



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

November 1, 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**

**U.S. Environmental Protection Agency
OAQPS/Sector Policies and Programs Division
Measurement Policy Group (D243-05)
Research Triangle Park, North Carolina 27711**

FOREWORD

This document provides EPA's responses to public comments on EPA's proposed revisions to Method 201A and 202 of 40 CFR Part 51, Appendix M. EPA published a Notice of Proposed Rulemaking in the Federal Register on March 25, 2009 (74 FR 12970). EPA received comments on this proposed rule via mail and e-mail. Copies of all comments submitted are available at the EPA Docket Center Public Reading Room. Comments letters are also available electronically through <http://www.regulations.gov> by searching Docket ID EPA-HQ-OAR-2008-0348.

This document provides the text of comments extracted from the original letters. For each comment, the name and affiliation of the commenter is provided, as well as the document control number (DCN) assigned to the comment letter. In some cases the same comment was submitted by two or more commenters. Rather than repeat these comment excerpts for each commenter, EPA has listed the comment excerpt only once and provided a list of the commenters who submitted the same comment.

The primary contact regarding questions or comments on this document is:

Ron Myers (919) 541-5407

U.S. Environmental Protection Agency
OAQPS/Sector Policies and Programs Division
Measurement Policy Group (D243-05)
Research Triangle Park, North Carolina 27711

myers.ron@epa.gov

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SECTION 1 INTRODUCTION & LIST OF COMMENTERS

On March 25, 2009, U.S. Environmental Protection Agency (EPA) proposed revisions to Methods 201A and 202 of 40 CFR Part 51, Appendix M (Recommended Test Methods for State Implementation Plans (SIPs)). The proposed amendments to Method 201A would add a particle-sizing device to allow for sampling of particulate matter (PM) with mean aerodynamic diameters less than or equal to 2.5 micrometers (μm) ($\text{PM}_{2.5}$ or fine PM). The proposed amendments to Method 202 would revise the sample collection and recovery procedures of the method to reduce the formation of reaction artifacts that could lead to inaccurate measurements of condensable particulate matter (CPM). Additionally, the proposed amendments to Method 202 would eliminate most of the hardware and analytical options in the existing method, thereby increasing the precision of the method and improving the consistency in the measurements obtained between source tests performed under different regulatory authorities.

This document contains summaries of the public comments that EPA received on the March 25, 2009 proposed amendments to Methods 201A and 202 and EPA's responses. This summary of public comments and EPA responses serves as the basis for revisions made between the March 25, 2009 proposed amendments and the subsequent promulgation of the amendments. Many commenters referred to EPA's Other Test Method (OTM) 027 and Conditional Test Method (CTM) 040, which were superseded by the proposed Method 201A, and OTM 028, which was superseded by proposed Method 202. In this document, the comments referring to OTM 027 and CTM 040 are addressed in the related sections of Method 201A and the comments referring to OTM 028 are addressed in the related sections of Method 202. EPA also received several comments regarding CTM 039 (Measurement of $\text{PM}_{2.5}$ and PM_{10} Emissions by Dilution Sampling (Constant Sampling Rate Procedures)). The CTM comments and EPA's responses are contained in the preamble to the final amendments.

1.1 LIST OF COMMENTERS

Table 1 lists the comment letters EPA received in Docket EPA-HQ-OAR-2008-0348 before the comment period closed on June 26, 2009. The table shows the commenter, affiliation,

and item number in Docket EPA-HQ-OAR-2008-0348. A list of acronyms and units of measure used in this document appears after the list of commenters.

**Table 1. Public Comments Contained in EPA-HQ-OAR-2008-0348
for Methods for Measurement of Filterable PM₁₀
and PM_{2.5} and Measurement of Condensable Particulate
Matter Emissions from Stationary Sources**

DCN	Commenter and Affiliate
EPA-HQ-OAR-2008-0348-0033	Comment submitted by J. Bruce Nemet, President, Resolution Analytics, Inc.
EPA-HQ-OAR-2008-0348-0033.1	Comment attachment submitted by J. Bruce Nemet, President, Resolution Analytics, Inc.
EPA-HQ-OAR-2008-0348-0034	Comment submitted by Lew Ballard and David Hendricks, Environmental Supply Co. Inc.
EPA-HQ-OAR-2008-0348-0034.1	Comment attachment submitted by Lew Ballard and David Hendricks, Environmental Supply Co. Inc.
EPA-HQ-OAR-2008-0348-0036	Comment submitted by Tom Gasloli, Air Quality Division (AQD), Michigan Department of Environmental Quality (MDEQ)
EPA-HQ-OAR-2008-0348-0037	Comment submitted by Tom Gasloli, Michigan Department of Environmental Quality (MDEQ)
EPA-HQ-OAR-2008-0348-0038	Comment submitted by Lee Carlson, Senior Research Associate, National Council for Air and Stream Improvement (NCASI) Southern Regional Center
EPA-HQ-OAR-2008-0348-0038.1	Comment attachment submitted by Lee Carlson, Senior Research Associate, National Council for Air and Stream Improvement (NCASI) Southern Regional Center
EPA-HQ-OAR-2008-0348-0039	Comment submitted by Michael Klein, Section Chief, Consultant Test Program, Bureau of Technical Services (BTS), New Jersey Department of Environmental Protection (NJDEP)
EPA-HQ-OAR-2008-0348-0039.1	Comment attachment submitted by Michael Klein, Section Chief, Consultant Test Program, Bureau of Technical Services (BTS), New Jersey Department of Environmental Protection (NJDEP)
EPA-HQ-OAR-2008-0348-0040	Comment submitted by Joe Jackson, AirNova, Inc.
EPA-HQ-OAR-2008-0348-0040.1	Comment attachment submitted by Joe Jackson, AirNova, Inc.
EPA-HQ-OAR-2008-0348-0040.2	Comment attachment submitted by Joe Jackson, AirNova, Inc.
EPA-HQ-OAR-2008-0348-0041	Comment submitted by Jim Serne, TRC Environmental Corporation
EPA-HQ-OAR-2008-0348-0041.1	Comment attachment submitted by Jim Serne, TRC Environmental Corporation
EPA-HQ-OAR-2008-0348-0042	Comment submitted by Terry Coughlin, We Energies, Pleasant Prairie Power Plant

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DCN	Commenter and Affiliate
EPA-HQ-OAR-2008-0348-0042.1	Comment attachment submitted by Terry Coughlin, We Energies, Pleasant Prairie Power Plant
EPA-HQ-OAR-2008-0348-0043	Comment submitted by Eric L. Hiser, Attorney, Jordan Bischoff & Hiser, PLC, on behalf of the Nucor Corporation
EPA-HQ-OAR-2008-0348-0043.1	Comment attachment submitted by Eric L. Hiser, Attorney, on behalf of the Nucor Corporation
EPA-HQ-OAR-2008-0348-0044	Comment submitted by Barry P. Boulianne, Senior Project Manager, BT Environmental Consulting, Inc. (BTEC)
EPA-HQ-OAR-2008-0348-0044.1	Comment attachment submitted by Barry P. Boulianne, Senior Project Manager, BT Environmental Consulting, Inc. (BTEC)
EPA-HQ-OAR-2008-0348-0045	Comment submitted by Naomi Goodman, Senior Project Manager, Electric Power Research Institute (EPRI)
EPA-HQ-OAR-2008-0348-0045.1	Comment attachment submitted by Naomi Goodman, Senior Project Manager, Electric Power Research Institute (EPRI)
EPA-HQ-OAR-2008-0348-0046	Comment submitted by Robin Edmiston-Bennett, ISB Environmental Network Leader, Roy Owens and Rick Cooper, Advanced Environmental Leads, Owens Corning Science and Technology, LLC
EPA-HQ-OAR-2008-0348-0046.1	Comment attachment submitted by Robin Edmiston-Bennett, ISB Environmental Network Leader, Roy Owens and Rick Cooper, Advanced Environmental Leads, Owens Corning Science and Technology, LLC
EPA-HQ-OAR-2008-0348-0047	Comment submitted by Catharine Fitzsimmons, Bureau Chief, State of Iowa, Department of Natural Resources
EPA-HQ-OAR-2008-0348-0047.1	Comment attachment submitted by Catharine Fitzsimmons, Bureau Chief, State of Iowa, Department of Natural Resources
EPA-HQ-OAR-2008-0348-0048	Comment submitted by Bruce Randall, Service Director, Emissions Testing, Performance, Remote and Emissions, and Andrew Dicke, Manager of Environmental and Acoustic Engineering, GE International, Inc.
EPA-HQ-OAR-2008-0348-0048.1	Comment attachment submitted by Bruce Randall, Service Director, Emissions Testing, Performance, Remote and Emissions, and Andrew Dicke, Manager of Environmental and Acoustic Engineering, GE International, Inc.
EPA-HQ-OAR-2008-0348-0049	Comment submitted by Lauren E. Freeman, Hunton & Williams LLP on behalf of the Utility Air Regulatory Group (UARG)

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DCN	Commenter and Affiliate
EPA-HQ-OAR-2008-0348-0049.1	Comment attachment submitted by Lauren E. Freeman, Hunton & Williams LLP on behalf of the Utility Air Regulatory Group (UARG) and their consultant RMB Consulting & Research, Inc. (RMB)
EPA-HQ-OAR-2008-0348-0050	Comment submitted by Filipa Rio, Manager, Environmental Affairs, Alliance of Automobile Manufacturers
EPA-HQ-OAR-2008-0348-0050.1	Comment attachment submitted by Filipa Rio, Manager, Environmental Affairs, Alliance of Automobile Manufacturers
EPA-HQ-OAR-2008-0348-0051	Comment submitted by Robert D. Bessette, President, Council of Industrial Boiler Owners (CIBO)
EPA-HQ-OAR-2008-0348-0051.1	Comment attachment submitted by Robert D. Bessette, President, Council of Industrial Boiler Owners (CIBO)
EPA-HQ-OAR-2008-0348-0052	Comment submitted by Leslie Sue Ritts, Counsel, National Environmental Development Association's Clean Air Project (NEDA/CAP)
EPA-HQ-OAR-2008-0348-0052.1	Comment attachment submitted by Leslie Sue Ritts, Counsel, National Environmental Development Association's Clean Air Project (NEDA/CAP)
EPA-HQ-OAR-2008-0348-0053	Comment submitted by Tyrone P. Wilson, Ph.D., Portland Cement Association (PCA)
EPA-HQ-OAR-2008-0348-0054	Comment submitted by Mark Petersen, Barr Engineering Company
EPA-HQ-OAR-2008-0348-0054.1	Comment attachment submitted by Mark Petersen, Barr Engineering Company
EPA-HQ-OAR-2008-0348-0055	Comment submitted by J. Roger Reehl, Senior Consultant and Billy R. Nichols, Senior Department Head, URS Corporation
EPA-HQ-OAR-2008-0348-0055.1	Comment attachment submitted by J. Roger Reehl, Senior Consultant and Billy R. Nichols, Senior Department Head, URS Corporation
EPA-HQ-OAR-2008-0348-0056	Comment submitted by Karin Ritter, Manager, Regulatory and Scientific Affairs, American Petroleum Institute (API)
EPA-HQ-OAR-2008-0348-0056.1	Comment attachment submitted by Karin Ritter, Manager, Regulatory and Scientific Affairs, American Petroleum Institute (API)
EPA-HQ-OAR-2008-0348-0057	Comment submitted by Thomas Schmelter, Project Manager, Health, Safety, and Environmental Services, Bureau Veritas North America, Inc.
EPA-HQ-OAR-2008-0348-0058	Comment submitted by Lawrence Odle, Director, Air Quality Department, Maricopa County

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DCN	Commenter and Affiliate
EPA-HQ-OAR-2008-0348-0058.1	Comment attachment submitted by Lawrence Odle, Director, Air Quality Department, Maricopa County
EPA-HQ-OAR-2008-0348-0059	Comment submitted by Paul M. Meeter, Air Quality Client Service Manager, Weston Solutions, Inc. (WESTON)
EPA-HQ-OAR-2008-0348-0059.1	Comment attachment submitted by Paul M. Meeter, Air Quality Client Service Manager, Weston Solutions, Inc. (WESTON)
EPA-HQ-OAR-2008-0348-0060	Comment submitted by Dean A. Kitchen, TETCO
EPA-HQ-OAR-2008-0348-0060.1	Comment attachment submitted by Dean A. Kitchen, TETCO
EPA-HQ-OAR-2008-0348-0061	Comment submitted by Tyrone Wilson, Director, Regulatory Affairs, Energy and Environment, Portland Cement Association (PCA)
EPA-HQ-OAR-2008-0348-0061.1	Comment attachment submitted by Tyrone Wilson, Director, Regulatory Affairs, Energy and Environment, Portland Cement Association (PCA)
EPA-HQ-OAR-2008-0348-0062	Comment submitted by Paul Cort, Staff Attorney, Earthjustice on behalf of Natural Resources Defense Council, et al.
EPA-HQ-OAR-2008-0348-0062.1	Comment attachment submitted by Paul Cort, Staff Attorney, Earthjustice on behalf of Natural Resources Defense Council, et al.
EPA-HQ-OAR-2008-0348-0063	Comment submitted by Kevin O'Halloren, Project Manager, Clean Air Engineering
EPA-HQ-OAR-2008-0348-0063.1	Comment attachment submitted by Kevin O'Halloren, Project Manager, Clean Air Engineering
EPA-HQ-OAR-2008-0348-0063.2	Comment attachment submitted by Kevin O'Halloren, Project Manager, Clean Air Engineering
EPA-HQ-OAR-2008-0348-0063.3	Comment attachment submitted by Kevin O'Halloren, Project Manager, Clean Air Engineering
EPA-HQ-OAR-2008-0348-0064	Comment submitted by Eric L. Hiser, Jorden Bischoff & Hiser, PLC on behalf of the Nucor Corporation
EPA-HQ-OAR-2008-0348-0064.1	Comment attachment submitted by Eric L. Hiser, Jorden Bischoff & Hiser, PLC on behalf of the Nucor Corporation
EPA-HQ-OAR-2008-0348-0065	Comment submitted by Peter M. Meeter, Air Quality Client Service Manager, Weston Solutions, Inc. (Weston)
EPA-HQ-OAR-2008-0348-0065.1	Comment attachment submitted by Peter M. Meeter, Air Quality Client Service Manager, Weston Solutions, Inc. (Weston)
EPA-HQ-OAR-2008-0348-0066	Comment submitted by Scott Evans, Clean Air Engineering
EPA-HQ-OAR-2008-0348-0066.1	Comment attachment submitted by Scott Evans, Clean Air Engineering

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DCN	Commenter and Affiliate
EPA-HQ-OAR-2008-0348-0067	Comment inadvertently included in this docket; was meant for EPA-HQ-OAR-2008-0334
EPA-HQ-OAR-2008-0348-0067.1	Comment attachment submitted by Jim Griffin, Senior Director, Regulatory and Technical Affairs, American Chemistry Council (ACC)
EPA-HQ-OAR-2008-0348-0067.2	Comment attachment submitted by Jim Griffin, Senior Director, Regulatory and Technical Affairs, American Chemistry Council (ACC)
EPA-HQ-OAR-2008-0348-0068	Anonymous public comment
EPA-HQ-OAR-2008-0348-0069	Comment submitted by Thomas Maza, Michigan Department of Environmental Quality (MDEQ)
EPA-HQ-OAR-2008-0348-0070	Comment submitted by Dean A. Kitchen, TETCO
EPA-HQ-OAR-2008-0348-0071	Comment submitted by J. Roger Reehl, Senior Consultant & Billy R. Nichols, P.E., Senior Department Head, URS Corporation
EPA-HQ-OAR-2008-0348-0072	Comment submitted by Robin Edmiston-Bennett, ISB Environmental Network Leader; Roy Owens and Rick-Cooper, Advanced Environmental Leads, Owens Corning Science And Technology, LLC
EPA-HQ-OAR-2008-0348-0073	Comment submitted by Barry R. Wallerstein, Executive Officer, Office of the Executive Director, South Coast Air Quality Management District (SCAQMD)
EPA-HQ-OAR-2008-0348-0074	Comment submitted by Kevin O'Halloren, Project Manager, Clean Air Engineering
EPA-HQ-OAR-2008-0348-0074.1	Comment attachment submitted by Kevin O'Halloren, Project Manager, Clean Air Engineering
EPA-HQ-OAR-2008-0348-0076	Comment submitted by Henry Hamanishi, P.E., Environmental Engineering Manager, J. R. Simplot Company
EPA-HQ-OAR-2008-0348-0077	Comment submitted by Barry R. Wallerstein, Executive Officer, South Coast Air Quality Management District (SCAQMD) (submitted as attachment for EPA-HQ-OAR-2008-0348-0073)
EPA-HQ-OAR-2008-0348-0078	Comment submitted by Stephen Anderson, Supervising Air Pollution Control Engineer, Source Emissions Monitoring, Connecticut Department of Environmental Protection (Connecticut DEP)
EPA-HQ-OAR-2008-0348-0079	Comment submitted by Maureen Barrett, Principle [sic], AERO Engineering Services
EPA-HQ-OAR-2008-0348-0079.1	Comment attachment submitted by Maureen Barrett, Principle [sic], AERO Engineering Services

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DCN	Commenter and Affiliate
EPA-HQ-OAR-2008-0348-0080	Comment submitted by Bruce Randall, Service Director, Emissions Testing, Performance, Remote and Emissions, and Andrew Dicke, Manager, Environmental and Acoustic Engineering, Global Projects Operation, Power Generation, GE International, Inc.
EPA-HQ-OAR-2008-0348-0080.1	Comment attachment submitted by Bruce Randall, Service Director, Emissions Testing, Performance, Remote and Emissions, and Andrew Dicke, Manager, Environmental and Acoustic Engineering, Global Projects Operation, Power Generation, GE International, Inc.
EPA-HQ-OAR-2008-0348-0081	Comment submitted by Anthony C. Sullivan, Barnes and Thornburg LLP
EPA-HQ-OAR-2008-0348-0081.1	Comment attachment submitted by Anthony C. Sullivan, Barnes and Thornburg LLP
EPA-HQ-OAR-2008-0348-0082	Comment submitted by Michael Pjetraj, P.E., Stationary Source Compliance Branch Supervisor, North Carolina Division of Air Quality (NCDAQ), North Carolina Department of Environment and Natural Resources (NCDENR)
EPA-HQ-OAR-2008-0348-0082.1	Comment attachment submitted by Michael Pjetraj, P.E., Stationary Source Compliance Branch Supervisor, North Carolina Division of Air Quality (NCDAQ), North Carolina Department of Environment and Natural Resources (NCDENR)
EPA-HQ-OAR-2008-0348-0083	Comment submitted by John Richards, Air Control Techniques, P.C.
EPA-HQ-OAR-2008-0348-0083.1	Comment attachment submitted by John Richards, Air Control Techniques, P.C.
EPA-HQ-OAR-2008-0348-0084	Comment submitted by Kevin J. Crosby, Technical Director, The Avogadro Group, LLC
EPA-HQ-OAR-2008-0348-0085	Comment submitted by Lawrence W. Kavanagh, Vice President, Environment and Technology Institute, American Iron and Steel Institute (AISI) and Bruce A. Steiner, President, American Coke and Coal Chemicals Institute (ACCCI)
EPA-HQ-OAR-2008-0348-0085.1	Comment attachment submitted by Lawrence W. Kavanagh, Vice President, Environment and Technology Institute, American Iron and Steel Institute (AISI) and Bruce A. Steiner, President, American Coke and Coal Chemicals Institute (ACCCI)
EPA-HQ-OAR-2008-0348-0086	Comment submitted by Karin Ritter, Manager, Regulatory and Scientific Affairs, American Petroleum Institute (API)
EPA-HQ-OAR-2008-0348-0086.1	Comment attachment submitted by Karin Ritter, Manager, Regulatory and Scientific Affairs, American Petroleum Institute (API)

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DCN	Commenter and Affiliate
EPA-HQ-OAR-2008-0348-0087	Comment submitted by Kurt A. Kissling, Pepper Hamilton LLP on behalf of Pilkington North America (PNA)
EPA-HQ-OAR-2008-0348-0087.1	Comment attachment submitted by Kurt A. Kissling, Pepper Hamilton LLP on behalf of Pilkington North America (PNA)
EPA-HQ-OAR-2008-0348-0088.1	Comment submitted by Randy Young, Weston Solutions
EPA-HQ-OAR-2008-0348-0089	Comment submitted by John W. Carroll, Pepper Hamilton, LLP, on behalf of Saint-Gobain Containers, Inc. (SGCI)
EPA-HQ-OAR-2008-0348-0090	Comment submitted by Connie Oldham, Ph.D., EPA Measurement Technology Group Leader
EPA-HQ-OAR-2008-0348-0091	Comment submitted by Stephen Page, Director, Office of Air Quality Planning and Standards
EPA-HQ-OAR-2008-0348-0093	Comment submitted by Jim Meador, Western Environmental Services and Testing, Inc.
EPA-HQ-OAR-2008-0348-0095.2	Comment submitted by Eric Hiser, Jordan Bischoff & Hiser, P.L.C. on behalf of Nucor Corporation
EPA-HQ-OAR-2008-0348-0096	Comment submitted by Jack Herbert, Oregon Department of Environmental Quality
EPA-HQ-OAR-2008-0348-0102	Comment submitted by Jace Shively, Avogadro Environmental Corporation
EPA-HQ-OAR-2008-0348-0103	Comment submitted by Thomas F. Mattei, Avogadro Environmental Corporation
EPA-HQ-OAR-2008-0348-0104	Comment submitted by Gregory Rock, GE Energy
EPA-HQ-OAR-2008-0348-0106	Comment submitted by Gammie Air Monitoring, LLC
EPA-HQ-OAR-2008-0348-0108	Comment submitted by Eric Grosjean, Atmospheric Analysis & Consulting, Inc
EPA-HQ-OAR-2008-0348-0110	Comment submitted by Randy Young, Weston Solutions
EPA-HQ-OAR-2008-0348-0111	Comment submitted by James A. Narens, Environmental Services Company, Inc.
EPA-HQ-OAR-2008-0348-0112	Comment submitted by Kris Hansen, AmTest Air Quality
EPA-HQ-OAR-2008-0348-0113	Comment submitted by Ned Shappley, METCO Environmental
EPA-HQ-OAR-2008-0348-0116	Comment submitted by James Christ, Airtech Environmental Services, Inc.
EPA-HQ-OAR-2008-0348-0117	Comment submitted by Arthur Werner, MACTEC
EPA-HQ-OAR-2008-0348-0118	Comment submitted by Joe Aldina, Covanta Energy

**Table 1. Public Comments Contained in EPA-HQ-OAR-2008-0348
for Methods for Measurement of Filterable PM₁₀
and PM_{2.5} and Measurement of Condensable Particulate
Matter Emissions from Stationary Sources**

DCN	Commenter and Affiliate
EPA-HQ-OAR-2008-0348-0119	Comment submitted by J. Bruce Nemet, President, Resolution Analytics, Inc.
EPA-HQ-OAR-2008-0348-0120	Comment submitted by Jeff Kaput, Airtech Environmental Services, Inc.
EPA-HQ-OAR-2008-0348-0121	Comment submitted by Angela Hansen, AmTest Air Quality
EPA-HQ-OAR-2008-0348-0122	Comment submitted by Paul Taverna, AECOM Environment

1.2 LIST OF ACRONYMS AND ABBREVIATIONS

ACC	American Chemistry Council
ACCCI	American Coke and Coal Chemicals Institute
AISI	American Iron and Steel Institute
API	American Petroleum Institute
AQD	Air Quality Division
AQMD	South Coast Air Quality Management District
BTEC	BT Environmental Consulting, Inc.
BTS	Bureau of Technical Services
C	Cunningham correction factor
CCM	Controlled Condensate Method
CIBO	Council of Industrial Boiler Owners
Connecticut DEP	Connecticut Department of Environmental Protection
CPM	condensable particulate matter
d	diameter
D ₅₀	Particle 50 percent cut diameter
DOT	Department of Transportation
DQOs	data quality objectives
dscf	dry standard cubic foot
dscm	dry standard cubic meter
EPA	U.S. Environmental Protection Agency
EPRI	Electric Power Research Institute
ERT	Electronic Reporting Tool
ESPs	electrostatic precipitators
FGD	flue gas desulfurization.
LOD	Limit of Detection
m _{cpm}	Total Mass of CPM
MDEQ	Michigan Department of Environmental Quality
MDLs	Method Detection Limits
MSHA	Mine Safety and Health Administration
M _w	Molecular weight of wet gas
MW	megawatts
NCASI	National Council for Air and Stream Improvement
NCDAQ	North Carolina Division of Air Quality

NCDENR	North Carolina Department of Environment and Natural Resources
ND	not detectable
NEDA/CAP	National Environmental Development Association's Clean Air Project
NIST	National Institute of Standards and Technology
NJDEP	New Jersey Department of Environmental Protection
N_{re}	Reynolds number
NTIS	National Technical Information Service
OSHA	Occupational Safety and Health Administration
OTM	Other Test Method
PCA	Portland Cement Association
PLQ	Practical Limit of Quantitation
PM	particulate matter
ppmw	parts per million by weight
P_s	Absolute stack gas pressure
Q_{IV}	Final sampling rate for cyclone IV
RCRA	Resource Conservation and Recovery Act
SCAQMD	South Coast Air Quality Management District
SEM	scanning electron microscope
SGCI	Saint Gobain Containers, Inc.
SI units	International System of Units
SIPs	State Implementation Plans
SO_3	sulfur trioxide
T_s	Absolute stack gas temperature
μ	Gas viscosity
μm	micrometer
UARG	Utility Air Regulatory Group
UHP	Ultra-high Purity
V_n	nozzle velocity
W_a	weight
WebFIRE	Web Factors Information and Retrieval - EPA's electronic emissions factors database
WinCIDRs	Windows-based Cascade Impactor Data Reduction System
μm	micrometers

SECTION 2.0

METHOD 201A - DETERMINATION OF PM₁₀ AND PM_{2.5} EMISSIONS FROM STATIONARY SOURCES (CONSTANT SAMPLING RATE PROCEDURE)

Section 2.0 of this document is organized by the section numbers of the proposed Method 201A.

M201A 1.1 SCOPE

Comment: (Comment No. 0056.1; Comment No. 0061.1) The American Petroleum Institute (API) and Portland Cement Association (PCA) support EPA's efforts to upgrade existing Method 201A by the addition of the PM_{2.5} cyclone. There is a clear need for a filterable PM_{2.5} emission test method to generate emissions data concerning the direct (primary) emissions of PM_{2.5} from stationary sources. Proposed Method 201A is a logical extension of OTM 027, which has been available on the EPA Emissions Measurement Center (EMC) website for several years. This method has previously been termed Method 201B2 and Proposed Method 4. The American Petroleum Institute and PCA understand that this method was originally based on the method summarized in the PCA Research and Development Publication SP2081 (Richards, J.R., "Test protocol: PCA PM₁₀/PM_{2.5} Emission Factor Chemical Characterization Testing," PCA R&D Serial No. 2081, Portland Cement Association, 1996), which in turn was based on a number of method iterations included in the draft publication prepared by Dawes and Farthing (Farthing and Dawes, "Application Guide for Source PM₁₀ Measurement with Constant Sampling Rate," EPA/600/3-88-057, U.S. Environmental Protection Agency, Research Triangle Park, N.C. 27711, 1988). The basic elements of this method have remained largely unchanged since 1996. This method has been used extensively by a number of organizations to simultaneously measure PM₁₀ and PM_{2.5}. The filterable PM_{2.5} data generated using this method are valid when the tests are conducted by qualified organizations adhering to the sampling procedures and quality assurance requirements specified in the method. PCA member companies have used this test method to compile the PM₁₀ and PM_{2.5} emissions data provided in the paper presented by Richards and Holder (Richards, J.T., Holder, and D. Goshow. "Optimized Method 202 Sampling Train to Minimize Biases Associated with Method 202

Measurement of Condensable Particulate Matter Emissions.” Paper presented at the Air and Waste Management Hazardous Waste Combustion Specialty Conference, St. Louis, Missouri, June 23-28, 1996). PCA believes that the filterable PM_{2.5} data generated using this method are valid when the tests are conducted by qualified individuals who are adhering to the sampling procedures and quality assurance requirements specified in the method.

Response: EPA agrees with the commenter’s assertion that the test method for measuring filterable PM_{2.5} emissions from stationary sources is needed and we appreciate their support for the addition of the PM_{2.5} cyclone to the Method 201A sampling train.

Comment: (Comment No. 0056.1) EPA should state more clearly that Proposed Method 201A can be used as a means to directly measure filterable coarse PM (PM₁₀ - PM_{2.5}). Coarse PM concentration is the difference between the measured PM₁₀ concentration and the measured PM_{2.5} concentration.

Response: EPA agrees with the commenter. Section 1.1 of Method 201A has been amended by adding the following sentence: “This method can be used to measure coarse particles (i.e., the difference between measured PM₁₀ concentration and the measured PM_{2.5} concentration).”

Comment: (Comment No. 0056.1) EPA should include guidance in proposed Method 201A concerning speciation of the constituents present in the PM₁₀, PM_{10-2.5}, and PM_{2.5} size fractions. This information should be provided to support the use of speciated PM₁₀, PM_{10-2.5}, and PM_{2.5} data in source apportionment studies.

Response: EPA did not revise the method to provide guidance for speciation of various particle fractions for source apportionment. EPA agrees with the commenter that with judicious selection of filter media and analytical finish, Method 201A could be used for speciating the less volatile metals. However, including details such as those in Method 29 to adapt this method for speciation analysis would introduce unnecessary complexity to this method without increasing the precision of the mass measurements. At this time, EPA believes that Appendix M is not a good choice for publication of guidance that may be misinterpreted and misused.

Comment: (Comment No. 0049.1) This technical review has clearly shown that CTM 40

has not been fully validated by EPA for the intended use. The method has application difficulties. Conditional Test Method 40 cannot be used on wet stacks, has undefined precision and will have very long run times on well-controlled sources. We do not believe that the method is suitable for use under the proposed rule. There are no technical references concerning the evaluation of CTM 40 cited in the proposed rule and only two rather oblique references in the method itself. There are no specifications for precision, accuracy or repeatability in the method so we expect these tests have never been done by EPA and the data do not exist. The lack of precision data is especially troubling because we have no idea whether two simultaneous sampling runs would obtain any level of agreement or what agreement should be expected.

Response: EPA agrees that Method 201A, which is based on CTM 40, is not applicable to source emissions containing water droplets as stated in Section 1.6 of the Method. (See additional comments and response in Section 1.6 Limitations of this document.) EPA is currently developing a method to measure PM in stacks with saturated water vapor and laboratory testing is ongoing. EPA has committed a significant budget and personnel to developing an acceptable method for sources with wet stacks and we plan to offer the method and protocol as soon as possible.

Regarding the precision of Method 201A, the precision uncertainty information available from a 1988 comparison between Method 201A and EPA Method 5 and 17 is still applicable because the filter media and support mechanisms are comparable. EPA evaluated the performance of Method 201A for PM₁₀ source measurements in 1988 and found the precision of the constant sampling rate method on which Method 201A is based to have precision of the same magnitude as Method 17 (5 percent relative difference) (PM₁₀ Source Measurement Methodology: Field Studies EPA 600/3-8-055; Docket item no. EPA-HQ-OAR-2008-0348-0027). EPA also found that the accuracy of the constant sampling rate PM₁₀ Method was -2 ± 4.4 percent compared to Method 17. EPA evaluated proposed Method 201A in a field test in 2009: *Field Evaluation of an Improved Method for Sampling and Analysis of Filterable and Condensable Particulate Matter* (see Docket no. EPA-HQ-OAR-2008-0348). Field test results show that the method meets Method 301 precision requirements for PM_{2.5}. Specifically, the precision resulting from 10 quadruplicate tests (40 test runs) was 7.9 percent Relative Standard Deviation, which meets the criteria for precision in Method 301. Thus, the method has been evaluated and found to be valid for the measurement of PM_{2.5}. The field test results demonstrate

that the revised method is comparable with the previous evaluation of Method 17 and the unrevised Method 201A requirements for measurement of size fractionated, filterable particulate. (See additional comments and response in Section 1.6 Limitations of this document). Regarding the length of run times, see additional comments and responses in this section.)

Comment: (Comment No. 0051.1) The Council on Industrial Boilers (CIBO) supports the Alliance suggestion for a general flexibility provision at the beginning of each method, as more fully described in the Alliance comments. Given the wide range of variables for any testing scenario, on-site operators need flexibility to ensure tests are accurate and meet all applicable methods and contain costs. The likelihood of increased complexity during field testing merits increased flexibility within Method 201A and Method 202 than is currently written.

One commenter (0050.1) stated that a general flexibility provision should be provided at the beginning of each method to respond to the experiences that will be acquired as the methods are field-applied.

Response: EPA did not revise the method to provide general flexibility because Method 201A produces method-defined results similar to Method 5 and Method 17. Both Method 201A and 202 have method-defined end points (i.e., the pollutant measured is defined by the test method's sampling and analytical procedures). In addition, the commenters did not suggest language that would provide the flexibility that they believe is needed and that would concurrently achieve the precision and accuracy that earlier stakeholders desired from this revision. In addition, EPA has learned through experience with Method 202 that general flexibility introduces imprecision and lack of confidence in the final method. EPA's alternative methods process provides a general flexibility that also incorporates a desirable measure of documentation needed for precision and confidence in the results.

EPA provided as much flexibility as possible for testers in the final method while still ensuring consistent results. One of the major changes in the methods was to eliminate optional procedures that were contained in the methods as originally promulgated. Removing the options results in more consistent measurements and improves the method's precision. In the final methods, we have also provided performance-based options that provide testers with some flexibility in obtaining PM measurements while ensuring that the methods will be applied consistently between different emission sources and between different testing contractors. For example, testers may choose to use analytical balances for particulate weight that have either 0.1 or 0.01 milligram (mg) sensitivity. Testers may choose between either baking the Method 202

glassware at 300 degrees Centigrade (°C) for 6 hours or conducting a field train proof blank to confirm their cleaning procedure results in acceptable residual mass contribution from glassware. Field train proof blanks are recovered on-site from a clean, fully-assembled sampling train prior to the first emissions test and provide the best indication of the lowest residual mass achievable by the tester. Field train recovery blanks are recovered from a sampling train after it has been used to collect emissions samples and has been rinsed in preparation for the second or third test in a series at a particular source. Field train recovery blanks include the additional uncertainty associated with how well the tester was able to clean the sampling train between test runs in the field. Therefore, we expect detection limits based on field train recovery blanks to be equal to or slightly greater than those determined using field train proof blanks.

Comment: (Comment No. 0058.1) As this is a technically demanding and complex method, an example calculation would be most helpful in understanding the application of the equations. Can you provide an example calculation showing the practical application of the equations for the stakeholders to aid them in achieving a thorough understanding of the concepts and procedures in the proposed method? This would serve as a useful instructional tool so that all parties can understand the practical application of the equations to a real-world example.

Response: Because affected facilities have a wide range of conditions (e.g., stack diameter, flow rate, moisture), it is not practical to provide a meaningful examples of the application of this method. EPA recommends that testers use available software to evaluate application of the equations for the conditions at their specific sources. Vendors of cyclone sets that meet the method requirements also provide software that allows testers to determine the necessary parameters for successful application of this method.

Comment: (Comment No. 0058.1) Can you supply stakeholders with an explanation of the origination of the equations in the proposed method and can you provide some specific references in the scientific literature that could help us understand the theoretical foundations of the method? A better understanding of the theoretical basis and origin of the equations will enable regulators to make informed decisions when evaluating the test results.

Response: The commenter should refer to National Technical Information Service (NTIS) Accession No. PB90247198, EPA Report No.: EPA 600/3-90/057 Application Guide for Measurement of PM_{2.5} at Stationary Sources (Docket item EPA-HQ-OAR-2008-0348-0002).

The guide contains the origination of the equations in the proposed method and specific references that will help with understanding the theoretical foundations of the method.

Comment: (Comment No. 0058.1) It is strongly recommended that a standardized data spreadsheet for use with the new Method 201A be provided by EPA for use by test companies and the regulatory agencies that will be reviewing the test results. This will ensure consistency among test companies in calculating the test results and will allow the regulatory agencies to evaluate the test results in the most efficient and consistent manner possible.

Response: EPA has taken steps to improve data consistency and data accessibility. EPA is providing the Electronic Reporting Tool (ERT), which is a product that replaces the paper source test reports, provides a consistent format, and uses primary recorded data to recalculate the test information each time the project data file is opened. The ERT was developed with input from stack testing companies that generally collect and compile performance test data electronically and offices within State and local agencies that perform field test assessments. The ERT is currently available, and includes calculations specified in the proposed Method 201A and 202. Future direct data submittal through EPA's Central Data Exchange for storage in EPA's electronic emissions database (WebFIRE) is expected to be available by December 31, 2011. One major advantage of submitting source test data with the ERT is that it provides a standardized method for test plan development and review, test report organization and calculation, test report submissions, test report review, and storage of completed reports. Another important benefit of submitting these data to EPA at the time the source test is conducted is that it has the potential to improve access to emissions test information and reduce the effort involved in data collection activities in the future.

To help ensure consistency among test companies in calculating the Method 201A and Method 202 test results, EPA also recommends that testers use available software to evaluate application of the equations for the conditions at their specific sources. Vendors of cyclone sets that meet the method requirements provide software that allows testers to determine the necessary parameters for successful application of Method 201A.

Comment: (Comment No. 0056.1) The commenter believes that EPA should specify the minimum solids catch weights needed in the PM₁₀ and PM_{2.5} size fractions. This information is

needed to help testing organizations determine the necessary sampling times. The commenter believes that at the sampling rates of 0.36 to 0.60 actual cubic feet per minute, adequate catch weights are obtained in these two size fractions in sampling runs of 2 to 4 hours.

(Comment No. 0050.1) Proposed Method 201A (74 FR 12980, Section 1.7) Conditions, states the following: "...you must extend the sampling time so that you collect the minimum mass necessary for weighing on each portion of this sampling train" and have the necessary sampling duration to obtain sufficient particulate catch weights. A reasonable limit must be put on sampling volume to contain potential unnecessary sampling time and exorbitant stack testing costs that could quickly escalate with such a requirement. The Alliance recommends the language be changed to the following: "Additional sample volume may be needed to collect enough mass necessary for weighting of each portion of this sampling train. If after collecting at least 90 cubic feet of sample, there is no measurable mass as compared to the blanks, the result should be reported as less than detection limit."

(Comment No. 0057) Regarding Section 8.3.4(c): The necessary sampling duration to obtain sufficient particulate catch weights, please define the minimum sample catch requirement for each particle size, greater than 10 μm , less than 10 μm but greater than 2.5 μm , and less than 2.5 μm .

(Comment No. 0048.1) Another commenter asked EPA to define "sufficient catch weights." Some states specify that sample times should be selected such that 50 mg would be collected based on the permitted emission limit. We recommend determining sample times such that 10 mg would be collected, based on the permitted emission limit.

(Comment No. 0076) Our source testers have also indicated that to obtain representative samples, each test may need to be increased as much as three times the current one hour test period.

(Comment No. 0057) Section 1.7 Conditions: "Further, to use this method in place of Method 5 or Method 17, you must extend the sampling time so that you collect the minimum mass necessary for weighing on each portion of the sampling train." Can you please provide guidance on how to calculate the sample time based on permit limits or established emission factors?

(Comment No. 0049.1) Even with dry stacks, we are concerned that the run time will be excessive on well controlled sources. During a series of tests sponsored by the RMC Research

Foundation in late 2003 and early 2004, Conditional Method 40 was used at the baghouse outlet of a ready mix loading operation. The total catch in the sample train was from 1.0 to 2.3 mg and was judged too low to make meaningful measurements because of weighing errors. It should be noted that the low catch included the PM₁₀ catch and not just the PM_{2.5}. This experience is consistent with past attempts to use particle sizing cyclones and impactors where 6 to 12 hour sampling times were required to obtain enough sample to weigh accurately.

Response: We agree with the commenters that collecting sufficient weighable mass is important for the method to be precise. We also understand that the sampling rate used to attain the cyclone cut-points is typically less than the rate used during Method 5 sampling. However, EPA did not revise the method to dictate a minimum sampling volume or minimum catch weight that would be necessary to obtain a valid sample. One reason for not specifying a minimum sampling volume or minimum catch weight is that different regulatory authorities and testing programs have differing measurement goals. For example, some regulatory authorities will accept less precision if results are well below compliance limits. State agencies or individual regulated facilities may develop data quality objectives (DQOs) for the test program, which may specify minimum detection limits and/or minimum sample volume and/or catch weight that would demonstrate that DQOs can be met. State agencies should consider the measurement capabilities, their program's data quality objectives, and the estimated sample durations when establishing emissions limitations. Stack samplers should take into consideration the compliance limits set by their regulatory authority and determine the minimum amount of stack gas needed to show compliance if the mass of particulate is below the detection limit.

Regarding stack gas volume, stack testers can use the minimum detection limit to determine the minimum stack gas volume. The stack tester may be able to estimate the necessary stack gas volume based on how much PM the source or source category is expected to emit (which could be determined from a previous test or from knowledge of the emissions for that source category). Alternatively, the minimum detection limit for a source can be determined by calculating the percent relative standard deviation for a series of field train recovery blanks. You will not be able to measure below the average train blank level, and EPA recommends calculating a source-specific detection limit by multiplying the standard deviation of field train recovery blanks by the appropriate 99 percent critical Student's $t_{(.99)}$ value (e.g., for seven field train recovery blanks, the standard deviation of the results would be multiplied by three).

Each stack tester will have different abilities as indicated by the results of their proof or field blank sample analyses. Proof train blanks result from on-site recovery of a clean, fully-assembled sampling train prior to conducting the first emission test. Proof train blanks provide the best indication of the lowest blank achievable by the tester because the only source of

residual mass in the blank sample is the clean sampling train. Historical proof train blanks provide a dataset to statistically determine the lowest mass that can be distinguished from zero (i.e., the tester's detection limit for this method). An alternative method detection limit may be determined from field train blanks that are recovered from sampling trains after they are used to collect emission samples and have been rinsed in preparation for the second or third test in a series at a particular source. Field train blanks (resulting from on-site recovery of a fully-assembled sampling train used for at least one emission test) include the additional uncertainty associated with how well the tester was able to clean the sampling train between test runs in the field. Therefore, we expect detection limits based on field train blanks to be equal to or slightly greater than those determined using proof train blanks. (See additional comments and response in this section regarding long sampling times, minimum catch weights, and sampling volume.)

An estimated detection limit was determined from an EPA field evaluation of proposed Method 201A (see *Field Evaluation of an Improved Method for Sampling and Analysis of Filterable and Condensable Particulate Matter*, Docket no. EPA-HQ-OAR-2008-0348). The estimated detection limit was calculated from the standard deviation of the differences from 10 quadruplicate sampling runs multiplied by the appropriate Students T value ($n-1 = 9$). Detection limits determined in this manner were (1) total filterable PM: 2.54 mg, (2) PM₁₀: 1.44 mg, and (3) PM_{2.5}: 1.35 mg. These tests showed more filterable particulate in the PM_{2.5} fraction and total filterable particulate detection limits may be biased high due to the small particulate mass collected in the fraction greater than PM₁₀. To achieve consistent results, significant care was made to minimize contamination.

Comment: (Comment No. 0049.1) Although the Utility Air Regulatory Group (UARG) believes that these proposed revisions are useful and should be finalized, UARG does not believe that these revisions are sufficient to provide accurate measurement of PM_{2.5} emissions at a large portion of electric generating facilities.

Response: The commenter did not provide enough information to determine why the proposed method would not work at a large number of electric generating facilities. Well-controlled emissions from electric generating facilities may require longer sampling times to meet the minimum regulatory requirements and/or allow reporting of non-detectable particulate emissions using this method. Facilities should work with their regulatory authorities to determine the minimum stack volume that must be sampled to demonstrate that a facility is in compliance with applicable permits and regulations.

Comment: (Comment No. 0044.1) EPA should clearly state what happens to OTM 27.

We are assuming that this OTM method will no longer be used, and would be deleted from the record.

Response: The commenter is correct. After the final rule and method revisions are approved, any references to CTM 40, OTM 27, or OTM 28 will direct users to the promulgated Method 201A or Method 202.

M201A 1.2 APPLICABILITY

Comment: (Comment No. 0041.1) EPA should encourage agencies to use common sense when judging the acceptability of Method 201A test results at sources where wide variations in gas velocity and temperature occur during the test run. It will not always be feasible for all of the Method 201A QA acceptance criteria to be met as a result of highly variable gas velocity and or temperature. Sampling for PM₁₀ and PM_{2.5} can be especially challenging.

Response: We agree with the commenter that air regulatory agencies should consider applicability criteria carefully when reviewing the Method 201A results. We also believe the limitations of the method are clearly specified, and thus, no change to the method is needed to address the commenter's concerns. We recognize that there is a limit to the variation in stack velocity that can be measured with one nozzle size and still meet the isokinetic sampling requirements of the method. The possible variations in the source emissions that may be tested makes providing detailed requirements for every test condition difficult. Thus, testers are encouraged to provide in their test plans a detailed assessment of the challenges they expect, as well as the methods they plan to employ to address those challenges and uncertainties in the measurements. The testers are further advised that regulatory authorities' acceptance of the proposed methods to address the challenges should be obtained prior to the test date. In addition, the quality assurance section of the test report should discuss the different conditions that were encountered, the success in achieving the objectives of the test, and the reliability of the emissions that were measured.

