



EPA Method 202

Best Practices Handbook

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EPA Method 202 Best Practices Handbook

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Forward

The EPA has developed this best practices handbook to assist stack testers in achieving the expected results when using EPA Method 202—Dry Impinger Method for Determining Condensable Particulate Emissions from Stationary Sources. Method 202¹ measures the concentration of condensable particulate matter (CPM) in stationary source emissions after filterable particulate matter (PM) has been removed from the sample gas. PM is divided into two fractions: PM with mean aerodynamic diameters less than or equal to 10 micrometers (μm) (PM_{10}) and PM with mean aerodynamic diameters less than or equal to 2.5 μm ($\text{PM}_{2.5}$).

What is CPM?

Condensable particulate matter is material that is a vapor at stack conditions, but that condenses and/or reacts upon cooling and dilution in the ambient air to form solid or liquid PM immediately after discharge from the stack, and is a component of primary PM. Primary PM is the sum of CPM and filterable PM. Filterable PM comprises particles that are directly emitted by a source as a solid or liquid (aerosol) at stack or release conditions and are captured on the filter of a stack test sampling train. All CPM is assumed to be in the $\text{PM}_{2.5}$ fraction.

When did the EPA begin requiring measurement of CPM?

After promulgating the PM_{10} National Ambient Air Quality Standards (NAAQS) in 1987, the EPA began recommending that, in certain circumstances, states consider including the condensable portion of PM_{10} emissions in the determination of total and fine PM emissions from major stationary sources. However, it was not until 2008 that the EPA codified a requirement to include CPM as part of the PM emissions measurement and control assessments for major stationary sources and major modifications. Specifically, in its 2008 regulation, known as the 2008 $\text{PM}_{2.5}$ New Source Review (NSR) Implementation Rule, the EPA revised the definition of “regulated NSR pollutant” for both Prevention of Significant Deterioration (PSD) and nonattainment NSR to require that CPM be considered in applicability determinations and in establishing emissions limits for “particulate matter emissions²,” “ PM_{10} emissions,” and “ $\text{PM}_{2.5}$ emissions.” See 73 FR 28321 (May 16, 2008). The definition was again revised in 2012 by removing the requirement that CPM be counted in the measurement and control of “particulate matter emissions,” but the CPM requirement was not removed from PM_{10} emissions or $\text{PM}_{2.5}$ emissions. See 77 FR 65107 (October 25, 2012). In the 2008 $\text{PM}_{2.5}$ NSR Implementation Rule, citing various concerns regarding the uncertainties associated with Method 202, the EPA announced that it would not require states to implement the requirement to account for CPM in establishing enforceable emissions limits for either PM_{10} or $\text{PM}_{2.5}$ in permits until the completion of a transition period that ended on January 1, 2011.

Since January 1, 2011, states have been required to account for CPM in establishing emissions limits for both $\text{PM}_{2.5}$ and PM_{10} in all applicable PSD and nonattainment NSR permits issued. The NSR regulations require that the PM measurement and control assessments for major stationary sources and major modifications include the condensable component for both $\text{PM}_{2.5}$ and PM_{10} emissions. Accordingly, CPM must be considered: (1) in the PSD program in areas that are classified attainment or unclassifiable for the 1997 annual secondary, 2008 24-

¹ Method 202 is contained in 40 CFR part 51, appendix M. See 75 FR 80118, December 21, 2010.

² Particulate matter emissions is a term that refers to the measurement of particles captured by a source test using EPA Method 5 source test. Particles in that size range includes $\text{PM}_{2.5}$ and PM_{10} as well as particles that have an aerodynamic diameter greater than PM_{10} . Such larger particles are not considered in the measurement of the indicators for the PM_{10} and $\text{PM}_{2.5}$ NAAQS.

hour primary or secondary or 2012 annual primary PM_{2.5} NAAQS or the PM₁₀ NAAQS, and (2) in nonattainment NSR in areas that are nonattainment for any of the PM_{2.5} or PM₁₀ NAAQS.

The EPA defines “PM₁₀ emissions” in 40 CFR 51.100(rr) as finely divided solid or liquid material less than or at that size fraction, “...as measured by an applicable reference method, or an equivalent or alternative method specified in this chapter or by a test method specified in an approved State implementation plan.” This definition applies to the use of the terms “PM₁₀ emissions” and “PM_{2.5} emissions” (which should be considered a subset of PM₁₀) in any regulations developed pursuant to Part 51, including the regulations for NSR (40 CFR 51.165) and PSD (40 CFR 51.166).

What method is used to measure CPM?

Although sources are required to count CPM in measurements of emissions of PM₁₀ and PM_{2.5}, the PSD and nonattainment NSR regulations do not specify the test method to be used for quantifying CPM emissions. The EPA has generally expected that sources and testing contractors measure CPM using Method 202 as promulgated in 1991 (56 FR 65433, December 17, 1991) and revised in 2010 (75 FR 80118, December 21, 2010). Alternative methods, such as Method 201A for ambient temperature sources and other methods approved by the appropriate regulatory authority, may be necessary to accommodate site-specific issues needed to measure CPM. Method 202 is the EPA’s reference test method for quantifying the CPM fraction of primary PM. This method applies to stationary sources that must meet new applicable CPM requirements established through federal or state permits or rules, such as New Source Performance Standards (NSPS) and NSR, which specify the use of Method 201A (Determination of PM₁₀ and PM_{2.5} Emissions from Stationary Sources (Constant Sampling Rate Procedure)) and/or Method 202 in compliance demonstrations. Method 202 also applies to stationary sources that are subject to applicable requirements to control or measure PM₁₀, or PM_{2.5}, and where Method 202 is incorporated as a component of the applicable test method.

How has the EPA refined the Method 202 CPM method?

Method 202, as promulgated in 1991, had the potential to create “sulfate measurement artifact” when the method was used to sample gas streams that contain sulfur dioxide. Over time, sulfur dioxide dissolved in the impinger water of the 1991 method would convert to sulfur trioxide and then to sulfuric acid, which was improperly quantified as CPM by the method. The EPA promulgated amendments to Methods 201A and 202 on December 21, 2010 (75 FR 80118) and finalized minor corrections in January 2014. The amendments to Method 202 revised the sample collection and recovery procedures to reduce the formation of reaction artifacts that could lead to inaccurate measurements of CPM. Additionally, the amendments to Method 202 eliminated most of the hardware and analytical options contained in the original 1991 method, thereby increasing the precision of the method and improving the consistency in the measurements obtained by source tests performed under different regulatory authorities.

As owners and operators have moved to meet the requirements implemented by states, the results from some CPM measurements have proven to be a significant portion of the fine particulate. The CPM can be especially important if the total fine particulate mass is low. Therefore, executing Method 202 following the best measurement practices known is important to generate accurate results that demonstrate compliance for NSR and PSD. This handbook contains the current best management practices to achieve the expected results from Method 202 testing.

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1.0 INTRODUCTION

This section identifies the purpose and intended audience of this handbook and explains the importance of achieving accurate and consistent measurements when using Method 202.

1.1 What is the purpose of this handbook?

This handbook provides the best practices currently known to promote accurate and consistent results when using EPA Method 202 for measuring CPM emissions from stationary sources. The procedures in this handbook significantly improve the results obtained when using the method for measurements at sources that have low concentrations of CPM emissions. The practices and recommendations identified in this handbook are not requirements, but are intended to assist stack testing contractors in achieving consistent, comparable, and accurate results when using Method 202. This handbook will also serve as a resource for continually improving the application of Method 202.

The EPA recommends that the testing community use this handbook because it contains thorough explanations of Method 202 requirements and procedures that are intended to reduce residual mass contamination that causes most of the difficulty that stack testing contractors experience when using Method 202, particularly when testing sources with low CPM emissions. Residual mass contamination can come from the laboratory glassware, the sampling train, the CPM filter, the nitrogen purge gas, the wash bottles, the sample containers, and the reagents used to recover and analyze samples. Residual mass can also unintentionally be introduced before, during, and after sampling or any time that a component or container containing a sample is exposed to the environment.

1.2 Who should use this handbook?

This handbook was written to address the needs of:

- Source testing contractors (stack testers) that use Method 202 to measure CPM emissions.
- Laboratory technicians involved in the processing and analysis of Method 202 samples.
- State and federal regulators that review and enforce air permits and regulations based on Method 202 results.
- Modelers that use Method 202 results in air quality analyses.
- Owners or operators of facilities that test for CPM and need reliable data to inform corporate or environmental decisions.

Condensable particulate matter is defined in 40 CFR 51.50 as material that is vapor phase at stack conditions, but which condenses and/or reacts upon cooling and dilution in the ambient air to form solid or liquid PM immediately after discharge from the stack. Note that all CPM, if present from a source, is typically in the PM_{2.5} size fraction and, therefore, all of it is a component of both primary PM_{2.5} and primary PM₁₀.

1.3 Why is this handbook necessary?

This handbook is necessary for several reasons, including:

- State and local agencies have reported inconsistencies in the application of Method 202 by stack testers, as well as a lack of understanding of how to achieve the performance requirements of the method.
- Owners and operators of facilities need data of sufficient quality to generate defensible compliance data at low fine particulate emission levels.
- Stack testers have reported that it is difficult to achieve blank levels at or below the method limit, particularly for emission sources where the residual or blank mass contributed from sampling train components and sample recovery materials is a significant portion of the CPM measurements obtained from Method 202.
- Modelers have reported that data biased by high blank levels affect the utility of resulting data.
- Accurate, updated emission factors for CPM are needed by industry.

Method 202 generates method-defined results that must meet specific performance criteria to be considered valid and representative. Owners and operators of facilities and stack testers have several choices or practices at their disposal for how they implement the method, as long as the performance-based criteria are met, especially the field train recovery blank allowance of 2.0 milligrams (mg). This handbook was created to compile and present practices and techniques that show stack testers how to continuously evaluate and improve their standard procedures, including selection of sampling and analytical materials, to reduce systematic error and achieve a blank result that is within the limits of the method.

The goal of this handbook is to promote national consistency in the execution of Method 202, which has shown wide variation in its implementation and allows many performance-based options and procedures. To advance that goal, this handbook provides technical information and recommendations that serve to promote continued improvement in the application of Method 202 by stack testers using the method at its lower limits of detection.

1.4 What does this handbook contain?

This handbook contains:

- Clear explanations of the method requirements
- Technical information and recommendations for producing accurate results
- Procedures to evaluate laboratory and sampling performance
- Information on how to interpret quality control (QC) check results to provide feedback that will improve implementation of the method.

1.5 Why was Method 202 developed?

The EPA promulgated Method 202 in 1991 (56 FR 65433, December 17, 1991) to measure CPM emissions from stationary sources. The CPM measurements were needed by state and local air pollution control agencies to include the full range of PM emissions in their State Implementation Plans (SIPs). Section 110 of the Clean Air Act, as amended in 1990 (42 U.S.C. 7410), requires state and local air pollution control agencies to develop, and submit for

EPA approval, SIPs that provide for the attainment, maintenance, and enforcement of the NAAQS in each air quality region (or portion thereof) within each state. The NAAQS pollutants include PM and its components, filterable PM and CPM.

