ANNOTATED BIBLIOGRAPHY OF ANALYTICAL METHODS FOR CERCLA HAZARDOUS SUBSTANCES

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EXECUTIVE SUMMARY

Measurement of the magnitude and distribution of releases of hazardous substances is required in support of efforts under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA). A Comprehensive Hazardous Substances List (the List), comprised of substances specified in Sections 311 and 307a of the Clean Water Act, Section 112 of the Clean Air Act, and the P and U Lists and F and K Waste Streams of the Resource Conservation and Recovery Act (RCRA), delineates the substances under the purview of CERCLA. The Comprehensive Hazardous Substances List contains approximately 617 unique listings, of which about two-thirds pertain to organic compounds and one-third to inorganic or organometallic substances.

Under Section 102(b) of CERCLA, each substance on the List is assigned a Reportable Quantity (RQ) value that specifies the amount of the substance, the release of which must be reported to the National Response Center. The U.S. EPA Office of Emergency and Remedial Response (OERR) is considering adjustment of these RQ values, and in order to do so requires information about the technical feasibility and financial impact of measurements of releases of hazardous substances. This report provides such information in support of the OERR rulemaking effort.

This report discusses the state of the art of analytical chemistry in the context of CERCLA, and presents the results of a search for specific analytical methods for each substance under the purview of CERCLA in three media - air, soil, and water - at two levels of sophistication - field analyses and laboratory methods. This report is not a critical review, but a non-critical survey, and deals only with demonstrated analytical methods. Specific facts about each analytical method located are presented as Method Descriptions. The Method Descriptions comprise an annotated bibliography, and each one gives a method summary, the interferences listed, the quality control prescribed, the EPA/ technical status or sensitivity of the method, the reference, and the cost of analysis by that method. The Method Descriptions are in two Appendices - A and B - for single- and multicompound methods, respectively. By a singlecompound method the authors mean a method demonstrated to be applicable to the determination of only one compound on the List, although it may also have been demonstrated to be applicable to compounds not on the List. Appendix C gives rudimentary guidance to appropriate analytical methods for the RCRA F and K Waste Streams, within the framework of Appendices A and B.

Overall, field methods have been located for 34 percent of the 1,851 possible compound/matrix combinations, and laboratory methods for 80 percent of them. Analytical methods seem better developed for inorganics than for organics at first glance, but most methods located for the determination of inorganics are for only a portion of the whole compound and, as such, do not unequivocally identify

or measure the CERCLA-hazardous substance, as required by CERCLA. For a total of 596, or 96 percent, of the 621 possible inorganic substance/matrix combinations, one or more laboratory methods were located. For organic compounds, laboratory methods were located for 71 percent of the possible substance/matrix combinations. These methods are usually satisfactory for at least tentative identification of the analyte. Field methods for organics, without the sophistication of laboratory equipment, were located for only 21 percent of the possible 1230 organic compound/matrix combinations.

The weakest technology for any analyte/matrix/level-of-sophistication combination was found to be in the field analysis of soil samples for organic compounds. This type of method was located for only 19 analytes. Field analysis of soil/sediment samples for inorganic substances was also found to be weak, with only 67 substances accounted for. As in nearly all of the inorganic procedures, these methods are typically elemental methods, not compound-specific ones, and do not identify the substance as a CERCLA compound, as required under CERCLA. Field water and air analytical methods, in combination with novel sample preparation procedures, could be applied to broaden the scope of both organic and inorganic field analyses. Similarly, empty spots in other technologies could be filled by the development of sample preparation procedures and However, the most promising way utilization of existing analytical methods. of conducting field site characterization is the use of mobile laboratories and laboratory equipment manufactured to be field-portable. These two types of analysis would allow methods of higher technology, once restricted to fixedsite use, to be used in the field. Additionally, use of the broad listings of all compounds of a given type (e.g., 'cyanides', 'arsenic and compounds') in regulations would help match the regulatory situation to analytical capabilities, since inorganic analytical capabilities would then match the regulations more closely.

The cost of analysis has been obtained from one of three sources: bid prices from government contracts, list prices from commercial analytical facilities, or price estimates based on individual cost of each step in the analysis. The cost for the determination of organic compounds ranges from approximately \$75.00 to \$300.00 per sample for laboratory analysis of lots of six samples, while field analysis costs vary from \$5.00 to \$40.00 per sample. Inorganic analysis costs vary from a range of approximately \$15.00 to approximately \$175.00 per sample for laboratory analysis of lots of six samples to a range of \$5.00 to \$40.00 for field analysis. Cost figures given do not include sampling costs, except for field analysis figures.

The availability of analytical methods is necessary, both for compliance by the regulated community with Superfund rules and regulations, and for enforcement of them by the EPA. With no field or laboratory analytical methods located for approximately 35 substances in air, water, or soil samples, strict enforcement of and compliance with Superfund regulations becomes impossible in cases of releases of those substances. In addition, EPA enforcement efforts and citizen compliance are made difficult or impossible by the sparseness of analytical methods for many of the other substances on the Superfund Comprehensive Hazardous Substances List. Furthermore, much of the technology located in the method-search effort for this report has not been evaluated by the EPA, and the location of the information is often not known to the regulated community, or

even to EPA personnel. This Annotated Bibliography should partially alleviate these problems. To make the information contained in this report easily available to regulators and users, it is at present being investigated if computerizing this report is feasible.

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GLOSSARY

| Absorption | - | the process of trapping an analyte in a liquid or solid. |
|----------------------|---|---|
| Adsorbent | - | the solid material on the surface of which adsorption occurs. |
| Adsorption | - | the process of trapping an analyte on the surface of a solid. |
| Analyte | - | molecule or ion for which analysis is being performed. |
| Breakthrough volume | - | the volume of air which, when sampled, begins to allow analyte to escape from the sampling medium. |
| Bubbler | - | sampling device consisting of a gas dispenser immersed in an absorbing liquid. |
| Calibration | _ | the establishment of a relationship between the response of a measurement system and the amount of analyte present, usually by measurement of the response due to several calibration standards, in order to quantify the amount of analyte in samples. |
| Composite sample | - | a sample composed of two or more increments. |
| Cumulative sample | - | a sample that is an average sample over a time period. The collected sample is retained in a single vessel or the separated component accumulated in one place. |
| Cryogenic collection | - | a sampling process wherein an air sample is passed through a trap cooled by liquid argon, ice, or similar material in order to collect volatile compounds. |
| Derivatization | - | process wherein the analyte is converted to another chemical species through chemical reaction. |
| Desorption | - | the process of releasing a substance from a sorbed (trapped or fixed) state by elution or heating. |
| Elute | - | to remove sorbed materials from a sorbent by use of a fluid. |

Filter

 a porous material used to separate solids from liquids or gases. (See Reference 12 for a detailed discussion of use and specifications of filters for air sampling.)

Functional group

- a particular configuration or group of atoms in an organic molecule.

Immiscible

- liquids that do not combine to make one phase, or a solution.

Impinger

- see "Bubbler".

Instantaneous sampling

 obtaining a sample in a time period that is negligible compared to the time scale of fluctuations in the level of analyte present.

Interference

- a substance that interferes with an analysis by obscuring the true signal from the analyte, making the analyte inactive towards detection, or making the analyte unavailable for detection.

Internal standard

- a known quality of a reference compound added to a collected sample, and generally to standards as well, for use in quantification of other compounds (see "calibration").

Limit of detection -

- the minimum quantity or concentration of analyte which produces a measurable response, that is, a signal above the noise level. The "instrumental detection limit" and "method detection limit" refer to the limit of detection in terms of the amount or concentration of analyte introduced into the instrumental measurement system and the amount or concentration of analyte carried through the complete analytical process, respectively.

Matrix

Sample matrix

- The type of material containing the analyte (e.g., air water, soil).

Matrix effects

- interferences in an analysis due to substances present in the specific sample matrix of a particular sample.

Method blank

 an artificial sample containing no analyte (e.g., distilled water), used to check for contamination of samples during analysis.

Moiety

- part or all of a compound (e.g., atom, ion, molecule).

Molar

- pertaining to a solution containing one mole (6 \times 10²³ atoms/ions/molecules) solute per liter of solution.

Phase a physically separate part of a heterogeneous system. Quantification - the assignment of a numerical value to the amount or concentration of analyte in a sample. Reference standard a solution or sample of a precisely known concentration, used to assess the accuracy of an analysis. Sensitivity the amount or concentration of analyte that will produce a certain level of response, such as 1 percent (0.044 absorbance units), from an analytical instrument. Solute the substance dissolved in a solvent. Sonication a method of adding energy, to aid in an extraction, in the form of sound waves. Sorbent a solid or liquid which traps or fixes an analyte by absorption or adsorption. Soxhlet extraction a particularly efficient manner of extracting analytes from solid samples, using continuous-flow contact of the sample with warm solvent. TFE a fluorocarbon plastic that does not contain plasticizers

that leach into solutions.

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INTRODUCTION

BACKGROUND

In 1980 the United States Congress enacted PL 96-510, the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA, the Act, or Superfund). CERCLA regulates hazardous substances, in order to minimize the chance of uncontrolled releases into the environment and to ensure speedy responses to such situations. Under Sections 103(a) and 103(b) of CERCLA, any party responsible for the release of an amount of a CERCLA-designated hazardous substance greater than or equal to the Reportable Quantity (RQ) for that substance must notify the National Response Center of the release. The 617 substances that fall under the purview of CERCLA are specified in Section 101(14) of the Act. Under Section 102 of CERCLA, the U.S. Environmental Protection Agency (EPA) may also designate other hazardous substances.

The substances designated in Section 101(14) of the Act are comprised of those specified in Sections 311 and 307(a) of the Clean Water Act (297 and 65 substances, respectively), Section 112 of the Clean Air Act (7 substances), and all of the hazardous substances identified in Section 3001 of the Resource Conservation and Recovery Act (RCRA). The substances designated in RCRA are contained in two lists of specific chemical products, wastes, or intermediates (the P and U lists; 107 and 233 entries, respectively) and two lists of processspecific industrial waste streams (the F and K lists; 13 and 67 waste streams, respectively). CERCLA also specifies that substances designated in Section 7 of the Toxic Substances Control Act (TSCA) are covered, but no compounds are currently regulated under that legislation. A Comprehensive Hazardous Substances List (the List) containing the 617 substances specified in Section 101(14) of the Act has been published, 1 and is reproduced at the end of this volume (see page 49). In addition to these chemical listings, the Act specifies 114 radionuclides of concern under CERCLA. The EPA has a memorandum of understanding with the Nuclear Regulatory Commission to deal with these. 2 and this report will not address them.

Under Section 102(b) of CERCLA, each substance is initially assigned either the same Reportable Quantity value as that given it in the Federal Water Pollution Control Act, or, if none is specified there, a value of one pound. RQ values assigned in the Federal Water Pollution Control Act range from 0.454 kilograms (1 pound) to 2270 kilograms (5000 pounds). Adjustment of these RQ values, in order to minimize unnecessary notifications of the National Response Center and still protect the public health and welfare, is being carried out by the Office of Emergency and Remedial Response of the U.S. EPA under Section 102 of the Act.

In order to determine whether release of a Reportable Quantity of a CERCLA hazardous substance has occurred, methods of chemical analysis for these substances in environmental samples are needed. For that reason, as well as to aid in the assessment of the feasibility of possible changes in RQ-values, a survey of the state of the art of analytical chemistry in the Superfund context has been undertaken. This report discusses the technical feasibility and cost of such analyses and presents a compound-by-compound cataloguing of analytical methods for three sample matrices: air, water, and soil/sediment.

In order to characterize a release of a hazardous substance properly, both quantitative and qualitative information is required. Field methods are necessary to rapidly characterize the site and to gauge the extent and location of contamination of air, soil, and water there. Such information can then be used to design a suitable sampling plan for the implementation of laboratory analyses. These analyses should yield information that is legally defensible about the amount of the hazardous substance(s) present in samples and should clearly and unequivocally identify it as a substance on the List.

The difficulties associated with satisfying these requirements for measurements made in support of the Act are compounded by the broad scenario encompassed by releases of CERCLA-hazardous substances. Under Superfund, such a release can be either episodic or continuous, and the analytical techniques necessary for such circumstances can range from those appropriate for very high concentrations to those suitable for ultra-trace levels. The hazardous substances involved can be solids, liquids, gases, or mixtures of more than one phase, and can be either nearly pure substances, which may be well characterized and labeled, or can be mixtures of unknown composition. The variability in the degree of knowledge of both the identity of the hazardous substances involved and the physical state of them is made even more vexing by the variability of the potential matrix into which they may be released. Soils and sediments can have extremely different characteristics that vary greatly from one area of the United States to another. Salt waters, brackish waters, and highly anaerobic waters may well have different characteristics that can hamper analysis to varying degrees. Different levels of particulates and humidity in air across the nation can affect the efficiency of air sampling. In addition, interactions with the sample matrix or other components of mixtures containing CERCLAhazardous substances can further complicate the situation. The broad scope of potential situations encompassed in the scenario of a release of CERCLA hazardous substances highlights the weaknesses and limitations of modern analytical technology.

Due to these reasons, the three general classifications of sample matrices (air, soil/sediment, and water) used in this report belie the complexities associated with them. Consideration of the general effects associated with each broad class of sample matrix is made in the individual Method Descriptions. However, individual interferences that may be present in the specific matrix of a particular sample cannot be considered, as such factors are release-specific. For example, sediment samples frequently contain sulfur and sulfides due to anaerobic conditions. This type of general interference may be considered in the discussion of an analytical method if it is specifically mentioned in the source of the method. However, particular organic compounds that may be present in one specific soil sample are not taken into account.

SCOPE

Since the Comprehensive Hazardous Substances List specified in Section 101(14) of the Act is a composite of other regulatory lists, substances are frequently listed by more than one regulatory synonym. In addition, several entries on the List are for broad classes of compounds, like "Arsenic and Compounds" and "Phthalate Esters". The F and K Waste Streams of RCRA present a situation even more vaguely defined because particular substances are not listed, but the origins of mixtures are specified. In addition, some substances are regulated under RCRA (and, therefore, CERCLA) due to their characteristics of ignitability, corrosivity, reactivity, or extraction procedure (EP) toxicity, but are not specified as particular compounds.

This report addresses only the specific compounds designated on the List. For these, searches were performed to obtain applicable analytical methods for air, water, and soil/sediment samples at two levels of sophistication: field methods and laboratory methods. Consideration of substances that fall under the purview of RCRA, and consequently CERCLA, due to the characteristics of ignitability, corrosivity, reactivity, or EP toxicity is beyond the scope of this study. This report is not intended to be a critical review and lists only information found. It does not attempt to assume applicability of a technique reported for determination of one compound to the determination of another or for a compound in a sample matrix other than the one for which the method was developed, with or without modification. An effort to provide guidance to the potential components of the F and K Waste Streams, within the framework of the List, is presented in Appendix C. The U.S. EPA Office of Solid Waste is currently studying the composition of many of these process-specific waste streams.

Releases of hazardous substances of concern under CERCLA can vary in nature. Both episodic and certain continuous releases are of concern. two classes can both be divided into well-characterized releases, where the hazardous substances present in the release are known, and poorly characterized releases, where the identities of the substances in the releases are not known. CERCLA requires that specific identification of a compound from the List be made, and the instance of a well-characterized release fits these requirements well. However, poorly characterized releases can present problems with verification of the presence of a CERCLA-designated hazardous substance. Because of problems associated with total analysis of samples to identify and measure all substances present, this report is for the most part aimed at methods for use in the context of well-characterized releases. This is not to say that methods that are suitable for poorly characterized releases are not included; in fact, analytical methods applicable to more than one CERCLA-designated hazardous substance have been put into a separate appendix, Appendix B, to emphasize their potential as screening methods. However, total analysis and characterization of a sample is a long and complex process, which must be designed based on particular sample characteristics, and this document does not provide any guidance for such efforts.

This report divides the compounds of interest under the Act into two groups - inorganic and organic compounds - due to the similarity of analytical methods

used within each group. In some instances, however, classification of a compound as a member of one group or the other is difficult. For example, this problem arises with organometallic compounds (e.g., tetraethyl lead) and some salts (e.g., ammonium picrate). The system used for such classifications in this report has an impact on the organization of the presentation of information and is described later in this report (See "Structure").

The analytical methods located for each compound/matrix combination are classified as one of two types - field methods, for rapid semi-quantitative measurement, and laboratory methods, for more precise and accurate determinations. The distinction between a field method and a laboratory method is nebulous, and could be based on either the quality of the results or on the equipment requirements for the analysis. This report arbitrarily defines field methods as those using portable equipment that does not require line voltage. The use of line voltage from nearby power lines or a gasoline-powered generator for power-intensive equipment such as a gas chromatograph/mass spectrometer (GC/MS) or an inductively coupled atomic plasma spectrometer (ICP) is a situation not considered to be part of a field method in this report. This decision is not a judgment that mobile laboratory responses are ineffective or inappropriate; many examples of rapid response by the U.S. EPA Municipal Environmental Research Laboratory in Edison, New Jersey (MERL-Edison) demonstrate the usefulness of such an approach to on-site analysis.²,³ However, in order to clearly delineate the distinction between field and laboratory methods and to highlight analytical methods that are an alternative to technology-intensive laboratory methods, we have chosen the above definition of a field method.

At the time this study was initiated, it was believed that the use of mobile laboratories was a minor consideration with regard to CERCLA analyses. Mobile laboratory analytical capabilities had been described elsewhere.^{2,3} However, during the two years of compiling this report, the use of mobile laboratories has increased. Therefore, a supplementary survey of the subject was prepared, and a portion of the information from the survey is included in the Results section of this report.⁴

The search for applicable analytical methods began with known sources of CERCLA, other EPA, and standard analytical methods, and continued through computerized searches of the literature. By the time computer-searching was ended, it became apparent that many of the methods located were in the early stages of development. A description of the method-search strategy used is presented later in this report.

The effort to determine the costs of cited analytical procedures relied on two sources of information. For standard environmental analyses, cost figures could be obtained from price lists and bid figures. However, for analytical procedures that are more infrequently encountered, cost figures were not so readily obtained and commercial analytical facilities were directly contacted to obtain a cost estimate. A description of the cost search is presented later in this report.

PURPOSE

The primary goal of this report is to inform the U.S. EPA-OERR of the status of analytical chemistry in the context of releases of Superfund-hazardous substances, in support of rulemaking efforts in the Regulations Branch of that office. In order to satisfy OERR's needs, specific information about analytical methods for each CERCLA-hazardous substance is presented in the Appendices of this report. Less-detailed gross statistical information about the results of our study is given in the main body of the report.

In planning the content and structure of the Appendices, attention was paid to the needs of both OERR and, secondarily, other potential users of the information presented. The Appendices comprise an annotated bibliography of potential analytical methods for Superfund-hazardous substances. As such, they could be useful to EPA managers as a decision-making resource, to EPA scientists and technicians as a source of analytical methods, and to the regulated community as a transfer of current technology.

The depth and scope of the Method Descriptions are intended to satisfy the needs of the EPA OERR. Again, an attempt to make the document useful to others was made, within the constraints of time and budgetary limitations. This document reports the results of a search for analytical methods and does not purport to be a critical review or to make any technical judgment about the analytical methods described or their applicability. In many cases, analytical methods are described that do not specifically identify the particular compound in the specified matrix, but measure hydrolysis or dissociation products. Examples are metallic sodium, sodium methylate, aluminum phosphide, and the phosphorus halides for inorganics, and certain acid halides for organics. Obviously these substances do not exist in an aqueous matrix, and only their hydrolysis products can be expected to be found.

CONTENTS AND USE OF THIS REPORT

CONTENTS

This report is composed of a main body, three Appendices, and an Index. The main body of the report summarizes the contents of the Appendices, gives an overview of the state of the art for analyses for hazardous substances under CERCLA, and highlights problems with the List and analytical chemical technology. The Appendices list analytical methods for substances on the Comprehensive Hazardous Substances List and for the RCRA F and K Waste Streams. dix A and Appendix B are primarily an annotated bibliography of analytical methods, furnishing a reference, a brief description of the reference contents, and cost information for each method. Appendix B describes methods applicable to more than one CERCLA hazardous substance, while Appendix A is a substanceby-substance listing of single-substance methods, or cross-references to Appendix B for each of the six matrix/level-of-sophistication combinations of concern. Appendix A also provides limited safety information and states the current reportable quantity (RQ) for each substance. Appendix C refers the user to parts of Appendix A and Appendix B which could be useful for specific RCRA F and K Waste Streams; these references are not meant to be exhaustive, but comprise the results of a limited investigation of the industrial processes involved. The Index, beginning on page 48 of this volume, gives the consensual name, or the name used in this report, for each regulatory synonym for substances on the Comprehensive Hazardous Substances List.

Since the Comprehensive Hazardous Substances List is a compilation of several individual regulatory lists of chemical species, no single system of nomenclature is consistently used, and there are many compounds for which more than one entry is given. The Index of this report lists each regulatory synonym for the chemical species of interest and specifies the name chosen for use in the Appendices. No single system of nomenclature has been used in the choice of these consensual names, but an attempt has been made to use the regulatory synonym that is the most common and widely accepted. In that way, the use of inverted structures is eschewed (e.g., prefer "1,1,2,2-tetrachloroethane" over "ethane, 1,1,2,2-tetrachloro-") and an attempt is made to avoid use of trivial names unless they are very common. In the case of many pesticides, the formal name is extremely long and convoluted and the trivial or trade name is the consensual name. For example, the name "Aldrin" is chosen over "1,2,3,4,10,10-Hexachloro-4,4a,5,8,8a-hexahydro-1,4:5,6-endo,exo-dimethanonaphthalene". The U.S. EPA Office of Emergency and Remedial Response requested public comment on the type of nomenclature and the number of synonyms to be used in the publication of the List. 1

As mentioned above, the Index to this report lists all of the regulatory synonyms for entries on the Comprehensive Hazardous Substances List, and provides the name used for each throughout the Report and Appendices. The entries in the Index and those in Appendix A are in alphabetical order. The prefixes n-, iso-, tert-, sec-, cis-, trans-, o-, m-, p-, N-, O- and numerals are not considered in alphabetizing, but bis-, di-, hexa-, mono-, penta-, poly-, tetra-, and tri- are used.

In order to use a consistent set of rules in the division into organic and inorganic classifications, several decisions were made. The use of the resulting rigid set of rules is intended to help users know in which part of Appendix A to look for a compound of interest. The rules have been chosen as an editorial choice and not as a scientific judgment. We list all metal-containing compounds as inorganic substances, all non-metal salts of organic acids as organic substances, and alkylated non-metallic inorganics as organic compounds. All carbonates and bicarbonates are considered to be inorganic substances. Under these rules dodecylbenzenesulfonic acid, ammonium benzoate, and methyl hydrazine are considered organic compounds, while calcium dodecylbenzenesulfonate, ammonia, and hydrazine are inorganic. While arguments could be raised over specific assignments of substances as organic or inorganic, these rules for classification were chosen solely for the purpose of systematically organizing the entries in the Appendices.

Reportable Quantity Values and Safety Information

As mentioned above, Appendix A is a substance-by-substance listing of CERCLA-hazardous substances which provides cross-references to Appendix B for multicompound methods, or a Method Description for single-compound methods. The first entry in each listing in Appendix A is the Reportable Quantity (RQ) value of the CERCLA-hazardous substance, as specified in the Federal Register. As discussed in the Background section of this document, each substance is assigned the same RQ value as in the Clean Water Act or, if none is specified for it there, a value of 0.454 kilograms (1 pound). Modifications of some of the resulting RQ values have been made, based on environmental and/or health factors. It is probable that further changes will be made in these values, particularly for those substances that are listed (in the Safety Information section) as potentially carcinogenic, potentially chronically toxic, or both. This is because, after the carcinogenicity and/or chronic toxicity of the substance is evaluated, a decision will be made as to a suitable RQ value. Until then, the statutory value of 0.454 kilograms (1 pound) applies.

Immediately following the RQ value for the CERCLA substance is a section that furnishes limited safety information. Factors about the substance that may be pertinent to field or laboratory personnel safety are listed here, such as if the substance has been shown to exhibit mammalian toxicity at or below certain levels. These levels are: an oral LD50 (dose lethal to 50 percent of the test population) of 10 milligrams per kilogram, a dermal LD50 of 4 milligrams per kilogram, or an inhalation LC50 (concentration lethal to 50 percent of the test population) of 40 parts per million. Where possible the route of entry for the substance has been specified. In addition, chronic toxicity was indicated for any substance which received a chronic toxicity score of 40 or higher in evaluations by the EPA Environmental Criteria and Assessment Office. Substances that

have been identified by EPA as having an appreciable chronic or subacute toxic effect, but that have not been evaluated, are listed as being potentially chronically toxic: those that have been identified as carcinogens, but have not yet been evaluated, are denoted as potentially carcinogenic. Specific hazards associated with a given substance are also mentioned, including reactivity, toxic products of reaction with water, chemical compatibilities, and flash points below 100°F. The major source of this information is a technical background document to support rulemaking pursuant to CERCLA 102(b), prepared by the U.S. EPA, OERR.

Readers may note that a particularly low RQ value is listed for a certain substance, but that no specific safety information is furnished. This situation can arise because the factors used to decide RQ values were not restricted to human (or mammalian) safety, but also included aquatic toxicity and other factors that would not appear in safety information. Additionally, the sources of information used for the compilation of these safety data are not comprehensive and the safety information is not purported to be exhaustive. Therefore, for those substances the toxicity or hazards of which are known to be severe, but for which little or no safety information is otherwise provided, the Safety Section contains a caution to obtain such data from other sources. The following are suggested:

Sittig, M. <u>Handbook of Toxic and Hazardous Chemicals</u>; Noyes Publications: Park Ridge, New Jersey, 1981.

Hazardous Chemicals Data Book, Environmental Health Review No. 4, G. Weiss, Ed.; Noyes Data Corporation: Park Ridge, New Jersey, 1980.

Sax, N. I. Dangerous Properties of Industrial Materials, 5th ed.; Van Nostrand Reinhold: New York, 1979.

The Condensed Chemical Dictionary, 10th ed.; G. G. Hawley, Ed.; Van Nostrand Reinhold: New York, 1981.

Following the Safety Information section of the Appendix A entry, Method Descriptions or cross-references to Appendix B for six matrix/level-of-sophistication combinations are covered.

Method Descriptions

In addition to RQ values and safety information, the compound listings in Appendix A contain descriptions of analytical methods for the CERCLA-hazardous substances. Therefore, Appendix A lends itself readily to compound-specific location of analytical methods. Some methods in Appendix A may be applicable to more than one compound, but are useful for the determination of only one compound specified on the List.

Appendix B is used to find methods applicable to more than one compound on the List. Such analytical methods have more versatility and greater potential for screening of poorly characterized releases. Each Method Description in Appendix B states the compounds from the List to which the method has been demonstrated to be applicable. Some entries on the List are broad, generic

classes of compounds, as "Phthalate Esters" and "Arsenic and Compounds". Currently, no RQ values are being set for such listings. Accordingly, such classifications are not considered in the assessment of capabilities for analysis for CERCLA substances.

The Method Descriptions presented in Appendices A and B contain varying amounts of information in one of two formats. Methods that were classified as Laboratory Methods contain six sections: Method Summary, Interferences, Quality Control, EPA/Technical Status, Reference, and Cost. Field Methods have the same six sections as Laboratory Methods, except that the EPA/Technical Status is replaced by a Sensitivity section.

The Method Summary section of each entry presents the general analytical technique used to inform readers of the equipment and personnel requirements. The section also gives salient method details, such as detector type for gas chromatographic methods and use of internal standards.

The Interferences section lists interferences that are specifically mentioned in the source of the method. Only what is present in the reference is noted in this section.

The Quality Control section gives quality control procedures dictated in the reference and frequently reports that "no procedures are given". This is because a fixed quality control protocol is very case-specific: the level of duplicate, replicate, and fortified samples can vary greatly, depending on the amount of confidence in the analytical results desired, the sample matrix, and other factors. For purposes of this report we follow the standard definition of quality control - those actions followed to ensure a specified quality of the product. 5,6 Therefore, steps given to demonstrate method precision and accuracy are listed in the Quality Control section. A rigid quality control protocol is not actually a part of an analytical method, but part of an overall quality assurance scheme that also includes a statistically valid sampling plan and valid end-use of the analytical results.

The EPA/Technical Status portion of the method description tells the reader whether a method is approved for use under a given EPA regulatory program and whether or not information on the precision and accuracy of the technique is given in the references. This information is provided to allow the reader to see how thoroughly the method has been evaluated. The precision and accuracy information mentioned is a part of method validation or technical review and, since it dictates no actions by the analyst, is not considered to be quality control. Because of variations in the form of such information from source to source and the high degree of dependence of such data on the characteristics of the particular individual sample matrix, no attempt is made to specifically list the precision and accuracy information furnished. Also included in the EPA/Technical Status part is the method detection limit, if provided. The method detection limit is defined as the concentration of analyte in a sample (as opposed to a standard or blank) that yields a detector signal equal to twice the standard deviation of the background noise in the signal of a sample with a concentration close to but above zero. The method detection limit must use information from a large number and wide variety of samples analyzed in many laboratories in order to be valid. The method

detection limit tells the performance of the method with average samples in the presence of normal interferences associated with them. The method detection limit may not be given in the reference(s) being described, but other types of data may be provided. The instrumental detection limit uses a reagent blank in place of the sample(s) used in measurement of the method detection limit. In this way, the instrumental detection limit gives a value for the ability of the instrument and procedure to detect an analyte in the absence of sample-matrix interferences. A third term, sensitivity, is used to indicate the concentration or amount of analyte that elicits a 1-percent (0.044 absorbance units) detector response. This value is a measure of instrument sensitivity only and does not take into account either sample treatment (e.g., extraction, digestion) efficiencies, or sample-matrix interferences. When a limit of detection is provided in the reference, and is defined differently than the above terms, that definition is specified. If a term is not defined in the reference, it is used undefined in the Method Description.

For field methods a Sensitivity section replaces the EPA/Technical Status section because of the lack of official EPA approval of field methods, and the importance of knowing the sensitivity of such methods. Strict use of analytical chemical terminology requires that the term "sensitivity" be used only to indicate the amount or concentration of a substance that will elicit a specified degree of response from an instrument. The use of "sensitivity" for this section of the Method Description is made because the term is easier to intuitively grasp by all of the potential users of this report than the more appropriate term "limit of detection".

The References section identifies the documents that were used to write the Method Description. If a bibliography was used and the primary reference not obtained, the bibliography is mentioned in the References Section and the primary reference is cited (parenthetically) in the Method Summary Section. This was necessary in the case of some foreign-language articles and articles in not easily accessible journals.

Appendix B entries present a list of compounds to which the particular method is applicable, along with their CAS Registry Numbers, before the text of the Method Description. In every instance, care was taken not to add any compounds to those specifically mentioned in the source(s) of the method, regardless of how reasonable that addition might seem. Therefore, it may well be that the lists of compounds preceding many method descriptions could contain more entries. This imposed limitation is expressed in the Appendix B Method Descriptions by including the adjective "above" in the phrase "for analysis of the above compounds". This wording is not meant to rule out the possibility that other subbstances may respond to the procedure. There are several Appendix B Method Descriptions that have the same name, but different matrices and/or lists of candidate compounds. For each, the list of compounds amenable to determination by the method should be checked carefully to be certain that the compound of interest is listed there. Several compounds are also included in more than one list for a given matrix/level-of-sophistication. This results from one of two circumstances: First, Method Descriptions were prepared for all Phase I methods located, regardless of how many times a compound is mentioned. (Phase I methods are from EPA, Federal, or other standard reference sources. See the Report Preparation section of this text for a full discussion of method sources.) Second, for Phase II methods, when a multicompound method was located that was applicable to a compound/matrix/level-of-sophistication combination for which no method had been located previously, that method was included. When such a Method Description was compiled, all CERCLA-hazardous substances to which it had been demonstrated to be applicable were included, whether another method had been located for them or not. In this way, several multicompound methods applicable to the determination of a particular compound in the same sample matrix may be provided, each with a different set of compounds listed to which the method is applicable.