Comment: (Comment No. 0056.1) The commenter recognized that some stationary sources have PM loadings that are too low to allow for the practical use of proposed Method 201A. For example, many gas turbines and most gas-fired combustion sources have very low

PM loadings, and run durations of 4 hours provide only minimal solids. The precision of proposed Method 201A is less than adequate for these especially low PM concentration sources. In these cases, more accurate data can be obtained by Method 5. The limitations of proposed Method 201A for low PM concentration sources should be discussed in the method.

(Comment No. 0048.1) The proposed revisions to Methods 201A and 202 for the measurement of PM, PM less than 10 μm in diameter (PM_{10}) and PM less than 2.5 μm in diameter ($\text{PM}_{2.5}$) are intended to provide the most accurate representation of primary PM emissions. The existing methods provide insufficient accuracy and precision for use on low PM emission sources, and the proposed revisions, in our opinion, will not significantly impact this deficiency. Method 201A will fundamentally use the same gravimetric method of analysis and will retain all the associated sources of errors. Although the dry Method 202 will likely reduce artifacts that can lead to erroneously high PM test results from certain sources categories, the proposed method will fundamentally use the same impinger and gravimetric method of analysis as the present wet Method 202, and will retain all the other associated sources of errors. Our fundamental concern is that the detection limits of these methods are not low enough to accurately measure very low levels of PM emissions, and their use will continue to contribute measurement noise in PM results even with the proposed revisions. Additionally 1) the added complexity of the new equipment and procedures will introduce opportunities for both negative and positive error, and, 2) being highly manual methods, there will be a learning curve for all test firms to become familiar with the equipment and procedures, again adding opportunity for both negative and positive error. It is our opinion that Method 201A and 202 in their existing form, or even with the improvements suggested, are inadequate and inappropriate for the measurement of very low emission sources; nor are they appropriate to support setting permit limits to implement particulate standards or ensure fair enforcement of those standards. We strongly urge that EPA expedite development of new fundamentally superior methods capable of measuring low emission sources.

Response: EPA recognizes that the proposed revisions to Method 201A for filterable particulate and Method 202 do not reduce the existing fundamental complexity associated with particle-sizing test procedures, which are exacerbated for fine PM. Well-controlled emissions from various sources may require longer sampling times to meet detection limit challenges. EPA recommends facilities work with their regulatory authorities to determine the minimum stack

volume that must be sampled to demonstrate that a facility is in compliance with applicable permits and regulations. State agencies and individual regulated facilities should develop data quality objectives (DQOs) for the regulatory demonstration program, which would include the achievable detection limits, expected minimum sample volume, desired particulate catch weight, and sample durations that would demonstrate that DQOs can be met.

EPA encourages advances in PM quantification and characterization, and also recognizes the need for PM test methods that offer improved detection limit capabilities and improved precision at low concentrations. EPA is aware that an ASTM-approved advanced particulate source test method for total filterable PM was being modified for quantifying PM₁₀ and PM_{2.5} but was not developed into a marketable product because of a lack of demand. EPA evaluated the use of the tapered element oscillating micro-balance (TEOM) technology for quantifying low concentrations of total filterable PM and determined that this technology met Method 301 criteria and was suitable for approval as an alternative for Method 5 or 17 for a wide range of sources. EPA believes that with the development of a viable market, the manufacturer of this technology will reintroduce this technology into the market and continue the development of this product for particle sizing. It is likely that when modified, this method could satisfy Method 301 validation for PM₁₀ or PM_{2.5} particle sizing.

M201A 1.5 ADDITIONAL METHODS

Comment: (Comment No. 0039.1) One commenter stated that Section 1.5 speaks of correcting results for ambient air contributions. The commenter stated that this section should be removed. The facility is responsible for what is emitted from the stack and must take this into account when establishing their allowables. This section is ripe for abuse as a means to blame non-compliance on ambient contributions and will result in legal challenges and disputes of test results.

(Comment No. 0048.1) Another commenter recommended that the sentence addressing correction of results be clarified. The commenter questioned whether it was the intent of the EPA not to allow the use of condensable particulate for low-temperature sources. The commenter also questioned if the process/method for making this adjustment was defined.

Response: We agree with the commenters that this section of the method was unclear. Therefore, Section 1.5 (Additional Methods) has been removed from the final method. The

decision to correct results for ambient air contributions is up to the permitting or regulatory authority and no further changes to the method are necessary. For sources that have very low PM emissions, such as processes that burn clean fuels (e.g., natural gas) and/or use large volumes of dilution air (e.g., gas turbines and thermal oxidizers), any ambient air particulate introduced into the process operation could be a large component of total outlet PM emissions.

M201A 1.6 LIMITATIONS

Comment: (Comment No. 0047.1) The Indiana Department of Natural Resources (IDNR) supports EPA's recommendation to use Method 5 to determine PM₁₀/PM_{2.5} filterable mass when measuring emissions following a wet scrubber. This procedure shares similarities with the IDNR's policy of equating all PM emissions to PM₁₀ if the source is unsuitable for Method 201A. Stating the procedure fairly explicitly in the method is beneficial.

(Comment No. 0046.1) Another commenter said that their experience when conducting emission testing at their facilities with similar stack conditions as described herein supports the EPA's position on the limitations of the proposed Method 201A testing protocol.

Response: EPA acknowledges and appreciates the commenters' support.

Comment: (Comment No. 0046.1) Is there any ongoing program to develop a particle-sizing method for sources with water droplets? The statement in the preamble refers to Technical Information Document 09 for determining PM₁₀ Emissions for sources with water droplets. It appears to require that we assume everything is PM₁₀. With all the current emphasis on PM_{2.5}, does EPA plan to offer any new guidance and if so when?

(Comment No. 0064.1) In its proposed Method 201A for direct measurement of filterable PM₁₀ and PM_{2.5}, EPA has stated that the method may not be suitable for saturated gas streams, and those streams containing water vapor, as would exist downstream of a wet scrubber (Section 4.0). Wet scrubbers are often utilized as the primary emission control device for sulfur dioxide (SO₂). For such sources, the revised Reference Method 201A disqualifies itself, due to its inherent design, for sampling wet scrubber effluent streams. It seems that suitable alternatives should have been developed to handle wet scrubber effluent streams, prior to proposing these modified methods given the research studies funded to date by EPA to address these issues.

(Comment No. 0049.1) Section 1.7 of Conditional Method 40 explicitly states that the method cannot be used on units with a wet scrubber that results in a stack gas containing liquid droplets. The reason for this limitation is that the method utilizes two cyclones to size the

particles and the gas entering the cyclones must be dry for the cyclones to work properly. Therefore, this method cannot be used on most scrubber-equipped units.

(Comment No. 0049.1) UARG urges EPA to continue its work to identify a method for measuring filterable (or total) PM at sources with entrained moisture droplets in the stack (e.g., units with wet stacks due to wet flue gas desulfurization (FGD)).

(Comment No. 0056.1) EPA has not developed a commercially ready test method for “wet stacks.” EPA has clearly stated that proposed Method 201A should not be used at emission sources that have entrained water droplets. API agrees with this limitation of the method. API is not satisfied with the use of Method 5 as the only acceptable method for sources with entrained water droplets. To provide more accurate emissions data for sources with “wet” stacks, API is sponsoring the development of an advanced manual sampling technique that can accurately measure filterable PM_{2.5} in stacks with entrained water droplets. API will complete field tests of this method during the next two months. API will share laboratory and field test evaluations of this new method. API believes that this new method for filterable PM_{2.5} emissions in “wet” stacks will be highly compatible with proposed Method 201A for filterable PM_{2.5} emission testing in “dry” stacks.

API maintains and EPA should acknowledge that there currently is not an acceptable method for the determination of PM_{2.5} primary and CPM in “wet stacks,” but API looks forward to sharing data from the advanced manual sampling technique we are currently developing for use in “wet stacks” in the near future.

(Comment No. 0049.1) UARG commenter stated that, at least in theory, the addition of a sizing device (i.e., cyclone) with a 2.5 µm cut-point is straightforward. However, as EPA notes in Section 1.6 of Method 201A, “you cannot use this method to measure emissions following a wet scrubber because this method is not applicable for in-stack gases containing water droplets.” UARG reminded EPA’s Measurement Group that approximately 175,000 megawatts (MW) of the electric utility 325,000 MW capacity is scrubbed. Because of cap-and-trade incentives, the scrubbed units tend to be larger and more utilized units. Thus, not only a significant percentage of the coal-fired capacity, but many highly dispatched units generating units, have no reference method for measuring filterable PM emissions by size fraction. UARG understands that this a non-trivial task; nevertheless, EPA needs to pursue expeditiously the development of such a reference method.

(Comment No. 0076) Another commenter stated that both fryer and dryer stacks have emissions that include very high moisture content. According to the commenter’s current third party source testers, performing in-stack cyclone separators and filters cannot be done on wet sources. Therefore, none of the commenter’s fryer and dryer stacks could be tested for PM₁₀ or PM_{2.5}, but would have to test for total particulate using Method 5, which could highly overstate

the amount of PM₁₀ and/or PM_{2.5}. EPA needs to state a viable alternative to the current proposed rule for our high moisture contact stacks before this rule is final.

Response: EPA is currently developing a method to measure PM in stacks with saturated water vapor and laboratory testing is ongoing. EPA has committed a significant budget and personnel to developing an acceptable method for sources with wet stacks, and we plan to offer the method and protocol as soon as possible. EPA's method development and evaluation is focused on the "Dried Particle Method" (See *Lab Work to Evaluate PM_{2.5} Collection With a Dilution Monitoring Device for Data Gathering for Emission Factor Development*; EPA-HQ-OAR-2008-0348) that directly measures the mass emission rate of particles with specified aerodynamic size. In the meantime, the promulgated amendments to Methods 201A and 202 improve their performance and reduce known artifacts. Testers should use these final, amended methods until a PM_{2.5} method for stack gases containing water droplets is promulgated.

In addition, regarding the need to increase test times, see the comments and response in Section 1.0 Scope. EPA agrees that increased sampling time is necessary to collect sufficient stack gas to adequately represent emissions on a dry standard volume basis. However, this is true for all sources in existing regulations and for State Implementation Plans (SIPs). See previous response on minimum detection limits and the need to work with regulatory authorities to determine how long to sample to meet compliance requirements.

Comment: (Comment No. 0064.1) The commenter noted that EPA states that "over time, the changes in the test methods proposed in this action will result in ... more accurate emissions inventories of direct PM emissions" (74 FR 12975). EPA concedes, however, that Method 201A is not usable for stacks with entrained moisture or high temperature stacks (74 FR 12974). Instead, EPA recommends using Method 5 and assuming "all of the collected material" is PM_{2.5}. EPA has provided no rationale in support of its conclusion that all of the collected material is PM_{2.5}. Further, this assumption provides erroneous information to the agencies, researchers, and the public, which will consider the entire PM levels detected by Method 5 to consist of PM_{2.5} with the correspondingly greater potential health threat that the finer PM may pose. Indeed, EPA itself agreed that Method 5 should not be used for this purpose, when it observed that Method 5 does not provide a reasonable estimate of PM₁₀ emissions and will provide a correspondingly poorer estimate of PM_{2.5} emissions (74 FR 12976).

(Comment No. 0049.1) There is one "recommendation" in the preamble regarding the

application of Method 201A that merits discussion. For stacks containing entrained water droplets (i.e., stacks where M201A is not applicable), EPA recommends using Method 5 and considering all the collected mass to be PM_{2.5}. UARG disagrees with what we consider a rather cavalier recommendation. For well-controlled coal-fired utility boilers, filterable PM_{2.5} is a small fraction (e.g., 30 to 50 percent) of the Method 5 mass. In fact, EPA's *Compilation of Air Pollutant Emission Factors* (AP-42) document tabulates the cumulative PM mass percent less than 2.5 gm for dry bottom boilers burning pulverized coal to be 29 percent for units with electrostatic precipitators (ESPs) and 53 percent for units with a baghouses. UARG believes EPA should withdraw its recommendation to use Method 5 in lieu of a specific PM_{2.5} methodology. Failing that, at a minimum the Agency should warn sources how large of a potential bias in PM_{2.5} emissions could result from following the Method 5 recommendation.

Although the commenter and EPA are not aware of any commercially available technology that can measure filterable (or total) PM in wet stacks, the lack of other methods is not sufficient reason to ignore the significant overstatement of emissions that would result from EPA's proposed policy. EPA should withdraw its proposed recommendation regarding measurement of filterable PM_{2.5} at units with wet stacks and develop a policy that either avoids the need for such a measurement or takes the overstatement into account. Such data should not be used for emission inventories without appropriate adjustment, and should only be used for compliance determinations if the emission limit was established using the same assumption (i.e., was established at a level consistent with the entire Method 5 catch).

(Comment No. 0086.1) API stated its objection to the use of Method 5 to measure total filterable PM emissions as a surrogate for filterable PM_{2.5} emissions. API has taken a constructive approach to addressing this issue by sponsoring the development of an advanced manual sampling technique that can accurately measure filterable PM_{2.5} in stacks with entrained water droplets. API appreciates the interest and time on the part of Mr. Dan Bivens, Mr. Ron Myers, and Mr. Jason DeWees to visit API's contractor, Air Control Techniques, P.C., on June 10, 2009 to evaluate the API prototype sampling system.

API reviewers of Docket ID EPA-HQ-OAR-2008-0348 found that a number of organizations have called for the development of a method to measure filterable PM_{2.5} in stacks with entrained water droplets. These organizations (Commenters 0038.1, 0064, 0076.1) agree with API's position that the categorization of all PM measured by U.S. EPA Method 5 as PM_{2.5}

overstates the true emissions. Only the INDR provided comments in support of the use of Method 5 data as a surrogate for PM_{2.5} and/or PM₁₀.

API suspects that the acceptance of Method 5 total filterable PM emissions data as a surrogate for PM_{2.5} filterable data has been necessitated previously by the lack of an appropriate test method to provide PM₁₀ and/or PM_{2.5} data in wet stacks. API believes that the new API sampling system being developed will satisfy the long-standing need for a wet stack filterable PM_{2.5} test method. API welcomes EPA's comments on the API method development program and requests expeditious approval of this new test method. As discussed above in Comment 1 with respect to the Transition Period, there will be very limited time to develop PM_{2.5} emissions inventories and control strategies. This new method is needed soon.

(Comment No. 0038.1) One commenter stated that another area of concern is the continuing lack of a test method for PM₁₀ or PM_{2.5} for sources with entrained water droplets. EPA's recommendation to use Method 5 on wet sources and assume the result is all PM_{2.5} unfairly penalizes these sources in terms of their contribution to ambient PM_{2.5} concentrations. EPA should accelerate its search for acceptable PM₁₀ and PM_{2.5} stack testing methods for wet sources.

(Comment No. 0056.1) API is not satisfied with the use of Method 5 as the only acceptable method for sources with entrained water droplets. When Method 5 is used, all of the PM is artificially classified as PM_{2.5}. In many emission sources this administrative procedure results in a significant overstatement of the actual primary PM_{2.5} emissions.

(Comment No. 0089.1) Saint Gobain Containers, Incorporated (SGCI), noted that, for stacks containing moisture, EPA recommended using Method 5 and treating 100 percent of the total filterable catch as PM_{2.5} (74 FR 12974-75). SGCI disagrees with this recommendation to the extent that it conflicts with and ignores the particle size distribution data set forth in AP-42 specific to glass melting furnaces. Therefore, SGCI would propose revising Section II(C)(1) that for industries with particle size distribution data defined in AP-42, the percentage of PM_{2.5} as established in AP-42 should be used in conjunction with Method 5 on hot stacks rather than treating the entire catch as PM_{2.5} as indicated below: "To measure PM₁₀ in stacks where water droplets are known to exist or where the stack gas exceeds 260°C (500°F), EPA recommends use of Method 5 of Appendix A-3 to 40 CFR Part 60 (or a comparable method) and consideration of the total particulate catch as PM₁₀ emissions, provided that the particle distributions set forth in

for a source category in AP-42 may be used in lieu of treating the entire catch as PM₁₀ (74 FR 12973, col. 2).” A similar statement appears in Section III(A) of the preamble (Id. at 12974-75), which should be revised in the same way. Industry has relied on these particle size distributions in the past and should be able to continue to do so in the context of Method 201A.

Response: EPA acknowledges that using Method 5 on stacks with entrained moisture and assuming that the catch is PM_{2.5} can potentially overestimate filterable PM_{2.5} concentrations. EPA Method 5 measures total filterable PM mass emissions from stationary sources. Method 5 does not specifically isolate PM₁₀ or PM_{2.5}. Method 17, similar to Method 5, measures total PM mass emissions, but it uses an in-stack filter operating at stack temperature instead of a heated probe and out-of-stack heated filter and thus is suitable only for dry sources. Water drops in stack gas streams typically originate from three sources: (1) condensation on particles within the stream if the stream is at or near saturation conditions (these droplets are generally smaller than 20 µm in diameter); (2) small water droplets that penetrate the mist eliminator of a scrubber (these particles are also generally smaller than 20 µm in diameter); and (3) large droplets formed by spray nozzles or mechanically entrained from mist eliminator surfaces in scrubbers and from wet structural and duct surfaces (these droplets are typically tens to hundreds of µm in diameter). (See *Development of Particle Size Test Methods for Sampling High Temperature and High Moisture Sources*, California Environmental Protection Agency, Air Resources Board Research Division, 1994; Docket EPA-HQ-OAR-2008-0348).

Monitoring the emission of PM_{2.5} and PM₁₀ emissions from a wet gas stream is a challenging problem that has not been addressed successfully despite considerable effort. A consensus method to provide this information has not emerged. EPA has determined that particulate from wet stacks is expected to be less than PM₁₀ under most test conditions of wet scrubbers. University of North Carolina particle physicists performed theoretical calculations based on a wet scrubber operating at 10,000 parts per million by weight (ppmw) total dissolved solids (TDS) with water droplets up to 50 µm in size (see *Development of Plans for Monitoring Emissions of PM_{2.5} and PM₁₀ from Stationary Sources With Wet Stacks*; Docket EPA-HQ-OAR-2008-0348). They determined that water droplets under these conditions, when dried, would generate particles of 10 µm or smaller. Using the same theoretical basis (i.e., the ratio of TDS to water droplet size), water droplets up to 10 µm in size would generate dried particles of 2 µm or less and water droplets up to 20 µm would be expected to generate dried particles up to 4 µm or

lessr.

Local regulatory agency authorities may allow Method 5 to be used in lieu of particle sizing in stacks containing entrained water droplets. In these cases, regulatory agencies should consider the limitations of such an approach and understand that the assumptions they make regarding the particle size distribution from samples collected in stacks with entrained water droplets may require emissions limitations consistent with the total filterable PM measured by Method 5 combined with CPM measured by Method 202. This approach for measurement of filterable and condensable particulate from stacks containing entrained water droplets should only be used as a stop-gap measure until the Agency develops or approves a wet stack particle sizing method that is scientifically defensible.

For the reasons stated in this response, EPA disagrees that using Method 5 on wet sources and assuming the result is an appropriate representation of $PM_{2.5}$ unfairly penalizes these sources in terms of their contribution to ambient $PM_{2.5}$ concentrations. The EPA work group for wet stack particle sizing concluded that the particulate in wet stacks was essentially all PM_{10} or less and that the entrained water droplets tend to form PM that includes the size range from ultrafine particulate to PM_{10} . EPA believes that this approach provides the best available technology currently available with the least penalty for the source categories that use wet scrubbers to control emissions. It is the States' or regulatory authorities' responsibility to interpret EPA's recommendation to use Method 5 when measuring PM in stacks containing water droplets and to decide if the collected particulate material best represents $PM_{2.5}$.

Because there is currently no completely acceptable method for measuring $PM_{2.5}$ in wet stacks, EPA understands the need to support the States with a $PM_{2.5}$ method for wet stacks. EPA is currently developing this method and laboratory testing is ongoing. EPA has committed a significant budget and personnel to developing an acceptable method for sources with wet stacks.

EPA's method development and evaluation is focused on the "Dried Particle Method" (See *Lab Work to Evaluate $PM_{2.5}$ Collection With a Dilution Monitoring Device for Data Gathering for Emission Factor Development*; EPA-HQ-OAR-2008-0348) that directly measures the mass emission rate of particles with specified aerodynamic size. EPA plans to offer the method and protocol as soon as possible. In the meantime, the promulgated amendments to Methods 201A and 202 improve their performance and reduce known artifacts. Testers should use these final, amended methods until a $PM_{2.5}$ method is promulgated.

Regarding the advanced manual sampling technique that API is currently developing for use in “wet stacks,” EPA acknowledges the sampling evaluations being conducted by API and will review the commenter’s data when they become available. In the meantime, the promulgated amendments to Methods 201A and 202 improve their performance and to reduce artifacts. Testers should use these final, amended methods until a PM_{2.5} method for source gas containing water droplets is promulgated.

Regarding the use of AP-42 as a replacement for PM₁₀ or PM_{2.5} compliance testing, EPA has determined that this is not appropriate because of the uncertainty in the data due to variations in the particle sizing used to generate AP-42 emission factors. EPA’s AP-42 particle-sizing data for sources controlled by wet scrubbers are based upon particle sizing methodologies that are affected by the same influences and uncertainties that make particle sizing in stacks with entrained water droplets a challenging technical issue. Particle-sizing information in AP-42 is based primarily upon data collected in the 1970’s and early 1980’s. The uncertainties associated with methods used during this period of time result in particle-sizing data that are dated and may not reflect the best sampling technology or the emissions from current control devices. Particle-sizing data from the 1970’s employed many measurement methodologies that were found to introduce difficult to quantify biases in the particle sizing data. Also, source testers implemented measurement methods in different ways to deal with source-specific measurement challenges. The inconsistencies associated with addressing measurement challenges and uncontrolled biases led to higher uncertainties associated with the measurement method results. Therefore, the use of AP-42 is a very poor choice as a replacement for contemporary emissions testing.

However, it may be acceptable to allow limited application of AP-42 particle size distributions as screening assessments when the underlying biases, uncertainties, and variations of the particle sizing data and test methodology are taken into consideration. For example, one simple method involves using terms that include factors such as TDS of the recirculating scrubber water, estimated water droplet size distribution of the exit gas, and total liquid mass that are used to calculate approximate emission factors. Instruments are already commercially available that can continuously monitor TDS, estimate water droplet mass, and estimate size distributions. Output from these instruments could be used to estimate an emission factor. However, the reliability and bias of this type of candidate estimation method will be uncertain until a reliable wet-stack, particle-sizing method is available. The required data inputs for this

type of estimation model need to be identified and the likelihood that these inputs can be provided by the emission source need to be confirmed. After the input data can be readily obtained, the estimation model(s) needs to be evaluated to bring the most promising methods to fruition. (See *Development of Plans for Monitoring Emissions of PM_{2.5} and PM₁₀ from Stationary Sources with Wet Stacks*, Department of Environmental Sciences and Engineering, University of North Carolina at Chapel Hill under subcontract to MACTEC Federal Programs, EPA Contact No: EP-D-05-096, Work Assignment 2-05, August 2007; Docket EPA-HQ-OAR-2008-0348).

Comment: (Comment No. 0064.1) Field experience indicates that for dilute streams, there will be difficulty in collecting enough material on the individual filters to allow accurate weighing. Because the captured mass on the filters will be extremely low under these conditions, it creates a likely source of increased experimental error and detrimental effects on repeatability and comparability between sources. The solution is to require an arbitrary increase in sampling duration that also adversely effects test repeatability and precision. If the sampling location does not physically allow for the use of the cyclones, the only option offered in the proposed reference method is to perform Method 5 and attribute all filterable and condensable fractions to PM₁₀. This approach has been shown to be substantially biased for steel industry sources, or other sources emission streams that may contain substantial quantities of soluble species such as SO₂. There is a known artifact due to SO₂ absorption in condensed waters that biases the condensable mass fraction high. For steel industry and other sources that, due to practical limitations, cannot adopt the revised method, there is no alternative offered that addresses the SO₂ absorption artifact.

Response: EPA understands the commenter has three concerns:

(1) *Emission sources with low particulate concentrations will require longer sampling time to collect measurable filterable PM.* While EPA recognizes the need for increased sampling times, these increases are not arbitrary and with proper DQO processes can be estimated with reasonable reliability. An increased sampling duration and associated increase in captured mass will result in an increase in precision and accuracy of the resulting data. (For a more complete response, see the comments and response in Section 1.1 of this document.)

(2) Longer sampling time and higher moisture sources generate more condensed water in Method 202 impingers, which may increase the likelihood of SO₂ artifact formation even in the

revised Method 202. Regarding this concern, the hardware, recovery and analysis procedures in the revised Method 202 have reduced the artifact issues associated with SO₂ conversion to sulfur trioxide (SO₃) to the extent that any artifact that we have been able to document is below the ability of stack testers to measure. Because sampled gas is not forced through impinger water and the temperature of condensable material collection is elevated to reduce SO₂ solubility, the revised method is much improved over the former Method 202. The Electric Power Research Institute (EPRI) supported a study to evaluate the affect of higher moisture and longer sampling times on the proposed Method 202 (*Evaluation of Alternative Condensible Particulate Matter Methods*. EPRI, Palo Alto, CA: 2009 1097976; see Docket no. EPA-HQ-OAR-2008-0348). These stakeholder studies required operating the train for twice the normal duration and at moisture levels up to 15 percent. The EPRI experiments involved measuring the sulfuric acid (H₂SO₄) artifact directly by titration. These results indicated the SO₂ artifact increased from below detection limit (BDL) to 0.25 mg/dscm when the moisture increased from 10 to 15 percent regardless of the sampled gas temperature. EPA concludes from this data that artifact formation increases as moisture increases. However, the increase is below the gravimetric detection limit of the method. The reduction in artifact formation shown in the stakeholder tests conducted using the proposed revisions to Method 202 demonstrates the improvements made to the final method. For sources with higher moisture than those evaluated in the EPA and EPRI tests, testers may need to pause sampling to empty and purge water collected in the Method 202 sampling train. As an alternative to the revised Method 201A/202 combination, the Agency would support the source's request to use OTM 039.

(3) *Existing sampling locations are not equipped with sufficient diameter sampling ports to allow Method 201A sampling.* Regarding this concern, EPA understands that many existing sources include sampling ports that are less than 6 inches in diameter. The stack port requirements to sample for filterable PM₁₀ have not changed with the revision of Method 201A to include the additional PM_{2.5} cyclone. While larger ports may be required at locations required to test for PM₁₀ and PM_{2.5} in stationary sources emission stacks, 4 inch ports are likely to be sufficient when only PM_{2.5} testing is required.

Comment: (Comment No. 0057) One commenter noted that Section 1.6 (Limitations) states, "You cannot use this method to measure emissions following a wet scrubber because this

method is not applicable for in-stack gases containing water droplets.” The commenter stated that not all wet scrubbers have exhausts that contain water droplets. Many wet scrubbers incorporate chevrons, filters, or devices as “mist eliminators” to remove water droplets prior to discharge through the stack. The commenter recommended the statement be re-stated: “You cannot use this method to measure emissions at sources where water droplets are present (i.e., wet scrubber stacks).” The commenter stated that the same comment would be addressed in Section 4.0 (Interferences).

Response: We agree with the commenter and Sections 1.5 and 4.0 of the final method have been revised to say that you cannot use this method to measure emissions where water droplets are present. We agree that not all wet scrubber controlled sources will have significant levels of water droplets to impact particle sizing. Method 4 would determine the presence of water in excess of the gas’s ability to hold that quantity of water. We also agree that wet scrubber controlled sources are not the only sources that may have entrained water droplets that make particle size testing incompatible with this method. Thus, the final method has been clarified to be consistent with the commenter’s suggestion.

Comment: (Comment No. 0039.1) One commenter stated that, in the past, he has been advised that the method limitations were approximately 800 degrees Fahrenheit (°F). The revised method now states this limitation as 500°F. The commenter questioned the reason for the lower temperature now specified in the proposed method.

(Comment No. 0046.1) Another commenter also noted that the type of metal used to construct the Method 201A cyclone may limit the applicability of the method when sampling at high stack temperatures (e.g., stainless steel cyclones are reported to gall and seize at temperatures greater than 260°C).

(Comment No. 0089.1) First, given the problems with using proposed Method 201A with hot exhausts, SGCI believes that these sources with high temperature exhaust stacks should be excluded from the method, similar to stacks containing water droplets. Just as there is not a reliable method for measuring particulate entrained in water droplets, an accurate method for measuring PM_{2.5} in stack gas hotter than 260°C does not exist, which EPA acknowledges in its proposal (74 FR 12973).

(Comment No. 0078) In Method 201A, we have some concerns with the ability to test for

fine particulates on simple cycle turbines with higher stack temperatures. We have at least one source in Connecticut with a simple cycle turbine that has a stack temperature of about 1,000°F. Due to the possibility of seizing or galling of the cyclones for PM₁₀ and PM_{2.5} particle sizing (Section 8.6.1), EPA has stated that the method may be impractical for sources with stack temperatures exceeding 500°F. We are asking that EPA further investigate this issue and look at a possible modification to the method to utilize sampling equipment that can withstand higher stack temperatures. One possibility may be to move the particle sizing device, at least for PM_{2.5} out of the stack and into a heated box, enabling use of a glass lined probe for sampling.

Response: Sampling from ducts at high temperatures presents challenges that should be addressed by the source tester in conjunction with the regulatory authority. Method 201A does not permit the use of a nozzle and probe extension leading to an external heated oven to house the cyclones that would otherwise block stack flow or be inoperative at stack temperatures beyond acceptable limits. Conventional screwed-together cyclones are designed to operate in stacks that have a blockage of less than 3 percent and have a temperature of less than 500°F. EPA revised Section 8.6.1 of Method 201A to allow the method to be used at temperatures up to 1,000°F using stainless steel cyclones that are bolted together, rather than screwed together. Using “break-away” stainless steel bolts facilitates disassembly and circumvents the problem of thread galling. If the stainless steel bolts seize, over-torquing such bolts causes them to break at the bolt head, thus releasing the cyclones without damaging the cyclone flanges (see *Review of Draft EPA Test Methods 201A and 202 Related to the Use of High Temperature and Out-of-Stack Cyclone Collection*, Southern Research Institute, EPA Docket number EPA-HQ-OAR-2008-0348). The method can be used at temperatures up to 2,500°F using specially constructed high-temperature stainless steel alloys (Hastelloy or Haynes 230) with bolt-together closures using break-away bolts (see also *Development of Particle Size Test Methods for Sampling High Temperature and High Moisture Sources*, California Environmental Protection Agency, Air Resources Board Research Division, 1994, NTIS PB95-170221).

Regarding the use of a heated box external to the stack to house the cyclones, EPA disagrees with this approach because of the potential for significant losses of particulate in the nozzle and probe liner. EPA expects that transport losses for particles in the size range of interest would be significant enough to materially affect the measurement results. These losses would be caused by deposition primarily by impaction in the sampling nozzle (and at the flow

rates used in PM₁₀ and PM_{2.5} sampling) settling losses in horizontal probes. (See *Review of Draft EPA Test Methods 201A and 202 Related to the Use of High Temperature and Out-of-Stack Cyclone Collection*, Southern Research Institute, EPA Docket number EPA-HQ-OAR-2008-0348.) EPA is currently evaluating a method to sample fine PM from sources with entrained water droplets. The sampling equipment designed for this experimental work includes a large, tapered nozzle that causes particulate velocity to slow sufficiently to be collected at 90° from the original flow direction. Because this experimental nozzle presents an obstruction area to the stack flow comparable to the PM₁₀ cyclone, EPA has determined that the nozzle has no benefit for sampling fine PM from small diameter stacks where current PM₁₀ and/or PM_{2.5} cyclones cause unacceptable obstruction. The use of a similarly designed tapered nozzle for high-temperature source sampling that allows particulate-sizing cyclones to be mounted outside the stack in a heated enclosure may be effective for PM_{2.5} sampling. However, this equipment configuration must be evaluated by interested stakeholders as an alternative procedure before it can be approved as an alternative method.

Comment: (Comment No. 0089.1) The operator of a hot stack should not be required to “take extraordinary measures” when such measures are not defined in the method - no less tested in the field for accuracy (e.g., Inconel)). In light of this, SGCI would propose that Sections 1.6 (Limitations) and 4.0 (Interferences) be revised to reflect that Method 201A cannot be used when stack gas temperatures exceed 260°C (500°F), as indicated below: “1.6 Limitations. This method is not suitable for sources with stack gas temperatures exceeding 260 °C (500 °F) since the threads of the cyclones may gall or seize, thus preventing the recovery of the collected PM and rendering the cyclone unusable for subsequent use.” SGCI would encourage EPA to develop an acceptable substitute method for hot stacks, but until such a method exists, an operator should not be required to take “extraordinary measures” in the interim when there are no data on the efficacy of such measures. Instead, Method 5 testing, in conjunction with particle size distribution data specific to glass furnaces, should be used for measurement of PM_{2.5} in hot stacks.

Response: (See also the response to Comment 0078 in this section.) EPA agrees that hot sources (greater than 250°C) are problematic for Method 201A because of galling of the cyclone threads. EPA initially set the maximum temperature for Method 201A at 500°F because higher

temperatures would cause galling of the cyclone threads. In these cases field testers report that the particle sizing components cannot be separated for sample recovery of subsequent use in field testing. Some testers recommended use of anti-seize preparations or anti-seize tape. (Emissions Test Report - *Research and Development PM₁₀ Testing, Melter and Holder Furnaces*, Davenport Iowa, Aluminum Company of America, January 1998, EPA Docket number EPA-HQ-OAR-2008-0348). Galling of the cyclone threads would likely prevent accurate recovery of the collected PM. Forced separation of the cyclone components would likely render the cyclone unusable for subsequent use and could lead to metal fragments and a high bias in the results. Anti-seizing agents can be used in stacks above 500°F, but the use of these agents can lead to a positive bias in the method results. Alternative cyclone configurations that use bolt together cyclones rather than threaded caps have been used successfully by testers up to the maximum temperature of the cyclone construction material. For example, PM measurement up to 1,371°C (2,500°F) can be accomplished with bolt-together specialty alloys (e.g., Hastelloy or Haynes 230). (See *Review of Draft EPA Test Methods 201A and 202 Related to the Use of High Temperature and Out-of-Stack Cyclone Collection*, Southern Research Institute, EPA Docket number EPA-HQ-OAR-2008-0348.) EPA revised the Section 8.6.1 of Method 201A to clarify the limitations involving high temperature stack testing and the use of alternative closure cyclones. Regarding an acceptable substitute method for hot stacks, EPA allows alternative closure hardware that is not subject to galling. EPA recognizes that some stack testing firms may need to invest in additional particle sizing cyclones that incorporate these alternative closure hardware and as a result the cost of testing at sources with hot stacks may be higher than lower temperature stacks.

Comment: (Comment No. 0112) In Section 1.6 of OTM-27 there is a statement regarding the maximum stack temperature: “This method may not be suitable for sources with stack gas temperatures exceeding 260°C (500°F).” For very clean sources that have good control devices it doesn’t seem that there would be any problem just using Method 5/OTM-28 and assuming that all PM collected is less than PM_{2.5}. However, for sources such as small refuse burning incinerators with only secondary combustion as a control device, using Method 5/OTM-28 would bias the PM_{2.5} results high. This might be fine for a “worse-case” compliance determination, but would not be ideal for establishing an emission standard. The outlet

temperatures may be in the range of 1,000 to 1,800°F.

Response: As noted in other comment responses in this section, we acknowledge that using Method 5 and assuming that the catch is PM_{2.5} can potentially overestimate filterable PM_{2.5} concentrations. In these cases, regulatory agencies should consider the limitations of such an approach and understand the assumptions they make regarding the particle size distribution from samples collected in high-temperature stacks.

M201A 1.7 CONDITIONS

Comment: (Comment No. 0039.1) We are not in favor of allowing this method to be used to determine total filterable particulate, as it will produce less accurate results. It would be preferred that this option be removed rather than allow the facility to seek approval for it.

(Comment No. 0056.1; Comment No. 0061.1) API and PCA do not agree with the possible use of proposed Method 201A for the measurement of total filterable PM (including particles with aerodynamic diameters exceeding 10 µm) as described in Section 1.7 of the proposed method. The configuration of the PM₁₀ cyclone close to the sampling nozzle could create non-ideal flow effects that influence particle capture in a manner similar to under-isokinetic sampling conditions.

Response: Although the detection limit and uncertainty when using Method 201A for total filterable PM is not as low as Method 5, EPA has determined that Method 201A continues to be applicable for measuring total filterable PM. The separate conduct of EPA Method 5 or 17 is the most direct and preferred method for quantifying total filterable particulate because these methods provide better precision and accuracy. The use of revised Method 201A for total filterable particulate still requires a tester to meet all of the measurement criteria of Method 5 or 17. We understand that it is unlikely that a significant number of source tests could meet these criteria. However, the collection of size fractionated particulate data provides valuable information on the percentage of each size fraction in the total particulate emissions. Simultaneous measurement of total and size fractionated particulate provides the most reliable measure of this distribution. Therefore, sources are not prohibited from conducting consecutive PM and PM₁₀/PM_{2.5} sampling. In previous studies, PM₁₀ sampling with the cyclone design specified in Method 201A has been compared to EPA Method 17. (See *Development of*

Sampling Methods for Source PM₁₀ Emissions, Williamson, et al., Southern Research Institute, PB89190375, 1989, EPA/600/3-88/056, Docket item no. EPA-HQ-OAR-2008-0348-0016; *PM₁₀ Source Measurement Methodology: Field Studies*, Farthing, et al., Southern Research Institute, 1988, PB89-194278/AS EPA 600/3-88/055, Docket item no. EPA-HQ-OAR-2008-0348-0027; and *Investigation of Source Emission PM₁₀ Particulate Matter Field Studies of Candidate Methods*, Farthing, et al., Southern Research Institute, Docket item no. EPA-HQ-OAR-2008-0348-0018). The design of the nozzles for the PM₁₀ sampling head addresses the non-ideal flow surrounding the PM₁₀ cyclone. The comparison study showed agreement within 10 percent. This agreement is acceptable based on Method 301 requirements for comparative methods. Therefore, EPA found that the use of Method 201A was acceptable for total filterable particulate as well size segregated particulate measurements.

In addition, EPA has reviewed the method regarding the percent obstruction for use of the combined PM₁₀ and PM_{2.5} cyclones or the PM_{2.5} cyclone alone. The area of typical PM₁₀/PM_{2.5}, 47 millimeter (mm) filter hardware has been recalculated to provide accurate blockage information (see Section 8.3.2). In both cases, EPA finds that the design of the sampling equipment when operated under isokinetic requirements of the method provides acceptable bias and precision.

Comment: (Comment No. 0057) Section 1.7 Conditions: “The acceptable range for the in-stack filter temperature is generally defined as the typical range of temperature for emission of gases. The acceptable range varies depending on the source and control technology.” Please establish general temperature guidelines. Perhaps re-state to include a table indicating “acceptable temperature ranges” and associated control technology (baghouse, scrubber, oxidizer, etc.).

Response: The proposed and final methods contain guidelines for sampling stacks at different temperatures. The method contains alternatives for temperatures outside this range. To summarize, use a combination of proposed Methods 201A and 202 with stack temperatures above 30°C (85°F). Method 201A can be used alone for filterable/condensable particulate for stacks with temperatures below 30°C (85°F), using a filter meeting the CPM filter specifications in Method 202. Because stainless steel’s maximum useful temperature limit is 1,000°F, sampling stacks at temperatures above 1,000°F requires the use of an alternative high

temperature cyclone alloy construction material. As stated in Section 8.6.1 of the method, at temperatures above 260°C (500°F) you may need to take extra measures, including the use of anti-seizing agents, hard chrome plating threaded surfaces, or bolt-together, rather than screw-together cyclone assemblies, to measure filterable PM. Particulate matter measurement up to 1,371°C (2,500°F) can be accomplished with bolt-together specialty high temperature alloys (e.g., Hastelloy or Haynes 230).

Comment: (Comment No. 0112) In Section 1.7 of OTM 27 there is a statement regarding the mass of PM that should be collected: "...you must extend the sampling time so that you collect the minimum mass necessary for weighing on each portion of this sampling train." This statement is referring to the use of OTM 27 as a replacement for Method 5 or Method 17; however it seems to imply that there is a requirement for a minimum mass to be collected for OTM 27. I don't see any place where the minimum mass is specified. Are there any guidelines for what the minimum mass should be?

Response: As with any test method, the minimum mass that the source tester should target for collection depends upon the minimum detection limit that the method is capable of measuring and the precision that is desired. As was demonstrated in several assessments of Method 201A, the detection limit and precision is comparable to Method 17. In the most recent assessment where ten quadruplicate sampling runs were performed, the detection limit was 2.54 mg for total filterable PM, 1.44 mg for filterable PM₁₀, and 1.35 mg for PM_{2.5}. Since measurement capabilities varies between source sampling crews depending on the crews' attention to cleanliness and attention to detail, some testers may be able to achieve slightly higher or lower detection levels. To achieve increased precision, source testers following the DQO process will target two to three times these masses. Sampling times in excess of Method 5 durations will be required to collect these masses due to the lower sampling rate required to achieve proper particle sizing and the partitioning of the particulate in the two or three particle sizes collected.

M201A 3.0 DEFINITIONS

Comment: (Comment No. 0048.1) The definitions section in Method 201A is

[Reserved]. The same section in Method 202 has definitions that do not apply to that method for Primary PM, Filterable PM, Primary PM₁₀ and Primary PM_{2.5}. It is our recommendation that these definitions should be moved to Method 201A and removed from Method 202. Also, with regard to Definitions for both methods, it is our opinion that this section needs to be expanded to include definitions for issues that are open to interpretation, or that said issues be better defined where they are discussed, e.g., new source category, field tests, all sampling train glassware.

Response: We revised Section 3.0 Definitions to add terms that appear in Method 201A (constant weight, primary PM, PM₁₀, PM_{2.5}, filterable PM, and condensable PM). The definitions are consistent with Section 3.0 in Method 202. These terms are relevant in Method 202 and remain in the definitions section of that method. Regarding terms such as new source category and all sampling train glassware, we revised the method to make the requirements clearer where those terms are discussed. For example, new source category is qualified by adding “at a single facility” to confirm that cleaned glassware must be used at the start of each new source category tested at a single facility. A field test is merely a test conducted at a source rather than in the laboratory.

M201A 6.0 EQUIPMENT AND SUPPLIES

Comment: (Comment No. 0037) One commenter did not understand the use of glass dishes and glass 250 milliliter (ml) beakers for drying the filter and rinses in proposed Method 201A. These seem like very heavy containers for the small amount of particulate catch. Would it be better not to use the lightweight metal tins used in Proposed 202? Some labs may have been using thin lightweight synthetic beaker liners for OTM 28. Something like that would be good too. But glass just seems too heavy for the PM we are likely to see.

Response: EPA recognizes that Method 201A may generate much smaller filterable particulate mass because the material is distributed between multiple fractions. We specify the use of glass containers for drying rinse samples because glass is inert to acid compounds collected in these wash fractions; however, any inert container would be acceptable. Our additional concern with allowing aluminum drying pans is that the rinse volume may be too large for the pan to accommodate. The additional handling needed to completely recover PM from the glass beakers to an aluminum pan could introduce potential bias in the results (the additional

handling steps would not change the results, but could change the uncertainty). Furthermore, EPA Methods 5 and 17 currently specify use of glass weighing dishes and 250 ml beakers. The same requirement was included in Method 201A to promote consistency between these methods.

We revised Sections 6.2, 11.2.4, and 11.2.7 of Method 201A to allow the use of fluoropolymer beaker liners for evaporating particulate rinse solvent and acetone rinse blank, desiccating particulate to constant weight, and weighing particulate samples in the final evaporation step. If testers have information about the source emission levels that indicate masses at or below 3 mg will be collected in any one of the filterable PM fractions, then it is acceptable to use pre-weighed Teflon® beaker liners for solvent evaporation and subsequent weighing to constant weight. Testers must be careful to eliminate static charge buildup on polymer beaker liners, especially when small residue masses are weighed.

Comment: (Comment No. 0038.1) One commenter stated that the proposed Method 201A should have a section listing the required equipment for sample recovery and analysis. At a minimum the method should specify glass beakers, 50 ml weighing tins, and an analytical balance with a resolution of 0.00001 g (0.01 mg).

(Comment No. 0050.1) Another commenter stated the the proposed method should include analytical balance specifications so that the minimum targeted method detection limit can be obtained. (See Section 6.3.7 of proposed Method 202 for an example.)

Response: We agree with the commenter and we revised Section 6.2 of Method 201A to list sample recovery and analysis equipment. The revisions are consistent with the commenters' suggestions and Method 5. However, we did not add weighing tins for the reasons stated in the previous response in this section.