The emissions inventories and analyses, including air quality modeling to assess compliance with the NAAQS, used in the state's attainment demonstrations must consider emissions from stationary sources that are significant contributors of primary PM₁₀ and PM_{2.5} emissions, which include both the filterable and condensable fractions of PM.

Since January 1, 2011, states have been required to include CPM emissions in new or revised emissions limits that they establish. Additionally, facilities that want to construct new or expand existing operations must conduct air quality analyses, which can include air quality dispersion modeling of CPM emissions, to demonstrate that the new emissions will not cause or contribute to a violation of any applicable NAAQS or PSD increment. Accurate measurement of primary CPM emissions is of the utmost importance. In order to accomplish this, the test method must collect everything that meets the definition of CPM and prevent anything that does not meet the definition of CPM from being measured erroneously as CPM.

1.6 What has the EPA done to improve Method 202?

Following promulgation of the original 1991 Method 202, the EPA identified two key factors that had led to inconsistent results:

- Different combinations of the optional procedures can cause large variations in the measurement of CPM.
- Sulfur dioxide (SO₂) gas (a typical component of emissions from several types of stationary sources) reacts chemically in the impinger water to form sulfur compounds that were not originally CPM. This "artifact" is not related to the primary emission of CPM from the source, but has been counted erroneously as CPM when using the 1991 version of Method 202.

As a result, the CPM concentrations obtained using the 1991 version of Method 202 and related emissions factors developed from these results could be biased high. In support of impending NAAQS rules for fine PM, the EPA completed several studies to assess artifact formation from Method 202:

- *1998 EPA laboratory study and field evaluation* ("Laboratory and Field Evaluation of EPA's Method 5 Impinger Catch for Measuring Condensable Matter from Stationary Sources" at <http://www.epa.gov/ttn/emc/methods/m202doc1.pdf>.) verified the need for conducting a nitrogen purge when SO₂ is present in stack gas and provided guidance for analyzing the collected samples. The study verified that artifact formation can be reduced by at least 90 percent if a 1-hour nitrogen purge of the impinger water is used to remove SO₂ before it can form sulfuric acid.
- *2005 EPA contractor study* ("Laboratory Evaluation of Method 202 to Determine Fate of SO₂ in Impinger Water" at <http://www.epa.gov/ttn/emc/methods/m202doc2.pdf>) replicated some of the earlier EPA work and addressed some additional issues. This report also verified the need for a nitrogen purge and identified the primary factors that affect artifact formation.

- *2005 private testing contractor study* (“Optimized Method 202 Sampling Train to Minimize the Biases Associated with Method 202 Measurement of Condensable Particulate Matter Emissions” at <http://www.epa.gov/ttn/emc/methods/m202doc3.pdf>) concluded that modification of the method to use dry impingers resulted in a significant additional reduction in the sulfate artifact.
- *2006-2009 EPA conducted studies and workshops* (“Field Evaluation of Condensable Particulate Matter Measurement” located in docket EPA–HQ–OAR–2008–0348) in collaboration with several stakeholders to characterize the artifact formation and other uncertainties. Laboratory study and field evaluation led to method revisions that were promulgated on December 21, 2010.

In response to the factors that had led to inconsistent results, the EPA promulgated major revisions to Method 202 (75 FR 80118, December 21, 2010) that were based on the results of the studies that investigated these factors. The revisions reduced the optional hardware and analytical procedures and addressed SO₂ artifact formation that was associated with the original 1991 Method 202. The 2010 revisions greatly improved the precision of the method and the consistency in the results achieved, which allow the method to be used to support implementation of PM NAAQS requirements for PSD and NSR purposes. In laboratory and field tests, the 2010 revisions reduced the SO₂ oxidation bias by 90 percent over the best results obtained using the original 1991 version of Method 202. The 2010 revisions contained the following key changes to the method:

- A condenser was added to the sampling train to cool the gas stream, replicating atmospheric cooling.
- The water was removed from the two impingers in front of the CPM filter, so sampling test runs begin with dry impingers to limit artifact formation.
- A post-test filtered nitrogen purge was added to reduce absorbed SO₂ and limit artifact formation.
- The sample collection temperature was changed to ambient (≤ 30 °C/85 °F) to reduce the solubility of SO₂ in condensed moisture.
- Performance-based, residual mass contribution specifications were added for containers and wash bottles, rather than specifying the type of container that must be used.
- Performance-based requirements were added for filter materials to limit the residual mass contribution from filters.

1.7 Why is achieving reliable results for Method 202 important?

Consistent application of Method 202 and achieving reliable results when using the method are important because modelers must have accurate CPM data when conducting the air quality analyses required for PM_{2.5} NAAQS PSD assessments. The condensable portion of PM_{2.5} has become a significant factor in PSD/NSR assessment failures. Additionally, regulatory agencies and sources rely on Method 202 results to determine compliance with current NSPS and maximum achievable control technology (MACT) rules that regulate PM defined as filterable PM collected by Method 5 or one of its versions. For example, the Mercury and Air Toxics Standards (MATS) for power plants and the Pulp and Paper National Emission Standards for Hazardous Air Pollutants (NESHAP) require measurement of CPM using Method 202 when determining PM₁₀ or PM_{2.5} emission estimates.

1.8 What are the implementation issues associated with Method 202?

As owners and operators of facilities have moved to meet fine particulate emission limitations issued by states, the CPM results from some Method 202 measurements proved to be a significant portion of fine particulate. The CPM can be especially important if the total fine particulate mass is low. Therefore, executing Method 202 following the best measurement practices known at the time is important in reducing interference, minimizing method blanks, and generating accurate results for NSR and PSD compliance demonstrations.

Method 202 allows for a correction to the total measured CPM mass for each test run, known as the blank correction. The field train recovery blank result (see Sections 2.3 and 2.7.3 of this handbook) provides the blank correction value, up to a maximum of 2.0 mg. Industry has reported difficulty achieving field train recovery blanks below the 2.0 mg method allowance. As a result, the allowable blank correction value in Method 202 is often exceeded by the field train recovery blank, especially by stack testers that are unfamiliar with the performance-based requirements in Method 202. On the other hand, an EPA laboratory evaluation and a survey of stack testing firms indicate that method blanks below the blank correction allowed in Method 202 are possible if the best measurement practices are used (see Appendix A of this handbook)³.

The blank correction is included in Method 202 to subtract the systematic error from the total CPM mass measured during a test run. In this method, the systematic error is the contribution to the measured CPM result that is attributable to the sampling media (e.g., glassware, filters, and reagents). High results for field train recovery blanks, above the 2.0 mg allowance, are indicators of positive bias in test samples. Even small positive bias can present challenges when facilities model emissions to determine compliance against the NAAQS. For example, if a source wants to expand, then it must apply for a permit from the state. The source must conduct dispersion modeling for its PM_{2.5} emissions (including CPM) to determine or demonstrate that the additional emissions will not exceed the NAAQS attainment for that air quality region.

High field train recovery blanks, above the method allowance, may dominate the modeling calculations used for NSR/PSD permit applications resulting in failure to meet emission requirements for new or replacement construction projects. Thus, it may be difficult for the source to demonstrate that emissions are within an acceptable level until the stack tester has resolved any issues needed to achieve low blank results.

When source emissions are close to the detection limit, blank values have a greater effect on results. Without the use of best practices, stack testing firms may attempt to minimize the effect of blanks by collecting larger volume of stack gas. However, sampling large volumes of gas using Method 202 has technical consequences (see Section 5.4 of this handbook).

³ U.S. EPA, 2015. Method 201A and 202 Best Practices to Reduce Blanks. Prepared by Eastern Research Group for EPA. October 27, 2015.

2.0 DISCUSSION OF QUALITY CONTROL PROCEDURES

This section identifies the QC procedures specified in Method 202, the purposes of each QC procedure, and how the results of the QC checks are used to improve CPM measurements.

2.1 What are the Method 202 quality control procedures?

Quality control (QC) procedures in Method 202 are specifications that promote proper implementation of the method and verify representative results. Method 202 includes performance-based QC procedures that add flexibility in implementing the method while maintaining quality and consistency of the measurements. Stack testers are free to choose from a variety of materials and to clean equipment in a variety of ways to meet the performance criteria specified in the method. When used properly, this performance-based approach to quality management enables continuous improvement in the control of systematic method bias.

There are several QC elements written into the method that work together to verify the quality of CPM measurements. The following QC checks are general and are used to control the overall test validity, but are not directly associated with sample collection, recovery, and analysis activities that may bias the sample results:

- Daily quality checks of the field log notebooks and data entries (Method 202, Section 9.1)
- Calculation verification (Method 202, Section 9.2)
- Documentation of sampling conditions (Method 202, Section 9.3)
- Thermocouple calibrations (Method 202, Section 10.1).

In addition to these general QC checks, Method 202 includes QC procedures that focus on limiting the residual mass contribution from the materials and equipment used in collecting, recovering, and analyzing samples. Because Method 202 measures CPM through gravimetric analysis (i.e., weighing) of evaporated samples, any extraneous mass contributed from the sample collection, recovery, and analytical procedures will add to the total CPM measurement collected from an emission source. The importance of quality weight measurements is magnified when operating Method 202 at sources that have low levels of CPM emissions and/or high volumetric flow. To control the extraneous mass, Method 202 specifies performance criteria for blank checks to limit the residual mass contribution from the materials and equipment used to collect samples, and for analytical balances used to conduct the gravimetric analysis.

2.2 What is a Method 202 blank check?

In Method 202 a blank check, or blank, is a QC assessment of a Method 202 sampling component (e.g., reagent, filter, glassware) either as received from the vendor and prepared as it would be for testing and before exposure to sample gas, or, as is the case for the field train recovery blank, after sampling test runs using the component have been conducted and recovered. Blank checks evaluate the systematic bias associated with the components and verify that the components meet the method specifications so that residual mass contributions

are sufficiently low and will not contribute significantly to CPM sample measurements. Blanks are recovered from sampling components according to the procedures specified in Method 202 and are measured using gravimetric analysis.

2.3 What is the Method 202 blank correction?

The Method 202 blank correction is the subtraction of systematic error from the final CPM sample result. This correction is an allowance for the amount of background residual mass that was found to be consistently present in Method 202 train recovery blanks when using best practices in a standard laboratory environment. The blank correction value is the field train recovery blank result, limited to a maximum of 2.0 mg. The residual mass determined by the field train recovery blank is assumed to be attributable only to the materials and procedures used in sample collection, recovery, and analytical procedures.

2.4 Why is there a limit to the blank correction?

The blank correction limit helps ensure that only CPM that comes from the source is included in the final sample result, not residual contamination from reagents or equipment that is exposed to the samples. Excess field train recovery blank values may be the result of incomplete recovery of samples from sampling runs. Therefore, field train recovery blanks also provide a benchmark for how well the method is conducted by your test team and provides an incentive to eliminate as much residual mass as possible from the components used in Method 202 testing. In this way, the blank correction limit promotes continuous improvement for stack testers, as removing the contamination potentials from the components and procedures used in Method 202 to meet the 2.0 mg limit leads to more accuracy and reliability. Minimizing the field train recovery blank is particularly critical when testing sources with low CPM concentrations, where a value of 2.0 mg can be significant compared to sample results.

