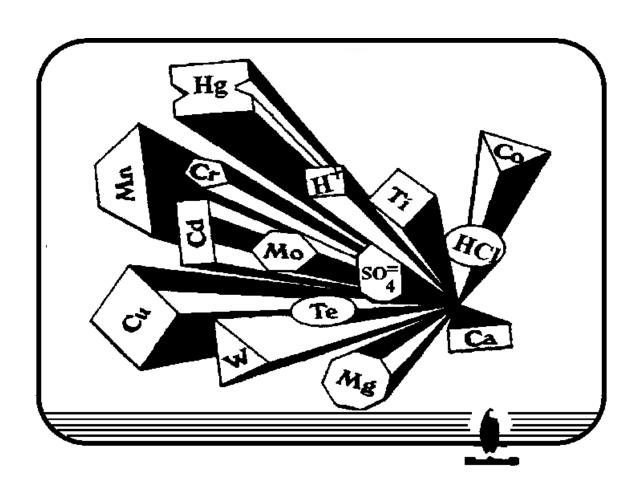
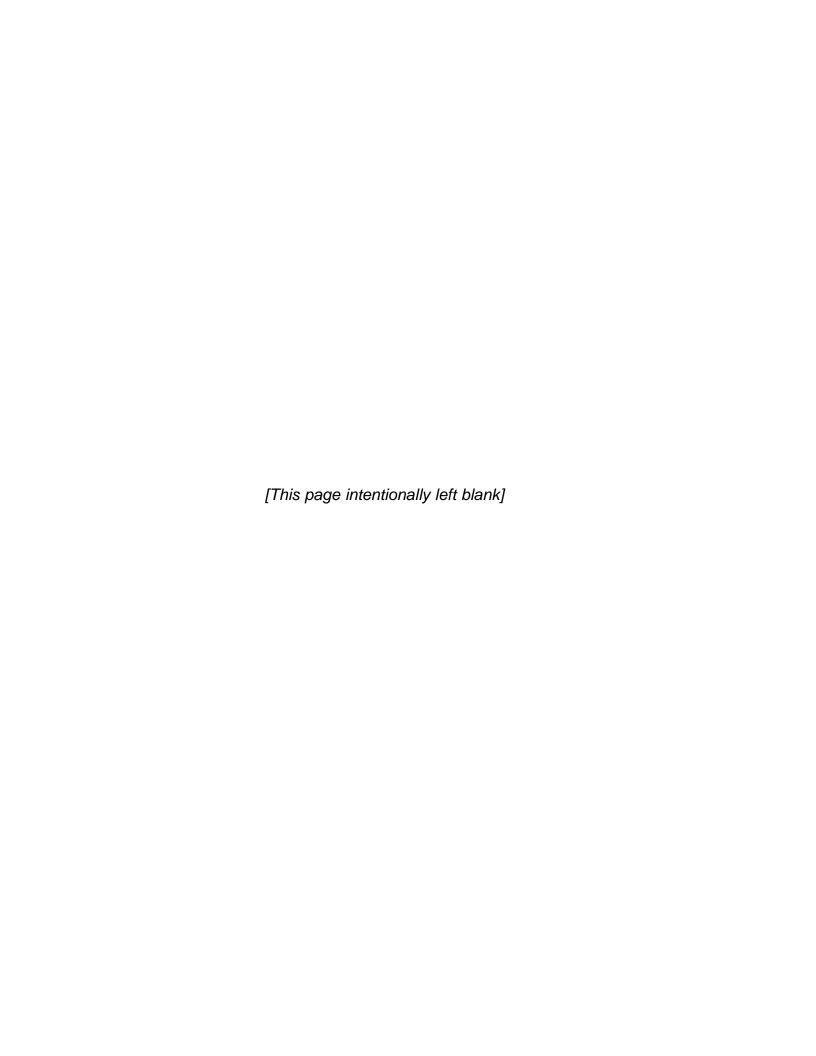
# **⇔**EPA Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air





Research and Development

EPA/625/SR-96/010a

June 1999



## **Project Summary**

# Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air

This Project Summary is the announcement of the availability of a collection of methods for measurement of inorganic pollutants of interest in ambient air. These methods have been prepared to provide regional, state and local environmental regulatory agencies and other users with step-by-step sampling and analysis procedures for the determination of selected inorganic pollutants in ambient air. The methods comprise the *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air*, which is intended to assist those persons responsible for sampling and analysis of ambient inorganic pollutants.

