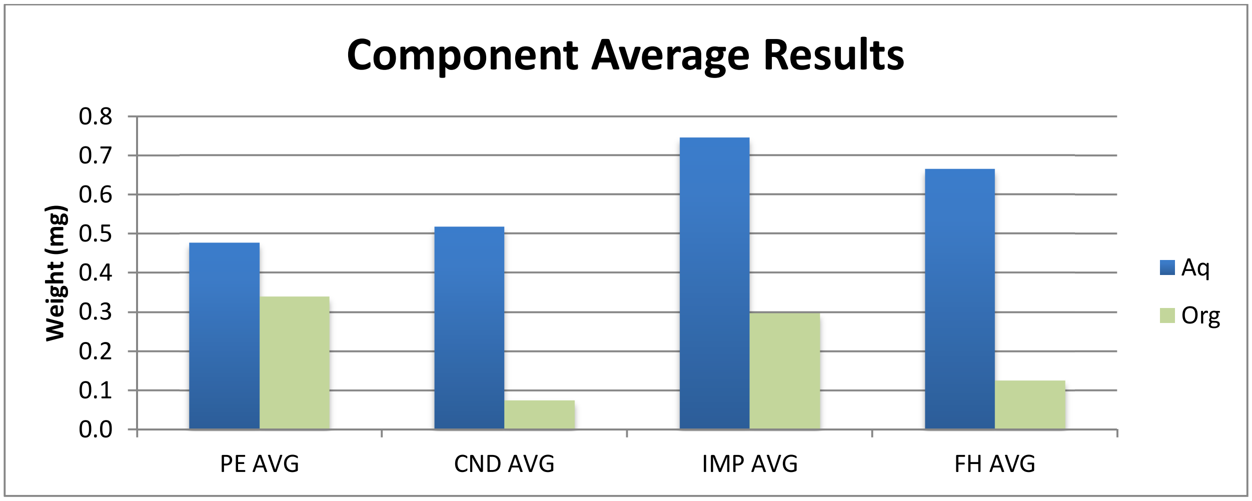

Figure 20. Component Average Inorganic and Organic Results (Net Average Weights)

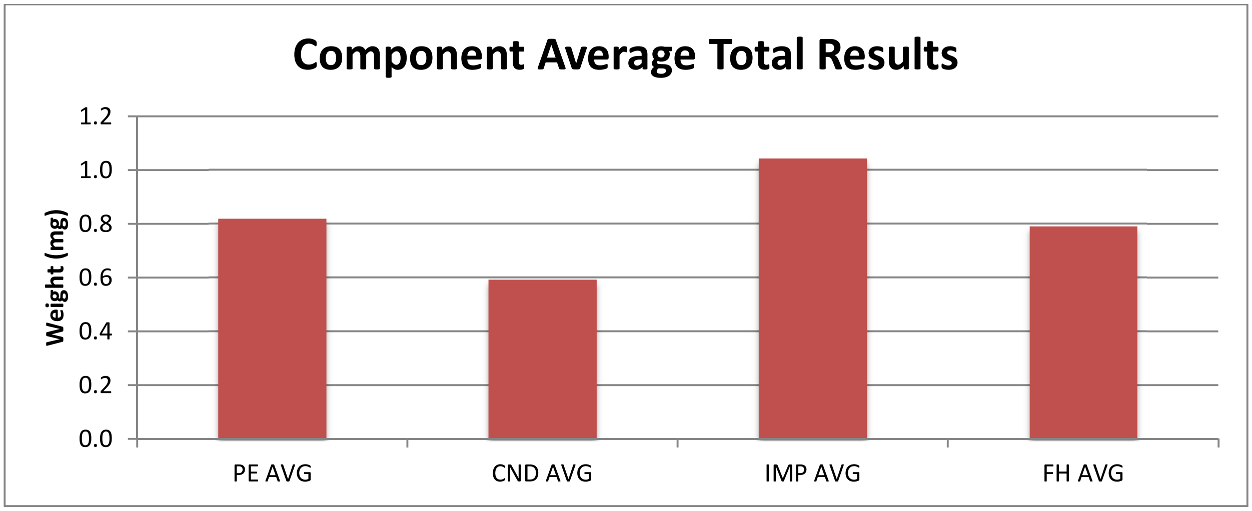

Figure 21. Component Average Total Results (Net Average Weights)