2.5 How can blank checks identify and limit sources of contamination in Method 202?

Collectively, the blank checks provide stack testers with tools to identify the specific components of the method that are contributing residual mass. This is information that the testers can use to eliminate sources of contamination and improve the implementation of the method. In cases where the field train recovery blank limit is not being met, these checks can be particularly useful in determining which aspects of the method need to be addressed. Blank results that are below the method criteria indicate that the component being evaluated is not a significant source of residual mass contamination, while results above the criteria indicate that the component is a source of contamination. After the sources of contamination are identified, stack testers can take steps to reduce the contamination, such as purchasing a different reagent or changing a glassware cleaning procedure.

Over time, as a stack testing team receives training and gains experience, incremental improvements associated with implementation of the method will lower blank results. These lower results verify good procedures and clean components for the method, while higher results will call stack tester's attention to areas that need improvement. Testers can use blank check results as continuous improvement markers by tracking performance and charting the results over time. Stack testing firms should document the practices and procedural improvements in their standard operating procedures (SOP) that are shown to result in lower blanks. Such documentation retains the information and ensures continued, successful implementation of the method.

2.6 What types of QC checks are in Method 202?

The QC checks in Method 202 fall into two categories: top-down and bottom-up. These categories represent two different approaches for verifying the quality and reliability of Method 202 sampling operations. Either approach can be used, depending on which aspect of the method is being targeted. These approaches can also be used in conjunction with each other for a more robust examination of the quality of samples generated by a stack tester.

2.7 What are the components of “top-down” QC checks in Method 202?

Top-down QC measures in Method 202 are performed in the field and provide an overall evaluation of residual mass contributions to sample blanks from equipment preparation, sample recovery, and analysis. The three top-down QC checks are: (1) field reagent blank, (2) field train proof blank and (3) field train recovery blank. Although top-down QC measures can indicate if there is a residual mass contamination problem, they do not necessarily identify the origin of the problem.

2.7.1 Field reagent blanks (Method 202, Section 9.8) are QC checks of the recovery reagents (e.g., water, hexane) that are taken in the field, directly from each reagent’s wash bottle. These blanks are an important addition to the field train proof blanks and field train recovery blanks because they differentiate the contamination originating in reagents from equipment contamination occurring before or during sample recovery. The field reagent blank quantifies the amount of residual mass contributed to the sample from the reagents and can confirm that low residual mass contributions from the reagents were maintained during testing, that the wash bottles used did not contribute residual mass, and that the reagents were not contaminated during testing. Field reagent blanks are not required by Method 202, but are recommended and included as part of the Method 202 sample recovery procedures.

Because top-down QC checks only help you identify blank issues after you have collected and processed CPM samples, you assume the risk that all of the materials used (e.g., reagents, CPM filter, and sampling train glassware) pass the low-residual blank quality requirements if you have not conducted a bottom-up QC review.

2.7.2 Field train proof blanks (Method 202, Section 9.9) evaluate equipment preparation and potential contamination during sample recovery. The intent of field train proof blanks is to measure the lowest achievable mass contribution background resulting from the entire Method 202 sampling and analysis process. While the proof blanks described in Method 202 can be improved as described later in this handbook, they still indicate whether a sampling train intended for use in Method 202 would contribute an acceptably low residual mass contribution to field samples. This blank is recovered at the test site from a clean, fully-assembled sampling train before it is used for testing. Stack testers must conduct field train proof blanks for sampling trains that use glassware that has not been baked after cleaning. For a field train proof blank, the train is set up as it would be for testing, including the CPM filter, and the train may then be leak checked. The aqueous and organic sample fractions and CPM filter are recovered from the train and returned to the laboratory for analysis according to the procedures in Method 202.

An alternative field train proof blank procedure has been developed by the National Council for Air and Stream Improvement (NCASI) (see Appendix B of this handbook). These

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additional best practices for proof blanks provide options to address field blank issues not originally covered in Method 202. This alternative procedure seeks to capture all of the sources of random and systematic error associated with Method 202 sampling. These sources of error include:

- Decontamination of the laboratory, sampling train, and sampling recovery equipment components.
- Transportation of the sampling train and sample recovery components to the field.
- Exposure of the sampling train to the sampling environment.
- Operation of the sampling train, including leak check.
- Recovery of samples.
- Transportation of samples from the site to the laboratory.
- Analysis of samples.

NCASI's alternative field train blank procedure consists of the following steps:

- 1) Take clean sampling train components to the field.
- 2) Assemble the sampling train at the sample collection site (e.g., on sampling platform).
- 3) Perform leak check on the sampling train.
- 4) Leave the sampling train exposed to the sampling environment for the same length of time as sample run (i.e., approximately 2 hours, typically).
- 5) Perform a post-run leak check.
- 6) Take the sampling train to sample recovery area.
- 7) Perform the post-test nitrogen purge (add water to dropout impinger).
- 8) Recover samples using the same procedures used for actual test samples.
- 9) Transport samples to laboratory.
- 10) Process and gravimetrically analyze samples according to the procedures in the method.

This NCASI train blank protocol addresses possible sources of contamination that are not included in the field train proof blank procedures of Method 202, specifically the leak checks, the amount of time the train is exposed to the sampling environment, and the post-test nitrogen purge. The NCASI believes their train blank protocol may be more representative of the error associated with Method 202. The EPA recommends using this procedure when collecting field train proof blanks as a Method 202 best practice.

Stack testers should note that permission from the EPA is required to use alternatives that are not explicitly stated in the method, such as this alternative field train proof blank procedure. Specific instructions on how to request approval for alternatives are in an Emission Measurement Center guideline document.⁴

2.7.3 Field train recovery blanks (Method 202, Section 9.10) are collected from a fully-assembled sampling train, prepared as it would be for testing at the test site after the first or second test run has been conducted. For this blank, 100 mL of water is added to the first impinger of the sampling train and the train is purged with nitrogen before sample recovery is

⁴ U.S. EPA, 2014. Emission Measurement Center Guideline Document: Requests for Approval of Alternatives/Modifications to Test Methods and Testing Procedures (EMC GD-022R4). Prepared by Robin R. Segall, USEPA/OAQPS/AQAD/EMC. April 21, 2014. Available at <http://www3.epa.gov/ttn/emc/guidlnd/gd22.pdf>.

performed. Field train recovery blanks evaluate the recovery of the field sample from the previous test run.

In addition to sample recovery procedures, this blank evaluates contamination from the sampling train, exposure to the sampling environment, the recovery reagents, and the glassware used for sample transportation and processing. This blank indicates the residual mass contribution from all of the materials and procedures used to collect Method 202 samples and the carryover or unrecovered mass from the previous sampling run. The field train recovery blank result, up to the maximum allowed by the method (2.0 mg), is the systematic blank correction to the field sample result. High field train recovery blanks alert stack testers that poor field recovery techniques are the cause of carryover contamination and contamination during sample recovery.

2.8 How can stack testers use the top-down QC in Method 202 to improve method performance?

Field reagent blanks, field train proof blanks, and field train recovery blanks are the main tools in top-down quality review and answer the question, “Are there any problems with my procedures that are causing blanks to be over the method limit?” Figure 2-1 shows a logical path to identify where improvements need to be made to reduce sample blank contamination using a top-down approach.

If field train proof and recovery blank results are less than or equal to the method limit, then stack testers can assume that their procedures are under control and they are generating reliable results. However, if field train proof and recovery blanks are not less than or equal to the method limit, then testers can use the improvements described in Sections 6 through 10 of this handbook to remove contamination, improve implementation of the method, and lower field blank results.

2.9 What are the components of bottom-up QC checks in Method 202?

Bottom-up QC measures in Method 202 are conducted in the analytical laboratory on a component-by-component basis to identify specific contributors to elevated blanks that originate in the preparation of materials and supplies prior to field testing and in the sample analysis following the field testing. In general, you can conduct bottom-up QC checks on each component used in the collection, recovery, and analysis of CPM samples.

Method 202 specifies the maximum residual mass contribution for the CPM filter, nitrogen purge gas, reagents, sample containers, and wash bottles. However, the components that have to greatest potential to contribute residual mass to the CPM measurements are:

- Reagents used for sample recovery
- CPM filters
- Glassware used in sample trains and sample processing
- Wash bottles

Performing bottom-up QC before a sampling campaign can identify issues with reagents, filters, materials, and equipment cleaning so that stack testers can be confident that they will generate reliable results in the field.

Figure 2-2 shows a typical sequence for performing bottom-up Method 202 QC checks. When using the bottom-up QC check flow chart, if a “No” answer is obtained for a reagent or a CPM filter, meaning that it does not meet the method specifications, the lot should be rechecked to confirm the result. If confirmed, then a different lot of the same type of reagent should be checked. If the second lot fails as well, then a reagent or filter with a lower residual mass on evaporation or of a higher quality should be obtained and checked. Repeat this process until the method criteria are met. The laboratory procedures used for checking the reagents, CPM filters, and glassware should be evaluated to verify that reliable results are being generated by the bottom-up QC checks.

2.9.1 Laboratory reagent blanks are taken from their storage containers, typically their original container, and processed according to the procedures in the method (Method 202, Section 9.7). These blank results determine whether or not the lot evaluated for each reagent is acceptable for use. The use of this blank reduces the potential for high field train recovery blanks by ensuring that only reagents that meet the residual mass specifications in the method are used.

2.9.2 Filter blanks are taken from an unused CPM filter and processed similarly to a CPM filter from a test run (Method 202, Sections 11.2.1.1 and 11.2.1.2); the filter is extracted by sonication using water (three times), extraction by sonication using hexane (three times); the aqueous fraction is extracted, and both fractions are evaporated then analyzed gravimetrically. This blank indicates whether or not the selected filter type is acceptable for use in the Method 202 sampling train and quantifies the amount of residual mass contributed to the sample from the CPM filter. This blank is not in Method 202; however, the EPA considers this QC measure to be a Method 202 best practice. The method requires that the CPM filter contribute 0.5 mg of residual mass or less (Method 202, Section 7.1.1).

2.9.3 Glassware cleaning blanks are taken from individual glassware components used in assembling the sampling train (e.g., condenser, impingers) and during sample recovery (e.g., beakers, separatory funnels) and processed similarly to field train proof blanks. Aqueous and organic rinses are collected, the aqueous fraction is extracted, and both fractions are evaporated then analyzed gravimetrically. This blank shows how effective the stack tester’s glass cleaning procedures are at removing residual mass. This blank is not in Method 202; however, the EPA considers this QC measure to be a Method 202 best practice.

2.10 How do you use the “bottom-up” QC to improve method performance?

Bottom-up quality review assesses the fundamental contributors to measurement blank that come from the reagents, glassware, and filter preparation prior to a field test. The bottom-up QC procedures in Method 202 also evaluate the technical capability of the materials used and the staff responsible for field test preparation to meet the performance-based blank requirements of the method. Bottom-up QC review helps identify blank issues before committing to a field measurement effort using Method 202. Therefore, bottom-up quality review identifies improvements that should be made before undertaking a Method 202 test run.

Blanks from reagents, sampling train glassware, and CPM filters are assessed during bottom-up quality review. Using the materials that meet or exceed the method specifications for residual mass helps reduce residual (blank) mass recovered in CPM samples. The sum of the blank results from these bottom-up QC checks represents the majority of the residual mass

2.0 Quality Control Procedures

that can be expected in a field train proof blank. Each of the blank checks provides key information about the materials and procedures used to prepare for Method 202 tests.