M201A 6.1.3 FILTER HOLDER

Comment: (Comment No. 0057) One commenter requested that the method recommend that a filter holder with an O-ring that will not bind to the filter when compressed be used. The use of compression type Environmental Supply Company 47 mm stainless steel filter holders have resulted in small fraction of the filter adhering to the O-ring. The small fraction is recovered during the rinse procedure, but that procedure introduces potential contamination and

may produce results with a negative filter gain (when not all of the filter is recovered). It is best to mandate a filter holder where the filter does not contact the O-ring or the potential for filter loss while maintaining a leak tight seal is reduced.

Response: EPA agrees that the filter should not be compressed into the components that seal the filter holder used in Method 201A. Section 6.1.3 of Method 201A has been modified to include the following change: “The filter must not be compressed between the Teflon® O-ring and the filter housing. EPA also requires the filter in Method 201A to meet Method 5 requirements. If Method 201A is used to collect filterable and condensable PM in low temperature (greater than 30°C) stacks, then the filter must meet proposed Method 202 requirements.”

M201A 6.1.5 Probe Liner

Comment: (Comment No. 0048.1) One commenter stated that if in-stack filtration is used, and no condensible [sic] analysis is performed, the material used for the probe extension is not an issue. The commenter recommended that a stainless steel liner be allowed in this situation.

(Comment No. 0057) Another commenter noted that the proposed method says, “The probe extension must be glass-lined or Teflon®.” But this would only be true if CPM were to be recovered in conjunction with the 201A sampling. The use of any type probe should suffice to sample PM₁₀ and PM_{2.5} with an in-stack cyclone and filter as the samples are not collected after the filter holder. The probe extension shall be glass-lined or Teflon® when CPM is to be determined.

Response: EPA agrees with the commenter that, under the limited use of Method 201A mentioned above for stacks operating at 30°C or less, a probe extension from the in-stack particulate filter would not contain reportable PM. Section 6.1.5 of Method 201A has been revised to clarify the use of probe extensions consistent with EPA Method 4, Section 6.1.1.

M201A 7.1.1 Filter

Comment: (Comment No. 0057) Three types of filters are available to use. Studies have been performed by Whatman and others that indicate placing a vacuum on a filter results in a loss of mass. The loss is likely not uniform on all three filter types. Mandating the use of one type would reduce this potential bias.

Response: We disagree with the commenter that it is necessary to specify the type of filter to be used in Method 201A. The commenter did not provide a citation for the studies by Whatman and others, and prior studies have confirmed that the use of the specified filter material will present no more bias or imprecision than already exists in previously collected data. (See *Development of Sampling Methods for Source PM₁₀ Emissions*, Williamson, et al, Southern Research Institute, PB89190375, 1989, EPA/600/3-88/056, Docket item no. EPA-HQ-OAR-2008-0348-0016; *PM₁₀ Source Measurement Methodology: Field Studies*, Farthing, et al, Southern Research Institute, 1988, PB89-194278/AS EPA 600/3-88/055, Docket item no. EPA-HQ-OAR-2008-0348-0027; and *Investigation of Source Emission PM₁₀ Particulate Matter Field Studies of Candidate Methods*, Farthing, et al., Southern Research Institute, Docket item no. EPA-HQ-OAR-2008-0348-0018). Moreover, EPA's specification for this method is the same as used with Method 5, Method 17, and ambient air sampling of PM_{2.5}. Accordingly, EPA has determined that no change should be made to the method.

M201A 7.2.1 Acetone

Comment: (Comment No. 0048.1) It is our recommendation that the use of polyethylene transfer/storage bottles be allowed to minimize the chance of breakage when in the field. Method 5, section 6.2.2 states, "Alternatively, polyethylene wash bottles may be used. It is recommended that acetone not be stored in polyethylene bottles for longer than a month." Also, our investigation has shown that the reagent blanks obtained from acetone in either glass or polyethylene storage containers are identical. The USEPA should not specify the type of container, but mandate the amount of residue that is acceptable. As long as the reagent blank meets the requirement specified in Section 7.2.1, then the type of container used should be acceptable.

Response: EPA agrees with the commenter that the contribution of the wash bottle or sample storage container to the blank residue is important. Therefore, EPA revised Section 6.2.1

of Method 201A by defining each of the sample recovery items in Method 201A consistently with Method 5, except for wash bottles and sample storage bottles. Any container material is acceptable for wash bottles, but the container must not contribute more than 0.1 mg of residual mass to the CPM measurements.

Comment: (Comment No. 0120) Section 7.2.1 says the maximum blank correction is 0.079mg/100ml. Can this be correct? If it is, we'll have to evaporate over 1,000 ml of acetone to get detectable blank masses or prove that our acetone is clean enough. We checked Method 5 and calculated the Method 5 allowable blank correction at 0.79mg/100ml.

Response: The blank correction limit specified in Section 7.2.1 of the proposed Method 201A is correct. Because of the possibility of collecting a lower mass for PM_{2.5} and for improved precision and accuracy, we are specifying a better grade of acetone for recovery of samples in Method 201A. Acetone is readily available with guaranteed residues below 1ppmw. An acetone with a residue of 1 ppmw is equivalent to residue of 0.1 mg/100 ml. With regard to the commenter's ability to quantify this level of residue in the acetone, evaporating 150 ml of acetone would provide the results needed to verify that the residue in the solvent is less than the guaranteed residue level of 1 ppmw.

**M201A 8.3.1 SAMPLING SITE LOCATION AND TRAVERSE POINT
DETERMINATION**

Comment: (Comment No. 0050.1) Section 8.3.1(d) (Sampling Ports) requires that a 6-inch sampling port be used, apparently in order to fit the cyclone, nozzle, and pitot tube into the stack. However, there should only be a concern regarding the port size in the event that the sampling apparatus includes both a PM₁₀ and a PM_{2.5} sampler with a nozzle less than 0.16 inches and that the typical 4-inch port should be acceptable for single PM_{2.5} or PM₁₀ samplers. The Alliance recommends that the language be changed to read as follows: "Sampling Ports. To accommodate the in-stack cyclones for this method, you may need larger diameter sampling ports than those used by Method 5 or Method 17 for total filterable particulate sampling. When you must use nozzles smaller than 0.16 inch in diameter and a combined PM₁₀ and PM_{2.5} sampling apparatus, the sampling port diameter may have to be 6 inches to accommodate the

entire apparatus as the conventional 4-inch diameter port may be too small for it to fit due to the combined dimension of the PM₁₀ cyclone and the nozzle extending from the cyclone, which will likely exceed the internal diameter of the port. A 4-inch port should be adequate for the single PM_{2.5} (or single PM₁₀) sampling apparatus and can be used when properly fitted.”

(Comment No. 0076) The physical dimensions of the cyclone would also cause problems with installation in the generally small fryer and dryer stacks.

(Comment No. 0064.1) Another commenter noted that the partitioning of the filterable solids using bulky, in-stack cyclones creates several logistical and practical problems. In the proposed Method 201A/202, partitioning the different fine particulate categories is accomplished by the use of two, relatively large cyclonic impactor devices. Low-porosity filter membranes are used to collect the individual size-segregated material cuts. The size of the in-stack separation cyclones requires 6-inch to 8-inch sampling ports that simply don't exist at the vast majority of stationary sources potentially affected by this rule.

Response: We acknowledge that the size of the sampling train PM₁₀ cyclones can potentially cause problems for some facilities that do not have sufficiently large sampling ports. However, facilities that are required to use Method 201A are responsible for ensuring that the stack has the appropriately sized sampling ports. EPA understands and agrees with the commenters' concerns regarding sampling port diameter requirements. EPA has revised Section 8.3.1(c) of the method to acknowledge that a 4-inch port may be too small in some cases to accommodate the PM₁₀ particle-sizing cyclone and the nozzle extending from the cyclone and to highlight the need for a larger port in such situations.

Comment: (Comment No. 0064.1) Site-specific physical limitations will not allow reported results between sources to be comparable because the reported values may differ simply due to sampling location clearances. The complexity of using either one or two separating cyclones will cause confusion in the reported values between sources. Depending on the capability of a given source to use the bulky in-stack apparatus, some sources will be reporting Method 5 filterable particulate as PM₁₀, while others will report Method 201A filterable catch as PM₁₀, some with the ability to fit two cyclone separators into their stack and test ports will be able to report PM₁₀ and PM_{2.5} separately. In all cases CPM will be considered PM_{2.5}. This variety of allowable reporting schemes will pose uncertainty when reported results between

sources are compared, because the reported values may differ simply due to sampling location clearances and other site-specific physical limitations. The reliance on the cyclone separators will have the result that data will not be comparable within source categories and particularly between different source categories.

Response: EPA acknowledges that source limitations, including duct diameter and port size, may influence the method selected to measure filterable and condensable particulate from stationary sources. EPA does not agree that Method 5 or Method 17 are equivalent to PM₁₀ and/or PM_{2.5} filterable PM measurements using Method 201A. Sampling from ducts that restrict the full use of Method 201A presents challenges that should be addressed by the source tester in conjunction with the appropriate regulatory authority. In all cases, test results must include a description of the test method and method options used during emission tests. Method 5 may be used to demonstrate compliance with filterable PM₁₀ and, in the future, filterable PM_{2.5} emissions limitations with the understanding that Method 5 measures PM larger than PM₁₀ and PM_{2.5}, thus providing an over estimate of PM₁₀ or PM_{2.5} emissions. The same logic holds for the use of Method 17 as an alternative to demonstrate compliance with filterable PM₁₀ and future filterable PM_{2.5} emissions limitation. If the regulated entity is able to demonstrate that the Method 5 measured PM emissions are below the applicable PM₁₀ or PM_{2.5} emissions limit, then it has also demonstrated that the PM₁₀ or PM_{2.5} emissions are below the applicable emissions limitation. EPA has determined that filterable particulate tests that are adequately documented to describe the method used to determine compliance in conjunction with State regulatory authority approval present no more confusion than currently experienced with the application of approved Method 5, Method 17, or Method 201A.

M201A 8.3.2 PROBE/CYCLONE BLOCKAGE CALCULATIONS

Comment: (Comment No. 0050.1) Add a new section in 8.3.2 that reads “8.3.2.3 Ducts with diameters less than 18 inches. Ducts with diameters less than 18 inches have blockage effects ranging from 5 to 10 percent, as illustrated in Figure 8 of Section 17. Therefore, when you conduct tests on these small ducts, you must adjust the observed velocity pressures for the estimated blockage factor whenever the combined sampling apparatus blocks more than three percent of the stack or duct (see Sections 8.7.2.2 and 8.7.2.3 on the probe blockage factor and the

final adjusted velocity pressure, respectively).”

(Comment No. 0046.1) Method 201A, Section 8.7.2.2 describes the probe blockage factor when combining the PM₁₀ and PM_{2.5} sizing cyclone. If only the PM_{2.5} cyclone and the filter are being used, do you need the blockage factor calculation on small stacks? Method 201A is not applicable for stacks with small diameters (i.e., 18 inches or less). The presence of the in-stack nozzle/cyclones and filter assembly in a small duct will cause significant cross-sectional area interference and blockage leading to incorrect flow calculation and particle size separation. The commenter (0046.1) stated that when conducting emission testing at facilities with similar small diameter stack conditions (less than 18 inches) as described in the proposal preamble (74 FR 12973), his experience supports EPA’s position on the limitations of the proposed Method 201A.

(Comment No. 0039.1) For stacks smaller than 18 inches, would there still be a blockage issue even when following the proposed Method 201A procedures, especially as the stack diameter gets smaller? Is there a lower limit of stack diameter where the method cannot be used? What is the average cross-sectional area blocked of the assembly when the pitot is removed?

(Comment No. 0052.1) There appears to be some confusion about the applicability of Proposed Method 201A to stacks with diameters less than 18 inches (Section C.1, par. 3 on page 12973, with page 12983, Section 8.7.2.3). We think EPA’s intent was to allow, but not require, RM 201A at smaller stacks at the discretion of the source but not to require it be used as blockage may occur from the instrument probe. It would be helpful if EPA would clarify this intent.

(Comment No. 0047.1) The IDNR would prefer that the small stack applicability threshold of Method 201A be explicitly stated in the method as an 18-inch stack diameter. The preamble states the 18-inch diameter minimum, while Section 8.7.2.3 of the method requires the use of Method 1A for stacks less than 18 inches in diameter.

Response: EPA agrees that the blockage factor for the combined PM₁₀/PM_{2.5} and pitot tube assembly was not correct in the proposed Method 201A. Revised measurements of the combined PM₁₀/PM_{2.5} sampling heads with the pitot tube show that the cross-sectional area is 31 square inches and measurements of the combined cyclones without the pitot tube show that the cross-sectional area is 25 square inches. The cross-sectional area includes the filter holder, but

does not include the probe extension in each case. We recalculated the blockage factor based on these areas. The following table outlines the acceptable minimum stack diameter when blockage is at 3 percent and 6 percent for the combined PM₁₀/PM_{2.5} cyclones, as well as for only the PM_{2.5} cyclone and only the PM₁₀ cyclone. These calculations were used to develop Figure 8 of the final Method 201A. For example, the blockage factor is determined by dividing the cross-sectional area of the sampling head by the cross-sectional area of the stack. Solving for the minimum stack diameter for the combined PM₁₀/PM_{2.5} sampling heads with the pitot tube (cross-sectional area of 31 square inches) at a 3 percent blockage factor yields a stack diameter of 36.4 inches.

Blockage	PM ₁₀ and PM _{2.5}	PM ₁₀ and PM _{2.5}	PM _{2.5}	PM _{2.5}	PM ₁₀	PM ₁₀
	Stack diameter allowed (inches)					
	w/o pitot	w/pitot	w/o pitot	w/pitot	w/o pitot	w/pitot
3%	32.4	36.4	20.8	26.5	24.9	29.8
6%	22.9	26.5	14.7	18.8	17.6	21.1

EPA revised the method to indicate the correct minimum stack diameter and blockage factor calculation in the method. Specifically, EPA revised Sections 8.3.1(a), 8.3.2.1, 8.3.2.2, 8.7.2.2, 8.7.2.3, Equation 24, and Figure 8, and added Figure 9. The combined PM₁₀/PM_{2.5} filter sampling head and pitot tube is not applicable for stacks with a diameter less than 26.5 inches because the blockage is greater than 6 percent. Blockage above 6 percent is not allowed for the combined PM₁₀, PM_{2.5} filter sampling head and pitot tube. For stacks with a diameter less than 26.5 inches, PM_{2.5} particulate measurements may be possible using only a PM_{2.5} cyclone, pitot tube, and in-stack filter. If the blockage exceeds 6 percent in that configuration, you must follow the procedures outlined in Method 1A to conduct tests in small stacks (stacks less than 26.5 inches in diameter). You must conduct the velocity traverse downstream of the sampling location or immediately before the test run. If only the PM_{2.5} cyclone and the filter are being used, you must use the blockage factor calculation described in 8.7.2.2 and 8.7.2.3.

Because the inlet to a PM_{2.5} cyclone is located about 2.75 inches from the end of the cyclone, the tester would be unable to collect a stack gas sample from traverse points 5 or 6. The traverse points would be located 2.6 and 0.8 inches from the far wall of the stack, respectively.

The source tester should use the procedures in Method 1, Section 11.3.2.2 for sampling these points. In this situation, three points would be sampled at the location of traverse point 4 for the duration required for all three points (4, 5, and 6).

M201A 8.3.4 PRELIMINARY VELOCITY PROFILE

Comment: (Comment No. 0048.1) It is our opinion that the first test run should be allowed to be used as the preliminary traverse for the second test run, etc.

Response: EPA agrees that an initial or trial test run is valuable for correcting issues with flow, moisture, and temperature that could potentially generate data that do not meet the method's requirements for isokinetic sampling. We believe it is imperative to establish proper sampling conditions for a test series using Methods 1 through 4 and to check for cyclonic flow. Testing contractors are not precluded from conducting initial or trial test runs to correct issues related to flow, moisture, and temperature. However, a trial run which does not meet the average and individual point isokinetic requirements would not be considered a valid run for compliance demonstration purposes.

M201A 8.3.4.3 PARTICULATE MATTER CONCENTRATION IN THE GAS STREAM

Comment: (Comment No. 0048.1) It is our opinion that Alternative Method 008 (Alternative Moisture Measurement Method – Midget Impingers) would also be an appropriate moisture estimation method and we suggest that it be included as an alternative in the method.

Response: EPA agrees with the commenter and has revised Section 8.3.4.3 of Method 201A to include the alternative to use Alternative Method 008 to estimate the moisture content of the stack gas.

Comment: (Comment No. 0050.1) One commenter supported the alternate use of wet bulb-dry bulb measurement or hand held hygrometer measurement to estimate the moisture content of sources with gas temperatures less than 71°C (160°F) with the use of Method 4.

Response: We acknowledge and appreciate the commenter's support.

Comment: (Comment No. 0058.1) The viscosity equation on page 12987 appears to differ significantly from the equation in the current version. Can you explain the difference and also what this equation is based on?

Response: The commenter is comparing Equation 3 for gas viscosity in the proposed Method 201A to the viscosity of stack gas equation in Section 6.3.4 of the original Method 201A. The commenter is correct that Equation 3 of the proposed Method 201A is not the same as the stack gas equation in Section 6.3.4 of the original Method 201A. Both equations result in stack gas viscosity values that are nearly identical. The equation in the proposed Method 201A changes the empirical constants to allow the use of percent oxygen measured in the wet stack rather than requiring oxygen to be corrected for stack moisture. The equation in the proposed method also corrects the viscosity for a cross term that involves the change in the affect of water viscosity with changes in stack temperature. The gas viscosity equation in the proposed and final Method 201A improves the accuracy of the viscosity term across a wider range of stack temperatures.

**M201A 8.3.4.4 PARTICULATE MATTER CONCENTRATION IN THE GAS
STREAM**

Comment: (Comment No. 0057) Would a scanning electron microscope (SEM) particle size analysis suffice for qualitative measurement of particle size distribution? The SEM analysis measures actual particle diameter and not aerodynamic diameter.

Response: A SEM particle size analysis would not suffice for qualitative measurement of particle size distribution because Method 201A determines particle size based upon all the characteristics that influence the aerodynamic properties of the particulate. Since Method 201A defines the particulate catch, the size cuts determined using an SEM would not be the same as that collected using Method 201A. Additionally, a different type of filter would be needed to allow the use of an SEM. Therefore, the final rule does not contain an allowance to use SEM particle size analysis.

For consistency with NAAQS particle sizing, several PM sizing characteristics are controlled by the cyclone design. The characteristics include the following: (1) the use

aerodynamic particle sizing rather than physical particle sizing; (2) a reference density of particulate of 1 gm/cubic centimeter (gm/cc); (3) a 50 percent particle sizing of the particles at the stated cut points; and 4) the slope of the particle sizing curve is comparable to the slope of the Federal Register reference method for ambient air quality determination. The first two characteristics affecting the size determined by this method incorporate the shape and the density of the particulate as they present themselves to the sampling device. Few particles are perfect spheres and thus have different aerodynamic properties depending on their orientation to the air flow. The last two characteristics result in different percentages of PM near the stated particle size being retained by the method for weighing. While half the particles of the stated size are retained for weighing, higher percentages of smaller particles (as defined by the slope of the size vs. percentage curve) than the stated particle size cut are retained and lower percentages of larger particles (as defined by the slope of the size vs. percentage curve) are retained for weighing. The particle sizing by SEM and other optical sizing devices do not address these complex relationships as is done with the cyclone and other dynamic particle sizing devices.

Even if SEM were allowed as a particle size indicator, the in-stack filter requirements are not compatible with filters used for SEM particle sizing and counting. The two types of filters used for SEM analysis are cellulose and Teflon® because particles must be captured on a flat surface to allow measurement by a SEM. Cellulose does not have the temperature stability characteristics and is reactive with stack gas components. Teflon® does not have the temperature stability characteristics. Therefore, filters used for SEM are not compatible with Method 201A.

M201A 8.4 PRE-TEST CALCULATIONS

Comment: (Comment No. 0057) Will the use of Windows-based Cascade Impactor Data Reduction System (WinCIDRs) software suffice for pre-test calculations. WinCIDRs was developed by Southern Research Institute when developing the cyclone PM sampling train. Alternatively, has EPA developed M201A operating software?

Response: EPA agrees that it is a complex task to complete the Method 201A calculations. Method 201A provides one way to perform in-stack particle sizing based on the use of cyclones and not cascade impactors. The use of impactors is another particle sizing

methodology. When performing particle sizing by impactor technology, testers must ensure that sufficient material is collected on each stage to weigh and to ensure that an excessive amount of material leading to produce particle bounce and re-entrainment is avoided. Cascade impactor particle size cutpoints are calculated using different constants due to the performance of the impactor stages.

We agree that any software that uses the equations specified in Method 201A is suitable for performing the pre-test calculations. Software that incorporates the equations provided in Method 201A as promulgated is acceptable for both pre-test and final calculations. Software that uses an alternative approach must be shown to be equivalent to the calculations required in Method 201A and may be proposed to EPA as an alternative. EPA has not developed software to complete the pretest calculations, but understands that several stack sampling firms and cyclone vendors have developed Excel spreadsheets or database software routines to perform the necessary calculations. EPA has developed the Electronic Reporting Tool (ERT) that includes post-test calculations to calculate the particle size cut as specified in the published method for both the PM₁₀ and PM_{2.5} cyclones.

M201A 8.5.1 THE ASSUMED REYNOLDS NUMBER

Comment: (Comment No. 0058.1) Section 8.5.1 specifies a procedure to verify the “assumed Reynolds number.” It appears that this initial Reynolds number would be determined by using preliminary test data in Equation 8. Please confirm that this is correct. If correct, it may be helpful to add text stating that the “assumed Reynolds number” is to be determined in this manner using preliminary test data.

Response: The Reynolds number (N_{re}) is estimated during pretest calculations to select the correct equation to calculate the cyclone cut point (D_{50}) lower limit in Equations 5 and 10. Samplers must determine during the pretest calculation process whether the Reynolds number is less than 3,162 or not. Once the sampling rate is chosen to satisfy the isokinetic sampling requirements, the Reynolds number can be recalculated based on the preliminary test data to verify that the correct equation for cyclone cut point was selected and the correct sampling rate has been selected. If the Reynolds number calculated using the test-specific parameters indicates the incorrect equation was used to determine the cyclone cut point, the tester must repeat the

calculation using the correct cut point formula. Therefore, we have added text to section 8.5.1 describing the initial selection of the correct cut point equation based on an assumed Reynolds number, but we have not required the use of Equation 8 to make this initial decision. Equation 8 is used to confirm the assumed Reynolds number (greater than or less than or equal to 3,162) and may be used to select the initial Reynolds number if you can estimate the sampling rate.

M201A 8.5.5 OPTIMUM SAMPLING NOZZLE

Comment: (Comment No. 0041.1) Section 8.5.5(b) states that when testing for PM_{2.5} only, you may have only two traverse points out of 12 that are outside the range of the minimum velocity pressure (Δp_{\min}) and the maximum velocity pressure (Δp_{\max}). If the coarse fraction for PM₁₀ determination is included, only one traverse point out of 12 can fall outside the range of the Δp_{\min} and Δp_{\max} . Many agencies require that more than the recommended maximum 12 traverse points be sampled if total filterable particulate is being determined (as noted in Section 1.7 of Method 201A). In cases where more than the recommended maximum 12 traverse points are sampled by Method 201A, then the allowable number of traverse points that fall outside of the range of the Δp_{\min} and Δp_{\max} should be adjusted accordingly to match the stated failure rates expressed as percentages. For example, if 24 traverse points are sampled by Method 201A, then for PM_{2.5} determination, you may have only four traverse points out of 24 that are outside the range of the Δp_{\min} and Δp_{\max} . This four out of 24 traverse points represents the same 16 percent failure rate criteria listed in Section 8.5.5. Similarly, for PM₁₀ determination by Method 201A, when 24 traverse points are sampled two traverse points can fall outside the range of the Δp_{\min} and Δp_{\max} . Again, the 8 percent failure rate cited in Section 8.5.5 is maintained. This clarification to the allowable number of traverse points that can fall outside the range of the Δp_{\min} and Δp_{\max} is necessary to prevent agencies from interpreting the intent of Section 8.5.5 of Method 201A to be 2 of 12 traverse points for PM_{2.5} rather than 16 percent of the traverse points. This is important for many sources where the gas velocity pressure varies during the test run and/or varies across the stack.

(Comment No. 0040.2) I endorse the author recommended changes to the PM₁₀/PM_{2.5} and Dry Impinger Condensable PM Methods that are summarized in comment letter 0041.1, including the author's recommendation to relax the number of traverse points outside of the

minimum-maximum velocity pressure range when stack velocity and gas temperature fluctuate (adjust the delta H sampling rate).

Response: EPA agrees that increasing the number of allowable traverse points outside the range Δp_{\min} and Δp_{\max} is appropriate when more than the appropriate number of traverse points are sampled. EPA has modified the method to allow 16 percent failure rate rounded to the nearest whole number for PM_{2.5} only and 8 percent failure rate rounded to the nearest whole number if the coarse fraction for PM₁₀ determination is included.

M201A 8.6 SAMPLING TRAIN PREPARATION

Comment: (Comment No. 0057) One commenter noted that Figure 1 shows a heated sampling probe. Is it necessary to heat the sampling probe? In addition, distilled water is presented, but if the use of the impingers in the figure is for moisture determination. If this is correct than any type of water would suffice; therefore, I would recommend removing “distilled” from the figure.

Response: The sampling probe must be heated to prevent moisture present in the sampled gas from condensing inside the sampling probe. The use of the heated probe and distilled water depends on the method that is paired with Method 201A. Typically, Method 202 would be paired with Method 201A. In this situation, the probe temperature must be maintained to minimize collection of CPM in this part of the sampling train. If the stack temperature is less than 85 °F, then the probe must be heated sufficiently to prevent moisture present in the sampled gas from condensing in the sampling probe extension, thus biasing moisture determination. While there is no requirement to use distilled or deionized, ultrafiltered water in the impingers used to determine moisture after the CPM filter when Method 201A is used to collect both the filterable and condensable PM, EPA has decided not to revise the method to avoid confusion when Method 202 is used to collect CPM.

M201A 8.6.1 SAMPLING HEAD AND PITOT TUBE

Comment: (Comment No. 0046.1) Method 201A, Section 8.6.1 discusses the use of

specialty metals if the temperature of the stack exceeds 260°C. There is no mention of a maximum temperature for the calculation of viscosity. Calculating a correct viscosity is critical in determining the cut point of the cyclone. The viscosity equation (Eq. 3) is an empirical equation. Assuming we could construct a specialty metal cyclone that would hold up to 600°C, would the equation calculate an accurate viscosity?

Response: The original empirical equation for viscosity was derived from a rigorous algorithm published by Wilke in 1950 (*A Viscosity Equation for Gas Mixtures?* Wilke, C. R., J. Chem. Phys., 18, pp. 517-519). Saksena also published an evaluation of empirical viscosity equations in 1963 (*Viscosity of Multicomponent Gas Mixtures*, Saksena, M. P. and Saxena, S. C., J. Sci, Industrial Res. (India) Volume 31, A, No. 1, pp. 18 – 25 (www.new.dli.ernet.in/rawdataupload/upload/insa/INSA_1/20005ab9_18.pdf)). In addition, Mathur (Mathur, S., and Saxena, S., C., *Applied Scientific Research* Volume 15, Number 1, January 1966, pp. 203-215) empirically evaluated the viscosity of gas mixtures at various temperatures and concluded that the predicted and experimental viscosity of polar and nonpolar mixtures of gases using equations derived from the original approach by Wilke compared within the experimental error of the measurement they performed. Therefore, EPA has determined that the equations provided in the revisions to Method 201A are adequate for application of the temperature range anticipated for the method.

M201A 8.6.2 FILTERABLE PARTICULATE FILTER HOLDER

Comment: (Comment No. 0057) Section 8.6.2 Filterable Particulate Filter Holder: "Weld the sensing lines to the outside of the probe to ensure proper alignment of the Pitot tube." I would recommend replacing the word "weld" with fasten. Welding Pitot tubes to method probes to accommodate a PM₁₀/PM_{2.5} sampling head would minimize alternative uses of the method probes. Replacing Pitot tips on a standard Method 5 sampling probe with longer Pitot tube tips may be an acceptable alternative.

Response: We agree with the commenter. EPA revised Section 8.6.2 of Method 201A to use the term "securely fasten" instead of "weld."

Comment: (Comment No. 0057) One commenter noted that the note in Section 8.6.2

(Filterable Particulate Filter Holder) says, “[Note: Calibrate the pitot tube on the sampling head because the cyclone body is a potential source flow disturbance].” Has a default Pitot calibration factor been established? If so, please present it in the text.

Response: EPA agrees with the commenter that accurate pitot calibration factors are essential to the method. EPA has not determined default pitot calibration factors because the method allows several variations in cyclone configuration and construction. Thus, EPA has revised Section 8.6.2 of Method 201A to make it a requirement to calibrate the pitot tube on the sampling head because the cyclone body has the potential to cause flow disturbance. For pitot calibration procedures, refer to the ASTM D3796.

M201A 8.6.7 MOISTURE TRAP

Comment: (Comment No. 0050.1) One commenter supported the proposed procedure in Section 8.6.7 that enables the tester to use an empty modified Greenburg Smith impinger followed by an impinger containing silica gel to collect moisture when measuring filterable PM that is maintained below 30°C (85°F).

Response: We acknowledge the commenter’s support.

Comment: (Comment No. 0056.1) API does not agree with the use of proposed Method 202 in combination with proposed Method 201A as described in Section 8.6.7 of proposed Method 201A. There is a lack of data concerning the possible catalytic oxidation effects of the large stainless steel cyclones and some common gases such as SO₂. These possible catalytic effects could create artifact sulfates.

(Comment No. 0061.1) The long sampling times sometimes required for Proposed Method 201A contribute to higher-than-desirable condensed moisture levels in the dry impingers. It is also important to note that there is a lack of data concerning the possible catalytic oxidation effects of the large stainless steel cyclones and some common gases such as SO₂. These possible catalytic effects could create some artifact sulfates. For both reasons, the Proposed Method 202 sampling train should be used separately from the Proposed Method 201A sampling train in cement kiln stacks. The sampling times for Proposed Method 202 at wet process cement kilns should be limited to one hour.

Response: EPA has determined that the pairing of proposed Method 201A and proposed Method 202 prevents many of the biases that occur due to the presence SO₂. The stainless steel specified for use in the manufacture of Method 201A cyclones was chosen for its resistance to reactivity with typical stack gas constituents such as SO₂. Sulfur trioxide converts readily to H₂SO₄ in the presence of water vapor and stainless steel. Although, stainless steel is relatively inert to attack by H₂SO₄, halogen acids are known to attack stainless steel. EPA understands that there may be rare circumstances in which reactive gases are present in selected facility stack gas. In these cases, testers and facility operators have knowledge of the reactive stack gas components and should use the flexibility provided in the test methods to avoid potential biases due to reactive stack gas components. EPA has modified Section 6.1.2 of Method 201A to allow the use of fluoropolymer-coated or stainless steel (316 or equivalent) PM₁₀ and PM_{2.5} sizing devices to parallel the use of similar construction materials shown in Method 201A (section 6.1.1) for nozzles. EPA has also modified Section 6.1.2 to allow the use of alternative high-temperature metal alloys. You must choose materials of construction for the cyclone that are compatible with the source you are sampling. As stated in the Limitations section of the method, you may measure PM at temperatures above 260°C (500°F) by using anti-seizing agents or hard chrome plating threaded surfaces. Particulate matter measurement up to 1,371°C (2,500°F) can be accomplished with bolt-together specialty alloys (e.g., Hastelloy or Haynes 230). With regard to the comments about long sampling times and higher condensed moisture levels, see the response under the Section 1.1.

M201A 8.6.8 LEAK CHECK

Comment: (Comment No. 0047.1) For Method 201A there should be an explicit vacuum for the initial leak check. Since the cyclones are removed for the post leak check, there needs to be an assurance that they are leak checked at an acceptable vacuum. Estimating a value is difficult as there are many variables.

Response: EPA disagrees with the commenter. Section 8.6.8 of Method 201A specifies that the procedures outlined in Section 8.4 of Method 5 must be used to leak-check the entire sampling system. EPA has determined that the leak check outlined in Section 8.4 of Method 5 is adequate. The most likely cause of vacuum increase during a sampling run is filter blockage.

Therefore, the most likely locations of leaks at higher vacuum are components of the train that follow the cyclones. These components are checked for leaks during post-sampling leak checks. High vacuum across the cyclones is not expected.

M201A 8.6.1 SAMPLING HEAD

Comment: (Comment No. 0048.1) It is our opinion that the reference in this section to unsaturated stacks is not necessary, since this method is already inappropriate for saturated stacks. In addition, the 30 to 40 minute in-stack warm-up time is excessive. Experience has shown that 10 to 15 minutes warm-up in the stack is sufficient to warm up the head to prevent condensation. We recommend that the wording be changed such that the head is left in the stack until moisture no longer condenses on the exterior surfaces.

Response: EPA agrees that reference to “unsaturated” stacks is unnecessary because the method applies only to stacks that do not contain condensed water droplets. Therefore, EPA has revised the section headings to read “8.6.6.1 Warm-up.” Sections 8.6.6.1 and 8.6.6.2 have also been revised to remove “unsaturated stacks.” However, EPA did not reduce the warm-up time. The 30 to 40 minute warm-up time is the time required to get the internal surfaces of the cyclone close to the internal stack temperature to prevent condensation of material on the surfaces. The warm-up time depends on the stack temperature, the thickness of the metal of the cyclone(s), and the initial temperature of the cyclone(s). The 30 to 40 minute in-stack warm-up time is required to ensure that the cyclones could reach a temperature to prevent condensation of material on the interior surfaces of the cyclones in hotter stacks (up to 500°F). Shortening the warm-up time by heating the cyclone assembly outside the stack is still allowed.

M201A 8.7.1 DWELL TIME AT FIRST SAMPLING POINT

Comment: (Comment No. 0034.1) There is some confusion in the testing community regarding the calculation of dwell time in OTM 27 (Method 201A). Most of this can be resolved by using the commenter’s equation for the calculation of dwell time at all points (see EPA-HQ-OAR-2008-0034.1 for equation). The suggested equation eliminates the confusion as to which

values you use at point 1 and accounts for the difference in Pitot coefficients in the preliminary traverse and the sample run. It is also a statistical improvement by preventing any unusual variation in ΔP_1 from transferring to the remaining points. This should result in the actual run time being closer to the estimated run time. In the text of OTM 27, Section 8.7.1.1 would be deleted and the wording of Section 8.7.1.2 modified to be grammatically correct. The commenter's equations replace Equations 22 and 23 and include any adjustments referred to in Equations 24, 25, and 26.

Response: EPA agrees with the commenter that simplification of the equations used for sampling times at each traverse point would benefit the method. The commenter's recommendation for simplifying the calculation of time at each traverse point is based on the assumption that the preliminary traverse for point 1 is performed by a separate pitot measuring device from the pitot used during the actual sampling. The simplification to the formula for sampling time requires the use of the preliminary traverse points to determine the average velocity pressure, and the velocity pressure at each of the traverse points (ΔP_n) is measured during the testing with a different pitot measuring device. EPA agrees that the combination of Equations 22 and 23 requires the pitot coefficient correction and has revised the method to simplify the equations and make these changes.

Regarding their suggestion to simplify the calculation in Equation 22, EPA has determined that their recommended simplification would not result in the same constant for all testers. Therefore, we did not further simplify Equation 22.

M201A 8.7.2.2 PROBE BLOCKAGE FACTOR

See Section 8.3.2 of this document for comments on blockage and minimum stack diameters.

M201A 8.7.2.3 FINAL ADJUSTED VELOCITY PRESSURE

Comment: (Comment No. 0050.1) Proposed Method 201A (74 FR 12973, Section C.1, paragraph 3) states that "Method 201A is also not applicable for stacks with small diameters (i.e., 18 inches or less)." However, page 12983, Section 8.7.2.3 reads as follows: "Final adjusted

velocity pressure. Calculate the final adjusted velocity pressure (Δ_{ps2}) using Equation 26. [Note: Figure 8 of Section 17 illustrates that the blockage effect of the large combined cyclone sampling head increases rapidly below diameters of 18 inches. Therefore, you must follow the procedures outlined in Method 1A to conduct tests in small stacks (< inches diameter). You must conduct the velocity traverse downstream of the sampling location or immediately before the test run.]" It may be useful for smaller stacks depending on the source specific circumstances. As such, the language in the text above should be modified to include 18 in the space "< inches diameter".

(Comment No. 0038.1) Another commenter noted that in the second sentence of this section, the number 18 was left out of the subtext in parentheses.

Response: We agree with the commenters that we inadvertently omitted “18” from the parenthetical in Section 8.7.2.3 of the proposed Method 201A. To clarify the conditions under which the method may be used in small stacks, EPA revised Sections 8.3.2.1, 8.3.2.2, 8.7.2.2, 8.7.2.3, and Figure 8 and added a new Figure 9 to Method 201A. (See Section 8.3.2 of this document for additional comments and responses on blockage and minimum stack diameters.) EPA recalculated the stack blockage factor for the combined PM₁₀/PM_{2.5}/filter assembly as well as the PM_{2.5}/filter assembly and revised the method with these corrections. The combined PM₁₀/PM_{2.5} filter sampling head with the pitot tube is not applicable for stacks less than 25.7 inches in diameter because the blockage factor exceeds 6 percent. However, PM_{2.5} particulate measurements may be possible using only the PM_{2.5} cyclone and in-stack filter as long as the blockage factor does not exceed 6 percent of the stack diameter (18.8 inches) (see Figure 8 of Method 201A). As stated in Section 8.7.2.3 of Method 201A, you must follow the procedures outlined in Method 1A to conduct tests if the blockage factor is greater than 3 percent.

M201A 8.7.3.3 SAMPLING PROCEDURES

Comment: (Comment No. 0050.1) Proposed RM 201A (74 FR 12984, Section 8.7.3.3) Sampling Procedures, suggests that any portion of the cyclone external to the stack will need to be heated or insulated to the stack temperature. This may or may not be necessary and flexibility should be provided to address the concern that many source stacks have a negative draft and create the potential to entrain insulating material into the stack and potentially into the sampling train near the outer wall sample points. The insulating step should either be omitted or that the

language should be changed to require that the insulating material be covered in a “lint free” material such as Gortex, Mylar, Kevlar or some other high temperature rated material.

Response: We disagree with the commenter that the heating requirements for portions of the cyclone and filter that are external to the stack should be removed from the final method. EPA has determined in order to prevent condensation in the cyclones, the cyclones must be maintained at stack temperature.

We agree that the testing contractor should use caution when insulating portions of the cyclone and filter that are external to the stack to avoid entrainment of insulating material. We are retaining the pre-heating requirement in the final method to maintain consistency in the application of Method 201A between different testing contractors and different source types. The tester must clean the sampling port to minimize or eliminate contamination for all test methods. This caution also extends for all material used to insulate exposed portions of the sampling head or any material used to seal the port to minimize biasing the stack gas collected at the first sampling point.

EPA appended the note in Section 8.7.3.3 of Method 201A to indicate that care should be taken to minimize contamination from material used to block the flow or insulate the sampling head during collection at the first sampling point.

Comment: (Comment No. 0057) Section 8.7.3.3 Sampling Procedures: "[Note: For many stacks, portions of the cyclones and filter will be external to the stack during part of the sampling traverse. Therefore, you must heat or insulate portions of the cyclones and filter that are not within the stack in order to maintain the sampling head temperature at the stack temperature...]" Does this mean $\pm 50^{\circ}\text{F}$ of the stack temperature? What is the guidance for heating this portion of the sampling train?

Response: EPA clarified Section 8.7.3.3 of the method to make it clear that you must maintain the cyclone sampling head within $\pm 10^{\circ}\text{C}$ ($\pm 18^{\circ}\text{F}$) of the stack temperature. The important aspect is to maintain the temperature of the sampled gas to achieve the proper particle sizing and to minimize the condensation of material on the internal surfaces of the cyclone. Maintaining the cyclone and filter assembly temperature can be accomplished by any means that does not contaminate the sample or cause PM not originating from the stack gas to enter the sampling system. EPA has determined that cyclones that are preheated to the stack temperature

(and insulated) have enough thermal mass combined with the temperature of the stack gas to maintain the proper temperature range. The final method continues to require that the sampler verify the combined cyclone sampling head temperature is at stack temperature.

Comment: (Comment No. 0057) Section 8.7.3.3 Sampling Procedures: There is a description of a running stop at the end of the test run. Is the sampling flow to be halted during port changes or is the flow to remain on? Can you please address this question in the text of the method?

(Comment No. 0048.1) It is our recommendation that a running stop is not necessary in all cases. In fact, a running stop risks picking up contamination from ports and stack walls, and should only be used when necessitated by negative static pressures as addressed already in section 8.7.3 of this method.

(Comment No. 0039.1) What is the purpose of the running stop?

(Comment No. 0049.1) There is a serious conflict between Sections 8.6.5.1 and 8.7.3 in this method. Section 8.6.9.1 states correctly that a warm-up period of 30 to 40 minutes is required before sampling starts to prevent condensation inside the cyclones. Section 8.7.3 states that a “running start” must be used if the negative pressure at the sampling location is higher than 5 inches of water column. Obviously, a running start and a warmup period are totally incompatible. The warmup is absolutely essential; however, we are not positive about the running start. EPA should rectify this conflict.

Response: The purpose of the running start is to maintain the flow in the system toward the pumps rather than a reverse flow. Most stacks are at a negative pressure (lower than ambient) and therefore need to have a running start and end to ensure that the flows in the cyclone, probe and impingers are always toward the pumps. The testers are always required to clean the ports to minimize the potential of contamination. Therefore, Section 8.7.3.3 has been clarified to state that for running starts and stops the flow must continue until the sampling head is completely removed from the port and that flow must be restarted prior to inserting the sampling head into the sampling port during port changes.

Comment: (Comment No. 0039.1) Should the velocity pressure measurements (Equations 20 and 21) be compared with the adjusted average blockage factor (b_f) preliminary

measurements (Equation 26)?

Response: Method 201A requires isokinetic sampling between the maximum velocity pressure and the minimum velocity pressure. EPA corrected velocity pressures to take into account the blockage effect on the velocity pressure, therefore, the preliminary adjusted velocity pressure in proposed Equation 26 must be compared to the minimum and maximum velocity pressure range to ensure isokinetic sampling was accomplished within the allowable error.

Section 8.7.2.3 requires the final velocity pressure calculated by Equation 27 (proposed Equation 26) to include the appropriate adjustment for pitot tube calibration on the associated cyclone head (either PM₁₀ and PM_{2.5} or PM_{2.5} alone) and adjustment for the appropriate blockage factor. Because the final velocity pressure is directly related to the nozzle size and flow rate selected for isokinetic sampling results as shown in Equation 20, they should be compared to the results from proposed Equation 26 to ensure the selected cyclone head assembly blockage factor has been considered. Requirements in Section 8.5.4 direct samplers to correct for blockage use of different pitot tube coefficients. Therefore, the final velocity pressure calculated in proposed Equation 26 must fall between the minimum and maximum velocity pressure calculated in proposed Equations 20 and 21. (For further details, refer to *Application Guide for Measurement of PM_{2.5} at Stationary Sources* EPA 600/3-90/057 in Docket No. EPA-HQ-OAR-2008-0348-0002.)

M201A 8.7.5.2 LEAK CHECK PROBE/SAMPLE TRAIN ASSEMBLY (POST-TEST)

Comment: (Comment No. 0057) Can leak rate adjustment calculations as described in Section 12.3 of Method 5 be utilized if the post-test leak check fails the specified criteria? If so, can this statement be added to the text?

Response: The leak rate adjustment calculations as described in Section 12.3 of Method 5 cannot be utilized if the post-test leak check fails the specified criteria. In Method 5, the leak rate adjusts the final volume collected by the sampling train. There is no limit to the amount of correction that can be applied. Leak correction similar to that allowed in Method 5 does not account for isokinetic sampling required in Method 201A. The requirements for an acceptable leak rate are clearly stated in Section 8.7.5.2 of Method 201A and no further clarification is

necessary.

M201A 8.7.5.5(g) RECOVERY OF PARTICULATE MATTER

Comment: (Comment No. 0038.1) Section 8.7.5.5(g) specifies that 100 ml of acetone be collected as an Acetone Rinse Blank and analyzed per instructions in Section 11.2.7. Further, instructions in Section 11.2.7 direct that 100 ml of acetone be reduced for determination of the dry residue mass. We feel that a 100 ml volume specified in Section 11.2.7 for reduction is too small. 100 ml of acetone weighs 79 grams at 20° C. To meet the 1 ppmw requirement for maximum residue by weight, the residue must be 0.000079 grams or less. The gravimetric Limit of Detection (LOD) (see Attachment 1 of EPA-HQ-OAR-2008-0348-0038.1) for the 50 ml aluminum weighing pans is in the range of 0.00009 to 0.00016 grams, and the Practical Limit of Quantitation (PLQ) is in the range of 0.00030 to 0.00053 grams. Therefore, in order to have a measured mass with minimal uncertainty in the measured value, the volume of acetone reduced for the percent weight residue determination should be no less than 450 ml, and the volume collected as the Acetone Rinse Blank should be no less than 500 ml.