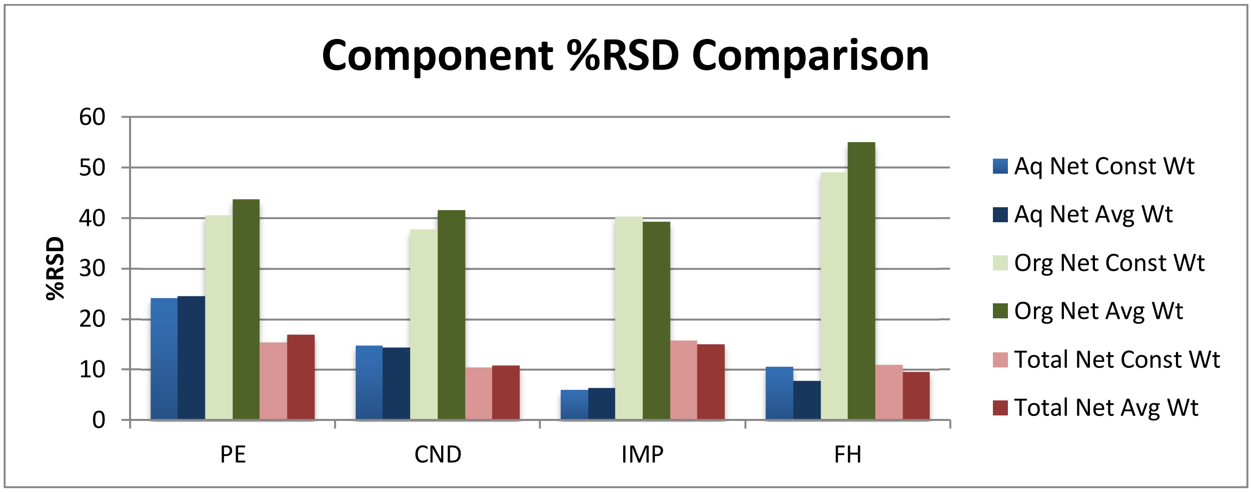

Figure 22. Component %RSD Comparison

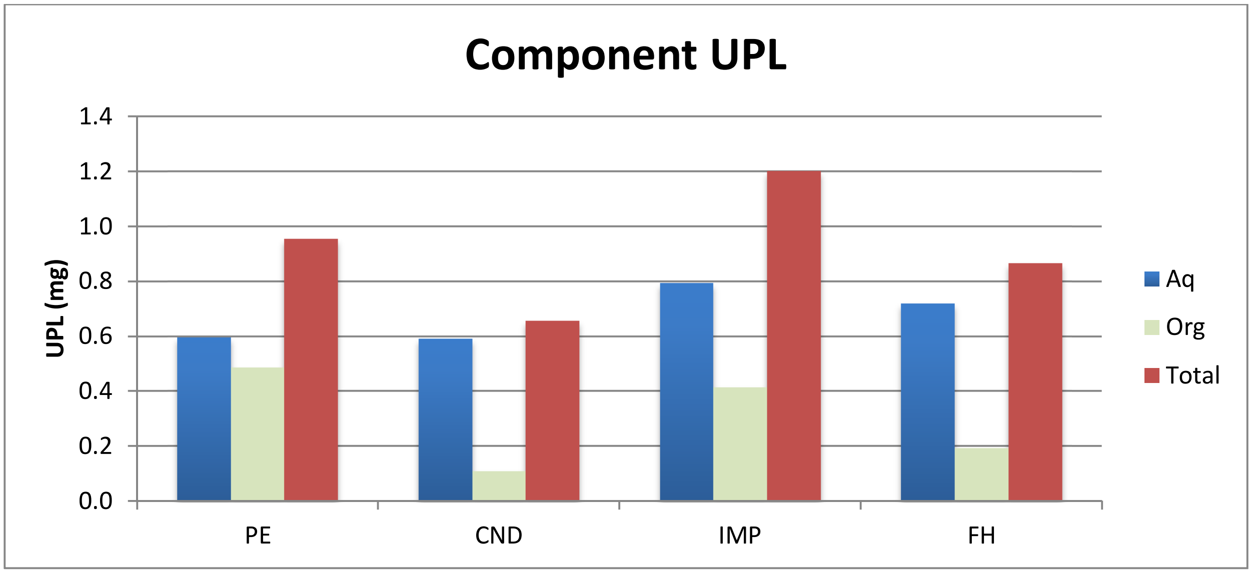

Figure 23. Component UPL Comparison (Net Average Weights)

Based on the analytical results found in this task several preparation procedures employed by Air-Tech Environmental were observed that were recognized to help limit contamination and residual mass contributions from the materials used in the blank recoveries. The cleaning procedure for the sampling trains which included washing with soap and water, scrubbing with a brush, rinsing with DI water, acetone, and hexane, and baking at 300 ºC for 6 hours (when possible). The recovery area, which was a mobile lab at Air-Tech Environmental for this task, was cleaned and the benchtop was covered with foil before performing recoveries. An adequate, but not excessive, volume of reagents was used for recoveries. This is impacts the residual mass contribution to blanks or samples because the larger the volume used the more residual mass is being contributed from that reagent. Also, the consistent application of procedures for all recoveries is good practice in maintaining data quality. All of these procedures are recommended as best practices for Method 202.