USE OF THIS REPORT

General

This report provides information to the OERR and others at three levels. The overview of the situation given in the Executive Summary may be sufficient for some intended uses. A more detailed discussion is offered in the text. When more detailed and specific information is required than is presented in the text, the user may consult the Appendices. The Appendices can be used to investigate analytical techniques for a single compound, or to assess capabilities for multicompound or screening analyses. Figure 1 is a flow chart that depicts the uses of the Index and Appendices A, B, and C.

Compound-Specific Use

To obtain information on analytical capabilities and costs for a specific chemical or compound, such as would be needed in a release where the specific hazardous substances are known, a stepwise procedure should be followed. First, one should consult the Index, where the name used in the report for that substance is given.

The Index gives the Chemical Abstracts Service (CAS) Registry Number for each substance and tells the consensual name used in the appendices. The CAS Registry Number can be used to ascertain the identity of the compound in question. Armed with the consensual name for the hazardous substance of interest, the user may then locate the listings of analytical methods for that compound in Appendix A.

The user will find six different classifications listed under each compound in Appendix A: field methods for three sample matrices - air, soil/sediment, and water - and laboratory methods for the same three matrices. Under each of the six classifications or matrix/level-of-sophistication combinations, one of three types of entry will be found: a blank space, indicating that no method was located; a Method Description, describing analysis for that compound; or a reference to a multicompound method listed in Appendix B. If the user is referred to Appendix B, the Table of Contents for Appendix B should be consulted to locate the page for the applicable Method Description. Since, in some cases, several Method Descriptions exist with the same title and different matrices and/or lists of analytes that have been demonstrated to be amenable to determination by that method, users should be certain to check the sample matrix and the analyte list above the Method Summary when consulting Appendix B.

USE OF APPENDICES/INDEX

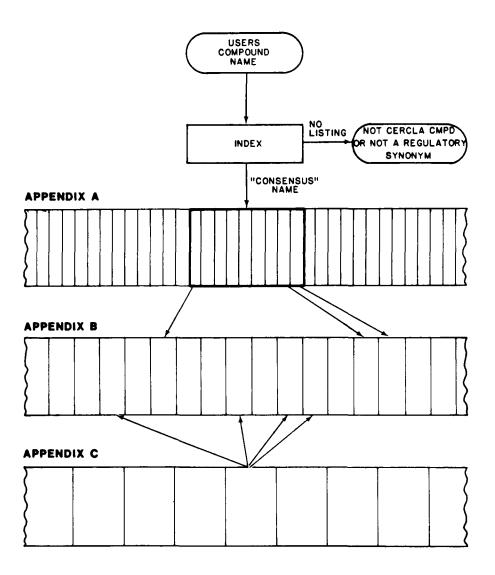


Figure 1. Flow chart depicting use of the Appendices.

Multicompound Use

In instances of poorly characterized releases, measurement of the degree and nature of environmental contamination will require extensive analysis. Analytical techniques that are suited to detection or measurement of more than one hazardous substance are attractive in these situations. Appendix B is a compilation of such analytical methods.

The methods of Appendix B are presented in alphabetical order by the method name within Section A (Inorganic) and B (Organic) of Parts I (Field) and II (Laboratory), and are listed in the Appendix B Table of Contents. The name used for each method attempts to be descriptive of the analytes to which the method is applicable.

The F and K Waste Streams from RCRA legislation are described in Appendix C. For each specific waste stream, substances that are potential components of the waste stream are listed. These listings are not exhaustive, both because they are limited to CERCLA-hazardous substances and because these waste streams are of a variable composition with their chemical components not specified. No particular single analytical technique is specific for these streams. From Appendix C, users may obtain the consensual names for appropriate analytes or the name of a particular multicompound method of interest for a given waste stream.

REPORT PREPARATION

METHOD SEARCH

The location of analytical methods for the substances of interest was performed in two phases, according to a hierarchy of method sources. A hierarchical system of sources of analytical methods was devised so as to use first those sources better demonstrated to be applicable to the CERCLA scenario. In Phase One of the method search, sources were utilized that were expected to yield well-developed methods with demonstrated method quality. Table 1 shows the hierarchy of method sources that comprised the method search. All Phase One methods located have been incorporated in this report. These include methods currently in use in the CERCLA program, such as those in Invitation for Bid documents; 7,8 methods used for the Resource Conservation and Recovery Act (RCRA), 9 the National Pollutant Discharge Elimination System of the Clean Water Act, 10,11 and other EPA programs; other Federal methods [e.g., National Institute for Occupational Safety and Health (NIOSH) methods 12]; and consensual methods [e.g., methods from the American Society for Testing and Materials $(ASTM)^{13,14}$ and the American Public Health Association $(APHA)^{15}$]. (The referenced documents used for Federal Methods are often available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402). If one or more analytical methods for a given substance/matrix/levelof-sophistication were located during Phase One, the search for that specific combination was ended. For substance/matrix/level-of-sophistication combinations still lacking an appropriate method, reference was made to Phase Two sources.

Phase Two of the method search process was composed of searches of sources of methods that are less well established and less tailored to environmental analyses. These sources began with specialized text and literature searches and progressed to contacts with commercial analytical facilities, experts in the field, and equipment manufacturers.

Due to the nature of the Phase One sources, early searches were performed in a more reference-specific manner than later ones. By the time Phase Two began, searches had become compound-specific. The nature of the sources in each phase of the search influenced greatly the information located. For methods from current CERCLA contracts, a complete quality control protocol is furnished, because the methods have reached a production-line status. However, for methods located in the literature, frequently no interferences are reported, no quality control protocol or precision and accuracy data are furnished, and even operating conditions are often not completely described. The degree of acceptance of a given method influences its level of use. When a method is more widely used, it becomes better developed and may eventually become part of a contract, with a rigid analytical and quality control protocol.

TABLE 1. METHOD SOURCES

| 1ABLE 1. | WEIHOD SOURCES |
|-----------------------------------|--|
| Type of Method | Phase One Sources |
| Current CERCLA Methods | Invitation for Bid ⁷ ,8 |
| EPA Methods | Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-8469 |
| | Proposed Modifications to SW-846 (10-17-83) |
| | Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples |
| | Methods for Chemical Analysis of Water and Wastes 10 |
| | Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater 11 |
| | Characterization of Hazardous Waste Sites - A Methods Manual, Vol. 3, "Avail- able Laboratory Analytical Methods" |
| Federal Methods (U.S. and Canada) | (OSHA) NIOSH Manual of Analytical Methods, Vol. $1-7^{12}$ |
| | (OSHA) NIOSH Analytical Methods Standard Completion Program |
| | (USGS) <u>Methods for Analysis of Organic</u> Substances in Water |
| | (EML) EML Procedures Manual |
| | (Alberta Environment) <u>Methods Manual for</u> Chemical Analysis of Atmospheric Pollutants |
| | (Alberta Environment) Methods Manual for Chemical Analysis of Water and Wastes |
| Consensus Methods | (AOAC) AOAC Methods, 13th ed. |
| | (APHA) Standard Methods for the Examination of Water and Wastewater, 14th ed. |
| | (APHA) Methods of Air Sampling and Analysis, 2nd ed. 15 |
| | (continued) |

TABLE 1. (Continued)

| Type of Method | Phase One Sources |
|--|--|
| Consensus Methods | (ASTM) Standards on Chromatography 13 |
| | (ASTM) ASTM Book of Standards, Vols. 11.01, 11.02, 11.03, 11.04, 14.0114 |
| | Phase Two Representative Sources |
| Other Methods | (Specialized References) Sampling and Analysis of Toxic Organics in the Atmosphere, STP-721, 1982. |
| | Handbook of PAH, Marcel-Dekker, 1983. |
| Recent Methods | (Journals, etc.) Analytical Chemistry |
| | Environmental Science and Technology |
| | Management of Uncontrolled Hazardous Waste Sites |
| Incipient Methods | (Manufacturers, Experts) <u>Draeger</u> <u>Detector Tube Handbook</u> |
| ~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~ | Personal Communications. |

Therefore, the source of an analytical method influences the information content of the Method Description provided.

Most of the Phase Two method search was performed by computer, utilizing the Dialog® On-line Information Retrieval Services and the CAS ONLINE® databases. The five Dialog® CA Search files, produced by the Chemical Abstracts Service (CAS) of the American Chemical Society, were used extensively to locate analytical methods for the Interim Report, the starting point for this document. References to compounds on the Comprehensive Hazardous Substances List (the List) were located in these databases by CAS Registry Number (CAS Number). Citations were received by mail (in 7 to 14 days), and the texts of the abstracts (from Chemical Abstracts) were each obtained and evaluated for method suitability. Articles corresponding to abstracts describing application methods were acquired from the University of Nevada, Las Vegas (UNLV) Library, the EPA EMSL-LV Library, or commercial sources. Method Descriptions were prepared from these articles, except in the rare case of foreign-language or unavailable articles, where the abstract from Chemical Abstracts was used.

When the computer search was initiated for the Interim Report, a great amount of information was being sought, and the search strategy reflected that situation, seeking any matrix/level of sophication combination for 144 substances, or 24 percent of the List. For the Final Report, the needed methods become fewer, as did possible references to them, and the amount of irrelevant material obtained increased. Thus, it was necessary to refine the search strategy, as the search progressed.

For the Final Report, most of the search was performed on CAS ONLINE®. The search results were printed online, including abstracts. This procedure saved substantial time, since the abstracts were available for evaluation as soon as the computer session was completed. The search was conducted in specific CA Sections, such as "Organic Analytical Chemistry", "Water," or "Agrochemical Bioregulators," in order to limit inappropriate output. The restriction of the search to about 20 of the 80 CA Sections also allowed a reduction of exclusionary terms in the search statement. These terms can exclude desired as well as undesired information. For example, if a term "not blood" is included in a search statement for a water method, a method suitable for both blood and water analyses will not be recognized. The use of CA Sections in searching can also permit a smaller number of search terms, reducing both the possibility of interferences and the time of the search. For the first part of the Final Report method search, all the CAS numbers of substances for which no methods had been found were entered in the computer and saved. These numbers were entered in subsets of five, so that changes would not require reentering the entire list.

A matrix-specific search statement, specifying English-language articles and the words "air" or "water" or "soil" or "sediment" or "environment," was also saved. Combination of the saved CAS Registry numbers and this search statement was made to locate references and to update search results. A search statement to locate methods without a specified sample matrix was also saved, so that such methods could be located for substances with no method identified for any specific matrix. After this search was completed and results evaluated, another one was initiated to locate methods for substances for which analytical information had been located for some, but not all, matrices. Separate lists were prepared for organic and inorganic substances that were without a method in a particular matrix (air, soil, or water), for a total of six lists. These lists were entered in the computer and saved, along with a search statement tailored for each set of substances. Each search was conducted in the applicable CA Sections. An example of a search is the follow-Organic substances requiring an analytical method for soil were saved in the computer as OSOIL/Q. This list was recalled and combined with the search statement English/LA and (soil? or sediment?) and (anal? or detn or determin? or trace or residu?). The question marks in the statement allow variations in the ends of the terms, such as analysis or analytical, soil or soils. Some of the terms were changed, omitted, or added for searching particular CA Sections.

An additional search was planned that used Dialog® databases other than the CA Search files to locate methods still needed. Accordingly, after the CAS ONLINE® search, the Dialindex™ file, a low-cost index to all of Dialog's® databases, was used to search for methods not found in the CA File. The names of five compounds were entered into the computer along with the numbers of the

eleven files to be scanned, and the computer provided the number of citations for each compound in each of the files. The individual files were then accessed and searched using the compound name and sample matrices of interest. It was found that the productivity of this type of searching was quite low. Because CAS Registry Numbers could not be used in most of these files, only the consensual name was searched, so a valid reference to a nonregulatory synonym would be lost. Also the commands had to be reentered for every file. The search was stopped after the first five substances due to the meager results obtained and the large amount of time expended.

As before, relevant journal articles were obtained from the library of the University of Nevada, Las Vegas (UNLV) and the library of the U.S. EPA Environmental Monitoring Sytems Laboratory (EMSL), Las Vegas. The CAS abstract numbers were used to order copies of hard-to-find articles from the CAS Document Delivery Service.

COST SEARCH

Costs of analyses performed internally vary widely from firm to firm and the figures are not readily available; therefore, the cost information about analytical methods in this report is based on list prices from commercial analytical firms. The price provided for each method is for one sample. An assumption is made that the sample is one of six submitted at the same time, both to minimize the large influence the number of samples has on the cost per analysis, and to partially reflect the fact that multiple samples are to be expected from a release site. A bid price is given as well, when available; it represents a fixed cost for an analysis following a rigid protocol, for a large quantity of samples. A bid price indicates the lowest probable cost for analysis by a particular method, but it also reflects the capacity and projected workload of the laboratory when the bid is made. It should be noted that most commercial laboratories offer discounts of 50 percent or more on many analyses for large numbers of samples.

Costs for common analyses were obtained from commercial price lists. 16 Further information or clarification was provided by specialists in various fields through personal communications (Mr. John Rhodes, Columbia Scientific Industries, Austin, Texas, June 23, 1983 and July 19, 1984; X-ray analysis. Mr. W. Gary Wilson, Environmental Research and Technology, Inc., Concord, Massachusetts, July 6, 1983; air sampling. Dr. Anthony Wong, California Analytical Laboratories, Inc., Sacramento, California, June 22, 1983; dioxin analysis). For methods about which no cost information had been found, a modular pricing structure was developed. A cost was obtained for each step involved in a particular sample preparation and analysis, and the costs were totaled to obtain an overall figure. Most of the modular prices were supplied by Mr. O. John Logsdon, Rocky Mountain Analytical Laboratory, Arvada, Colorado, and Mr. Jack Custer and Mr. Dan Grove, Alert Laboratories, Canton, Ohio; some were found in price brochures. Sources for bid prices were primarily government literature 17 and the Environmental Protection Agency Superfund Sample Management Office (Ms. Linda Haas, EPA Sample Management Office, Viar and Co., personal communication, June 24, 1983, October 25, 1983, and August 8, 1984).

Errors can occur in developing a cost estimate from modules for two reasons. The Method Summary might omit or simplify the description of a procedure, so the cost for the complete procedure may not be represented. Also, the cost of an analysis may not be given in a price list because it is inherently difficult to define - perhaps due to a large number of variables - and these difficulties exist with modular pricing as well.

In a few instances, the cost information for a method description is followed by the notation (est.). For these methods, cost data were incomplete but an estimate was attempted from what was available. When it was not possible to update cost information from the 1983 Interim Report, the notation (1983) follows the cost figure. All the cost information in this section has been documented; however, many of the sources do not wish for their firms to be identified with a specific price quotation. It must be emphasized that the cost information presented in this report should not in any way be considered to constitute a price list.

Factors Influencing Cost Figures

Quantity Considerations--

It has already been noted that increasing the number of samples submitted at one time reduces the cost per sample, within limits. The number of components analyzed per sample, as well as the number of related analyses performed at one time, can also affect the cost.

Gas chromatography (GC), gas chromatography/mass spectrometry (GC/MS), X-ray fluorescence spectrometry, and inductively coupled plasma spectrometry (ICP), among other techniques, can provide information simultaneously about several components in a sample. It is common for laboratories to charge a base price for such analyses, plus an additional charge for each component over a certain number. Therefore, in this report, GC and GC/MS cost information is valid for five or fewer components per sample, and X-ray fluorescence spectrometry for ten components. Pricing structures for analysis by ICP were found both with and without a base price; the system without one is used here because it indicates more clearly the cost for determination of just the component in question.

Another cost consideration is that a laboratory may discount a group of analyses. For example, if the acid extractable and base/neutral extractable fractions of a sample are both analyzed at once, it may cost less than if each fraction were analyzed individually.

Sampling Considerations--

The cost information provided with the method descriptions in this report does not include sampling costs. The hourly fee of \$40.00 to \$70.00 charged by commercial laboratories for field work applies to sampling.

Water and Soil/Sediment Samples—The cost of the actual collection of water or soil/sediment samples is normally small because it does not require much time. Samples for laboratory analysis need appropriate containers for collection and shipment. Sample container kits for RCRA analyses can be purchased from the laboratory that will perform the analysis, or the cost may be

included in the cost of the analysis. The charge for a kit containing two bottles and a cooler, shipped via UPS to the sampling site, is \$35.00 to \$60.00 (list). Small augers and dredges for soil/sediment collection may be included in the cost of the field work; larger ones can be rented.

<u>Air Samples</u>—For air samples, the sampling situation changes. It may take several hours to collect the sample. Also, air sampling is actually part of the sample preparation since the analyte is removed from the sample matrix by filtration, dissolution, adsorption, or absorption as the sample is collected. The costs of air sampling are mostly due to the hourly charges for labor in the field. The fee does not include travel or related expenses, which would differ with each combination of laboratory and field site.

The time required for collecting an air sample depends on the concentration of the analyte and the analytical method to be used. If a four-hour collection time is assumed, the cost for labor alone is \$160.00 to \$280.00. As stated earlier, costs are based on six samples. If all six samples can be taken during four hours, the labor cost per sample for collection time is then \$27.00 to \$47.00.

Another consideration in air sampling costs is the cost of supplies. The cost of chemicals is usually negligible. Use of equipment such as impingers (bubblers) and pumps are generally included in labor charges. The cost of consumable supplies may be added to the sampling cost. These supplies include Tenax® cartridges, about \$10.00 each, and charcoal tubes, about \$1.00 each.

Quality Considerations--

Properties of the sample can affect the cost of analysis by influencing the necessary sample preparation steps. These properties include the sample matrix (air, water, or soil), complexity of the matrix, sample toxicity, and analyte concentration.

The sample matrix greatly influences the sample preparation required. Filters containing particulates from air samples usually undergo dissolution, acid digestion, or ashing. The cost for these procedures may already be included in the cost of the analysis. An X-ray fluorescence determination of metals can be performed directly on the filter with no sample preparation required. When analytes must be desorbed from the sample cartridges; the charge is about \$10.00. Solutions containing gaseous or soluble analytes from air samples are often already in a suitable form for the analytical procedure. Water samples may be ready for analysis as supplied, or may require filtration or a simple extraction. Soil/sediment samples usually need longer extractions or additional preparatory steps. Costs for soil/sediment samples are typically 10-25 percent higher than those for water samples.

The complexity of the sample, referring to the number of potential interferences it contains, also affects sample preparation. The removal of these interferences, or special procedures to avoid them, can add up to 100 percent to the basic cost of the analysis.

Samples containing highly hazardous materials frequently require special handling and use of special equipment or a containment facility, and may increase the cost of analysis by up to 100 percent.

The analysis of substances at concentrations near the limits of detection may call for additional or more sophisticated sample preparation or instrumentation, thus adding to the cost of analysis. For instance, graphite furnace atomic absorption (AA) spectrometry might be used rather than flame AA to analyze a metal concentration. The cost would then increase from approximately \$10.00 to \$20.00 to approximately \$18.00 to \$50.00. Analysis by high resolution mass spectrometry costs about 50 percent more than by low resolution mass spectrometry, mainly because of higher labor costs.

Considerations Relating to Field Analysis--

By far the largest portion of field costs are due to the hourly charge for labor, typically \$40.00 to \$70.00 per hour. Travel and other expenses are additional charges. Colorimetric and other wet chemical analyses take varying amounts of time, depending on the method used. The analysis may call for a period when active work is not performed; for example, time may be required to allow a reaction to occur. If this period is over twenty minutes, it is assumed that analysis is being performed on another sample. With this exception, it is assumed that only one sample at a time can be analyzed in the field. The cost information for wet chemical methods for field use includes a higher degree of estimation than the information provided for other methods. Time estimates for some field methods are based in part on those provided in Wastewater Analysis Handbook, Hach Company, Loveland, CO, April 1978.

An additional cost in field analyses is that of equipment. Some equipment may be supplied by the laboratory; instruments may have to be rented or purchased. The cost information for instrumental field methods includes rental charges, prorated to the time estimated for sample preparation and analysis. The use of rental fees for costs of instrumentation, rather than purchase prices was made because rental costs can be correlated to an individual analysis more readily.

Cost figures for non-routine analyses have been developed by use of a modular pricing system. The costs of the specific sampling (as applicable), sample preparation, and sample analysis steps were added to arrive at an estimated total analytical price. Table 2 gives some examples of the figures used and obtained by such a procedure. For example, laboratory analysis of air samples by HPLC/UV for polycyclic aromatic hydrocarbons requires Soxhlet extraction, solvent exchange and instrumental analysis by HPLC/UV, at costs of \$20-45, \$10-15, and \$100 respectively. The total cost, \$130.00-\$160.00, is the sum of these figures.

Considerations Relating to Laboratory Analysis--

The costs of laboratory analyses are particularly sensitive to two factors: turnaround time and the level of quality control required. A rush request may incur a surcharge on the cost of the analysis of 50 percent. Alternatively, an hourly surcharge may be made; \$75.00 per hour is typical.

TABLE 2. EXAMPLES OF COSTS FOR FREQUENTLY MENTIONED METHODS

Costs of Sampling
(assume 4 hours for air sampling, except for detector tube methods)

Sample Matrix

| A.1 | Time | Instrument | <u>Supplies</u> | <u>Total</u> |
|-------------------|-----------------|------------|-----------------|-----------------|
| Air (Charcoal) | \$27.00 - 47.00 | | \$1.00 | \$28.00 - 48.00 |
| Soil | n* | | n(\$18-30)** | n(\$18-30)** |
| Water | n | | n(\$18-30)** | n(\$18-30)** |

Costs of Laboratory Analyses

| Method | Mat | <u>rix</u> | Preparation | Analysis | Total |
|----------------|--------------|--|--|--------------------|---------------------|
| ICP | soil/se | diment | \$10.00 | \$10.00 \$10.00 | \$20.00 \$10.00 |
| GC/FID HPLC/UV | air e.g.: | bromoform | \$10.00 (desorption) | \$60.00- 80.00 | \$70.00- 90.00 |
| | air e·g·: | polycyclic aromatic hydrocarbons | \$20.00-45.00 (Soxhlet extraction) \$10.00-15.00 (solvent exchange) | \$100.00 | \$130.00- 160.00 |

Costs of Field Analyses

| Method | Matrix | Time | Instrument | <u>Supplies</u> | <u>Total</u> |
|------------------|--------|---------------|------------|-----------------|---------------|
| Detector Tube | air | \$10.00-18.00 | | \$2.00-3.00 | \$12.00-21.00 |
| Test Stick | water | \$3.00-5.00 | | n | \$3.00-5.00 |

^{*} n = negligible

^{**} If the cost of sampling containers is not included in the analytical cost charged by the laboratory, the cost given in parentheses applies.

Quality control is included to some degree in most commercial analyses, and is generally a requirement in government contracts. To determine quality control costs, it is necessary to determine what quality control procedures are included in the cost of the analysis. If more standard, duplicate, or fortified samples are desired, they will be analyzed as additional samples, with commensurate charges. Texts are available that describe sampling plans to ensure a constant level of risk to either the producer or consumer and thus maintain a consistent level of quality in the product (analysis). However, most quality control dictated in the methods located does not use such an approach. 6

RESULTS

BACKGROUND

General

The evaluation of a release of a hazardous substance will include both determination of the occurrence of a release and characterization of the Characterization of the release is the concern in this report and can involve both identification of the hazardous substance involved and measurement of the magnitude and distribution of the release. Identification of the hazardous substances released is very release-specific, and is largely determined by the sample matrix characteristics, the particular substances released, and the degree of knowledge of the nature of the released materials. and specific identification of hazardous substances of unknown origin in a complex mixture released into a difficult matrix can be beyond the capabilities of modern analytical technology. This is due to limitations in time, database, In addition, in the instance of a compound that reacts with or and technology. dissociates in water, only analysis for the products of the process is possible, and such an analysis can never specifically identify the compound from which the analyte(s) came. Such element-specific analyses give the best information that can be obtained, but are not specific methods for individual compounds. If it is known that a strong, exothermic reaction will occur when the substance of interest contacts water, and that only decomposition products will be present in the water sample, the phrase "reacts with water" is placed under the Appendix A water sample headings. The Appendix B methods that apply to such compounds are, accordingly, only used to determine reaction products; to avoid any presumption that analysis is for a substance never actually present in its original chemical state, the Method Description describes the analysis as for ions, not for compounds. The substances in question may react with moisutre in water or soil as well.

Measurement of the distribution of contamination from a release involves collection and analysis of air, water, and soil/sediment samples in the vicinity of the release. Sample collection requires physical collection of a representative sample, while sample analysis involves two phases: sample preparation and analyte determination. The sample preparation process involves removal of potential interferences and transfer of the analyte into a state amenable to the chosen method of analysis, and the determinative process then involves the ultimate analysis of a suitably prepared sample. A weakness in any of these three steps will result in a faulty measurement. Sample collection methods and their sophistication frequently depend upon the matrix being sampled - air, soil, or water - and, to a lesser extent, upon the compounds of interest. Sample preparation procedures, such as digestion, extraction, or hot-water leaching, depend upon both the sample matrix and the determinative

method(s) to be used, while the choice of a determinative method is largely dictated by the analyte, and is frequently the same for all three sample matrices. For many air analyses, the sample collection step and part of the sample preparation process are combined in that the analyte is removed from the air sample matrix into a solution or onto a stationary sorbent. Because of the dependence of each section of the overall measurement process on these different factors, consideration is made of the factors involved in sampling each type of sample matrix, in sample preparation, and in determination of the compounds of interest before specific discussion begins.

Sampling

Sampling of air differs substantially from sampling of water or soil. With air sampling devices, substances in two physical states can be collected particulates and gases. Collection of particulates involves mechanically trapping particles on a filter medium with pores smaller than the size of the particles. This procedure is straightforward and does not involve rapid diffusion, chemisorption, or other physical/ chemical processes. When gases are to be collected from air, the compound of interest must have a significant vapor pressure to be collected at any appreciable distance from the source of the vapors. Non-volatile hazardous substances, such as inorganic salts, metals, and organic compounds like dioxin, have negligible vapor pressures. Such compounds exist as particulate substances in air only if very finely divided. Low-volatility substances would slowly bleed into the atmosphere and the sampling system. In such a steady-state situation, collection of a cumulative sample is ideal. Cumulative sampling utilizes the accumulation of an analyte on some stationary phase, and is usually used in air sampling, due to the large sample volumes which must be processed in order to obtain a reasonable amount of analyte. For substances of high vapor pressure, such as hydrogen fluoride, a release would have to be slow and steady to create a situation amenable to composite sampling. If a rapid discharge of such a substance occurred, any composite sample taken would only be a dilution of an instantaneous sample. Due to the open nature of the atmospheric system, rapid releases of highly volatile compounds could be dissipated before sampling begins. The well-mixed, open-system characteristics of the atmosphere create special problems in sampling. The variability in chemical properties and vapor pressures of the approximately 617 hazardous substances under the purview of CERCLA requires several techniques for sampling from air.

Many organic compounds and a few inorganic substances have sufficiently high vapor pressures to be sampled as vapors. Such sampling usually involves passage of air past a stationary solid phase, or through a liquid. The analyte is thus either adsorbed on the solid or chemically fixed by the liquid. Removal of the analyte from the solid or liquid can then be carried out by standard techniques for analysis of solid or liquid samples.

Water bodies can be considered to be closed systems in the short term, and are frequently not well mixed. When a water body is not well mixed, sampling is not straightforward, particularly in the instance of insoluble or immiscible substances. The difficulty associated with sampling in such an instance is due to problems associated with obtaining a representative sample that truly reflects the condition of the water body. Due to the vastly smaller volumes of

water that are collected in comparison to air samples, sample collection times are greatly decreased. Since the system is essentially closed, the only compounds that will not be collected when in contact with a body of water will be those that are insoluble, forming a separate phase or layer, and those that degrade in water to become different substances.

Soil and sediment can also be considered to approximate a closed system that is not well mixed. Due to the virtual lack of mixing, variations between samples are expected to be large, and the contaminated soil near a release will not be homogeneous. Since smaller volumes of soil than air are needed for sampling, it can be sampled more quickly and easily. For sampling of both soil and water, the sample is usually placed in a container for subsequent sample preparation steps, in contrast to air sampling, which is done by removal of the analyte from the (air) sample matrix. In some instances, soil sampling may not be appropriate. For example, the release of a gas with a density less than that of air would usually not be expected to contaminate nearby soil.

Sample Preparation

Sample preparation techniques usually involve the elimination of potentially interfering substances from the sample. For example, this is done by total oxidation of organic matter in the sample in the case of metals analyses, or by column cleanup to eliminate certain interferences in organic analyses. In addition, sample preparation can include modification of the analyte to make it amenable to the determinative steps to follow. For example, metals in water samples are oxidized so that all of the metal present is in the same oxidation state, and certain organic compounds are often esterified to make them suitable for gas chromatography. These techniques will depend on both the analyte of interest and on the sample matrix, because the analyte must not be destroyed, and because the potential interferences which must be removed are specific for particular sample matrices.

Analyte Determination

The determinative procedures used on a suitably prepared sample are usually dictated by the identity of the analyte. This is because a characteristic of the analyte must ultimately be measured. In organic compounds this is often a characteristic imparted by the presence of a certain functional group, and, less frequently, the presence of a certain element such as nitrogen or phosphorus. These characteristics may be measured by an appropriate detector after chromatographic separation. On the other hand, in metal-containing compounds, use is usually made of the high sensitivity and specificity possible in determination of the metal content of samples by atomic spectrometry. The place of element-specific inorganic analyses in the CERCLA program is discussed in the sub-section of this report "Inorganic Listings - General Considerations."

TECHNICAL RESULTS

This report gives Method Descriptions for 749 analytical procedures applicable to CERCLA hazardous substances. Field methods comprise 167 of the methods, and 582 laboratory methods are described. The cost of laboratory analyses ranges from approximately \$15.00 to \$300.00 per sample in lots of six,

and that of field analyses is from about \$5.00 to \$40.00 per sample in lots of six. Sampling costs are not included in the costs given for laboratory analyses, due to their extremely variable nature.

Of the 582 analytical procedures classified as laboratory methods, 153 cite an EPA-published reference, 178 describe a National Institute for Occupational Safety and Health (NIOSH) or Occupational Safety and Health Administration (OSHA) method, and 251 cite non-NIOSH/OSHA non-EPA publications. NIOSH/OSHA references are singled out here because of the large amount of work done by the Department of Health, Education, and Welfare (later Department of Health and Human Services) in air analytical technology. It is also appropriate to point out that these methods are intended for use in work place applications, where high levels of known target compounds are anticipated in the absence of comparable levels of interfering compounds.

Table 3 shows the numbers of substances for which single- or multi-compound methods were located. Single-compound methods for laboratory (field) analysis were located for 155(67), 13(3), and 22(12) CERCLA-hazardous substances in air, soil, and water samples, respectively. For the multicompound methods, 326(214), 438(57), and 507(263) compounds had methods located for air, soil, and water, respectively, in the laboratory (field).