- Laboratory reagent blanks help in the selection of reagents of sufficient quality (tracked by lot).
- CPM filter blanks help in proper filter selection (tracked by lot).
- Glassware cleaning blanks help evaluate the sampling train and sample processing glassware cleaning and preparation procedures. Individual Method 202 sampling train components can be barcoded or otherwise identified to allow for tracking of the trains themselves.

Bottom-up and top-down QC checks can be used together to identify sources of contamination. For instance, if a laboratory reagent blank is sufficiently low, indicating that the reagent meets the method specification, but the field reagent blank is out of specification, then the reagent was likely contaminated in some way, either from the wash bottles in which they were used or from the environment. This information can prevent stack testers from unnecessarily taking steps to remove a possible source of contamination, such as replacing a reagent, when the contamination is actually being introduced from another source.

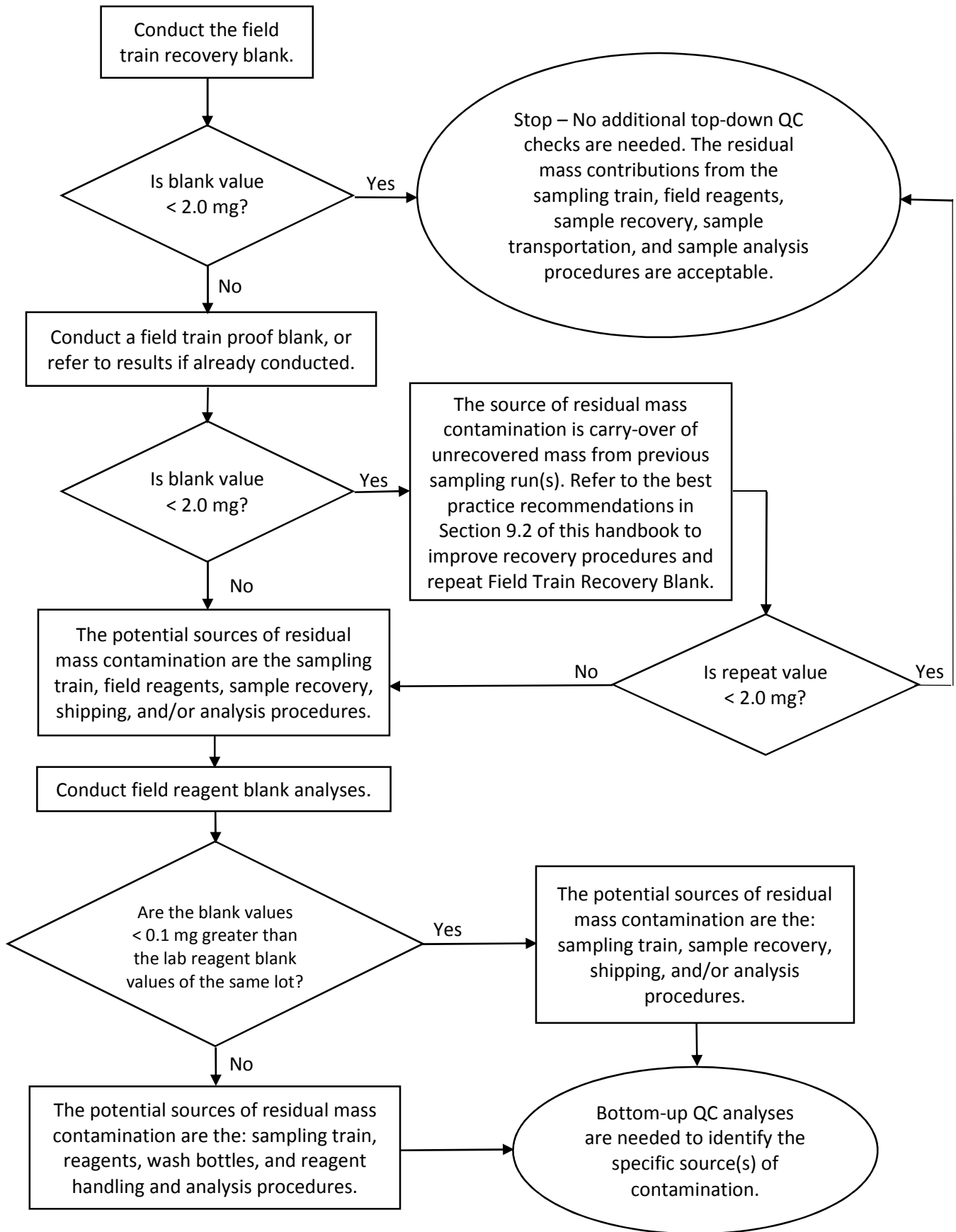


Figure 2-1. Method 202 Top-Down QC Flow Chart

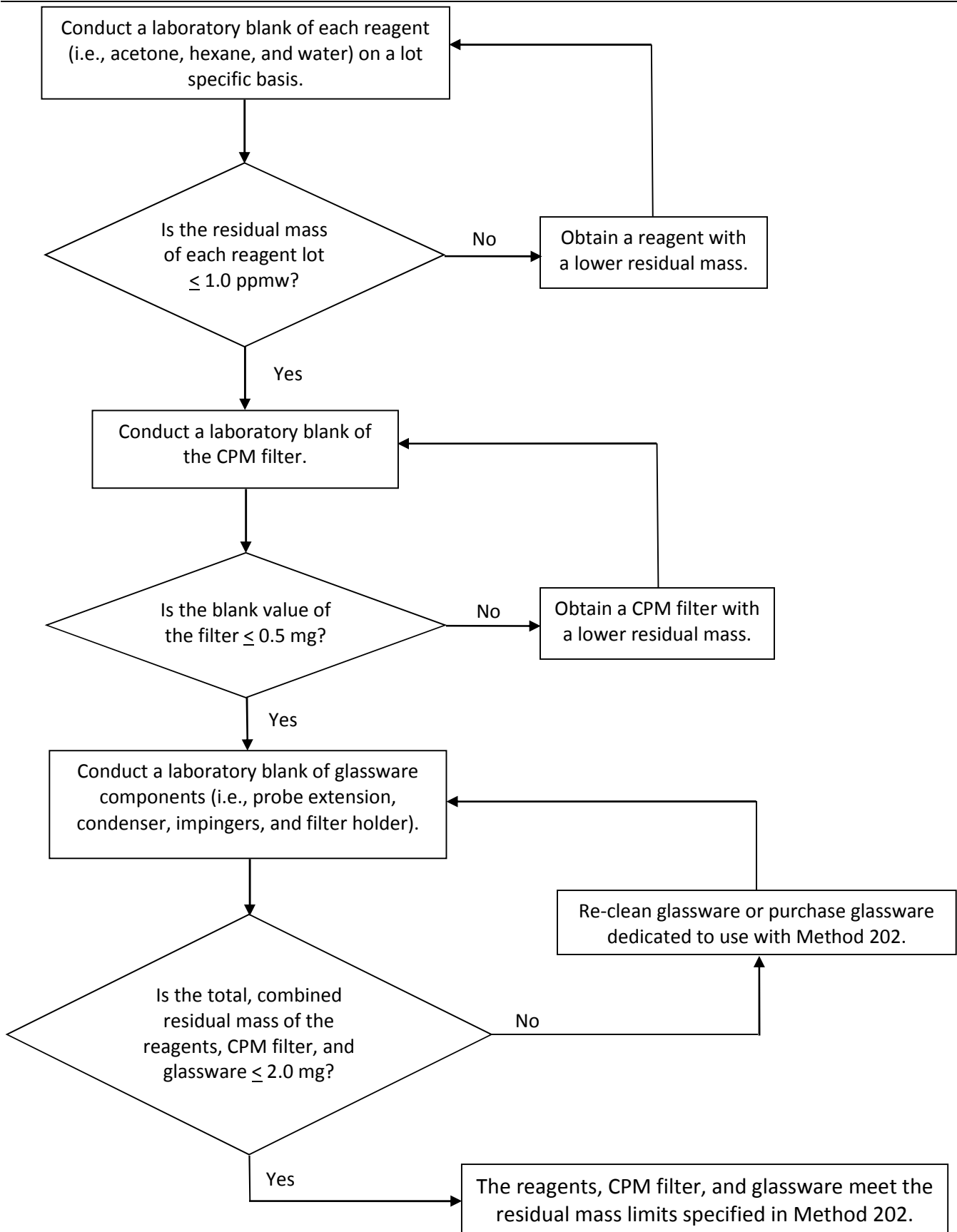


Figure 2-2. Method 202 Bottom-Up QC Flow Chart

3.0 ANALYTICAL DETECTION LIMITS

This section presents information regarding the analytical detection limits of Method 202.

3.1 What are analytical detection limits and how do they apply to Method 202?

An analytical detection limit is the lowest amount of an analyte that can be distinguished from the absence of that analyte. This limit is the value at which detection of an analyte cannot be differentiated from random error. In Method 202, the analyte is CPM and it is measured as the residual mass remaining after samples are evaporated. The residual mass of the samples is determined gravimetrically using analytical balances.

Detection limits are dependent on many factors, including the source matrix and the proficiency of the test team performing the measurement. Therefore, the method detection limit (MDL) provided in this handbook is an estimate that is based on experiments performed in the laboratory using Other Test Method (OTM) 28, a precursor to Method 202. The Method 202 analytical detection limit—the lowest amount of CPM that can be measured after correcting for the systematic bias, was experimentally determined from seven replicate analyses to be approximately 2 mg. This value comes from a laboratory report that was generated to support the 2010 revision to Method 202⁵.

The mass correction allowance of up to 2.0 mg in the method is based on this experimentally determined detection limit value. The intent of the blank correction allowance was to eliminate the systematic bias from the measurement of the two fractions generated for gravimetric analysis in Method 202.

Actual Method 202 analytical detection limits can vary greatly between sources, depending on which options are chosen and how stack testers execute the method. To account for the multitude of differences and variations between sources and testers, the EPA recommends that stack testers determine individual tester-specific detection limits.

3.2 How are source-specific and tester-specific analytical detection limits determined?

Procedures for determining source- or tester-specific analytical detection limits are described in the Summary of Public Comments and Responses for the 2010 Methods 201A and 202 revision proposal⁶. Source-specific detection limits can be calculated by recovering and analyzing multiple field train recovery blanks at a particular source and multiplying the standard deviation of the blank results by the single-sided critical Student's t value at 99 percent confidence that corresponds to the number of blanks performed. Tester-specific detection limits are determined similarly, using field train proof blanks performed. Testing firms

⁵ U.S. EPA, 2009. Draft Project Report: Evaluation and Improvement of Condensable Particulate Matter Measurement. Prepared by Eastern Research Group and E. H. Pechan & Associates for EPA. March 5, 2009.

⁶ U.S. EPA, 2010. Summary of Public Comments and Responses for Methods for Measurement of Filterable PM₁₀ and PM_{2.5} and Measurement of Condensable Particulate Matter Emissions from Stationary Sources at <http://www.epa.gov/ttnemc01/methods/comments201a202.pdf>.

may want to determine one stack test team's performance against another to ensure uniform quality among teams or company locations. The equation below can be used for these determinations.