Response: We disagree with the commenter and have increased the blank volume specified in Section 11.2.7 to 150 ml. Samplers are free to collect more acetone for their field reagent blanks and correct the blank to 150 ml if they choose to use more acetone field blank volume. Because the typical amount of acetone used in field recovery of the Method 201A cyclone assemblies is approximately 150 ml, EPA has determined that this volume better represents the blank contribution to the method results. If the dry residue mass cannot be measured using 150 ml for the acetone rinse blank, then it will not affect the test results. Solvents are required to have a residue less than 1 ppmw, as specified by the vendor certification. Acetone meeting these specifications will not contribute weighable mass to samples and no correction of the particulate mass for acetone residue will be necessary as specified in Section 12.3.2.2 and Equation 42. Acetone field recovery blank measurement is performed to allow the tester to identify contributions to gross contamination during field measurements. Therefore, EPA has determined that 150 ml of solvent is adequate to confirm that recovery blank contribution to sample weight above the method constant weight requirement of 0.5 mg. If the dry residue mass cannot be measured using 150 ml for the acetone rinse blank, then it will not

affect the test results.

Comment: (Comment No. 0050.1) Proposed Method 201A (74 FR 12985, Section 8.7.5.5(g)) Container # 7, Acetone Rinse Blank, requires the tester to take 100 ml of acetone directly from the wash bottle. The Alliance recommends the following language: "Take *approximately* 100 ml of acetone to avoid using a clean measuring flask in the field."

Response: EPA agrees and clarified Section 8.7.5.5(g) of Method 201A to state that *approximately* 200 ml may be taken from the acetone wash bottle. EPA understands the need to minimize glassware in the field for sample and blank collection. EPA agrees that an approximate volume greater than 150 ml is acceptable for the acetone field recovery blank sample because the laboratory will measure exactly 150 ml of acetone from this sample container for analysis (see Section 11.2.7).

M201A 10.1 GAS FLOW VELOCITIES

Comment: (Comment No. 0047.1) Section 10 of Method 201A requires an S-type pitot tube. If testing of stacks with diameters less than 18 inches is still allowed and the testers are required to use Method 1A, the option of using a standard pitot tube should remain.

Response: As discussed in Section 8.3.2 of this document, EPA recalculated the area blocked by the combined sampling head and determined that the 6 percent blockage occurs at stacks with a diameter of 26.5 inches instead of 18 inches. If testing is performed in stacks less than 26.5 inches in diameter (as specified in Section 8.7.2.3 of this document and Method 201A), then testers are allowed to use a standard pitot tube according to the requirements in Method 1A and 5. Method 1A and Method 5 allow the use of a standard pitot to minimize the effects of duct blockage when the velocity measurement is conducted at a downstream location. EPA has modified Section 10.1 of Method 201A to allow standard pitot tubes to be used downstream when significant blockage exists.

M201A 10.2 THERMOCOUPLE CALIBRATION

Comment: (Comment No. 0048.1) It is our recommendation that alternatives, or National Institute of Standards and Technology (NIST) equivalents, to mercury-in-glass thermometers should be allowed. Many companies are eliminating the use of anything with mercury in it due to the environmental and safety concerns. The options in Method 2, section 10.3.1 would be appropriate. (The section of Method 2 that should be referenced is 10.3.1, not 10.1.4.1.2)

Response: EPA agrees with the commenter. Section 10.2 of the final method has been revised to allow the use of alternatives to mercury-in-glass thermometers. EPA added an alternative to use a reference thermocouple and potentiometer (calibrated against NIST standards). EPA also agrees that the cross reference should be Section 10.3.1 of Method 2.

M201A 10.3 NOZZLES

Comment: (Comment No. 0047.1) Section 10.3 of Method 201A allows Teflon® coated nozzles. Only stainless steel nozzles should be allowed because Teflon® flakes at higher temperatures.

Response: EPA has determined that Teflon® coated nozzles and cyclones are acceptable for use in lower temperature stacks to avoid corrosion issues. As part of the inspection process prior to sampling, testers should inspect the physical condition of the sampling probes as well as assessing the physical dimensions to ensure that bias from the probe is minimized.

M201A 11.2.1 CONTAINER #1, < PM_{2.5} MICROMETER FILTERABLE PARTICULATE

Comment: (Comment No. 0116) Section 11.2.1 instructs us to weigh filter to the nearest 0.1 mg. Is this correct?

Response: Methods 201A and 202 require weighing the filter and other components to 0.1 mg. This is the approximate level of precision that could be expected without using a very expensive analytical scale and weigh room. We did not propose more stringent weighing requirements because we believe that the source tester's ability to obtain the consistency in recoveries (i.e., obtaining near zero mass differences in blanks) would not justify more stringent

minimum weight capabilities of the scale.

**M201A 11.2.2 AND 11.2.3 DRY WEIGHT OF PARTICULATE MATTER
CONTAINER #2, > PM₁₀ MICROMETER FILTERABLE PARTICULATE
ACETONE RINSE AND CONTAINER #3, FILTERABLE PARTICULATE ≤
10 MICROMETER AND > 2.5 MICROMETERS ACETONE RINSE**

Comment: (Comment No. 0039.1) Both of these acetone rinse containers say to treat these like Container #1 (filter). Should they be treated like Container #4 (another acetone rinse) instead?

(Comment No. 0108)) My question is in the analytical procedures 11.2.2 and 11.2.3 the method says to treat containers 2 and 3 like container 1. The problem is that container 1 is just the PM_{2.5} filter that we dessicate and weigh while containers 2 and 3 contain the PM from the cyclones plus acetone. We would assume that the correct procedure is to transfer the contents of containers 2 and 3 into pre-weighed beakers, evaporate the acetone, dessicate, and weigh similar to the procedures for container 4.

(Comment No. 0050.1) The following language should be added to Sections 11.2.2 and 11.2.3: “Note the level of liquid in the container, and confirm on the analysis sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use the methods, subject to the approval of the Administrator to correct the final result.”

(Comment No. 0038.1) Another commenter noted that the instructions in the sections for Containers #2 and #3 direct the user to process the acetone rinses from cyclones I and IV like Container #1, which is a fiber filter. To make these sections consistent with instructions for handling liquid samples throughout the proposed Methods 201A and 202, it should contain language similar to that in the proposed Method 202 Section 11.2.2 – Organic Fraction Weight - “Quantitatively transfer the contents to a clean glass beaker, and evaporate at room temperature (not to exceed 30°C (85°F)) and pressure in a laboratory hood to approximately 10 ml. Quantitatively transfer the beaker contents to a 50 ml pre-weighed tin, and evaporate to dryness at room temperature (not to exceed 30°C (85°F)) and pressure in a laboratory hood. Following evaporation, desiccate the residue for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh at intervals of at least 6 hours to a constant weight (i.e., ≤ 0.5 mg change from

previous weighing), and report results to the nearest 0.1 mg.”

Response: Regarding the request to treat Containers 2 and 3 in Method 201A consistently, EPA agrees with the commenters that the instructions for handling liquid samples should be consistent throughout the method. EPA revised Sections 11.2.2 and 11.2.3 to correct a typographical error, thus making treatment of Containers 2 and 3 consistent with Container 4, not Container 1. Regarding treatment of acetone rinses the same in Method 201A and Method 202, EPA has determined that quantitative transfer of particles in acetone from beaker to weighing tin is not necessary and adds unnecessary imprecision to final sample weight. Therefore, EPA has not revised the method (Section 11.2.4) to require this final transfer step consistent with Method 202 organic sample handling. Alternatively, EPA has changed the method to allow fluoropolymer beaker liners to be used to evaporate and weigh the samples.

Comment: (Comment No. 0050.1) Proposed Method 201A (74 FR 12985, Sections 11.2.2 and 11.2.3) Analytical Procedures (for Container # 2 and Container # 3 respectively): “Separately treat this container like Container # 1.” The Alliance recommends the following language be added to Sections 11.2.2 and 11.2.3: “Note the level of liquid in the container, and confirm on the analysis sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use the methods, subject to the approval of the Administrator to correct the final result.”

Response: EPA has revised the method to require Container #2 and #3 to be treated like Container #4. Section 11.2.4, which describes the treatment of Container #4, includes the description of how containers should be handled after receipt from the field. EPA has determined that Section 11.2.4 already includes this language suggested by the commenter. Therefore, no additional changes to the method are necessary.

M201A	11.2.4 CONTAINER #4, ≤ PM2.5 MICROMETERS ACETONE RINSE OF THE EXIT TUBE OF CYCLONE IV AND FRONT HALF OF THE FILTER HOLDER
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Comment: (Comment No. 0038.1) Instructions direct that the contents of Container #4 be quantitatively transferred to a tared 250 ml beaker, evaporated to dryness at ambient

temperature and pressure, and weighed to a constant weight, presumably in the 250 ml beaker. It has been our experience that this portion of the sampling train often yields the smallest sample mass when compared to Containers #2 and #3. To expect the laboratory to get a quantifiable mass from this section of the sampling head while using a 250 ml beaker is not practical, as the gravimetric Limit of Detection (LOD) will quite often be higher than the actual mass of the residue present. To make this section consistent with instructions for handling liquid samples throughout the proposed Methods 201A and 202, it should contain language similar to that in the proposed Method 202 Section 11.2.2 – Organic Fraction Weight - “Quantitatively transfer the contents to a clean glass beaker, and evaporate at room temperature (not to exceed 30°C (85°F)) and pressure in a laboratory hood to approximately 10 ml. Quantitatively transfer the beaker contents to a 50 ml pre-weighed tin, and evaporate to dryness at room temperature (not to exceed 30°C (85°F)) and pressure in a laboratory hood. Following evaporation, desiccate the residue for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh at intervals of at least 6 hours to a constant weight (i.e., ≤ 0.5 mg change from previous weighing), and report results to the nearest 0.1 mg.”

Response: EPA agrees with the commenter that weighing small masses in high mass containers leads to increased bias. EPA has modified the Section 11.2.4 of the method to allow the use of tared PFTE beaker liners.

Comment: (Comment 0120) Section 11.2.4 says to report the results to the nearest 0.1 g. We assume this is a typographical error and should read 0.1mg.

Response: We agree with the commenter that Section 11.2.4 contains a typographical error in the reporting units. The final rule was revised to change the reporting units from "g" to "mg."

M201A 11.2.7 CONTAINER #7, ACETONE RINSE BLANK

Comment: (Comment No. 0038.1) Section 8.7.5.5(g) specifies that 100 ml of acetone be collected as an Acetone Rinse Blank and analyzed per instructions in Section 11.2.7. Further, instructions in Section 11.2.7 direct that 100 ml of acetone be reduced for determination of the dry residue mass. We feel that a 100 ml volume specified in Section 11.2.7 for reduction is too

small.

Response: See response to previous comment on Section 8.7.5.5 of this document.

Comment: (Comment 0120) Section 11.2.7 has us evaporating our acetone blank to 10 ml, then transferring the contents of the beaker to a pre-weighed tin for final evaporation and weights. Section 11.2.4 has us fully evaporate and weigh the sample in the 250 ml beaker. Is this correct? Why would we handle a blank any differently than handling a sample?

Response: We have revised Sections 11.2.4 and 11.2.7 to be consistent and allow evaporation of the acetone rinse for both the sample and the blank at room temperature in a tared glass beaker or a PFTE beaker liner.

Comment: (Comment No. 0047.1) The proposed blank procedure in Method 201A, Section 11.2.7, will likely skew the blanks low due to sample loss due to residual material remaining in the equipment after transfers. EPA should utilize a method of determining blanks which will minimize sample loss.

Response: EPA understands the commenter's concern that multiple transfers of blank samples during the evaporation can add bias to the results. EPA has determined that transfer of dissolved solids can be performed quantitatively within the bias and precision requirements of the method based on laboratory experiments (*Evaluation and Improvement of Condensable Particulate Matter Measurement* (see Docket no. EPA-HQ-OAR-2008-0348-0028)). However, EPA has added the alternative of using tared fluoropolymer beaker liners to 11.2.7 of Method 201A for evaporation and residual weight determination.

M201A 12.0 CALCULATIONS AND DATA ANALYSIS

Comment: (Comment No. 0058.1) The term Q_{IV} (Sampling rate for cyclone IV to achieve D_{50}) appears in the nomenclature list in Section 12 (Calculations and Data Analysis) but does not appear anywhere else in the method. Is Q_{IV} supposed to be used in any other calculations?

Response: Section 8.4 of the method requires pre-test calculations to help select the appropriate gas sampling rate (Q_s) through cyclone I and cyclone IV. The sampling rate must

meet the requirements in Table 2 of Section 17, which lists the range in particle size allowed for the D_{50} of each cyclone. Proposed Figure 9 of Section 17 shows the acceptable sampling rate for the combined cyclone heads. Pretest calculations in Table 3 are based on the sampling rate of cyclone I because the PM_{10} D_{50} is more sensitive to the sampling rate than cyclone IV. Adding the $PM_{2.5}$ cyclone to the sampling head reduces the range of acceptable D_{50} values by removing the lower portion of the 9 to 11 μm PM_{10} range. As required in Section 8.4.2, testers must confirm that the sampling rate they select as Q_s based on cyclone IV equations meets the requirements for both cyclones as shown in final Figure 10. A number of approaches may be used to choose a flow rate within the limits of Q2.25 and Q11 for the combined $PM_{2.5}/PM_{10}$ cyclone head, however, the approaches chosen for this method use the limiting flowrates corresponding to cyclone I D_{50} values and converts the chosen D_{50} into a corresponding flowrate.

If sampling is done for only $PM_{2.5}$, then additional terms are necessary to calculate the sampling rate in the method. Testers can either use the same approach to select sampling rate for $PM_{2.5}$ -only cyclone assemblies and recognize that at the lower temperatures the range flow rate is more restrictive than necessary or use the additional equations in this method. Calculations of flow rate based on cyclone I assumes that the Cunningham correction factor is unity (1) removing that term from the flow rate calculation. The Cunningham factor is used in equations to calculate the sampling rate when $PM_{2.5}$ cyclone sampling heads are used without the PM_{10} cyclone. To determine the full extent of the flow rate that meets the limits for the cut point using cyclone IV only, EPA has added requirements and calculations necessary to perform only $PM_{2.5}$ measurements when the PM_{10} cyclone is not necessary.

For Reynolds numbers less than 3,162:

$$Q_{IV} = (0.0060639 \times (\mu/C^{0.4242}) (P_s M_w/T_s)^{-0.5759} (1/D_{50})^{0.8481}$$

For Reynolds numbers greater than 3,162:

$$Q_{IV} = (0.007657 \times (\mu/C^{0.6205}) (P_s M_w/T_s)^{-0.3795} (1/D_{50})^{1.241}$$

Where

Comment: (Comment No. 0058.1) Section 12 describes the iterative process to determine the correct D_{50} value (using Z-ratio) on cyclone IV. Is this same iterative process also to be used on the D_{50} value for cyclone I? If so please clarify in the method text. (note that on

page 12990 the text for Re-Estimated Cunningham Correction Factor refers to Eq. 32 — the cyclone I D_{50} equation).

Response: EPA agrees with the commenter that the final method should be clarified. Because the Cunningham correction factor for a 10 μm particle is so close to the unity, iteration is not required for Cyclone I. However, this is not the case for $\text{PM}_{2.5}$ particulate. Therefore, iteration as required in the method is needed for Cyclone IV, where both the Reynolds number and Cunningham correction can change significantly.

M201A 12.1 NOMENCLATURE

Comment: (Comment No. 0039.1) For V_{aw} , the word “blank” should be removed from this definition.

Response: EPA agrees with the commenter that “blank” should be removed from V_{aw} . In addition, EPA corrected the definition of the term V_{aw} to clarify that V_{aw} is the volume of acetone used in the sample recovery wash.

M201A 12.3.2.3 PARTICULATE WEIGHT CATCH PER SIZE FRACTION

Comment: (Comment No. 0038.1) Specification for the maximum allowable acetone blank value needs to be changed from 0.001 percent by weight to either 1 ppmw or 0.0001 percent by weight to be consistent with the reagent specification stated in Section 7.2.1.

Response: We agree with the commenter that maximum allowable acetone recovery blank value should be consistent with the reagent specification stated in Section 7.2.1. Thus, we revised Section 12.3.2.3 of the final method to specify the maximum allowable residual mass in the acetone recovery blank in terms of weight per volume of acetone (0.1 mg per 100 ml solvent), rather than percent weight.

Comment: (Comment No. 0038.1) Subtracting the acetone blank mass from the individual sample masses would be fine if the volumes of the acetone rinses are all exactly 100 ml. However, this is not reality, and the accuracy of determining the blank correction suffers

from this approach. Also, given the previous discussion on Sections 8.7.5.5(g) and 11.2.7 above, regarding gravimetric LOD and the suggested Acetone Rinse Blank volume, it is suggested that rather than subtracting the mass of the Acetone Rinse Blank dry residue directly from the sample masses M2, M3, and M4, the concentration of the Acetone Rinse Blank be calculated as the mg of dry residue per ml of Acetone Rinse Blank Volume limited to a maximum of 0.00079 mg/ml (concentration of residue at 1 ppmw). [See DCN: EPA-HQ-OAR-2008-0038.1 for equation.] This concentration of the dry residue would be multiplied by the volume of the acetone in ml used to collect and recover each sample from the sampling head. The resulting mass would be subtracted from the dry residue mass determined for the sample of interest. This approach will provide: 1) a more accurate determination of the dry residue mass from the Acetone Rinse Blank due to processing a larger volume of acetone, and 2) the blank mass correction for each sample will be more accurately assessed as it will be proportional to the amount of acetone used to collect each sample. The liquid volume of the samples and blanks could be determined by either direct volumetric measurement or by multiplying the wet weight of the sample or blank by the density of the reagent at 20°C. The equation above and suggested procedure for determining the blank correction for each of the sample masses M2, M3, and M4 would replace Equation No. 41 for determining the dry residue concentration for the acetone blank (Ca). The commenter suggested modifications to Equation No. 42 (see DCN:EPA-HQ-OAR-2008-0038.1). Where $W_{a(2,3,4)}$ denotes the weight correction for each individual sample mass M2, M3, and M4, respectively, and $V_{(2,3,4)}$ denotes the volume for the individual acetone rinses, containers 2,3, and 4, respectively. For example, to determine the mass correction for the dry residue from container #3, the equation would be of the following form: [See DCN:EPA-HQ-OAR-2008-0038.1 for equation.]

(Comment No. 0038.1) Section 12.3.2.3 directs that the mass of the acetone blank weight determined in Section 12.3.2.2 be subtracted from each size fraction and “Do not subtract a blank value of greater than 0.001 percent of the weight of the acetone used from the sample weight.” The term “acetone used” is ambiguous as it does not specify whether it is referring to acetone used for the Acetone Rinse Blank or the acetone used in collecting the individual samples.

(Comment No. 0038.1) The language “Subtract the weight of the acetone blank from the particulate weight catch in each size fraction” in Section 12.3.2.3 is ambiguous when combined with the note in Section 12.3.2.2 to “Correct each of the particulate matter weights per size

fraction by subtracting the acetone blank weight (that is, $M_{2,3,4} - W_a$).” It is unclear whether the acetone blank weight is to be subtracted from each of the sample masses M_2 , M_3 , and M_4 individually, or whether the acetone blank weight is to be subtracted from the summation of masses for each particle size fraction for PM ($M_{1,2,3,4} - W_a$), PM_{10} ($M_{1,3,4} - W_a$), and $PM_{2.5}$ ($M_{1,4} - W_a$). Language in Sections 12.3.2.2 and 12.3.2.3 need to be revised by dropping the term “size fraction” and using language that clearly directs the user to “subtract the Acetone Blank Mass from each sample mass (M_2 , M_3 , and M_4) individually.”

Response: EPA has revised Sections 12.3.2.2 and 12.3.2.3 of the method to clarify how acetone field blank values are used to correct the particulate mass collected in each fraction of the sampling train catch. Both the text and formulas have been revised to provide this clarification.

We agree with the commenter and agree with the commenter’s suggested equation. EPA revised Section 12.3.2.3 of the final the method to accommodate different acetone rinse volumes, as follows.

$$M_{(2,3,4)} = W_{(2,3,4)} - W_{a(2,3,4)}$$

However, the correction must be proportional to the amount of solvent used. Some testers may use more solvent due to heavy deposits that are difficult to remove, while other testers may use less solvent. We revised Section 12.1 of the final method to add variables (m_2 , m_3 , m_4) for the uncorrected residual weight of each fraction. This allows correction of the mass in each fraction for the weight (W_a) of the residual acetone to recover samples in each fraction.

We have clarified the definition of each of the factors used in the equations for blank correction due to acetone residual. The acetone used for the blank determination should be taken from the field container used to rinse and recover PM from sampling train components. The acetone field recovery blank should be used only to establish the potential residue per volume of acetone solvent used to recover the sample. The maximum field recovery blank value that can be subtracted is 0.0001 percent (1 ppmw or 0.1 mg/100 ml of acetone). (See also Section 7.2.1 of this document and the method, which repeat this maximum allowable correction for acetone residual: Any container material is acceptable for wash bottles and storage bottles, but the container must not contribute more than 0.1 mg of residual mass to the CPM measurements.)

Comment: (Comment No. 0037) The proposed 201A does not very clearly state the requirement for isokinetic. Section 8.3.4 says to void the sample if the range is not met, but does not state the range. It is not until Section 8.5.4 where selecting a nozzle is discussed that a range is mentioned. I think it would be a good idea to add language specifying that the isokinetic must meet 80 to 120 percent either at Equation 40 in Section 12.5 or in Table 6. This will save state agencies from a lot silly arguments with testing firms.

Response: EPA agrees with the commenter that the required deviation from isokinetic sampling is not clearly communicated in the method. While the acceptable minimum and maximum deviation from isokinetic sampling is stated in Section 8.5.4, we have added a clarification to Section 8.3.4 (b) to specify the acceptable variation from isokinetic sampling as 80 to 120 percent and no more than 29 percent (2 out of 12 or 5 out of 24) sampling points out of this criteria.

Comment: (Comment No. 0038.1) Equation 13 could be simplified by using the previously calculated value for nozzle diameter (D) and the previously calculated sampling rate (Q_s) along with the constant 3.506 from Equation No. 12 to determine the nozzle velocity (V_n). [See DCN:EPA-HQ-OAR-2008-0038.1 for equation.]

Response: We acknowledge the commenter's suggestions and agree that the velocity of gas in the nozzle V_n can be calculated using the commenter's equation. We also acknowledge that the commenter's equation can be found in the original Method 201A and in the literature that supports this method revision. Therefore, EPA has revised Equation 13 using the simplification recommended by the commenter.

Comment: (Comment No. 0056.1) API questions some of the equations presented in the March 25, 2009 Federal Register presentation of Proposed Method 201A. The following corrections are needed: (1) Equation 4 uses D_p instead of D_{50N} (2) Equation 10 should be identified as being used for N_{re} greater than 3162 just below the Equation 10 label. (3) Equation 24 does not differentiate the Pitot constants (C_p and C_p') for the preliminary and sampling probes. C_p' should be in the denominator. The equation was stated correctly in the EMC

website version of the method but was not correct in the March 25, 2009 Federal Register version. (4) Equation 26 does not subscript the numbers in Aps1 and Aps2. (5) Equation 34 should be identified as being used for N_{re} greater than 3162 just below the Equation 34 label. (6) Equation 37 should be identified as being used for N_{re} greater than 3162 just below the Equation 37 label. (7) Equation 40 is missing the sampling time (0) in the denominator. This equation was stated properly in the EMC website version of the method but was incorrect in the March 25, 2009 Federal Register version of the method.

Response: Regarding (1) Equation 4, the commenter is correct. While the equation is correctly written for any particle size, this application should use D_{50} for Cylone IV (2.5 μm). Regarding (2) Equation 10, Equation 5 covers N_{re} less than 3162. While the commenter correctly advises that the equation is different from the Southern Research Institute reference on the subject, excluding N_{re} equal to 3162 presents a logical gap in the calculations and EPA has chosen to include this condition in Equation 10. Inclusion of the N_{re} designation is redundant with the header label for the equation.

Regarding (3) Equation 24, this was a typographical error in the Federal register. OTM 27 posted on the EPA Web site is correct. The equation was stated correctly in the EMC Web site version of the method, but was not correct in the March 25, 2009 Federal Register version. Regarding (4) Equation 26, the commenter is correct, the numbers should be subscripted as follows: ΔP_{s1} . OTM 27 posted on the EPA Web site is correct. Regarding (5) and (6) Equations 34 and 37, the commenter is correct. We revised the final method to remove the N_{re} notes under the equations. OTM 27 posted on the EPA Web site is correct. While the commenter correctly advises that the equation is different from the Southern Research Institute reference on the subject, excluding $N_{re} = 3162$ presents a logical gap in the calculations and EPA has chosen to include this condition in Equation 34. Inclusion of the N_{re} designation is redundant with the header label for the equation.

Regarding (7), the commenter is correct; Equation 40 is missing the sampling time (0) in the denominator. This equation was stated properly in the EMC Web site version of the method, but was incorrect in the March 25, 2009 Federal Register version of the method. EPA revised the final method to include the sampling time in the denominator.

Comment: (Comment No. 0112) If only the PM_{2.5} head is used (without the PM₁₀ head), does this affect the equations shown in the method? OTM 27 states that sampling can be performed using only the PM_{2.5} head, but I don't see where it mentions if the equations need to be modified. I am talking specifically about equations that directly affect the sampling rate and PM_{2.5} cut diameter. I am assuming that the equations don't need modification, but want to verify that this is correct. We are starting our 1st test program on Monday and the test port will not accommodate a PM₁₀/PM_{2.5} head configuration.

Response: In the final Method 201A, the equations are the same for all the configurations of the particle-sizing cyclones. However, if the tester does not use the PM₁₀ cyclones Equation 33 (Actual Particle Cut Diameter for Cyclone I) is a not applicable.

M201A 17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Comment: (Comment No. 0056.1) API believes that it might be necessary to adjust the sampling rate curve shown in Figure 9 of Proposed Method 201A when gas stream conditions differ significantly from the ambient air-oriented carbon dioxide, oxygen, and moisture levels listed at the right corner of Figure 9.

Response: EPA has determined that the differences in gas density and gas viscosity between ambient air and flue gas are small enough that the figure is correct as a starting point for calculating the compromise flow rate. An alternative way of calculating the compromise flow rate is to calculate the flow required for both a 10 µm cut for cyclone I and 2.5 µm cut for cyclone IV and averaging the two values. Regardless of the method used to calculate the compromise flow rate, testers must determine the compromise flow rate that meets the isokinetic requirements stated in the method.

SECTION 3.0
METHOD 202—DRY IMPINGER METHOD FOR DETERMINING
CONDENSABLE PARTICULATE EMISSIONS FROM STATIONARY
SOURCES

Section 3.0 of this document is organized by the section numbers of the proposed Method 202.

M202 GENERAL COMMENTS

Comment: (Comment No. 0061.1) PCA supports EPA’s efforts to upgrade “old” Method 202. PCA is aware that numerous researchers have determined that existing Method 202, as developed in the early 1990s, is subject to significant bias in some applications due to the absorption and aqueous phase oxidation of dissolved SO₂ to form sulfates. This artifact problem goes well beyond SO₂ and includes other soluble gases including, but not necessarily limited to ammonia and nitrogen dioxide. For example, the absorption of ammonia present in low concentrations in some effluent gas streams can enhance the absorption and aqueous phase oxidation of SO₂.

(Comment No. 0056.1) API supports EPA’s efforts to upgrade existing Method 202. API is aware that numerous researchers have determined that Method 202, as developed in the early 1990s, is subject to significant bias in some cases due to the absorption and subsequent aqueous phase oxidation of dissolved SO₂. API believes that this artifact problem goes well beyond SO₂ and includes other soluble gases including, but not necessarily limited to ammonia, nitrogen dioxide, alcohols, aldehydes, and other water-soluble organic compounds. While API recognizes the benefits of rapid post-test purging advocated by EPA, API agrees with statements in a number of published technical papers that rapid purging is not sufficient to avoid significant artifact problems.

Response: We acknowledge the commenter’s support for our efforts to improve the existing method. With regard to the commenter’s assertion that rapid purging is not sufficient to avoid significant artifact problems, the laboratory experiments conducted by EPA (Evaluation and Improvement of Condensable Particulate Matter Measurement (Docket no. EPA-HQ-OAR-

2008-0348-0028)) show that the nitrogen purge reduces the artifact to the blank level specified in the method.

Comment: (Comment No. 0083.1) Air Control Techniques, P.C. appreciates the open and constructive process used by EPA in the development of Proposed Method 202. Since we proposed the dry impinger modifications to Method 202 in 2005 (June 13, 2005 letter to Mr. Tom Logan, November 2, 2005 paper at the AWMA Hazardous Waste Combustion Conference), EPA and other stakeholders participating in the development effort have added a number of enhancements to this method. Proposed Method 202 as published on March 25, 2009 provides an effective and economical means to compile CPM emissions data for stationary sources. Adoption of Proposed Method 202 based on some of the recommendations included in the docket will also improve this method. When we first proposed the dry impinger sampling train modifications that provided the foundation for Proposed Method 202, we envisioned it as an improvement to existing Method 202. The dry impinger method retains the good features of existing Method 202 while minimizing the bias to higher-than-true measured concentrations caused by the aqueous phase oxidation of soluble gases to form CPM. The mandatory inclusion of the filter minimizes the potential bias to lower-than-true measured concentrations of condensed PM due to incomplete capture of submicrometer-sized particles forming in the impingers.

Response: We acknowledge the commenter's support.

Comment: (Comment No. 0086.1) API has previously commented that Proposed Method 202 can be performed effectively by testing/engineering organizations having experience with complex test methods. API noted that no special sampling equipment is needed. The sampling run times needed in this method are consistent with many other EPA reference methods. Proposed Method 202 provides an effective and economical way to obtain CPM emissions data while dilution-based methods are being developed. In response to the Barr Engineering comments, it is important to note that Proposed Method 202 uses standard glassware that has been available for over twenty-five years, involves sample recovery procedures similar to existing Method 202, and requires the use of the nitrogen purge that was previously considered optional. API is aware that some test companies have been using the predecessor method, OTM

028, for over three years without any significant problems. Proposed Method 202 is considerably simpler than many other test methods already in common use. Furthermore, Proposed Method 202 is far less complicated and costly than EPA Method CTM 039 based on high volume dilution. API believes that those testing organizations inexperienced with Proposed Method 202 can quickly become proficient with this moderately complex test method.

(Comment No. 0083.1) There are several comments included in Docket EPA-HQ-OAR-0348 that suggest that Proposed Method 202 is too complex. We disagree. Air Control Techniques, P.C. has been using the predecessor method that is presently designated as OTM 028 for four years in a variety of industrial applications and find that this method is no more complex than many other EPA reference methods. We do not believe that most other experienced testing firms have had major problems with this method.

Response: We acknowledge the commenter's support for the proposed revisions to Method 202.

Comment: (Comment No. 0044.1) EPA should clearly state what happens to OTM 028 (Dry Impinger Method for Determining Condensable Particulate Emissions from Stationary Sources). We are assuming that this OTM method will no longer be used, and would be deleted from the record.

Response: The commenter's assumption is correct. After the proposed revisions to Method 202 have been finalized, we will remove OTM 028 from EPA's website for Other Methods (<http://www.epa.gov/ttn/emc/prelim.html>).

Comment: (Comment No. 0050.1) The Alliance recommends that EPA number the final approved test method as Method 202A. Replacing the existing Method 202 with a revised testing method with the same number (202) will create tremendous confusion in the future as testing results are compared and summarized. Other organizations, such as ASTM International normally renumber or add a date, for example, to the new or modified testing method in order to distinguish it from other similar or previous methods. Several EPA approved testing methods employ this concept, using a different numbering sequence to ensure there is no confusion in which test method and procedure applies. For example, EPA Method 3 includes Methods 3a, 3b, and 3c. EPA testing Methods 6 includes 6a, 6b, and 6c. Yet another example would be Methods

201 and 201A. These are all unique tests methods, as will be the modified Method 202.

Therefore, the Alliance suggests the revised method be numbered Method 202A.

(Comment No. 0064.1) If EPA allows substantial variation in conducting Method 202, then EPA must assign each substantial variant a separate method number (e.g., 202A, 202B, etc.) to avoid data interchangeability issues. The variations allowed in Method 202 create the possibility of divergent results merely because different allowable techniques were used. EPA's Emission Measurement Center, in fact, has acknowledged that Method 202 is more a "family" of emission measurement techniques than a true method. In its "Frequently Asked Questions" page (<http://www.epa.gov/ttnlenc/methods/method202.html>), EMC states:

Does EPA Method 202 provide reproducible results? When conducted consistently and carefully, EPA Method 202 does provide acceptable precision for most emission sources. However, several options are allowed by the method to accommodate State/local test methods that existed at the time the method was proposed and promulgated in the Federal Register. Each of these options may change the mass that would be counted as condensable particulate matter. As a result, when the same source is tested using different options allowed by the method there may appear to be a large variation of the condensable particulate emissions. In addition, the flue gas characteristics may exacerbate the perception of the amount of variation that is introduced by the optional procedure. For example, under specified conditions, EPA Method 202 allows the one hour nitrogen purge to be replaced with air or not conducted when specified conditions exist. Each of these options results in more SO₂ remaining dissolved in the impinger water. The dissolved SO₂ slowly converts to SO₃ and then to H₂SO₄. While the SO₂ should not be counted as condensable particulate matter, both SO₃ and H₂SO₄ form particulate matter. As a result, EPA Method 202 should not be considered to be a single standardized test method, but should be considered to be a collection of test methods. Therefore, when EPA Method 202 is specified as the applicable test method, any optional procedures should also be specified in order to achieve results that are more in agreement with the basis of the specified emission limitation.

While EPA states that it has reduced the number of these variants, significant variations still remain. EMC has stated that "any optional procedures should also be specified" to preserve replicability. Because there is no effective way to specify optional procedures, EPA should establish separate method numbers to reflect significant variations where there is reason to believe that use of one method variant would not lead to the same (or substantially similar) value as another variant.

Comment: (Comment No. 0044.1) EPA should consider designating the proposed method as Method 202A and retain the existing Method 202 as an optional test method. In some

situations, the existing Method 202 may be more reasonable to use and would be much more cost effective.

Comment: (Comment No. 0056.1) In compiling emission inventories, API recommends that EPA consider CPM emissions data measured in accordance with OTM 028 to be equivalent in quality with Proposed Method 202. CPM data obtained using the previous version of Method 202 should be categorized separately due to the potential artifact biases and the inconsistencies in the method application by various testing organizations.

Response: We disagree that EPA should number the final approved test method as Method 202A. The final Method 202, reflecting the proposed revisions, will replace the version of Method 202 originally promulgated on December 17, 1991 (56 FR 65433) as contained in Appendix M of 40 CFR Part 51. The final Method 202 will effectively replace the original (1991) method to provide a test method for measuring CPM from stationary sources. Additionally, we will post the final Method 202 on EPA's list of reference test methods located at <http://www.epa.gov/ttn/emc/promgate.html>. We disagree with commenters that the original Method 202 promulgated in 1991 should be retained after the changes proposed to Method 202 have been finalized. We believe that retaining both versions of method would lead to confusion among the testing and regulatory communities. Additionally, retaining the current (1991) version of Method 202 would potentially propagate the measurement errors caused by artifact formation that the final method seeks to address. In addition, regulatory agencies and regulated source operators will avoid the time and expense of modifying existing permits to change the name of the test method requirements.

Comment: (Comment No. 0056.1) API supports the designation of Proposed Method 202 as a replacement for the previously adopted Method 202. Proposed Method 202 is a logical and effective enhancement to "old" Method 202 and should not be considered as a different or parallel method. There is no value in retaining "old" Method 202 considering that the biases and variations in this old method are well documented. There is no benefit to the public, regulatory agencies, or the regulated community to continue to compile CPM data that can be substantially in error. By designating this enhanced CPM measurement method as Proposed Method 202, EPA has helped regulatory agencies and regulated source operators avoid the significant time and expense involved in modifying existing permits with respect to test method requirements.

Following the promulgation of Proposed Method 202, the previous version of this method should be removed from the list of EPA Reference Methods.

(Comment No. 0061.1) PCA supports the designation of Proposed Method 202 as a replacement to the previously adopted Method 202. Proposed Method 202 is a logical and effective enhancement to “old” Method 202 and should not be considered as a different or parallel method. There is no benefit to the public, regulatory agencies, or the regulated community to continue to compile CPM data that can be substantially in error. By designating this enhanced CPM measurement method as Proposed Method 202, EPA has helped regulatory agencies and regulated source operators avoid the significant time and expense involved in modifying existing permits with respect to test method requirements.

(Comment No. 0086.1) API disagrees with the continued use of existing Method 202. Existing Method 202 is subject to well-documented bias problems in many applications and these problems cannot be adequately avoided by rapid purging or other quality assurance steps. Following the promulgation of Proposed Method 202, for serious data quality reasons, the previous version of this method should be removed from the list of EPA Reference Methods.

(Comment No. 0083.1) There is no need to retain existing Method 202 as an alternative to Proposed Method 202.

Response: We agree with the commenters that the final Method 202, reflecting the proposed revisions, should replace the version of Method 202 originally promulgated on December 17, 1991 (56 FR 65433). The promulgation of amendments to Method 202 will revise the appropriate sections of Method 202 currently contained in Appendix M of 40 CFR Part 51. The changes to Appendix M will effectively replace the original (1991) method to provide a test method for measuring CPM from stationary sources. Additionally, we will post the final Method 202 on EPA’s list of reference test methods located at <http://www.epa.gov/ttn/emc/promgate.html>.

Comment: One commenter (0050.1) stated that a general flexibility provision should be provided at the beginning of the method to respond to the experiences that will be acquired as the methods are field-applied. A general flexibility provision should be provided at the beginning of each method to respond to the experiences that will be acquired as the methods are field-applied.

(Comment No. 0051.1) CIBO supports the Alliance suggestion for a general flexibility provision at the beginning of each method, as more fully described in the Alliance comments. Given the wide range of variables for any testing scenario, on-site operators need flexibility to ensure tests are accurate and meet all applicable methods and contain costs. The likelihood of increased complexity during field testing merits increased flexibility within RM 202 than is currently written.

Response: The commenters did not provide any examples of flexibility that they believe should be in the methods. In addition, because the end point is defined by the method, this method is not amenable to a general flexibility measurement approach statement. The unrevised methods provided more flexibility, but they were criticized for lack of precision. However, based upon field experiments and method evaluations, we have revised the final methods to include as much flexibility as possible while retaining precision.

If the source has unique characteristics that require procedures that vary from the published method, the source should petition the appropriate regulatory agency for approval to use an alternative procedure or test method.

Comment: (Comment No. 0056.1) Proposed Method 202 for the measurement of primary CPM has been in use since 2005 as OTM 028. API believes that this method provides accurate and precise data when used by qualified individuals who adhere to OTM 028 sampling procedures and quality assurance steps. This new method eliminates more than 90 percent of the artifact bias due to the aqueous phase reactions of soluble gases. Furthermore, the use of the between-impinger filter eliminates the negative bias that has often been suspected to occur due to the formation and penetration of submicrometer-sized condensed particles in the water-filled impingers of “old” Method 202.

(Comment No. 0061.1) Proposed Method 202 for the measurement of primary CPM has been in use since 2005 as OTM 028 (40 CFR part 51, appendix A). This method provides accurate and precise data when used by qualified individuals who adhere to the Proposed Method 202 sampling procedures and quality assurance steps. This new method eliminates approximately 90 percent of the artifact bias due to the aqueous phase reactions of soluble gases. Furthermore, the use of the between-impinger filter eliminates the negative bias that has often

been suspected to occur to the formation and penetration of submicrometer-sized condensed particles in the water-filled impingers of old Method 202.

Response: We acknowledge the commenters' support for OTM 028.

Comment: One commenter (0051.1) stated that he believes that the proposed amendments to Method 202 may still be lacking with regard to the method precision in extreme source conditions such as where ammonia, SO₂, and relatively high moisture content are all present. The commenter encouraged EPA to provide more evidence than the one limited experiment to date, that the amended method satisfactorily perform as proposed where ammonia, SO₂, and relatively high moisture content are all present.

The commenter said that consideration should be given to mimicking the parameters of a source category (e.g., secondary aluminum metallurgical furnaces with intermittent burner flame direct impingement onto furnace contents, and employing free ammonia injection for hydrogen chloride emission control) when selecting the synthetic gas stream test matrix for evaluating the performance of Method 202. The commenter said that the diluent gas would be much closer to ambient air, with oxygen content near 20 percent by volume (dry basis), rather than 7.5 percent. The 11 to 12 percent moisture content and 22 to 25 parts per million by volume (ppmv) SO₂ concentration from the previously described experiment would continue to be representative in extreme here. However, for purely stoichiometric purposes, the volumetric concentration of ammonia should be at least twice the SO₂ concentration, since two molecules of ammonia per molecule of SO₂ are required to form the ammonium sulfate artifact. A target concentration of 75 ppmv ammonia would be within the realm of reasonableness for the metallurgical furnace source category, since the intermittent burner flame direct impingement onto chloride fluxes in the furnace results in wide fluctuation in off gas stream acidity, and in associated wide fluctuation in ammonia injection regulated by gas stream pH controller. Such fluctuation can lead to greater ammonia slip, due to response lag time with the pH controller. With the ammonia target level being 1.5 times the stoichiometric balance with SO₂ for ammonium sulfate formation, this could provide an alkaline condition to condensed moisture in the dry impingers that may have been lacking before.

The commenter also believed that further experimentation should include a study of the post-test nitrogen purge efficacy. Two identical parallel sampling trains should be employed

(with no need seen to continue the 160°F filter component). At the conclusion of the sample collection period, the two Method 202 impingers should be quickly post weighed, with measured amounts of the reagent water added to both "dry impingers" of each sampling train to above the level of the impinger tips. After swirling the added water around each impinger to solubilize as much of the analytes on glassware surfaces as possible, the solution pH of each is measured and an aliquot from each impinger solution is taken for IC analysis. As quickly as these steps can be completed the one-hour post test nitrogen purge is commenced on only one of the two sampling trains, with the other train held for a period of time (two hours) before commencing its post test nitrogen purge. After the purges have been completed, the impingers are again weighed for determining any changes in solution volumes, and an additional aliquot from each impinger solution is taken for IC analysis. Remaining sample recoveries and analyses can be completed as normal. The comparison of the pre and post purge IC analytical results for the ions NH_4^+ and SO_4^- will answer questions about the effectiveness of the post test nitrogen purge for SO_2 removal when ammonia is present, and about the necessity for expediency in commencing the nitrogen purge. If the ions NH_4^+ and SO_4^- are significantly present in the aliquots collected prior to purging, but are absent from the aliquots collected after purging, this, for example, would alleviate much of the concern about the results of the previous experiment. We are not aware in the record where EPA has yet addressed these questions with regard to the presence of ammonia in stoichiometric excess.

(Comment No. 0049.1) UARG's consultant RMB Consulting & Research, Inc. (RMB) is disappointed that EPA elected to "declare victory" in 2007 after limited laboratory testing and failed to conduct any field evaluations of the proposed amendments to Method 202. RMB believes EPA's statements in the preamble regarding the effectiveness of the Agency's proposed modifications to Method 202 are far too optimistic – especially considering the paucity of field data. RMB encourages EPA to not be prescriptive with respect to mandating the use of amended Method 202; rather, the Agency should not only be receptive to but should encourage further improvements for measuring CPM emissions. In the meantime, RMB will continue to advise clients who are attempting to quantify CPM from coal-fired EGUs to consider simultaneously conducting amended Method 202 and CCM runs. The reason for this recommendation is RMB is not convinced (and we certainly have not seen data from coal-fired EGUs, especially those employing wet control technologies) that amendments to Method 202 have solved the bias issues

with existing Method 202. RMB believes the following procedure, although not as straightforward as we would like, is the most accurate approach for quantifying CPM from coal-fired EGUs: (1) Simultaneously conduct Method 202 and CCM test runs. (For wet FGD applications and assuming the nitrogen purge is used, RMB is not convinced there is significant benefit to amended Method 202 relative to the existing Method 202.); (2) For each test run, quantitatively determine the mass of sulfate collected in the Method 202 impingers and subtract this mass from the Method 202 inorganic fraction; and (3) From the corresponding CCM run, add the mass of $\text{SO}_3/\text{H}_2\text{SO}_4$ collected in the CCM train to the Method 202 inorganic fraction. RMB's rationale for this approach is as follows. The only certain inorganic condensable sulfate specie from coal combustion is $\text{SO}_3/\text{H}_2\text{SO}_4$. If the Method 202 results are free from sulfate artifacts, then we are basically subtracting x units of sulfate collected in the Method 202 train only to add back the same x units of sulfate from the CCM results. More often, sulfate artifacts do exist so we are subtracting y units of sulfate collected in the Method 202 train and adding back z units of sulfate from the CCM results and y is greater than z. RMB does not like having to recommend additional test runs, but it remains a better alternative than obtaining biased CPM results. RMB believes our approach also benefits EPA in that we cannot understand how the Agency's air quality planning challenge would benefit from inaccurate (biased) emission inventory data.