TABLE 3. SUBSTANCES FOR WHICH SINGLE- OR MULTICOMPOUND METHODS WERE LOCATED

| Type of Analysis | Number of Substances | | | | |
|------------------|--|--|--|--|--|
| | Single-Compound (Appendix A) Methods | Multicompound (Appendix B) Methods | | | |
| Laboratory - | | | | | |
| Air | 155 | 326 (99)* | | | |
| Soil/Sediment | 13 | 438 (1)* | | | |
| Water | 22 | 507 (8)* | | | |
| Field - | | | | | |
| Air | 67 | 214 (24)* | | | |
| Soil/Sediment | 3 | 57 | | | |
| Water | 12 | 263 (1)* | | | |

^{* ()} Number of additional compounds with a single-compound method in Appendix A as well as a multicompound method.

If all 6 compound/matrix/level-of-sophistication combinations were located for each inorganic compound, 1242 would be compiled. The actual number found was 942, or 75 percent of the total. For organic compounds, 2460 entries are possible, and 1134, or 46 percent of the total possible, were located. The overall percentage of methods located was 57 percent. Table 4 shows the number and percent of total possible compound methods located for field and laboratory levels of sophistication for all three sample matrices. These figures do not include duplicate compound/matrix/level-of-sophistication entries. No methods were located for any matrix/level-of-sophistication for 31 organic substances; a method was found for at least one matrix/level-of-sophistication for each inorganic substance.

TABLE 4. NUMBER OF SUBSTANCES FOR WHICH METHODS WERE LOCATED

| Substances | Number for Which Method(s) Located | Percent of Total Possible | |
|-----------------|---------------------------------------|------------------------------|--|
| Inorganic (207) | | | |
| Field | | | |
| Air | 133 | 64 | |
| Soil/Sediment | 45 | 22 | |
| Water | 168 | 81 | |
| Laboratory | | | |
| Air | 203 | 98 | |
| Soil/Sediment | 190 | 92 | |
| Water | 203 | 98 | |
| Organics (410) | | | |
| Field | | | |
| Air | 137 | 33 | |
| Soil/Sediment | 15 | 4 | |
| Water | 107 | 26 | |
| Laboratory | | | |
| Air | 278 | 68 | |
| Soil/Sediment | 261 | 64 | |
| Water | 336 | 82 | |

Organic Listings

General Considerations--

Organic compounds are characterized by the presence of carbon-carbon bonds, which are nonpolar. Many organic compounds have similar physical and chemical properties, due to structural similarities. Minor differences between them exist due to the presence or absence of various functional groups, but in general their solubilities, reactivity, and miscibility are similar. Since these compounds are so numerous and many are so similar, detection by functional group is possible, but compound-specific detection or measurement is very difficult. Currently, mass spectrometry and Fourier transform infrared spectrometry are the only detection/measurement methods with which as organic compounds can usually be identified at trace levels; however, both of these techniques require separation of the compound of interest from other compounds present in the sample. Usually, this separation is performed by gas chromatography, a technique which requires that the analyte of interest be sufficiently volatile and separable from other compounds in the sample. Novel methods of sample introduction are being explored to attempt to make high performance liquid chromatography (HPLC) a suitable separation step preceding mass spectrometry. This would obviate the requirement that exists with gas chromatography that the analyte by volatile or semi-volatile. Modifications of the HPLC apparatus to encompass microbore columns are also being explored. Mass spectrometry frequently cannot differentiate between isomers, while Fourier transform infrared spectrometry does not allow the analyst to differentiate between homologs, or compounds that differ only in the length of a hydrocarbon chain.

In addition, for mass spectrometry to furnish completely unequivocal compound identification, the mass spectrometer should be a high resolution instrument, and certified pure compounds must be available or the database used by the instrument computer must include a reference library of mass spectra that contains information on the target compounds. The EPA/NIH database of mass spectral data contains data for 38,971 compounds and their mass spectra, and is usually furnished by the manufacturer when mass spectrometers are purchased. However, of these compounds, only 348 are of concern under CERCLA. In order to generate such information, the compounds of interest must be available in pure form. The U.S. EPA Quality Assurance Reference Materials Project (QARMP) has the responsibility of making such materials and their standard solutions available to EPA and EPA-contractor laboratories. Of the 410 organic compounds on the Comprehensive Hazardous Substances List, most should be amenable to mass spectrometric analysis for identification purposes. However, many are currently unavailable through the QARMP.

The use of Fourier transform infrared spectrometry for environmental analyses is still in the developmental stage. Unlike gas chromatography/mass spectrometry (GC/MS), gas chromatography/Fourier transform infrared spectrometry (GC/FTIR) has not reached the stage of common use for environmental analyses. A study by the U.S. EPA of the technique has been performed, and it shows promise when used in conjunction with GC/MS. However, use of the technique is currently limited by the size and quality of the database of reference gas phase infrared spectra available.

At higher concentrations than trace levels, more compound-specific methods are available including nuclear magnetic resonance (NMR), (non-Fourier transform) infrared spectrometry, and X-ray diffraction. However, for all of these methods a nearly pure sample is required and, in analysis of environmental samples, the presence of additional compounds could preclude their use. In addition, no articles, books or other sources of information located mentioned use of these methods in the CERCLA scenario. This report deals only with techniques that have been shown to be somewhat applicable to use in analysis of environmental samples; analysis of concentrated neat samples (e.g., drum or tank residues) is not within the scope of this study.

Without compound-specific detection, analysis must rely even more heavily upon separation procedures, to isolate the compounds of interest from other interfering compounds present so that the signal from a non-specific detector can be attributed to a single compound. This is particularly true for low concentrations of analyte. Since the signal of an analytical instrument usually results from monitoring a property that many organic compounds possess, the analyte of interest must be removed from potentially interfering compounds. Both gas chromatography and liquid chromatography are used extensively for this purpose.

Other spectral techniques can provide less extensive information regarding compound identity, but can be very useful as complementary techniques to each other and/or to GC/MS. 18,19 For example, ultraviolet-visible (uv-vis) spectrometry and fluorescence spectrometry yield information about electronic distribution in a molecule, and can be quite valuable as ancillary techniques to mass spectrometry. In addition, confirmation of tentative compound identification can be accomplished by use of techniques including chemical derivatization, use of dual GC columns or dual semi-specific GC detectors, thin layer chromatography (TLC), and others.

Air samples--

In the context of releases of CERCLA-hazardous substances into the air, measurement of a wide range of concentrations, from 'ambient' levels to 'source' levels, can be required. Ambient air is a complex and dynamic mixture with very low levels of contaminants resulting in many potential physical/chemical interactions. Airborne inorganic compounds can function as oxidants, water can induce hydrolysis reactions, solid particles can act as catalytic surfaces for reactions, and sunlight can promote photochemical transformations. In addition, organic species in air are difficult to sample and analyze because there are a large number of compounds present in ambient air that can interfere with measurements of typically low levels of other organic compounds. 'Source' analytical procedures, as opposed to those for ambient air, are intended for high levels of single, known contaminants without equivalently high levels of interfering compounds.

Field monitoring for hazardous substances in air probably began with the use of canaries in the mining industry. More recently, the Occupational Safety and Health Administration (OSHA) has provided an impetus for development of portable methods for measurement of levels of organic vapors in the workplace. Semi-specific detector tubes, through which a known volume of air is pumped,

are available for 78 of the 410 organic compounds on the Comprehensive Hazardous Substances List. Table 5 lists the organic compounds on the List for which gas detector tubes are available. In all, 137 of 410 possible methods were located for field analysis of organics in air.

TABLE 5. ORGANIC COMPOUNDS FOR WHICH GAS DETECTOR TUBES ARE AVAILABLE

| ======================================= | | |
|---|----------------------------|---------------------------|
| Acetaldehyde | Cyclohexanone | Methylene Chloride |
| Acetic Acid | 1,2-Dichlorobenzene | Methyl Ethyl Ketone |
| Acetic Anhydride | 1,3-Dichlorobenzene | Methyl Isobutyl Ketone |
| Acetone | l,4-Dichlorobenzene | Methyl Mercaptan |
| Acrolein | l,1-Dichloroethylene | Methyl Methacrylate |
| Acrylonitrile | 1,2-trans-Dichloroethylene | Monoethylamine |
| Allyl Chloride | 1,2-Dichloropropane | Monomethylamine |
| Amyl Acetate | Dichlorvos | Propionic Acid |
| Aniline | Diethylamine | Propylene Oxide |
| Aziridine | Dimethylamine | 1,2-Propyleneimine |
| Benzene | 1,1-Dimethylhydrazine | Pyridine |
| Benzyl Chloride | l,2-Dimethylhydrazine | 1,1,2,2-Tetrachloroethane |
| Butyl Acetate | Dimethyl Sulfate | Tetrachloroethylene |
| iso-Butyl Acetate | 1,4-Dioxane | Tetrahydrofuran |
| Butylamine | Epichlorohydrin | Toluene |
| iso-Butyl Alcohol | Ethyl Acetate | Toluene Diisocyanate |
| n-Butyl Alcohol | Ethyl Acrylate | 1,2,1-Trichloroethane |
| Carbon Disulfide | Ethylbenzene | Trichloroethylene |
| Chlorobenzene | Ethylenediamine | Triethylamine |
| Chloroform | Ethylene Oxide | Trimethylamine |
| Cresol | Ethyl Ether | Vinyl Acetate |
| <u>m</u> -Creso1 | Formaldehyde | Vinyl Chloride |
| o-Cresol | Formic Acid | Xylene |
| p-Cresol | Furfural | <u>m</u> -Xylene |
| Cumene | Methacrylonitrile | o-Xylene |
| Cyclohexane | Methyl Bromide | <u>p</u> -Xylene |

For quantitative analysis of organic compounds in air, sampling and sample preparation procedures become critical. The major obstacle in the development of these procedures is the identification of a sampling process and medium which, when combined, give quantitative recovery of the analyte from the air volume sampled. The Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina, has demonstrated acceptable methods for the determination of 43 volatile and semi-volatile organic compounds in air. If the recovery of the analyte from the sampling medium is not quantitative, questionable analytical results will be obtained (Mr. Steve Bromberg, Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina, personal communication, 19 September 1982). However, partial recovery does not rule out these media for qualitative identification. Laboratory procedures for the determination of 278 of 410 organic compounds in air were located.

The limitations of laboratory determinations of organic compounds in air are very closely related to the weaknesses in sampling and sample preparation steps mentioned above. For the vast majority of organic compounds on the List, no sampling medium/preparation has been devised that has been shown to give quantitative absorption and desorption of analyte. Once an efficient process has been demonstrated for both removal of the analyte from air onto or into a sampling medium and for quantitative recovery from that medium, standard organic determinative methods will usually be applicable. Development of such a sampling process is difficult for environmental, or low-level, samples since laborious cleanup procedures to control blank problems are often required for current solid-phase collection systems.²⁰ However, for many compounds, such sampling methods could be devised and validated readily, with some research and development work. One alternative method to current sampling procedures is to concentrate organic gases into liquids at low temperature, obviating unwanted reactions and artifacts. 20 The most notable problem associated with sampling air is that the large volumes of air needed for sampling in such instances can often exceed the breakthrough volume for a given analyte and sampling medium, which is the volume of air that will move the analyte completely through the sampling system. Other problems associated with the sampling process are irreversible sorption, vaporization, and potential decomposition or reaction of the analyte caused by other atmospheric constituents during sampling. 20

The limitations of field analytical techniques for the determination of organics in air are related to the similarity in physical/chemical properties of these substances. Since field equipment usually does not have the sophisticated chromatographic or detection/measurement capabilities of laboratory equipment, it frequently cannot differentiate between similar organic compounds. For this reason, most of the field methods located for organic compounds in air detect/measure more than one compound without differentiating between them.

Field-portable gas chromatographs, which can differentiate between compounds, are available commercially with a wide variety of detectors and have been used in emergency and remedial response actions. 21 In addition, field-portable infrared spectrometers are available and have been evaluated for field use in spill responses. 22

Water Samples--

Without the use of sophisticated laboratory instrumentation, field measurement of organic compounds in water is not a well-developed technology. Due to the much greater concentration of contaminants in and higher chemical activity of water compared to air and to the complications posed by the presence of more molecules/ions in solution, matrix interferences are much more prevalent in water than in air. Because of the level of sophistication needed to separate and detect or measure organics, the development of field methods for these compounds has only recently been pursued.²³ Thin-layer chromatographic (TLC) methods have been devised that are amenable to field use²⁴ and successful use of portable gas chromatographs²¹ has been demonstrated for a few compounds, but the major thrust in field analysis for such analytes has been in the development of mobile laboratories.³ Some progress could probably be made in the development and testing of methods to remove the analyte from the problematic water matrix and to utilize existing technology for actual analysis.²⁵

In addition, gas chromatographic static-headspace analytical methods, which offer a large savings in analysis time compared to dynamic-headspace or purge-and-trap methods, 26 , 27 could be useful for initial site-characterization efforts. Microextraction analytical techniques, 28 , 29 which minimize the time spent in sample preparation, could be valuable methods in conjunction with field-portable instruments. In all, 107 organic compounds of 410 can be detected or determined in water samples by the field methods located in the appendices of this report.

Laboratory methods for the determination of organic substances in water use separation and detection/measurement techniques which are more sophisticated than those usually used for field analyses. Analytical methods for water samples have been developed due to both the emphasis on water pollution, through the Clean Water Act, and the fact that the water matrix presents less of a sampling problem than air. Currently, laboratory analysis for organics in water in the CERCLA Contractor Laboratory Program is carried out for approximately 130 compounds. Methods for the measurement of 336 of 410 organics were located for water samples at the laboratory level of sophistication. Without the use of GC/MS, unequivocal identification for the majority of compounds would be almost impossible, and complementary identification data would have to be obtained by other methods. Future progress should be expected in the development of a combined GC/MS-GC/FTIR instrument, which could utilize the strengths of each of these powerful techniques. 10

The major limitation for field methods is the lack of sophistication in available separation and detection methods. However, by development of novel sample preparation procedures, use could be made of existing field technologies, like high-performance thin layer chromatography (HPTLC), paper chromatography, and other techniques. ²⁰ Demonstration of applicability of various portable instruments to field use and/or use of air field methods with modified sample preparation techniques could expand field capabilities. On the other hand, the technology-intensive laboratory is limited by reference data: the certified standards, the pure materials needed to obtain reference mass spectra, and computer data-handling capabilities. Progress on these fronts could come from the EPA QARMP, the EPA/NIH mass spectral database, the GIFTS commercial FTIR database, ³⁰ and from instrument manufacturers.

Soil/Sediment Samples--

Field methods for only 15 organic compounds in soil/sediment samples were located. This is probably at least partially due to the complexity and variability of the soil/sediment matrix, and the commensurate difficulties in removal of interferences during sample preparation. However, methods for laboratory analysis of soil/sediment samples exist, and the sample preparation steps from these should usually be suitable for field use in combination with existing field methods.

The technology for laboratory analysis of soil/sediment samples is the same as that for analysis of water samples, except that the procedures used to isolate the analyte from the matrix usually consist of a liquid-solid extraction step to dissolve the analyte in a liquid phase. The soil/sediment matrix is more complex and variable than the water matrix, so that in most instances additional cleanup steps are required for soil/sediment sample extracts.

However, the determinative procedures carried out on a prepared sample are the same. The same 130 compounds currently measured in water samples under CERCLA are also measured in soil/sediment samples. Methods for 261 of 410 organics in soil/sediment samples at the laboratory level of sophistication were located.

Inorganic Listings

General Considerations--

The List contains 207 entries which are classified as inorganic substances in this report. Several of these compounds are organometallic compounds or metal salts of organic acids, and a sizeable part of the molecule is organic in nature. However, frequently the metallic part of the compound is the more readily identified and measured one.

Laboratory methods for inorganic compounds can use sophisticated laboratory instrumentation to obtain better precision, accuracy, limits of detection and certainty of identification of various ions than field methods provide. Unfortunately, identification and measurement of a given ion often does not satisfy the requirement of CERCLA that a particular CERCLA-hazardous substance be identified. However, the Comprehensive Hazardous Substances List contains 14 inorganic species, with all compounds containing that moiety included. this time, no reportable quantity is being assigned to such listings, although, if one were assigned, a significant diminution of the technological burden of analysis would be removed. This is because many of the inorganic entries on the Comprehensive Hazardous Substances List are salts that will dissociate or hydrolyze in water or can undergo oxidation/reduction, acid/base, or complex formation reactions quite readily 31 in aqueous solution or in soil/sediment. In such an instance, the CERCLA-hazardous substance is transformed into another chemical species, the history of which cannot be known. Several of the molecules and/or ions which are parts of CERCLA-hazardous substances can be indigenous to the sample collection area (e.g., sodium and phosphate in water, iron and sulfides in soil/sediment).

Air Samples--

Field methods for the determination of inorganics in air are limited. Methods were located for 133 of the 207 inorganics on the List. One limitation is that many of these inorganic compounds are salts, with negligible vapor pressures. Such compounds would be present in air only as particulates, and such contaminations of air should be short-lived. The Occupational Safety and Health Administration (OSHA) has been a major force in the development of portable sensing devices. These are directed at monitoring workplace air, to ensure worker safety. The U.S. EPA Municipal Environmental Research Laboratory in Edison, New Jersey, has not yet performed extensive air monitoring for inorganics, except for analysis for hydrogen cyanide, hydrogen sulfide, and ammonia for personnel safety. It has been suggested that humans smell the vapors emanating from the ground at release sites, in order to gauge the concentration of the vapors. 32 This method does not seem acceptable and, hopefully, development of responsible field methods for gases will continue. For 17 of the 207 inorganic entries on the Comprehensive Hazardous Substances List. gas detector tubes are available (see Table 6). However, several of these actually detect hydrolysis products of salts and not the hazardous substance itself. Field air analysis capabilities for particulate inorganic compounds

TABLE 6. INORGANIC SUBSTANCES FOR WHICH GAS DETECTOR TUBES ARE AVAILABLE

| | :52==================================== | ======================================= |
|-------------------|---|---|
| Ammonia | Hydrogen Cyanide | Nitrogen Dioxide |
| Chlorine | Hydrogen Fluoride | Pheno1 |
| Cyanogen Bromide | Hydrogen Sulfide | Phosgene |
| Cyanogen Chloride | Mercury | Phosphine |
| Hydrazine | Nickel Tetracarbonyl | Potassium Cyanide |
| Hydrochloric Acid | Nitric Acid | Sodium Cyanide |
| | | |

are mostly X-ray fluorescence methods, which are element-specific and do not identify the particular compound containing the metal detected. Table 7 lists the substances that are determined by field X-ray fluorescence. Use of the same sampling procedures as for laboratory air analyses, with minor modifications of the sample preparation procedures used, combined with the use of developed field methods for the analysis of water samples could give more specific information about the prepared sample in the field air analyses.

TABLE 7. ELEMENTS DETERMINED
BY X-RAY FLUORESCENCE

| Analyte | | Number of Compounds |
|-----------|-------|---------------------|
| Silicon | | 1 |
| Antimony | | 7 |
| Barium | | 1 |
| Cadmium | | 4 |
| Calcium | | 7 |
| Chromium | | 14 |
| Copper | | 10 |
| Iron | | 10 |
| Lead | | 14 |
| Nickel | | 7 |
| Manganese | | 1 |
| Selenium | | 7 |
| Zinc | | 16 |
| | Total | 99 |

The National Institute for Occupational Safety and Health has developed and validated a large number of analytical methods for inorganic compounds in air. These are mainly element-specific methods, and they have been validated over a specified concentration range. Laboratory methods that are applicable to at least one portion of inorganic salts and compounds in air have been located for 203 of the 207 inorganic substances on the Comprehensive Hazardous Substances List. These methods do not give any information at all about the identity of the compound containing the element determined.

Many field and laboratory analytical methods for inorganic substances in air lack specificity. Differentiation between valence states is, in the case of chromium, possible but difficult, while unequivocal identification of the particular metal-containing compound in a sample is beyond the reach of current technology. X-ray diffraction methods could be applicable to such situations, but no references citing demonstration of applicability were located in this study. Field analytical methods for particulates in air are especially weak, and validation of sampling/sample preparation procedures to allow use of field water analysis determinative technology for air samples would improve this situation greatly.

Water Samples--

Field analysis for inorganic substances in water are the best developed of any of the field methods considered in this report. Methods were located for 168 of the 207 substances of interest. A major impetus for the development of this technology came from the National Pollutant Discharge Elimination System, and the need for field effluent testing it created. Analytical methods are readily developed for inorganics in water, since water is an ideal solvent for most inorganic substances. Methods for salts typically focus on one of the constituents of the salt, so that methods frequently do not identify the particular compound present. Field kits exist for most heavy metal ions and, therefore, for their compounds, for ammonia and ammonium salts, and for cyanides, inorganic acids, and fluorides. An EPA study found that a kit put together from commercially available field test modules could detect most of the inorganics listed in Federal Register, Vol. 40, No. 250, December, 1975. This study relied very heavily upon highly non-specific methods like conductivity. A field kit has been made available based on the results of this study, and it is claimed to detect "over 300 compounds" in waterways. 33 CERCLA requires specific identification of substances from the List, so that non-specific measurements are of limited use under the Act. However, such methods are useful in field analyses, in order to gauge the extent of migration of a release and thereby to plan a thorough and efficient sampling plan. Field kits, including test papers, are available for the substances listed in Table 8.

Laboratory analytical methods for water samples have been located for all but 9 of the 207 inorganic entries on the Comprehensive Hazardous Substances List. However, most of these methods are applicable to only one portion of the inorganic compound and are therefore of limited utility under CERCLA, since they do not specifically identify a CERCLA compound. Table 9 lists the inorganic species for which water and soil/sediment samples are analyzed in the current CERCLA program.⁸

Soil/Sediment Samples--

Field methods for 45 of the 207 inorganic substances on the Comprehensive Hazardous Substances List have been located for the soil/sediment matrix. Unfortunately, most of the methods located were elemental methods, which detect or measure one element present in the compound.

TABLE 8. ANALYTES THAT CAN BE DETERMINED IN WATER SAMPLES USING TEST KITS

| ======================================= | |
|---|---------------------|
| Analyte | Number of Compounds |
| Aluminum | 2 |
| Aluminum (P) | 2 |
| Ammonia | 31 |
| Antimony (P) | 7 |
| Cadmium | 4 |
| Chromates | 6 |
| Chromium(VI) | 12 |
| Chromium (P) | 19 |
| Chromium | 14 |
| Copper | 10 |
| Copper (P) | 10 |
| Fluorides and others | 17 |
| Halides, cyanides, and others* | 44 |
| Hydrazines* | 3 |
| Lead | 14 |
| Lead (P) | 14 |
| Mercury | 7 |
| Nickel ions (P) | 8 |
| Nitrate ions | 14 |
| Potassium (P) | 10 |
| Selenium | 7 |
| Silver | 4 |
| Sulfite ions (P) | 3 |
| Zinc (P) | 16 |
| Zinc | 16 |
| Zirconium | 4 |
| Total | 298 |
| by field kit: | |
| by test paper | |

(P) Test Paper method

Analytical capabilities for inorganics in soil/sediment at the laboratory level of sophistication are nearly as well developed as for water, with 190 of the possible 207 entries accounted for. Table 9 shows the inorganic species for which analysis of water and soil/sediment samples are performed under current CERCLA contracts. As with determinations of inorganic substances in air and water samples, specific identification of compounds is difficult to impossible.

^{* 10} of the 47 compounds measured by these tests are organics

TABLE 9. INORGANICS MEASURED IN WATER AND SOIL SAMPLES UNDER CERCLA

| ======================================= | **===================================== | ======================================= | |
|---|---|---|---|
| Aluminum | Cadmium | Manganese | Tin |
| Antimony | Chromium | Mercury | • Vanadium |
| Arsenic | Cobalt | Nickel | Zinc |
| Barium | Copper | Selenium | Ammonia |
| Beryllium | Iron | Silver | Cyanide |
| Boron | Lead | Thallium | Sulfide |
| | ======================================= | | ======================================= |

FINANCIAL RESULTS

Specific cost information is included as part of each Method Description in the appendices. Average costs for analysis by each method are shown in Table 10. Cost averages for miscellaneous instrumental methods are not included, because the cost figures have a wide range and the average is not very meaningful. For the same reasons, overall cost averages have not been compiled. Laboratory instrumental analysis for organic compounds is substantially more expensive than other types of analysis. Readers should keep in mind that prices for analysis can vary widely, depending upon current economic conditions, laboratory equipment and specialty, and other factors. For that reason, the contents of this report should not be considered a rigid price list.

TABLE 10. METHOD- AND COST-SEARCH RESULTS

| Laboratory Methods | Non-NIOSH Non-EPA | NIOSH | EPA | Average Cost |
|----------------------------|----------------------|------------------|------------|--------------|
| AA or ICP | 3 | 13 | 75 | \$ 20.00 |
| X-Ray fluorescence | 17 | 1 | - | 32.00 |
| Colorimetry | 31 | 26 | 14 | 35.00 |
| IR | 60 | - | - | 70.00 |
| Wet methods | 6 | 2 | - | 25.00 |
| Miscellaneous instrumental | 30 | 13 | 5 | |
| GC | 83 | 107 | 37 | 90.00 |
| GC/MS | 7 | 1 | 10 | 285.00 |
| HPLC | 13 | 15 | 11 | 130.00 |
| Other | 1 | _ | 1 | |
| TOTA | $L \overline{251}$ | $\overline{178}$ | <u>153</u> | |

| Field Methods | Number of Methods | Average Cost |
|------------------------------|-------------------|--------------|
| X-Ray fluorescence | 19 | \$32.00 |
| Test sticks/indicator papers | 18 | 4.50 |
| Wet methods/colorimetry | 48 | 20.00 |
| Miscellaneous instrumental | 10 | |
| Detector tubes | 64 | _ 16.50 |
| TOTAL | 159 | |
| | | |

MOBILE LABORATORY SURVEY RESULTS

Instrumentation

The appendices of this report describe laboratory and field analyses, but the definition of neither classification incorporates the special cases of analyses by mobile laboratories. Therefore, a study was undertaken to explore this topic. The following information is from the resulting report.

A commercial mobile laboratory is typically a vehicle with appropriate support systems and a selection of sophisticated instruments that varies with the needs of a particular situation. Table 11 is a list of instruments used in selected commercial mobile laboratories. Gas chromatographs in mobile laboratories employ one or more of a variety of detectors: flame ionization detector (FID), thermal conductivity detector (TCD), photoionization detector (PID), electron-capture detector (ECD), flame photometric detector (FPD), and Hall electrolytic conductivity detector (HECD). Brands and models of gas chromatograph identified as having been used in commercial mobile laboratories are the Hewlett-Packard HP5840, Perkin-Elmer P-E 3920, HNU, and Tracor Instruments Inc. Model 560. Many smaller instruments and pieces of equipment, such as the Miran® IR or bomb calorimeter, are also used routinely. The U.S. Coast Guard has successfully used a Fourier transform infrared spectrometer (FTIR) in a mobile laboratory in response to an environmental incident.

A Canadian firm, Sciex Ltd., has developed instruments with mass spectrometric detector systems that can sample air directly, and has begun manufacturing a mobile laboratory equipped with them. The mobile laboratory is available with either a TAGA™ 3000 GC/MS or a TAGA™ 6000 (GC) MS/MS system. Demonstrated uses of TAGA™ systems include the following: tracing the chlorine from a train derailment; determining PCB's in cement kiln stack gas, ambient air, and soil; air monitoring at landfills for 20 compounds and in the workplace for bis-(chloromethyl)ether; analyzing hazardous waste barrel contents; direct soil surface sniffing; hazardous waste emission monitoring; and continuous on-line monitoring of combustion gases and automobile engine exhausts. A4,35 Direct MS/MS analysis cannot be used alone when certain interferences are present. In such instances, flash gas chromatography is used before MS/MS as a gross cleanup step. The ionization sources for the TAGA™ 6000 are not completely effective in all situations, but the instrument has significant applications in many instances.

Procedures

The U.S. EPA Municipal Environmental Research Laboratory, Oil and Hazardous Materials Spills Branch, Edison, New Jersey (MERL-Edison or MERL-OHMSB) has pioneered EPA efforts to provide rapid on-site mobile laboratory analytical capabilities. Table 12 lists past MERL-Edison mobile laboratory responses and the principal analytes measured at each site.

In order to process the potentially large number of samples expected at release sites and/or to obtain timely results, many of MERL-Edison's mobile laboratory analytical procedures attempt to save time by modifying traditional sample workup procedures (e.g., APHA, ASTM, EPA) and/or through use of more

TABLE 11. INSTRUMENTS USED IN SELECTED COMMERCIAL MOBILE LABORATORIES

| Firm* | GC | GC/MS | AA | HPLC | voc | TOX | TOC | X-Ray Fluor- escence | Chemi- lumin- escence NOX Analyzer | UV/VIS Spec- trometer |
|--------|----|------------------------|----|-----------------|-----|-----|-----|----------------------------|--|-----------------------------|
| Alert | х | | x | | x | - | x | ** | | |
| EAL | x | | | | x | | | x | x | |
| ES | x | | | | x | | x | | | x |
| ESE | x | | x | x | | x | x | | x | |
| GCA | x | | | | | | | | | |
| IT | x | \mathbf{x}^{\dagger} | x | x | x | x | x | | x | x |
| ОНМ | x | \mathbf{x}^{\dagger} | x | x ^{††} | x | x | x | | | x |
| Radian | x | | x | x | | | | | | |
| RTS | x | | x | x | | | | | | |
| SCA | x | | x | | | x | x | | | |

* = Acronyms refer to the following companies:

EAL = EAL Corporation, Richmond, California.

ES = Engineering-Science, Arcadia, California.

ESE = Environmental Science and Engineering, Inc., Gainesville, Florida.

GCA = GCA Corporation, Bedford, Massachusetts.

IT = IT Corporation, Wilmington, California.

OHM = 0. H. Materials, Findlay, Ohio.

RTS = Resource Technology Service, Inc., Devon, Pennsylvania.

SCA = SCA Waste Chemical Co., Inc., Cheektowaga, New York.

** = Will be used in a pending job.

t = Finnigan OWA GC/MS.

tt = Usage has been limited, but capability is present.

rapid instrumental determinative steps. The procedures at MERL-Edison are designed to minimize losses in precision and accuracy, to use less space, and to generate less waste.

Traditional methods developed for lower levels of contamination in environmental matrices are often tedious and time consuming. Such procedures frequently involve extraction, evaporative concentration, chromatographic cleanup,

TABLE 12. MERL-EDISON MOBILE LABORATORY RESPONSES AND ANALYTES

| Location | Date | Analyte(s) | | |
|-------------------------|-----------|----------------------------------|--|--|
| Haverford, Pennsylvania | Nov. 1976 | Pentachlorophenol | | |
| Dittmer, Missouri | Apr. 1977 | PCB's | | |
| Oswego, New York | Oct. 1977 | PCB's | | |
| Niagara, New York | Oct. 1978 | Chlorinated benzenes, BHC, PCB's | | |
| Elizabeth, New Jersey | May 1979 | PCB's | | |
| Pittston, Pennsylvania | Aug. 1979 | o-Dichlorobenzene | | |
| Crosby, Texas | Feb. 1980 | PCB's | | |
| Moira, New York | Oct. 1980 | PCB's | | |
| Seymour, Indiana | Nov. 1980 | Priority pollutants | | |
| Kingston, New Hampshire | Jan. 1981 | PCB's, pesticides | | |
| La Marque, Texas | Sep. 1981 | Purgeable compounds | | |

and other steps to remove interferences and concentrate the analyte in order to obtain a reliable instrumental response for the analyte of interest. In spill responses, however, the analyte-to-interference concentration ratio is much higher, and the sample preparation procedure is usually not so critical. In addition, the identity of the spilled material is often known, so that use of more rapid and direct sample preparation procedures and analytical methods is possible. For example, MERL-Edison has developed procedures for organics with a rapid extraction step, such as simply spinning the sample with the extraction solvent $^{37-42}$ For certain situations, the extraction step has been completely omitted 43 Streamlined cleanup techniques and use of positive displacement micropipets to eliminate multiple dilutions of samples and standards have also been adopted 39 , 40

MERL-Edison has not yet been requested to perform analyses with the results to be used in litigation but has always concentrated on site characterization and the monitoring of remedial response efforts. However, standard analytical procedures are performed in the mobile laboratory when it is located in Edison, New Jersey. This indicates that the laboratory is capable of generating high-quality analytical data that can be used in litigation activities, provided support systems equivalent to those in Edison are available on site. It is expected that this situation applies to all mobile laboratories now in use.