The analytical detection limit formula is as follows:

$$DL = (S)(t)$$

Where:

- DL = Source-specific or tester-specific analytical detection limit, mg.
- S = Standard deviation of the field train recovery or proof blank results, mg.
- t = Critical Student's t value at 99 percent confidence, single-sided, for the number of field train blanks evaluated.

Initial tester-specific detection limits can be estimated by generating a minimum of seven field train proof blanks prior to field testing. Tester specific detection limits can be compared to the EPA's estimate of detection limit to assess the effectiveness of implementing best practices for Method 202. Since field train proof blanks are performed before each source test has begun and, therefore, are not exposed to source emissions, subsequent proof blank results can be included in an ongoing source tester-specific detection limit calculation. This will result in a very reliable tester-specific detection limit with a robust dataset as more proof blank results are added to the calculation over time.

3.3 How can stack testers reduce their analytical detection limits?

Stack testers can achieve lower detection limits by reducing the residual mass contributions from the components and materials used in Method 202, including the sampling train glassware, filters, and reagents, and by implementing procedures that prevent contamination. See Sections 6 through 10 of this handbook for best practice recommendations that can help reduce residual mass contributions. Reducing the residual mass lowers both the random and systematic error contributing to higher detection limits.

3.4 How is the residual mass contribution of a particular material determined?

Blank checks provide a way to determine the residual mass contribution of a material used in Method 202 (see Sections 2.2 and 2.9 of this handbook). Performing blank checks is critically important in ensuring low detection limits. Blank checks can verify that a material meets the method specification for residual mass and locate sources of excessive residual mass.

3.5 What role does the analytical balance play in determining detection limits?

Because Method 202 samples are analyzed gravimetrically, the analytical balance used can influence the detection limits. The environmental conditions that the balance and desiccator are exposed to affect their performance and, thus, any corresponding detection limits that are determined using the balance. Controlling the environmental conditions and following the best practices will help to ensure accurate mass measurements (see Section 10 of this handbook). Also, the sensitivity of the balance may be a minor contributor to the detection limit. The method specifies that the analytical balance must be capable of weighing

3.0 Analytical Detection Limits

at least 0.0001 g (0.1 mg) and requires that calibration checks be performed with NIST traceable weights each day that CPM samples are measured. Proper balance checks to evaluate accurate weight measurement are critical to maintaining high-quality gravimetric analysis and the appropriate detection limits.

4.0 METHOD 202 OPTIONS

This section presents options that are available in Method 202.

4.1 Why are options allowed in Method 202?

Method 202 has several options that are available for collecting, recovering, processing, and analyzing samples, as well as for cleaning the sampling train. The method also allows different filterable PM methods (e.g., Method 201A, 5, 17, etc.) to be used in conjunction with Method 202. These performance-based options provide flexibility for stack testers in the implementation of the method. Specifying performance-based criteria means that the method does not tell the tester exactly what to use or how to perform a particular method procedure. Rather, the method defines the criteria that must be met for a specific method element so that alternatives can be used while maintaining the quality and reliability of the CPM measurement results.

4.2 What options are available for Method 202 sample collection?

Method 202 samples are collected using a sampling train that consists of stainless steel and glass components that are standard equipment for conducting isokinetic stack sampling. These components include the following:

- Method 23-type condenser
- Water dropout impinger or bottle
- Modified Greenburg-Smith back-up impinger
- CPM filter with filter holder
- Moisture trap
- Connecting glassware

The EPA recommends a vertical configuration for the condenser as a best practice to prevent water from collecting in the condenser coils and sample gas from then bubbling through it, which could promote SO₂ artifact formation. If necessary, the water dropout impinger can be replaced with a dropout bottle to accommodate the vertical condenser. The moisture trap that follows the CPM filter consists of a modified Greenburg-Smith impinger containing 100 mL water and an impinger containing silica gel. The moisture trap equipment housed in a separate impinger box is cooled as is typically done in Method 5. See Figures 4-1 and 4.2 for schematics of combined Method 201A and Method 202 sampling trains with the condenser in both configurations. Depending on which filterable PM method (e.g., Method 201A, Method 17, and Method 5) is being used in conjunction with Method 202, a probe extension may be needed for the sampling train. If a probe extension is used, it should be heated sufficiently to prevent condensation. The CPM sample includes all sampling components after final filterable PM collection through the CPM filter. The following options are available for Method 202 sample collection, some of which have performance-based specifications:

- Sampling train configuration: The condenser can be set up vertically (which is the EPA's best practice recommendation) or at 45 ° from vertical.

- Type of CPM filter: Any nonreactive, nondisintegrating polymer filter that does not have an organic binder, does not contribute more than 0.5 mg residual mass, and is 99.95 percent efficient on 0.3 μm dioctyl phthalate particles (Method 202, Section 7.1.1).
- Material for the CPM filter holder: Glass, stainless steel, or fluoropolymer-coated stainless steel (Method 202, Section 6.1.3).
- Type of support filter (if used): Must meet the same specifications as the CPM filter; can be used to reduce the pressure drop (Method 202, Section 6.1.3).
- Type of probe extension (when used): Glass-lined or fluoropolymer-lined (Method 202, Section 6.1.1).
- Procedure to conduct the filtered nitrogen purge: Using the meter box or passing nitrogen through the train under pressure (Method 202, Section 8.5.3).
- Portion of the train included in a pressurized nitrogen purge (when conducted): May be conducted using the entire train from the inlet of the condenser to the outlet of the CPM filter holder or using only the back-up impinger and CPM filter holder (Method 202, Section 8.5.3.1).

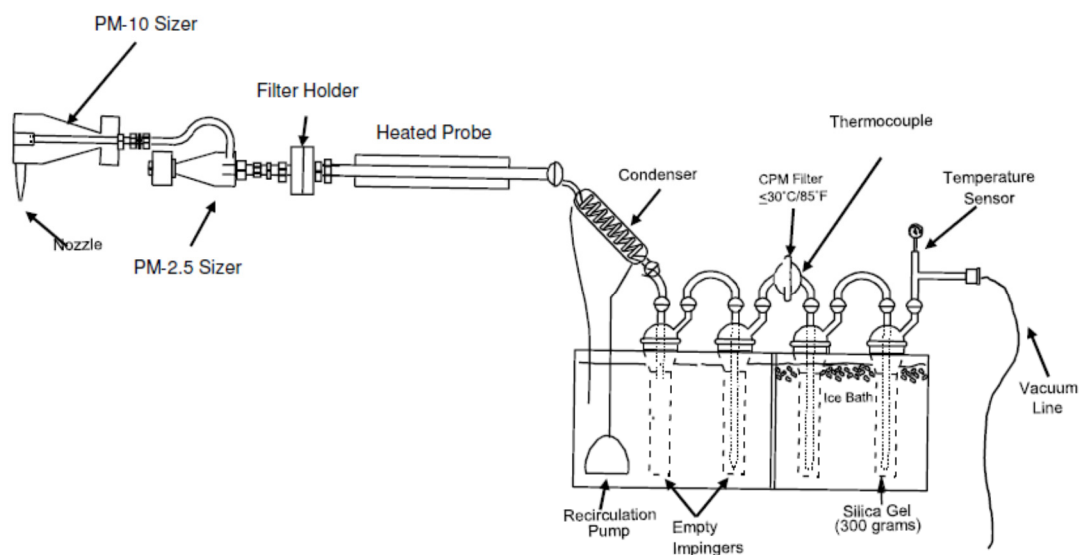


Figure 4-1. Schematic of Combined Method 201A & 202 Field Sampling Train

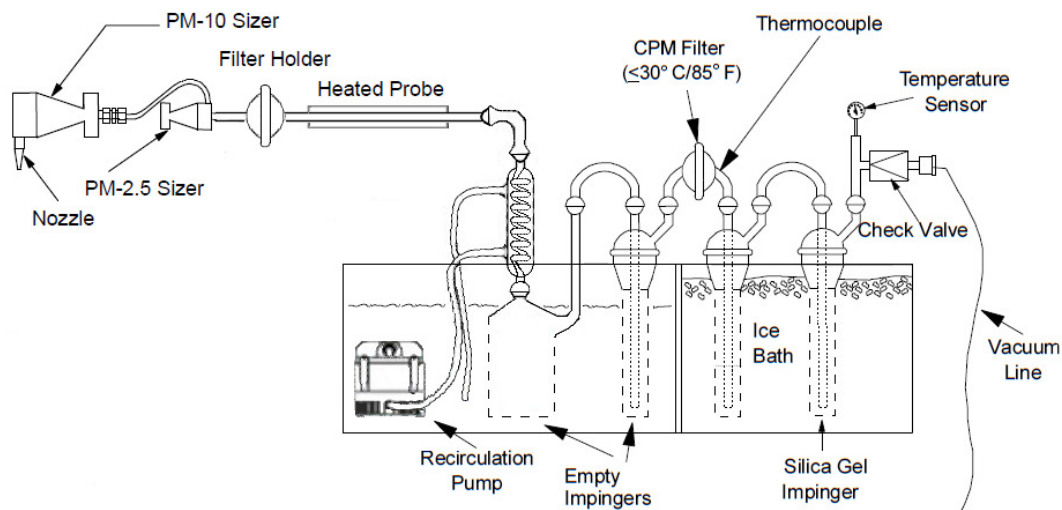


Figure 4-2. Schematic of Method 201A & 202 Sampling Train with Vertical Condenser

4.3 What options are available for Method 202 sample recovery?

Method 202 CPM samples are recovered as follows:

- 1) Transfer the liquid from the CPM sample portion of the train into the aqueous sample container.
- 2) Rinse the CPM sample portion of the train with water twice and collect those rinses in the aqueous sample container.
- 3) Rinse the train with acetone and collect that rinse in the organic sample container.
- 4) Rinse the train with hexane twice and collect those rinses in the organic sample container.
- 5) Transfer the CPM filter into the CPM filter container (typically Petri dishes are used).

The CPM sample portion of the train includes the back half of the filterable PM filter holder, the probe extension (depending on the train configuration), the condenser, the water dropout impinger, the back-up impinger, the front half of the CPM filter holder, the connecting glassware, and the CPM filter. See Figure 4-1 for the schematic of a combined Method 201A and Method 202 sampling train. Method 202 sample recovery procedures include the following performance-based options:

- Use of any deionized, ultra-filtered water containing 1.0 part per million by weight (ppmw) (1.0 mg/L) residual mass or less (Method 202, Section 7.1.3).
- Use of any acetone that is stored in glass containing 1.0 ppmw (0.791 mg/L) residual mass or less (Method 202, Section 7.2.1).
- Use of any hexane that is American Chemical Society Grade with 1.0 ppmw (0.655 mg/L) residual mass or less (Method 202, Section 7.2.2).
- Use of wash bottles made of any material, provided that the bottle has a residual mass contribution of less than 0.1 mg (Method 202, Section 6.2.2).

- Use of clean amber glass bottles with residual organic plus inorganic mass contribution of less than 0.05 mg as sample containers (Method 202, Section 6.2.2).