(Comment No. 0064.1) Several aspects of the proposed Methods will, in our opinion, result in significant experimental variability in measured mass emission rate of CPM. Emissions from steel mini-mills, such as operated by Nucor, and many other types of sources will contain soluble gases that are not properly categorized as CPM. However, the present Method 202 uses chilled, water-filled impinger devices that efficiently capture both the condensable constituents and some portion of these soluble gases, which include SO_2 . As pointed out in our comments contained herein the proposed Method 201A/202 may not offer a practical method to avoid this so-called "sulfate artifact," which tends to bias the CPM measurement high. Also, the practicality of the method for field use in a wide range of specific situations may be questioned. It is unfortunate that EPA apparently plans to promulgate an extensively revised reference method, while acknowledging it has numerous technical shortcomings.

Response: We acknowledge the concerns expressed by the commenters for using this method to sample and analyze CPM emitted under extreme source conditions. The EPA and a

stakeholder (EPRI) have evaluated a range of conditions that included high moisture and SO₂ concentrations. We have also evaluated the effect of ammonium from well-controlled sources and found no artifact formation. If significant CPM is found in emissions from sources operating at extreme conditions, we believe that these measurements represent particulate material that will form upon release to the ambient air. Therefore, this material would be counted as CPM when using the final method.

Numerous field tests of the revised method and EPA's evaluations of the revised test procedures at a hog-fuel boiler (see *Field Evaluation of an Improved Method for Sampling and Analysis of Filterable and Condensable Particulate Matter*; Docket no. EPA-HQ-OAR-2008-0348) showed acceptable performance of the fine particulate test methods under a variety of source conditions.

Devising a suite of controlled laboratory experiments to evaluate extreme ranges and unique operating conditions that are not representative of the vast majority of emissions sources conditions would be complex and highly uncertain. EPA has determined that the value of evaluating these extreme conditions is doubtful because there is no basis for determining beforehand which of the potential byproducts of the reactive gases would remain as gases in the ambient air or would combine to form PM. In some cases, reactive gases in these extreme process conditions are added and intended to combine with other gases included in the emissions to form particulate that is more easily controlled.

While SO₃ may be the most abundant CPM emitted from coal fired combustion, there is indication that other compounds comprise CPM. Few speciation tests of coal and oil combustion have been performed, but those that have indicated the presence of not only sulfate (SO₄²⁻) but also chloride (Cl⁻), nitrate (NO₃⁻), ammonium ion (NH₄⁺), and a range of inorganic elements that are potentially components for CPM including phosphorous, arsenic, and selenium. Therefore, the specific correction for H₂SO₄ from coal combustion source emissions proposed by the commenter adds to the complexity of the method for all sources while providing an advantage to only one specific source category. EPA recommends that such facilities handle this proposed alternative method with their regulatory authority.

EPA has determined that the revisions to Method 202 present significant improvements over the previous version of the method. Therefore, EPA believes that the revised method is generally applicable for measuring total CPM from stationary sources. However, EGU's can

petition their regulatory authority for approval of an alternate measurement procedure.

Comment: (Comment No. 0042) In response to the proposed method development work conducted by EPA staff, we conducted unique tests for the measurement of CPM on Unit 1 at our Pleasant Prairie Power Plant in January 2009. The Pleasant Prairie Plant is sub-bituminous coal-fueled and is equipped with SCR for NO_x reduction; cold side ESPs for particulate capture and wet FGDs for SO₂ capture. Tests were conducted simultaneously at the exit of the wet FGD in a “wet” stack using the three individual methods sampling trains. The tests included the former (Methods 201A and 202) as well as the amended EPA CPM measurement methods. In addition, we also included tests using the Controlled Condensate Method (CCM) for H₂SO₄ mist. A complete copy of this test program and its results is attached to our comment submittal. The test results indicate that the amended condensable particulate measurement method results are not significantly different from results obtained from the existing version of Method 202. When both of these results are compared with the H₂SO₄ mist measurements obtained with the CCM, a high bias relative to the CCM results was observed. (See EPA-HQ-OAR-2008-0042.1 for test data).

Response: We appreciate the test data submitted by the commenter. As stated in the method, we recognize the limitations of the revised method when it is applied to wet stack sampling conditions. We also would expect that the results obtained using the CCM, which measures only SO₃/H₂SO₄, would be different from the measurement of filterable and total CPM. The CCM tests performed by the commenter indicated that SO₃/H₂SO₄ analysis was by ion chromatography and included only sulfate analysis without analysis for chlorides, fluorides, phosphates, borates, carbonates, etc. For example, as indicated in Chow, J. C., et al. (see Docket No. EPA-HQ-OAR-2008-0348), additional elements such as Cl⁻, NO₃⁻, NH₄⁺, and a range of inorganic elements that are potentially components of CPM including phosphorous, antimony, arsenic, mercury, and selenium are contained in the inorganic samples obtained by dilution sampling. Chow determined that SO₃/H₂SO₄ accounted for 45 to 62 percent of the fine particulate mass. Based upon these findings, 38 to 55 percent of the fine particulate mass could be excluded in CCM measurement. The additional chemical species would be expected to be components of the CPM in samples from both the unrevised and revised Method 202.

Comment: (Comment No. 0064.1) The sampling train design and sample recovery steps may introduce experimental error such that even carefully performed tests have unacceptable precision. For condensables measured by Method 202, EPA acknowledges the increased complexity and effort for glassware cleaning prior to each test and the numerous additional steps required to recover, extract, and analyze the samples. It is noted that each additional analytical step, particularly when handling the CPM filter, has the potential to increase method error. Due to sheer number of such transfer and sample analysis steps, even a carefully conducted test may fail to achieve the stated precision goal. Experience in the field suggests some methodology changes that will improve consistency, but not address the flaws in the revised Method 202.

Response: We disagree with the commenter that the stated precision of the method cannot be achieved due to the complexity of the sampling and analysis procedures. Our laboratory studies and field evaluations have demonstrated that the procedures of the revised method, when properly applied, generate results of acceptable precision. (See *Evaluation and Improvement of Condensable Particulate Matter Measurement* (Docket no. EPA-HQ-OAR-2008-0348-0028) and *Field Evaluation of an Improved Method for Sampling and Analysis of Filterable and Condensable Particulate Matter*; Docket no. EPA-HQ-OAR-2008-0348). In addition, other commenters (0056.1, 0061.1, 0083) support revisions to Method 202 with minor adjustments. Commenters 0056.1 and 0061.1 stated that the method provides accurate and precise data when used by qualified individuals who adhere to the sampling procedures and quality assurance steps. Commenter 0083.1 added that the proposed method provides an effective and economical means to compile condensable particulate matter emissions data for stationary sources. In addition, commenter 0083.1 stated the proposed method retains the good features of existing Method 202 while minimizing the bias to higher-than-true measured concentrations caused by the aqueous phase oxidation of soluble gases to form condensable particulate matter. Commenters 0056.1 and 0061.1 stated that the proposed method eliminates approximately 90 percent of the artifact bias due to the aqueous phase reactions of soluble gases. All three commenters (0056.1, 0061.1, and 0083.1) stated that the mandatory inclusion of the between-impinger filter eliminates the negative bias that has often been suspected to occur due to the formation and penetration of submicrometer-sized condensed particles in the water-filled impingers of the existing version of Method 202. Commenter 0083.1 disagreed with other commenters that state the method is too complex. Commenter 0083.1 found that the method is

no more complex than any other EPA method. Revised Method 202 contains improved techniques and procedures that prevent or reduce the contamination of the sampling device and associated analytical hardware. These improved techniques are not beyond the capabilities of source testing companies that have the goal of controlling contamination. As indicated by other commenters, the required techniques are not that dissimilar from other EPA test methods and are less demanding than some EPA test methods. We have included the use of field recovery blanks for use by the tester and the regulatory agency to assess field and laboratory conformance with good practices for field and laboratory equipment preparation, recovery, and analysis. Also, between the reagent blank determination and the field recovery blank assessments, information is provided that indicates initial areas to investigate to improve techniques.

Comment: (Comment No. 0059.1) The proposed method did not list anything for QC blank and blank spikes. We suggest specifying 1 blank and 2 blank spike per batch of 20 samples.

Response: We disagree with the commenter's suggestions for blanks and blank spikes. The revised method requires field reagent blanks and a field train recovery blank to provide data to testing contractors to assess contributions to residual mass measurements from reagents. The reagent blanks are not directly used to correct the CPM measurements obtained from the revised method. Mass values determined with the field train recovery blank are used up to a point to correct the CPM measurements. The combination of reagent blanks and the field train recovery blanks provide some limited indication about whether the testers' quality control process requires improvement. The method also contains a requirement to conduct a field train proof blank, if the tester chooses an alternative procedure to prepare their glassware rather than baking at 300°C. While high field recovery blanks may be due to contamination from the reagents, hardware or field crew, high field recovery blanks may also provide an indication that material was not recovered completely and might constitute carryover between sampling runs. EPA believes that field recovery blanks performed at least once for each compliance test of three sampling runs is adequate for this method.

Comment: (Comment No. 0077) Our concerns are that the proposed modifications to Method 202 may measure less of the PM and PM precursor emissions than the existing South

Coast Air Quality Management District (AQMD) test methods (including Method 202) and, therefore, may lessen the stringency of existing AQMD regulations. EPA cites a test report from the Alliance of Automobile Manufacturers that concludes there is no statistical difference between results by the existing and proposed modified Method 202. This test may not represent the same comparison for facilities in the South Coast including those that involve natural gas combustion and ammonia injection. We do not agree, however, that the term artifact applies to many sources in AQMD since our inorganic catches typically consist of both cations and anions that combine to make a solid ionic salt when the water is removed.

Response: EPA has improved Method 202 to reduce the byproduct of SO₂ conversion to H₂SO₄ in the water-cooled impinger solutions used in the original promulgated version of Method 202. We have also evaluated the retention of condensable inorganic and organic particulate using physical chemical predictions, supported by laboratory tests. The revised Method 202, as proposed, has been improved to capture CPM that would be formed under ambient air conditions immediately after emission from the stationary source. The modifications minimize, if not eliminate, slower fate and transport reactions of gases in the atmosphere. The revised method also eliminates many of the options in the original procedure which will improve the consistency of data generated nationally on stationary source CPM emissions. EPA has developed this method in combination with a revision to Method 201A which allows sampling and analysis of both filterable and CPM less than 2.5 μm in aerodynamic diameter. EPA believes these method revisions significantly improve the bias and precision of PM_{2.5} measurement from stationary sources by resolving well known errors with the methods. States should consider how and if these method revisions are applied to stationary sources they regulate.

EPA recognizes that South Coast Method 5 measures more mass than the revised Method 202. The primary objective of the revised Method 202 is the quantification of primary PM emissions. Laboratory assessments of the additional mass quantified by South Coast Method 5 show that it consists of SO₂ that has been oxidized into SO₃. There are a range of secondary reactions that may occur in the atmosphere to convert SO₂ to PM. However, measurement of this secondary particulate material formation is not the objective of Method 202. EPA's ambient air particulate models are programmed to account for these secondary reactions and Method 202 was revised to provide the fundamental input for these models for use in predicting particulate

mass in ambient air. For example, waters of hydration for H₂SO₄ have been eliminated from the weight of H₂SO₄ collected in the method to avoid double counting the hydration water mass. Using South Coast Method 5 may provide some indication of the secondary reactions and related particulate mass attributable to the source. However, there is no requirement to require any State agency to adopt EPA Method 202 as the measurement method for condensable PM for use in SIPs nor for use as the specific test method used to determine compliance with past or future particulate emissions limitations. If one or more States decides to continue the use of an existing CPM method or adopt a method other than Method 202, the Agency will assess the States' decisions based upon the merits of the methods and the ability of the methods to achieve the emissions goals.

Comment: (Comment No. 0049.1) Although EPRI's research report is not yet finalized, the results suggest that the reduction in bias under conditions more representative of power plant flue gas are not as large as suggested by EPA's tests. (See Attachment A, p. 3-4; EPRI Comments, OAR-2008-0348-0045.) Perhaps of greater concern, the EPRI testing showed a significant low bias in the measurement of actual H₂SO₄ in the gas stream. These results suggest the worst of both worlds -- that the method may not only continue to have a high bias when applied to some source categories in the field, but also may not collect all of the known condensable PM at those same sources. Because EPA has only just proposed the method revisions, few sources have attempted to apply the proposed "fix" in the field. And, where it has been used, sources (having been led to believe through the stakeholder process that revised Method has little bias) generally have not yet collected data to test that assertion. In assisting UARG in the preparation of these comments, RMB Consulting & Research was able to obtain one recent dataset that compares simultaneous testing with EPA's existing Method 202, the proposed revisions to Method 202, and the controlled condensation method (CCM) for measuring H₂SO₄ in the gas stream at a coal-fired electric utility boiler with a wet FGD. The results of those tests are summarized in Attachment A at 4-5. The results indicate not only similar performance between existing Method 202 and the proposed revision, but also a significant increase in the amount of H₂SO₄ measured by the existing and proposed revised Method 202 as compared to that measured by CCM. If the CCM results are correct, the results indicate not only that the revised Method 202 did not significantly reduce the bias as compared

to existing Method 202, but that both methods continue to significantly overstate condensable PM at units with wet stacks.

Response: EPA understands that the commenter is concerned regarding the comparison between the CCM measurement of H₂SO₄ and the total CPM in Method 202 (existing and revised). Based on the EPRI stakeholder laboratory tests, we recognize that the revised Method 202 is not 100 percent effective in capturing SO₃ and that the CCM captured a greater percentage (100 percent) of the H₂SO₄ in laboratory tests. The estimated collection efficiency of SO₃ for the proposed revision to Method 202 is less than the CCM method EPA used for comparison. The revised Method 202 recovered an average of 45 percent at 7 ppmv and 65 percent at 34 ppmv SO₃, respectively. EPA considered modifications to Method 202 to improve the collection efficiency but decided in favor of the current proposal due to the increased complexity that would be required in a further-modified method.

While we understand that selected emission tests comparing CCM to Method 202 (original and revised) show results for H₂SO₄ that are less than the CPM in Method 202, we do not agree that CCM measures all of the CPM, since H₂SO₄ is only one of the potential condensable inorganic species that have been identified in coal combustion emission source samples, sources to refute that SO₃ is the only CPM. Therefore, we do not find a contradiction between controlled laboratory experiments that include only H₂SO₄ condensable particulate and the results of field tests at functioning stationary sources. Furthermore, the EPA has reviewed data from several other tests on a variety of source categories and finds that results from the revised Method 202 provide significant improvement in CPM measurements and significant reduction in SO₂ artifact compared to the method as originally promulgated. Therefore, EPA has determined that the revised Method 202 provides the best method currently available to measure CPM emissions from stationary sources.

Comment: (Comment No. 0045.1) EPRI's research indicates that the procedures in OTM 28 significantly reduced bias associated with conversion of SO₂ to H₂SO₄, compared to the current Method 202. Therefore, in the absence of any true H₂SO₄ in flue gas, OTM 28 will provide a more accurate measure of CPM than the original Method 202. However, OTM 28 is inefficient at capturing H₂SO₄ aerosol. Thus, in the presence of SO₃/H₂SO₄, lower CPM values will be measured with OTM 28 compared to Method 202, due to a combination of reduced bias

and poor capture efficiency. EPRI did not test the original Method 202 with an H₂SO₄ spike; therefore, we cannot say definitively how much of the CPM reduction observed in the EPA and EPRI tests of OTM 28 with SO₃ present is due to reduction in artifactual sulfate formation and how much is due to reduced capture efficiency. OTM 28 also appears susceptible to smaller biases associated with the SO₂ concentration and moisture content of the flue gas. In contrast, controlled condensation measurements of H₂SO₄ are relatively unaffected by these factors. EPRI therefore concludes that a more accurate measurement of CPM for sources that emit H₂SO₄ would be obtained by running a parallel controlled condensation system (CCS) train along with OTM 28. The total sulfate from the OTM 28 impinger catch and CPM filter would be replaced by the sulfate measured in the CCS train, as follows: Total CPM = organic CPM + inorganic CPM – OTM 28 sulfate + CCS sulfate. In order to implement the sulfate correction approach, EPA would need to add an option to Method 202 to measure H₂SO₄ in each method fraction by ion chromatography, prior to evaporation of the solvent for gravimetric analysis. This approach does not meet the EPA's request for suggestions of a less demanding or costly modification, as it requires running two sample trains simultaneously, with consequent addition of manpower, equipment, glassware cleaning, and chemical analysis. However, it completely eliminates the bias from conversion of SO₂ to H₂SO₄. The CCS train is run at a temperature above the water dew point, so there is no opportunity for SO₂ to dissolve and become oxidized within the condenser coil. CCS also does not suffer from the poor capture efficiency for SO₃/ H₂SO₄ that appears to affect the OTM 28 method. [See DCN: EPA-HQ-OAR-2008-0348.0045.1.]

(Comment No. 0045.1) Based on EPRI's test results, OTM 28 significantly reduces the sulfate artifact compared to Method 202; however, the extent of reduction in the presence of true H₂SO₄ cannot be determined due to the confounding effect of poor recovery. OTM 28 is impacted by a negative bias from inefficient capture of H₂SO₄, and its accuracy is affected to a smaller extent by other factors including SO₂ and moisture content of the flue gas. Finally, EPRI notes that data and results of the EPRI studies discussed in this letter were included in a draft report from ERG to EPA, which was posted to this docket as item EPA-HQ-OA1-2008-038. EPRI did not review that report before it was posted, and it does not represent the final verified data set from the EPRI work. EPRI has not had the opportunity to review the ERG report in detail, and we are not prepared to state whether we agree with the conclusions of the report. EPRI plans to publish

the results of the EPRI studies as an EPRI report at a future date. (See DCN: EPA-HQ-OAR-2008-0348.0045.1.)

Response: Although EPRI published the report under its own cover, the results are the same in both the EPA and EPRI reports. The conclusions drawn by both parties are similar and EPA acknowledges that EPRI has the right to develop its own recommendations from these results. EPA also acknowledges that, based on the data from the collaborative work with EPRI, the correction for H₂SO₄ emissions proposed by EPRI would provide a less-biased emissions estimate. However, the proposed addition of a second sampling train specifically for H₂SO₄ correction and the additional analysis required to correct proposed Method 202 accurately for H₂SO₄ from coal-fired electric utility sources adds significant complexity and cost to the procedures in every Method 202 test. In addition, an EPA reference method or accepted consensus method to standardize CCM testing is not available. Therefore, we have not modified the proposed method. If the source has unique characteristic that require procedures that vary from the published method, the source should petition the appropriate regulatory agency for approval to use an alternative procedure or test method.

M202 1.2 APPLICABILITY

Comment: (Comment No. 0044.1) The proposed method states that a Method 17 filter can be used to collect filterable and condensable particulate from sources operating below 85°F if the filter is treated as described in Sections 8.5.4.4 and 11.2.1. Sections 8.5.4.4 and 11.2.1 describe the filter extraction procedure. If using Method 17, filterable PM could be determined gravimetrically rather than performing the filter extraction. The added analytical step can only lead to positive bias due to the extra sample handling with no added benefit for sources with little sulfur.

(Comment No. 0054.1) The proposed method states that a Method 17 filter can be used to collect filterable and condensable particulate from sources operating with stack temperatures below 85°F if the filter is treated as described in Sections 8.5.4.4 and 11.2.1. Could filterable and condensable particulate be determined gravimetrically and not perform the filter extraction step? The added steps for the filter extraction would seem to add bias in the results.

Response: We agree in part with the commenters. If a high percentage of H₂SO₄ is present in the sample gas, waters of hydration that combine with the H₂SO₄ can produce inconsistent weight measurements. To maintain consistency in application of Method 202 at different emission sources and by different testing contractors, we have revised Sections 1.2(b) and 11.2.1 of the final method to specify that, in cases where the Method 17 is used in conjunction with Method 202 and the filter can be brought to constant weight, testers can determine the filterable PM and CPM gravimetrically.

Comment: (Comment No. 0044.1) EPA should consider clarifying the language in Section 1.2 allowing the use of a "cooling probe" technique for sources that discharge above 30°C with little moisture, provided that the cooling probe allows for a CPM filter temperature less than 30°C. This modified Method 5 technique has been successfully used on many sources. It has been demonstrated to be a simple and cost effective method.

Response: We agree with the commenter that the "cooling probe" technique should be allowed in Method 201A. We believe that the language in Method 201A does not prohibit the use of a modest level of cooling of the sampled gas to achieve a filtration temperature below 30°C and so are not making a change to the final method.

Comment: (Comment No. 0050.1) EPA has correctly identified that ambient stacks (30°C or less) do not require measurement using Method 202 to determine condensable emissions as such emissions will be measured by the filterable method (i.e., Method 201A). However, during certain months of the year or in certain areas of the country it is not uncommon for ambient conditions to rise above 30°C or 85°F. If a tested process obtains air from an ambient source, it will not be able to meet the temperature limitation proposed in the method even though condensation and condensable emissions are not expected to occur once the emissions reach the atmosphere. The Alliance proposes that the references to a temperature of 30°C or 85°F be amended everywhere they appear (e.g., FR 13003, Sections 1.2 and 2.1) in the proposed reference test method to read as follows: "30°C (85°F) or ambient whichever is greater."

(Comment No. 0052.1) NEDA/CAP is pleased that EPA has clarified that use of Method 202 is not required where gas exhausting a stack is less than 30°C or 85°F and that Method 201A

will apply under those conditions. We urge EPA to state that Method 202 is never appropriate under 30°C or 85°F. We also suggest revising this condition to reflect how seasonal temperature variations affect conditions under which condensables will form, by amending the various references to the condition in proposed Sections 1.2 and 2.1, so that it is clear that Method 202 is to be utilized at "30°C (85°F) or ambient temperature, whichever is greater."

Response: We acknowledge the commenter's support for specifying that the use of Method 202 is not required where the gas filtration temperature does not exceed 30°C (85°F). However, we disagree with the commenters that the temperature limit for Method 202 should account for ambient conditions. Although we acknowledge that the total primary PM emissions measured from certain processes (e.g., dust emissions from crushing cement aggregate) would be unaffected by the gas filtration temperature, we believe that test methods, such as Method 202, where the pollutant measured is defined by the method must be conducted within the specified parameters to promote consistency in the results obtained by stack testers (consistency is defined as the method precision or comparative results between tests at the same source type).

Revisions to Method 201A require that the temperature never exceed 30°C (85°F) to approximate the average temperature in ambient air where CPM releases could form particulate. Maintaining this requirement provides a consistent basis to compare CPM measured at the same source category in different locations and at different times of the year. Collection of CPM below 30°C (85°F) will be normalized to a consistent weight since the analysis method requires evaporation of recovered sample to constant weight in a temperature and humidity controlled weighing room. Other method-defined endpoints specify the sample collection temperature (e.g., Method 5) to maintain the consistency of the measured mass. Therefore, to ensure that the gas filtration temperature used by stack samplers will be not be affected by seasonal or regional differences, we are not providing an allowance in Section 1.2 for establishing the applicability of Method 202 based upon the ambient temperature. Stack testers have the ability and responsibility to operate the sampling method within the parameters specified in the method. However, as acknowledged above, we recognize that the characteristics of some processes make the mass of PM collected insensitive to the filtration temperature and we leave open the possibility for regulatory authorities to prospectively allow for collection of PM samples at the ambient temperature.

Comment: (Comment No. 0050.1) This method can be used with Method 17 to collect condensable and filterable particulate material from sources operating at stack temperatures and/or samples collected below 30°C (85°F) with the filter being treated as specified in this method. (74 FR 13003, Section 1.2)

Response: EPA acknowledges the comments and appreciates the support for the proposed requirements. We believe it is appropriate for regulatory authorities to allow sources the option of using a gravimetric analysis (as opposed to the filter extraction procedure) for source tests where the stack filtration temperature is less than or equal to 30°C (85°F) at all times and the source tester is able to achieve constant weight during analysis following the procedures in the Methods 201A and 202. EPA believes that it is prudent for regulatory authorities to require the use of procedures specified in the final methods that stabilize the material collected on the in-stack filter using the extraction and weighing procedure in the Method 202 if the sampling temperature and constant weight requirements cannot be met.

Comment: (Comment No. 0056.1) In the proposed rule, EPA requested comments concerning the appropriateness of requiring Proposed Method 202 for sources generating gas streams at temperatures equal to or less than 30°C. API finds little purpose in requiring Proposed Method 202 for this application. At these temperatures, all of the potential CPM forms prior to the sampling system, and the filter provided in Proposed Method 201A will effectively capture the CPM. API agrees with the requirements stated in Section 1.1 of Proposed Method 201A.

(Comment No. 0061.1) Another commenter noted that in the proposed rule, EPA requested comments concerning the appropriateness of requiring Proposed Method 202 for sources generating gas streams at temperatures equal to or less than 30°C. PCA finds little purpose in requiring Proposed Method 202 for this application. At these temperatures, all of the potential CPM will have formed prior to the sampling system and the filter provided in Proposed Method 201A will effectively capture the CPM. PCA agrees with the requirements stated in Section 1.1 of Proposed Method 201A.

Response: EPA acknowledges the comments and appreciates the support to allow Method 201A to be used without Method 202 for measurement of PM in cases where the stack temperatures are less than 30°C .

Comment: (Comment No. 0059.1) The significance of the specification to maintain the filter below 30°C (85°F), if any, should be explained in the applicability portion of the method.

Response: The intent of the method is to measure CPM that would form upon release of stack emissions to the ambient air near the stack release point. While there is no single overriding justification for selecting 30°C (85°F) as the benchmark for the maximum filtration temperature, EPA used several general reasons for selecting this temperature. A temperature of 30°C (85°F) is in the general range of average ambient conditions, is reasonably near the standard temperature used to correct sample volumes, is easily achievable with available stack sampling glassware, and is achievable with dilution sampling methods (i.e., CTM 039) under high-humidity, high-temperature conditions. Consequently, we believe that 30°C (85°F) is a reasonable temperature to use as the basis of primary particulate matter emissions for use in dispersion modeling used to assess the impact of stack gases on the ambient air. We see no compelling reason for changing to another temperature that would represent the variable ambient air conditions. With regard to the commenter's suggestion to explain the significance of the temperature cutoff in Section 1.2, we do not believe that such an explanation is necessary or appropriate in the method. Consequently, we are not revising Section 1.2 to address this comment.

Comment: (Comment No. 0061.1) Requiring the use of Proposed Method 202 for sources such as material handling operations, crushers, and bagging operations results in unnecessary expenses. Proposed Method 202 should not be required for sources that clearly do not generate condensable vapors. In the cement industry, sources such as clinker coolers and finish mills operate at elevated temperature but have no possible source of condensable vapors. Method 5, "Determination of Particulate Matter Emissions from Stationary Sources," provides an adequate measurement of total particulate matter emissions for these types of sources.

Response: The final Methods 201A and 202 allow for collection of PM at sources with sample gas temperatures less than 30°C (85°F) using a simple filtration method that adequately addresses the needs expressed by the commenter. Although we agree with the commenter that certain processes may not be sources of CPM emissions, we believe that sources that operate at higher temperatures should demonstrate, through source testing, that their emissions do not

contain CPM before they are exempted from testing for CPM. Therefore, we do not believe that a change to the final method is necessary. However, we recognize that the characteristics of some processes make the mass of PM collected insensitive to the filtration temperature and we leave open the possibility for regulatory authorities to prospectively allow for collection of PM samples at the ambient temperature.

Comment: (Comment No. 0064.1) Nucor fully supports EPA's concern that the present Method 202 is unreliable in evaluating CPM, at least from high temperature metal operations. Theoretically, the ratio of condensable:filterable emissions should be relatively constant from the same furnace under similar operating conditions. Instead, Nucor has found that Method 202 has consistently delivered inconsistent condensable:filterable emissions ratios, certainly between units, and sometimes even between different runs of the same test. The attached presentation, *Condensable Particulate Matter and NSR: A Problem Deferred*, was given at the Energy and Environment Conference held in Phoenix, Arizona on February 2-4, 2009. In it, we presented the results of Method 202 testing at eight steel mills on the electric arc furnace baghouse. In four cases only the overall test result was available; in the other four cases, results from each test run were available. The testing showed that the condensable:filterable ratio could vary from as much as 3.8:1 to 13.5:1 in the same test. Further, parallel testing using IDEM Modified Method 5 (cooling the sampling train to atmospheric temperature rather than using a heated filter) showed both lower and more consistent values than Method 202, with the standard deviation of the test runs being a full order of magnitude lower than the variability seen in Method 202.

Based upon these results, Nucor believes that Method 202 is deeply flawed as applied to high temperature metals operations. Even if EPA should elect not to promulgate the proposed revisions to Method 202, EPA should disapprove existing Method 202 for application to iron and steel facilities until a new, better method can be adopted.

Response: We disagree with the commenter that the proposed Method 202 retains flaws that were associated with many of the alternatives of the method that it replaces. We have evidence (*Evaluation and Improvement of Condensable Particulate Matter Measurement* (Docket no. EPA-HQ-OAR-2008-0348-0028)) that the proposed revisions to Method 202 achieves an additional 90 percent reduction in H₂SO₄ artifact formation (compared to the current Method 202 using the nitrogen purge option), provides testing contractors with a more

standardized application of the method, improves the precision of the method, and quantifies more accurately direct PM emission to the ambient air. These revisions to Method 202 are a substantial improvement over the previous method. With regard to the source test emissions data submitted by the commenter, insufficient information was provided with the emissions test results by the commenter to evaluate the source of variability in the test results. We believe there are additional sources of variability of the CPM emissions from electric arc steel furnaces. The dynamics of steel production with an electric arc furnace include temperature, oxygen, and recycled or raw material characteristics that influence the quantity of sulfates, nitrates, chlorides, semivolatile organic matter that are potentially emitted during the charging, melting, and refining of steel to meet production specifications. In addition to the variations in the process operation, several aspects of the control device operation influence CPM emissions. Lastly, there are aspects of the emissions testing that are critical to achieve consistent results and information was not available in the test reports for EPA to assess any of these variables. We believe that to begin to explain some of the variability that the commenter observed in its data, the following information would be needed to assess the influence that may be attributable to the source test method:

- Blank train results,
- Field reagent bank results,
- Data on the mass of the organic and inorganic CPM (to determine the consistency of the split),
- The temperature of the Method 5 filter hot-box/stack gas exit temperature during collection,
- The total cubic feed or cubic meters collected in each train (to evaluate how close to the results are to the method detection limit),
- The CPM exit temperature,
- Process data including stack temperature taken periodically during each run, and
- Water/moisture train results

Additionally, the following data would also be useful for the evaluation:

- The volume of solvent (organic and water) used to recover the samples, and
- The laboratory narrative - were there any issues or observations such as samples taken to dryness at elevated temperature, etc.

To supplement the test method information, detailed process information may help assess the influence that may be attributable to the variability in the organic and inorganic vapors and gasses that may condense to form particulate matter. Some of the process information that may impact the formation of CPM include:

- Emissions of acid gases such as SO_x, NO_x, and HCl,
- Emissions of volatile organic compounds,
- The presence of volatile metals such as arsenic and selenium,
- Emissions of CO that may indicate the generation of semi-volatile organic compounds, and
- Any differences in the process operation that occurred during the source test period.

Comment: (Comment No. 0068) The third sentence in Section 1.2 of Method 202 is confusing. The Method 17 alternative would be better placed in a later section.

Response: We agree with the commenter that the discussion of Method 17 in Section 1.2 is misplaced. Requirements for handling and extracting the filter upstream of the Method 202 sampling train should be addressed in Method 201A. Consequently, we have revised Section 1.4 of Method 201A to clarify that the Method 202 is not required to measure total primary PM if the gas collection temperature does not exceed 30°C (85°F). We also revised Section 11.2.1 to specify additional CPM filter handling procedures.

M202 1.6 LIMITATIONS

Comment: (Comment No. 0063.1) We believe that performing this method using a jumper to connect the outlet of the heated filter (Method 5 or equivalent) to the CPM portion of the sample train needs to be addressed. Direct connection of the impinger train can be difficult or impossible with probes longer than 6 ft depending on the amount of clearance behind test ports. Furthermore, sampling vertically in ducts does not allow for a direct connection. We have been asked to perform this method in both of the above scenarios. Our general approach is to use a heat-traced Teflon® line (to prevent condensation) and to recover the Teflon® line as part of the CPM fraction of the train (inorganic and organic rinses). As written, any kind of jumper

technically would have to undergo the same preparation as the CPM portion of the sample train (including baking at 300°C). This would obviously melt a Teflon® line. The sample analytical data from testing at two coal-fired units (CleanAir_OTM 28 Analytical_SampleData03.pdf) that we are waiting on approval to submit will be of interest for this point as well. The OTM 28 was performed using a jumper during this test program. The two field blanks (one on an unused train, the other on a “used” train at the end of the test program) both included a jumper rinse. What’s more, these jumpers were not baked prior to use. Both had organic field blank residues below detection limit for both field blanks. Inorganic blank residue was non-detectable for the preliminary field blank, and about 1 mg for the post-test (“used”) field blank. Again, I should have approval to send you this data by the end of the week. Our recommendation is to avoid using jumpers as much as possible. If a jumper is absolutely necessary, it does not appear that baking the line is absolutely necessary. As a precaution, we suggest baking Teflon® jumper lines at up to 350°F in an oven and circulating hot air through the line prior to use.

(Comment No. 0106) With the combined OTM 027/028 sampling train can a heated or unheated Teflon® transfer line (~20 feet in length) be placed in between the heated probe exit and the spiral condenser? Sample recovery of the transfer line would include the DI water, acetone, and methylene chloride rinses in addition to the spiral condenser, first two impingers, and unheated filter front half. If possible which type of transfer line is recommended heated or unheated?

Response: We acknowledge that a transfer line to connect the sampling probe to the condenser may be needed at certain locations so that the impinger box(es) can remain level or stationary. In such cases, the entire transfer line must be heated to a temperature that is at least 300°F or 10°F greater than the temperature of the stack gases being sampled (whichever is less) to keep acid gases in the vapor state and to minimize the CPM caught in the transfer line upstream of the condenser. The transfer line must be recovered as specified in Section 8.5.4 of Method 202.

Comment: (Comment No. 0048.1) We recommend that some provision or alternate procedure be added to address the problem of high moisture content sources. It is possible to entirely fill the first dropout impinger on these sources. Since the intent of the method is to prevent artifact formation, having the water in the first dropout surging into the second may

encourage mixing with the stack gas and promote artifact creation. In our opinion a provision for stopping the test, leak checking, and replacing the full impinger with an empty one needs to be added. Another option would be to add an extra empty impinger at these sources. We recommend that EPA provide guidance on the best practices for high-moisture sources.

Response: For stacks with high moisture content, where the formation of artifact may be higher, testing contractors may need to use a larger impinger to provide for additional capture condensate volume. Section 6.1.2 of the method does not limit the size or shape of the knock-out vessel used in the method as long as the specified nitrogen purge can be performed on the liquid collected in the knock-out vessel. Also, if accumulation of water in the first impinger is excessive (e.g., when sampling moist gases for extended periods), testing contractors are not prohibited from recovering, quantifying the volume or mass of water removed and purging (with a back up filter to retain PM) the water collected in the first impinger before the end of the emission test.

Comment: (Comment No. 0056.1) Proposed Method 202 should include a summary of the practical limits of applicability. For example, it is difficult to conduct Proposed Method 202 when the sampling equipment is exposed to ambient temperatures less than -10 °F due to water freezing problems in the condenser.

(Comment No. 0113) The method states that the exit temperature of the CPM filter should be below 85 degrees F. Is there a lower temperature threshold we should not fall below?

(Comment No. 0086.1) API has previously submitted a comment (Comment 20, May 26, 2009) concerning the practical limits of Proposed Method 202 for tests in extremely cold conditions. API noted that Clean Air Engineering has submitted comments that are compatible with and expand the condenser temperature issue. Clean Air Engineering (Docket Document 0063.1) recommends that a minimum and maximum condenser temperature be specified to reduce test-to-test differences caused by the extent of vapor phase condensation. Mr. Evans of Clean Air Engineering has also asked the following: "Which leads into the second issue: how does one perform this test method when the ambient temperature is below 32°F? This test condition obviously requires supplemental heating of the water bath, which may not be indicative of ambient conditions and subsequent CPM formation. Mr. S. Evans, Clean Air Engineering, Docket Document 0063.1" The API and Clean Air Engineering comments both

point toward the need for a dry impinger case heater to maintain the gas sample temperatures at the filter above freezing and in a range of 32°F to 85°F. This does not add substantial complexity to the method. API recommends that EPA revise Proposed Method 202 to specify this sample gas temperature range.

Response: We acknowledge the potential challenges associated with operating Method 202 at low ambient temperatures. EPA has therefore revised the Section 8.5.1 of the final Method 202 to set a lower limit of 20°C (65°F) on the CPM filter. We believe that our rationale for establishing the temperature criteria and the impact on the method results that can be caused by operating Method 202 at significantly different temperatures has been adequately presented in the proposal and promulgation preambles. The testing contractor should take the necessary steps (e.g., heated water impinger bath) to meet the method requirements when the method is used at low ambient temperatures.

Comment: (Comment No. 0056.1) Proposed Method 202 has previously been termed OTM 028. It can be performed by numerous testing/engineering organizations having experience with complex test methods. No special sampling equipment is needed. The sampling run times needed in this method are consistent with many other EPA reference methods. No major changes are needed to this proposed method.

Response: We acknowledge the commenter's support.

Comment: (Comment No. 0057) "You can use this method to measure emissions following a wet scrubber only when this method is combined with a filterable particulate method that operates at high enough temperatures to cause water droplets sampled through the probe to become gaseous." Consider re-stating this sentence to include other sources besides wet scrubbers which may have water droplets present in the stack. Not all wet scrubber sources have water droplets in the stack.

Response: We agree with the commenter's suggestion to clarify the language in Section 1.6. Our intent in the proposed method was to specify that Method 202 can be used to sample gases that contain water droplets as long as the filterable PM test method was operated at a sufficiently high temperature to maintain the entrained moisture in the vapor phase. Therefore,

we have revised Section 1.5 of the final method (proposed Section 1.6) to remove the emphasis on wet scrubbers.

Comment: (Comment No. 0066) Based on paired testing we have performed on fluid catalytic cracking units (FCCUs) we have seen that the proposed method modifications may result in lower values compared to the existing method on dry stacks. However, on wet stacks we see no difference between the existing method and the proposed modifications. Please see the attached chart showing the results of this testing. [See EPA-HQ-OAR-2008-0348-0066 for chart.]

Response: We acknowledge that the results obtained when using the revised Method 202 can be higher, lower, or the same as the results obtained using the existing Method 202. However, the modifications proposed for Method 202 were those necessary to reduce the bias of the method (as indicated by SO₂ conversion to SO₃) and to obtain consistent results between different emission source types and testing contractors.

M202 2.1 SUMMARY

Comment: (Comment No. 0059.1) In Section 2.1 of the method, there is a reference to filterable particulate collected on filters maintained above 30°C (85°F). The intent of the condensable method is to combine with Methods 5, 17, and 201A. The filter temperature will be operated at that prescribed in the method (i.e., 248 ± 25°F for Method 5 or at stack temperature for 17 and 201A). This should be clarified.

Response: We agree with the commenter that the language in Section 2.1 of the proposed revisions to Method 202 regarding the temperature of the filter used to collect filterable PM should be revised to allow for cases where the filter must be maintained at temperatures other than 30°C (85°F) (e.g., the temperature of the PM filter in Methods 17 or 201A is maintained at stack temperature). Therefore, Section 2.1 of the final method has been revised to remove the reference to a specific temperature and to clarify that the filter temperature must be maintained as specified in either Method 5, 17, or 201A or such other temperature as specified by an applicable standard or approved by the authority that established the regulatory requirement for a particular application.

Comment: (Comment No. 0064.1) The design of the CPM capture train does not allow for less efficient condensation on the relatively dry walls of the Method 23 condenser and the two dry impingers. In the proposed Method 202, the first half of the impinger train consists of a “Method 23,” water-cooled glass condenser followed by the two dry, controlled-temperature impingers. This design is apparently intended to minimize the volume of water which contacts the stack gas sample, rather than having the sample gas bubble through solution-containing chilled impingers as in the current Method 202. Because the sample gas contacts less water surface, there is presumably less potential for SO₂ absorption and thus the high-biased CPM due to sulfate artifact is reduced. For many sources, this is not necessarily an improvement. The sample train design trades off reduced sulfate artifact for potentially low-biased CPM in relatively dry streams due to heat/mass transfer resistances. Generally, boundary-layer heat and mass transfer resistances at a dry wall are greater than between a bubble traveling a high velocity through a surrounding liquid. Thus, there may be relatively less-efficient condensation on the relatively dry walls of the Method 23 condenser and the two dry impingers. This is especially the case where a dry sampled stream contains insufficient moisture to coat the condenser walls. For this condition, there is likelihood that a portion of the condensable material will not be condensed, and may pass through the condenser tube and dry impingers. If such “bypassed” condensables do drop out in the chilled water impinger (CPM container #4), there is no analysis in the method to quantify.

Response: The EPA recognizes the commenter’s concern regarding the efficiency of CPM collection on the dry gas surfaces of the sampling train when sources do not contain some minimum moisture level to allow PM condensation. We recognize this phenomenon may include polar organic material and condensable acid and base gases that do not have sufficient moisture in which to dissolve and ionize. Condensation will depend upon the availability of nuclei and surface area to begin condensation once the saturation concentration is reached. However, the proposed revisions to Method 202 include a CPM filter maintained at temperatures between 20°C and 30°C to simulate stack dilution in ambient air. The amount of material collected depends upon the saturation concentration of the vapor, the temperature and the ability of the condensed material to pass through this filter. Because the CPM filter increases the surface area and nucleation sites, the mass measured by this method defines CPM regardless of the dry conditions of the emissions source.

M202 2.1.1 CONDENSABLE PARTICULATE MATTER

Comment: (Comment No. 0039.1) Regarding purging the impingers with nitrogen, Section 2.1.1 says "immediately" after sample collection and Section 8.5.3 says "as soon as possible" after sample collection. There are no definitions or limits as to what this means. Also, what about test disruptions that delay the completion of a test run? There should be outside limits defined whereby a run is voided if that timeframe is exceeded.

Response: We do not believe that it is necessary for the Agency to specify an acceptable time frame over which the nitrogen purge of the impinger water should be conducted. Rather, the testing contractor has incentive to conduct the nitrogen purge in a timely manner because the longer the purge is delayed, the greater the potential artifact formation from conversion of dissolved SO₂ to H₂SO₄. Therefore, we are not revising the final rule to specify the time frame for conducting the purge.

M202 6.0 EQUIPMENT AND SUPPLIES

Comment: (Comment No. 0048.1) It is our recommendation that EPA should specify the container type for each container (i.e., glass or plastic) and also whether the lid should have a Teflon® liner or other liner is acceptable. This includes the correct bottle for each type of reagent blank, since these are not specified in Sections 8.5.4.7, 8.5.4.8, or 8.5.4.9.

Response: We disagree with the commenter that the method should specify the material of construction of containers used for sample and blank recovery procedures. Although we believe that the most appropriate containers are constructed of glass and equipped with a PTFE lid, we also believe that testing contractors should have the flexibility to select the type of containers that meet the performance specifications of the method. Therefore, the final method has been revised to add a performance-based specification for sample and blank containers. We also made accompanying edits to the CPM container language in Sections 8.5.4 (Sample Recovery).

Comment: (Comment No. 0057) We have operated the sampling train as shown in Figure 1 and have determined a more effective (less glass breakages and easier to fit) arrangement as presented below. This alternative positions the condenser upstream of a knockout impinger "flask" in the vertical plane. [See DCN EPA-HQ-OAR-2008-0348-0057]

Response: The method provides stack testers with the flexibility to use alternative glassware. Therefore, we are not revising the final rule. However, the glassware configuration selected by the testers must allow for the nitrogen purge to be conducted as specified in the method.

Comment: (Comment No. 0102) Is it required to use a Method 23 coil type condenser or can a straight condenser be used?

Response: In the proposed method, we specified the use of a Method 23-type condenser because this equipment is a readily available piece of glassware and the condenser coils accomplish the objective of cooling the sample gas quickly without undue mixing of the sample gas and the condensed water. The most important aspect is that the temperature achieved at the drop out impinger is at or near the temperature required at the exit of the back up filter. Achieving this will result in almost all of the liquid water being in the drop out impinger and that the second impinger will be a "finishing" step with little water such that the back up impinger does not collect a lot of water droplets (with the potential of becoming blinded by this water). We do not believe that a straight condenser can provide sufficient cooling of the sample gas to ensure that moisture will be collected in the first two impingers. Consequently, the final method specifies the use of a Method 23-type condenser.

Comment: (Comment No. 0069) Allow the CPM filter holder to contain more than 1 filter. Suggestion: A Teflon® coated filter may be placed between the CPM filter and the filter support.