MERL-Edison is currently assembling a manual of their tested analytical protocols for mobile-laboratory use that will be applicable to approximately 240 of the CERCLA hazardous substances in air, water, or soil. Table 13 lists those substances on the CERCLA Comprehensive Hazardous Substances List that MERL-Edison has measured in the field with a mobile laboratory, and for which protocols are being prepared. The instrumentation required to perform the analyses for the substances listed in Table 13 include GC/MS, GC/ECD or GC/HECD, GC/NPD, GC/FID, spectrofluorometers, infrared spectrometers, emission spectrometers, carbon analyzers, inductively coupled atomic plasma emission

TABLE 13. HAZARDOUS COMPOUNDS ANALYZED BY THE MERL-EDISON MOBILE LABORATORY

| Compound Name | CAS No. | Compound Name | CAS No. |
|----------------------------|---------------------------|----------------------------|------------|
| Acenaphthene | 83-32-9 | 2-Chloronaphthalene | 91-58-7 |
| Acenaphthylene | 208-96-8 | Chlorophenol (all Isomers) | |
| Acrolein | 107-02-8 | 2-Chlorophenol | 95-57-8 |
| Aldrin | 309-00-2 | Chromium (and | 7440-47-3 |
| Aluminum Sulfate | 10043-01-3 | Compounds) | |
| Aniline | 62-53-3 | Chrysene | 218-01-9 |
| Anthracene | 120-12-7 | Copper (and Compounds) | 7440-50-8 |
| Antimony | 7440-36-0 | Cresol | 1319-77-3 |
| Aroclor 1016 | 12674-11-2 | Cyclohexane | 110-82-7 |
| Aroclor 1221 | 11104-28-2 | DDD | 72-54-8 |
| Aroclor 1232 | 11141-16-5 | DDE | 72-55-9 |
| Aroclor 1242 | 53469-21-9 | DDT (and Metabolites) | 50-29-3 |
| Aroclor 1248 | 12672-29-6 | Dibenz[a,h]anthracene | 53-70-3 |
| Aroclor 1254 | 11097-69-1 | Dibenz[a,i]pyrene | 189-55-9 |
| Aroclor 1260 | 11096-82-5 | Dichlorobenzene | 25321-22-6 |
| Arsenic | 7440-38-2 | (Mixed) | |
| Benzal Chloride | 98-87-3 | 1,2-Dichlorobenzene | 95-50-1 |
| Benzene | 71-43-2 | 1,3-Dichlorobenzene | 541-73-1 |
| Benzidine | 92-87-5 | 1,4-Dichlorobenzene | 106-46-7 |
| Benzo[a]anthracene | 56-55-3 | Dichlorobromomethane | 75-27-4 |
| Benzo[a]pyrene | 50-32-8 | Dichloroethane | 1300-21-6 |
| Benzo[b]fluoranthene | 205-99-2 | 1,1-Dichloroethane | 75-34-3 |
| Benzo[k]fluoranthene | 207-08-9 | 1,2-Dichloroethane | 107-06-2 |
| Benzo[ghi]perylene | 191-24-2 | Dichloroethylene (all | 25323-30-2 |
| Benzoic Acid | 65-85-0 | Isomers) | 23323 30 2 |
| Benzonitrile | 100-47-0 | 1,1-Dichloroethylene | 75-35-4 |
| Benzotrichloride | 98-07-7 | 1,2-Dichloroethylene | 156-60-5 |
| Benzyl Chloride | 100-44-7 | 2,4-Dichlorophenol | 120-83-2 |
| Beryllium (and | 7440-41-7 | 2,6-Dichlorophenol | 87-65-0 |
| Compounds) | , 110 12 , | Dichloropropane (all | 26638-19-7 |
| α-BHC | 319-84-6 | Isomers) | 20030 17 7 |
| β-BHC | 319-85-7 | 1,2-Dichloropropane | 78-87-5 |
| Bromoform | 75-25-2 | Dieldrin | 60-57-1 |
| Cadmium (and | 7440-43-9 | Diethyl Phthalate | 84-66-2 |
| Compounds) | 7440 13 7 | 2,4-Dimethylphenol | 105-67-9 |
| Carbon Tetrachloride | 56-23-5 | Dimethyl Phthalate | 131-11-3 |
| Chlordane | 57 - 74 - 9 | Di-n-butyl Phthalate | 84-74-2 |
| Chlorobenzene | 108-90-7 | Dinitrobenzene (all | 25154-54-5 |
| Chloroethane | 75-00-3 | Isomers) | 27174-74-7 |
| Chloroform | 67-66-3 | 4,6-Dinitro-o-cresol | 534-52-1 |
| p-Chloro- <u>m</u> -cresol | 59-50 - 7 | Dinitrophenol (all | 25550-58-7 |
| F QUIDIO TE CLESOI | ,, <u>,</u> , , | Isomers) | 47770-70-7 |
| | | • | E1 20 E |
| | | 2,4-Dinitrophenol | 51-28-5 |

(continued)

TABLE 13. (Continued)

| | CAS No. | Compound Name | CAS No. |
|--------------------------|------------|---------------------------|------------|
| Dinitrotoluene (all | 25321-14-6 | Parathion | 56-38-2 |
| Isomers) | | Pentachlorobenzene | 608-93-5 |
| 2,4-Dinitrotoluene | 121-14-2 | Pentachloroethane | 76-01-7 |
| 2,6-Dinitrotoluene | 606-20-2 | Pentachloronitrobenzene | 82-68-8 |
| Endosulfan | 115-29-7 | Pentachlorophenol | 87-86-5 |
| Endrin (and Metabolites) | 72-20-8 | Phenanthrene | 85-01-8 |
| Ethylacetate | 141-78-6 | Phenol | 108-95-2 |
| Ethylbenzene | 100-41-4 | Polynuclear Aromatic | |
| Ethylene Dibromide | 106-93-4 | Hydrocarbons | |
| Fluoranthene | 206-44-0 | Pyrene | 129-00-0 |
| Fluorene | 86-73-7 | Pyridine | 110-86-1 |
| Halomethanes | | Quinoline | 91-22-5 |
| Heptachlor | 76-44-8 | Resorcinol | 108-46-3 |
| Heptachlor Epoxide | 1024-57-3 | Selenium (and Compounds) | 7782-49-2 |
| Hexachlorobenzene | 118-74-1 | Silver (and Compounds) | 7440-22-4 |
| Hexachlorobutadiene | 87-68-3 | Sodium | 7440-23-5 |
| Hexachloroethane | 67-72-1 | Sodium Hydroxide | 1310-73-2 |
| Kepone | 143-50-0 | 1,2,4,5-Tetrachlorobenzen | |
| Lead Acetate | 301-04-2 | 1,1,1,2-Tetrachloroethane | |
| Lead (and Compounds) | 7439-92-1 | 1,1,2,2-Tetrachloroethane | |
| Lindane | 58-89-9 | 2,3,4,6-Tetrachlorophenol | |
| Malathion | 121-75-5 | Thallium (and Compounds) | 7440-28-0 |
| Mercury (and Compounds) | 7439-97-6 | Toluene | 108-88-3 |
| Methanol | 67-56-1 | Toxaphene | 8001-35-2 |
| Methoxychlor | 72-43-5 | 1,2,4-Trichlorobenzene | 120-82-1 |
| Methyl Bromide | 74-83-9 | 1,1,1-Trichloroethane | 71-55-6 |
| Methyl Chloride | 74-87-3 | 1,1,2-Trichloroethane | 79-00-5 |
| Methyl Parathion | 298-00-0 | Trichloroethylene | 79-01-6 |
| Methylene Bromide | 74-95-3 | Trichloromonofluoro- | 75-69-4 |
| Methylene Chloride | 75-09-2 | methane | |
| Naphthalene | 91-20-3 | Trichlorophenol | 25167-82-2 |
| Nickel (and Compounds) | 7440-02-0 | 2,4,5-Trichlorophenol | 95-95-4 |
| Nitrobenzene | 98-95-3 | 2,4,6-Trichlorophenol | 88-06-2 |
| Nitrophenol (all | 25154-55-6 | sym-Trinitrobenzene | 99-35-4 |
| Isomers) | | Xylene | 1330-20-7 |
| 2-Nitrophenol | 88-75-5 | Xyleno1 | 1300-71-6 |
| 4-Nitrophenol | 100-02-7 | Zinc (and Compounds) | 7440-66-6 |
| Nitrotoluene | 1321-12-6 | , | |

spectrometers (ICP), and atomic absorption spectrometers (AA). In addition to providing field operation guidance for the instruments listed, each protocol provides guidance on sample isolation, fractionation, screening, and the use of appropriate safety devices.

The MERL OHMSB mobile laboratory has developed specific quality assurance protocols for use in mobile laboratory responses. Several QA/QC protocols are designed to generate method validation data concurrently with sample results in order to give the on-site coordinator real-time feedback on analytical quality. Use of a single standard to define the linear dynamic calibration range for a large number of compounds eliminates the need for extensive preanalysis instrument calibration. A series of complementary QA protocols that describe the preparation of synthetic samples for method validation have been prepared. These make use of aqueous, non-aqueous, or sediment media that resemble the site samples and include detailed instructions for fortification of samples with water-soluble, partially water-soluble, water-insoluble, and volatile materials.

Several commercial firms have designed their own mobile laboratory sampling, sample work-up, and analytical methods, but most firms use procedures designed for standard laboratory analyses, or make minor modifications. Typically, the same quality assurance procedures that are employed in the main laboratory are employed in the field units.

Cost Factors

According to one firm that provides both mobile and fixed-site laboratory services, the first two factors considered in setting a price for a mobile laboratory activation are how rapid a response is required and how many samples per day will be processed. If the mobile laboratory support effort is of sufficient magnitude, analytical costs may be lower than those incurred at a fixedsite laboratory. For example, an on-site compatibility screening (PCB, oxidant, reductant, cyanide, and sulfide) of up to 200 samples per day was performed at a cost of approximately \$15.00 per sample. Such large numbers of samples can help distribute the capital costs of mobilization. A 2-day job would have prohibitive mobilization costs, while a stay of 1 to 2 weeks could be cost -effective. If laboratory trailers were located in several areas of the country, mobilization costs could be greatly reduced. For nearly all on-site analytical programs, costs are determined on the basis of outfitting a mobile laboratory with men and equipment. Normally, for the use of a mobile laboratory, a fixed daily rate is charged that includes glassware, extractors, and miscellaneous supplies. Any reagents or supplies that must be expended are charged. Major equipment such as a gas chromatograph is charged at a daily rate with discounts according to the duration of the job. Besides the probability of lower charges when a sufficient number of samples is processed per day, an added benefit of on-site analysis is quick turnaround time that may significantly lower the costs of other contractors on the site.

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Index and Table of Contents for Appendix A

CAUTION

The following information is presented in each volume containing the appendices to the report. These facts are presented to ensure that the information presented in the appendices is clearly defined and that what is not intended to be included is outlined.

The Method Descriptions presented in Appendix A and Appendix B are not intended to be critical reviews of analytical methods, but comprise an annotated bibliography. The Method Descriptions are only a brief summary of the information contained in the reference(s) being described. Efforts were purposely made to avoid speculation about applications of methods to analytes or sample matrices not documented by a reference. There is an Index/Table of Contents which tells the name for each substance used throughout the report. See the text of the report for a detailed description of the contents of the Method Descriptions and for the reasons for preparation of the appendices and report.

The cost figures furnished for each method are presented following the Method Descriptions, and should not be used as a price list. For a discussion of the factors involved in figuring the cost figures cited, see the text of the report.

Use of this Index

This Index contains each regulatory synonym for specific hazardous substances designated under the Federal Water Pollution Control Act (Clean Water Act), the Clean Air Act, and the Resource Conservation and Recovery Act. To the right of each entry is the Chemical Abstracts Service (CAS) Registry Number, to aid in unequivocal identification of the substance. To the right of the CAS number is the consensual name, or the synonym chosen for use throughout this report, followed by the page in Appendix A where method descriptions and/or references to Appendix B can be found.

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| o-Dichlorobenzene | 95-50-1 | 1,2-Dichlorobenzene | A-50 |
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| Dodecylbenzenesulfonic Acid | 27176-87-0 | | A-60 |
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| Ethyl Acrylate | 100-41-4 | | A-628 |
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| Ethylene Dichloride | 107-06-2 | 1,2-Dichloroethane | A-51 |
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| Ferric Dextran | 9004-66-4 | | A-108 |
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| Ferric Nitrate | 10421-48-4 | | A-110 |
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| 1,2,3,4,10,10-Hexachloro-6,7-epoxy- 1,4,4a,5,6,7,8,8a-octahydro-endo,exo 1,4:5,8-dimethanonaphthalene | 60-57-1 - | Dieldrin | A-54 1 |
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| 1,2,3,4,10,10-Hexachloro-1,4,4a,5, 8,8a-hexahydro-1,4:5,8-endo,exo- dimethanonaphthalene | 309-00-2 | Aldrin | A-324 |

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| N-Nitrosodiethylamine | 55-18-5 | | A-792 |
| N-Nitrosodimethylamine | 62-75-9 | | A-793 |
| N-Nitrosodiphenylamine | 86-30-6 | | A-795 |
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| Phenol, 2,6-Chloro- | 87-65-0 | 2,6-Dichlorophenol | A-528 |
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| Potassium Permanganate | 7722-64-7 | | A-206 |
| Potassium Silver Cyanide | 506-61-6 | | A-212 |
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| Propanal, 2-Methyl-2-(methylthio)- O-[(methylamino) carbonyl]oxime | 116-06-3 | Aldicarb | A-323 |
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| 1-Propanamine, N-Propyl- | 142-84-7 | Dipropylamine | A-597 |
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| Propanenitrile | 107-12-0 | | A-837 |
| Propanenitrile, 3-Chloro- | 542-76-7 | 3-Chloropropionitrile | A-456 |
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| 1,2,3-Propanetriol, Trinitrate 1-Propanol, 2,3-Dibromo-, Phosphate (3:1) | 55-63-0 126-72-7 | Nitroglycerine Tris(2,3-dibromopropyl) Phosphate | A-781 A-941 |
| 1-Propanol, 2-Methyl- | 78-83-1 | iso-Butyl Alcohol | A-4 05 |
| 2-Propagone | 67-64-1 | Acetone | A-298 |
| 2-Propanone, 1-Bromo- | 598-31-2 | Bromoacetone | A-394 |
| Propargite | 2312-35-8 | Pt Omogcerotte | A-839 |
| Propargyl Alcohol | 107-91-7 | 2-Propyn-1-ol | A-846 |
| 2-Propenal | 107-02-8 | Acrolein | A-311 |
| 2-Propenamide | 79-06-1 | Acrylamide | A-315 |
| Propene, 1,3-Dichloro- | 542-75-6 | 1,3-Dichloropropene | A-536 |
| 1-Propene, 1,1,2,3,3,3-Hexachloro- | 1888-71-7 | Hexachloropropene | A-686 |
| 2-Propenenitrile | 107-13-1 | Acrylonitrile | A-318 |
| 2-Propenenitrile, 2-Methyl- | 126-98-7 | Methacrylonitrile | A-710 |
| 2-Propenoic Acid | 79-10-7 | Acrylic Acid | A-316 |
| 2-Propenoic Acid; Ethyl Ester | 140-88-5 | Ethyl Acrylate | A-625 |
| 2-Propenoic Acid, 2-Methyl-, Ethyl Ester | 97-63-2 | Ethyl Methacrylate | A-651 |
| 2-Propenoic Acid, 2-Methyl-, Methyl Ester | 80-62-6 | Methyl Methacrylate | A-751 |
| 2-Propen-1-o1 | 107-18-6 | Allyl Alcohol | A-326 |
| Propionic Acid | 79-09-4 | | A-840 |
| Propionic Acid, 2-(2,4,5-Trichloro- phenoxy)- | 93-72-1 | 2,4,5-TP Acid | A-914 |
| Propionic Anhydride | 123-62-6 | | A-841 |
| <u>n</u> -Propylamine | 107-10-8 | | A-842 |
| Propylene Dichloride | 78-87-5 | 1,2-Dichloropropane | A-530 |
| Propylene Oxide | 75-56-9 | | A-843 |
| 1,2-Propylenimine | 75-55-8 | 2-Methylaziridine | A-718 |
| 2-Propyn-1-o1 | 107-19-7 | | A-846 |
| Pyrene | 129-00-0 | | A-847 |
| Pyrethrins | 121-29-9 | | A −848 |
| 4-Pyridinamine | 121-21-1 504-24-5 | 4-Aminopyridine | A-332 |

| 110-86-1 91-80-5 100-75-4 109-06-8 54-11-5 | Methapyrilene N-Nitrosopiperidine 2-Picoline | A-85 |
|--|---|--|
| 91-80-5 100-75-4 109-06-8 | N-Nitrosopiperidine | A-71 |
| 109-06-8 | | |
| | 2-Picoline | A-80 |
| 54-11-5 | | A-83 |
| | Nicotine and Salts | A-77 |
| 56-0402 | Methylthiouracil | A -75 |
| 107-49-3 | Tetraethyl Pyrophosphate | A-88 |
| 930-55-2 | N-Nitrosopyrrolidine | A-80 |
| 91-22-5 | | A-85 |
| 50-55-5 | | A-85 |
| 108-46-3 | | A-85 |
| 81-07-2 | | A-85 |
| 94-59-7 | | A-86 |
| 7783-00-8 | | A-21 |
| 7782-49-2 | | A-21 |
| 7446-08-4 | | A-21 |
| 7488-56-4 | | A-21 |
| 7446-08-4 | Selenium Dioxide | A-21 |
| 630-10-4 | | A-21 |
| 115-02 -6 | Azaserine | A-35 |
| 7440-22-4 | | A-21 |
| 506 - 64-9 | | A-21 |
| 7761-88-8 | | A-22 |
| 93-72-1 | 2,4,5-TP Ac1d | A-91 |
| | | A-22 |
| 7631-89-2 | | A-22 |
| 7784-46-5 | | A-22 |
| 26628-22-8 | | A-22 |
| 10588-01-9 | Sodium Dichromate | A-23 |
| | | A-22 |
| | | A-23 |
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| 10361-89-4 | | |
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| | 107-49-3 930-55-2 91-22-5 50-55-5 108-46-3 81-07-2 94-59-7 7783-00-8 7782-49-2 7446-08-4 630-10-4 115-02-6 7440-22-4 506-64-9 7761-88-8 93-72-1 7440-23-5 7631-89-2 7784-46-5 26628-22-8 10588-01-9 1333-83-1 7631-90-5 7775-11-3 143-33-9 10588-01-9 25155-30-0 7681-49-4 16721-80-5 1310-73-2 7681-52-9 10022-70-5 124-41-4 7632-00-0 7558-79-4 10039-32-4 10028-24-7 10140-65-5 7601-54-9 7785-84-4 10101-89-0 | 107-49-3 930-55-2 91-22-5 50-55-5 108-46-3 81-07-2 94-59-7 7783-00-8 7782-49-2 7446-08-4 7488-56-4 7446-08-4 630-10-4 115-02-6 7440-22-4 506-64-9 7761-88-8 93-72-1 7440-23-5 7631-89-2 7784-46-5 26628-22-8 10588-01-9 1333-83-1 7631-90-5 7775-11-3 143-33-9 10588-01-9 25155-30-0 7681-49-4 16721-80-5 1310-73-2 7681-52-9 10022-70-5 124-41-4 7632-00-0 7558-79-4 10039-32-4 10028-24-7 10140-65-5 7601-54-9 7785-64-4 10101-89-0 10361-89-4 7758-29-4 |

| Hazardous Substance | CAS No. | Consensual Name | Page |
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| Sodium Selenite | 10102-18-8 | | A-24 |
| | 7782-82-3 | | |
| 4,4'-Stilbenediol, a,a-Diethyl | 56-53-1 | Diethylstilbestrol | A-55 |
| Streptozotocin | 18883-66-4 | | A-86 |
| Strontium Chromate | 7789-06-2 | | A-24 |
| Strontium Sulfide | 1314-96-1 57-24-9 | Commobadae and Color | A-24 |
| Strychnidin-10-one, and Salts Strychnidin-10-one, 2,3-Dimethoxy- | 357-57 - 3 | Strychnine and Salts Brucine | A-86 A-39 |
| Strychnine and Salts | 57-24-9 | prucine | A-86 |
| Styrene | 100-42-5 | | A-86 |
| Sulfuric Acid | 7664-93-9 | | A-24 |
| Sulfuric Acid, Dimethyl Ester | 77-78-1 | Dimethyl Sulfate | A-57 |
| Sulfur Hydride | 7783-06-4 | Hydrogen Sulfide | A-13 |
| Sulfur Monochloride | 12771-08-3 | | A-24 |
| Sulfur Phosphide | 1314-80-3 | Phosphorus Pentasulfide | A-19 |
| Sulfur Selenide | 7488-56-4 | Selenium Disulfide | A-21 |
| Sulfuric Acid, Thallium(I) Salt | 7446-18-6 | Thallium(I) Sulfate | A-25 |
| 2,4,5-T | 93-76-5 | 2,4,5-T Acid | A-86 |
| 2,4,5-T Acid | 93-76-5 | | A-86 |
| ,4,5-T Amines | 2008-46-0 | | A-86 |
| 2,4,5-T Esters | 93-79-8 | | A-87 |
| | 2545-59-7 | | |
| | 61792-07-2 | | |
| | 1928-47-8 | | |
| DE | 25168-15-4 72-54-8 | DDD | |
| .2,4,5-Tetrachlorobenzene | 95-94-3 | טטט | A-47 |
| 2,3,7,8-Tetrachlorodibenzo- p-dioxin(TCDD) | 1746-01-6 | | A-87 A-87 |
| .1,1,2-Tetrachloroethane | 630-20-6 | | A-87 |
| ,1,2,2-Tetrachloroethane | 79-34-5 | | A-87 |
| etrachloroethylene | 127-18-4 | | A-87 |
| ,3,4,6-Tetrachlorophenol | 58-90-2 | | A-88 |
| etraethyldithiopyrophosphate | 3689-24-5 | | A-88 |
| etraethyl Lead | 78-00-2 | | A-25 |
| etraethyl Pyrophosphate | 107-49-3 | | A-88 |
| etrahydrofuran | 109-99-9 | | A-88 |
| etranitromethane | 509-14-8 | | A-89 |
| etraphosphoric Acid, Hexaethyl Ester | 757-58-4 | Hexaethyl Tetraphosphate | A-68 |
| hallic Oxide | 1314-32-5 | | A-25 |
| hallium | 7440-28-0 | | A-25 |
| hallium(I) Acetate | 563-68-8 | | A-25 |
| hallium(I) Carbonate | 6533-73-9 | | A-25 |
| hallium(I) Chloride hallium(I) Nitrate | 7791-12-0 | | A-25 |
| hallium(II) Oxide | 10102-45-1 | W-114- A-41- | A-25 |
| hallium(III) Oxide hallium(I) Selenide | 1314-32-5 | Thallic Oxide | A-25 |
| hallium(I) Sulfate | 12039-52-0 7446-18-6 | | A-25 |
| hioacetamide | 62-55-5 | | A-25 |
| hiofanox | 39196-18-4 | | A-89 A-89 |
| hioimidodicarbonic Diamide | 541-53-7 | 2,4-Dithiobiuret | A-60 |
| hiomethanol | 74-93-1 | Methylmercaptan | A-74 |
| hiophenol | 108-98-5 | merny imercaptan | A-89 |
| Thiosemicarbazide | 79-19-6 | | A-89 |
| hiourea | 62-56-6 | | A-89 |

| Hazardous Substance | CAS No. | Consensual Name | Page |
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| Thiourea, (2-Chlorophenyl)- | 5344-82-1 | 1-(o-Chlorophenyl)thiourea | A-455 |
| Thioures, 1-Naphthalenyl- | 86-88-4 | a-Naphthylthiourea | A-773 |
| Thiourea, Phenyl- | 103-85-5 | N-Phenylthiourea | A-823 |
| Chiram | | • | A-899 |
| Coluene | 108-88-3 | | A-901 |
| Coluenediamine | 95-80-7 | | A-904 |
| Toluene Diisocyanate | 584-84-9 | | A-906 |
| -Toluidine Hydrochloride | 636-21-5 | | A-910 |
| Toxaphene | 8001-35-2 | | A-911 |
| 2,4,5-TP Acid | 93-72-1 | | A- 914 |
| 2,4,5-TP Acid Esters | 32534-95-5 | | A-915 |
| H-1,2,4-Triazo1-3-amine | 61 - 82-5 | Amitrole | A-333 |
| Trichlorfon | 52-68-6 | | A-916 |
| l,2,4-Trichlorobenzene | 120-82-1 | | A-917 |
| l,l,l-Trichloroethane | 71-55-6 | | A-918 |
| 1,1,2-Trichlorethane | 79-00-5 | | A-921 |
| Trichloroethene | 79-01-6 | Trichloroethylene | A-924 |
| Frichloroethylene | 79-01-6 | | A-924 |
| Trichloromethanesulfonyl Chloride | 594-42-3 | | A-928 A-929 |
| Trichloromonofluoromethane | 75-69-4 | | A-929 |
| Irichlorophenol | 25167-82-2 | | A-931 |
| 2,4,5-Trichlorophenol | 95-95-4 | | A-933 |
| 2,4,6-Trichlorophenol | 88-06-2 93-76-5 | 2,4,5-T Acid | A-867 |
| 2,4,5-Trichlorophenoxyacetic Acid | 27323-41 - 7 | 2,4,5-1 ACIU | A-934 |
| Triethanolamine Dodecylbenzene Sulfonate | | | A-935 |
| Triethylamine | 121-44-8 | | A-937 |
| Trimethylamine | 75-50-3 | | A-940 |
| sym-Trinitrobenzene | 99-35-4 | Paral Jahrada | A-808 |
| 1,3,5-Trioxane, 2,4,6-Trimethyl- | 123-63-7 | Paraldehyde | A-941 |
| Tris(2,3-dibromopropy1) Phosphate | 126-72-7 72-57 - 1 | | A-942 |
| Trypan Blue | 13560-99-1 | | A-943 |
| 2,4,5-T Salts | 66-75-1 | Uracil Mustard | A-944 |
| Uracil, 5-[bis(2-chloroethyl)amino]- | 66-75-1 | DIACII Mustare | A-944 |
| Uracil Mustard | 541-09-3 | | A-260 |
| Uranyl Acetate | 10102-06-4 | | A-261 |
| Uranyl Nitrate | 36478-76-9 | | |
| Vanadic Acid, Ammonium Salt | 7803-55-6 | Ammonium Vanadate | A-27 |
| Vanadium (V) Oxide | 1314-62-1 | Vanadium Pentoxide | A-262 |
| Vanadium (V) Oxide Vanadium Pentoxide | 1314-62-1 | | A-262 |
| Vanadyl Sulfate | 27774-13-6 | | A-264 |
| Vinyl Acetate | 108-05-4 | | A-9 4 |
| Vinyl Chloride | 75-01-4 | | A-94 |
| Vinylidene Chloride | 75-34-5 | 1,1-Dichloroethylene | A-52 |
| Warfarin | 81-81-2 | · · · · · · · · · · · · · · · · · · · | A-95 |
| Xylene | 1330-20-7 | | A-95 |
| m-Xylene | 108-38-3 | | A-95 |
| o-Xylene | 95-47-6 | | A-95 |
| p-Xylene | 106-42-3 | | A-95 |
| Xvlenol | 1300-71 - 6 | | A-95 |
| Yohimban-16-carboxylic Acid, 11,17- Dimethoxy-18-[(3,4,5-trimethoxy- | 50-55-5 | Reserpine | A- 85 |
| benzoyl)oxy]-methyl Ester Zinc | 7440-66-6 | | A-26 |
| | | | (cont |

| Hazardous Substance | CAS No. | Consensual Name | Page |
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| Zinc Acetate | 557-34-6 | | A-266 |
| Zinc Ammonium Chloride | 52628-25-8 | | A-267 |
| | 14639-97-5 | | |
| | 14639-98-6 | | |
| Zinc Borate | 1332-07-6 | | A-268 |
| Zinc Bromide | 7699-45-8 | | A-269 |
| Zinc Carbonate | 3486-35-9 | | A-270 |
| Zinc Chloride | 7646-85-7 | | A-271 |
| Zinc Cyanide | 557-21-1 | | A-272 |
| Zinc Fluoride | 7783-49-5 | | A-273 |
| Zinc Formate | 557-41-5 | | A-274 |
| Zinc Hydrosulfite | 7779-86-4 | | A-275 |
| Zinc Nitrate | 7779-88-6 | | A-276 |
| Zinc Phenolsulfonate | 127-88-2 | | A-277 |
| Zinc Phosphide | 1314-84-7 | | A-278 |
| Zinc Silicofluoride | 16871-71-9 | | A-280 |
| Zinc Sulfate | 7733-02-0 | | A-281 |
| Zirconium Nitrate | 1374-89-9 | | A-282 |
| Zirconium Potassium Fluoride | 16923-95-8 | | A-283 |
| Zirconium Sulfate | 14644-61-2 | | A-284 |
| Zirconium Tetrachloride | 10026-11-6 | | A-285 |

Appendix A

Analytical Methods Available

Part I: Inorganic Compounds

ALUMINUM PHOSPHIDE (CAS Number 20859-73-8)

RQ: 45.4 kg

SAFETY INFORMATION: Liberates phosphine on contact with water. Toxic (inhala-

tion). Flammable.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Aluminum," Appendix B.

Air Samples
Laboratory Method

See "Aluminum," Appendix B. See "Phosphorus," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Aluminum," Appendix B. See "Phosphorus," Appendix B.

Water Samples Laboratory Method

See "Aluminum," Appendix B. See "Phosphorus," Appendix B.

ALUMINUM SULFATE (CAS Number 10043-01-3)

RQ: 2270 kg

SAFETY INFORMATION:

Air Samples Field Method

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Aluminum," Appendix B.

See "Fluorides and Other Compounds," Appendix B.

Air Samples Laboratory Method

See "Aluminum," Appendix B. See "Nitrates and Sulfates," Appendix B.

See "Sulfates (Particulate)," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Aluminum," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B.

See "Aluminum," Appendix B. See "Sulfates," Appendix B.

AMMONIA (CAS Number 7664-41-7)

RQ: 45.4 kg

SAFETY INFORMATION: Toxic gas.

> CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

Analysis for ammonia in air may be performed by use of a detector tube. 0.1 to 1 liter sample is collected and analyzed with a hand-operated bellows pump (Bendix/Gastec®, Draeger®, or equivalent) and an ammonia gas-detector tube (Bendix/Gastec® No. 3H, 3M, or 3L, Draeger® No. CH20501, 25501, or 31901, or equivalent). A positive result is indicated by a change in color in the tube (purple to yellow, purple to yellow, pink to yellow, orange to dark blue, yellow to grey, or yellow to violet, respectively). The color change is from an acid-base reaction and an acid-base indicator in all cases but one (Draeger® CH25501) where formation of the millon base nitrate produces the grey color.