4.4 What options are available for Method 202 sample processing and analysis?

Sample processing for Method 202 includes procedures for extracting the CPM filter with water and hexane, extracting the aqueous sample with hexane, and evaporating the aqueous and organic samples to dryness in weighing vessels. Method 202 sample analysis is conducted gravimetrically using analytical balances. The following options are available:

- Hot plates or ovens can be used to assist evaporation and reduce aqueous samples to approximately 10 mL (Method 202, Section 6.2.2).
- Gravimetric analysis can be conducted using glass evaporation vials, fluoropolymer beaker liners, or aluminum weighing tins (Method 202, Section 6.2.2).

4.5 What options are available for sampling train cleaning?

Method 202 requires sampling trains to be cleaned before stationary source testing begins. The cleaning procedure includes washing the train components with soap and water and rinsing with tap water, deionized water, acetone, and hexane. There are two options for the final step in the cleaning process. Stack testers can either: (1) bake the glassware at 300 °C for 6 hours or (2) perform a field train proof blank on the sampling train. The field train proof blank procedures are specified in Method 202, Section 8.5.4.10. Section 7.4 of this handbook recommends best practices for cleaning sampling train glassware.

4.6 What filterable PM methods can be used with Method 202?

Method 202 is operated in conjunction with a filterable PM method. This filterable PM method is the front portion of the sampling train and Method 202 is the back portion (see Figure 4-1). Method 202 collects CPM after the filterable PM is removed. The filterable PM filter marks the boundary between the filterable PM sample and the Method 202 CPM sample. The following filterable PM methods may be used with Method 202:

- Method 5, 5A, 5D, and 5I (Method 202, Section 1.4)
- Method 17 (Method 202, Section 1.4)
- Method 201A (Method 202, Section 1.4)

At ambient sources, where the temperature of the stack gas never exceeds 30 °C (85 °F), Method 202 is not needed to measure total primary PM because all PM is filterable at these temperatures.

5.0 LIMITATIONS OF METHOD 202

This section identifies limitations of Method 202.

5.1 What are the limitations of Method 202?

Method 202 has limitations that affect how the method can be implemented and what is detected by the method. There are limitations associated with “wet” sources, the filterable PM filter temperature, sample gas volume, and sampling train cleaning, as described below.

5.2 What filterable PM method should be used in conjunction with Method 202 for sources that have emissions with entrained moisture droplets?

For sources with emissions that have entrained moisture droplets (i.e., “wet” sources) an out-of-stack filter method that operates at temperatures high enough to cause water droplets to become vaporous, such as Method 5, must be used as the accompanying filterable PM method. Wet sources are not compatible with filterable PM methods that use in-stack filters, including Method 17 and Method 201A, because entrained water droplets containing dissolved solids are not fractionated into the proper filterable particulate fractions by these methods.

5.3 How does the filterable PM filter temperature affect Method 202 CPM measurement?

Because CPM originates from vapor phase materials present in the stack, that condense and/or react upon cooling and dilution in the ambient air, the formation of CPM depends on the temperature that the sample gas is exposed to. In the Method 202 sampling train, the temperature of the CPM filter must be maintained as specified in the method (i.e., maintain the CPM filter temperature between 20 °C (68 °F) and 30 °C (85 °F)). However, the filter associated with the filterable PM method that precedes the Method 202 sampling train depends on the filterable PM method specifications or on the stack conditions (if the filterable PM method does not specify a filter temperature).

Filterable PM methods that have an out-of-stack filter require stack testers to maintain filter temperature according to the specification of the method. For filterable PM methods with in-stack filters, the temperature of the source determines the filter temperature. Condensable compounds that are sufficiently cooled at the filter associated with the filterable PM method to form solid or aerosol PM are captured by the filter and are not quantified as CPM (this material would be counted as filterable PM). Condensable compounds in the sample gas that remain in the vapor phase after the filter associated with the filterable PM method proceed through the sampling train and are potentially captured on the CPM filter.

5.4 What issues are associated with increasing the volume of sample gas collected?

When the volume of sample gas is dramatically increased, the amount of moisture collected in the water dropout and back-up impingers increases. Sulfur dioxide accumulates in the collected water over time even for sources containing low (1 ppm) SO₂. This can lead to the formation of artifacts—these artifact materials are measured as CPM by Method 202, but are not attributable to the source, much like the artifact formed in the pre-2010 version of

Method 202. Larger amounts of accumulated moisture and longer sampling times lead to greater potential for interaction with the SO₂ in stack gas and larger amounts of any artifacts that are generated. When stack testers must collect large volumes of sample gas, the method recommends that stack testers remove the accumulated moisture from the sampling train if it is greater than half the capacity of the water dropout impinger or above the tip of the back-up impinger (Method 202, Section 8.5.1.1).

A parallel issue with increasing the volume of sample gas is the increased time required for higher volume sampling runs. Longer test runs allow more time for the slow SO₂ reactions that lead to artifact formation to take place. The post-test nitrogen purge is designed to remove dissolved gasses from the accumulated moisture and reduce the formation of artifacts. Longer sample times delay the start of the purge, increasing the potential for artifact formation. In order to limit this potential for artifact formation, the EPA's best practice recommendation is that stack testers limit the sampling time to 2 hours for Method 202 testing when acid gases are present or moisture exceeds 2 percent causing it to condense with CPM.

5.5 What is the limitation for Method 202 sampling train cleaning?

Method 202 is operated in conjunction with a filterable PM method, such as Method 5, 17, or 201A (see Section 4.6 of this handbook). Some of the combined sampling train configurations require the use of a heated probe extension in the Method 202 portion of the train and the probe extension is recovered as part of the CPM sample. These probe extensions can often be too large to fit into ovens and, therefore, cannot be baked. Method 202 specifies that if the full sampling train is not baked, then a field train proof blank must be performed (Method 202, Section 8.4). Furthermore, the size and shape of probe extensions can make basic cleaning more difficult. Stack testers should be as thorough as possible when cleaning probe extensions (see Section 7.3 of this handbook).

6.0 BEST PRACTICES FOR REAGENTS

This section identifies the reagents used in Method 202, the method requirements, and the EPA's recommended best practices for minimizing the contribution of residual mass from reagents.

6.1 What reagents are used in Method 202?

Method 202 requires the use of the following reagents for glassware cleaning, sample recovery, and analysis:

- Deionized water
- Acetone
- Hexane

These reagents are used extensively throughout Method 202, presenting multiple opportunities for any residual mass contained in the reagents to contaminate the sampling train or recovered samples. Furthermore, because they are used to clean the glassware, the reagents determine the baseline cleanliness of the sampling train itself. Thus, it is very important to use reagents that contain as little residual mass as possible, follow the method requirements, and use best practices to limit potential contamination.

6.2 What are the method requirements for the reagents used in Method 202?

As specified in Section 7.2 of Method 202, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. If such specifications are not available, then you must use the best grade available. Each reagent must not contribute more than 1.0 ppmw (0.1 mg/100 g) of residual mass on evaporation (Method 202, Sections 7.2.1, 7.2.2 and 7.2.3). With regard to storage, acetone must be stored in a glass bottle and not a metal container (Method 202, Section 7.2.1). Laboratory and field reagent blanks are not required by the method, but the EPA recommends that you run such blanks as a way to determine the residual mass contribution that can be expected when using the reagent (see Sections 2.7.1 and 2.9.1 of this handbook).

6.3 What are the EPA's recommended best practices regarding Method 202 reagents?

The EPA recommends the following best practices to minimize the residual mass contributions from the reagents used in Method 202:

- Purchase and use the highest quality reagents that are available. When listed, use the manufacturer's stated residual mass on evaporation value to determine if the reagent is below the residual mass on evaporation limit of 1.0 ppmw specified in Method 202. If not listed, the residual mass value can be determined using the laboratory reagent blank procedure.
- Run the laboratory reagent blanks (Method 202, Section 9.7) before conducting field tests to confirm that the residual mass concentration is below the method limit for each reagent lot obtained. You should use a volume of 500 mL so that the amount of residual mass generated can be measured on the analytical balance.

6.0 Best Practices for Reagents

- Store each reagent in its original container until it is needed for sample recovery and analysis.
- Do not use acetone that has been shipped or stored in a metal container because this reagent can acquire metal from the container walls that can show up as residual mass.
- Wear gloves when handling reagents and minimize the amount of time that reagents are exposed to the environment to prevent contamination of reagents in the laboratory and in the field.
- Perform field reagent blanks (Method 202, Section 9.8) to verify that: (1) the residual mass concentrations of the reagents used are below the method limit throughout testing, (2) the wash bottles used for the reagents do not contribute residual mass, and (3) the reagents are not contaminated during testing. For field reagent blanks, a volume of 500 mL, taken directly from the wash bottle used during sample recovery, should be used so that the amount of residual mass generated can be measured on the analytical balance.
- After opening a reagent, label the bottle with date opened and expiration date. You may choose any reasonable expiration date if none is specified by the manufacturer but we recommend that it be no more than a year after the bottle was opened to avoid unexpected contamination and/or evaporation.
- Never store reagents or solvents in recovery bottles; after use, any residual reagent or solvent should be disposed of accordingly.

7.0 BEST PRACTICES FOR SAMPLING TRAINS

This section identifies the EPA's recommended best practices for minimizing the contribution of residual mass from the sampling train, including the CPM filter, and highlights pre-test procedures that prevent contamination.

7.1 What is the Method 202 sampling train configuration?

Method 202 collects CPM from stationary source emissions after filterable PM has been removed from the sample gas. This is accomplished by operating a sampling train for filterable PM upstream of the Method 202 sampling train (see Figure 4-1 of this handbook). Method 202 sampling trains consist of the following components:

- Probe extension (when required by the filterable PM sampling train being used in conjunction with Method 202; should be heated sufficiently to prevent condensation)
- Condenser
- Water dropout impinger
- Modified Greenburg-Smith impinger
- CPM filter with filter holder
- Modified Greenburg-Smith impinger containing 100 mL water
- Modified Greenburg-Smith impinger (optional)⁷
- Modified Greenburg-Smith impinger containing silica gel
- Connecting glassware

The exit of the Method 202 sampling train (i.e., the outlet of the silica gel impinger) is connected to the sampling system meter box and vacuum pump. The CPM is collected on and recovered from the portion of the sampling train between the filterable PM filter and the CPM filter.

7.2 What are the Method 202 requirements for the sampling train?

In addition to the configuration requirements described above, Method 202 has the following requirements for the components of the sampling train:

- The heated probe extension, when used, must be glass-lined or fluoropolymer-lined (Method 202, Section 6.1.1).
- The CPM filter must be a nonreactive, nondisintegrating polymer filter that does not have an organic binder, does not contribute more than 0.5 mg residual mass, and is 99.95 percent efficient on 0.3 μm dioctyl phthalate particles (Method 202, Section 7.1.1).
- The support filter, if used, must meet the same specifications as the CPM filter; can be used to reduce a pressure drop if present (Method 202, Section 6.1.3).

⁷ Optional impinger not included in Figures 4-1 and 4-2. By using this optional impinger, you may operate the "dry side" of the sample train at a higher temperature (<85°F) in an effort to reduce the amount of moisture collected in these impingers.