Determination of pollutants in ambient air is a complex task, primarily because of the wide variety of compounds of interest coupled with the lack of standardized sampling and analysis procedures. Many inorganic compounds can be sampled and analyzed by several techniques, often with different interferences and detection limitations. This Compendium contains a set of 17 methods (in 5 categories) presented in a standardized format, with a variety of applicable sampling methods and various analytical techniques for specific classes of inorganic pollutants, as appropriate to their ambient levels and potential interferences. Consequently, this treatment allows flexibility in selecting alternatives to complement the user's background and laboratory capability. These methods may be modified from time to time as advancements are made.

This Project Summary was developed by EPA's Center for Environmental Research Information (CERI), National Risk Management Research Laboratory (NRMRL), Office of Research and Development (ORD), U.S. EPA, Cincinnati, OH, with assistance from the ORD's National Exposure Research Laboratory (NERL) at Research Triangle Park, NC. Its purpose is to announce key findings of the research project, which is fully documented in a separate report of the same title (see Project Report ordering information on the last page).

### Introduction

The Clean Air Act Amendments of 1990 (CAAA of 1990) have significantly increased the responsibilities of both federal and state agency programs for evaluating and maintaining air pollutant emissions compliance. In turn, this increased responsibility has generated a need for more personnel trained to interpret, enforce, and respond to regulatory initiatives. Consequently, the Agency has restructured its technology transfer program to more effectively provide technical assistance in the form of publication of technical documents, presentations and workshops, and development of tools to assist Agency personnel in keeping their skills up-to-date so that they may efficiently cope with the many changes evolving in new programs, equipment, sampling and analytical methodology, and available enforcement tools.

Limited guidance has been available to state and local agencies or to other organizations concerned with the determination of inorganic pollutant concentrations in ambient air. As a result, state and local agencies and others responding to air pollution problems have had to develop their own monitoring strategies, including selection of monitoring methods, sampling plan designs, and specific procedures for sampling, analysis, logistics, calibration and quality control. For the most part, these procedures were based on professional judgments rather than adherence to any documented uniform guidelines. Many governmental agencies and professional or research organizations have developed ambient air monitoring methods and procedures, mostly to respond to specialized needs. But these methods and procedures have, *in general*, been neither standardized nor readily available to other agencies involved with ambient air monitoring for various pollutants.

To meet these needs, EPA's ORD, through CERI and NERL has supported technology transfer programs involving standardized, peer reviewed monitoring methods for regulatory and industrial personnel via publications of a series of methods Compendia. Other Compendia in this series, reflecting EPA's commitment to technology transfer, are:

- Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA 600/4-89-017, June 1988.
- Compendium of Methods for the Determination of Air Pollutants in Indoor Air, EPA 600/4-90-010, April 1990.
- Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, EPA 625/R-96/010b, January 1999.

These Compendia have historically assisted Federal, State, and local regulatory personnel in developing and maintaining necessary expertise and up-to-date technology involving sampling and analysis of both indoor and organic hazardous air pollutants (HAPs). The objective of this project was to develop and standardize methods for measuring inorganic pollutants of interest in ambient air and publish them in this *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air*. This fourth Compendium adds much needed methods for measurement of inorganic pollutants in ambient air to the series.

Consistent with past practices, the Compendium methods are provided as guidance only in appropriate monitoring situations. In particular, these methods are not intended to be used as specific regulatory guidance for measurement or monitoring purposes and are offered with <u>no endorsement for suitability or recommendation for any specific application</u>; rather, this is merely to document the methods and to make them more widely available.

### Structure and Content of the Inorganic Compendium

This Compendium has been prepared to provide regional, state and local environmental regulatory agencies, as well as other interested parties, with specific guidance on the determination of selected inorganic compounds and pollutants in ambient air. A visual guide to the organization of the Inorganic Compendium is illustrated in Figure 1, while Table 1 lists the 17 methods which comprise the 5 chapters of the Compendium. The 17 methods have been compiled from the best elements of methods developed or used by various research or monitoring organizations. They are presented in a standardized format, and each one has been extensively reviewed by several technical experts having expertise in the methodology presented.