INTERFERENCES:

Aliphatic amines produce positive results, but with lower sensitivity. Hydrazines react with the same sensitivity as ammonia.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is sensitive to concentration levels of ammonia of approximately 200 to 200,000 milligrams per cubic meter (3H and CH31901), 1 to 100 milligrams per cubic meter (3M and CH 25501), and 0.1 to 4 milligrams per cubic meter (3L and CH 20501) with the above sample sizes in the absence of interferences.

REFERENCES:

National Draeger, Inc., Detector Tube Handbook, 4th ed.; K. Leichnitz, Ed.; National Draeger, Inc.: Pittsburgh, 1979, pp. 35-37.

Bendix Corporation, Bendix Gastec Precision Gas Detector System Manual, Blue Book; Bendix Corporation: Largo, Florida, no date, pp. 21-23.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$13-20 (list).

See also: "Hydrazines and Other Compounds," Appendix B.

See also: "Organic Vapors," Appendix B. See also: "Volatile Species," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Ammonia," Appendix B.

Air Samples
Laboratory Method 1

METHOD SUMMARY:

Ammonia in air samples may be measured colorimetrically. The ammonia is collected from a known volume of air in a midget impinger containing dilute sulfuric acid. Nessler reagent is added to produce a yellow-brown complex, which is measured at 440 nm on a spectrophotometer.

INTERFERENCES:

Ammonium salts will react with the reagent to give a false high reading, but filtration of the air before its passage into the impinger will remove them.

QUALITY CONTROL:

A blank should be processed with the samples.

EPA/TECHNICAL STATUS:

This method is classified as tentative by the National Institute of Occupational Safety and Health (NIOSH) for use over the range of 14 to 94 milligrams per cubic meter of air with a 10-liter sample. Precision and accuracy information is not furnished. The method is applicable to concentration of 10 to 80 milligrams of ammonia per cubic meter according to the APHA.

REFERENCES:

U.S. Department of Health, Education and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 205; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

American Public Health Association Methods of Air Sampling and Analyses, 2nd ed., Method 801; APHA: Washington 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$25-30 (list), plus sampling costs.

See also: "Ammonia," Appendix B.

Air Samples
Laboratory Method 2

METHOD SUMMARY:

Infrared absorption spectrometry can be used to determine the concentration of ammonia in air samples. The absorbance at 10770 nm (929 cm⁻¹) is measured, using a 10-meter path length cell. Air can be sampled in a Saran® or Mylar® plastic bag and approximately 5 liters of the sample drawn into an evacuated cell. Continuous monitoring of air flowing through the cell is possible if the spectrometer is set to one particular wavelength. Some vapors can be adsorbed on silica gel and quantitatively desorbed into the absorption cell later. Tentative qualitative identification of compounds can be made based on a full infrared absorption spectrum.

INTERFERENCES:

Compounds present in the sample which absorb infrared radiation at the wavelength of interest will interfere.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The sensitivity of the method, using a 10-meter pathlength cell, is 17 milligrams per cubic meter. No precision or accuracy information is furnished.

REFERENCE:

American Public Health Association "Infrared Absorption Spectroscopy" In Methods of Air Sampling and Analysis, 2nd ed., M. Katz, Ed.; APHA: Washington, D.C., 1977, pp. 79-84.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$75-85 (list).

Note: NIOSH Method S347 (ion specific electrode analysis) is applicable.

(Reference provided by reviewer.)

See also: "Ammonia," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Ammonia," Appendix B.

Water Samples
Laboratory Method

See "Ammonia," Appendix B.

AMMONIUM BICARBONATE (CAS Number 1066-33-7)

<u>RQ</u>: 2270 kg

SAFETY INFORMATION:

Air Samples Field Method

Soil/Sediment Field Method

Water Samples Field Method

See "Ammonia," Appendix B. See "Volatile Species," Appendix B.

Air Samples Laboratory Method

See "Ammonia," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Ammonia," Appendix B.

Water Samples
Laboratory Method

See "Acid Anions," Appendix B. See "Ammonia," Appendix B.

AMMONIUM BIFLUORIDE (CAS Number 1341-49-7)

RQ: 2270 kg

SAFETY INFORMATION: Toxic (inhalation).

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Fluorides," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Ammonia," Appendix B.

See "Fluorides," Appendix B.

See "Fluorides and Other Compounds," Appendix B.

See "Volatile Species," Appendix B.

Air Samples Laboratory Method

See "Ammonia," Appendix B.

See "Fluorides," Appendix B.

See "Fluorides and Hydrogen Fluoride," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Ammonia," Appendix B.

Water Samples
Laboratory Method

See "Ammonia," Appendix B.

See "Fluoride Compounds," Appendix B.

See "Fluoride Ions," Appendix B.

See "Fluorides," Appendix B.

AMMONIUM BISULFITE (CAS Number 10192-30-0)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Ammonia," Appendix B.

See "Sulfite Ions," Appendix B.

See "Volatile Species," Appendix B.

Air Samples Laboratory Method

See "Ammonia," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Ammonia," Appendix B.

Water Samples
Laboratory Method

See "Ammonia," Appendix B. See "Sulfites," Appendix B.

AMMONIUM CARBONATE (CAS Number 506-87-6)

RQ: 2270 kg

SAFETY INFORMATION:

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Ammonia," Appendix B. See "Volatile Species," Appendix B.

Air Samples Laboratory Method

See "Ammonia," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Ammonia," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B. See "Ammonia," Appendix B.

AMMONIUM CHLORIDE (CAS Number 12125-02-9

RQ: 2270 kg SAFETY INFORMATION:

Air Samples Field Method

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Ammonia," Appendix B.

See "Halides. Cyanides, and Other Compounds," Appendix B. See "Volatile Species," Appendix B.

Air Samples Laboratory Method

See "Ammonia," Appendix B. See "Chlorides," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Ammonia," Appendix B.

Water Samples Laboratory Method

See "Ammonia," Appendix B. See "Chlorides," Appendix B.

AMMONIUM CHROMATE (CAS Number 7788-98-9)

RQ: 454 kg

CAUTION: The user should obtain additional toxicity/hazard SAFETY INFORMATION: information before handling this substance or using this analytical procedure.

> Air Samples Field Method

See "Chromium," Appendix B.

Soil/Sediment Samples Field Method

See "Chromium," Appendix B.

Water Samples Field Method

See "Ammonia," Appendix B.

See "Chromates," Appendix B. See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

See "Volatile Species," Appendix B.

Air Samples Laboratory Method

See "Ammonia," Appendix B.

See "Chromic Acid and Chromates," Appendix B.

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Ammonia," Appendix B. See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

Water Samples Laboratory Method

See "Ammonia," Appendix B.

See "Chromium," Appendix B.

See "Chromium, Trivalent," Appendix B.

See "Hexavalent Chromium," Appendix B.

AMMONIUM DICHROMATE (CAS Number 7789-09-5)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

See "Chromium," Appendix B.

Soil/Sediment Samples Field Method

See "Chromium," Appendix B.

Water Samples Field Method

See "Ammonia," Appendix B.

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

See "Volatile Species," Appendix B.

Air Samples Laboratory Method

See "Ammonia," Appendix B.

See "Chromic Acid and Chromates," Appendix B.

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Ammonia," Appendix B. See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

Water Samples Laboratory Method

See "Ammonia," Appendix B.

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

AMMONIUM FLUOBORATE (CAS Number 13826-83-0)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Ammonia," Appendix B. See "Volatile Species," Appendix B.

Air Samples
Laboratory Method

See "Ammonia," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Ammonia," Appendix B.

Water Samples
Laboratory Method

See "Ammonia," Appendix B.

AMMONIUM FLUORIDE (CAS Number 12125-01-8)

RQ: 45.4 kg

SAFETY INFORMATION: Exhibits chronic toxicity.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Fluorides," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Ammonia," Appendix B.

See "Fluorides and Other Compounds," Appendix B.

See "Volatile Species," Appendix B.

Air Samples
Laboratory Method

See "Ammonia," Appendix B.

See "Fluorides," Appendix B.

See "Fluorides and Hydrogen Fluoride," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Ammonia," Appendix B.

Water Samples
Laboratory Method

See "Acid Anions," Appendix B.

See "Amines," Appendix B.

See "Ammonia," Appendix B.

See "Fluorides," Appendix B.

AMMONIUM HYDROXIDE (CAS Number 1336-21-6)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

See "Hydroxides," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Ammonia," Appendix B.
See "Volatile Species," Appendix B.

Air Samples Laboratory Method

See "Ammonia," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Ammonia," Appendix B.

Water Samples
Laboratory Method

See "Ammonia," Appendix B.

AMMONIUM SILICOFLUORIDE (CAS Number 16919-19-0)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

Analysis for ammonium silicofluoride in air can be performed by filtration of air, followed by X-ray fluorescence analysis of the filter for silicon, using a portable X-ray fluorescence spectrometer with a radioisotope excitation source. Sampling time may be up to 8 hours, but analysis may be performed in approximately 1 minute. This method is non-destructive, so that further analysis of the filtered material may be undertaken following this measurement. This method is not specific for ammonium silicofluoride, but measures the total silicon content of the sample.

INTERFERENCES:

Polyvinyl chloride filters are unacceptable for sampling, and silica quartz filter materials contain silicon, aluminum, and phosphorus. Cellulose membrane or fiber filters and polycarbonate or fluorocarbon membrane filters are acceptable, while glass fiber filters are satisfactory for sampling only if of the highest purity.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

The method is sensitive to amounts of ammonium silicofluoride over 150 micrograms, or approximately 0.1 to 10.0 micrograms per cubic meter, depending on the volume of air sampled.

REFERENCES:

Rhodes, J. R.; Stout, J. C.; Schindler, J. S.; Piorek, S. "Portable X-ray Survey Meters for in Situ Trace Element Monitoring of Air Particulates," In Toxic Materials in the Atmosphere, STP-786; American Society for Testing and Materials: Philadelphia, 1981, pp. 70-82.

Rhodes, J. R.; Pradzynski, A. H.; Hunter, C. B.; Payne, J. S.; Lindgren, J. L. "Energy Dispersive X-ray Fluorescence Analysis of Air Particulates in Texas," Environ. Sci. Technol., 6(10), 1972, pp. 922-927.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$30 (list), plus sampling costs.

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Ammonia," Appendix B.

See "Fluorides and Other Compounds," Appendix B.

See "Volatile Species," Appendix B.

Air Samples Laboratory Method

METHOD SUMMARY:

Analysis for ammonium silicofluoride in air can be performed by filtration of air, followed by X-ray fluorescence analysis of the filter for silicon, using an X-ray fluorescence spectrometer. This method is not specific for ammonium silicofluoride, but measures the total silicon content of the sample. Sampling time may be up to 8 hours, but analysis may be performed in approximately 1 minute. This method is non-destructive, so that further analysis of the filtered material may be undertaken following this measurement.

INTERFERENCES:

Polyvinyl chloride filters are unacceptable for sampling, and silica quartz filter materials contain silicon, aluminum, and phosphorus. Cellulose membrane or fiber filters and polycarbonate or fluorocarbon membrane filters are acceptable. This method is not specific for ammonium silicofluoride, but measures silicon.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision information is furnished. The method is used to measure silicon only, and is sensitive to amounts of ammonium silicofluoride over 150 micrograms, or approximately 0.1 to 10.0 microgram per cubic meter, depending on the volume of air sampled.

REFERENCES:

Rhodes, J. R.; Stout, J. C.; Schindler, J. S.; Piorek, S. "Portable X-ray Survey Meters for in Situ Trace Element Monitoring of Air Particulates," In Toxic Materials in the Atmosphere, STP-786; American Society for Testing and Materials: Philadelphia, 1981, pp. 70-82.

Rhodes, J. R.; Pradzynski, A. H.; Hunter, C. B.; Payne, J. S.; Lindgren, J. L. "Energy Dispersive X-ray Fluorescence Analysis of Air Particulates in Texas," Environ. Sci. Technol., 6(10), 1972, pp. 922-927.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$30 (list), \$20 (bid), plus sampling costs.

Note: NIOSH Method S348 (analysis by ion chromatography) is applicable. (Reference furnished by reviewer.)

See also: "Ammonia," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Ammonia," Appendix B.

Water Samples
Laboratory Method

See "Ammonia," Appendix B. See "Fluorides," Appendix B.

AMMONIUM SULFAMATE (CAS Number 7773-06-0)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Ammonia," Appendix B. See "Volatile Species," Appendix B.

Air Samples
Laboratory Method

See "Ammonia," Appendix B. See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Ammonia," Appendix B.

Water Samples
Laboratory Method

See "Acid Anions," Appendix B. See "Ammonia," Appendix B.

AMMONIUM SULFIDE (CAS Number 12135-76-1)

RQ: 45.4 kg

SAFETY INFORMATION: Exhibits oral toxicity. Flashpoint 72°C.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Sulfides," Appendix B.

Water Samples Field Method

See "Ammonia," Appendix B.

See "Halides, Cyanides, and Other Compounds," Appendix B.

See "Sulfides," Appendix B.

See "Volatile Species," Appendix B.

Air Samples Laboratory Method

See "Ammonia," Appendix B.

See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Ammonia," Appendix B. See "Sulfides," Appendix B.

Water Samples Laboratory Method

See "Ammonia," Appendix B. See "Sulfides," Appendix B.

AMMONIUM SULFITE (CAS Number 10196-04-0)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Ammonia," Appendix B.

See "Sulfite Ions," Appendix B.

See "Volatile Species," Appendix B.

Air Samples
Laboratory Method

See "Ammonia," Appendix B.

See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Ammonia," Appendix B.

Water Samples
Laboratory Method

See "Acid Anions," Appendix B.

See "Ammonia," Appendix B.

See "Sulfites," Appendix B.

AMMONIUM THIOCYANATE (CAS Number 1762-95-4)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Ammonia," Appendix B. See "Volatile Species," Appendix B.

Air Samples
Laboratory Method

See "Ammonia," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Ammonia," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B.

See "Ammonia," Appendix B.

See "Thiocyanates," Appendix B.

AMMONIUM THIOSULFATE (CAS Number 7783-18-8)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Ammonia," Appendix B. See "Volatile Species," Appendix B.

<u>Air Samples</u> Laboratory Method

See "Ammonia," Appendix B.
See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Ammonia," Appendix B.

Water Samples
Laboratory Method

See "Acid Anions," Appendix B. See "Ammonia," Appendix B.

AMMONIUM VANADATE (CAS Number 7803-55-6)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Ammonia," Appendix B. See "Volatile Species," Appendix B.

Air Samples
Laboratory Method

See "Ammonia," Appendix B. See "Vanadium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Ammonium," Appendix B. See "Vanadium," Appendix B.

Water Samples
Laboratory Method

See "Ammonia," Appendix B. See "Vanadium," Appendix B.

ANTIMONY (CAS Number 7440-36-0)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Antimony," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Antimony," Appendix B.

Air Samples Laboratory Method

Note: NIOSH Method P&CAM 351 (analysis by ICP) is applicable. (Reference

furnished by reviewer.)

See also: "Antimony," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Antimony," Appendix B.

Water Samples Laboratory Method

See "Antimony," Appendix B.

ANTIMONY PENTACHLORIDE (CAS Number 7647-18-9)

RQ: 454 kg

SAFETY INFORMATION: Reacts vigorously with water to form HC1.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Antimony," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

Reacts with water.

See "Antimony," Appendix B.

See "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples
Laboratory Method

See "Antimony," Appendix B. See "Chlorides," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Antimony," Appendix B.

Water Samples Laboratory Method

Reacts with water.

See "Antimony," Appendix B. See "Chlorides," Appendix B.

ANTIMONY POTASSIUM TARTRATE (CAS Number 28300-74-5)

RQ: 45.4 kg

SAFETY INFORMATION: Exhibits chronic toxicity.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Antimony," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Antimony," Appendix B. See "Potassium," Appendix B.

> Air Samples Laboratory Method

See "Antimony," Appendix B. See "Potassium," Appendix B.

> Soil/Sediment Samples Laboratory Method

See "Antimony," Appendix B. See "Potassium," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B.

See "Antimony," Appendix B. See "Potassium," Appendix B.

ANTIMONY TRIBROMIDE (CAS Number 7789-61-9)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Antimony," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

Reacts with water.

See "Antimony," Appendix B. See "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples
Laboratory Method

See "Antimony," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Antimony," Appendix B.

Water Samples
Laboratory Method

Reacts with water.

See "Antimony," Appendix B. See "Iodides and Bromides," Appendix B.

ANTIMONY TRICHLORIDE (CAS Number 10025-91-9)

RQ: 454 kg

SAFETY INFORMATION: Reacts vigorously with water to form HCl.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Antimony," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

Reacts with water.

See "Antimony," Appendix B.

See "Chlorides," Appendix B.

See "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples
Laboratory Method

See "Antimony," Appendix B. See "Chlorides," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Antimony," Appendix B.

Water Samples
Laboratory Method

Reacts with water.

See "Antimony," Appendix B. See "Chlorides," Appendix B.

ANTIMONY TRIFLUORIDE (CAS Number 7783-56-4)

RQ: 454 kg

SAFETY INFORMATION: Toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Antimony," Appendix B. See "Fluorides," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Antimony," Appendix B.

See "Fluorides and Other Compounds," Appendix B.

Air Samples Laboratory Method

See "Antimony," Appendix B.

See "Fluorides," Appendix B.

See "Fluorides and Hydrogen Fluoride," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Antimony," Appendix B.

Water Samples
Laboratory Method

See "Acid Anions," Appendix B.

See "Antimony," Appendix B.

See "Fluorides," Appendix B.

ANTIMONY TRIOXIDE (CAS Number 1309-64-4)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Antimony," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Antimony," Appendix B.

Air Samples
Laboratory Method

See "Antimony," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Antimony," Appendix B.

Water Samples Laboratory Method

See "Antimony," Appendix B.

ARSENIC (CAS Number 7440-38-2)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY

Arsenic in air samples can be measured by spectrophotometry. The arsenic, converted to arsine, is adsorbed on mercuric iodide crystals, eluted with iodine solution, and reacted with molybdate reagent and hydrogen sulfate solution at 100°C. The color is read at 720 nm on a portable spectrophotometer and the arsenic concentration is calculated from a standard curve. (This method is described by Olivee, W.T.; Funnell, H. S. Anal. Chem. 31, 1959, p. 259, and is summarized in the reference given below.)

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is a source method only.

REFERENCE:

Quantitative Analysis of Gaseous Pollutants, W. Ruch, Ed.; Ann Arbor-Humphrey Science: Ann Arbor, 1970, p. 35.

COST INFORMATION:

No cost information has been obtained.

Soil/Sediment Samples Field Method

Water Samples Field Method

Air Samples Laboratory Method

Note: NIOSH Methods P&CAM 286 (analysis by furnace AA) and P&CAM 320 (ion chromatography, hydride generation, analysis by quartz furnace AA) are applicable. (References provided by reviewer.)

See also: "Arsenic," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Arsenic," Appendix B.

Water Samples
Laboratory Method

ARSENIC ACID (CAS Numbers 1327-52-2, 7778-39-4)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Arsenic," Appendix B.

Water Samples Field Method

Air Samples Laboratory Method

See "Arsenic," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Arsenic," Appendix B.

Water Samples
Laboratory Method

ARSENIC DISULFIDE (CAS Number 1303-32-8)

RQ: 2270 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Arsenic," Appendix B. See "Sulfides," Appendix B.

Water Samples Field Method

See "Sulfides," Appendix B.

Air Samples
Laboratory Method

See "Arsenic," Appendix B.
See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Arsenic," Appendix B. See "Sulfides," Appendix B.

Water Samples
Laboratory Method

Insoluble in water.

ARSENIC PENTOXIDE (CAS Number 1303-28-2)

RQ: 2270 kg

SAFETY INFORMATION: Exhibits toxicity (oral).

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Arsenic," Appendix B.

Water Samples Field Method

Air Samples Laboratory Method

See "Arsenic," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Arsenic," Appendix B.

Water Samples Laboratory Method

ARSENIC TRICHLORIDE (CAS Number 7784-34-1)

RQ: 2270 kg

SAFETY INFORMATION: Reacts vigorously with water to form HC1.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Arsenic," Appendix B.

Water Samples Field Method

Reacts with water.

See "Halides, Cyanides and Other Compounds," Appendix B.

Air Samples
Laboratory Method

See "Arsenic," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Arsenic," Appendix B.

Water Samples
Laboratory Method

Reacts with water.

ARSENIC TRIOXIDE (CAS Number 1327-53-3)

RQ: 2270 kg

SAFETY INFORMATION: Exhibits toxicity (oral). Potentially carcinogenic.

Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Arsenic," Appendix B.

Water Samples Field Method

Air Samples
Laboratory Method

See "Arsenic," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Arsenic," Appendix B.

Water Samples
Laboratory Method

ARSENIC TRISULFIDE (CAS Number 1303-33-9)

RQ: 2270 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Arsenic," Appendix B. See "Sulfides," Appendix B.

Water Samples Field Method

See "Sulfides," Appendix B.

Air Samples
Laboratory Method

See "Arsenic," Appendix B. See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Arsenic," Appendix B. See "Sulfides," Appendix B.

Water Samples
Laboratory Method

See "Sulfides," Appendix B.

ASBESTOS (CAS Number 1332-21-4)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

METHOD SUMMARY:

A colorimetric test may be used to detect the possible presence of asbestos-bound magnesium and iron in soil/sediment samples. A 100-milligram sample is treated with acid to remove any iron and/or magnesium present that is not asbestos-bound, then reacted to release the asbestos-bound iron and magnesium. A positive result is indicated by a color change from clear to red.

INTERFERENCES:

A few other compounds that contain similarly bound magnesium and iron will produce false positive results.

QUALITY CONTROL:

Non-asbestos control samples for magnesium and non-asbestos control samples for iron should also be analyzed.

SENSITIVITY:

This method is sensitive to concentration levels of asbestos as low as 1 percent can be detected.

REFERENCES:

E.C. Apparatus Corporation, Asbestest® Test Kit for Screening Sample Materials for Possible Presence of Asbestos, EC Apparatus Bulletin No. 110, EC Apparatus Corp., St. Petersburg, Florida.

Gorman, Jr., W. EC Apparatus Corp., St. Petersburg, Florida, personal communication, April 1983.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$10-18 (list).

Water Samples Field Method

Air Samples Laboratory Method 1

METHOD SUMMARY:

Asbestos mineral particles in air samples can be measured by scanning transmission electron microscopy (STEM). The intensity of the X-ray spectrum obtained is compared to that of a standard. The analysis will differentiate particles of the three asbestos minerals: chrysotile, amosite, and crocidolite.

INTERFERENCES:

Variations in the diameter of the asbestos fiber can affect the intensity ratios under certain conditions.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information is not furnished.

REFERENCE:

Grasserbauer, M. In <u>Analysis of Airborne Particles by Physical Methods</u>, H. Malissa, Ed.; CRC Press: West Palm Beach, 1978, pp. 125-178.

COST INFORMATION:

The cost per sample for analysis by this method is \$250 and up, plus sampling costs.

Air Samples Laboratory Method 2

METHOD SUMMARY:

Asbestos fibers in air samples may be measured by microscopy. The sample is collected on a cellulose ester membrane filter. The filter is dissolved into a transparent, optically homogenous gel by treatment with a reagent

containing dimethyl phthalate and diethyl oxalate. The asbestos fibers are sized and counted using a phase-contrast microscope at 400-450x magnification.

INTERFERENCES:

All particulates with a length-to-diameter ratio of 3 to 1 or greater and a length greater than 5 micrometers are, in the absence of other information, considered to be asbestos if the atmosphere is known to contain asbestos.

QUALITY CONTROL:

A blind recount should be performed for about 1 in every 10 samples.

EPA/TECHNICAL STATUS:

This method is classified as operational by the National Institute for Occupational Safety and Health (NIOSH). The lowest limit of reliable quantification is 100,000 fibers per cubic meter (NIOSH). The test method is generally applicable for the number-count determination of asbestiform particulates ranging from 0.5 to 20 particulates per cubic centimeter of air (ASTM). Precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 239; Publication No. 77-157-A, U.S. DHEW, 1977.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.03, Method D4240-83; ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$30 (list).

Note: NIOSH Method P&CAM 309 (filter collection, redeposition on a silver filter, analysis by X-ray diffraction) is applicable for chrysotile asbestos.

Soil/Sediment Samples
Laboratory Method

Water Samples
Laboratory Method

METHOD SUMMARY:

Analysis for asbestos in water may be performed by electron-microscopic examination of a membrane filter after a known quantity of water has been passed through it.

INTERFERENCES:

Misidentification of fibers as asbestos can cause incorrect results, and obscuration by large amounts of extraneous material can also interfere.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is an EPA interim method, updating the 1967 EPA method, and is approved for use under the Clean Water Act. No precision or accuracy information is furnished.

REFERENCE:

Anderson, C. H.; Long, J. McA. <u>Interim Method for Determining Asbestos in</u> Water; EPA-600/4-80-005, U.S. EPA: Athens, Georgia, 1980.

COST INFORMATION:

The cost per sample for analysis by this method is \$250 and up.

BARIUM CYANIDE (CAS Number 542-62-1)

RQ: 4.54 kg

SAFETY INFORMATION: Highly toxic (ingestion).

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

Analysis for barium cyanide in air can be performed by filtration of air, followed by X-ray fluorescence analysis of the filter for barium, using a portable X-ray fluorescence spectrometer and a radioisotope excitation source. Sampling time may be up to 8 hours, but analysis may be performed in approximately 1 minute. This method is non-destructive, so that further analysis of the filtered material may be undertaken following this measurement. The method is not specific for barium cyanide, but measures the total barium content of the sample.

INTERFERENCES:

Polyvinyl chloride filters are unacceptable for sampling, and silica quartz filter materials contain silicon, aluminum, and phosphorus. Cellulose membrane or fiber filters and polycarbonate or fluorocarbon membrane filters are acceptable, while glass fiber filters are satisfactory for sampling only if of the highest purity.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

The method is sensitive to amounts of barium cyanide over 280 micrograms, or approximately 0.1 to 10.0 microgram per cubic meter, depending on the volume of air sampled.

REFERENCES:

Rhodes, J. R.; Stout, J. C.; Schindler, J. S.; Piorek, S. "Portable X-ray Survey Meters for in Situ Trace Element Monitoring of Air Particulates," In Toxic Materials in the Atmosphere, STP-786; American Society for Testing and Materials: Philadelphia, 1981, pp. 70-82.

Rhodes, J. R.; Pradzynski, A. H.; Hunter, C. B.; Payne, J. S.; Lindgren, J. L. "Energy Dispersive X-ray Fluorescence Analysis of Air Particulates in Texas," Environ. Sci. Technol., 6(10), 1972, pp. 922-927.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$30, plus sampling costs.

Soil/Sediment Samples Field Method

See "Cyanides," Appendix B.

Water Samples Field Method

METHOD SUMMARY:

Analysis for barium cyanide in water can be performed by turbidimetric analysis for barium. A 25-milliliter water sample is treated with a reagent, causing barium to precipitate as the sulfate. The precipitate is held in suspension by a colloid, and the turbidity is analyzed with a portable spectrophotometer. This method is not specific for barium cyanide, but measures the total barium content of the sample.

INTERFERENCES:

Calcium concentrations over 100 milligrams per liter interfere. Strontium interferes and is read as barium.

QUALITY CONTROL:

A barium standard solution can be used to check the accuracy of the test.

SENSITIVITY:

The method is sensitive to concentration levels of barium above approximately 25 milligrams per liter in the absence of interferences.

REFERENCE:

Hach Chemical Company, <u>Procedures, Chemical Lists</u>, and <u>Glassware for Water and Wastewater Analysis</u>, Hach: Ames, IA, 1975, pp. 2-17, 2-18.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$15-23 (list).

See also: "Cyanides," Appendix B.

See also: "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples Laboratory Method 1

METHOD SUMMARY:

Analysis for barium cyanide in air can be performed by filtration of air, followed by X-ray fluorescence analysis of the filter for barium, using an X-ray fluorescence spectrometer. Sampling time may be up to 8 hours, but analysis may be performed in approximately 1 minute. This method is non-destructive, so that further analysis of the filtered material may be undertaken following this measurement. This method is not specific for barium cyanide, but measures the total barium content of the sample.

INTERFERENCES:

Polyvinyl chloride filters are unacceptable for sampling, and silica quartz filter materials contain silicon, aluminum, and phosphorus. Cellulose membrane or fiber filters and polycarbonate or fluorocarbon membrane filters are acceptable, while glass-fiber filters are satisfactory for sampling only if of the highest purity.

QUALITY CONTROL:

No quality control procedure is given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision information is furnished. The method is sensitive to concentration levels of barium cyanide over 100 micrograms, or approximately 0.1 to 10.0 micrograms per cubic meter, depending on the volume of air sampled.

REFERENCES:

Rhodes, J. R.; Stout, J. C.; Schindler, J. S.; Piorek, S. "Portable X-ray Survey Meters for in Situ Trace Element Monitoring of Air Particulates," In Toxic Materials in the Atmosphere, STP-786; American Society for Testing and Materials: Philadelphia, 1981, pp. 70-82.

Rhodes, J. R.; Pradzynski, A. H.; Hunter, C. B.; Payne, J. S.; Lindgren, J. L. "Energy Dispersive X-ray Fluorescence Analysis of Air Particulates in Texas," Environ. Sci. Technol., $\underline{6}(10)$, 1972, pp. 922-927.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$30 (list), \$20 (bid), plus sampling costs.

Air Samples Laboratory Method 2

METHOD SUMMARY:

Analysis for barium cyanide in air can be performed by cellulose-membrane filtration of a known volume of air, followed by hot water leaching of soluble compounds and subsequent analysis of the resulting solution for barium by atomic absorption spectrometry (AA). The method is not specific for barium cyanide, but measures all those barium compounds that are dissolved from the collection filter in boiling water.

INTERFERENCES:

Addition of sodium chloride to the sample is required to suppress ionization.

QUALITY CONTROL:

A method blank should be processed with each set of 10 samples and the results used in the calculations.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 0.281 to 1.084 milligrams of barium per cubic meter for a 168-liter air sample. The method may be extended to lower concentrations by longer sampling times or increased instrumental response. A method classified as operational by NIOSH is for use over the range of 42 to 1.650 milligrams per cubic meter in a 240-liter air sample. Precision and accuracy information is furnished.

REFERENCES:

- U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set N, Method S198; PB-258 433 (NTIS), U.S. DHEW: Cincinnati, 1976.
- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 173; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$10-20 (list), plus sampling costs.

Note: NIOSH Method 351 (acid digestion, analyses for barium by ICP) is applicable. (Reference provided by reviewer.)

See also: "Cyanides," Appendix B.

Soil/Sediment Samples Laboratory Methods

METHOD SUMMARY:

Analysis of soil/sediment samples for barium cyanide can be performed by analysis for barium by atomic spectrometry. A 2-gram sample is digested with nitric acid and hydrogen peroxide and analyzed by inductively coupled plasma (ICP) or flame atomic absorption spectrometry (AA). These methods are not specific for barium cyanide, but measure the total barium present in the sample.

INTERFERENCES:

Potassium chloride must be added to the sample and standards to suppress the ionization of barium.

QUALITY CONTROL:

For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods. A reference standard should be run weekly and at least 10 percent of the samples should be duplicates and/or standards.