6.0 CONCLUSIONS AND BEST PRACTICES

The survey and laboratory evaluations conducted in this work assignment were successful in meeting their objectives. Best practices for the materials and procedures used in Methods 201A and 202 were determined and representative data of the residual mass concentrations of filters, reagents, and sampling trains were achieved. The data produced confirmed findings from the survey and the laboratory evaluations revealed best practices themselves. The full list of best practice findings from the survey responses in Task 2 are in Table 2. In particular, Method 202 filter selection, reagent selection, sampling train cleaning, and sample recovery location considerations are critical in limiting residual mass contributions. These factors were confirmed in the laboratory evaluations. The CPM filters used should be 100% Teflon® filters that do not contain any substrate or support ring other than Teflon® for Method 202. CPM filter blanks that demonstrate that filters to be used in source testing meet the method limit should be performed. Likewise, reagent blanks that demonstrate the purity of reagents proposed for use meets the method limit should be performed. The cleaning procedure for the sampling trains which should include washing with soap and water, scrubbing with a brush, rinsing with DI water, acetone, and hexane, and baking at 300 °C for 6 hours (when possible). Recovery areas should be cleaned, free of dust, and access should be limited. Also, not using excessive volumes of reagent and consistent application of the procedures should be employed. All of these items are recommended as best practices for conducting source testing using Methods 201A and 202 based on the survey responses and the analytical data generated in this work assignment.

7.0 REFERENCES

U.S. Environmental Protection Agency Work Assignment 3-07 Contract EP-D-11-006

U.S. Environmental Protection Agency, Washington, D.C., Method 5 – Determination of Particulate Matter Emissions from Stationary Sources, 40 CFR Part 60 Appendix A, July 1, 1989.

U.S. Environmental Protection Agency, Washington, D.C., Method 202 – Dry Impinger Method for Determining Condensable Particulate Emissions from Stationary Sources, 40 CFR Part 51 Appendix M, December 8, 2010.

8.0 APPENDIX

ERG Standard Operating Procedure for Gravimetric Determination for Particulate Emissions Measurements (ERG-MOR-002)

ERG
EASTERN RESEARCH GROUP, INC.

ENGINEERING AND SCIENCE DIVISION

TITLE: Standard Operating Procedure for Gravimetric Determination for Particulate Emissions Measurements	EFFECTIVE DATE: MAY 15 2013
REFERENCES: NA	
SATELLITE FILES: ICP-MS, Prep	
REASON FOR REVISION: Added other method applicability	
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1.0 PURPOSE

The purpose of this document is to present a general procedure for the gravimetric analysis of particulate matter. This procedure is not written for use with a specific analytical balance. For instruction for a specific balance, refer to the operations manual for that balance.

2.0 SCOPE AND APPLICABILITY

2.1 Scope

This SOP covers standard preparations for determining gravimetric weight using ovens, desiccators, and analytical balances.

2.2 Applicability

This SOP is applicable to all gravimetric procedures performed by ERG personnel requiring particulate emissions measurements.

3.0 INTERFERENCES

Impurities in the reagents may interfere with results. Refer to the specific method for more information.

4.0 SAFETY

Normal laboratory safety procedures will apply.

5.0 MATERIALS AND APPARATUS

5.1 Desiccator.

5.2 Analytical Balance. Used to measure within 0.0002 g for all weight determinations (0.00001 g for Particle Size Distribution [PSD] determinations).