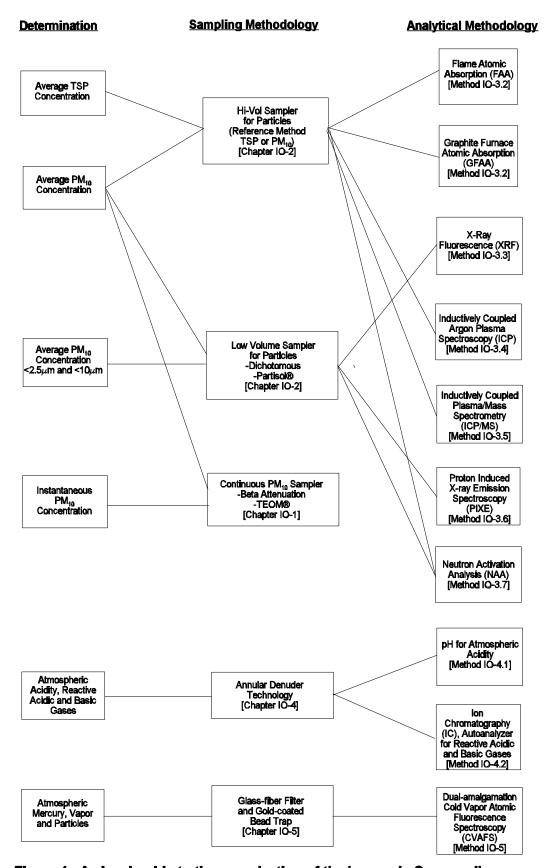


Figure 1. A visual guide to the organization of the Inorganic Compendium.

### Table 1. Methods in the Inorganic Compendium

### Chapter IO-1 Continuous Measurement of PM<sub>10</sub> Suspended Particulate Matter (SPM) in Ambient Air

- Method IO-1.1 Determination of PM<sub>10</sub> in Ambient Air Using the Andersen Continuous Beta Attenuation Monitor
- Method IO-1.2 Determination of PM<sub>10</sub> in Ambient Air Using the Thermo Environmental Inc. (formerly Wedding and Associates) Continuous Beta Attenuation Monitor
- Method IO-1.3 Determination of PM<sub>10</sub> in Ambient Air Using a Continuous Rupprecht and Patashnick (R&P) TEOM® Particle Monitor

### Chapter IO-2 Integrated Sampling of Suspended Particulate Matter (SPM) In Ambient Air

- Method IO-2.1 Sampling of Ambient Air for Total Suspended Particulate Matter (SPM) and PM<sub>10</sub> Using High Volume (HV) Sampler
- Method IO-2.2 Sampling of Ambient Air for PM<sub>10</sub> Using an Andersen Dichotomous Sampler
- Method IO-2.3 Sampling of Ambient Air for PM<sub>10</sub> Concentration Using the Rupprecht and Patashnick (R&P) Low Volume Partisol® Sampler
- Method IO-2.4 Calculations for Standard Volume

### **Chapter IO-3** Chemical Species Analysis of Filter-Collected Suspended Particulate Matter

- Method IO-3.1 Selection, Preparation and Extraction of Filter Material
- Method IO-3.2 Determination of Metals in Ambient Particulate Matter Using Atomic Absorption (AA) Spectroscopy
- Method IO-3.3 Determination of Metals in Ambient Particulate Matter Using X-Ray Fluorescence (XRF) Spectroscopy
- Method IO-3.4 Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma (ICP) Spectroscopy
- Method IO-3.5 Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)
- Method IO-3.6 Determination of Metals in Ambient Particulate Matter Using Proton Induced X-Ray Emission (PIXE) Spectroscopy
- Method IO-3.7 Determination of Metals in Ambient Particulate Matter Using Neutron Activation Analysis (NAA) Gamma Spectrometry

# Chapter IO-4 Determination of Reactive Acidic and Basic Gases and Strong Acidity of Atmospheric Fine Particles in Ambient Air Using the Annual Denuder Technology

- Method IO-4.1 Determination of the Strong Acidity of Atmospheric Fine Particles  $(2.5\mu m)$
- Method IO-4.2 Determination of Reactive Acidic and Basic Gases and Strong Acidity of Atmospheric Fine Particles

### Chapter IO-5 Sampling and Analysis for Atmospheric Mercury

Method IO-5 Sampling and Analysis for Vapor and Particle Phase Mercury in Ambient Air Utilizing Cold Vapor Atomic Fluorescence Spectrometry (CVAFS)

The 17 specific methods, covering a variety of inorganic pollutants and compounds, are categorized into 5 chapters. Nearly all the procedures have considerable flexibility and assume that the analyst has a substantial air monitoring background and expertise. Consequently, users are responsible for preparing a specific standard operating procedure (SOP) when incorporating the Inorganic Compendium methods into their air monitoring program.