EPA/TECHNICAL STATUS:

These methods are in use in the CERCLA program and are approved for the National Pollutant Discharge Elimination System (NPDES). Precision and accuracy information is furnished.

REFERENCES:

- U.S. Environmental Protection Agency, Chemical Analytical Services for Low and Medium Concentration Inorganics in All Media, Exhibit D; Invitation for Bid (Solicitation Number WA 82-A072), U.S. EPA: Washington, April 23, 1982.
- U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 208.1; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.
- U.S. Environmental Protection Agency, <u>Test Methods for Evaluating Solid Waste</u>: <u>Physical/Chemical Methods</u>, 2nd ed., <u>Methods 7080</u>, 7081; SW-846, U.S. EPA: <u>Washington</u>, 1982.

Federal Register, 44 (233), December 3, 1979, pp. 69559-69564.

COST INFORMATION:

Cost per sample for analysis by flame AA is approximately \$24 (list); by ICP, it is approximately \$20 (list).

See also: "Cyanides," Appendix B.

Water Samples Laboratory Methods

METHOD SUMMARY:

Analysis of water samples for barium cyanide can be performed by atomic spectrometry. A 100-milliliter sample is digested with nitric acid and hydrogen peroxide and analyzed by inductively coupled plasma (ICP) or flame or furnace atomic absorption (AA) spectrometry. This method is not specific for barium cyanide, but measures the total barium content of the sample.

INTERFERENCES:

Potassium chloride must be added to the sample and standards to suppress the ionization of barium.

QUALITY CONTROL:

For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods. A reference standard should be run weekly and at least 10 percent of the analyses should be duplicates and/or standards.

EPA/TECHNICAL STATUS:

These methods are in use in the CERCLA program and are approved for the National Pollutant Discharge Elimination System (NPDES) of the Clean Water Act. Precision and accuracy information is furnished. Limits of detection are 0.1 milligram barium per liter for flame AA, and 2 micrograms barium per liter for furnace AA or ICP (EPA). The working range for flame AA is 1 to 5 milligrams per liter (ASTM).

REFERENCES:

- U.S. Environmental Protection Agency, Chemical Analytical Services for Low and Medium Concentration Inorganics in All Media, Exhibit D; Invitation for Bid (Solicitation Number WA 82-A072), U.S. EPA: Washington, April 23, 1982.
- U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Methods 200.7, 208.1, 208.2, EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.
- U.S. Environmental Protection Agency, <u>Test Methods for Evaluating Solid Waste</u>: <u>Physical/Chemical Methods</u>, 2nd ed., Methods 7080, 7081; SW-846, U.S. EPA: Washington, 1982.

Federal Register, 44 (233), December 3, 1979, pp. 69559-69564.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, D3051-78, D5986-81; ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost per sample for analysis by flame AA is approximately \$10-20 (list); by furnace AA, approximately \$18-50 (list), by ICP, approximately \$10 (list).

See also: "Acid Anions," Appendix B.
See also: "Cyanides," Appendix B.

BERYLLIUM (CAS Number 7440-41-7)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

Exhibits toxicity (inhalation).

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Beryllium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

Note: NIOSH METHOD 288 (graphite furnace analysis) is applicable. (Reference

furnished by reviewer.)

See also: "Beryllium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Beryllium," Appendix B.

Water Samples Laboratory Method

See "Beryllium," Appendix B.

BERYLLIUM CHLORIDE (CAS Number 7787-47-5)

RQ: 2270 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

Reacts vigorously with water to form HCl and to evolve

heat.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Beryllium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples Laboratory Method

See "Beryllium," Appendix B. See "Chlorides," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Beryllium," Appendix B.

Water Samples Laboratory Method

See "Beryllium," Appendix B. See "Chlorides," Appendix B.

BERYLLIUM FLUORIDE (CAS Number 7787-49-7)

RQ: 2270

Potentially carcinogenic. Potentially chronically toxic. SAFETY INFORMATION:

The user should obtain additional toxicity/hazard CAUTION:

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Beryllium," Appendix B. See "Fluorides," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Fluorides and Other Compounds," Appendix B.

Air Samples Laboratory Method

See "Beryllium," Appendix B.

See "Fluorides," Appendix B.

See "Fluorides and Hydrogen Fluoride," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Beryllium," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B. See "Beryllium," Appendix B.

See "Fluorides," Appendix B.

BERYLLIUM NITRATE (CAS Numbers 13597-99-4, 7787-55-5)

RQ: 2270 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Beryllium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Nitrate Ions," Appendix B.

Air Samples Laboratory Method

See "Beryllium," Appendix B. See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Beryllium," Appendix B. See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B. See "Beryllium," Appendix B.

See "Nitrates," Appendix B.

CACODYLIC ACID (CAS Number 75-60-5)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially carcinogenic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Arsenic," Appendix B.

Water Samples Field Method

Air Samples
Laboratory Method

See "Arsenic," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Arsenic," Appendix B.

Water Samples
Laboratory Method

CADMIUM (CAS Number 7440-43-9)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Cadmium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Cadmium," Appendix B.

Air Samples
Laboratory Method

METHOD SUMMARY:

Atomic spectrometry may be used for the analysis of cadmium fumes in air samples. Analysis is performed by membrane-filtration of 140 liters of air, followed by acid digestion of the sample and sampling medium and flame atomic absorption (AA) analysis of the resulting solution at 228.8 nm.

INTERFERENCES:

Silicon has been reported to interfere with the analysis. Cadmium dust and other cadmium compounds interfere.

QUALITY CONTROL:

A method blank should be analyzed with each set of 10 or fewer samples. The percent recovery should be determined and, if it is less than 95 percent, results corrected accordingly. Duplicate determinations should agree within 5 percent.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the ranges of 0.04 to 0.175 milligrams per cubic meter in a 140-liter air sample, and 0.122 to 0.175 milligrams per

cubic meter in a 25-liter sample. Precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, <u>Ten NIOSH Analytical Methods</u>, Set 1, Method S313; PB-271 712 (NTIS). U.S. DHEW: Cincinnati, 1977.

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, Method S313; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.

Note: NIOSH Method P&CAM 345 (filter collection and analyses by X-ray fluorescence) is applicable. (Reference provided by reviewer.)

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$10-20 (list), plus sampling costs.

See also: "Cadmium," Appendix B.

See also: "Lead and Cadmium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Cadmium," Appendix B.

Water Samples
Laboratory Method

See "Cadmium," Appendix B.

CADMIUM ACETATE (CAS Number 543-90-8)

RQ: 45.4 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Cadmium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Cadmium," Appendix B.

Air Samples
Laboratory Method

See "Cadmium," Appendix B.

See "Lead and Cadmium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Cadmium," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B.

See "Cadmium," Appendix B.

CADMIUM BROMIDE (CAS Number 7789-42-6)

RQ: 45.4 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Cadmium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Cadmium," Appendix B.

See "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples Laboratory Method

See "Cadmium," Appendix B.

See "Lead and Cadmium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Cadmium," Appendix B.

Water Samples Laboratory Method

See "Cadmium," Appendix B.

See "Iodides and Bromides," Appendix B.

CADMIUM CHLORIDE (CAS Number 10108-64-2)

RQ: 45.4 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic. CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Cadmium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Cadmium," Appendix B.

See "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples Laboratory Method

See "Cadmium," Appendix B. See "Chlorides," Appendix B.

See "Lead and Cadmium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Cadmium," Appendix B.

Water Samples Laboratory Method

See "Cadmium," Appendix B. See "Chlorides," Appendix B.

CALCIUM ARSENATE (CAS Number 7778-44-1)

RQ: 454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Calcium," Appendix B.

Soil/Sediment Samples Field Method

See "Arsenic," Appendix B.

Water Samples Field Method

Air Samples
Laboratory Method

See "Arsenic," Appendix B. See "Calcium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Arsenic," Appendix B. See "Calcium," Appendix B.

Water Samples Laboratory Method

See "Arsenic," Appendix B. See "Calcium," Appendix B.

CALCIUM ARSENITE (CAS Number 52740-16-6)

RQ: 454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Calcium," Appendix B.

Soil/Sediment Samples Field Method

See "Arsenic," Appendix B.

Water Samples Field Method

Air Samples Laboratory Method

See "Arsenic," Appendix B. See "Calcium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Arsenic," Appendix B. See "Calcium," Appendix B.

Water Samples Laboratory Method

See "Arsenic," Appendix B. See "Calcium," Appendix B.

CALCIUM CARBIDE (CAS Number 75-20-7)

RQ: 4.54 kg

SAFETY INFORMATION: Forms flammable and explosive gas on exposure to moisture.

The user should obtain additional toxicity/hazard CAUTION:

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Calcium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

See "Calcium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Calcium," Appendix B.

Water Samples Laboratory Method

See "Calcium," Appendix B.

CALCIUM CHROMATE (CAS Number 13765-19-0)

RQ: 454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Calcium," Appendix B. See "Chromium," Appendix B.

Soil/Sediment Samples Field Method

See "Chromium," Appendix B.

Water Samples Field Method

See "Chromium," Appendix B.

See "Chromates," Appendix B.

See "Hexavalent Chromium," Appendix B.

Air Samples Laboratory Method

See "Calcium," Appendix B.

See "Chromic Acid and Chromates," Appendix B.

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Calcium," Appendix B.

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

Water Samples Laboratory Method

See "Calcium," Appendix B.

See "Chromium," Appendix B.

See "Chromium, Trivalent," Appendix B.

See "Hexavalent Chromium," Appendix B.

CALCIUM CYANIDE (CAS Number 592-01-8)

RQ: 4.54 kg

SAFETY INFORMATION:

Forms toxic cyanide in water. Evolves highly toxic hydrogen cyanide on contact with acids. Fire risk if exposed to moisture or combined with calcium carbide. CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

See "Calcium," Appendix B.

Soil/Sediment Samples Field Method

See "Cyanides," Appendix B.

Water Samples Field Method

See "Halides, Cyanides, and Other Compounds," Appendix B. See "Cyanides," Appendix B.

Air Samples
Laboratory Method

See "Calcium," Appendix B. See "Cyanides," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Calcium," Appendix B. See "Cyanides," Appendix B.

Water Samples
Laboratory Method

See "Acid Anions," Appendix B. See "Calcium," Appendix B. See "Cyanides," Appendix B.

CALCIUM DODECYLBENZENE SULFONATE (CAS Number 26264-06-2)

RQ: 454 kg

SAFETY INFORMATION:

Air Samples Field Method

See "Calcium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Anionic Detergents," Appendix B.

Air Samples Laboratory Method

See "Calcium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Calcium," Appendix B.

Water Samples Laboratory Method

See "Alkyl Benzene Sulfonates," Appendix B. See "Calcium," Appendix B.

CALCIUM HYPOCHLORITE (CAS Number 7778-54-3)

RQ: 4.54 kg

SAFETY INFORMATION: Strong oxidizer; can readily cause fires.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Calcium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Calcium," Appendix B.
See "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples
Laboratory Method

See "Calcium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Calcium," Appendix B.

Water Samples Laboratory Method

See "Calcium," Appendix B. See "Chlorine," Appendix A.

CHLORINE (CAS Number 7782-50-5)

RQ: 4.54 kg

SAFETY INFORMATION: Toxic (inhalation). Dangerous if in contact with

reducing agents.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

Analysis for chlorine in air may be performed by use of a detector tube. A 2-liter sample is collected and analyzed with a hand-operated bellows pump and a chlorine gas detector tube (Draeger® 67 28411 or equivalent). A positive result is indicated by a color change in the tube from white to either pale yellow or pale yellowish-orange. The test is based on the reaction of chlorine with o-toluidine.

INTERFERENCES:

Chlorine dioxide will also give a positive result.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

The lower detection limit of the tube varies from approximately 0.5 to 1 milligram per cubic meter.

REFERENCES:

National Draeger, Inc., <u>Draeger® Detector Tube Handbook</u>, "Air Investigations and Technical Gas Analysis with Draeger® Tubes", 4th ed.; Draeger: Pittsburgh, August 1979.

National Draeger, Inc., Bulletin No. ND-105, Draeger: Pittsburgh, January 1, 1982.

National Draeger, Inc., Measurements with Draeger® Tubes in the Threshold Limit Value Range, Draeger® Gas Analysis Product Information; Leaflet 4340.3e, Draeger: Pittsburgh, December 1981.

COST INFORMATION:

The cost per sample by this method is approximately \$13-18 (list).

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples Laboratory Method 1

METHOD SUMMARY:

Free chlorine in air may be determined colorimetrically. A known volume of air is passed through a chromic acid scrubber and two impingers. The sample is treated with an alkaline solution of 4-nitroaniline to produce an orange-brown color that is measured at 485 nm with a spectrophotometer. Six standard solutions, with concentrations from 10 micrograms to 2 milligrams of free chlorine per liter, are used for calibration.

INTERFERENCES:

Sulfur dioxide interference is removed by the chromic acid scrubber.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved, but is used by the Alberta Environmental Centre, Alberta, Canada. Precision and accuracy information is not furnished. The method is sensitive to concentration levels of chlorine in the range of 0.2 to 50 micrograms per milliliter in the absorbing solution.

REFERENCES:

Alberta Environment, Methods Manual Chemical Analysis of Atmospheric Pollutants, 2nd ed., Method 11515, Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

Geabbay, J.; Davidson, M.; Donagi, A. "Spectrophotometric Determination of Free Chlorine in Air," Analyst, 101, February 1976, p. 128-135.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$20-25 (list), plus sampling costs.

Air Samples Laboratory Method 2

METHOD SUMMARY:

Free chlorine in air samples may be measured colorimetrically. A known volume of air is passed through a fritted bubbler containing dilute methyl orange. The methyl orange dye is bleached quantitatively by free chlorine, and the extent of bleaching is determined with a spectrophotometer at 505 nm.

INTERFERENCES:

Free bromine, nitrogen dioxide, manganese, and possibly ozone interfere positively. Sulfur dioxide in solution interferes negatively. Nitrites impart an off-color orange to the methyl orange reagent.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is classifed as tentative by the National Institute of Occupational Safety and Health (NIOSH) for use over the range of 145 to 2900 micrograms per cubic meter in a 30-liter sample. Limited precision information is furnished.

REFERENCE:

U.S. Department of Health, Education and Welfare, NIOSH Manual of Analtyical Methods, 2nd ed., Vol. 1, Method P&CAM 209; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$20-25 (list), plus sampling costs.

Soil/Sediment Samples
Laboratory Method

Water Samples
Laboratory Method 1

METHOD SUMMARY:

Water samples may be analyzed for chlorine by spectrophotometry. A 10-milliliter sample is treated with potassium iodide at a pH below 4; the liberated iodine reacts with N,N-diethyl-p-phenylenediamine (DPD) to produce a red-colored solution, which is measured spectrophotometrically.

INTERFERENCES:

Turbidity or color in the sample can preclude use of the method.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is approved for use in the National Pollutant Discharge Elimination System (NPDES) of the Clean Water Act. Precision and accuracy information is furnished.

REFERENCE:

U.S. Environmental Protection Agency, Methods for the Chemical Analysis of Water and Wastes, Method 330.5, EPA-600/4-79-020; U.S. EPA: Cincinnati, 1979.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$20-25 (list).

Water Samples Laboratory Method 2

METHOD SUMMARY:

Water samples may be analyzed for chlorine by one of three iodometric titrimetric methods. In each of the methods, iodine is stoichiometrically liberated from potassium iodide by the chlorine in a 100- or 200-milliliter sample. In the first method, the iodine is titrated with phenylarsine oxide, and an amperometer or a starch indicator can be used to detect the endpoint. Stirring may lower the chlorine value, and copper, silver or substantial amounts of organic matter must not be present. In the second method, an excess of phenylarsine oxide is added and the excess is determined by back-titration with a standard iodine titrant. Either a starch indicator or an amperometer can be used to determine the endpoint. In the third method, the liberated iodine is titrated with N,N-diethyl-p-phenylenediamine (DPD), using ferrous ammonium sulfate (FAS) as an indicator. All three methods will measure chlorine concentrations above 1 milligram per liter.

INTERFERENCES:

Color and turbidity in the sample can make the endpoint difficult to detect when a starch indicator is used, while copper and silver ions can interfere with amperometric endpoint detection. Buffering the sample at pH 4 minimizes interferences for the starch-indicator methods from iron(III), manganese(VI), and nitrite ions. Copper and dissolved oxygen are prevented from interfering with the FAS/DPD procedure by addition of disodium ethylene-diaminetetraacetate.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

These methods are approved for use in the National Pollutant Discharge Elimination System (NPDES) of the Clean Water Act. Precision and accuracy information is furnished.

REFERENCE:

U.S. Environmental Protection Agency Methods for the Chemical Analysis of Water and Wastes, Methods 330.1, 330.2, 330.4; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$13-18 (list).

CHLOROSULFONIC ACID (CAS Number 7790-94-5)

RQ: 454 kg

SAFETY INFORMATION:

Dangerously reactive with water or moisture; reacts to form hydrochloric acid and sulfuric acid. Strong oxidizing agent. Highly toxic (inhalation). Strong irritant (eye, skin). Flammable in contact with combustable material; evolves hydrogen gas on contact with most metals.

CAUTION:

The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

Laboratory Method Field Method

> Water Samples Field Method

Reacts with water.

See "Cholinesterase-Inhibiting Compounds," Appendix B. See "Fluorides and Other Compounds," Appendix B. See "Halides, Cyanides, and Other Compounds, Appendix B.

Air Samples
Laboratory Method

Soil/Sediment Samples
Laboratory Method

Water Samples
Laboratory Method

Reacts with water.

CHROMIC ACETATE (CAS Number 1066-30-4)

RQ: 454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Chromium," Appendix B.

Soil/Sediment Samples Field Method

See "Chromium," Appendix B.

Water Samples Field Method

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

Air Samples Laboratory Method

See "Chromic Acid and Chromates," Appendix B.

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B.

See "Chromium," Appendix B.
See "Chromium, Trivalent," Appendix B.

CHROMIC ACID (CAS Number 11115-74-5)

RQ: 454 kg

SAFETY INFORMATION: Strong oxidizer; can readily cause fires. Potentially

carcinogenic. Potentially chronically toxic. Strong

irritant to skin (corrosive).

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Chromium," Appendix B.

See "Chromic Acid and Strontium Chromate," Appendix B.

Soil/Sediment Samples Field Method

See "Chromium," Appendix B.

Water Samples Field Method

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

Air Samples Laboratory Method

METHOD SUMMARY:

Chromic acid mist in air samples may be measured colorimetrically. The chromic acid is collected on a polyvinyl chloride (PVC) filter, the filter is washed in 0.5N sulfuric acid, diphenylcarbazide is added, and additional acid is added to bring the solution to volume. The absorbance of the solution is read at 540 nm on a spectrophotometer.

INTERFERENCES:

Many of the heavy metals may interfere at high concentrations.

QUALITY CONTROL:

Spiked filters should not be used for standards or other purposes since such samples are not very stable. A blank should be processed and its value, if any, should be used in calculation of results.

EPA/TECHNICAL STATUS:

This method is classified as operational by the National Institute for Occupational Safety and Health (NIOSH) for use over the range of 0.004 to 0.2 milligrams chromic acid per cubic meter, using a 100-liter air sample. Precision and accuracy information is furnished.

REFERENCES:

- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 169; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.
- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, Method P&CAM 169; Publication No. 75-121, U.S. DHEW: Cincinnati, 1974.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$25-30 (list), plus sampling costs.

See also: "Chromic Acid and Chromates," Appendix B.

See also: "Chromium," Appendix B.

See also: "Hexavalent Chromium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Chromium," Appendix B.
See "Hexavalent Chromium," Appendix B.

Water Samples

Laboratory Method

See "Chromium," Appendix B. See "Chromium, Hexavalent," Appendix B.

CHROMIC SULFATE (CAS Number 10101-53-8)

RQ: 454 kg

Potentially chronically toxic. SAFETY INFORMATION:

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Chromium," Appendix B.

Soil/Sediment Samples Field Method

See "Chromium," Appendix B.

Water Samples Field Method

See "Chromium, Hexavalent," Appendix B.

See "Chromium," Appendix B.

Air Samples Laboratory Method

See "Nitrates and Sulfates," Appendix B.

See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Chromium," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B.

See "Chromium," Appendix B. See "Chromium, Trivalent," Appendix B.

CHROMIUM (CAS Number 7440-47-3)

 \underline{RQ} : 0.454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Chromium," Appendix B.

Soil/Sediment Samples Field Method

See "Chromium," Appendix B.

Water Samples Field Method

See "Chromium," Appendix B.

Air Samples Laboratory Method

See "Chromium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Chromium," Appendix B.

See "Chromium, Hexavalent," Appendix B.

Water Samples
Laboratory Method

See "Chromium," Appendix B.

See "Chromium, Hexavalent," Appendix B.

CHROMOUS CHLORIDE (CAS Number 10049-05-5)

RQ: 454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Chromium," Appendix B.

Soil/Sediment Samples Field Method

See "Chromium," Appendix B.

Water Samples Field Method

See "Chromium," Appendix B.

Air Samples Laboratory Method

See "Chromium," Appendix B. See "Chlorides," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Chromium," Appendix B.

Water Samples
Laboratory Method

See "Chlorides," Appendix B. See "Chromium," Appendix B.

COBALTOUS BROMIDE (CAS Number 7789-43-7)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Cobalt," Appendix B.

Water Samples Field Method

See "Cobalt," Appendix B.
See "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples
Laboratory Method

See "Cobalt," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Cobalt," Appendix B.

Water Samples
Laboratory Method

See "Cobalt," Appendix B.

COBALTOUS FORMATE (CAS Number 544-18-3)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Cobalt," Appendix B.

Water Samples Field Method

See "Cobalt," Appendix B.

Air Samples Laboratory Method

See "Cobalt," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Cobalt," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B. See "Cobalt," Appendix B.

COBALTOUS SULFAMATE (CAS Number 14017-41-5)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Cobalt," Appendix B.

Water Samples Field Method

See "Cobalt," Appendix B.

Air Samples Laboratory Method

See "Cobalt," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Cobalt," Appendix B.

Water Samples Laboratory Method

See "Cobalt," Appendix B.

COPPER (CAS Number 7440-50-8)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Copper," Appendix B.

 $\frac{\texttt{Soil/Sediment Samples}}{\texttt{Field Method}}$

See "Copper," Appendix B.

Water Samples Field Method

See "Copper," Appendix B.

Air Samples
Laboratory Method

See "Copper," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Copper," Appendix B.

Water Samples
Laboratory Method

See "Copper," Appendix B.

COPPER CYANIDE (CAS Number 544-92-3)

RQ: 4.54 kg

SAFETY INFORMATION: Toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Copper," Appendix B.

Soil/Sediment Samples Field Method

See "Copper," Appendix B. See "Cyanides," Appendix B.

Water Samples Field Method

See "Copper," Appendix B. See "Cyanides," Appendix B.

Air Samples Laboratory Method

See "Copper," Appendix B. See "Cyanides," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Copper," Appendix B. See "Cyanides," Appendix B.

Water Samples Laboratory Method

See "Copper," Appendix B. See "Cyanides," Appendix B.

CUPRIC ACETATE (CAS Number 142-71-2)

RQ: 45.4 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

See "Copper," Appendix B.

Soil/Sediment Samples Field Method

See "Copper," Appendix B.

Water Samples Field Method

See "Copper," Appendix B.

Air Samples Laboratory Method

See "Copper," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Copper," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B. See "Copper," Appendix B.

CUPRIC ACETOARSENITE (CAS Number 12002-03-8)

RQ: 45.4 kg

SAFETY INFORMATION: Highly toxic (ingestion).

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Copper," Appendix B.

Soil/Sediment Samples Field Method

See "Arsenic," Appendix B. See "Copper," Appendix B.

Water Samples Field Method

See "Copper," Appendix B.

Air Samples Laboratory Method

See "Arsenic," Appendix B. See "Copper," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Arsenic," Appendix B. See "Copper," Appendix B.

Water Samples Laboratory Method

See "Arsenic," Appendix B. See "Copper," Appendix B.

CUPRIC CHLORIDE (CAS Number 7447-39-4)

RQ: 4.54 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Copper," Appendix B.

 $\frac{\texttt{Soil/Sediment Samples}}{\texttt{Field Method}}$

See "Copper," Appendix B.

Water Samples Field Method

See "Copper," Appendix B.

See "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples
Laboratory Method

See "Chlorides," Appendix B.

See "Copper," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Copper," Appendix B.

Water Samples Laboratory Method

See "Chlorides," Appendix B.

See "Copper," Appendix B.

CUPRIC NITRATE (CAS Number 3251-23-8)

 \underline{RQ} : 45.4 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

> Air Samples Field Method

See "Copper," Appendix B.

Soil/Sediment Samples Field Method

See "Copper," Appendix B.

Water Samples Field Method

See "Copper," Appendix B. See "Nitrate Ions," Appendix B.

> Air Samples Laboratory Method

See "Copper," Appendix B. See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Copper," Appendix B. See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Copper," Appendix B. See "Nitrates," Appendix B.

CUPRIC OXALATE (CAS Number 5893-66-3)

RQ: 45.4 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

See "Copper," Appendix B.

Soil/Sediment Samples Field Method

See "Copper," Appendix B.

Water Samples Field Method

See "Copper," Appendix B.

Air Samples Laboratory Method

See "Copper," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Copper," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B. See "Copper," Appendix B.

CUPRIC SULFATE (CAS Number 7758-98-7)

RQ: 4.54 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

See "Copper," Appendix B.

Soil/Sediment Samples Field Method

See "Copper," Appendix B.

Water Samples Field Method

See "Copper," Appendix B.

Air Samples Laboratory Method

See "Copper," Appendix B.

See "Nitrates and Sulfates," Appendix B. See "Sulfates (Particulate)," Appendix B.

See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Copper," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Copper," Appendix B. See "Sulfates," Appendix B.

CUPRIC SULFATE, AMMONIATED (CAS Number 10380-29-7)

RQ: 45.4 kg

The user should obtain additional toxicity/hazard SAFETY INFORMATION: CAUTION: information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Copper," Appendix B.

Soil/Sediment Samples Field Method

See "Copper," Appendix B.

Water Samples Field Method

See "Ammonia," Appendix B. See "Copper," Appendix B.

See "Volatile Species," Appendix B.

Air Samples Laboratory Method

See "Ammonia," Appendix B. See "Copper," Appendix B.

See "Nitrates and Sulfates," Appendix B.

See "Sulfates," Appendix B.

See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Ammonia," Appendix B. See "Copper," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Ammonia," Appendix B.

See "Copper," Appendix B.

See "Sulfates," Appendix B.

CUPRIC TARTRATE (CAS Number 815-82-7)

RQ: 45.4 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Copper," Appendix B.

Soil/Sediment Samples Field Method

See "Copper," Appendix B.

Water Samples Field Method

See "Copper," Appendix B.

Air Samples Laboratory Method

See "Copper," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Copper," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B.

See "Copper," Appendix B.

CYANIDES (CAS Number 57-12-5)

RQ: 4.54 kg

SAFETY INFORMATION: Can form very toxic hydrogen cyanide upon contact with

water or acids.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Cyanides," Appendix B. See "Hydrogen Cyanide," Appendix A.

Soil/Sediment Samples Field Method

See "Cyanides," Appendix B.

Water Samples Field Method

See "Cyanides," Appendix B.

Air Samples
Laboratory Method

See "Cyanides," Appendix B.

See "Hydrogen Cyanide," Appendix A.

Soil/Sediment Samples
Laboratory Method

See "Cyanides," Appendix B.

Water Samples
Laboratory Method

CYANOGEN (CAS Number 460-19-5)

RQ: 45.4 kg

SAFETY INFORMATION: Highly toxic. Highly flammable gas.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Cyanides," Appendix B.

Water Samples Field Method

See "Cyanides," Appendix B.

Air Samples Laboratory Method

See "Cyanides," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Cyanides," Appendix B.

Water Samples Laboratory Method

CYANOGEN BROMIDE (CAS Number 506-68-3)

RQ: 454 kg

SAFETY INFORMATION: Highly toxic. Strong irritant to eyes and skin.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

A cyanogen chloride gas detector tube (Draeger® CH 19801, or equivalent) can be used for measurement of cyanogen bromide in air. The cyanogen bromide reacts with pyridine, barbituric acid, and glutaconaldehyde cyanamide in the indicating layer. A positive result is indicated by a color change from white to brownish pink in the indicating layer of the tube.

INTERFERENCES:

Chlorine dioxide and cyanogen chloride can give a positive interference.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is sensitive to concentration levels of cyanogen bromide over 5 milligrams per cubic meter.

REFERENCE:

National Draeger, Inc., Draeger® Detector Tube Handbook, "Air Investigations and Technical Gas Analysis with Draeger® Tubes", 4th ed.; Draeger: Pittsburgh, August 1979.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$13-(list).

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Cyanides," Appendix B.

Air Samples Laboratory Method

See "Cyanides," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Cyanides," Appendix B.

Water Samples Laboratory Method

See "Substituted Nitriles and Tetranitromethane," Appendix B.

CYANOGEN CHLORIDE (CAS Number 506-77-4)

RQ: 4.54 kg

SAFETY INFORMATION: Highly toxic (inhalation and injestion).

Air Samples Field Method

METHOD SUMMARY:

Analysis for cyanogen chloride in air may be performed by use of a detector tube. A 0.1- to 2-liter sample is collected and analyzed with a hand-operated bellows pump and a cyanogen chloride gas detector tube (Draeger® CH 19801 or equivalent). A positive result is indicated by a color change in the tube from white to pink.

INTERFERENCES:

High concentrations of cyanogen bromide will cause a brownish to pink color change.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is sensitive to concentration levels of approximately 0.5 to 15 milligrams per cubic meter.

REFERENCES:

Snyder, R. E.; Jonkin, M. E.; McKissick, A. M. <u>Development of Hazardous Toxic Wastes Analytical Screening Procedures</u>; Atlantic Research Corporation for U.S. Army Medical Research and Development Command: Fort Detrick, Maryland, July 16, 1982, p. 4.

National Draeger, Inc., <u>Draeger® Detector Tube Handbook</u>, "Air Investigations and Technical Gas Analysis with Draeger® Tubes", 4th ed.; Draeger: Pittsburgh, August 1979.

National Draeger®, Inc., Bulletin No. ND-105; Draeger: Pittsburgh, January 1, 1982.

National Draeger, Inc., Measurements with Draeger® Tubes in the Threshold Limit Value Range, Draeger® Gas Analysis Product Information, Leaflet 4340.3e; Draeger: Pittsburgh, December 1981.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$13-20 (list).

Soil/Sediment Samples Field Method

See "Cyanides," Appendix B.

Water Samples Field Method

METHOD SUMMARY:

Cyanogen chloride in water samples can be measured spectrophotometrically. Phosphate buffer and pyridine-barbituric acid reagent are added to the water sample, and the color is read at 578 nm. The analysis should be conducted immediately after sampling, since cyanogen chloride is volatile and labile. This method may involve hazardous materials and operations.

INTERFERENCES:

Substances that create color or turbidity may interfere.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

The limit of detection is 0.005 milligrams of cyanide ion per liter.

REFERENCE:

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, D4165-82; ASTM: Philadelphia, 1983, pp. 132-135.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$20 (list).

See also: "Cyanides," Appendix B.

Air Samples
Laboratory Method

See "Chlorides," Appendix B. See "Cyanides," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Cyanides," Appendix B.

Water Samples Laboratory Method

See "Chlorides," Appendix B. See "Cyanides," Appendix B.