5.3 Beakers. 150 mL or 250 mL. (150 mL beakers stabilize faster on an analytical balance.

5.4 Hygrometers. Used to measure the relative humidity of the laboratory equipment room.

5.5 Temperature Gauge. Used to measure the temperature of the laboratory equipment room.

5.6 Wash Bottles. Teflon® bottles are recommended. Polyethylene wash bottles may be used at the option of the tester. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

5.7 Petri Dishes. For filters, glass or polyethylene, unless otherwise specified by the Project Manager (PM).

5.8 Oven. Temperature of 105°C needs to be achieved.

5.9 Weighing Pans. Aluminum dishes for weighing samples.

5.10 Class "S" Weights. Used for checking the calibration of the balance.

6.0 REAGENTS

6.1 Acetone. Reagent grade. <0.001% residue, in glass bottles, is required. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes suppliers transfer acetone to glass bottles from metal containers; thus, acetone blanks shall be run prior to field use and only acetone with low blank values (<0.001%) shall be used. The EPA specifies that in no case shall a blank value of greater than 0.001% of the weight of acetone used be subtracted from the sample weight (e.g., A 100 mL blank will need to weigh less than 0.001 gram).

6.2 Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Task Leader or the PM.

7.0 DEFINITIONS

7.1 Constant Weight. The constant weight is considered as the agreement between successive readings, with no less than six hours between readings, i.e., a 0.0005 g change from previous weighing for solids and ambient air filters and a 0.00005 g change for stationary source PSD (47 mm) filters.

7.2 Final Weight. The final weight is considered the last reading taken after a constant weight is established. **Do not use the average constant weight.**

8.0 PROCEDURES

Record balance room relative humidity and temperature each time gravimetric measurements are made. Check the level of the balance with the bubble meter located on the balance.

The balance calibration is checked using class "S" calibration weights. Check calibration of the balances with weights that bracket the sample weight (e.g., a 1.5 gram sample should have a calibration range of 0.5, 1.0, and 2.0 gram weights). Record the calibration check in the appropriate logbook or notebook. When performing the calibration check, a deviation of more than ±0.0002 grams at any weight level will require that the zero be checked or balance maintenance. Acceptable deviation for a 5 place balance is ≤0.00005g.

For stationary source samples, all fractions (i.e., filters, thimbles, etc.) are initially tare weighed. After sampling, acetone fractions are evaporated and cooled. All fractions used in the method are then reweighed to a constant weight to determine the final particulate matter.

Before filters or thimbles are used in sampling methods, they should be visually inspected against light for irregularities and flaws or pinhole leaks. Label the shipping containers (glass or plastic petri dishes, or the outside of the thimble). Keep the filters and thimbles in these containers at all times except during sampling and weighing.

Desiccate the filters, thimbles, beakers, or pans at 20 ± 5.6°C (68 ± 10°F) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 hours to a constant weight, i.e., 0.0005 g change (0.00005 g change for PSD determination) from previous weighing. Record results to the nearest 0.0001 g (0.00001 g for PSD determination.) During each weighing, the filter, thimble, beaker or pan must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50 percent.

Alternatively, the filters, thimbles, or beakers may be oven dried at 105°C (220°F) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Task Leader and/or PM.

The oven may not be used with any other type of sample besides particulate determination (i.e., silica gel, chemical compounds, etc., are not allowed in this oven).

8.1 EPA Method 5 Analysis

Record the data required on a sheet for initial and final weights as shown in Figure 1 and 2. A final weight should be determined on the filter and an acetone probe rinse for each sampling run.

At the option of the tester, the contents of the acetone container may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature must be below the boiling point of the solvent. Also, to prevent “bumping”, the evaporation process must be closely supervised, and the contents of the beaker must be swirled occasionally to maintain an even temperature.

Use extreme care, as acetone is highly flammable and has a low flash point.

8.2 EPA Method 17 Analysis

Record the data required on a sheet for initial and final weights as shown in Figures 1 and 3. A final weight should be determined on the filter and the acetone probe rinse for each sampling run. The tester may combine acetone and the filter by transferring the contents of the acetone into a tared 150 mL beaker and evaporating to dryness. After the acetone is dry, the respective filter can be placed into the beaker, and the combined sample can be desiccated and weighed to a constant weight. The weight of the particulate would be determined by subtracting the sum of the initial filter and beaker weights from the final weight.

8.3 Particle Size Distribution Sampling Method Analysis

Record the data required on a sheet for initial and final weights as shown in Figures 1 and 2. A final weight should be determined on each filter in the filter set and on the acetone probe rinse for each sampling run. Acetone probe rinses can be evaporated to dryness in pre-weighed 100m to aluminum baking tins.

8.4 Air Pollution Sampling Method Analysis (High Volume (HiVol) Ambient Air Filters)

To weigh the 8 x 10" filters, the balance needs to be equipped with a large area weighing chamber. The Satorius LA 120S in the ERG laboratory's Balance Room is equipped with this device.