### **Ordering Information**

The Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, EPA-625/R-96/010a, may be purchased in hard copy from:

National Technical Information Service (NTIS) 5285 Port Royal Road, Springfield, VA 22161 Telephone: 703-487-4650; Fax: 703-321-8547 E-mail: Info@NTIS.FEDWORLD.GOV

Internet: http://www.ntis.gov

► U.S. EPA: Office of Air Quality Planning and Standards (OAQPS)

Electronic versions of the individual Compendium (IO-) Methods are available for downloading from the "AMTIC" section of EPA's OAQPS Technology Transfer Network via the Internet at the "AMTIC, Air Toxics" section of the TTN Web:

### http://www.epa.gov/ttn/amtic/

Methods IO-1 to IO-5 are posted in the portable document format (PDF). The downloaded files can be read using an Acrobat Reader. Acrobat readers are available from Adobe®, free of charge, at:

### http://www.adobe.com/prodindex/acrobat/readstep.html

and are required to read Acrobat (PDF) files. Readers are available for Windows, Macintosh®, and DOS.

- ▶ U.S. EPA: Office of Research and Development,
- Center for Environmental Research Information (CERI)

New technology transfer documents may be ordered on-line through the CERI Technology Transfer web site -- "http://www.epa.gov/ttbnrmrl/". Once the web site has been assessed, click on the publication list and follow the menu-driven ordering instructions.

### Acknowledgments

The Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air (EPA/625/R-96/010a) was prepared under Contract No. 68-C3-0315, Work Assignments Nos. 2-10 and 3-10, by Midwest Research Institute (MRI), as a subcontractor to Eastern Research Group, Inc. (ERG), and under the sponsorship of the U.S. Environmental Protection Agency (EPA). Final revisions were performed by Science Applications International Corporation (SAIC) under Contract No. 68-C7-0011, W.A. 1-82. Justice A. Manning, John O. Burckle, and Scott Hedges, Center for Environmental Research Information (CERI), and Frank F. McElroy, National Exposure Research Laboratory (NERL), all in the EPA Office of Research and Development, were responsible for overseeing the preparation of the Compendium. Further support was provided by other members of the Compendia Workgroup, which included:

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# COMPENDIUM OF METHODS FOR THE DETERMINATION OF INORGANIC COMPOUNDS IN AMBIENT AIR

U. S. Environmental Protection Agency Office of Research and Development Center for Environmental Research Information National Risk Management Research Laboratory Cincinnati, Ohio 45268

### **DISCLAIMER**

The information in this document has been compiled wholly or in part by the United States Environmental Protection Agency (EPA) under Contract No. 68-C3-0315, W.A. 2-10 to Eastern Research Group (ERG). The work was performed by Midwest Research Institute (MRI) under subcontract to ERG. Final revisions were performed by Science Applications International Corporation (SAIC) under Contract No. 68-C7-0011, W.A. 1-82. It has been subjected to the Agency's peer and administrative review, and it has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

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### Acknowledgments

These Methods were prepared for publication in the *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air* (EPA/625/R-96/010a), which was prepared under Contract No. 68-C3-0315, WA No. 2-10, by Midwest Research Institute (MRI), as a subcontractor to Eastern Research Group, Inc. (ERG), and under the sponsorship of the U. S. Environmental Protection Agency (EPA). Final revisions were performed by Science Applications International Corporation (SAIC) under Contract No. 68-C7-0011, W.A. 1-82. Justice A. Manning, John O. Burckle, and Scott Hedges, Center for Environmental Research Information (CERI), and Frank F. McElroy, National Exposure Research Laboratory (NERL), all in the EPA Office of Research and Development, were responsible for overseeing the preparation of this Compendium. Other support was provided by the following members of the Compendia Workgroup:

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These Methods are the result of the efforts of many individuals. Gratitude goes to each person involved in the preparation and review of this methodology.