Response: The method provides stack testers the flexibility to use appropriate non-contaminating equipment. The EPA's laboratory experience indicated that larger filters and/or sintered filter supports performed well. However, filter backers or supports, including an additional non-contaminating filter between the CPM filter and the filter support, may be used to reduce the pressure drop across the CPM filter assembly.

M202 6.1.3 LONG STEM IMPINGER INSERT

Comment: (Comment No. 0069) Allow equivalent long-stem impinger inserts.
Suggestion: "long stem Greenburg-Smith insert or equivalent made of inert material, such as Teflon®.

Response: We disagree with the commenter's suggestion to use a long stem Greenburg-Smith insert or equivalent component. We specified the use of the long-stem Greenburg Smith impinger in the proposed method to ensure that the inside diameter of the impinger inlet was sufficiently large so as to not become plugged when sampling gases that contain high concentrations of organic compounds. Therefore, we are not revising the final rule requirements with regard to alternatives to a long-stem Greenburg Smith impinger.

M202 6.2.1.3 ULTRA-HIGH PURITY (UHP) NITROGEN GAS

Comment: (Comment No. 0050.1) Proposed RM 202 (74 FR 13004, Section 6.2.1.3) Ultra-High Purity (UHP) Nitrogen Gas, is required for purge. The Alliance understands that there are circumstances where such a high grade of nitrogen will be desirable to virtually eliminate sulfur compound-induced interferences in measuring PM_{2.5} emissions. However, flexibility should be provided to allow the option for a lower purity nitrogen gas purge, or no purge, especially for situations where there is little if any sulfur of concern.

(Comment No. 0061.1) PCA concurs that the use of ultra high purity (UHP) nitrogen is needed for purging the Proposed Method 202 impingers. There are isolated cases when very high levels of hydrocarbons have been found in cylinders reported to be UHP quality. It would be helpful to have some general quality assurance procedures to screen UHP cylinders prior to a test program in a manner analogous to the reagent blank pretest analyses.

(Comment No. 0056.1) API concurs that the use of ultra high purity nitrogen is needed for purging the Proposed Method 202 impingers. API is aware of isolated cases when very high levels of hydrocarbons have been found in cylinders incorrectly labeled as UHP quality. The capture of organics in the Proposed Method 202 sampling train could bias the test results to substantially higher-than-true levels. It would be helpful to have some general quality assurance procedures to screen UHP cylinders prior to a test program in a manner analogous to the reagent blank pretest analyses. API proposes that testing organization be required to conduct a blank test using the ultra high purity nitrogen prior to the field tests. At least one pretest blank run should be conducted for each cylinder of nitrogen to be used in the test program. The blank runs should be recovered and analyzed in a manner identical to those specified in Proposed Method 202. The ultra high purity nitrogen cylinder should be rejected if the measured catch weights exceed 2 mg.

Response: We disagree with the commenter who asserted that lower purity nitrogen gas is acceptable and concur with the commenter that asserts that UHP nitrogen is necessary. In our laboratory studies, we have found that high-purity nitrogen is necessary to eliminate artifacts from the nitrogen gas used to purge the train. Several studies have demonstrated measurable artifacts at very low SO₂ concentrations with the 1990 version of Method 202. Therefore, the requirement to use UHP nitrogen in the purging procedure is retained in the final rule.

We believe that the required field train recovery blank samples respond to the commenter's concerns regarding a quality assessment of the UHP nitrogen. We also disagree with the commenter that the final rule should specify general quality assurance procedures to screen UHP cylinders prior to testing because any artifact contributed from UHP nitrogen are assessed as part of the field train recovery blank procedure. We believe that such a requirement could be overly burdensome for testing contractors who are unable to purchase nitrogen cylinders in advance of an emission test, conduct an evaluation of the gas quality, and transport the cylinders to the test site. However, some stack testers may find it desirable to qualify or test the nitrogen prior to conducting the field sampling activities.

M202 6.3 ANALYSIS

Comment: (Comment No. 0040.2) I would like to suggest adding "Sonication device" to Section 6.3 analysis equipment.

Response: We agree with the commenter that sonication devices should be added to the list of analytical equipment. Therefore, the equipment list in Section 6.2.2 of the final method (proposed Section 6.3) includes a sonication device.

Comment: (Comment No. 0122) Can you recommend what type of sonicator (i.e., brand and size) would be appropriate for this test method?

Response: We cannot recommend a particular brand of sonicator. However, in addition to the specifications in Section 6.2.2(i) of the final method (proposed Section 6.3.9), the sonication device selected for extracting the CPM filter should be large enough to completely immerse the extractor tube.

M202 6.3.2 WEIGHING TINS

Comment: (Comment No. 0040.1) Laboratory procedures conducted by AirNova evaluated the ability to use fluoropolymer beaker liners as weighing containers instead of volume reduction in beakers, followed by weighing tin evaporation. This gravimetric methodology has already been approved as EPA EMC method Alt-005 since 1998. Beaker liners are relatively low weight and allow for analytical balance determinations on the order of tenths of a mg. Their use could also reduce beaker-to-weighing tin liquid transfer errors. See EPA-HQ-OAR-2008-0040.1 for description of laboratory procedures. Additionally, when demonstrated to provide reliable results, the method should allow alternate weighing procedures to the proposed EPA202 beaker/weighing tin requirement. AirNova has been endorsing and using Teflon® baggie procedures since May of 1992.

(Comment No. 0054.1) This method has a lot of transferring of the sample from one sample container to another which lends itself to loss of sample between each step. The last line in Section 2.1.4 of the ‘Evaluation and Improvement of Condensable Particulate Matter Measurement’ report recommended changing the method to include “Transfer to aluminum weighing pans for final dry down.” One of the statements prior to this conclusion says “Samples dried down in a single glass jar (versus transfer to an aluminum weighing pan) contained 0.4 mg more CPM.” This may be a true statement, but it does not address that the glass jar may have still contained 0.4 mg after the transfer to an aluminum weighing pan or that small sample

reduction could be lost during the transfer. It should be kept in mind that a stable weight is considered ± 0.5 mg. We do not feel that using glass beakers is reducing the accuracy of this method, but they should be an acceptable alternative especially when used in a temperature and humidity controlled environment. In summary, glass sample beakers should be allowed for final dry down and could help reduce sample loss.

(Comment No. 0040.2) I would like to propose changing the text “Weighing Tins, 50ml” in Section 6.3.2 to “weighing tins, fluoropolymer beaker liners or any other chemically inert gravimetric weighing container. Any weighing methodology used needs to demonstrate consistent reproducible results on the order of tenths of a milligram.” The AirNova reduced temperature glassware baking paper also provides backup data for support. This change would accommodate the use of EPA EMC Alt-005 in determining the weight of the residual masses to Section 6.3.2. Teflon® (or fluoropolymer equivalent) bag beaker liners provide gravimetric Method Detection Limits (MDLs) of approximately 0.3 mg and are already accepted as an alternative to Weighing Tins. Our lab’s last three annual MDLs for EPA Method 202 using FEP beaker liners were 0.35 mg, 0.3 mg, and 0.3 mg.

Response: We agree with the commenter that fluoropolymer beaker liners or Teflon® baggies can be used instead of weighing tins in the final evaporation step. Therefore, we have revised Section 6.2.2 of the final method (proposed Section 6.3) to specify that fluoropolymer beaker liners or baggies can be used instead of weighing tins. However, we disagree with the commenter’s suggestion to allow the use of glass beakers in the weighing process. The results generated by Environment Canada’s experiments (Docket No. EPA-HQ-OAR-2008-0348-0028) documented an uncertainty greater than 0.5 mg when glass beakers were used for this measurement. The combined uncertainty caused by the relatively heavy glass beakers and the much lighter sample weight is too great based upon the studies conducted by Environment Canada. Additionally, the Environment Canada study showed that the changes in barometric pressure affected the weight of the beaker when measured during the weighing process. We also disagree with the suggestion to revise Section 6.3.2 to allow for the use of other chemically inert gravimetric weighing containers because the suggested revision is too open ended.

Comment: (Comment No. 0040.2) In Section 6.3.4 (Drying Equipment), a desiccator and desiccant should be added. I suggest making “Hot plate or oven with temperature control” Subsection 6.3.4.1 and adding two more subsections: 6.3.4.2 “Desiccator with a humidity monitor. Maintain relative humidity below 50 percent” and 6.3.4.3 “Active Desiccant: Anhydrous Calcium Sulfate or both Silica Gel and Anhydrous Calcium Sulfate. Regenerate according to manufacturer’s specifications.”

Response: We agree with the commenter that the terms “desiccator” and “desiccant” should be added to Section 6.3.4. However, the EPA requires that use of anhydrous calcium sulfate because of its superior performance over silica gel. Consequently, we have revised Section 2.2 of the final method (proposed Section 6.3.4) to add specifications for a desiccator.

M202 6.3.7 ANALYTICAL BALANCE

Comment: (Comment No. 0038.1) Method 202 should require a balance with a resolution of 0.00001 g (0.01 mg). The specification for maximum residue in the acetone reagent requires getting a detectable weight below the resolution of a four place balance (0.000079 g, see comments on Section 8.5.4.7). The use of a four place balance to determine masses below the resolution of the instrument is not consistent with good laboratory practice.

Response: We have evaluated the impact of requiring a sensitivity of 0.01 mg for the CPM mass measurement and the cost of a new, more sensitive analytical balance for sample analysis against the need for precision sensitivity in the revised method. Based upon our laboratory and field evaluations, the precision of the revised method is less than 0.5 mg. Consequently, we do not believe that cost of a 5-place balance and the stringent weight room requirements necessary to realize the benefit of the lower resolution scale, are warranted. However, the analytical laboratory has the option of using a more sensitive balance if desired.

Comment: (Comment No. 0048.1) Please define “extremely low sources” and set up a different procedure for cases when the samples have reached a constant weight, etc.

Response: We agree with the commenter that the use of the term “extremely low sources” is unclear; therefore, we have removed this term from Section 6.3.7 of the final method. We have also revised Section 6.2.2 (proposed Section 6.3.7) to allow the use of an analytical

balance with a minimum sensitivity of 0.0001 mg. However, we disagree with the commenter that a different constant weight procedure should be provided in the final method. We believe that the Method 5 constant weight criteria of ± 0.5 mg is adequate for this method and that an alternative procedure is not necessary in the final rule.

M202 6.3.8 pH METER

Comment: (Comment No. 0119) EPA CTM 028 specifically requires the use of a pH meter to titrate re-suspended aqueous samples to pH 7 (see Section 11.2.2.4). Although our lab is equipped with a pH meter, I prefer the analyst to use a colorimetric endpoint determination (i.e., phenolphthalein) in place of the meter. Phenolphthalein is much more reliable and does not require calibration. A meter, although calibrated, may have "response drift" since its last calibration). Phenolphthalein is consistently reliable and never needs calibration. I would like approval from the EPA for its use in place of a meter for this specific procedure.

Response: We agree with the commenter that a colorimetric endpoint determinant, such as phenolphthalein, is an acceptable alternative to using a pH meter. Our intent in the proposal was to neutralize the acids in the sample without adding more ammonium hydroxide that was necessary for neutralization. The use of phenolphthalein, or other indicator, achieves that objective. Therefore, Section 6.2.2(h) of the final method (proposed Section 6.3.8) was revised to allow for the use of colorimetric pH indicators. Accompanying edits were also made to Section 11.2.2.2.

M202 7.1.1 FILTER

Comment: (Comment No. 0040.2) In Section 7.1.1, I would like to suggest replacing "Filter" with "CPM Filter" and replace "Teflon®" with "Teflon®, PTFE or chemically equivalent" when mentioning the trade marked/registered material Teflon®.

(Comment No. 0068) It would be better to offer alternatives like quartz, TFE coated, or TFE filters.

(Comment No. 0117) Problems with clogging of the back cellulose filter (after the impingers) on OTM 027/028 sampling trains have been noted in field tests of uncontrolled oil-fired boilers. Apparently the moisture condensing on the filter absorbs SO₂ and forms a H₂SO₄ mist that clogs the filter and drastically increases the pressure drop.

Response: Section 7.1.1 of the proposed method specified the use of a Teflon® membrane filter that does not have an organic binder. The intent of this requirement was to specify the type of CPM filter that would not contribute mass to the CPM measurements (e.g., by chemical reaction with the sample gas, splintering during filter extraction). In light of the commenters' suggestions, and because it is not feasible for the Agency to evaluate all possible permutations of filter type and sample gas composition, we have determined that the CPM filter requirements in the final rule should be based upon performance rather than a specific type of filter. Therefore, we have revised the language in Section 7.1.1 to specify the use of nonreactive, nondisintegrating filter that do not contribute more than 0.5 mg of residual mass to the CPM measurements. In selecting the appropriate CPM filter, testing contractors should avoid the mistake of equating the dioctyl phthalate (DOP) size for the test particles to the pore size for the filter. Filters with pore sizes larger than the test particles can retain a high percentage of very small particles. In our evaluation of different types of filters, we determined that filter sizes of 47 mm are marginal if not unacceptable for use. We have also identified a manufacturer who provides both hydrophilic (water absorbing) and hydrophobic filter media that meet the DOP particle collection criteria.

Comment: (Comment No. 0048.1) It is our recommendation that the EPA either provide a procedure that can be used to determine whether a filter is non-reactive to SO₂ or SO₃ or provide a recommendation of a filter material that meets the criteria.

(Comment No. 0057) Section 7.1.1 of the proposed method says that "You must use a Teflon® membrane filter that does not have an organic binder" and that "If the source you are sampling has SO₂ or SO₃ emissions, then you must use a filter that will not react with SO₂ or SO₃." Does a Teflon® filter react with SO₂ or SO₃? What is the recommended filter type for SO₂ and SO₃ emitting sources?

Response: We believe that the specifications for the CPM filter in the final method are adequate with regard to the particulate capture and chemical inertness and provide testing contractors with flexibility when selecting appropriate filter media. We also disagree with the commenter that the final method should specify a procedure for determining filter reactivity or a

specification for filter material. However, based upon published reports, SO₂ and oxidation products do not react with fluoropolymer filters.

Comment: (Comment No. 0068) You might want to explain why the glass fiber Method 17 filter is okay but the CPM filter must be Teflon®.

Response: The final method requires that the CPM filters be constructed of nonreactive, nondisintegrating material to prevent filter shards from biasing the CPM filter extraction results. Because the sample recovery process requires that the CPM filter be placed in an extractor tube and subjected to two extractions (organic and inorganic) using sonication, the filter must be constructed of materials that are not prone to releasing shards. In contrast, the sample recovery process for the Method 17 filter involves much less handling of the filter (i.e., the filter is simply transferred to a petri dish and evaluated gravimetrically). Therefore, glass fiber filters are appropriate for use in Method 17.

M202 7.1.3 WATER

Comment: (Comment No. 0048.1) It is our recommendation that the EPA should not specify an ASTM Type of water for this procedure, but should specify a residue level as is done for acetone and methylene chloride in Section 7.2.1 and Section 7.2.2. Most testing contractors will be able to process a blank and determine a residue level, but will have to buy HPLC grade water for use on these projects which is expensive and unnecessary if the blank residue can be reached another way. The specification should be the same as for the acetone, which is less than 1 ppmw residue. For a 150 ml blank, this will result in a residue of 0.0002 grams.

Response: The EPA's experimental results (Evaluation and Improvement of Condensable Particulate Matter Measurement (Docket no. EPA-HQ-OAR-2008-0348-0028)) indicate that ultra-filtered, de-ionized water is necessary to reduce the blank residual mass for this method. However, we agree with the commenter that an ASTM specification is not necessary. Therefore, we have revised the final method as suggested to specify that ultra-filtered, deionized water that contains 1 ppmw (1 mg/L) residual mass is acceptable and remove the incorporation by reference citation in Section 7.1.3.

Comment: (Comment No. 0036) You may want to add a mg/100 ml value for the Acetone and methylene chloride blanks in Proposed Method 202 so users do not have to mess with density calculations to translate the ppmw limits into an allowable mass correction in mg.

(Comment No. 0069) Specify maximum blank values for acetone and methylene chloride in mg/100 ml.

Response: In the proposed Method 202, we chose to specify the residual mass and blank values in terms of ppmw because this unit of measure is commonly used by vendors. However, we acknowledge that specifying the residual mass and blank values in terms of mg/ml would allow for direct comparison to the residual mass values determined during the sample recovery process. Consequently, the final rule specifies the residual mass and blank values in terms of ppmw and mg/ml.

Comment: (Comment No. 0039.1) The method has you take and analyze reagent blanks, but this information is not utilized. The calculations only deal with the field train blank determined from processing the field train blank as you would a field train sample (or at least that is what it implies). There is nothing that says you need to either use the exact same amount of rinse volumes as the samples or do volume corrections from the blank volumes to that used for samples. For example, Method 29 specifies using the exact same rinse volumes as blank volumes, so there is no need to volume correct. If we wish to keep a field blank train, then the method should specify exact volumes for reagents used in sample recovery and the reagent blanks should be the same volumes. These reagent blanks should be used to process the field train blank. If water is added prior to the purge, this same volume should be added during recovery of the field train blank. Alternatively, eliminate the field train blank and just use volume-adjusted reagent blanks. These blanks would be volume-adjusted based on the rinse volumes, any water added prior to the purge if needed and the 100-ml of water charged to the 1st impinger. Volumes would need to be measured and recorded, which the method does not specify doing for the rinses. As currently written, the blank corrections are open to interpretation and could over or under estimate blanks if volumes used are not consistent with those used for samples.

(Comment No. 0048.1) It should also be noted that the blank values do not enter into the calculations, but are only there for the purpose of assessing the residual levels in the reagents.

Response: Reagent blanks specified in the final method are not used to correct the CPM sample results. Rather, they are used to help testing contractors identify the source of the blank contamination in samples. Field train recovery blanks are the best indicator of the total blank contamination for field tests. We believe that a small amount of contamination above the reagent blank is expected in field samples and we have limited the blank correction to the level that we were able to demonstrate in the field train precision evaluation. To improve the clarity of the final method, we have used consistent terminology to refer to the various laboratory and field reagent blanks and field train recovery blanks throughout the method.

With regard to the volume of solvent used, testing contractors should use the same volume for laboratory and field reagent blanks as was used for sample recovery to ensure that the blank correction is not biased.

M202 7.2.1 ACETONE

Comment: (Comment No. 0048.1) The requirements specified for the amount of residue from 100 ml blanks for acetone (0.0000789 grams) and methylene chloride (0.000132) is at or below the limit of the 4-place balance, which is allowed under the method. This is the same issue with the new Method 201A. All previous promulgated methods have allowed blanks of 0.001 percent rather than the 10 times more restrictive 0.0001 percent (1 ppmw) allowed in these two methods. It is our recommendation that either the volume of the blank be increased by 10 times so that there is a more measurable amount of residue, or the amount of residue allowed be increased for the 100 ml blanks to what has been acceptable under all other promulgated methods. Since the amount of residue that is allowed for the entire train blank, which is subtracted from the sample, is 0.002 grams, then it would be appropriate to increase the amount of allowed residue in the reagent blanks to something more like less than 0.001 percent for water, acetone and methylene chloride.

Response: The residual mass values for reagents specified in the proposed method were selected based upon: (1) the level of residue that would not impact the detection limit, precision, and accuracy of the method and (2) the residue level specifications used by vendors. The

residual mass levels specified in the method are substantially lower than the detection limit that can be obtained under well-controlled conditions. Consequently, the reagent analyses specified in the proposed method would show if the residual mass of a reagent were 20 percent above the vendor's guarantee.

In light of the above comment, and other similar comments received regarding the reagent blank volume, we have increased the reagent blank volume in the final method to 150 ml. This volume ensures that the residual mass would be measurable if the reagent was at the maximum guaranteed value.

**M202 7.2.2 METHYLENE CHLORIDE, AMERICAN CHEMICAL SOCIETY
(ACS) GRADE**

Comment: (Comment No. 0083.1) We encourage EPA to conduct future studies to identify a solvent to replace methylene chloride in Proposed Method 202 and in other EPA reference methods. As you are aware, methylene chloride is a suspected human carcinogen. A less hazardous solvent is needed. The adoption of Proposed Method 202 should not be delayed while alternative solvents are evaluated. On an interim basis, EPA should allow the use of a less hazardous solvent.

(Comment No. 0086.1) The toxicity issue raised by GE Energy goes well beyond the possible hazards created by the use of methylene chloride by central office laboratory personnel conducting extractions of the samples received from the field. Health hazards are much easier to minimize in a well-controlled laboratory setting than in the more challenging conditions at the test site. The use of methylene chloride creates significant safety issues for emission testing crews recovering Method 202 samples. Methylene chloride is a highly toxic compound that rapidly breaks through most, if not all, types of chemically resistant gloves. Methylene chloride poses a risk to field testing personnel who must rinse the Method 202 glassware after each test run. In addition to the significant skin absorption hazards, test crews must provide hoods and other types of local ventilation to minimize inhalation exposure. However, the hoods sometimes do not provide 100 percent efficient capture due to the bulky characteristics of the glassware. Furthermore, the ventilation systems simply transfer the methylene chloride from the point of glassware rinsing to occupied areas immediately adjacent to the mobile laboratory/field testing

trailer. Methylene chloride poses a risk to field test personnel, plant personnel working in the area of the mobile laboratory/trailer, and agency test observers. On an interim basis, EPA should allow and encourage the use of a less toxic solvent such as n-hexane. EPA should sponsor a set of tests to confirm that n-hexane or another less-toxic solvent provides the sample rinse effectiveness as methylene chloride. [For supplemental to this document, See DCN: EPA-HQ-OAR-2008-0348-0056.1.]

(Comment No. 0118) The use of methylene chloride (a known carcinogen) as the cleaning and recovery solvent will require safety departments to develop procedures for appropriate handling on-site and the use of personal protection equipment for personnel that may be exposed to the solvent. The use of toluene, as specified in EPA Method 23, is a technically acceptable alternative. Please review the use of this solvent as a replacement for methylene chloride in Method 202 (and OTM 028).

Response: The extraction solvent specified in a particular test method is dependent on the analyte(s) of interest. If the target analyte is known, an appropriate solvent can be identified that has the desired recovery performance for that analyte. For Method 202, the pollutant measured by the method, CPM, is defined by the method (i.e., whatever remains after the sample recovery procedures is considered to be CPM regardless of its analyte group). Although no single solvent is universally applicable to all analyte groups, methylene chloride was chosen for the proposed method based upon studies (IERL-RTP Procedures Manual: Level 1 Environmental Assessment; EPA-600/2-76-160a; June 1976) that showed it was the optimum solvent to recovery polar and non-polar CPM.

We acknowledge the commenters' concerns regarding the toxicity of methylene chloride and the exposure hazards associated with its use and we agree that the use of an alternative solvent justified. However, because the recovery performance of solvents has been previously evaluated to support various EPA programs, we disagree with the commenters that additional studies are necessary to identify a suitable alternative solvent.

In identifying an alternative solvent, we initially considered specifying toluene because its extraction performance for polar and non-polar compounds is similar to methylene chloride. However, because the vapor pressure of toluene is lower than methylene chloride, additional time would be needed to evaporate the organic samples to dryness at room temperature (30°C or

less). Because the additional evaporation time would be an additional burden on testing contractors, we rejected toluene as the replacement solvent.

We also evaluated the solvents used for organic compound recovery in the analytical methods under the EPA's Office of Solid Waste (http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/3_series.htm). We reviewed EPA's "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" (SW-846), which was developed to support the Resource Conservation and Recovery Act (RCRA) program, to identify test methods that covered the same types of compounds expected to comprise CPM. Based upon our review of SW-846, we identified Method M-3550c (Ultrasonic Extraction) as a comparable method (M-3550c is used to extract semi-volatile organic compounds from waste samples). Section 7.4 of M-3550c, which discusses extraction solvents, lists the following extraction solvents by class of compound:

- Acetone/hexane or acetone/methylene chloride can be used to extract semivolatile organics;
- Acetone/hexane or acetone/methylene chloride can be used to extract organo-chlorine pesticides; and
- Acetone/hexane, acetone/methylene chloride, or hexane can be used to extract polychlorinated biphenyls (PCBs).

Of the above compound classes, the class that most closely relates to the type of high-molecular weight hydrocarbons expected to comprise organic CPM is PCBs. Hexane is listed as one of the solvents that can be used for extracting PCBs. Hexane is also listed as an alternative solvent (when used in combination with acetone) for the other compounds classes discussed in Section 7.4. Consequently, based upon this analysis, we have replaced methylene chloride with hexane as an acceptable alternative to methylene chloride in the final method.

M202 7.2.3 WATER

Comment: (Comment No. 0038.1) Section 7.2.3 specifies that the water used in this method conform to the standards for ASTM D1 193-06, Type I water. Section 11.2.6 (Container #7) directs that the Water Rinse Field Blank be processed to determine the dry residue mass. This mass is to then be entered in the table in Figure 3. However, this ASTM standard does not

specify a criterion for maximum percent residue by weight, which is the criterion applied to the acetone and methylene chloride reagents, and there is no evaluation of the residue mass determined in the analysis of the Water Rinse Field Blank. To make this section consistent with the reagent specifications for acetone (Section 7.2.1) and methylene chloride (Section 7.2.2), the maximum percent residue by weight should be specified.

(Comment No. 0039.1) What level of residue is allowed for Type 1 water? Maximum residue levels are specified for acetone and methylene chloride, but not water. It was noted that the maximum residue allowed for acetone is 10 percent of that currently allowed (for Method 201A as well) and for methylene chloride the maximum residue allowed is 15 percent of that currently allowed. Was that the intent?

Response: We specified the more stringent residual mass requirements in Method 202 due to the need for improved method performance (i.e., improved method detection limit, improved precision, improved accuracy at low concentrations). While we acknowledge that the solvent requirements are more stringent than the current Methods 202 and 201A, the requirements are comparable with the Method 315 which is essentially the same test method for the organic extractable matter as Method 202.

The purpose of the field reagent blanks is to provide testing contractor with information to target corrective actions, if necessary, if they have difficulty in meeting the residual mass allowance in the method. The method does not require analysis of field reagent blank samples and the field reagent blank values are not used in correcting CPM measurements. However, we acknowledge that Figure 3 could be misleading with regard to the field reagent blanks and we have revised Figure 3 of the final method to note that the entries for the field reagents are not used to correct the CPM measurements.

We agree with the commenter that the method should specify a residual mass level for water used to prepare glassware and recover samples. Therefore, we have revised Sections 7.1.3 and 7.2.3 the final method to specify the glassware preparation and sampling recovery must be conducted using deionized, ultra-filtered water that meets a residual blank value of 1 ppmw or less.

Comment: (Comment No. 0040.2,) In Section 7.2.3, change the text from “Water” to “Low Residue Water”. ASTM Type 1 refers to the production method and does not include a maximum residue value.

Response: We disagree that the commenter’s suggested editorial change to Section 7.2.3 is necessary because the final method has been revised to specify the residual mass level of the water.

M202 7.2.4 CONDENSABLE PARTICULATE SAMPLE DESICCANT

Comment: (Comment No. 0068) Why only sodium sulfate why not a choice of silica gel or sodium sulfate?

Response: The final method does not allow the use of silica gel because it does not reduce the relative humidity in desiccators to sufficient levels to adequately dry the samples in the recovery process. Laboratory evaluations of silica gel and calcium sulfate demonstrate that silica gel is much less effective at removing water/humidity from air in desiccators. Measurements show relative humidity is reduced to approximately 25 percent with silica gel which is higher than ambient relative humidity during cold dry weather conditions. Calcium sulfate reduces relative humidity below 6 percent making it much more effective at removing water from samples. Also, Section 7.3.2 of Method 5 specifies the use of calcium sulfate and we are maintaining this consistency.

Comment: (Comment No. 0040.2) The amount of water that will need to be evaporated from each sample will require larger desiccant adsorptive capacities and regenerative ability. Silica gel has very large adsorptive and regenerative capacities and is extremely efficient at ambient temperatures. Having the option to use both would utilize the best qualities of each desiccant type.

Response: We disagree with the commenter that large amounts of water must be desiccated from samples because the method specifies that samples be evaporated to dryness before desiccation. We also disagree with the commenter’s suggestion to allow the use of silica gel. The final method specifies the use of calcium sulfate because of its superior drying capability.

M202 **7.2.5 AMMONIUM HYDROXIDE**

Comment: (Comment No. 0040.2) In Section 7.2.5, I would suggest adding “low residue” to the Ammonium Hydroxide quality requirement.

Response: We disagree with the commenter that the final rules needs a low-residue qualifier for ammonium hydroxide and note that NIST-traceable ammonium hydroxide is prepared using deionized or distilled water that meets the low residual requirements of the final method.

M202 **8.2 PREPARATIONS**

Comment: (Comment No. 0039.1) Sections 8.2 and 8.4 speak about baking the glassware for 6 hours prior to use for each "source category." Should this requirement be prior to each source test (or source tested)? This removes interpretation as to when the baking is required and provides a greater level of bias elimination for each test.

(Comment No. 0044.1) The proposed method requires that baking of glassware for a minimum of six hours at a minimum of 600°F prior to field tests and that cleaned, baked glassware be used at the start of each new source category tested. BT Environmental Consulting, Incorporated (BTEC) recommends that EPA define source category as "similar sources at a single test site."

(Comment No. 0048.1) We request that EPA further clarify glassware-baking specifications. For example, is it allowed for an air emission testing body to set aside one set of glassware that is baked one time and then use it only for one source category forever?

Response: Stack testers must use clean glassware, prepared as described in Section 8.4, at the start of each testing campaign for each new source category tested at a facility. For example, if CPM measurements are to be taken from two natural-gas fired turbines, the same set of glassware could be used for both emission tests. However, if more than one source category at a facility is to be tested, a new set of clean glassware must be used for each different source category. It is unacceptable to use the same set of glassware for source categories located at

different facilities unless the glassware is cleaned as described in Section 8.4 between facility tests.

We acknowledge that the language regarding the glassware cleaning requirement is unclear. Therefore, we have revised section 8.2 to specify that cleaned glassware must be used at the start of each new source category tested at a single facility.

Comment: (Comment No. 0040.1) The proposed EPA Method 202 Dry Impinger Method incorporates a 6-hour glassware bake at 300°C. Ovens that achieve such a high temperature and are large enough to accommodate all the glassware necessary for typical stack testing projects are expensive and uncommon. The proposed six-hour baking duration is time consuming for stack testing firms that have many projects occurring simultaneously. Based on correspondence with EPA, glassware baking temperatures of 100°C were producing total blank mass values in the 4 to 8 mg range with large variability. There is no laboratory data to determine if a lower temperature could be sufficient to achieve low background masses. This laboratory scope of work covers two lower temperature scenarios. The first is baking the glassware at 200°C for 6 hours and the second is 125°C for 3 hours. In order to obtain an overall estimated total CPM background mass, one set of fluoropolymer filter membranes were processed according to the proposed EPA Method 202 methodology. I suggest allowing the use of baking at 125°C for 3 hours in the proposed EPA202 glassware preparation as a minimum.

(Comment No. 0040.2) In support of lowering the glassware baking temperature and duration, AirNova has performed two additional laboratory studies (additionally provided). Based on this work, I recommend changing the minimum baking requirement to 125 °C for 3 hours.

(Comment No. 0110) We would generally expect that the soap and water and two different polarity solvent rinses would effectively clean the glassware. There is a host of cleaning methods and solutions available in the literature to clean glassware for any conceivable purpose. Is there some technical basis for the 300°C baking temperature, such as volatilizing vacuum greases or other common contaminants? Or was this simply what was used during method development? Has the 300°C step been shown in field work to be necessary? What is the temperature tolerance on the 300°C? Are there blank considerations such as cut-off levels that might allow for streamlining certain steps, most notably, the high-temperature baking?

(Comment No. 0048.1) GE Energy has conducted experiments (attached) with baked and unbaked glassware and has found no benefit to the baking of glassware prior to use. It is our recommendation that these requirements be removed from the method. If the air emission testing body reports values significantly higher than 2.0 mg already allowed and that results in the source failing the test, the client will be able to exercise their right to hire a more diligent air emission testing body.

(Comment No. 0050.1) Proposed RM 202 (74 FR 13004, Section 8.2) Preparations, requires baking the glassware at 300°C for 6 hours. The data supporting the need for glassware to be baked at this high temperature for an extended time period cannot be readily obtained from the supporting information in the docket. The Alliance understands that the "Draft Project Report – Evaluation and Improvement of Condensable Particulate Measurement" may contain this information and recommends that the effect of pre-bake temperature and time on cleanliness of blanks be clearly presented in this report and include a table comparing the effect of 300°C for 6 hours versus lower glassware preparation temperatures. The requirement to bake glassware at 300°C for 6 hours should be optional because it has not been possible to fully evaluate the supporting data and the need for such high temperature is not readily apparent for all situations. Otherwise, this requirement would require the stack tester to bring to the testing site a large amount of pre-cleaned glassware, much more than what is currently normal for such testing. As an option, the Alliance recommends the tester start with baked glassware for the first test and then be allowed to perform additional tests reusing the same glassware after it has been cleaned by chemical methods. If the chemical cleaning of the glassware is not adequate, the blank values would likely elevate, possibly eliminating the test from consideration. If the blanks do not elevate, this scenario would be very cost effective and would conserve resources.

(Comment No. 0054.1) The requirement for baking the glassware at 300°C for six hours is a topic that needs to be addressed. We have not conducted experiments to see if the glassware baking is necessary or to what advantage this might have over just rinsing clean glassware with the same types of solvents that will be used for the sample recovery. Without the glassware baking we have conducted numerous test runs on non-combustion sources that have had results that would be acceptable blank results. There might be some sources that would require a 300°C baking of the glassware after testing to render the glassware clean, but this does not seem to be necessary for the majority of tests. Could this be reworded to help explain better why this

requirement was created? What compounds do we think will not come off the glassware with washing, methylene chloride and acetone rinsing, but needs to be baked off at 300°C because it will end up in the sample? If it is some acid reaction that is expected, perhaps an acid bath could be an alternative?

(Comment No. 0056.1) On page 42 of the Preamble and in Proposed Method 202, EPA proposes glassware cleaning at 300°C (572°F) for a period of 6 hours. This is excessive. API believes that glassware is sufficiently clean when the glassware is baked in a manner consistent with EPA Reference Method 23 (450°F for two hours).

(Comment No. 0061.1) On page 42 of the Preamble and in Proposed Method 202, EPA proposes glassware cleaning at 300°C (572°F) for a period of 6 hours. Glassware is sufficiently cleaned when it is baked in a manner consistent with EPA Reference Method 23 (450°F for two hours).

(Comment No. 0066) Regarding the glassware baking temperature, EPA has established performance-based criteria for allowable contamination for this method of 2 mg. However, EPA still clings to the prescriptive requirement of baking glassware at 300°C. There is no need for this. Testers should be allowed to achieve the 2 mg blank requirement in any way they choose. While we would have no objection to a note in the method stating that the 2 mg blank value was derived from glassware cleaning at 300°C, we do not feel there is any purpose in maintaining the prescriptive requirement for that temperature, particularly in light of the fact that there is no data showing that the 2 mg blank cannot be achieved at lower temperatures or through other means. Furthermore, most testing companies do not currently have ovens that can achieve these temperatures. Ovens that can achieve these temperatures cost between \$4000 and \$6000.

(Comment No. 0041.1) Section 8.4 states that removal of all silicone grease from areas that will be exposed to the methylene chloride rinse during sample recovery. After cleaning, you must bake glassware at 300°C for 6 hours prior to each source type sampled. The requirement to bake the glassware at 300°C for 6 hours is unnecessary. For more than 15 years, sample train glassware manufacturers have offered impingers with Viton O-rings which are much more effective at preventing leaks than silicone grease. Very few stack test companies use glassware that does not have O-rings. Very few stack test companies use silicone grease on any glassware due to potential contamination issues. As a result, the presence of silicone grease on impingers or glassware is highly unlikely. The sample train glassware cleaning procedure stated in Section

8.4 of Method 202 (i.e., soap and water, and rinsed using tap water, deionized water, acetone, and finally MeCl_2 will remove organic or inorganic residues. After this cleaning procedure, baking the glassware at 125°C for 3 hours is adequate to finish the sample train glassware cleaning process. Joe Jackson of AirNova has submitted comments to this docket along with laboratory data demonstrating that baking the sample train glassware at 125°C for 3 hours resulted in acceptably clean glassware. Revision of the 300°C and 6 hour requirement is important for the following reasons: (1) Modern sampling train glassware is equipped with Viton O-rings. Baking at 300°C will destroy or deteriorate the Viton O-rings. The effort to remove these O-rings before baking and then replace them after baking is time consuming. (2) Laboratory ovens capable of reaching 300°C and maintaining it for 6 hours are much more costly than standard laboratory ovens that generally achieve 150 to 200°C temperatures. An oven capable of 300°C costs \$ 4,000 to \$ 5,000, compared to the cost of an oven capable of 200°C which costs about \$ 1,000.

(Comment No. 0068) Section 8.4 requires baking of glassware after cleaning at 300°C for 6 hours. The requirement for glassware baking only prior to the test makes little sense. Why not rinse with the recovery solvents as we will in between runs? You already mandate a reagent blank why not change that to a proof blank with a limit? If above the limit, re-rinse and re-weigh.

(Comment No. 0044.1) The recommended high temperature baking and cleaning adds cost and unnecessary time delays as it requires the test firm to keep multiple sampling trains, and other related equipment.

(Comment No. 0110) We would generally expect that the soap and water and two different polarity solvent rinses would effectively clean the glassware. There is a host of cleaning methods and solutions available in the literature to clean glassware for any conceivable purpose. Is there some technical basis for the 300°C baking temperature, such as volatilizing vacuum greases or other common contaminants? Or was this simply what was used during method development? Has the 300°C step been shown in field work to be necessary? What is the temperature tolerance on the 300°C ?

(Comment No. 0110) In general, the method is quite proscriptive [sic] instead of being performance based. Are there blank considerations, such as cut-off levels, that might allow for streamlining certain steps (most notably, the high-temperature baking)?

Response: Method 202 has the potential to measure CPM at very low levels. Consequently, the glassware used in the sampling train must be free from contamination to maximize the precision of the accuracy of the CPM measurements. The glassware cleaning requirements contained in the proposed revisions to Method 202 were based upon experimental results that indicated that the maximum blank correction of the method (2 mg) could not be achieved without thorough cleaning and baking of the glassware at 300°C for 6 hours. However, based upon our review of the comments, we have determined that it is appropriate to provide a performance-based option for demonstrating the cleanliness of glassware.

As an alternative to baking glassware as required in the proposed changes to Method 202, Section 8.4 of the final method has been revised to allow testing contractors to perform a field train proof blank of the assembled glassware in the field prior to conducting emission tests using the sampling train.

Comment: (Comment No. 0048.1) Section 8.2 says to analyze reagent blanks (water, acetone, and methylene chloride) before field tests to verify low blank concentrations. What is the intent here? If an air emission testing body has three or four different field jobs in one week, do they need to have reagent blanks for each “field test” or can we just run one set. It is our recommendation that EPA change this requirement to allow running reagent blanks on each new lot of reagent.

Response: The purpose of the laboratory reagent blanks is to confirm the residual mass contained in the reagents that will be used for sample recovery. In the proposal, we intended to require testing for each lot of reagents received by the testing contractor. However, we agree with the commenter that the language in Section 8.2, as proposed, is unclear with regard to reagent testing. Therefore, we have revised Sections 8.2 and 9.7 of Method 202 to clarify the use of laboratory reagents.

Comment: (Comment No. 0056.1) API agrees with the pretest reagent blank analyses. This is a prudent procedure that will minimize tests with unacceptable blank levels.

Response: EPA acknowledges the commenter’s support.

M202 8.3 SITE SETUP

Comment: (Comment No. 0057) Section 8.3 (Site Setup) specifies that, "You must follow the procedures required by filterable particulate sampling method setup run in conjunction with this method including: . . ." Because 8.3(b) may not always be applicable, depending on the filterable method used, please revise this section to state "...in conjunction with this method, which may include: . . ."

Response: Our intent in Section 8.3 is to prescribe the site setup procedures. However, we acknowledge that Section 8.3(b) may not always be applicable. Therefore, we have revised Section 8.3 to accommodate tests that do not require the use of cyclone or particulate separators.

M202 8.3.1.2 TRAVERSE POINTS

Comment: (Comment No. 0048.1) We recommend that the EPA change “recommended maximum” number of traverse points to “required” number of traverse points.

Response: We agree with the commenter’s editorial suggestion. Method 1 (Traverse Points) specifies the minimum number of traverse points for sampling locations and the term “maximum” is not used in Method 1. Therefore, section 8.3.2 has been revised to remove the language regarding the recommended maximum number of traverse points.

M202 8.4 SAMPLING TRAIN PREPARATION

Comment: (Comment No. 0048.1) In Section 8.4, EPA refers to “all sampling train glassware must be cleaned...” We assume that this requirement only applies to the back-half glassware prior to the CPM filter, but whatever the intent, it is our recommendation that it be specifically stated.

Response: We acknowledge that the language used in Section 8.4 of the method to specify the glassware to be cleaned could potentially be confusing. Our intent was that all glassware used in Method 202 be cleaned prior to use at each facility for the same type of source.

To clarify our intent, we have revised the text of the second sentence in Section 8.4 of the final method to remove the reference to “sampling train.”

Comment: (Comment No. 0048.1) It is our recommendation that EPA should ban the use of silicone or any other grease for this or any other method that uses glassware. Impingers are available with Viton O-rings or Teflon® tape can be used on ground glass joints. These alternatives should be mandated.

Response: We disagree with the commenter that it is necessary to ban the use of silicone or other types of grease in the Method 202 sampling train. Although we acknowledge the alternatives suggested by the commenter, we believe that the requirement in Section 8.4 to completely remove all grease from areas that will be exposed to the extraction solvent during sample recovery and that the maximum allowable residual mass for field train recovery blanks in Section 9.10 are sufficient to prevent biasing the CPM measurement values.

M202 8.4.2 BACKUP IMPINGER

Comment: (Comment No. 0057) Section 8.4.2 (Backup Impinger) says to, "Place the dropout and other impingers in an insulated box...." What are the "other impingers?" Please state or remove from text.

Response: We agree with the commenter that the text in Section 8.4.2 is not clear with regard identifying the impingers. Therefore, we have revised Section 8.4.2 of the final method to clarify references to the backup impinger.

Comment: (Comment No. 0059.1) The proposed Method 202 suggests the use of two insulated impinger boxes. The first contains water and the recirculation pump used to maintain a CPM filter temperature of less than 30°C/85°F. The second contains ice water to maintain a silica gel exit gas temperature of less than 20°C/68°F. An acceptable alternative should include a single insulated box containing all impingers, the recirculation pump and containing an ice bath capable of maintaining both the CPM and silica gel temperature requirements.

(Comment No. 0113) If a lower temperature limit is not specified for the exit temperature of the CPM filter, is it necessary to have the dropout impingers separate from the ice bath impingers?

Response: The purpose of using divided impinger boxes with somewhat different temperature requirements is to minimize the potential to form sulfate artifact in the first two impingers and to maximize the collection of water vapor for calculating the moisture content of the sampled gas. To provide stack testers with flexibility, Method 202 does not specify the configuration of the insulated boxes needed to contain the pre- and post-CPM filter impingers. Testing contractors are free to use any configuration of impinger boxes that comply with the method requirements (e.g., place the first two impingers in the same box or part of the box for the impingers that are intended to collect water vapor passing through the first two impingers and the back up filter). However, testing contractors should use discretion when configuring the impinger boxes. Reducing the temperature of the first impingers increases the concentration of SO₂ in the water collected in the impingers which could potentially increase artifact formation.

M202 8.4.3 CPM FILTER

Comment: (Comment No. 0040.2) In Section 8.4.3, I would like to suggest adding the following text to the end of the paragraph: “An optional support media can be inserted behind the CPM filter to aid in vacuum reduction. This media is not analyzed as part of the CPM but should meet the chemically inert requirements of Section 6.1.3.” Our studies have demonstrated an effective decrease in the initial system vacuum of approximately 2 inches of mercury by supporting the 64-mm diameter PTFE filter membrane with a 64 mm-diameter quartz filter backing.

Response: We agree with the commenter that the use of a support for the filter media in the CPM filter can be helpful and we have revised Section 6.1.3 (proposed Section 6.1.2) to allow the use of a support for the filter. The filter holder and support filter would not be analyzed as part of the CPM measurements.

M202 **8.4.4 MOISTURE TRAP**

Comment: (Comment No. 0069) Clarify that the post-CPM impingers do not have to contain Type 1 water. Suggestion: The water used in the post-CPM impingers is not required to be ASTM D1193-06 Type 1 water or equivalent.

Response: We believe that the text in Section 8.4.4 requiring the use of water, or the alternative in Section 6.1.1.1 of Method 5, is sufficient and provides the stack tester with flexibility when using Method 202.