Before beginning calibration, insert the filter weighing stand. Check the leveling of the balance. If necessary, adjust with leveling feet of the chamber.

Calibration is performed as explained in Section 8.0.

Ambient filters are equilibrated for 24 hours under balance room conditions prior to weighing. To weigh samples, check that the balance and filter support are tarred to zero. Open the chamber door and slide in the filter paper onto the weighing support, placing the filter behind the front support rod so that is rests on the three back support rods. Close the chamber door. Weigh the filter paper.

Record the data required on a sheet for initial and final weights as shown in Figures 1 and 4. Handle each sample as explained in 40 CFR 50, Appendix J, Section 9.16 - 9.17.

8.5 EPA Method 202 Analysis

Samples generated from the recovery of Method 202 sampling trains and filters using water, acetone, and hexane will be evaporated to dryness in tared aluminum weighing pans. Reagent blanks will likewise be evaporated to dryness in weighing pans. The pans will be desiccated for 24 hours and then weighed to constant weight.

9.0 QUALITY CONTROL

Data sheets should serve as a checklist for proper operating procedures. All entries should be completed in full. Any abnormalities encountered during collection, recovery, or analysis of the sample should be documented to assist in the interpretation of the data.

Each time a balance calibration is checked, the actual and determined weight must be recorded in a Calibration Logbook located beside each balance. Each entry should be initialed and dated.

Perform balance zero check to ensure the balance returns to zero between samples. Temperatures above 29°C (85°F) and relative humidities above 50% can affect the balance operation and accuracy. Therefore, the temperature and humidity should be included in the Analysis Logbook.

Balances are serviced and calibrated with NIST traceable weights annually by a service representative. A Certificate of Weight Verification is kept on file by the QA Coordinator.

10.0 DOCUMENTATION

Personnel will follow required documentation procedures and maintain complete and accurate records. All data should be recorded in a notebook.

11.0 REFERENCES

U.S. Environmental Protection Agency, Washington, D.C., Method 5 - Determination of Particulate Emissions from Stationary Sources, 40 CFR Ch. 1, July 1, 1989.

U.S. Environmental Protection Agency, Washington, D.C., Method 17 - Determination of Particulate Emissions from Stationary Sources (In-Stack Filtration Method), 40 CFR Ch. 1, July 1, 1989.

U.S. Environmental Protection Agency, Washington, D.C., Method 202 – Dry Impinger Method for Determining Condensable Particulate Emissions from Stationary Sources, 40 CFR Ch. 1, July 1, 1989.

Balfour, W.D., Procedures for the Determination of Particle Size Distributions using an In-Stack Cascade Impactor, Radian Corporation, Austin, Texas, August 1980.

Andersen 2000 Inc., Operating Manual for Andersen 2000 Inc., Mark II and Mark III, Particle Sizing Stack Samplers, Andersen 2000 Inc, Atlanta, Georgia, January, 1976.

EPA Methods, Code of Federal Regulations, Title 40, Part 50, Appendix J. October 16, 1992.

Initial Tare Weight Log						
Client			Technician			Page
Charge #			Date			
Filter Beaker #	**Weight 1 Date**	**Weight 2 Date**	**Weight 3 Date**	**Weight 4 Date**	**Final Weight (g)**	**Comments**

Figure 1. Initial Tare Weight Log

Final Weight Log								
Client				Technician				Page
Plant				Date				of
Sample Type								
Run #	**Sample ID**	**Volume (mL)**	**Weight 1 Date**	**Weight 2 Date**	**Weight 3 Date**	**Weight 4 Date**	**Final Weight (g)**	**Comments**

Figure 2. Final Weight Log

Method 17—Analysis Data Sheet

		PAGE OF	
PROJECT:		DATE:	
SAMPLE TYPE:		TECHNICIAN:	

Run #	Sample ID#	Initial Filter & Beaker (F & B) Weight (g)		Total F & B Weight (g)	Final Weight (g)	Sample Weight (g)	Comments
		filter					
		beaker					
		filter					
		beaker					
		filter					
		beaker					
		filter					
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Figure 3. Method 17—Analysis Data Sheet

Total F & B Weight = Initial Filter Weight (g) + Initial Beaker Weight (g)

HiVol Filter Final Weight Log

Client		Technician		Page
Plant		Date		of
Sample Type				

Sample ID	**Tare Weight (g) Date**	**Sample Weight (g) Date**	**Net Weight (g) Date**	**Zero Check**	**Comments**

Figure 4. Air Pollution Sampling—Final Weight Log