# Chapter IO-1: CONTINUOUS MEASUREMENT OF PM<sub>10</sub> SUSPENDED PARTICULATE MATTER (SPM) IN AMBIENT AIR

# Method IO-1.1: Determination of $PM_{10}$ in Ambient Air Using the Andersen Continuous Beta Attenuation Monitor

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# Method IO-1.2: Determination of $PM_{10}$ in Ambient Air Using the Thermo Environmental Instruments (formerly Wedding and Associates) Continuous Beta Attenuation Monitor

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# Method IO-1.3: Determination of PM<sub>10</sub> in Ambient Air Using a Continuous Rupprecht and Patashnick (R&P) TEOM® Particle Monitor

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# Chapter IO-2: INTEGRATED SAMPLING OF SUSPENDED PARTICULATE MATTER (SPM) IN AMBIENT AIR

# Method IO-2.1: Sampling of Ambient Air for Total Suspended Particulate Matter (SPM) and PM<sub>10</sub> Using High Volume (HV) Sampler

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### Method IO-2.2: Sampling of Ambient Air for PM<sub>10</sub> Using an Anderson Dichotomous Sampler

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# Method IO-2.3: Sampling of Ambient Air for PM<sub>10</sub> Concentration Using the Rupprecht and Patashnick (R&P) Low Volume Partisol® Sampler

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### Method IO-2.4: Calculations for Standard Volume

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# Chapter IO-3: CHEMICAL SPECIES ANALYSIS OF FILTER-COLLECTED SUSPENDED PARTICULATE MATTER

### **Method IO-3.1:** Selection, Preparation, and Extraction of Filter Material

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# Method IO-3.2: Determination of Metals in Ambient Particulate Matter Using Atomic Absorption (AA) Spectroscopy

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# Method IO-3.3: Determination of Metals in Ambient Particulate Matter Using X-Ray Fluorescence (XRF) Spectroscopy

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### Method IO-3.4: Determination of Metals in Ambient Particulate Matter Using Inductively

### Coupled Plasma (ICP) Spectroscopy

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# Method IO-3.5: Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)

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# Method IO-3.6: Determination of Metals in Ambient Particulate Matter Using Proton Induced X-Ray Emission (PIXE) Spectroscopy

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# Method IO-3.7: Determination of Metals in Ambient Particulate Matter Using Neutron Activation Analysis (NAA) Gamma Spectrometry

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# Chapter IO-4: DETERMINATION OF REACTIVE ACIDIC AND BASIC GASES AND STRONG ACIDITY OF ATMOSPHERIC FINE PARTICLES IN AMBIENT AIR USING THE ANNULAR DENUDER TECHNOLOGY

### **Method IO-4.1:** Determination of the Strong Acidity of Atmospheric Fine-Particles (<2.5 Fm)

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# Method IO-4.2: Determination of Reactive Acidic and Basic Gases and Strong Acidity of Atmospheric Fine Particles (<2.5 Fm)

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### Chapter IO-5: SAMPLING AND ANALYSIS FOR ATMOSPHERIC MERCURY

# Method IO-5: Sampling and Analysis for Vapor and Particle Phase Mercury in Ambient Air Utilizing Cold Vapor Atomic Fluorescence Spectrometry (CVAFS)

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### **FOREWORD**

The U. S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, EPA's research program is providing data and technical support for solving environmental problems today and building a science knowledge base necessary to manage our ecological resources wisely, understand how pollutants affect our health, and prevent or reduce environmental risks in the future.

The National Risk Management Research Laboratory (NRMRL) is the Agency's center for investigation of technological and management approaches for reducing risks from threats to human health and the environment. The focus of the Laboratory's research program is on methods for the prevention and control of pollution to air, land, water, and subsurface resources; protection of water quality in public water systems; remediation of contaminated sites and ground water; and prevention and control of indoor air pollution. The goal of this research effort is to catalyze development and implementation of innovative, cost-effective environmental technologies; develop scientific and engineering information needed by EPA to support regulatory and policy decisions; and provide technical support and information transfer to ensure effective implementation of environmental regulations and strategies.

Measurement of inorganic pollutants in ambient air is often difficult, in part because of the variety of inorganic substances of potential concern, the variety of potential techniques for sampling and analysis, and lack of standardized and documented methods. This Compendium is one of three Compendia of methods which provide documented and technically reviewed methodology for determining concentrations of selected pollutants of frequent interest in ambient and indoor air. The methods contained in this Compendium provide sampling and analysis procedures for a variety of inorganic pollutants and suspended particulate matter in ambient air. As with the previous Compendia methods, these methods are provided only for consideration by the user for whatever potential applications for which they may be deemed appropriate. In particular, these methods are not intended to be associated with any specific regulatory monitoring purpose and are offered with no specific endorsement for fitness or recommendation for any particular application, other than for an attempt at standardization.

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E. Timothy Oppelt, Director National Risk Management Research Laboratory