DICHLOROPHENYLARSINE (CAS Number 696-28-6)

RQ: 0.454 kg

SAFETY INFORMATION: Exhibits toxicity (skin). Potentially carcinogenic.

Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

See "Arsenic," Appendix B. See "Arsines," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Arsenic," Appendix B.

Water Samples Laboratory Method

See "Arsenic," Appendix B.

DIETHYLARSINE (CAS Number 692-42-2)

RQ: 0.454 kg

Pyrophoric. Potentially carcinogenic. Potentially SAFETY INFORMATION:

chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

See "Arsenic," Appendix B. See "Arsines," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Arsenic," Appendix B.

Water Samples Laboratory Method

See "Arsenic," Appendix B.

FERRIC AMMONIUM CITRATE (CAS Number 1185-57-5)

RQ: 454 kg

SAFETY INFORMATION:

Air Samples Field Method

See "Iron Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Ammonia," Appendix B. See "Iron," Appendix B.

See "Volatile Species," Appendix B.

Air Samples Laboratory Method

See "Ammonia," Appendix B.

See "Iron," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Ammonia," Appendix B.

See "Iron," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B.

See "Ammonia," Appendix B.

See "Iron," Appendix B.

FERRIC AMMONIUM OXALATE (CAS Numbers 55488-87-4, 2944-67-4)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

See "Iron Compounds (Particulate), " Appendix B.

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Ammonia," Appendix B. See "Iron," Appendix B.

See "Volatile Species," Appendix B.

Air Samples Laboratory Method

See "Ammonia," Appendix B. See "Iron," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Ammonia," Appendix B. See "Iron," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B.

See "Ammonia," Appendix B.

See "Iron," Appendix B.

FERRIC CHLORIDE (CAS Number 7705-08-0)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Iron Compounds (Particulate)." Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Halides, Cyanides, and Other Compounds," Appendix B. See "Iron," Appendix B.

Air Samples
Laboratory Method

See "Chlorides," Appendix B. See "Iron," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Iron," Appendix B.

Water Samples Laboratory Method

See "Chlorides," Appendix B. See "Iron," Appendix B.

FERRIC DEXTRAN (CAS Number 9004-66-4)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially toxic.

Air Samples Field Method

See "Iron Compounds (Particulate), " Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Iron," Appendix B.

Air Samples Laboratory Method

See "Iron," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Iron," Appendix B.

Water Samples Laboratory Method

See "Iron," Appendix B.

FERRIC FLUORIDE (CAS Number 7783-50-8)

RQ: 45.4 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

See "Fluorides," Appendix B.
See "Iron Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Fluorides," Appendix B. See "Fluorides and Other Compounds," Appendix B. See "Iron," Appendix B.

Air Samples Laboratory Method

See "Fluorides," Appendix B.
See "Fluorides and Hydrogen Fluoride," Appendix B.
See "Iron," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Iron," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B. See "Fluorides," Appendix B. See "Iron," Appendix B.

FERRIC NITRATE (CAS Number 10421-48-4)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

See "Iron Compounds (Particulate), "Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Iron," Appendix B. See "Nitrate Ions," Appendix B.

Air Samples
Laboratory Method

See "Iron," Appendix B.

See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Iron," Appendix B.

See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Water Samples
Laboratory Method

See "Iron," Appendix B.

See "Nitrates," Appendix B.

FERRIC SULFATE (CAS Number 10028-22-5)

RQ: 454 kg

SAFETY INFORMATION:

Air Samples Field Method

See "Iron Compounds (Particulate), "Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Iron," Appendix B.

Air Samples Laboratory Method

See "Iron," Appendix B.

See "Nitrates and Sulfates," Appendix B. See "Sulfates (Particulate)," Appendix B.

See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment_Samples Laboratory Method

See "Iron," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Iron," Appendix B.

See "Sulfates," Appendix B.

FERROUS AMMONIUM SULFATE (CAS Number 10045-89-3)

RQ: 454 kg SAFETY INFORMATION:

Air Samples Field Method

See "Iron Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Ammonia," Appendix B.
See "Iron," Appendix B.
See "Volatile Species," Appendix B.

Air Samples Laboratory Method

See "Ammonia," Appendix B.
See "Iron," Appendix B.
See "Nitrates and Sulfates," Appendix B.
See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Ammonia," Appendix B.
See "Iron," Appendix B.
See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Ammonia," Appendix B. See "Iron," Appendix B. See "Sulfates," Appendix B.

FERROUS CHLORIDE (CAS Number 7758-94-3)

RQ: 45.4 kg SAFETY INFORMATION:

Air Samples Field Method

See "Iron Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Halides, Cyanides, and Other Compounds," Appendix B. See "Iron," Appendix B.

Air Samples Laboratory Method

See "Chlorides," Appendix B. See "Iron," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Iron," Appendix B.

Water Samples Laboratory Method

See "Chlorides," Appendix B. See "Iron," Appendix B.

<u>RQ</u>: 454 kg

SAFETY INFORMATION:

Air Samples Field Method

See "Iron Compounds (Particulate), " Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Iron," Appendix B.

Air Samples Laboratory Method

See "Iron," Appendix B.

See "Nitrates and Sulfates," Appendix B.

See "Sulfates (Particulate)," Appendix B.

See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Iron," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Water Samples
Laboratory Method

See "Iron," Appendix B.

See "Sulfates," Appendix B.

FLUORINE (CAS Number 7782-41-4)

RQ: 4.54 kg

SAFETY INFORMATION: Strong oxidizer capable of igniting substances and sustaining combustion.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

Reacts with water-

Water Samples Field Method

Reacts with water.

Air Samples Laboratory Method

METHOD SUMMARY:

Molecular fluorine in air samples can be determined electrochemically. The air sample is passed through a bubbler containing lithium chloride solution. The bubbler is part of a unit that acts as a reaction cell. The current generated by the cell when fluorine passes through is measured amperometrically, and is proportional to the concentration of the fluorine.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

Calibration is limited to measurements of the sampling pump flow rate.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No specific precision and accuracy data are supplied, although a general statement is furnished.

REFERENCE:

Kaye, S.; Griggs, M. "Electroanalytical Determination of Molecular Fluorine in the Atmosphere," <u>Anal. Chem.</u>, 40(14), 1968, pp. 2217-2218.

COST INFORMATION:

Cost information has not been obtained.

Soil/Sediment Samples
Laboratory Method

Reacts with water.

Water Samples Laboratory Method

Reacts with water.

HYDRAZINE (CAS Number 302-01-2)

RQ: 0.454 kg

SAFETY INFORMATION: Explosion hazard. Potentially carcinogenic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Hydrazines and Other Compounds," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Hydrazines and Other Compounds," Appendix B.

Air Samples Laboratory Method

METHOD SUMMARY:

Colorimetry may be used for the analysis of hydrazine in air samples. A known volume of air is drawn through a bubbler containing 0.1 M hydrochloric acid. The resulting solution is made alkaline with 1.2 M sodium hydroxide and reacted with p-dimethylaminobenzaldehyde. Glacial acetic acid is added, followed by colorimetric analysis at 480 nm.

INTERFERENCES:

Hydrazine derivatives can interfere with the analysis.

QUALITY CONTROL:

A method blank should be processed with each 10 samples.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 0.6 to 3.4 milligrams per cubic meter. Precision and accuracy information is furnished.

REFERENCES:

- U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set Q, Method S237; PB-258 435 (NTIS), U.S. DHEW: Cincinnati, September 1976.
- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, Method S237; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$20-25 (list), plus sampling costs.

See also: "Hydrazines," Appendix B.

Soil/Sediment Samples
Laboratory Method

Water Samples Laboratory Method

METHOD SUMMARY:

Hydrazine in water samples can be determined by gas chromatography with nitrogen/phosphorus detection (GC/NPD). The sample is treated with an excess of acetone to form acetone azine and is extracted three times with methylene chloride. The extract is dried with sodium sulfate, concentrated, and an aliquot is injected into the GC.

INTERFERENCES:

Aldehydes and unsymmetrical ketones can react with hydrazine to form synamd anti-isomers that can cause interferences with each other.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Accuracy information is furnished. This method is sensitive to concentration levels of hydrazine as low as 0.1 micrograms per liter of sample.

REFERENCE:

Selim, S.; Warner, C. R. "Residue Determination of Hydrazine in Water by Derivatization and Gas Chromatography," J. Chromatogr., 166, 1978, pp. 507-511.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$175-215 (list).

HYDROCHLORIC ACID (CAS Number 7647-01-0)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

Analysis for hydrochloric acid in air may be performed by use of a detector tube. A 1-liter air sample is collected with a hand-operated bellows pump and analyzed with a hydrochloric acid gas detector tube (Draeger® CH 29501 or equivalent). A positive result is indicated by a color change in the tube from blue to yellow.

INTERFERENCES:

A very high relative humidity may cause the hydrochloric acid determination to be low. Chlorine will produce a pale blue discoloration, but it will still be possible to measure the hydrochloric acid concentration.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is sensitive to concentration levels of approximately 1.5 to 15 milligrams per cubic meter.

REFERENCES:

National Draeger, Inc., <u>Draeger® Detector Tube Handbook</u>, "Air Investigations and Technical Gas Analysis with Draeger® Tubes", 4th ed.; Draeger: Pittsburgh, August 1979.

National Draeger, Inc., Bulletin No. ND-105; Draeger: Pittsburgh, January 1, 1982.

National Draeger, Inc., Measurements with Draeger® Tubes in the Threshold Limit Value Range, Draeger® Gas Analysis Product Information, Leaflet 4340.3e; Draeger: Pittsburgh, December 1981.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$13-20 (list).

See also: "Inorganic Acids," Appendix B.

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Fluorides and Other Compounds," Appendix B.

Air Samples
Laboratory Method

METHOD SUMMARY:

Analysis for hydrochloric acid in air can be performed by collection of the analyte from a known volume of air, using a midget bubbler charged with a sodium acetate solution, followed by analysis of the resulting solution with a chloride ion-specific electrode. This method is not specific to hydrogen chloride, but measures all chlorides.

INTERFERENCES:

Bromide, iodide, and cyanide ions can interfere. High concentrations of silver-complexing ligands (e.g. thiosulfate, ammonia) can interfere. The presence of sulfide can poison the electrode, and sulfide ions must therefore be removed by addition of cadmium carbonate.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), has been evaluated and reviewed by that agency, and validated over the range of 3.5 to 14 milligrams per cubic meter in a 15-liter sample. Precision and accuracy information is furnished.

REFERENCES:

- U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set R, Method S246, PB-262 403 (NTIS). U.S. DHEW: Cincinnati, December 1976.
- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, Method S246; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$15, plus sampling costs.

Note: NIOSH Method P&CAM 310 (silica tube collection, eluent desorption, analyses by ion chromatography) is applicable. (Reference furnished by reviewer.)

See also: "Inorganic Acids," Appendix B.

See also: "Chlorides," Appendix B.

Soil/Sediment Samples
Laboratory Method

Water Samples Laboratory Method

See "Chlorides," Appendix B.

HYDROGEN CYANIDE (CAS Number 74-90-8)

RQ: 4.54 kg

SAFETY INFORMATION: Exhibits high toxicity (oral inhalation). Releases toxic

cyanide ion in water. Flash point -17.7°C (0°F).

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

Analysis for hydrogen cyanide in air may be performed by use of a detector tube. A 0.5-liter air sample is collected and analyzed with a hand-operated bellows pump and a hydrocyanic acid gas detector tube (Draeger® CH 25701 or equivalent). A positive result is indicated by a color change in the tube from yellow to red. The method is based on liberation of hydrochloric acid from mercuric chloride, and subsequent reaction of the hydrochloric acid with methyl red.

INTERFERENCES:

Both acidic and basic interfering gases (hydrogen sulfide, hydrogen chloride, sulfur dioxide, nitrogen dioxide, and ammonia) are retained in the precleanse layer of the tube and will not affect the hydrogen cyanide determination.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is senesitve to concentration level of approximately 2 to 35 milligrams per cubic meter.

REFERENCES:

Snyder, R. E.; Jankin, M. E.; McKissick, A. M. <u>Development of Hazardous Toxic</u> Wastes Analytical Screening Procedures, Atlantic Research Corporation for U.S. Army Medical Research and Development Command, Fort Detrick, Maryland, July 16, 1982, p. 4.

National Draeger, Inc., <u>Draeger® Detector Tube Handbook</u>, "Air Investigations and Technical Gas Analysis with Draeger® Tubes", 4th ed.; Draeger: Pittsburgh, August 1979.

National Draeger, Inc., Bulletin No. ND-105; Draeger: Pittsburgh, January 1, 1982.

National Draeger, Inc., Measurements with Draeger® Tubes in the Threshold Limit Value Range, Draeger® Gas Analysis Product Information, Leaflet 4340.3e; Draeger Pittsburgh, December 1981.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$13-20 (list).

See also: "Fumigants," Appendix B.

Soil/Sediment Samples Field Method

See "Cyanides," Appendix B.

Water Samples Field Method

See "Cyanides," Appendix B. See "Halides," Appendix B.
See "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples Laboratory Method 1

METHOD SUMMARY:

Infrared absorption spectrometry can be used to determine the concentration of hydrogen cyanide in air samples. The absorbance at 3000 nm (3333 $\rm cm^{-1}$) is measured, using a 10-meter pathlength cell. Air can be sampled in a Saran® or Mylar® plastic bag and approximately 5 liters of the sample drawn into an evacuated cell. Continuous monitoring of air flowing through the cell is possible if the spectrometer is set to one particular wavelength. Some vapors can be adsorbed on silica gel and quantitatively desorbed into the absorption cell later. Tentative qualitative identification of compounds can be made based on a full infrared absorption spectrum.

INTERFERENCES:

Compounds present in the sample that absorb infrared radiation at the wavelength of interest will interfere.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The sensitivity of the method, using a 10-meter pathlength cell, is 50 milligrams per cubic meter or 50 parts per million. No precision or accuracy information is furnished.

REFERENCE:

American Public Health Association, "Infrared Absorption Spectroscopy," In Methods of Air Sampling and Analysis, 2nd ed., M. Katz, Ed.; American Public Health Association: Washington, 1977, pp. 79-84.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$75-85 (list), plus sampling costs.

Air Samples Laboratory Method 2

METHOD SUMMARY:

Hydrogen cyanide in air samples can be measured using an ion selective electrode. A known volume of air is drawn through a sodium hydroxide-charged tube, then the analyte is desorbed. Alternatively, the sample may be passed through a filter and midget bubbler containing 0.1 N potassium hydroxide, which is later diluted. The resulting solution is analyzed with a cyanide ion selective electrode.

INTERFERENCES:

Sulfide ions irreversibly poison the electrode and must be removed if present in the sample. Some halogen and metal ions may interfere.

QUALITY CONTROL:

A blank should be processed with every 10 samples. Recovery and blank corrections should be included in the calculations.

EPA/TECHNICAL STATUS:

The filter/bubbler method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 5.2 to 21.0 milligrams per cubic meter, using a 12-liter sample. The sodium hydroxide-charged tube method is sensitive to levels above approximately 500 micrograms per cubic meter. Precision and accuracy information is furnished.

REFERENCES:

Cadoff, B. C.: Taylor, J. K. <u>Development of a Solid Sorption Tube and Analytical Procedures for Hydrogen Cyanide in the Workplace Atmosphere</u>, PB-253 228, (NTIS), U.S. Department of Health, Education, and Welfare: Cincinnati, 1976.

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, Method S288; Publication No. 78-175, U.S. DHEW: Cincinnati, August 1978.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$25 (list), plus sampling costs.

Soil/Sediment Samples
Laboratory Method

See "Cyanides," Appendix B.

Water Samples Laboratory Method

HYDROGEN FLUORIDE (CAS Number 7664-39-3)

RQ: 45.4 kg

SAFETY INFORMATION: Exhibits acute and chronic toxicity.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

Analysis for hydrogen fluoride in air may be performed by use of a detector tube. A 2-liter air sample is collected with a hand-operated bellows pump and analyzed with a hydrogen fluoride gas detector tube (Draeger® CH 30301 or equivalent). A positive result is indicated by a color change in the tube from pale blue to pale pink. This method is based on the formation of quinalizarin from a zirconium hydroxide and 1,2,5,8-tetrahydroxyanthraquinone lake and hydrogen fluoride.

INTERFERENCES:

A high relative atmospheric humidity may cause the hydrogen fluoride determination to be low.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is sensitive to concentration levels of approximately 1 to 15 milligrams per cubic meter.

REFERENCES:

National Draeger, Inc., <u>Draeger® Detector Tube Handbook</u>, "Air Investigations and Technical Gas Analysis with Draeger® Tubes", 4th ed.: Draeger: Pittsburgh, August 1979.

National Draeger, Inc., Bulletin No. ND-105; Draeger: Pittsburgh, January 1, 1982.

National Draeger, Inc., Measurements with Draeger® Tubes in the Threshold Limit Value Range, Draeger® Gas Analysis Product Information, Leaflet 4340.3e; Draeger: Pittsburgh, December 1981.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$13-20 (list).

See also: "Fluorides," Appendix B.

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Fluorides and Other Compounds," Appendix B.

Air Samples Laboratory Method

METHOD SUMMARY:

Analysis for hydrogen fluoride in air is performed by passage of 45 liters of air through a midget bubbler containing 0.1 N sodium hydroxide, followed by sample dilution with Total Ionic Strength Activity Buffer (TISAB) and analysis of the resulting solution for fluoride ions with a fluoride ion specific electrode. This method is not specific for hydrogen fluoride, but measures the total fluoride content of the sample.

INTERFERENCES:

Very high amounts of fluoride-complexing metals can interfere with the analysis.

QUALITY CONTROL:

A method blank must be processed and results adjusted accordingly.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 1.3 to 4.5 milligrams per cubic meter in a 45-liter sample. Precision and accuracy information is furnished.

REFERENCES:

- U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set L, Method S176; PB-250 159 (NTIS), U.S. DHEW: Cincinnati, January 1976.
- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, Method P&CAM 117; Publication No. 75-121, U.S. DHEW: Cincinnati, 1974.

- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 117; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.
- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, Method S176; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is \$10-20 (list), plus sampling costs.

See also: "Fluorides," Appendix B.

See also: "Fluorides and Hydrogen Fluoride," Appendix B.

Soil/Sediment Samples
Laboratory Method

Water Samples Laboratory Method

See "Fluorides," Appendix B.

HYDROGEN SULFIDE (CAS Number 7783-06-4)

RQ: 45.4 kg

SAFETY INFORMATION: Flammable gas. Toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method 1

METHOD SUMMARY:

Analysis for hydrogen sulfide in air may be performed by use of a detector tube. A 0.1- to 1-liter air sample is collected and analyzed with a hand-operated bellows pump and a hydrogen sulfide gas detector tube (Draeger® 67 19001 or equivalent). A positive result is indicated by a color change in the tube from white to pale brown. The method is based on the formation of lead sulfide.

INTERFERENCES:

High concentrations of sulfur dioxide may cause the hydrogen sulfide determination to be too high, although sulfur dioxide alone will not discolor the tube.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is sensitive to concentration levels of approximately 1 to 300 milligrams per cubic meter for the prescribed sample size.

REFERENCES:

National Draeger, Inc., <u>Draeger® Detector Tube Handbook</u>, "Air Investigations and Technical Gas Analysis with Draeger® Tubes", 4th ed.: Draeger: Pittsburgh, August 1979.

National Draeger, Inc., Bulletin No. ND-105; Draeger: Pittsburgh, January 1, 1982.

National Draeger, Inc., Measurements with Draeger® Tubes in the Threshold Limit Value Range, Draeger® Gas Analysis Product Information, Leaflet 4340.3e; Draeger: Pittsburgh, December 1981.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$13-20 (list).

Air Samples Field Method 2

METHOD SUMMARY:

Analysis for hydrogen sulfide in air can be performed by photorateometric analysis, which utilizes the color developed from the reaction of hydrogen sulfide with lead acetate-impregnated paper to indicate the concentration of hydrogen sulfide in air.

INTERFERENCES:

There are no reported interferences.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

The method is sensitive to one part per billion hydrogen sulfide and requires use of an electrical monitoring device [Model 825 R-d Rateometric Analyzer, Houston Atlas, Inc., Houston, Texas].

REFERENCE:

Kimbell, C. L. "Atmospheric Monitoring for Hydrogen Sulfide by Photorateometric Analysis" In <u>Toxic Materials in the Atmosphere</u>, STP-786, American Society for Testing and Materials: Philadelphia, 1981, pp. 60-69.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$8 (list), plus sampling costs.

See also: "Organic Vapors," Appendix B.

Soil/Sediment Samples Field Method

See "Sulfides," Appendix B.

Water Samples Field Method

See "Sulfides," Appendix B.

Air Samples Laboratory Method 1

METHOD SUMMARY:

Analysis for hydrogen sulfide in air can be performed spectrophotometrically. Two liters of air are aspirated through an aqueous cadmium hydroxide suspension, to which Stractan 10° 0 is added to minimize photo-decomposition. The collected sulfide is determined by spectrophotometric measurement of the methylene blue produced by the reaction of the sulfide with N,N-dimethyl-p-phenylenediamine and ferric chloride.

INTERFERENCES:

Strong reducing agents (e.g. sulfur dioxide) inhibit color development. High concentrations of nitrogen dioxide interfere, and ozone can cause a negative interference.

QUALITY CONTROL:

A method blank should be analyzed with each set of samples.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute of Occupational Safety and Health (NIOSH), and validated over the range of 8.5 to 63 milligrams per cubic meter in 2 liters of air. Precision and accuracy information is furnished.

REFERENCES:

- U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set A, Method S4; PB-245 850 (NTIS), U.S. DHEW: Cincinnati, October 1975.
- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, Method S4; Publication No. 77-157-B, U.S. DHEW: Cincinnati, 1977.
- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 126; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost information has not been obtained.

See also: "Sulfur-Containing Gases," Appendix B.

Air Samples Laboratory Method 2

METHOD SUMMARY:

Hydrogen sulfide in air samples can be measured by gas chromatography. A known volume of air is drawn through a desiccant tube, then through a tube containing molecular sieve that traps the hydrogen sulfide. The hydrogen sulfide is released by thermal desorption, and the sample is analyzed by gas chromatography with flame photometric detection (GC/FPD).

INTERFERENCES:

Humidity interferes, but its effect is eliminated by use of the desiccant tube. Any sulfur-containing compound with the same retention time as hydrogen sulfide can interfere.

QUALITY CONTROL:

Since no internal standard is used in this method, standard gases must be analyzed at the same time the sample analysis is done.

EPA/TECHNICAL STATUS:

This method is proposed by the National Institute for Occupational Safety and Health (NIOSH) for use over the range of 15 to 60 milligrams per cubic meter for a 5-liter air sample. Precision and accuracy information is furnished.

REFERENCE:

U.S. Department of Health and Human Services, NIOSH Manual of Analytical Methods, Vol. 6, Method P&CAM 296; Publication No. 80-125, U.S. DHHS: Cincinnati, August 1980.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$100-120 (list), plus sampling costs.

See also: "Sulfur-Containing Gases," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Sulfides," Appendix B.

Water Samples Laboratory Method

See "Sulfides," Appendix B.

LEAD (CAS Number 7439-92-1)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Lead and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Lead," Appendix B.

Water Samples Field Method

See "Lead," Appendix B.

Air Samples Laboratory Method

See "Lead," Appendix B.

See "Lead and Cadmium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Lead," Appendix B.

Water Samples Laboratory Method

LEAD ACETATE (CAS Number 301-04-2)

RQ: 2270 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Lead and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Lead," Appendix B.

Water Samples Field Method

See "Lead," Appendix B.

Air Samples Laboratory Method

See "Lead," Appendix B. See "Lead and Cadmium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Lead," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B. See "Lead," Appendix B.

LEAD ARSENATE (CAS Numbers 7784-40-9, 7645-25-2, 10102-48-4)

RQ: 2270 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic. CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Lead and Compounds (Particulate)." Appendix B.

Soil/Sediment Samples Field Method

See "Arsenic," Appendix B. See "Lead," Appendix B.

> Water Samples Field Method

See "Lead," Appendix B.

Air Samples Laboratory Method

See "Arsenic," Appendix B.

See "Lead," Appendix B.

See "Lead and Cadmium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Arsenic," Appendix B. See "Lead," Appendix B.

> Water Samples Laboratory Method

See "Arsenic," Appendix B. See "Lead," Appendix B.

LEAD CHLORIDE (CAS Number 7758-95-4)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Lead and Compounds (Particulate), " Appendix B.

Soil/Sediment Samples Field Method

See "Lead," Appendix B.

Water Samples Field Method

See "Halides, Cyanides, and Other Compounds," Appendix B. See "Lead," Appendix B.

Air Samples Laboratory Method

See "Chlorides," Appendix B.

See "Lead," Appendix B.

See "Lead and Cadmium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Lead," Appendix B.

Water Samples Laboratory Method

See "Chlorides," Appendix B. See "Lead," Appendix B.

LEAD FLUOBORATE (CAS Number 13814-96-5)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Lead and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Lead," Appendix B.

Water Samples Field Method

METHOD SUMMARY:

Field analysis for lead fluoborate in water samples can be performed by colorimetric analysis for boron. A 2-milliliter sample is treated with concentrated sulfuric acid and a carmine color reagent, and the intensity of the resulting bluish-red or blue color is measured with a portable spectrophotometer at 585 nm.

INTERFERENCES:

No specific interferences are given. With this method no specific boron-containing compound can be identified, only the total boron content of the sample is determined.

QUALITY ASSURANCE:

No quality assurance procedures are given.

SENSITIVITY:

The method is applicable to concentrations above approximately 5 milligrams per liter.

REFERENCE:

Hach Chemical Co., Procedures, Chemical Lists and Glassware for Water and Wastewater Analysis, 2nd ed.; Hach: Ames, Iowa, 1978, pp. 2-19. 2-20.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$25-40 (list).

See also: "Lead," Appendix B.

Air Samples Laboratory Method

See "Lead," Appendix B. See "Lead and Cadmium," Appendix B.

Soil/Sediment Samples
Laboratory Method

METHOD SUMMARY:

Soil/sediment samples may be analyzed for lead fluoborate by inductively coupled plasma atomic spectrometry (ICP) or colorimetric analysis for boron. A 2-gram sample is digested with nitric acid and hydrogen peroxide and analyzed. For colorimetric analysis, 1 milliliter of the digested sample is acidified and evaporated in the presence of curcumin, and the resultant color is measured photometrically. These methods are not specific for lead fluoborate, but the total boron concentration of the sample is measured.

INTERFERENCES:

High concentrations of calcium and magnesium compounds in the sample may make it necessary to pass the sample through a cation exchange resin.

QUALITY CONTROL:

For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods when using ICP. Using either method, a reference standard should be analyzed weekly and at least 10 percent of the analyses should be duplicates and/or standards.

EPA/TECHNICAL STATUS:

These methods are in use in the CERCLA program and are approved for the National Pollutant Discharge Elimination System (NPDES). Precision and accuracy information is furnished.

REFERENCES:

- U.S. Environmental Protection Agency Chemical Analytical Services for Low and Medium Concentration Inorganics in All Media, Exhibit D, Invitation for Bid (Solicitation Number WA 82-A072), U.S. EPA: Washington, April 23, 1982.
- U.S. Environmental Protection Agency Methods for Chemical Analysis of Water and Wastes Method 212.3; EPA-600/4-79-020, U.S. EPA: Cincinnati, March 1979.

U.S. Environmental Protection Agency <u>Technical Additions to Methods for Chemical Analyses of Water and Wastes</u>, Method 200.7; EPA-600/4-82-055, U.S. EPA: Washington, 1982.

Federal Register, 44 (233), December 3, 1979, pp. 69559-69564.

COST INFORMATION:

Cost per sample for analysis by ICP is approximately \$20 (list); by the colorimetric method, it is approximately \$35-45 (list).

See also: "Lead," Appendix B.

Water Samples Laboratory Method

METHOD SUMMARY:

Colorimetric analysis or inductively coupled plasma atomic spectrometric (ICP) analysis for boron may be used to analyze water samples for lead fluoborate. A 100-milliliter sample is digested with nitric acid and hydrogen peroxide and analyzed. For colorimetric analysis, 1 milliliter of the digested sample is acidified and evaporated in the presence of curcumin and the resultant color is measured photometrically. These methods are not specific for lead fluoroborate, but the total boron content of the sample is weighed.

INTERFERENCES:

Total calcium and magnesium hardness exceeding 100 milligrams per liter as calcium carbonated may make it necessary to pass the sample through a cation exchange resin.

QUALITY CONTROL:

For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods. Using either method, a reference standard should be analyzed weekly and at least 10 percent of the analyses should be duplicates and/or standards.

EPA/TECHNICAL STATUS:

These methods are in use in the CERCLA program and are approved for the National Pollutant Discharge Elimination System (NPDES). Precison and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Chemical Analytical Services for Low and Medium Concentration Inorganics in All Media, Exhibit D; Invitation for Bid (Solicitation Number, WA 82-A072), U.S. EPA: Washington, April 23, 1982.

- U.S. Environmenal Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 212.3; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.
- U.S. Environmental Protection Agency <u>Technical Additions to Methods for Chemical Analyses of Water and Wastes</u>, Method 200.7; EPA-600/4-82-055, U.S. EPA: Washington, 1982.

Federal Register, 44 (233), December 3, 1979, pp. 69559-69564.

COST INFORMATION:

Cost per sample for analysis by ICP is approximately \$10 (list); by the colorimetric method, it is approximately \$35-40 (list).

See also: "Lead," Appendix B.

LEAD FLUORIDE (CAS Number 7783-46-2)

RQ: 454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Fluorides," Appendix B. See "Lead and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Lead," Appendix B.

Water Samples Field Method

See "Fluorides and Other Compounds," Appendix B. See "Lead," Appendix B.

Air Samples Laboratory Method

See "Fluorides," Appendix B.

See "Fluorides and Hydrogen Fluoride," Appendix B.

See "Lead," Appendix B.

See "Lead and Cadmium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Lead," Appendix B.

Water Samples Laboratory Method

See "Fluorides," Appendix B. See "Lead," Appendix B.

LEAD IODIDE (CAS Number 10101-63-0)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Lead and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Lead," Appendix B.

Water Samples Field Method

See "Halides, Cyanides, and Other Compounds," Appendix B. See "Lead," Appendix B.

Air Samples Laboratory Method

See "Lead," Appendix B.

See "Lead and Cadmium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Lead," Appendix B.

Water Samples Laboratory Method

See "Lead," Appendix B.

See "Iodides and Bromides," Appendix B.

LEAD NITRATE (CAS Number 10099-74-8)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Lead and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Lead," Appendix B.

Water Samples Field Method

See "Lead," Appendix B. See "Nitrate Ions," Appendix B.

Air Samples Laboratory Method

See "Lead," Appendix B.

See "Lead and Cadmium," Appendix B.

See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Lead," Appendix B.

See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Lead," Appendix B. See "Nitrates," Appendix B.

LEAD PHOSPHATE (CAS Number 7446-27-7)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Lead and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Lead," Appendix B.

Water Samples Field Method

See "Lead," Appendix B.

Air Samples Laboratory Method

See "Lead," Appendix B.

See "Lead and Cadmium," Appendix B.

See "Phosphorus," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Lead," Appendix B.

See "Phosphorus," Appendix B.

Water Samples Laboratory Method

See "Lead," Appendix B.

See "Phosphorus," Appendix B.

LEAD STEARATE (CAS Numbers 7428-48-0, 1072-35-1, 56189-09-4)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Lead and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Lead," Appendix B.