M202 **8.4.6.1 SAMPLING TRAIN**

Comment: (Comment No. 0054.1) To be consistent with other EPA methods the text “Sampling Train. You must pretest the entire sampling train for leaks.” could be replaced with this part from Method 5, “8.4.2 Pretest Leak Check. A pretest leak check of the sampling train is recommended, but not required. If the pretest leak check is conducted, the following procedure should be used.” I agree that it is a good idea to do this, but the way it is worded it is unclear if the run would be invalidated if the pre-test leak check was not done at a high enough vacuum or if it was leaking at greater than 4 percent of the sample flow rate even though it was done at a much higher vacuum.

Response: The revised Method 202 has the potential to measure CPM concentrations at very low levels. The intent of the pre-test leak check is to minimize the potential for creating an upward bias in CPM measurements due to contaminants that are drawn into the sampling train from the ambient air at the test location. We believe that the pre-test leak requirements in Section 8.4.6.1 for the maximum allowable leak rate and the vacuum to be used during the leak-check, as proposed, are clear. Therefore, we are not revising the pretest leak-check requirements.

Comment: (Comment No. 0044.1) The proposed method requires that the CPM filter readings be less than 30°C (less than 85°F). However, if the CPM filter temperature is substantially lower than 85°F, additional organics could condense and be counted as CPM. BTEC has provided technical data documenting this finding (see "Potential Effect of Condensation Temperature on Condensable Particulate matter Measurements via Method 202," J. Moldovan and M. Distler, AWMA National Conference, June 2007.). BTEC recommends that the temperature downstream of the CPM filter be within a range from 10 to 30°C (50 to 85°F). This range will help to "standardize" the CPM data with respect to sampling temperature.

(Comment No. 0050.1) Proposed Method 202 (Section 8.5.1) specifies that the sampling air temperature at the filter must be less than 30°C during sample collection. The draft method does not provide a lower sampling air temperature criteria which is important. One of the EPA docket reports (EPA Contract No. EP-D-07-097) provides technical information that some larger organic compounds will be collected by Method 202. Although some maybe lost with purging, it is possible that some compounds will react forming larger or more stable compounds. These compounds will not be lost with purging and will be counted as PM_{2.5} rather than VOC. In general, the lower the air sampling temperature at the CPM filter, the more organics will condense as they reach their dew points. One of the Alliance member companies has provided technical data documenting that CPM increases with decreases in air sampling temperature. (See "Potential Effect of Condensation Temperature on Condensable Particulate Matter Measurements via Method 202," J. Moldovan and M. Distler, AWMA National Conference, June 2007). This issue is important when testing a source that contains organics and has a stack temperature exceeding 30°C. For example a coal-fired boiler may exhaust to the atmosphere at 120°C. If air Test No. 1 was performed with a CPM filter temperature of 30°C and Test No. 2 with a CPM filter temperature of 0°C, results from the two tests will not be identical due to the large difference in air sampling temperatures. Therefore, the Alliance recommends that EPA consider setting a temperature range at the CPM filter, from 15 to 30°C, rather than simply less than 30°C, which leaves no lower bound. This temperature range will help to "standardize" the collected CPM data with respect to air sampling temperature, which we feel is a goal of EPA.

Response: Although much of the commenters' arguments are conjecture, we agree with the concern raised by the commenters to standardize testing. The EPA experimental work conducted at a hog fuel-fired boiler (*Field Evaluation of an Improved Method for Sampling and Analysis of Filterable and Condensable Particulate Matter*; Docket no. EPA-HQ-OAR-2008-0348) indicates that CPM material above theoretical estimate of vapor pressure was lost which confirms the need for a lower temperature limit. We also determined that laboratory analysis of CPM, including evaporation to constant weight at standard laboratory temperature, normalizes the retention of CPM. To improve precision, EPA has revised Section 8.5.1 of the final method to set a lower temperature bound of 20°C (65°F) for the CPM filter based upon similar requirements in methods (e.g., Method 0010) used to collect organic material from stationary sources.

Comment: (Comment No. 0111) Looking over this method, it seems to be vague about the specifics of the temperature of the external filter and how it is achieved. I know that it is stated in Section 8.5.1 to maintain the CPM filter temperature less than or equal to 30°C (less than or equal to 85°F) during sample collection. Figure 1 in Section 18 clearly shows two sections in the cold box portion of the train. Why is this? The iced portion of the box (back half), used to drop out any remaining moisture, will be below 68°F. Does this not satisfy the condition of being less than or equal to 30°C (less than or equal to 85°F)? Could we not simply use a non-divided box and circulate the ice water through our condenser to achieve a filter temperature of less than or equal to 30°C (less than or equal to 85°F)? The entire premise of the method deals with this condensation temperature of 85°F. Any gas passing through the train at temperatures less than 68°F will more than satisfy this condition. Again, why have two separate temperature requirements when 68°F is obviously less than or equal to 30°C (less than or equal to 85°F)?

(Comment No. 0112) Is there any reason that ice water can't be used in the first section of the sample box where the knockout impinger needs to be kept at less than 85°F? Section 8.4.2 has the less than 85°F requirement, but I don't see why it would hurt to use ice water to cool the impinger.

Response: EPA's intention in the proposed Method 202 was to have the CPM collection surfaces maintained as close to 30°C (85°F) as practicable. The purpose of using divided

impinger boxes with somewhat different temperature requirements is to allow an elevated temperature for CPM collection and a reduced temperature for moisture collection. The elevated temperature for CPM collection is intended to minimize the potential to form sulfate artifact in the first two impingers while maximizing the collection of water vapor after the CPM filter. If the increased potential to form sulfate artifact in the first two impingers is not a concern, then the first two impingers can be contained in the same box or part of the box with the impingers that are intended to collect water vapor. If one impinger box is used and maintained at ice water temperature, then the temperature at the exit of the CPM filter should be much lower than 85°F which satisfies the requirements of the method. In response to other comments received, EPA has modified the method to specify a lower bound temperature on the CPM collection temperature. Testers have the flexibility to assemble the required sampling train components in one or more impinger boxes to meet the CPM and moisture collection temperature requirements in the method.

M202 8.5.2 LEAK-CHECK PROBE/SAMPLE TRAIN ASSEMBLY

Comment: (Comment No. 0057) If a post-test leak-check fails to meet the specified criteria, can leak rate adjustment calculations (EPA Method 5, Section 12.3) be used to correct the sample volume and provide a valid test run?

Response: The use of leak rate adjustment calculations, such as the modifications specified in Section 12.3 of EPA Method 5, is not allowed in Method 202. Because Method 202 has the potential to measure very low levels of CPM concentrations, we believe that the error introduced in the CPM concentration measurements by correcting the dry gas volume measurements based upon pre- or post-test leak-checks is unacceptable. When Method 202 is used in conjunction with Method 201A, the volume correction does not correct for nonisokinetic sampling. Therefore, for consistency, EPA does not allow correction of dry gas volume based upon pre- and post-test leak checks for Method 202.

M202 8.5.3 POST-TEST NITROGEN PURGE

Comment: (Comment No. 0089.1) SGCI supports EPA's proposal concerning Method 202 that the stack tester purge the impingers with nitrogen gas for one hour as soon as possible after the post-test leak check. Data reported by EPA support that the one hour nitrogen purge should reduce, but not eliminate, CPM testing artifact. Thus, SGCI supports this proposed amendment to improve the accuracy of the test results.

Response: We acknowledge the commenter's support for the nitrogen purge requirements.

Comment: (Comment No. 0041.1) Section 8.5.3 should be revised to clarify that the sample train may be purged without having the meter box in place as shown in Figure 2 of the proposed method. In practice very few sample trains are purged at the stack sampling location and very few are purged with a meter box / dry gas meter following the impingers. To require a meter box/dry gas meter during the purge is unnecessary. To do so places an unnecessary burden on test firms to bring extra meter boxes/dry gas meters to job sites. It is not practical to delay the start of the next test run while the post-test purge of the previous test run is underway. The pressure in the nitrogen purge cylinder is more than adequate to force the nitrogen through the sample train. Most test firms bring the sample train glassware to the sample recovery location (usually cleaner and safer than the stack location) or an onsite trailer for nitrogen purging and sample recovery. Typically, there is not enough room at the stack sampling location to purge sample trains, and it is usually impractical to haul nitrogen purge cylinders up to the stack locations. Once at the sample recovery location, the water dropout or knockout impinger needs to be inspected to determine if any moisture was condensed. At that time it can be weighed along with the other impingers, to determine the moisture content. Weighing the impingers typically takes a few minutes (certainly less than 5 minutes). By weighing the impingers prior to the nitrogen purge a more accurate moisture catch is determined and the need to measure the amount of degassed deionized water that is added (if any) is eliminated.

(Comment No. 0086.1) A number of organizations have submitted comments recommending changes in the required purging procedures. For example, Owens Corning Science and Technology, Inc. (Docket Document 0072) has recommended that test crews move the Proposed Method 202 sampling train to a clean-up trailer or laboratory prior to starting the purge. They are also concerned about transporting a nitrogen cylinder to the stack or roof

sampling location. Organizations submitting comments concerning purging include Barr Engineering (Docket Document 0054.1), TETCO (Docket Document 0060.1), TRC Environmental (Docket Document 0041.1), and New Jersey DEP (Docket Document 0039.1). These comments appear to result simply from a lack of clarity concerning EPA's intent with respect to the nitrogen purge. Testing firms experienced with the predecessor method (OTM 028) immediately move the sampling train to a mobile laboratory and begin purging using a nitrogen cylinder. The short time required to move the sampling train does not substantially increase the time available for aqueous phase oxidation of dissolved gases. EPA should eliminate the portion of Figure 2 that shows the meter box and revise the text in Proposed Method 202 to require purging in a clean environment without the need for a meter box.

(Comment No. 0060.1) Section 8.5.3.3 of the proposed Method 202 requires that the impinger train be purged while still connected to the meter box and vacuum line. I suggest allowing the impinger train to be brought down off the stack prior to purging and allow the use of a rotameter to monitor the volume of nitrogen purge rather than requiring the impinger train to stay connected to the meter box. After the final leak check, the open ends of the impinger train can be capped during transport to reduce the possibility of oxygen contamination; by capping the ends the sample will not be exposed to any more air than when immediately connecting to the nitrogen purge line. The first advantage is that it uses a rotameter to regulate the flow and avoids using the meter box to regulate the nitrogen purge flow rate and reduces the chance of pressurizing the impinger train. Pressurizing the impingers and trying to regulate the volume with the meter box will lead to "popping" open some impingers during the purge. I have observed impingers popping open while conducting Method 11 tests for H₂S samples at refinery fuel gas lines that are under pressure when the flow rate is regulated with the meter box. The flow is better regulated upstream of the impingers and not downstream with the meter box. The second advantage is that disconnecting the impinger train from the meter box for the purge will lessen down time on the stack for the test crew while waiting for the purge before continuing with the next sample test run. This will save money for industries and will not reduce the effectiveness of the nitrogen purge.

(Comment No. 0050.1) The proposed method requires that the purging be conducted across the entire sampling train, from probe nozzle to the dry gas meter as shown in Figure 2. The Alliance sees two potential problems with this requirement. First, if the air pulled by the gas

meter does not exactly balance the bottle gas purge of 20 liters/min there may be a system air imbalance. This may result in leaks or even impinger failures or ruptures, and could create safety issues. Second, by having to purge through the entire air sampling train as shown in Figure 2, it forces the tester to have extra sample trains specifically for purging. This can delay testing and will increase cost.

(Comment No. 0054.1) It would be more efficient and require less equipment if you could use the pressure from the nitrogen cylinder to deliver the 20 liters/min instead of using another meter setup. This would also reduce the possibility of passing ambient air through the impingers.

(Comment No. 0057) Section 8.5.3.3 says "To avoid over-or under-pressurizing the impinger array, slowly commence the nitrogen gas flow through the line while simultaneously opening the meter box pump valve(s)." Could the impinger train be purged without being connected to a meter box and pump? Purging while connected to a meter box and pump occupies this equipment.

(Comment No. 0069) If nitrogen is pushed through the train during the purge, the method should specifically allow the post-CPM impingers to be removed from the train before starting the purge. If the train will be purged by pushing nitrogen through the sampling train, then the components after the thermocouple of the CPM filter may be disconnected from the train before beginning the purge.

(Comment No. 0059.1) Suggest that EPA consider allowing a positive pressure nitrogen purge (similar to that in EPA 202) as an option instead of utilizing a meter box.

(Comment No. 0041.1) Figure 2 should be revised to illustrate the nitrogen Purge Train without the vacuum line to a meter box and without passing through the condenser.

(Comment No. 0044.1) The Method 5 sample train and pump are not necessary and may cause back pressure on the impinger train. Purging of the entire test train also forces the operator to be equipped with a second sample train specifically for purging.

(Comment No. 0103) The purge flow and equipment configuration seems a bit ambiguous. Section 8.5.3.3 requires the nitrogen flow to be either the delta-Hat of the meter box or 20 liters/minute and show a bypass ahead of the impingers. Figure 2 shows the impinger train connected to a meter box and a high-range rotameter that's labeled for use when pushing nitrogen through the impingers. Does the nitrogen have to be pulled through the impingers? If

nitrogen can be pushed through, is the meter box needed? If nitrogen can be pushed, what is the purpose of the bypass rotameter ahead of the impinger train?

Response: It was our intent in the proposed Method 202 to allow testing contractors the option of conducting either a pressurized purge (i.e., without the dry gas meter box and pump attached to the sampling train) or a vacuum purge (i.e., with the dry gas meter box attached to the sampling train). However, we acknowledge that the language in Section 8.5.3 and the sampling train depicted in Figure 2 of the proposed method were unclear. Consequently, we have revised Section 8.5.3 and Figure 2 of the final method to clarify that a pressurized purge is an acceptable alternative.

With regard to the commenters' suggestion to allow testing contractors to conduct the nitrogen purge at the sample recovery location, instead of at the sampling location, we did not specify in the method where the purge had to be conducted to provide flexibility to testing contractors. We continue to believe that testing contractors should have the flexibility to conduct the nitrogen purge at the location of their choosing; therefore, the final rule does not specify where the purge must be conducted. However, testing contractors should conduct the purge as soon as practicable after the post-test leak-check to reduce the potential for artifact formation in the impinger water.

Comment: (Comment No. 0044.1) The proposed method does not allow for disassembly of the condenser and impinger train prior to initiating the purge steps. BTEC proposes that disassembly of the condenser and impinger train be allowed prior to initiating the purge steps. Specifically, we recommend that the impingers be weighed first and that the nitrogen purge be performed only on the two impingers upstream of the CPM filter, the CPM filter, and the one downstream impinger. This procedure will save time and cost and minimize the equipment needed for re-assembly and further stack testing (See the attached Figure 1). (Comment No. 0044.1) BTEC proposes that Figure 2 in the proposed method be modified to only include purging of the first two impingers and the CPM filter. The third impinger is only used to hold the CPM filter.

(Comment No. 0046.1) Owens Corning recommends that the proposed testing protocol be modified to allow for the tester to disassemble the impinger train to measure for moisture content prior to conducting the required nitrogen purge. It has been our practice to clean-up the

impinger train in a clean environment. We have purposely avoided doing so on the roof because of the inconvenience and the potential contamination resulting from the proximity to the sampling stack and other emission sources. The change will allow us to continue this practice. We are also concerned that being required to transport a nitrogen cylinder to a roof, to conduct the purge there, unnecessarily raises significant safety issues. Those issues can be avoided by allowing the tester the option to conduct the nitrogen purge in a clean environment away from the roof. We recommend that the tester also be allowed to purge the impinger train without the use of a sampling meter box as long as it is done in a clean environment. It has been our experience with this method that because the purging process is performed in a clean environment, there have not been problems with contamination of the sample.

(Comment No. 0050.1) We would like to offer an alternate procedure for purging. We recommend that after the final leak-check, if required, the stack tester be allowed to disassemble the sampling train glassware and purge only the two impingers upstream of the CPM filter, the CPM filter and the one downstream impinger. This procedure will eliminate the noted problems, save time, cost and minimize the equipment needed for re-assemble, reduce the possibility of contamination during this part of the stack test procedure, and then continue with the next stack test run. (See attached drawing or Figure No. 5 of the recommended purge train).

(Comment No. 0059.1) On a recent test program using the proposed 202 revisions, we transferred all of the condensate catch into the second dry impinger prior to purging. Our intention was to reduce potential sources of contamination wherever possible. The proposed method suggests inserting a new long-stem impinger insert into the first dry impinger and then performing the nitrogen purge. Our approach eliminates "opening" the first impinger and inserting a new piece of glassware. Is this approach acceptable?

(Comment No. 0048.1) The manipulation of the impinger stem and the addition of water in Section 8.5.3.2 seem counterproductive to greater resolution of minute quantities of CPM likely to be entrained in these impingers. We recommend transferring any liquid in the first (dry) impinger to the second impinger, then purging.

(Comment No. 0050.1) Section 8.5.3.2 requires that, if water was collected, the first short stem impinger be replaced with a long stem modified Greenburg Smith insert and that enough water be added to cover the tip of the impinger. This step has the potential to introduce sample bias into the sample train by installing a new piece of glassware at the conclusion of the test run.

As an alternative, the Alliance recommends that the language be changed to require any liquid collected be transferred to the second impinger and that the first impinger be removed from the train prior to purging. The Alliance recommends the following language in section 8.5.3.2: “In the event that water is collected in the first impinger, remove the impinger from the sample train and transfer its contents [collected water] to the second impinger. The impinger tip length must extend below the water level in the impinger catch. If insufficient water was collected, you must add a measured amount of degassed, deionized, distilled ultra-filtered ASTM D1193-06, Type 1 or equivalent (incorporated by reference) water until the impinger tip is at least 1 cm below the surface of the water. You must record the amount of water added to the dropout impinger (see Figure 4 of Section 18) to correct the moisture content of the effluent gas. (Note: Prior to use, water must be degassed using a nitrogen purge bubbled through the water for at least 15 minutes to remove dissolved oxygen.)”

(Comment No. 0041.1) Figure 2 shows the purge line connected to the condenser. The narrative in Section 8.5.3 describes replacing the short stem impinger stem with a long stem so that the nitrogen purge passes through the condensed water in the water dropout impinger. Little or no (less than 1 ml) moisture will reside in the condenser as they are [sic] designed to drain into the water dropout impinger. Therefore, it should not be necessary to nitrogen purge the condenser. The nitrogen purge should pass through the water in the water dropout impinger. The method should allow the purge line to be connected to the water dropout impinger. The condenser should be rinsed and recovered as described in Sections 8.5.4.

Response: We agree with the commenters that testing contractors should be allowed the option of transferring any water collected in the first impinger to the second impinger and performing the nitrogen purge on the condenser, second impinger, and the CPM filter. The condenser must be included as part of this purge option to maintain the purge gas temperature between the specified temperature limits. However, to use this option, testing contractors must determine the amount of moisture collected in the first two impingers and the moisture trap impingers in the field. To provide this option, we have revised Section 8.5.3.2 of the final method.

Comment: (Comment No. 0060.1) Section 8.5.3.2 of the proposed Method 202 requires the replacement of a short stem impinger insert with a modified Greenberg Smith impinger insert

prior to the nitrogen purge. I suggest changing this, or at least allowing the option to replace the entire first impinger with a complete, clean impinger with a modified Greenberg Smith insert. Any collected water from the knock-out impinger should be transferred to the replacement impinger. This change has two advantages over the current proposal. The first advantage is that replacing the entire impinger lessens the possibility of ambient air dust contaminating the sample while opening the impinger. Sampling locations are often less than ideal (dusty), and dust settles on the neck and crack between the impinger bottle and insert, and changing the insert will allow dust to enter the sample. The second advantage is that changing the insert will lead to broken inserts while trying to separate the knock-out insert from the impinger bottle. Almost all current impingers are made to use O-rings between the impinger bottle and insert. The advent of O-ring impingers avoids the use of vacuum grease and provides better leak checks than the old, ground-glass fittings; however, the O-ring connection can become very stiff and hard to disconnect, especially in the field and on the stack. Changing the entire impinger rather than the insert will avoid these two problems.

Response: We agree with the commenters' suggestion regarding the transfer of liquid from the first impinger to the second impinger prior to purging and we have revised the final method accordingly. However, we disagree with the commenter's suggestion to remove the first impinger from the sampling train and replacing the original impinger with a second impinger with a long stem insert. Using a second impinger, as the commenter suggests, risks introducing additional glassware contamination which negates the advantages of the commenters suggestions.

Comment: (Comment No. 0048.1) To avoid compounding errors associated with adding water for the nitrogen purge, we recommend the option of inserting a Teflon® tube (1/4 inch or 1/8 inch) through a stopper into the impinger arm, and then into the liquid. This would alleviate the need to break the fitting or add water, and prevent the potentially compounding error of water addition.

(Comment No. 0057) Section 8.5.3.2 says to "Replace the short stem impinger insert with a modified Greenberg Smith impinger insert." As an alternative, could a Teflon® line be inserted down and through the short stem impinger extending below the water level in the impinger catch? The Teflon® line would be inserted through a compression fitting in a socket

fitting and clamped on top of the short stem impinger. Nitrogen would then be purged through the sample line. This would reduce the potential for breaking glassware and contamination when removing/inserting glassware stems.

(Comment No. 0044.1) The proposed method states that the short stem impinger should be replaced with a modified Greenberg Smith impinger so that the tip of the probe insert is below the water level. The current Method OTM 28 impinger train utilizes a vertical condenser followed by a 1-piece pot-belly knock out impinger which has no stem. BTEC proposes an option to use a Teflon® socket with a precleaned Teflon® line that can be placed in the impinger to facilitate the purge (See the attached Figure 2).

Response: We disagree with the commenter's suggestion to insert a Teflon tube into the first impinger for conducting the nitrogen purge. Using the configuration suggested by the commenters, there is no provision to maintain the temperature of the purge gas. Therefore, EPA believes that a Teflon® or other inert line used to purge the CPM train is not an acceptable alternative. Consequently, Section 8.5.3.2 has not been revised to allow the use of a Teflon® tube or other inert lines when conducting the nitrogen purge.

Comment: (Comment No. 0054.1) The nitrogen purge should not be required at sources where no SO₂ is present (or other APM). The nitrogen purge should be optional just like in Method 202 because this step has no benefit at many sources and will just add to the cost of testing those sources.

(Comment No. 0040.2) The commenter recommends that the method state that a purge is necessary for the field blank train when nitrogen purges are not used.

(Comment No. 0058.1) The bulk of the power plants in Maricopa County are natural gas-fired combined cycle units that use pipeline-quality natural gas with very low sulfur content. The lab analyses of the natural gas fuel samples at these plants typically show a very low sulfur concentration (1 or 2 ppmv). The department generally allows the omission of the nitrogen purge of the Method 202 due to the very low SO₂ content of the fuel. Nitrogen purging in these cases is therefore unnecessary since there is practically no SO₂ in the test train. The new method as proposed appears to require the nitrogen purge for all cases. A nitrogen purge will add over 1-hour per test run (1-hour nitrogen purge time plus set up time). Is it possible to leave the nitrogen purge optional (as it is in the current Method 202) for those sources where SO₂ artifact

formation is not a concern? If not, then it is recommended that language be added allowing exemption of the nitrogen purge in cases where the SO₂ is not likely to be present.

Response: We disagree with the commenters. Method 202 requires a nitrogen purge to ensure that the operation of the method is consistent among different stack testers. We believe the likelihood of no artifact formation using the unpurged Method 202 is very small based on work performed by Wien (e.g., Wien, S., et. al., 2004). Their studies showed a majority of the fine particulate from low sulfur gas fired sources was due to condensable material. Their results also showed 60 to 90 percent of the condensable inorganic mass from these low sulfur fuel combustion sources was sulfate. We have determined that it is too difficult for stack testers to judge whether the nitrogen purge is necessary based upon characteristics of the emission source. With regard to the additional time required for the nitrogen purge, the sampling train glassware can be removed and purged separately which would remove the delay.

Comment: (Comment No. 0059.1) On a recent test program using the proposed 202 revisions, we collected approximately 150 ml of water in the first dry impinger and approximately 5 ml of water in the second dry impinger. These liquid catch volumes were low enough that during the test run the sample gas never bubbled through the liquid catch in dry impingers 1 and 2. Since sample gas never bubbled through the condensate catch, is it necessary to perform the nitrogen purge and does this nitrogen purge potentially introduce a high bias?

(Comment No. 0040.2) In Section 8.5.3.1, I would like to suggest changing the text from "If no water was collected before the CPM filter, then . . ." to "If less than 25 ml of water was collected before the CPM filter or if the emission source is known to contain less than 25 ppmv of SO₂, then . . ."

(Comment No. 0050.1) Section 8.5.3.1 states that, "if no water was collected before the CPM filter, then you may skip the remaining purge steps..." The Alliance recommends the following language: "If no measurable liquid is in the bottom of the impinger before the CPM filter then you may skip the remaining purge steps..."

(Comment No. 0044.1) The proposed method specifies that, if no water was collected before the CPM filter, then you may skip the remaining purge steps and proceed with sample recovery. At times, there are very small quantities of water before the CPM filter. BTEC

proposes that the method quantify a water volume threshold amount at which the remaining purge steps can be skipped. BTEC recommends a threshold amount of 20 to 30 ml.

(Comment No. 0059.1) On a recent test program using the proposed Method 202 revisions, and as suggested in the proposed method, the pH of the condensate catch is not measured. Since the sample gas never bubbled through the condensate and the pH is not measured, how do we confirm that a nitrogen purge is necessary? Should the pH of the condensate be measured?

Response: We disagree with the commenters that Method 202 should specify a de minimis volume of water, below which the nitrogen purge is not required. We also do not believe that requiring measurement of the pH of collected condensate is necessary to confirm that the nitrogen purge is necessary. The nitrogen purge is required to reduce the bias caused by artifact formation if any water is visible in the bottom of a knock-out impinger.

Comment: (Comment No. 0068) Section 8.5.3.2 requires the use of degassed water. Why degassed water? Why not recovery water since we are going to purge it any way?

(Comment No. 0040.2) In Section 8.5.3.2, I would like to propose deleting the note concerning nitrogen purging the water that is added prior to nitrogen purging the impinger solutions. This step would have to be performed daily on-site since oxygen would re-dissolve into the purged water. Any dissolved oxygen would be removed quickly during the first moments of the sample train nitrogen purging.

(Comment No. 0054.1) Are there good data to support that degassing Type 1 water (or equivalent) is necessary to produce reliable results? This seems like it could be more effort for no benefit.

Response: We disagree with the commenters that water used to supplement the moisture catch does not need to be degassed prior to the nitrogen purge. Small amounts of oxygen can play an important role in artifact formation at the detection limit of the method. Method 202 requires the use of degassed water because dissolved oxygen in water that has not been degassed can contribute to artifact formation through the oxidation of sulfur compounds in the impinger water. Therefore, for consistency, all stack testers must degas any water added to the impingers prior to the nitrogen purge. With regard to the commenter's concern that the degassing procedure would have to be conducted daily, degassed water can be stored under a nitrogen

blanket to prevent oxygen from dissolving into the water after it has been degassed. With regard to the commenter's request to use recovery water, we have revised Section 8.5.3.2 of the final method to allow combination of the moisture collected in the condenser and first (short stem) impinger with the moisture collected in second (modified Greenburg Smith) impinger. Subsequent purge of the condenser, second impinger, and CPM filter is a simplifying step that meets the goals of the nitrogen purge to reduce SO₂ artifacts.

Comment: (Comment No. 0059.1) Prior to the nitrogen purge, degassed distilled water is added to the impinger. Following the purge, the impingers are rinsed twice with distilled water. Should the rinse water be degassed? The method is not clear.

Response: There is no need to degas the water used to rinse the impinger after the sampled moisture has been poured into the recovery container. The purpose of degassing the water used for the nitrogen purge is to remove dissolved oxygen that could react with any SO₂ present in the water collected in the impingers. Degassed water is not needed to recover residual water-soluble CPM from the impinger after the purged sample has been poured into the recover container because atmospheric oxygen has already been added to the post-purge moisture catch during the transfer process.

Comment: (Comment No. 0050.1) Section 8.5.3.3 requires a post-test nitrogen purge. The Alliance recommends that the purge time be set to a minimum of one hour to allow for additional time for purging if necessary to fully strip the SO_x.

Response: Method 202 requires that the nitrogen purge be conducted for one hour based upon the work reported by Dewees et al., (Dewees, and Steinsberger, K., 1990). That study found that the SO₂ concentrations in Method 202 samples collected from various stationary sources and exposed to various SO₂ concentrations were negligible after a 60-minute purge at a gas flow rate 20 liters per minute. Based on this work, we believe that one hour is a sufficient time period to minimize the potential formation of artifact mass. The method, as written, does not prevent stack testers from conducting the purge for longer time periods, if desired. However, we acknowledge that the proposed language may not be clear; therefore, we are revising Section 8.5.3.3 to clarify that the nitrogen purge must be conducted for at least one hour.

Comment: (Comment No. 0054.1) Section 8.5.3.3 mentions using the purged inline filter. I have had a hard time locating the raw data that supports the need of the filter. In the *Evaluation and Improvement of Condensable Particulate Matter Measurement* report it is mentioned that a fine black residue was present from unfiltered gas, but I can not find the data to see if the cause of this was investigated or if this was repeatable. It could be suggested to use best practices when setting up the purge system by first cracking the cylinder before attaching the regulator and then purging the regulator prior to connecting to a sample. I did not locate the detailed description of the setup of the experiment that this conclusion was based on.

Response: Testing conducted by Environment Canada related to the nitrogen purge gas particulate contamination was summarized in the EPA laboratory assessment of the dry impinger modification to Method 202. The Environment Canada work included an assessment of the fine particulate residue captured from the compressed nitrogen gas used in the purge of condensable particulate samples. Inorganic analysis of this residue identified iron as the primary metal constituent. Other stakeholders including PCA and API found compressed gas nitrogen cylinders that were contaminated. Because contamination has been observed by several of the stakeholders and the fine particulate caused by iron in the nitrogen cylinder will be a potential contaminant each time the same cylinder is used, EPA has determined that an in-line particulate filter must be used to remove the potential fine filterable particulate bias from compressed gas cylinders. .

Comment: (Comment No. 0069) In Section 8.5.3.3, the method should emphasize that the temperature of the CPM filter must be no greater than 85°F during nitrogen purge. Suggestion: periodically checking the temperature at the exit of the CPM filter, the rotameter and delta H.

Response: We do not believe that the additional monitoring requirements suggested by the commenter are needed. Section 8.5.3.3 of the final method states the following with regard to the CPM filter temperature:

“During either purge procedure, continue operation of the condenser recirculation pump, and heat or cool the water surrounding the first two impingers to maintain the gas temperature measured at the exit of the CPM filter greater than 20°C (65°F), but less than or equal to 30°C (85°F).”

We believe that this language is sufficiently explicit to ensure that the CPM filter is maintained during the nitrogen purge. Therefore, we are not revising the CPM filter temperature specifications in Section 8.5.3.3.

Comment: (Comment No. 0064.1) During actual test runs in the field with relatively high ambient temperature, it was noted that the ice bath was reduced during the lengthy nitrogen purge and some moisture could be potentially lost. It is recommended that the ice bath temperature be monitored during the nitrogen purge. It has been observed that when the proposed Method 202 is performed in the field there can be water droplet carryover out of the chilled water impinger (#3). Quantitative recovery of this carryover is difficult and it has the potential to saturate the silica gel. It is recommended that a dry, blank impinger be placed between the chilled water impinger and the final silica gel impinger to prevent water carryover from saturating the silica gel.

Response: We disagree with the commenter that the method should require that the temperature of the ice bath used to cool the moisture traps should be monitored during the nitrogen purge. Section 8.4.4 of the final method specifies the gas temperature at the exit of the moisture traps must be maintained below 20°C (68°F). Section 8.5.3.3 further specifies that the gas temperature measured at the exit of the silica gel impinger must maintained below 20°C (68°F) to prevent removal of moisture during the purge. We believe that the requirements to maintain the temperature of the exit gas below 20°C (68°F) achieves the same objective as the commenter's suggestion. Therefore, we are not adding a requirement to the final method to require monitoring of the moisture trap ice bath temperature during the nitrogen purge.

Also, because the determination of the amount of moisture in the sample gas is made either by volumetric or gravimetric analysis, we disagree with the commenter that a quantitative recovery of moisture carried from the chilled water impinger to the silica gel impinger is difficult.

Comment: (Comment No. 0103) Section 8.5.3 requires the silica-gel impinger outlet temperature to be monitored and maintained at less than 68°F during the post-test purge to prevent loss of moisture. If the impingers are weighed for moisture gain prior to the purge, do these conditions still have to be maintained during the purge?

Response: If the volume of liquid collected in the moisture traps is determined prior to conducting the nitrogen purge, it is not necessary to maintain the temperature at the exit of the silica-gel impinger below 20°C (68°F). We have revised Section 8.5.3 of the final method to clarify the requirements regarding maintenance of the silica gel impinger exit temperature.

Comment: (Comment No. 0041.1) If the ambient temperature in the sample recovery area is less than 85 °C, there is no reason to record the CPM filter temperature during the nitrogen purge for quality checks, as some agencies might require.

Response: We acknowledge that the commenter’s suggestion makes intuitive sense. However, to maintain consistency between testing contractors, the final method requires that the temperature of the CPM filter be maintained below 30°C (85°F).

M202 8.5.4 SAMPLE RECOVERY

Comment: (Comment No. 0080.1) Consistent terminology is strongly recommended. If the intent is that a “rinse blank” and a “field rinse blank” serve the same purpose, they should share the same descriptive name as well.

(Comment No. 0121) Are the “Rinse Blanks” discussed in Sections 8.5.4.7 through 8.5.4.9 (labeled Containers 6, 7, and 8) different from the “Field Rinse Blanks” discussed in Sections 11.2.5 through 11.2.7 (also called Containers 6, 7, and 8)? Are “Rinse Blanks” the same thing as “Reagent Blanks?”

Response: We agree with the commenter that the use of consistent terminology is important to assessing accurately the requirements and procedures for any test method, and we acknowledge that the language used to designate the procedures for field rinse blanks was not always consistent. Consequently, we have revised the final method to use consistent terminology for laboratory reagent, field reagent, field train recovery, and field train proof blanks.

Comment: (Comment No. 0080.1) We recommend that the proposed Method 202 be consistent with all other EPA methods (e.g., Method 5) with regard to types of blanks, number of blanks and use of blanks in adjusting resultant data. Whatever blanks and blank handling

procedures remain after the proposed Method 202 are finalized, every other EPA method should be revised to include the same language and requirements.

Response: The solvent blank procedures in the proposed Method 202 were made as consistent as possible with EPA Method 5 and the current version of Method 202. Method 5 has no requirement for field train recovery blanks. We disagree with the commenter that the blank requirements and procedures for all methods should be the same. We believe that method-specific blank procedures are appropriate for different methods. The final Method 202 has the potential to measure very low levels of CPM. Accordingly, the blank residue and handling requirements in Method 202 are more stringent than other PM test methods. We have revised the procedure for blank correction in the proposed Method 201A/202 to use the field train blank value not to exceed 2.0 mg. However, we are not further revising the blank requirements and handling procedures of the final method.

Comment: (Comment No. 0080.1) We suggest that since the field train blank is the only blank that is allowed to be subtracted from the sample results under the proposed Method 202, the other blanks (except the reagent blank) do not serve any purpose and should not be collected.

(Comment No. 0121) Is the water, acetone, and DCM blank mass subtracted from anything?

Response: We disagree with the commenter that the blanks required by Method 202 do not serve any purpose. The laboratory and field reagent blanks and the field train recovery and field train proof blanks provide valuable quality indicators for assessing the diligence of the test contractor to minimize contamination and provide thorough sample recovery. The laboratory and field reagent blanks required by Method 202 are used to target corrective actions, if necessary, to reduce the residual mass contribution from using reagents that do not meet the residual mass specifications or from field contamination of reagents in subsequent tests. The field train proof blank (added in response to comments) quantifies the residual mass contribution from the clean glassware. The field train recovery blank characterizes between run residual mass contribution from external contamination or from incomplete recovery of previous sample trains and is the only blank mass that is used to correct the CPM measurement. Without these blanks, stack testers would not be able to track the source of high residual mass values.

Comment: (Comment No. 0080.1) The amount of particulate that would be determined from each type of blank is already reflected in the amount of particulate found in the train blank. However, if EPA wishes to retain the other blanks, then we have the following specific comments about the blanks as specified in the draft Method 202. In general, the language used to describe blanks in the draft Method 202 is confusing. The method identifies: (1) Rinse blanks for acetone, water, and methylene chloride to be collected from the wash bottles in the field as specified in Section 8.5.4. This section requires one rinse blank for each liquid for each run, or a total of nine rinse blanks for a three-run testing program. (2) Reagent blanks for each reagent are specified in Section 9.9. This Section requires that one blank of each reagent be collected and analyzed “before you begin testing.” (3) Field reagent blanks are specified in Section 9.9. This section seems to imply that a blank must be collected from each primary (as opposed to wash) container. The intent appears to be to determine whether the reagent(s) become contaminated between the laboratory and the field. (4) Field blank trains are specified in Section 9.10. Only mass found in the field blank trains less than or equal to 2.0 mg may be subtracted from test results. (5) Field rinse blanks are specified in Section 11.2.

Response: The blanks required by Method 202 are used to target corrective actions by the testing contractor, if necessary, in subsequent tests. There are four types of blanks identified in Method 202: 1) reagent blanks straight from the vendor supply, 2) field reagent blanks taken from the wash bottles or other equipment used to recover samples from the collection train, 3) field train recovery blanks that are rinses of sampling trains after samples have been recovered during field testing, and 4) field train proof blank collected from a clean, fully assembled train prior to the first sample collection as an option to baking glassware. With regard to the reagent blanks for water, acetone, and extraction solvent (comment no. 1), we have revised Sections 8.5.4.7, 8.5.4.8, and 8.5.4.9 of the method to clarify that one set of field reagent blanks per lot(s) of solvent used per test is required rather than the commenter’s assumption of one blank per train run. EPA’s experience shows that well-managed field tests can demonstrate the following wash bottle blanks: for acetone 0.6 to 0.7 mg/100 ml, for methylene chloride or hexane, 0.05 to 0.1 mg/100 ml, and for water 0.6 mg/ 100 ml. With regard to comment numbers 3 and 5, the field water, acetone, and hexane blanks required in Section 9.9 are the same blanks analyzed according to the procedures specified in Sections 11.2.5, 11.2.6, and 11.2.7.

With regard to the requirement for reagent blanks in Section 9.8 (note: the commenter referred to Section 9.9 but we assume that the reference was intended to be to Section 9.8), the commenter's description is accurate (i.e., one reagent blank must be collected and analyzed before testing begins to verify low blank concentrations). Also, the commenter's description of the field train blanks in comment no. 4 is correct (i.e., the method allows for a maximum of 2 mg of residual mass to be subtracted from the test results). We have revised Sections 9.7 and 9.8 of the method to provide clarification and flexibility with regard to reagent blanks. The use of laboratory reagent blanks and field reagent blanks are strongly recommended in the method as a means to identify the cause for any high blank values identified in the mandatory field train blanks.

Comment: (Comment No. 0104) If a site contains four flues in a common stack, is each flue required to be treated as a new source category? If the boilers operate with the same coal, can one field blank be used for all four flues? Also, for a project that will be running nine tests on four flues, is it required to have a rinse blank for every test? The amount being brought back will be substantial. The rinse bottles will be the same for the entire project, and the reagents will be coming from the same lots.

Response: Our intent in OTM 28 and the proposed revision to Method 202 was that a single set of field reagent and field train recovery blanks (and a field train proof blank, if applicable) be collected for a sampling campaign at emissions sources that were essentially identical in design and operational characteristics. The sampling equipment preparation procedures specified in Section 8.4 of the method are not required to be repeated for each stack/source of the same source type at the same facility. If reagents from multiple lots are used, the residual mass for each lot should be determined to ensure that the supplier is providing a consistent product.

With regard the commenter's questions regarding how a specific flue configuration at a site should be tested, the commenter did not provide sufficient information regarding the processes at the site for us to make a determination.

Comment: (Comment No. 0038.1) The language describing which components to rinse for recovery of the aqueous sample does not encompass all possible permutations for arrangement of the sampling train components, in particular the case when an out-of-stack heated filter is used for the front half of the sampling train. The statement “Rinse the probe extension, condenser, each impinger, and the connecting glassware, and the front half of the CPM filter housing twice with water” should be replaced with the following language, “Rinse all components between the CPM filter and the filter in the front half of the sampling train twice with water.” This will accommodate all possible sampling train combinations while being clear on which components to include in the rinse and recovery procedures.

Response: We agree with the commenter that the language in Method 202 is unclear with regard to the rinse and recovery procedures for sampling train components. The probe extension begins with the back-half of the filterable PM filter holder. To improve the clarity of the rinse procedure, we have revised the Section 8.5.4.2 to specify explicitly the sampling train components that must be recovered.

Comment: (Comment No. 0040.2) In Section 8.5.4.2 (the impinger rinse procedure), clarify whether to also rinse the replaced long stem nitrogen purging impinger insert. It should be cleaned according to Section 8.4. Since it does not participate during the entire sampling duration, its rinsing as a sample fraction may be unnecessary and may also become a source of addition background mass.

Response: Because the long-stem impinger insert is not used to collect samples and only a small portion of the impinger tip will typically contact the sample during the nitrogen purge, we do not believe that it is necessary to rinse this additional glassware as part of sample recovery.

Comment: (Comment No. 0040.2) In Section 8.5.4.3, I would suggest replacing the text “glass container” with “pre-rinsed glass container with a PTFE lined lid.” This should reduce the contamination from bottle production and minimize residues extracted from lids made of other plastics that dissolve with methylene chloride exposure. I would also like to suggest adding the text “For methylene chloride, use a PTFE wash bottle” to Section 8.5.4.3.

(Comment No. 0050.1) Section 8.5.4.3 reads: "Follow the water rinses of the probe extension, condenser, each impinger and all of the connecting glassware and the front half of the CPM filter with an acetone rinse. Then repeat the entire procedure with two rinses of MeCl₂, and save both solvents in a separate glass container identified as CPM Container #2. Mark the liquid level on the jar." The Alliance recommends the following language: "Follow the water rinses of the probe extension, condenser, each impinger and all of the connecting glassware and the front half of the CPM filter with an acetone rinse. Save the solvent in a glass container identified as CPM Acetone Container #2. Then repeat the entire procedure with two rinses of methylene chloride, and save the solvent in a separate glass container identified as CPM Methylene Chloride Container #2. Mark the liquid levels on both the jars."

Response: Based upon other comments we received regarding the container specifications, we have revised the container requirements in Section 6.2 (proposed Section 6.3) and Section 8.5.4 to provide a performance-based option. Rather than specify that the containers must be constructed of a specific material (e.g., glass), the revised method allows you to use whichever leak-proof container and lid that contribute less than 0.05 mg of residual mass to the CPM measurements. Notwithstanding the changes we have made to the container requirements, we agree with the commenter’s suggested edits. With regard to the commenter’s suggestion to specify the solvent wash bottle, the testing contractor has the option of using his preferred non-contaminating wash bottles.

Comment: (Comment No. 0040.1) The use of an additional field acetone rinse during sample cleanup is not essential and under non-ideal conditions may promote additional contamination. The methylene chloride promulgated EPA Method 202 cleanup can be retained.

Response: We disagree with the commenter that the field acetone rinse is not needed. The acetone rinse was added to the proposed method to completely remove water and to enhance recovery of organic CPM in the final solvent rinse. Furthermore, EPA laboratory experiments (Evaluation and Improvement of Condensable Particulate Matter Measurement (Docket no. EPA-HQ-OAR-2008-0348-0028)) showed that using the first rinse as the initial extraction solvent created emulsions that interfered with the organic-to-inorganic separation.

M202 8.5.4.7 CPM CONTAINER #6, ACETONE RINSE BLANK

Comment: (Comment No. 0038.1) Section 8.5.4.7 specifies that 150 ml of acetone be used as an Acetone Rinse Blank and analyzed per instructions in Section 11.2.5. Further, instructions in Section 11.2.5 direct that only 100 ml of acetone be reduced for determination of the dry residue mass. We feel that a 100 ml volume specified in Section 11.2.5 for reduction is too small (100 ml of acetone weighs 79 grams at 20°C). To meet the 1 ppmw requirement for maximum residue by weight the residue must be 0.000079 grams or less. The gravimetric LOD for the 50 ml aluminum weighing pans is in the range of 0.00009 to 0.00016 grams and the PLQ is in the range of 0.00030 to 0.00053 grams. Therefore, in order to have a measured mass with minimal uncertainty in the measured value, the volume of acetone reduced for the percent weight residue should be no less than 450 ml, and the volume collected as the Acetone Rinse Blank should be no less than 500 ml.

Response: We disagree with the commenter's suggestion to increase the volume of the field blanks. However, in light of the above comment, and other similar comments received regarding the reagent blank volume, we have increased the reagent blank volume in the final method to 150 ml. Evaporating 150 ml of reagent and weighing the residual mass will provide testing contractors with the ability to verify that the residue in the solvent was less than the guaranteed residue level of 1 ppmw.