7.0 Best Practices for Sampling Trains

- The CPM filter holder must be either glass, stainless steel, or fluoropolymer-coated stainless steel (Method 202, Section 6.1.3).
- You must clean the sampling train using soap and water and rinse with tap water, followed by deionized water, acetone, and hexane, in that order (Method 202, Section 8.4).
- Baking the sampling train glassware at 300 °C for 6 hours is strongly recommended, but if any component of the sampling train is not baked (e.g., probe extension), then you must run a field train proof blank (Method 202, Section 8.4).
- You must complete a leak test on the sampling train and the Pitot tube assembly before beginning a test run (Method 202, Section 8.4.6).

7.3 What are the EPA's recommended best practices for sampling trains beyond the requirements of Method 202?

Minimizing residual mass contamination of the sampling trains is critical in generating reliable CPM measurements. The EPA recommends the following best practices to minimize the residual mass contributions from sampling trains:

- Purchase new sampling train glassware for use in Method 202 sampling exclusively.
- Dedicate sampling train components solely for use in Method 202 to avoid contamination from other test methods.
- Clean sampling trains according to the best practices glassware cleaning procedure (see Section 7.4 of this handbook).
- Pre-wash the condenser to ensure its cleanliness, using one of the two following pre-washing procedures:
 - 1) Sonicate the condenser in an ultrasonic bath, or
 - 2) Flush the condenser overnight with hot soapy water using a recirculation pump.
- Set-up the sampling train with the condenser positioned vertically to prevent water from collecting in the coils and increasing the contact between sample gas and water (see Figure 4-2 of this handbook).
- Clean the probe extension, if used, by sealing the inlet of the probe with an airtight fitting, adding the reagent, sealing the outlet with an airtight fitting, and rotating the probe end over end at least three times. Repeat this process three times with each reagent.
- Run a field train proof blank to provide a quantitative assessment of stack testers' glassware cleaning and handling procedures and to determine if the sampling train has a sufficiently low residual mass contribution.

Tests to evaluate selected best practices completed by the EPA in 2013 showed the importance of dedicating sampling components to only Method 202 testing. A probe that was previously used for another test method contributed hundreds of milligrams to the train blank result even though it was cleaned before use. Two more recoveries were required to achieve results below the 2.0 mg blank correction limit (See Appendix A of this handbook, Section 5.4).

7.4 What are the EPA's recommended best practices for cleaning sampling train glassware?

Cleaning the glassware removes residual mass, thereby preventing bias in samples. The EPA recommends the following best practices for cleaning analytical glassware as well as sampling train glassware:

- 1) Soak the components in water with detergent to make contamination easier to remove.
- 2) Wash in water with detergent using a brush.
- 3) Rinse with tap water.
- 4) Rinse with DI water three times.
- 5) Rinse with acetone three times.
- 6) Rinse with hexane three times.
- 7) Bake all glassware components that fit into an oven at 300 °C for 6 hours.

Use the following best practices when using this glassware cleaning procedure:

- Avoid using tap water that has a high mineral content (i.e., "hard" water).
- Use hot tap water for soaking and washing. (Note: Hot tap water may have high levels of minerals. If this is observed or suspected, use cold water.)
- Use a non-abrasive, no-residue detergent.
- Use a no-scratch brush.
- For rinsing, use reagents of the same grade as the sample recovery reagents.
- Cover all openings with polytetrafluoroethylene (PTFE) tape, rinsed foil, or cleaned caps to prevent contamination while handling, transporting, and/or storing the glassware.

7.5 What are the EPA's recommended best practices regarding the Method 202 CPM filter?

In addition to the method requirements, the EPA recommends the following best practices to minimize contributions of residual mass from CPM filters:

- All CPM filters used in Method 202 should be made of 100 percent PTFE, including support rings and support layers.
- Protect filters from contamination in laboratory and field environments at all times.
- Minimize handling of CPM filters as much as possible.
- Wear gloves and use clean forceps when handling filters.
- Inspect for filter chards generated during filter manufacture and cutting.
- Place the filters in sterile pre-cleaned glass Petri dishes for storage and transport.
- Ensure that the size and specifications of the CPM filter are adequate for the source type being measured. Using filter sizes of 80 to 100 mm and/or using a support filter can help address pressure drop and filter blinding issues.
- Conduct in-house experiments to determine the type of filters (e.g., manufacturer and model) that perform best in achieving low blank results in Method 202. This can be conducted by performing filter blank determinations (see Section 2.9.2 of this handbook) on a representative portion (e.g., 5 percent or 10 percent) of a package of filters and comparing the results of different manufacturers or models. An

7.0 Best Practices for Sampling Trains

evaluation of CPM filters of different types was performed (see Appendix A of this handbook, Section 3 and Figure 7).

- Run filter blanks from the lot to be used in testing to demonstrate that filters meet the requirements of the method. These filter blanks should be conducted for each lot of filters before they are used in testing.

7.6 What are the EPA's recommended best practices for pre-test preparation and assembly of Method 202 sampling trains?

The EPA recommends the following best practices in preparing and assembling the sampling trains to minimize the residual mass contributions from the sampling trains.

- Set up the sample train preparation area in the cleanest environment available and use a dust-free enclosure whenever possible. Choose a location that is away from the stack or any other areas with potential for contamination. If the entire facility could be a significant source of contamination, then find a location that is well away from the site if possible. Trailers work well as recovery areas because stack testers have more control over the recovery trailer than they do over a facility. Additionally, testers can actively take measures to prevent contamination in recovery trailers.
- Clean the train preparation area, including all surfaces, before assembling the sampling train.
- Cover the countertop where the train is set up with laboratory bench paper or pre-cleaned aluminum foil.
- When possible, filter the ambient air of the train preparation area using a high-efficiency particulate air (HEPA) filter.
- Limit access to the train preparation area to only essential staff to reduce the potential for contamination.
- Before assembling the sampling train, visually inspect all covers used on Method 202 glassware to confirm that the covers remained in place during storage and transport.
- Visually inspect the impingers, condensers, CPM filter/filter holder, connector pieces, probe extensions, probe liners, sampling nozzles, filterable PM filter/filter holder, and O-rings for signs of contamination, scratches, cracks, or tears. Do not use any glassware or sampling train components that do not pass the visual inspection.
- After the visual inspection, rinse the sampling train glassware, including the probe extension, impingers, condenser, and filter housing, with water, acetone, and hexane prior to assembling the sampling train.
- Verify that the CPM filter is not damaged, is properly centered in the CPM filter assembly, and that the gasket used to seal the filter holder is properly seated.
- Pre-weigh impingers so that the volume of moisture collected in the sampling train can be determined gravimetrically. This prevents the need to use graduated cylinders and reduces the potential for introducing residual mass.
- Set-up the sampling train up with the condenser positioned vertically to prevent water from collecting in the coils (see Figure 4-2 of this handbook).

8.0 BEST PRACTICES FOR POST-TEST NITROGEN PURGE

This section identifies the Method 202 requirements for the post-test nitrogen purge and presents the EPA's best practice recommendations for limiting mass contribution.

8.1 What are the Method 202 requirements for the post-test nitrogen purge?

Method 202 requires a post-test nitrogen purge at a minimum of 14 liters per minute (L/min) for 1 hour if water was collected in the sampling train. You must perform the purge as soon as possible after the post-test leak check. The purpose of the purge is to remove gases that can be partially absorbed, including SO₂, from the accumulated water. Purging limits the amount of time that gases can interact with the water and, therefore, reduces potential artifact formation.

You may conduct the post-test nitrogen purge either by using the sampling system meter box or by passing nitrogen through the train under pressure (Method 202, Section 8.5.3). Method 202 specifies the requirements for the materials used, the train configuration and water level of the impingers, the purge procedures, and the two methods of performing the purge.

8.2 What are the requirements for the materials used in a nitrogen purge?

Method 202 has the following requirements for the materials needed to conduct a nitrogen purge:

- The purge line must be made of inert tubing and fittings that are capable of delivering at a flow rate of 14 L/min from a standard gas cylinder (Method 202, Section 6.2.1).
- The rotameter must be capable of measuring gas flow up to 20 L/min and be accurate to 5 percent of full scale (Method 202, Section 6.2.1).
- The nitrogen gas cylinder, pressure regulator, and purged in-line filter must be capable of delivering a flow rate of at least 14 L/min (Method 202, Section 6.2.1).
- The nitrogen gas must be ultra-high purity or equivalent, containing no more than 1 ppmv oxygen, 1 ppmv total hydrocarbons, and 2 ppmv moisture and must not contribute more than 0.1 mg of residual mass per purge (Method 202, Section 7.1.5).

8.3 What are the Method 202 requirements for the impinger configuration and water level?

To ensure effective purging when using either purge method, you must submerge the tips of the impinger inserts so that they extend to at least one centimeter below the level of the accumulated water in all impingers being purged. If the water dropout impinger is included in train configuration for the purge, then you must replace the short stem insert with a modified Greenburg-Smith impinger insert. The longer stem purge insert must be cleaned to the same level as all other glassware. If additional water is needed to submerge the impinger insert tips, then you may add a measured amount of water that meets the method specifications. The

8.0 Best Practices for Post-Test Nitrogen Purge

added water must be degassed to remove dissolved oxygen by bubbling nitrogen through the water for at least 15 minutes before use. You must record the amount of water added.

8.4 What are the Method 202 requirements for the nitrogen purge operation?

For the post-test nitrogen purge, Method 202 has the following specifications (Method 202, Section 8.5.3):

- Measure and record the amount of water collected during sampling.
- Filter the nitrogen gas during the purge.
- Maintain the temperature of the condenser between 20 °C (68 °F) and 30 °C (85 °F) using the recirculation pump.
- Maintain the temperature of the gas exiting the silica gel impinger below 20 °C (68 °F) if the amount of water collected in the moisture trap has not been determined before the purge.
- Check the rotameter and orifice meter pressure differential ($\Delta H@$) values every 15 minutes.

8.5 What are the Method 202 requirements for performing a nitrogen purge using the meter box?

If you conduct the nitrogen purge with the sampling system meter box and vacuum pump, then you must conduct the purge on the CPM portion of the sampling train, from the inlet of the condenser, omitting the probe extension, through the exit of the silica gel impinger (Method 202, Section 8.5.3.2). Replace the short stem impinger insert in the water dropout impinger with a modified Greenburg-Smith impinger insert prior to the purge. Verify that the impinger insert tips are submerged below the water level in the impingers; degassed water may be added if needed. Begin the purge with no nitrogen flow and gradually increase the flow rate while simultaneously opening the meter box pump valve to avoid over- or under-pressurizing the sampling train. Maintain the nitrogen gas flow at 14 L/min with a positive overflow rate of less than 2 L/min. After the purge, you must simultaneously turn off the nitrogen flow and the vacuum pump without back-flushing the moisture impinger into the CPM filter housing.

8.6 What are the Method 202 requirements for performing a pressurized nitrogen purge?

Pressurized nitrogen purges may be conducted either by using the entire CPM train from the inlet of the condenser to the outlet of the CPM filter holder or by using only the back-up impinger and CPM filter holder (Method 202, Section 8.5.3.1). If you use the water dropout impinger for the purge, then you must replace the short stem impinger insert with a modified Greenburg-Smith impinger insert. If you use only the back-up impinger in the purge, then you must transfer the water collected in the condenser and water dropout impinger to the back-up impinger before the purge. You must verify that the impinger insert tips are submerged below the water level in the impingers; degassed water may be added if needed. Begin the pressurized nitrogen purge with no nitrogen gas flow and increase the flow gradually to avoid applying too much pressure to the sampling train.