M202 8.5.4.8 CPM CONTAINER #7, WATER RINSE BLANK

Comment: (Comment No. 0038.1) Section 8.5.4.8 specifies that 150 ml of water be used as a Water Rinse Blank and analyzed per instructions in Section 11.2.6. Further, instructions in Section 11.2.6 direct that only 100 ml of water be reduced for determination of the dry residue mass. We feel that a 100 ml volume specified in Section 11.2.6 for reduction is too small (100 ml of water weighs 99.82 grams at 20°C). To meet the 1 ppmw requirement for maximum residue by weight, the residue must be 0.000010 grams or less. The gravimetric LOD for the 50 ml aluminum weighing pans is in the range of 0.00009 to 0.00016 grams, and the PLQ is in the range of 0.00030 to 0.00053 grams. Therefore, in order to have a measured mass with minimal uncertainty in the measured value, the volume of water reduced for the percent weight residue should be no less than 450 ml, and the volume collected as the Water Rinse Blank should be no less than 500 ml.

Response: We disagree with the commenter's suggestion to increase the volume of the field reagent blanks to 500 ml. However, in light of the above comment, and other similar comments received regarding the reagent blank volume, we have increased the reagent blank volume in the final method to 150 ml. This volume approximates the volume of water used during field train recovery. Evaporating 150 ml of reagent and weighing the residual mass will provide testing contractors with the ability to verify that the residue in the solvent was less than the guaranteed residue level of 1 ppmw.

M202 8.5.4.9 CPM CONTAINER #8, METHYLENE CHLORIDE RINSE BLANK

Comment: (Comment No. 0038.1) Section 8.5.4.9 specifies that 150 ml of methylene chloride be used as a Methylene Chloride Rinse Blank and analyzed per instructions in Section 11.2.7. Further, instructions in Section 11.2.7 direct that only 100 ml of methylene chloride be reduced for determination of the dry residue mass. We feel that a 100 ml volume specified in Section 11.2.7 for reduction is too small (100 ml of methylene chloride weighs 132.6 grams at 20°C). To meet the 1 ppmw requirement for maximum residue by weight, the residue must be 0.000133 grams or less. The gravimetric LOD for the 50 ml aluminum weighing pans is in the range of 0.00009 to 0.00016 grams and PLQ is in the range of 0.00030 to 0.00053 grams. Therefore, in order to have a measured mass with minimal uncertainty in the measured value, the volume of methylene chloride reduced for the percent weight residue should be no less than 450

ml, and the volume collected as the Methylene Chloride Rinse Blank should be no less than 500 ml.

Response: We disagree with the commenter's suggestion to increase the volume of the field blanks. However, in light of the above comment, and other similar comments received regarding the reagent blank volume, we have increased the reagent blank volume in the final method to 150 ml. This volume approximates the volume of solvent used during field train recovery. Evaporating 150 ml of reagent and weighing the residual mass will provide testing contractors with the ability to verify that the residue in the solvent was less than the guaranteed residue level of 1 ppmw.

M202 9.0 QUALITY CONTROL

Comment: (Comment No. 0058.1) Section 9.0 of the proposed method includes a provision for audit samples. This is especially desirable since proposed changes to the method will increase the complexity of both the sampling and analysis activities of the method.

Response: We acknowledge the commenter's support for the use of audit samples. However, the audit procedures specified in Section 9.11 of the proposed method have been removed from the final method in light of the proposed changes to the General Provisions of Parts 51, 60, 61, and 63 requiring the use of commercially-available audit samples.

M202 9.4 HEALTH AND SAFETY PLAN

Comment: (Comment No. 0054.1) The requirement to prepare a Health and Safety Plan seems out of place in a Quality Control section of a stationary source test method and should be regulated elsewhere.

Response: We agree with the commenter that the health and safety plan requirements should not be placed under the quality control section of the method. Consequently, we have replaced the language in Section 5.0 (Safety) with the language from Section 9.4 (Health and Safety Plan) and made this a non-mandatory recommendation.

M202 9.5 CALIBRATION CHECKS

Comment: (Comment No. 0048.1) Are Sections 9.5 and 9.7 redundant, or do they refer to a field balance (Section 9.5) and a laboratory balance (Section 9.7)? It is our recommendation that these sections either be combined, or that the guidance specify which balance(s) these procedures apply.

Response: Section 9.4 of the final method (Section 9.5 in the proposal) pertains to calibration checks performed on the field analytical balance and Section 9.6 of the final method (Section 9.7 of the proposal) pertains to calibration checks performed on the analytical balance used in the laboratory. We acknowledge that the section titles and language used in the sections is unclear with regard to the purpose of the calibrations. Therefore, we have revised these sections to improve clarity of the method requirements.

M202 9.8 REAGENT BLANKS

Comment: (Comment No. 0080.1) We recommend that the language regarding reagent blank specifications in Section 9.8 be modified such that a blank is collected and analyzed for each lot/batch of reagent(s) received by the testing firm. This would establish a baseline for cleanliness of each lot of reagents, which is what we believe the intent of this requirement to be. We submit that all EPA methods should be updated to require that the testing contractor maintain records of each batch of purchased reagents and the blank residue value obtained from each purchased batch, regardless of the lot number. This would ensure that the “as received” material met the requirements. The requirements should be specified as a not to exceed amount of residue, not as a particular brand or type of reagent (e.g., HPLC grade, Type I).

Response: We agree with the commenter that the language in Section 9.8, as proposed, is unclear with regard to reagent testing. In the proposal, we intended to require testing for each lot of reagents received. Therefore, we revised Section 9.7 of the final method (Section 9.8 of the proposed method) to explicitly state that at least one reagent blank must be analyzed for each lot of reagents that will be used for sample recovery and analysis. With regard to the commenter’s

suggestion to extend this requirement to all EPA reference test methods, we agree that there is some merit to the commenter's suggestion; however, it is not appropriate to address that comment in this action because the proposal was limited to the revisions to EPA Methods 201A and 202.

M202 9.9 FIELD REAGENT BLANKS

Comment: (Comment No. 0080.1) The method appears to use the terms “field reagent blank” and “rinse field blank” interchangeably, at least for reporting. Section 9.9 specifies that three field reagent blanks and nine field rinse blanks are required. At the end of sections 11.2.5, 11.2.6, and 11.2.7, it is specified that the field rinse blank data be “reported to the nearest 0.1 mg on Figure 3.” Figure 3 indicates that the results of analysis of one field blank train and one each of the field reagent blanks are to be reported - no mention is made of field rinse blanks. The number of blanks required to be collected, analyzed, and reported must be consistent throughout the method. The field reagent blanks (or field rinse blanks) should be correlated to the specific lot/batch of reagent so that the two reagent blanks can be evaluated to determine if any degradation occurs through handling in the field.

We support the use of “reagent blanks” to determine the acceptability of the reagents prior to their use in the field and encourage the EPA to require the reporting of this data in each test report. We also support the collection and analysis of “field reagent blanks” collected near the end of the testing to demonstrate that the reagents brought into the field maintained their integrity throughout the “compliance testing.” Finally, we support the collection and analysis of “rinse field blanks” during the recovery of each sample train as a way to demonstrate that the reagents actually being used during the recovery have also maintained their integrity. However we would encourage EPA to use these rinse blanks to adjust the sample results and also to adjust the results found in the field train blank as is currently done in Method 5 for example. In the latter case, if the rinse blanks are subtracted from the field train blank, then the field train blank result should be close to zero.

Response: We acknowledge the commenter's support for the use of blanks in the proposed Method 202. We also agree with the commenter that the terminology used for the various blanks required in the method is inconsistent. Therefore, we have revised the method,

where appropriate, to use the terms “laboratory reagent blank,” “field reagent blank,” and “field train blank.” In response to other comments on the proposal, we have also added a proof blank of the sampling train prior to testing to confirm the cleanliness of glassware as an option to the baking requirement. However, we disagree with the commenter that reagent blanks should be collected near the end of testing to demonstrate that the reagents brought into the field maintained their integrity throughout the test. We have revised the method to make the use of laboratory reagent blanks and field reagent blanks a recommended option to provide flexibility to testers. Although we acknowledge that confirming the integrity of reagents before and over the duration of a test could be useful information to the testing contractor, we do not believe that requiring such blanks in Method 202 is necessary because the required field train recovery blank provides a measure of the combined blank contribution to the samples.

We disagree with the commenter that Section 9.9 of the proposed method requires three field reagent blanks and nine field rinse blanks. Section 9.8 of the final method (proposed Section 9.9) recommends that testing contractors must run at least one field reagent blank of water, acetone, and hexane per test series on each source type at a facility or site. We disagree with the commenter’s recommendation to use the reagent blanks to adjust the sample results and to adjust the results found in the field train recovery blank. The purpose of the field reagent blanks is to provide testing contractors with information that they can use to target corrective actions, if necessary, to achieve the residual mass requirements of Method 202. The method allows for up to 2 mg of the residual mass in the field train recovery blank to be subtracted from the CPM measurements. Because the field train recovery blank includes residual mass contributions from the reagents, the sampling train glassware and potential field contamination, we believe that the commenter’s suggestion to correct the field train recovery blank using the field reagent blank results is unnecessary.

M202 9.10 FIELD TRAIN BLANK

Comment: (Comment No. 0050.1) Section 9.10 allows for a test field blank credit of the lesser of 2 mg or the actual blank value. For example, if the blank is 5 mg, the credit allowed is only 2 mg. We understand that the value of 2 mg was taken from laboratory studies using pure solvents and good laboratory techniques. The Alliance has reviewed one of the EPA docket

reports (EPA Contract No. EP-D-07-097) which reported on several studies dealing with determining the field train blanks. Specifically, Table 6-5 through Table 6.8 in the noted docket report provided raw data from a series of testing to understand the level of the blanks. This report shows 10 data points, ranging from 10.7 to 0.46 mg. The average of this data is 3.5 mg. The Alliance recommends that rather than using the data average, statistical methods be used to establish a reasonable statistical upper limit for the field blank. As such, the Alliance recommends that a value of 5 mg be allowed as the field blank.

(Comment No. 0086.1) Several organizations have submitted comments concerning the reasonableness of this 2 mg limit. Comments submitted by Resolution Analytics (Docket Document 0033), TRC Environmental (Docket Document 0041.1), and GE Energy (Docket Document 0048.1) indicated that the presently specified blank correction limit of 2 mg is at least 2 to 3 mg below the levels experienced by a number of testing firms. API concurs with the recommendation to increase the allowable blank correction value to a maximum value of 5 mg.

Response: The field train recovery blank is unique to air emissions source test methods. Other test methods allow only for subtraction of the contaminant analyte from the field reagent blank. We established the 2.0 mg blank correction based upon laboratory tests (*Evaluation and Improvement of Condensable Particulate Matter Measurement* (Docket No. EPA-HQ-OAR-2008-0348-0028)) that indicated SO₂ artifact formation during a typical compliance test at a well-controlled source would be 2.0 mg. We were able to demonstrate much lower laboratory blanks using the method procedures, as proposed, during these tests.

We have evaluated data from stakeholder field tests and EPA-sponsored field evaluations of the revised method (*Field Evaluation of an Improved Method for Sampling and Analysis of Filterable and Condensable Particulate Matter*; Docket no. EPA-HQ-OAR-2008-0348) and found that final field blanks taken before sampling (often called proof blanks) meet the method requirements. Other field train blank data taken between sampling runs demonstrates variable blank recovery between 1 mg and as much as 10 mg. We believe that the higher field train blank results mentioned by the commenter may occur because the sample has not been completely recovered between sampling runs. Contamination of glassware or recovery solvents, or other sampling and recovery issues, are likely the cause of the higher blank values cited by the commenter.

We have found that there is inherent blank residual in Method 202 originating primarily from the inorganic fraction of the samples. We have determined that the blank results achieved by proper application of Method 202 in both laboratory and field tests is approximately 2 mg of blank residue. Therefore, we do not believe the data cited by the commenter constitute grounds for increasing the blank correction value. We also expect that testers will be able to consistently achieve field blank levels of approximately 2 mg or less with the assistance of the various blanks (reagent, recovery, and proof) and further experience with the method.

Comment: (Comment No. 0044.1) The proposed method allows for a test blank credit of the lesser of 2.0 mg or the actual blank value. BTEC proposes that the actual blank value be employed in all cases. It is possible to get elevated reagent and train blanks even though the glassware for these varying results had been cleaned using the same procedures and personnel, with the same reagents and analytical laboratory.

(Comment No. 0048.1) Rather than specifying a limit on the residue, it is our opinion that the EPA should consider allowing a percentage of the amount of CPM found in the source (e.g., 10 percent or 1 percent).

Response: Because Method 202 has the potential to measure very low levels of CPM, the sampling train components and reagents used to recovery samples must contribute very little residual mass to the CPM measurements. Ideally, the field recovery blank includes only the laboratory reagent blank plus any residue contributed from clean glassware. Elevated residue levels are indicators of contamination of: (1) reagents, glassware, or other components used in sampling, or (2) indicators of incomplete sample recovery that would appear as sample mass in subsequent tests. We have limited the allowable blank subtraction to account for the residue associated with the best available field train recovery blanks achieved by test crews. We believe that requiring a consistent blank level for all tests is an important factor in generating consistent results across different testing contractors and emission sources. Consequently, we disagree with the commenter's suggestion to allow for a site-specific blank correction factor.

Comment: (Comment No. 0057) Section 9.10 says that "You must recover a minimum of one field train blank for each set of compliance tests at the facility." Does this mean one field blank per source, per facility, per week? If so, can you please revise the text to make this clear?

(Comment No. 0059.1) If multiple tests are performed on a single source (i.e., three tests at various conditions), is a single blank train for the test program sufficient (assuming that all tests are performed during the same mobilization)?

(Comment No. 0080.1) Section 9.10 requires that a field train blank be collected and analyzed just as a regular sample for each “set of compliance tests” at the facility. This includes the requirement to purge the sample with nitrogen for 1 hour. The term “set of compliance tests” should be defined. We suggest that a field train blank be collected for every set of three runs that constitutes one compliance test.

(Comment No. 0069) Specify that the field train blank is to be recovered from a sampling train used to collect a sample to demonstrate that clean up was adequate.

Response: We acknowledge that the language in Section 9.10 regarding field train recovery blanks is unclear. Our intent in the proposed method was to require that a field train recovery blank be conducted on each collection of cleaned sampling train components that are used to measure CPM emissions from a source type at a facility. This requirement correlates to the requirement to use clean glassware for each type of source tested at a facility.

The method requires that testing contractors recover at least one field train recovery blank per field test and respective condition. That means one field train recovery blank must be recovered at each stationary source emission point that is tested for compliance (a compliance test equals a minimum of three test runs plus one field train blank). Therefore, we have revised the text to Section 9.10 of the final method to specify when the field train recovery blanks must be recovered.

M202 9.11 AUDIT PROCEDURE

Comment: (Comment No. 0037) Why does the proposed Method 202 use the EPA Audit Program language in Sections 9.11, 9.12, and 9.13? This program is scheduled to end September 30, 2009.

(Comment No. 0039.1) EPA is privatizing the current audit program and audits are not expected to be available from EPA after September 30, 2009. This section should recognize this fact and reference audit purchases from private entities, as further specified by EPA.

(Comment No. 0082.1) The language in the proposed M202 sections 9.11 through 9.13 lists the audit requirements. EPA has proposed to privatize the current audit program (see 74 FR 28451). This fact should be reconciled in the proposed method to agree with the privatization proposal.

Response: We recognize that the EPA-funded audit program will not be available in the near future. On June 16, 2009 (74 FR 28451), we proposed amendments to the General Provisions of Parts 51, 60, 61, and 63 to allow accredited providers to supply stationary source audit samples and to require sources to obtain and use these samples from the accredited providers instead of from EPA. The proposal revises EPA test methods 5I, 6, 6A–C, 7, 7A–D, 8, 15A, 16A, 18, 23, 25, 25C, 25D, 26, 26A, 104, 106, 108, 108A–C, 204A–F, 306, 306A, and 308 to delete any language pertaining to audit samples. The proposal also incorporated by reference Volume 3, *General Requirements for Environmental Proficiency Test Providers* adopted December 22, 2007, as an example of an acceptable accredited proficiency test sample provider (APTSP) technical criteria document. This document outlines the criteria an accredited provider program must meet for the samples to be acceptable.

To maintain consistency between EPA test methods, we have removed Sections 9.11, 9.12 and 9.13 from the final method.

M202 10.1 THERMOCOUPLE CALIBRATION

Comment: (Comment No. 0048.1) We recommend that the EPA change the reference to Method 2, Section 10.1.4.1.2 to Section 10.3.1. We also recommend that the EPA allow the use of Alternate Method 11 for the calibration of thermocouples. As explained in the alternate method, a thermocouple that works at any temperature is expected to work at any other temperature, so all that is required is to calibrate at a single temperature (e.g., ambient, against an NIST traceable thermometer). The user then must make sure the polarity is correct by holding the thermocouple and ensuring that it records a positive increase in temperature.

Response: We agree with the commenter’s suggestion. Therefore, we have revised Section 10.1 of the method to change the reference from “Method 2, Section 10.1.4.1.2” to “Method 2, Section 10.3.1.” We also revised Section 10.1 to allow the use of Alternative Method 2 (Thermocouple Calibration Procedure).

M202 10.2 AMMONIUM HYDROXIDE

Comment: (Comment No. 0040.2) Section 10.2 references Section 5.5 of Method 6 of Appendix A-4 to 40 CFR Part 60. Method 6 does not have this section. I believe the reference should be to Method 6, Section 10.5.

Response: We agree with the commenter's edit. Therefore, we have revised Section 10.2 of the method to change the reference from "Method 6, Section 5.5" to "Method 6, Section 10.5."

M202 11.2 CONDENSABLE PARTICULATE MATTER ANALYSIS

Comment: (Comment No. 0059.1) We do not understand the requirement of 30°C evaporation to dryness. We would propose a standard drying temperature of 105°C before and after the neutralization step. We have seen a very acidic stack where we added a lot of NH₄OH. The resulting weight contribution from (NH₄)₂SO₄ can be more than half the gross weight before correction (to NH₃ added). Since we chose to use the aluminum dish because of its low background weight for gravimetric analysis, we have seen signs of corrosion by (NH₄)₂SO₄ on aluminum. The aluminum dish provides more surface area for evaporation at 30°C. If the drying temperature is elevated to 105°C we could use a glass vial which is more robust than aluminum.

(Comment No. 0040.1) The final blank residues would not be affected if the aqueous fractions were taken to dryness at 105°C as performed in the current promulgated EPA Method 202.

Response: We disagree with the commenters' suggestions regarding the evaporation and drying procedures. Method 202 is designed to quantify all CPM compounds that may exist in ambient air. This includes compounds that would vaporize at the higher temperatures cited by the commenters such as ammonium nitrate, and ammonium chloride. These inorganic compounds are commonly found on filters used in the EPA's ambient air speciation sampling network. To the extent that these compounds are collected and retained in the Method 202

sampling train, it is our intent to provide an analytical finish that retains these compounds. Although the method allows for evaporation of the majority of the sample volume at elevated temperature, we believe that lighter components of CPM could be evaporated from the samples if the catches are completely evaporated or dried at elevated temperatures (the last 10 ml of sample must be evaporated at ambient conditions). Therefore, we are not revising the evaporation and drying procedures in the final method.

M202 11.2.1 CONTAINER #3, CPM FILTER SAMPLE

Comment: (Comment No. 0044.1) The proposed method requires that the CPM filter be extracted, apparently due to concern regarding H₂SO₄. BTEC proposes that this extraction be optional because, for many non-combustion sources, extraction of the CPM filter is unnecessary. In utilizing gravimetric analysis, handling errors are minimized, positive CPM bias due to filter breakdown during the sonication and extraction process are minimized, and costs are reduced.

(Comment No. 0050.1) Section 11.2.1 states that the CPM filter be chemically processed, apparently due to concern for H₂SO₄. The Alliance recommends that processing of the CPM filter be an option as the processing may be unnecessary when the source does not contain H₂SO₄ or nitrates. Since for many sources, the processing of the CPM filter is unnecessary especially if sulfate and nitrates are low or do not exist, we recommend an alternative method of simply weighing the filter.

Response: We disagree with the commenters' assertion that the filter extraction should be an optional procedure in Method 202. The primary issues of mass determination of the CPM filter involve waters of hydration and the ability to achieve constant weight during mass measurements. Method 202 measures the mass of a complex mixture of compounds that comprise CPM emissions. We do not believe it is technically feasible or appropriate for the EPA to specify the types of emissions sources that would not require extraction of the CPM filter.

We believe that requiring extraction and analysis of the CPM filter promotes consistency in method application across different source categories. The CPM filter must be extracted and analyzed as specified in the method to avoid subjective decisions about the presence or absence of H₂SO₄ (acidic) CPM. Therefore, we are not revising the final method to make the filter extraction procedure optional. However, Method 201A allows testers to use gravimetric

procedures to quantify total PM emissions in cases where the in-stack filter can be maintained below 30°C (85°F) and the filters can be brought to constant weight.

Comment: (Comment No. 0054.1) With regard to Section 11.2.1.1, the amount of power varies by location in the sonication bath (or at least some styles). Are there more specific recommendations on how to operate the bath? Neither the sonication bath nor the extraction tubes are mentioned in the required equipment Section 6.3.

Response: We agree with the commenter that the method should contain specifications for sonication devices. Therefore, the list of analytical equipment in Section 6.2.2 of the final method (proposed Section 6.3.9) includes frequency and dimensional specifications for the sonication device.

Comment: (Comment No. 0040.2) Section 11.2.1.2 specifies the methylene chloride extraction procedure. The solvent quoted in parentheses should be methylene chloride, not water.

Response: We agree with the commenter's suggested edit. Accordingly, we have revised Section 11.2.1.2 to reflect the editorial correction. Also, in response to comments requesting the use of a less toxic solvent than methylene chloride, the final method replaces methylene chloride with hexane.

M202 11.2.2 CPM CONTAINER #1, AQUEOUS LIQUID IMPINGER CONTENTS

Comment: (Comment No. 0040.2) I would like to recommend that the proposed EPA Method 202 extraction procedures mentioned in Sections 11.2.2 and 11.2.2.1 and shown in Figure 6 be modified to agree with the original EPA Method 202 Section 5.3.2.1 extraction procedure. The Organic Train Rinse (container #2) will always contain small quantities of water that is residual in the impingers. In the proposed method, this residual water will not undergo the extraction process and will have to be evaporated in the Organic Fraction (significantly reducing the evaporation rate). EPA Method 315 Section 11.2.5.1 evaporates these similar fractions at an elevated temperature below the boiling point of the methylene chloride solvent (104°F).

Response: We disagree with the commenter suggestion regarding evaporation of the organic train rinse. Method 202 has the potential to measure very low levels of CPM and we believe that lighter components of CPM could be evaporated from the samples if the catches are evaporated at elevated temperatures. Therefore, to promote consistency in application of the method, the final evaporations of hexane (formerly methylene chloride) and water fractions are performed at ambient temperature.

Comment: (Comment No. 0040.2) In accordance with the original EPA Method 202 Section 5.3.2.1, I would like to recommend using Container #2 as the first extraction quantity in Section 11.2.2. In order to keep the organic and inorganic phases separate during extraction, the proposed organic rinse sequence (which includes the initial rinse of acetone) would need to be changed back to the original methylene chloride only rinses (no acetone). I believe the acetone rinse performed in EPA Method 315 Section 8.7.6.4.3 was added only to reduce the water retention on the surface of the impingers prior to the methylene chloride rinses and as an additional solvent, will not benefit EPA Method 202.

Response: We agree with the commenter that the acetone rinse was added to the proposed method to completely remove water and to enhance recovery of organic CPM in the final solvent rinse. EPA laboratory experiments (*Evaluation and Improvement of Condensable Particulate Matter Measurement* (Docket No. EPA-HQ-OAR-2008-0348-0028)) showed that using the organic train rinse as the initial extraction solvent created emulsions that interfered with the organic-to-inorganic separation. The proposed change to hexane from methylene chloride will enhance the separation of organic and inorganic phases and will address the commenter's concern.

Comment: (Comment No. 0048.1) The proposed method goes to great lengths to ensure that the glassware used in the field is cleaned and baked and handled so as to minimize any contamination. It also specifies the level of residue allowed in the reagents used, requires a nitrogen purge using a specified purity for the nitrogen, etc. Nowhere in the method does it specify what the cleaning procedure should be for the separatory funnel that is used in the analytical phase of the project. We recommend that there should be some specified procedure

provided for ensuring the cleanliness of the separatory funnel, or alternately eliminate this step completely.

Response: We consider the separatory funnel to be part of the glassware used in the method and, as such, must be cleaned as specified in Section 8.4. However, we acknowledge the language as proposed was unclear. Therefore, we have revised Sections 8.2 and 8.4 to clarify that all glassware used in the method must be cleaned prior to testing.

Comment: (Comment No. 0048.1) We question the value of “speciating” the CPM. The method requires that 90 ml of highly carcinogenic methylene chloride be used to extract what may be a very small amount of water-soluble organics from the water fraction. In addition to being carcinogenic, methylene chloride has a high environmental toxicity and is expensive. It is our recommendation that the aqueous fraction simply be evaporated without extraction and the organic fraction be evaporated. The total will still be the CPM in the source and it would reduce the amount of manipulation of the sample. Does anyone really care how much of the CPM is aqueous and how much is organic?

(Comment No. 0086.1) In most cases, there is no compelling need to determine if the fractions of the CPM that are inorganic or organic. In most categories of stationary sources, the CPM is almost entirely inorganic (e.g., fossil fuel-fired boilers and fluidized catalytic cracking units) or almost entirely organic (e.g., surface coatings). In the few cases where there is a need to determine the inorganic/organic partitioning of condensable material, a limited number of additional test runs could be conducted specifically for this purpose.

Response: We disagree with the commenters regarding the usefulness of extracting the organic fraction of CPM. Regardless of the end use of the measurement data, experimental results developed by Environment Canada (*Evaluation and Improvement of Condensable Particulate Matter Measurement* (Docket No. EPA-HQ-OAR-2008-0348-0028)) show that the organic CPM fraction must be separated from the inorganic CPM to obtain an accurate, unbiased measurement of total CPM. Environment Canada found that combining the organic and inorganic fractions into one solution followed by evaporation to dryness caused a negative bias in the recovery of organic CPM. Therefore, as specified in the final method, the organic and inorganic CPM fractions must be separated and evaporated individually. Organic extraction transfers the majority of organic compounds to a solvent that can be evaporated at a temperature

that reduces the loss of organic material classified as CPM from the Method 202 samples. The results of organic solvent evaporation at ambient temperature over 24 hours, as reported by EPA, results in acceptable recovery of organic material typically classified as CPM. Water evaporation from CPM samples at elevated temperatures limits the evaporation temperature to 100°C or less for the majority of the inorganic CPM evaporation step and to ambient temperature for the final 10 ml of sample. Constant boiling mixtures or azeotropes of the inorganic compounds/acids associated with water are never reached and, therefore, the acidic components are retained under these conditions. Therefore, the final method specifies that the samples must be extracted into organic and inorganic components and separately evaporated to dryness.

Comment: (Comment No. 0059.1) Section 11.2.2 of the proposed Method 202 requires that the inorganic fraction be extracted 3 times with 30 ml of methylene chloride each time (the total methylene chloride used is 90 ml). We suggest that the inorganic fraction be extracted only twice with 60 ml of methylene chloride each time, plus 30 ml of methylene chloride for final rinse (the total DCM used is 150 ml).

Response: We disagree with the commenter regarding the number solvent rinses specified in Section 11.2.2. We believe that three rinses with hexane are needed to achieve an acceptable recovery of organic CPM. Therefore, we are not revising Section 11.2.2 with regard to the required number of solvent rinses.

M202 11.2.2.2 ORGANIC FRACTION WEIGHT DETERMINATION

Comment: (Comment No. 0040.2) I would like to suggest the text “weighing tin or equivalent” instead of “50ml tin” in Sections 11.2.2.2, 11.2.2.5, 11.2.6, and 11.2.7.

Response: We agree with the commenter that alternatives to weighing tins should be allowed in the method. In response to previous comments received regarding this issue, we have revised Section 6.2.2(b) (proposed Section 6.3.2) to allow for the use of fluoropolymer beaker liners as an alternative to weighing tins.

Comment: (Comment No. 0040.2) Replace the term “anhydrous calcium sulfate” with “active desiccant” in Sections 11.2.2.2 and 11.2.2.5.

Response: We disagree with the commenter's suggested edit. The term "active desiccant" is too open ended and does not provide the level of performance of the desiccant. Active silica gel used in sampling trains is not adequate for the evaporation of water from CPM samples. An alternative desiccant that achieves the same or lower level of humidity for drying samples is allowable. However, there are few other desiccants that perform as well and are as inexpensive and safe to use. Therefore, EPA has not modified the method and has determined that the explicit reference to anhydrous calcium sulfate in the analytical procedures in Sections 11.2.2.2 and 11.2.2.5 reduces the potential for using incorrect types of desiccant.

Comment: (Comment No. 0040.2) The ambient evaporation procedures mentioned in the proposed EPA Method 202 Sections 11.2.2.2 and 11.2.2.3 require a larger quantity of laboratory time and labor than the original EPA Method 202 methodology. The Inorganic Fractions will need to be constantly monitored when they are in a 105°C (220°F) oven in order to ensure that the volumes do not go to dryness. Both the Inorganic and Organic Fractions will require a significant amount of fume hood and desiccator time to evaporate the final residual water at ambient temperatures. The Inorganic Fraction would be required to undergo this ambient drying step twice to obtain the final residual mass determination. I am concerned that commercial laboratories will shortcut the ambient evaporative steps when confronted with projects with large numbers of samples. My opinion is that this bottleneck in the sample analysis procedure will pressure labs to accelerate the evaporation without deviation documentation. Since the majority of volatile CPM will be extracted into the organic phase for ambient drying, I would like to suggest retaining the original EPA Method 202 inorganic fraction drying procedure that uses 105°C until dryness. The 16+ hours of elevated oven temperatures needed to reduce the aqueous volume to close to 10 ml may drive off the aqueous volatile CPM as much as oven drying to dryness. The AirNova reduced temperature glassware baking paper also provides backup data demonstrating little effect of 105°C oven drying on the background residue mass.

Response: We acknowledge that the evaporation procedures in the proposed method will require additional time and attention as compared to the requirements currently specified in Method 202. However, we disagree with the commenter's suggestion regarding evaporation of the inorganic train rinse. The final method allows elevated-temperature evaporation of water from the inorganic CPM samples and limits the evaporation temperature to 100°C or less for the

majority of the inorganic CPM evaporation process. Evaporation of constant-boiling mixtures of the inorganic compounds/acids in the sample does not occur because the soluble inorganic CPM is ionized in water. The final 10 mL of water evaporation is limited to ambient temperature to ensure inorganic CPM is not lost. We believe that the more volatile components of inorganic CPM could be evaporated from the samples if the catches are evaporated to dryness at elevated temperatures. Therefore, to promote consistency in application of the method, the final evaporations of hexane and water fractions are performed at ambient temperature.

Comment: (Comment No. 0048.1) Sections 11.2.2.2 and 11.2.2.5 require that after the sample has been evaporated to not less than 10 ml at room temperature the sample is to be transferred “quantitatively” to a pre-tared weighing tin where the evaporation continues at room temperature until all of the sample is evaporated. It is our recommendation that this step be eliminated from the process altogether. There is no way in the method to evaluate whether the sample has been transferred “quantitatively” and there is also a great risk of an accident occurring with all this additional handling. It is our opinion in general that the glass beaker that the sample is placed in after extraction should be the beaker it stays in until it is finally weighed. A 150 ml beaker is of sufficient size and with a tare weight of about 60 to 65 grams should not be too large to throw off the final weighings (Refer to Attachment 1 of NCASI’s comments).

Response: We disagree with the commenter’s suggestion to allow the use of glass beakers in the weighing process. The results generated by Environment Canada’s experiments (*Evaluation and Improvement of Condensable Particulate Matter Measurement* (Docket No. EPA-HQ-OAR-2008-0348-0028)) documented an uncertainty greater than 0.5 mg when glass beakers were used for this measurement. The combined uncertainty caused by the relatively heavy glass beakers and the much lighter sample weight is too great based upon the studies conducted by Environment Canada. Additionally, the Environment Canada study showed that the changes in barometric pressure affected the weight of the beaker when measured during the weighing process. Therefore, the final method specifies that the evaporation and weighing procedures must be conducted using weighing tins, fluoropolymer beaker liners, or fluoropolymer baggies.

Comment: (Comment No. 0059.1) Section 11.2.2.2 requires that the combined organic fraction, from both Container #1 (organic extract) and Container #2 (organic fraction) be transferred to a clean glass beaker. We suggest that the combined organic fraction from both Container #1 (organic extract) and Container #2 (organic fraction) be transferred to a clean flat bottom flask.

Response: We have determined that a conical beaker is necessary to reduce the time required for evaporation and to approximate the condensable material as it would form in ambient air. Therefore, we continue to recommend and specify clean, conical glass beakers for sample evaporation.

Comment: (Comment No. 0059.1) Section 11.2.2.2 requires that the organic fraction samples be dried at room temperature (less than 30°C) in fume hood. We suggest that the organic fraction samples be transferred to a vial for weight determination. Samples are then dried using an N-evap in fume hood at ambient temperature.

Response: We agree that transferring the organic fraction samples to an evaporation vial and the use of a nitrogen blowdown at ambient temperature complies with the method requirements for organic fraction weight determinations.

Comment: (Comment No. 0059.1) Section 11.2.2.2 requires that organic fraction samples be evaporated at room temperature in a fume hood to no less than 10 ml. We suggest that the organic fraction samples be evaporated using a rotary evaporator at 50°C to no less than 10 ml.

Response: We disagree with the evaporation of the organic fraction at 50°C and/or reduced pressure based upon Henry's Law calculations performed by Environment Canada (*Evaluation and Improvement of Condensable Particulate Matter Measurement* (Docket No. EPA-HQ-OAR-2008-0348-0028)). Therefore, we have not revised Section 11.2.2.2 to incorporate the commenter's suggestions

Comment: (Comment No. 0059.1) Section 11.2.2.2 requires that the organic fraction samples are transferred to a pre-weighed tin dish. We suggest that the organic fraction samples be transferred to a pre-weighed glass vial.

Response: In response to comments received regarding the use of alternatives to weighing tins, we have revised Section 6.3.2 to allow the use of fluoropolymer beaker liners as an alternative to weighing tins. However, the commenter's suggestion is not sufficiently specific with regard to the shape (e.g., tube, dish) of the glass vial to warrant a change to Section 11.2.2.2.

M202 11.2.2.3 INORGANIC FRACTION WEIGHT DETERMINATION

Comment: (Comment No. 0048.1) It is our opinion that the water that is used to reconstitute the sample for the titration does not have to be Type I, but should be the same water that produced an acceptable blank.

Response: We agree with the commenter that the same type of water can be used to redissolve the inorganic residue for the titration procedures as was used for sample recovery. We also agree with the commenter that the requirement to use Type I water is not necessary. Therefore, in response to this and other comments received on water specifications, the final rule requires the use of deionized, ultra-filtered water that meets a residual blank value of 1 ppmw in the sample collection, recovery, and analytical procedures.

Comment: (Comment No. 0059.1) Section 11.2.2.3 requires that 10 ml of remaining inorganic fraction be dried at room temperature (less than 30°C) in a fume hood. We suggest that 10 ml of remaining inorganic fraction be dried in an oven set between 28 and 30°C.

Response: We agree that the commenter's approach meets the method requirements specified in Section 11.2.2.3 that require sample evaporation be performed at room temperature less than 30°C. If samples are evaporated in controlled enclosures, the temperature inside the enclosure must be recorded and documented.

Comment: (Comment No. 0059.1) Section 11.2.2.3 requires that the inorganic fractions be transferred to a clean 500 ml beaker. We suggest that the inorganic fractions be transferred back to its original container. If a plastic container has been received then a clean beaker or a clean glass bottle equivalent in size is used.

Response: We disagree with the commenter's suggestion. The final method specifies the use of glass beakers for sample evaporation because aqueous CPM fractions will be evaporated at elevated temperature to expedite sample preparation. Evaporation of the aqueous sample fractions should take no longer than 24 hours to retain inorganic CPM based upon the experimental laboratory work done by Environment Canada (*Evaluation and Improvement of Condensable Particulate Matter Measurement* (Docket No. EPA-HQ-OAR-2008-0348-0028)). Open-mouth laboratory beakers withstand elevated evaporation temperatures and allow for faster evaporation due to the lack of restriction that is typical of screw-cap, wide-mouth sample bottles.

M202 11.2.2.4 TITRATION TO NEUTRALIZE ACID IN THE SAMPLE

Comment: (Comment No. 0059.1) Section 11.2.2.4 requires that the inorganic fraction of the samples be titrated using a burette filled with 0.1 normal (N) NH_4OH . We suggest that the inorganic fractions be titrated using calibrated 100 μl Eppendorf Pipet adding increments of 20 μl or larger aliquots, of 0.1 N NH_4OH .

Response: Section 11.2.2.2 of Method 202 (proposed Section 11.2.2.4) does not specify the manner (e.g., burette, pipet) in which the NH_4OH titrant must be added to the sample. Consequently, testing contractors have flexibility in selecting the equipment used for titration.

Comment: (Comment No. 0119) EPA CTM 028 specifically requires the use of a pH meter to titrate re-suspended aqueous samples to pH 7 (sec. 11.2.2.4). Although our lab is equipped with a pH meter, I prefer the analyst to use a colorimetric endpoint determination (i.e., phenolphthalein) in place of the meter. Phenolphthalein is much more reliable and does not require calibration. A meter, although calibrated, may have "response drift" since its last calibration). Phenolphthalein is consistently reliable and never needs calibration. I would like approval from the EPA for its use in place of a meter for this specific procedure.

Response: We agree with the commenter that a colorimetric endpoint determinant, such as phenolphthalein, is an acceptable alternative to using a pH meter. Our intent in the proposal was to neutralize the acids in the sample without adding more ammonium hydroxide that was necessary for neutralization. The use of phenolphthalein, or other indicator, achieves that

objective. Therefore, Sections 6.2 and 11.2.2.2 of the final method allow for the use of a colorimetric pH indicator.

Comment: (Comment No. 0122) Do we need to test the pH level of the samples in the lab if the pH levels are measured in the field during recovery? I did not see in the method where this step was required to be done in the lab.

Response: The method does not require testing contractors to measure the pH of the samples until the aqueous portion is rehydrated and then neutralized using 0.1 N sodium hydroxide (see Section 11.2.2.2).

M202 11.2.2.5 AQUEOUS PHASE EVAPORATION

Comment: (Comment No. 0056.1) API generally agrees with the proposed Method 202 sample processing procedures in Section 11.2.2.5 that provide for air drying of the last 10 ml of the sample. This is a prudent procedure.

Response: We acknowledge the commenter's support for the ambient evaporation requirements.

M202 12.1 NOMENCLATURE

Comment: (Comment No. 0040.2) Section 12.1 needs the addition of: V_i = Volume of inorganic fraction, ml; V_{ib} = Volume of inorganic field blank, ml; V_o = Volume of organic fraction, ml; and V_{ob} = Volume of organic field blank, ml.

Response: We agree with the commenter's edits. Therefore, we have revised Section 12.1 of the final method accordingly.

Comment: (Comment No. 0054.1) In the current Method 202, the constant for calculating m_c is 17.0026 (0.354 x 48.03). Should the 17.03 mg/milliequivalents be 17.00?

Response: The value of 17.03 mg/milliequivalents for the ammonium ion is correct. Therefore, no change was made to the final method.

M202 12.2.2 MASS OF FIELD BLANK (mg)

Comment: (Comment No. 0038.1) The title of this section should be “Mass of the Field Train Blank.” This section also incorrectly references Section 9.9 and should reference Section 9.10 – Field Train Blank.

Response: We agree with the commenter’s edits. Therefore, we have revised Section 12.2.2 accordingly.

Comment: (Comment No. 0041.1) Section 12.2.2 states that the mass of the field blank shall not exceed 2 mg. This 2 mg value does not agree with actual field blank values reported by several test firms. Comments and data were provided to the docket by Bruce Nemet of Resolution Analytics, Inc regarding the results of approximately a dozen field blanks collected by several different test firms. These field blank data demonstrate that the average field blank is approximately 5 mg. The basis for EPA selecting 2 mg as the maximum amount for the field blank correction appears arbitrary. Section 12.2.2 should be revised to state that the field blank correction shall not exceed 5 mg. EPA should evaluate the sources or key contributors of the field blank. The importance of each of the following should be evaluated: Teflon® Membrane Filters; Solvents (acetone, methylene chloride, and de-ionized water); Sample Train Glassware; Nitrogen Purge; and Analytical Laboratory Technique. The appropriate value for the maximum field blank correction should not be established without knowing which factors or items are the key contributors to the field blank results.

Response: We established the 2.0 mg blank correction based upon laboratory tests (*Evaluation and Improvement of Condensable Particulate Matter Measurement* (Docket No. EPA-HQ-OAR-2008-0348-0028)) that indicated SO₂ artifact formation during a typical compliance test at a well-controlled source would be 2.0 mg. We were able to demonstrate much lower laboratory blanks using the method procedures, as proposed, during these tests.

We have evaluated data from stakeholder field tests and EPA-sponsored field evaluations of the revised method (*Field Evaluation of an Improved Method for Sampling and Analysis of*

Filterable and Condensable Particulate Matter; Docket no. EPA-HQ-OAR-2008-0348) and found that final field blanks taken before sampling (often called proof blanks) meet the method requirements. Other field train recovery blank data taken between sampling runs demonstrate variable blank recovery between 1 and 10 mg. We believe that the higher field train recovery blank results mentioned by the commenter may occur as a result of one or more causes. One cause may be because the sample has not been completely recovered between sampling runs. Another cause may be due to contamination of glassware or recovery solvents. Other sampling and recovery issues may also cause higher blank values cited by the commenter.

We recognize there is inherent blank residual in Method 202 originating primarily from the inorganic fraction of the samples. We have determined that the blank results achieved by proper application of Method 202 in both laboratory and field tests is approximately 2 mg of blank residue. We believe that source testers that routinely have field train recovery blanks in excess of 2 mg should investigate the cause of the excess. Therefore, we do not believe the data cited by the commenter constitute grounds for increasing the blank correction value.

Comment: (Comment No. 0080.1) In Method 5, “field rinse blanks” are used to adjust the results of each component of the sample to account for the error introduced by the residue in the reagents. In Method 202, the field train blank serves this purpose. However, no more than 2 mg may be subtracted from the test run results, depending on the results of analysis of the field blank train. We support the use of a field train blank, and encourage EPA to do further studies on the upper limit of the amount that should be allowed to be subtracted as we feel that insufficient data was used to arrive at the current 2 mg quantity. However, we are concerned that inconsistencies between Method 5 and Method 202 in how blanks are handled may lead to confusion and problems, since the two Methods are frequently performed concurrently. We would encourage EPA to harmonize Method 5 and 202 blank practices.

Response: We disagree with the commenter that the allowable correction for field train recovery blank mass in Methods 5 and 202 should be the same value. Method 202 has been revised to modernize the approach for blank correction and make it more relevant to actual field contamination detection. In Method 202, the field train recovery blank results accommodate not only the residue in the recovery reagents, but also residue that is associated with well cleaned glassware that can not be eliminated. Consequently, the quality of the data from Method 202

measurements will be more representative of field sample contamination than those from using Method 5.

M202 **12.2.4 TOTAL MASS OF CPM (mg)**

Comment: (Comment No. 0040.2) I would like to suggest rearranging Equation 4 in Section 12.2.4 to read: “Where the sample and blank volumes of each fraction are the same, $m_{\text{cpm}} = (m_i - m_{\text{ib}}) + (m_o - m_{\text{ob}})$. Where the sample and blank volumes of each fraction are different, $m_{\text{cpm}} = (m_i - m_{\text{ib}} * V_i / V_{\text{ib}}) + (m_o - m_{\text{ob}} * V_o / V_{\text{bo}})$.” By blank correcting each fraction separately, the risk of over applying a high inorganic blank value to the Total Mass of CPM would be minimized. This would also allow the Inorganic and Organic Fractions to be reported separately if desired by the Administrator.

(Comment No. 0041.1) The CPM results obtained with the proposed Method 202 represent a single value (i.e., total CPM). By summing the inorganic and organic field blank values and subtracting the total field blank (m_{fb}), it is no longer possible to separately report organic CPM and inorganic CPM. EPA may want to consider making separate field blank corrections for the inorganic field blank and the organic field blank, such that the proposed Method 202 yields results for both types of CPM (i.e., organic CPM and inorganic CPM). Categorizing the CPM results as organic or inorganic may provide useful information about various types of emission sources.

Response: The blank correction for this method has been generalized and simplified to correct the total CPM measurement for field train recovery blanks up to a limit of 2.0 mg. The complexity of adjusting the inorganic and organic fractions individually and limiting the total correction to 2.0 mg is not necessary for the intended use of the method results. However, it is likely that alternative reporting can be negotiated with the appropriate regulatory authority. Therefore, we do not believe that it is necessary to correct the blank mass separately for the organic and inorganic fractions. Consequently, we are not revising Equation 4 in Section 12.2.4 of the final method.