8.7 What are the potential sources of residual mass contamination from the Method 202 post-test nitrogen purge?

The post-test nitrogen purge presents some possible sources of contamination. These sources include:

- The nitrogen gas used for purge.
- The materials needed to conduct the purge (e.g., tubing, pressure regulator).
- The modified Greenburg-Smith impinger insert (if used).
- Exposure to the environment while setting up for and performing the purge.
- The amount of time that the moisture collected during the sampling run is allowed to sit before the purge is conducted.

8.8 What are the EPA's best practice recommendations for limiting mass contribution during the Method 202 post-test nitrogen purge?

In addition to following the requirements of the method, the EPA recommends that you follow these best practices for conducting the post-test nitrogen purge:

- Handle the sampling train and purge equipment with care. Setting up the purge requires disconnecting components of the train and exposing the sample portion of the train to the environment. Connect sampling train components to the purge equipment or cap and cover them quickly to limit exposure. Be careful not to allow dust and debris to settle in or on the sampling train. Wear gloves to prevent dirt or oils from your skin from being deposited on the sampling train components.
- Use PTFE membrane or cellulose filters to filter the nitrogen gas during the purge. Do not use fiberglass filters, which can cause contamination from filter abrasion.
- Evaluate the nitrogen gas you plan to use for the purge to verify that it meets the Method 202 specifications, including the residual mass contribution limit of 0.1 mg. A nitrogen purge conducted using clean sampling train glassware that is then recovered and analyzed according to the procedures of Method 202 can serve as a "purge blank." Alternatively, a purge blank can be performed by passing the nitrogen purge gas through a tared CPM filter for the same amount of time and same volume of gas as is used for testing. Run a purge blank on every nitrogen gas cylinder to be used in Method 202 testing before deployment to the field.
- If you are purging the train using the sampling system meter box and you are using the modified Greenburg-Smith impinger insert, then prepare the insert according to the sampling train glassware cleaning procedures and best practices outlined in Sections 7.3 and 7.4 of this handbook. Rinse the insert with water, acetone, and hexane before conducting the purge.
- If you are purging the train by passing nitrogen through the train under pressure and you are using only the back-up impinger and CPM filter holder, then transfer the water collected in the condenser and water dropout impinger directly into the back-up impinger without the use of any additional containers. This eliminates the potential contamination that an additional container may contribute.

9.0 BEST PRACTICES FOR SAMPLE RECOVERY

This section identifies Method 202 requirements and presents the EPA's best practice recommendations for CPM sample recovery.

9.1 What are the Method 202 requirements for sample recovery?

Method 202 CPM samples are recovered by collecting the CPM filter, collecting the liquid from the CPM sample portion of the train into the aqueous sample container, rinsing the CPM sample portion of the train with water twice, collecting those rinses in the aqueous sample container, rinsing the train with acetone, collecting that rinse in the organic sample container, rinsing the train with hexane twice, and collecting those rinses in the organic sample container.

The CPM sample portion of the train consists of the back half of the filterable PM filter holder, the probe extension (if required for the filterable PM method), the condenser, the water dropout impinger, the back-up impinger, the front half of the CPM filter holder, the connecting glassware, and the CPM filter. The method requires sample recovery to be performed after the amount of water collected during the test run has been determined and the post-test nitrogen purge has been conducted. Method 202 specifies the following requirements for the materials used to recover samples:

- Deionized, ultra-filtered water must contain 1 ppmw (1 mg/L) residual mass or less (Method 202, Section 7.1.3).
- Acetone must be stored in glass and must contain 1 ppmw (0.791 mg/L) residual mass or less (Method 202, Section 7.2.1).
- Hexane must be American Chemical Society Grade and must contain 1 ppmw (0.655 mg/L) residual mass or less (Method 202, Section 7.2.2).
- Wash bottles must have a residual mass contribution of less than 0.1 mg and can be made of any material (Method 202, Section 6.2.2).
- Sample containers must be leak-proof and have a residual mass contribution of less than 0.05 mg (Method 202, Section 6.2.2).

9.2 What are the EPA's best practice recommendations for limiting mass contribution during Method 202 sample recovery procedures?

The sample recovery procedures present many opportunities for sample contamination. Stack testers must be diligent in following the method requirements to limit contamination to the greatest extent possible. The EPA recommends the following best practices to reduce the potential for contamination from external sources during sample recovery:

- Recover samples immediately, if possible, but no more than 30 minutes after the sampling run and the post-test nitrogen purge have been completed. Quickly recovering samples will ensure the most accurate results by limiting the amount of time that sampling trains are exposed to the environment of the site, thus reducing the amount of dust and debris that could bias the results.
- Set up the sample recovery area at a clean location or isolate it in a dust-free enclosure whenever possible. Set up the recovery area well away from the stack

9.0 Best Practices for Sample Recovery

and if possible, well away from the site itself if the source could be a significant source of contamination. Trailers work well as recovery areas because stack testers can actively take measures to prevent contamination.

- Clean the recovery area, including all surfaces, before recovering the samples.
- Cover the countertop where recovery is conducted with laboratory bench paper or cleaned aluminum foil.
- If possible, filter the ambient air of the sample recovery area using a HEPA filter.
- Limit access to the sample recovery area to only necessary staff to reduce the potential for contamination.
- Decontaminate the exterior of the sampling trains by wiping them down to remove any dust or debris. Wear gloves for this step, but discard them after the exterior decontamination is complete.
- Wear gloves during sample recovery. Gloves should be made of a material that has good compatibility with the reagents used in Method 202, such as nitrile. Latex gloves are soluble in acetone and should not be used. Rinse the gloves with deionized water before use. When wearing gloves, be careful to prevent transfer of contaminants from the surroundings.

The EPA recommends the following best practices for sample recovery procedures and the materials that come in direct contact with the samples:

- Determine the moisture of test runs by weighing the impingers rather than by using a graduated cylinder. This eliminates the need to transfer the contents of the moisture trap in sample jars and minimizes the potential for residual mass from additional containers and glassware.
- Use PTFE wash bottles. PTFE is compatible with the reagents used in Method 202 and does not have the leaching potential, that other polymers do for the same reagents. Triple rinse the bottles with the intended reagent and fill it prior to use. Do not store reagents in the wash bottles. Evaluate the wash bottles following the field reagent blank procedures to verify that the residual mass contribution is less than 0.1 mg.
- You may use glass capillary spigot rinse bottles as alternative wash bottles.
- Use only glass sample containers with PTFE lined lids for organic samples and reagent blanks. Wash and prepare the glass sample containers according to the sampling train glassware cleaning procedures and best practices outlined in Sections 7.3 and 7.4 of this handbook, including baking (do not bake the PTFE-lined lids).
- Rinse sample containers with water, acetone, and hexane, three times for each reagent before use.
- Evaluate sample containers using the field reagent blank procedures to verify that the residual mass contribution is less than 0.05 mg.
- When recovering samples, be careful to use adequate but not excessive amounts of reagents to minimize the potential contribution of residual mass to the samples by the reagents.
- If a probe extension is part of the CPM sampling train, stack testers can conduct the sample recovery for that component by sealing the inlet of the probe with an airtight fitting, adding the reagent, sealing the outlet with an airtight fitting, rotating the probe

9.0 Best Practices for Sample Recovery

end over end at least three times, and recovering the rinse in the appropriate sample container using a funnel. Repeat this process for each rinse specified in the method.

- Ensure that the brushes and any other supplies or materials that may be used for sample recovery are clean and protect them from contamination, particularly if they are used at the stack. Do not use any brushes or materials that have been used for the filterable PM recovery.
- Seal sample container lids using PTFE tape to avoid contamination from adhesive tapes (e.g., electrical tape).
- Optional: Chill aqueous sample bottles to 6 °C after recovery to reduce the rate of SO₂ conversion to sulfate artifact.

10.0 BEST PRACTICES FOR ANALYTICAL PROCEDURES

This section identifies Method 202 requirements for the analytical procedures and presents the EPA's best practice recommendations to prevent mass contribution from sample analysis procedures.

10.1 What analytical procedures are conducted in Method 202?

The Method 202 procedures for processing and analyzing CPM samples include moisture determination, CPM filter extraction, aqueous sample extraction, titration of the aqueous fraction (the titration step is not always necessary, see Method 202, Section 11.2.2.1), aqueous and organic sample evaporations, and weight determinations for the aqueous and organic sample fractions.

10.2 What are the Method 202 requirements for the analytical procedures?

The following requirements are specified in the method:

- All glassware used for the analytical procedures must be cleaned before use (Method 202, Section 8.2). This glassware includes:
 - Separatory funnel
 - Graduated cylinder
 - Beakers
 - Pipettes
 - Burette
- Sonicate the filter during extraction for a minimum of 2 minutes (Method 202, Section 11.2.1.1).
- Desiccate the weighing vessels with anhydrous calcium sulfate for 24 hours prior to gravimetric analysis (Method 202, Sections 11.2.2 and 11.2.3).
- The analytical balances must be capable of weighing 0.0001 g (0.1 mg).
- Calibration checks of the analytical balances must be performed with NIST traceable weights each day that CPM samples are measured, using reference weights that closely approximate to the combined weight of the sample and container (Method 202, Sec 9.4).
- The gravimetric analysis must achieve constant weight, which is defined as a change of 0.5 mg or less from the previous weighing with an interval of at least 6 hours.

10.3 What are the EPA's recommended best practices for the analytical procedures?

The EPA recommends the following best practices for implementing the Method 202 analytical procedures:

- Wash and prepare all of the laboratory glassware that is used to process the samples for analysis according to the sampling train glassware cleaning procedures and best practices outlined in Sections 7.3 and 7.4 of this handbook, including baking.

10.0 Best Practices for Analytical Procedures

- When performing aqueous sample evaporations the samples can be heated to expedite evaporation, but do not allow them to reduce below 10 mL in volume while heated. Allow the sample to cool and evaporate the sample to dryness at ambient temperature.
- When using an oven rather than hot plates for evaporation, do not heat the samples above 105 °C and check the samples frequently to avoid taking them to dryness under heated conditions.
- Use fluoropolymer beaker liners or other non-reactive low mass containers for sample weighing. Note that aluminum weighing tins have been observed to present a potential for oxidation.
- Set out empty tared weighing vessels to serve as dust blanks any time that the samples are exposed to the environment during the sample processing and analysis procedures.
- Use an analytical balance with a resolution of 0.00001 g (0.01 mg) for gravimetric analysis. The additional sensitivity is necessary to obtain accurate results for samples with very low mass, including blank checks.
- Maintain the temperature of the weighing room at 20 °C ± 3 °C (68 °F ± 5 °F).
- Maintain the relative humidity of the weighing room at 35 to 50 percent. The lower the relative humidity of the weighing room the greater the potential for static problems, particularly when using fluoropolymer beaker liners.
- Do not use balances with a weigh error of 0.05 percent or greater.
- Use an anti-static device during gravimetric analysis to prevent static from interfering with the analysis.
- Reduce the constant weight criteria to a change of 0.2 mg or less from the previous weighing.

NOTE: Appendices are not included in this file to limit file size.