Water Samples Field Method

See "Lead," Appendix B.

Air Samples Laboratory Method

See "Lead," Appendix B.

See "Lead and Cadmium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Lead," Appendix B.

Water Samples Laboratory Method

LEAD SUBACETATE (CAS Number 1335-32-6)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronially toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Lead and Compounds (Particulate)." Appendix B.

Soil/Sediment Samples Field Method

See "Lead," Appendix B.

Water Samples Field Method

See "Lead," Appendix B.

Air Samples Laboratory Method

See "Lead," Appendix B.

See "Lead and Cadmium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Lead," Appendix B.

Water Samples Laboratory Method

LEAD SULFATE (CAS Numbers 15739-80-7, 7446-14-2)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Lead and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Lead," Appendix B.

Water Samples Field Method

See "Lead," Appendix B.

Air Samples Laboratory Method

See "Lead," Appendix B.

See "Lead and Cadmium," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Lead," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Water Samples
Laboratory Method

LEAD SULFIDE (CAS Number 1314-87-0)

RQ: 2270 kg

SAFETY INFORMATION: Forms toxic H2S when in contact with acids. Potentially

chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Lead and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Lead," Appendix B. See "Sulfides," Appendix B.

Water Samples Field Method

Insoluble in water.

Air Samples
Laboratory Method

METHOD SUMMARY:

Lead sulfide in air samples can be measured by X-ray diffraction. A known volume of air is drawn through a membrane filter. The filter is dissolved in tetrahydrofuran then redeposited on a silver membrane filter. The filter samples are scanned qualitatively by X-ray powder diffraction to determine the presence of lead sulfide. The mass of lead sulfide present is then determined by measuring the diffraction peak intensity for the analyte and for the silver filter.

INTERFERENCES:

Using copper K α X-ray radiation, lead oxide, lead sulfate, and copper iron sulfide will interfere with the primary lead sulfide peak. Lead oxide and lead sulfate will also interfere with the secondary lead sulfide peak. The tertiary lead sulfide peak interferes with the secondary silver peak.

QUALITY CONTROL:

Method blanks and standards are processed along with the samples.

EPA/TECHNICAL STATUS:

This is an operational method, as classified by the National Institute for Occupational Safety and Health (NIOSH), for use over the range of 0.06 to 40 milligrams per cubic meter in a 500-liter sample. Precision and accuracy information is furnished.

REFERENCE:

U.S. Department of Health and Human Services, NIOSH Manual of Analytical Methods, Vol. 7, Method P&CAM 350; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

COST INFORMATION:

Cost information has not been obtained.

See also: "Lead," Appendix B.

See also: "Lead and Cadmium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Lead," Appendix B. See "Sulfides," Appendix B.

Water Samples Laboratory Method

Insoluble in water.

LEAD THIOCYANATE (CAS Number 592-87-0)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Lead and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Lead," Appendix B.

Water Samples Field Method

See "Lead," Appendix B.

Air Samples Laboratory Method

See "Lead," Appendix B. See "Lead and Cadmium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Lead," Appendix B.

Water Samples Laboratory Method

See "Lead," Appendix B. See "Thiocyanates," Appendix B.

LITHIUM CHROMATE (CAS Number 14307-35-8)

RQ: 454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Chromium," Appendix B.

Soil/Sediment Samples Field Method

See "Chromium," Appendix B.

Water Samples Field Method

See "Chromates," Appendix B.
See "Chromium," Appendix B.
See "Hexavalent Chromium," Appendix B.

Air Samples Laboratory Method

METHOD SUMMARY:

Atomic absorption (AA) spectrometric analysis for lithium may be used to analyze air samples for lithium chromate. Samples are collected on cellulose membrane filters. The filter medium is digested with nitric acid and the digest is diluted and analyzed by AA. This method does not differentiate between lithium-containing compounds, but measures the total lithium content of the sample.

INTERFERENCES:

Cesium should be added to samples and standards to control ionization interference. The method of standard additions and/or background monitoring may be needed to correct for interferences.

QUALITY CONTROL:

A minimum of 1 filter blank for every 10 filter samples must be analyzed. Standard solutions should match the sample matrix as closely as possible. Standards should be analyzed in duplicate.

EPA/TECHNICAL STATUS:

This method is classified as operational by the National Institute of Occupational Safety and Health (NIOSH) for use over the range of 4.2 to 84 micrograms per cubic meter of air in a 240-liter sample. Precision information is furnished.

REFERENCE:

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 173; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$10-20 (list), plus sampling costs.

See also: "Chromic Acid and Chromates," Appendix B.

See also: "Chromium," Appendix B.

See also: "Chromium, Hexavalent," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Chromium," Appendix B.

See "Chromium, Hexavalent," Appendix B.

Water Samples Laboratory Method

See "Chromium," Appendix B.

See "Chromium, Hexavalent," Appendix B.

MERCURIC CYANIDE (CAS Number 592-04-1)

RQ: 0.454 kg

SAFETY INFORMATION: Releases toxic cyanide ions in water. Exhibits chronic

toxicity.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Cyanides," Appendix B.

Water Samples Field Method

See "Cyanides," Appendix B.

See "Halides, Cyanides, and Other Compounds," Appendix B.

See "Mercury," Appendix B.

Air Samples Laboratory Method

See "Cyanides," Appendix B. See "Mercury," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Cyanides," Appendix B. See "Mercury," Appendix B.

Water Samples Laboratory Method

See "Cyanides," Appendix B. See "Mercury," Appendix B.

MERCURIC NITRATE (CAS Number 10045-94-0)

RQ: 4.54 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Mercury," Appendix B. See "Nitrate Ions," Appendix B.

Air Samples Laboratory Method

See "Mercury," Appendix B.
See "Nitrates," Appendix B.
See "Nitrates and Sulfates," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Mercury," Appendix B.
See "Nitrates," Appendix B.
See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Mercury," Appendix B. See "Nitrates," Appendix B.

MERCURIC SULFATE (CAS Number 7783-35-9)

RQ: 4.54 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Mercury," Appendix B.

Air Samples Laboratory Method

See "Mercury," Appendix B.

See "Nitrates and Sulfates," Appendix B. See "Sulfates (Particulate)," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Mercury," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Mercury," Appendix B. See "Sulfates," Appendix B.

MERCURIC THIOCYANATE (CAS Number 592-85-8)

RQ: 4.54 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Mercury," Appendix B.

Air Samples Laboratory Method

See "Mercury," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Mercury," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B.

See "Mercury," Appendix B. See "Thiocyanates," Appendix B.

MERCUROUS NITRATE (CAS Numbers 7782-86-7, 10415-75-5)

RQ: 4.54 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Mercury," Appendix B.

Air Samples Laboratory Method

See "Mercury," Appendix B.
See "Nitrates," Appendix B.
See "Nitrates and Sulfates," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Mercury," Appendix B.
See "Nitrates," Appendix B.
See "Nitrates and Sulfates," Appendix B.

Water Samples
Laboratory Method

See "Mercury," Appendix B. See "Nitrates," Appendix B.

MERCURY (CAS Number 7439-97-6)

RQ: 0.454 kg

SAFETY INFORMATION: Exhibits toxicity (inhalation). Exhibits chronic

toxicity.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

Analysis for mercury vapor in air may be performed by use of a detector tube. A 0.1- to 2-liter air sample is collected and analyzed with a hand-operated bellows pump and a mercury vapor detector tube (Draeger® CH 23101 or equivalent). A positive result is indicated by a color change in the tube from pale yellowish-grey to pale yellowish-orange. The method is based on the reaction of mercury with copper(I)iodide to form a copper-mercury complex.

INTERFERENCES:

Chlorine will cause the mercury determination to be low.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is sensitive to concentration levels of approximately 0.1 to 2 milligrams per cubic meter.

REFERENCES:

National Draeger, Inc., <u>Draeger® Detector Tube Handbook</u>, "Air Investigations and Technical Gas Analysis with Draeger® Tubes", 4th ed.; Draeger: Pittsburgh, August 1979.

National Draeger, Inc., Bulletin No. ND-105; Draeger: Pittsburgh, January 1, 1982.

National Draeger, Inc., <u>Measurements with Draeger® Tubes in the Threshold Limit Value Range</u>, Draeger® Gas Analysis Product Information, Leaflet 4340.3e; Draeger: Pittsburgh, December 1981.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$13-20 (list).

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Mercury," Appendix B.

Air Samples Laboratory Method

METHOD SUMMARY:

Mercury in air samples can be measured by cold vapor atomic absorption (AA) spectrometry. Elemental mercury vapor is collected on silver wool or silvered Chromosorb P and released by heating. The released mercury is swept through the absorption cell of an AA spectrometer, and the response at 253.7 nm is measured. Calibration of the instrument with a series of five standards is required.

INTERFERENCES:

High levels of chlorine and sulfur dioxide will poison the silver wool. A tube packed with scarite® placed before the collector will remove the chlorine. Hydrogen sulfide may interfere.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 0.046 to 0.18 milligrams per cubic meter, using a 3-liter sample. The American Public Health Association considers the procedure applicable to the determination of elemental mercury in the range of 0.02 to 10.0 micrograms per cubic meter, at a collection flow rate of 100 to 200 cubic centimeters per minute over a 24-hour period. The limit of detection is approximately 0.001 microgram mercury. Precision and accuracy information is furnished.

REFERENCES:

American Public Health Association, Methods of Air Sampling and Analysis, 2nd ed., M. Katz, Ed.; APHA: Washington, 1977, pp. 488-492.

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, Method S199; Publication No. 78-175, U.S. DHEW: Cincinnati, August 1978.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$18-50 (list), plus sampling costs.

See also: "Mercury," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Mercury," Appendix B.

Water Samples Laboratory Method

See "Mercury," Appendix B.

MERCURY FULMINATE (CAS Number 628-86-4)

RQ: 0.454 kg

SAFETY INFORMATION: Explosive. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Ammonia," Appendix B. See "Mercury," Appendix B.

Air Samples
Laboratory Method

See "Mercury," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Mercury," Appendix B.

Water Samples Laboratory Method

See "Ammonia," Appendix B. See "Mercury," Appendix B.

NICKEL (CAS Number 7440-02-0)

RQ: 0.454 kg

SAFETY INFORMATION: Exhibits toxicity (oral). Potentially carcinogenic.

Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Nickel and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Nickel Ions," Appendix B.

Air Samples
Laboratory Method

See "Nickel," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Nickel," Appendix B.

Water Samples Laboratory Method

See "Nickel," Appendix B.

NICKEL AMMONIUM SULFATE (CAS Number 15699-18-0)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

> CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Nickel and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Ammonia," Appendix B. See "Nickel Ions," Appendix B.

See "Volatile Species," Appendix B.

Air Samples Laboratory Method

See "Ammonia," Appendix B. See "Nickel," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Ammonia," Appendix B.

See "Nickel," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Ammonia," Appendix B.

See "Nickel," Appendix B.

NICKEL CHLORIDE (CAS Numbers 7718-54-9, 37211-05-5)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Nickel and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Halides, Cyanides, and Other Compounds," Appendix B. See "Nickel Ions," Appendix B.

Air Samples Laboratory Method

See "Chlorides," Appendix B. See "Nickel," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Nickel," Appendix B.

Water Samples Laboratory Method

See "Chlorides," Appendix B. See "Nickel," Appendix B.

NICKEL CYANIDE (CAS Number 557-19-7)

RQ: 4.54 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Nickel and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Cyanides," Appendix B.

Water Samples Field Method

See "Cyanides," Appendix B. See "Nickel Ions," Appendix B.

Air Samples Laboratory Method

See "Cyanides," Appendix B. See "Nickel," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Cyanides," Appendix B. See "Nickel," Appendix B.

Water Samples Laboratory Method

See "Cyanides," Appendix B. See "Nickel," Appendix B.

NICKEL HYDROXIDE (CAS Number 12054-48-7)

RQ: 454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

Air Samples Field Method

See "Nickel and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Nickel Ions," Appendix B.

Air Samples
Laboratory Method

See "Nickel," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Nickel," Appendix B.

Water Samples Laboratory Method

See "Nickel," Appendix B.

NICKEL NITRATE (CAS Number 14216-75-2)

RQ: 454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Nickel and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Nickel Ions," Appendix B. See "Nitrate Ions," Appendix B.

Air Samples Laboratory Method

See "Nickel," Appendix B.

See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Nickel," Appendix B.

See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Nickel," Appendix B. See "Nitrates," Appendix B.

NICKEL SULFATE (CAS Number 7786-81-4)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Nickel and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Nickel Ions," Appendix B.

Air Samples Laboratory Method

See "Nickel," Appendix B.

See "Nitrates and Sulfates," Appendix B.

See "Sulfates (Particulate)," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Nickel," Appendix B.

See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Nickel," Appendix B. See "Sulfates," Appendix B.

NICKEL TETRACARBONYL (CAS Number 13463-39-3)

RQ: 0.454 kg

SAFETY INFORMATION: Exhibits toxicity (inhalation). Flash point -4°F.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

Analysis for nickel tetracarbonyl in air may be performed by use of a detector tube. A 2-liter air sample is collected and analyzed with a hand-operated bellows pump and a nickel tetracarbonyl gas detector tube (Draeger® CH 19501 or equivalent). A positive result is indicated by a color change in the tube from pale brown to pink, caused by the reaction of the nickel tetracarbonyl with iodine and a dioxime compounds.

INTERFERENCES:

Hydrogen sulfide and sulfur dioxide may lower the nickel tetracarbonyl indication and iron pentacarbonyl may cause a brown discoloration.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is sensitive to concentration levels of approximately 1 to 10 milligrams per cubic meter.

REFERENCES:

Snyder, R. E.; Jankin, M. E.; McKissick, A. M. <u>Development of Hazardous Toxic Wastes Analytical Screening Procedures</u>, Atlantic Research Corporation for U.S. Army Medical Research and Development Command, Fort Detrick, Maryland, July 16, 1982, p. 4.

National Draeger, Inc., <u>Draeger® Detector Tube Handbook</u>, "Air Investigations and Technical Gas Analysis with Draeger® Tubes", 4th ed.; Draeger: Pittsburgh, August 1979.

National Draeger, Inc., Bulletin No. ND-105; Draeger: Pittsburgh, January 1, 1982.

National Draeger, Inc., Measurements with Draeger® Tubes in the Threshold Limit Value Range, Draeger® Gas Analysis Product Information, Leaflet 4340.3e; Draeger: Pittsburgh, December 1981.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$13-20 (list).

Soil/Sediment Samples Field Method

Water Samples Field Method

Air Samples
Laboratory Method

METHOD SUMMARY:

Atomic absorption spectrometry may be used for the analysis of nickel tetracarbonyl in air samples. The analysis is performed by passage of a known volume of air through an acid-washed charcoal-tube sampling device, followed by desorption of the analyte with dilute nitric acid and analysis of the resulting solution by graphite furnace atomic absorption spectrometry (AAS).

INTERFERENCES:

Particulate nickel compounds will interfere unless a pre-filter is used.

QUALITY CONTROL:

Method blanks and standards are processed with the samples.

EPA/TECHNICAL STATUS:

This method is proposed by the National Institute for Occupational Safety and Health (NIOSH), for use over the range of 2 to 60 micrograms per cubic meter in an 80-liter sample. Precision and accuracy information is furnished.

REFERENCE:

U.S. Department of Health and Human Services, NIOSH Manual of Analytical Methods, Vol. 7, Method P&CAM 344; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$23-55 (list), plus sampling costs.

See also: "Nickel," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Nickel," Appendix B.

Water Samples
Laboratory Method

See "Nickel," Appendix B.

NITRIC ACID (CAS Number 7697-37-2)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

Analysis for nitric acid in air may be performed by use of a detector tube. A 1- to 2-liter air sample is collected and analyzed with a hand-operated bellows pump and a nitric acid gas detector tube (Draeger® 67 28311 or equivalent). A positive result is indicated by a color change in the tube from blue to yellow. The test is based on the reaction of nitric acid with bromophenol blue.

INTERFERENCES:

Nitrogen dioxide will also give a positive result.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

The tube will measure concentrations of approximately 3 to 130 milligrams per cubic meter.

REFERENCES:

National Draeger, Inc.. <u>Draeger® Detector Tube Handbook</u>, "Air Investigations and Technical Gas Analysis with Draeger® Tubes", 4th ed.; Draeger: Pittsburgh, August 1979.

National Draeger, Inc., Bulletin No. ND-105; Draeger: Pittsburgh, January 1, 1982.

National Draeger, Inc., Measurements with Draeger® Tubes in the Threshold Limit Value Range, Draeger® Gas Analysis Product Information, Leaflet 4340.3e; Draeger: Pittsburgh, December 1981.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$13-20 (list).

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Nitrate Ions," Appendix B.

Air Samples Laboratory Method 1

METHOD SUMMARY:

Analysis for nitric acid in air can be performed by sampling air through a 47-millimeter nylon filter at 12 liters per minute, followed by analysis of the filter medium for nitrate ion by ion chromatography (IC). If sampling is performed at 1 liter per minute and analysis performed by a chemiluminescence analyzer, the method can be used for continuous monitoring.

INTERFERENCES:

This analysis responds to any nitrate-containing salt trapped on the filter disc, and is not specific to nitric acid.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

These methods have been evaluated, but are not EPA-approved. Precision and accuracy information is furnished.

REFERENCES:

Forrest, J; Tamer, R.L; Spandau, D.; D'Ottavio, T.; Newman, L. "Determination of Total Inorganic Nitrate Utilizing Collection of Nitric Acid on NaCl-Impregnated Filters," Atmos. Environ., 14, 1980, pp. 137-144.

Current Methods to Measure Atmospheric Nitric Acid and Nitrate Artifacts, R. K. Stevens, Ed.; EPA-600/2-79-051, U.S. Environmental Protection Agency: Research Triangle Park, March 1979.

COST INFORMATION:

The cost per sample for analysis by IC is approximately \$70-80 (list), plus sampling costs; information about the chemiluminescence method has not been obtained.

Air Samples Laboratory Method 2

METHOD SUMMARY:

Nitric acid in air samples may be measured by drawing a known volume of air through a midget impinger containing distilled water, then analyzing the contents of the impinger by direct potentiometry using a nitrate ion selective electrode. This method is not specific for nitric acid, but measures the total concentration of nitrates in the sample.

INTERFERENCES:

Anions, including bromide, chloride, fluoride, iodide, phosphate, and nitrite, will interfere, but use of a buffer solution removes the interferences. However, high concentrations of these species should be avoided.

QUALITY CONTROL:

A calibration standard is analyzed before and after each sample to assure reliable results. A blank should be processed with every 10 samples.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 2.60 to 108 milligrams per cubic meter, using a 180-liter air sample. Precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, Method S319; Publication No. 78-175, U.S. DHEW: Cincinnati, August 1978.

Note: NIOSH Method P & CAM 339 (silica get tube collection, elution, analyses by ion chromatography) is applicable. (Reference provided by reviewer.)

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$10-15 (list), plus sampling costs.

See also: "Inorganic Acids," Appendix B.

See also: "Nitrates," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Nitrates," Appendix B.

Water Samples Laboratory Method

NITRIC OXIDE (CAS Number 10102-43-9)

RQ: 4.54 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples
Laboratory Method 1

METHOD SUMMARY:

Infrared absorption spectrometry can be used to determine the concentration of nitric oxide in air samples. The absorbance at 5250 nm (1905 cm⁻¹) is measured, using a 10-meter pathlength cell. Air can be sampled in a Saran® or Mylar® plastic bag; approximately 5 liters of the sample are drawn into an evacuated cell. Continuous monitoring of air flowing through the cell is possible if the spectrometer is set to one particular wavelength. Some vapors can be adsorbed on silica gel and quantitatively desorbed into the absorption cell later. Tentative qualitative identification of compounds can be made based on a full infrared absorption spectrum.

INTERFERENCES:

Compounds present in the sample that absorb infrared radiation at the wavelength of interest will interfere.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The sensitivity of the method, using a 10-meter pathlength cell, is 200 milligrams per cubic meter or 100 parts per million. No precision or accuracy information is furnished.

REFERENCE:

American Public Health Association, "Infrared Absorption Spectroscopy," In Methods of Air Sampling and Analysis, 2nd ed., M. Katz, Ed.; APHA: Washington, 1977, pp.79-84.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$75-85 (list), plus sampling costs.

Air Samples Laboratory Method 2

METHOD SUMMARY:

Nitric oxide in air samples can be determined spectrometrically. A known volume of air is drawn through a sampling tube containing (in series) triethanolamine-impregnated molecular sieve, Draeger oxidizer, and additional triethanolamine-impregnated molecular sieve. The first section removes ambient nitrogen dioxide. The Draeger oxidizer transforms nitric oxide to nitrogen dioxide, and the last section adsorbs the nitrogen dioxide. The nitrogen dioxide in the last section is desorbed and hydrolyzed in aqueous triethanolamine solution to yield nitrite ion. An aliquot is treated with hydrogen peroxide, sulfanilamide, and N-(1-naphthyl) ethylenediamine dihydrochloride, and the intensity of the color of the reacted nitrite ion is determined on a spectrophotometer at 540 nm.

INTERFERENCES:

High levels of nitrogen dioxide can interfere.

QUALITY CONTROL:

One blank should be processed with every 5 samples.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 13.6 to 58.9 milligrams per cubic meter, using a 1.5-liter sample. Precision and accuracy information is furnished.

REFERENCE:

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, Method S321; Publication No. 78-175, U.S. DHEW: Cincinnati, August 1978.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$55 (list), plus sampling costs.

See also: "Hazardous Organic Emissions," Appendix B.

See also: "Nitrogen Dioxide and Nitric Oxide," Appendix B.

Soil/Sediment Samples Laboratory Method

Water Samples Laboratory Method

METHOD SUMMARY:

Nitric oxide in water samples can be measured by chemiluminescence. The nitric oxide is stripped from the water and the chemiluminescence of the nitric oxide in the gaseous phase is determined.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This procedure is not EPA-approved. Some precision information is furnished.

REFERENCE:

Zafiriou, O. C.; McFarland, M. "Determination of Trace Levels of Nitric Oxide in Aqueous Solution," Anal. Chem., 52(11), 1980, pp. 1662-1667.

COST INFORMATION:

Cost information has not been obtained.

NITROGEN DIOXIDE (CAS Number 10102-44-0)

RQ: 4.54 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

Analysis for nitrogen dioxide in air may be performed by use of a detector tube. A 0.2- to 0.5-liter air sample is collected and analyzed with a hand-operated bellows pump and a nitrogen dioxide gas detector tube (Draeger® CH 30001 or equivalent). A positive result is indicated by a color change in the tube from pale gray to bluish-gray. The analysis is based on reaction with N,N-diphenylbenzidine.

INTERFERENCES:

Ozone and chlorine will also cause color changes. Ozone causes a color change to pale gray. Chlorine is indicated with approximately one-half to one-third the sensitivity of NO2.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is sensitive to concentration levels of approximately 1 to 50 milligrams per cubic meter.

REFERENCES:

Snyder, R. E.; Jankin, M. E.; McKissick, A. M. <u>Development of Hazardous Toxic Wastes Analytical Screening Procedures</u>, Atlantic Research Corporation for U.S. Army Medical Research and Development Command, Fort Detrick, Maryland, July 16, 1982, p. 4.

National Draeger, Inc., <u>Draeger® Detector Tube Handbook</u>, "Air Investigations and Technical Gas Analysis with Draeger® Tubes", 4th ed.; Draeger: Pittsburgh, August 1979.

National Draeger, Inc., Bulletin No. ND-105; Draeger: Pittsburgh, January 1, 1982.

National Draeger, Inc., Measurements with Draeger® Tubes in the Threshold Limit Value Range, Draeger® Gas Analysis Product Information, Leaflet 4340.3e; Draeger: Pittsburgh, December 1981.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$13-22 (list).

See also: "Organic Vapors," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Fluorides and Other Compounds," Appendix B.

Air Samples
Laboratory Method 1

METHOD SUMMARY:

Analysis of air for nitrogen dioxide can be performed by passage of a known volume of air through an impinger filled with Griess-Saltzman reagent, followed by colorimetric analysis of the resulting solution. Five standard solutions, with concentrations of 0.1 to 1 micrograms nitrogen dioxide per milliliter should be used for calibration.

INTERFERENCES:

Sulfur dioxide interference is eliminated by addition of 1 percent acetone to the color reagent; nitric oxide and ozone do not interfere.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

Method 13535 is used by the Alberta Environmental Centre and is not EPA-approved. The method is suitable over the concentration range of 2 to 600 micrograms of nitrogen dioxide per liter of air. The detection limit is 2 milligrams per liter of test solution. Method 108 has been tentatively approved by the National Institute for Occupational Safety and Health (NIOSH) for use over the range of 0.01 to 10 micrograms per cubic meter. Precision and accuracy information is furnished.

REFERENCES:

Alberta Environment, Methods Manual for Chemical Analysis of Atmospheric Pollutants, 2nd ed.; Method 13535, Alberta Environmental Centre, Vegreville, Alberta, Canada, 1981.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.03, D1607-76; ASTM: Philadelphia, 1983.

- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, Method P&CAM 108; Publication No. 75-121, U.S. DHEW: Cincinnati, 1974.
- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 108; Publication No. 77-157-A, U.S. DHEW, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$20-25 (list), plus sampling costs.

Air Samples Laboratory Method 2

METHOD SUMMARY:

Nitrogen dioxide in air samples can be measured spectrophotometrically. A known volume of air is drawn through triethanolamine-impregnated molecular sieve. The nitrogen dioxide is desorbed from the molecular sieve with aqueous triethanolamine. An aliquot of the solution is treated with hydrogen peroxide, sulfanilamide, and N-(1-naphthyl)ethylenediamine dihydrochloride, and the color intensity is determined on a spectrophotometer at 540 nm.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

A blank should be processed with every 10 samples.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 5.8 to 21.6 milligrams per cubic meter, using a 3.9-liter sample. The detection limit is approximately 0.2 milligrams per cubic meter. Precision and accuracy information is furnished.

REFERENCE:

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, Method S320; Publication No. 78-175, U.S. DHEW: Cincinnati, August 1978.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$25-30 (list), plus sampling costs.

Air Samples Laboratory Method 3

METHOD SUMMARY:

Infrared absorption spectrometry can be used to determine the concentration of nitrogen dioxide in air samples. The absorbance at 7900 nm (1266 cm⁻¹) is measured, using a 10-meter pathlength cell. Air can be sampled in a Saran® or Mylar® plastic bag; approximately 5 liters of the sample are drawn into an evacuated cell. Continuous monitoring of air flowing through the cell is possible if the spectrometer is set to one particular wavelength. Some vapors can be adsorbed on silica gel and quantitatively desorbed into the absorption cell later. Tentative qualitative identification of compounds can be made based on a full infrared absorption spectrum.

INTERFERENCES:

Compounds present in the sample that absorb infrared radiation at the wavelength of interest will interfere.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The sensitivity of the method, using a 10-meter pathlength cell, is 10 milligrams per cubic meter or 5 parts per million. No precision or accuracy information is furnished.

REFERENCE:

American Public Health Association, "Infrared Absorption Spectroscopy." In Methods of Air Sampling and Analysis, 2nd ed., M. Katz, Ed.; APHA: Washington, 1977, pp. 79-84.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$75-85 (list), plus sampling costs.

See also: "Hazardous Organic Emissions," Appendix B.

See also: "Nitrogen Dioxide and Nitric Oxide," Appendix B.

Soil/Sediment Samples Laboratory Method

Water Samples Laboratory Method

OSMIUM TETROXIDE (CAS Number 20816-12-0)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should

The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

Soil/Sediment Samples Laboratory Method

METHOD SUMMARY:

Analysis of soil/sediment samples for osmium tetroxide can be performed by atomic spectrometric analysis for osmium. The sample is digested with nitric acid and hydrogen peroxide, and the osmium content of the resulting solution is determined by flame-atomization atomic absorption spectrometry (flame AA), furnace atomization AA, or inductively coupled atomic plasma emission spectrometers (ICP). This method does not identify any particular osmium compound, but measures the total osmium content of the sample.

INTERFERENCES:

Substances that emit or absorb light at the frequency being monitored can interfere in AA. The method of standard additions can eliminate many matrix effects.

QUALITY CONTROL:

Ten percent of the samples should be processed in duplicate and one method blank should be analyzed with each set of samples.

EPA/TECHNICAL STATUS:

This method is EPA-approved for analyses under the Resource Conservation and Recovery Act (RCRA). No precision and accuracy information is furnished.

REFERENCE:

U.S. Environmental Protection Agency, <u>Test Methods for Evaluating Solid Waste</u>: <u>Physical/Chemical Methods</u>, 2nd ed. Methods 3050, 7550, 7551; SW-846, U.S. EPA:Washington, 1982.

COST INFORMATION:

Cost per sample for analysis by flame AA is approximately \$20-30 (list); by furnace AA, it is approximately \$28-60 (list); by ICP, approximately \$20 (list).

Water Samples Laboratory Method

METHOD SUMMARY:

Analysis for osmium tetroxide in water samples may be performed by atomic absorption spectrometry (AA). A 100-milliliter sample is mixed with acid, heated, filtered, and the resulting solution analyzed for osmium by flame or furnace AA. Concentrations of osmium below 0.5 milligrams per liter should be analyzed by furnace AA.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require background correction or standard addition methods. A check standard should be analyzed each time a set of samples is run.

EPA/TECHNICAL STATUS:

These methods are approved for analyses under the National Pollutant Discharge Elimination System (NPDES) and Resource Conservation and Recovery Act (RCRA). Precision and accuracy information is furnished (Methods 252.1, 252.2).

REFERENCES:

- U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Methods 252.1, 252.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.
- U.S. Environmental Protection Agency, <u>Test Methods for Evaluating Solid Waste: Physical/Chemical Methods</u>, 2nd ed., Methods 3050, 7550, 7551; SW-846, U.S. EPA: Washington, 1982.

COST INFORMATION:

Cost per sample for analysis by flame AA is approximately \$10-20 (list); by furnace AA, it is approximately \$18-50 (list).

PHENYLMERCURIC ACETATE (CAS Number 62-38-4)

RQ: 0.454

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

<u>Air Samples</u> Laboratory Method

See "Mercury," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Mercury," Appendix B.

Water Samples
Laboratory Method

See "Mercury," Appendix B.

PHOSPHINE (CAS Number 7803-51-2)

RQ: 45.4 kg

SAFETY INFORMATION: Exhibits toxicity (inhalation). Flammable gas.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Methods

METHOD SUMMARY:

Analysis for phosphine in air may be performed by use of a detector tube. A 1-liter air sample is collected and analyzed with a hand-operated bellows pump and a phosphine gas detector tube (Draeger® CH 31101, or equivalent). A positive result is indicated by a color change in the tube from white to weak grayish-violet. The method is based on the reaction of phosphine with a gold compound to form colloidal gold.

INTERFERENCES:

Arsine and antimony hydride will also give positive results. Ammonia, hydrogen chloride, hydrogen selenide, hydrogen sulfide, and mercaptans are retained by the precleanse layer of the tube.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is sensitive to concentration levels of approximately 0.1 to 5 milligrams per cubic meter for the sample size given above.

REFERENCES:

Snyder, R. E.; Jankin, M. E.; McKissick, A. M. <u>Development of Hazardous Toxic Wastes Analytical Screening Procedures</u>, Atlantic Research Corporation for U.S. Army Medical Research and Development Command, Fort Detrick, Maryland, July 16, 1982, p. 4.

National Draeger, Inc., <u>Draeger® Detector Tube Handbook</u>, "Air Investigations and Technical Gas Analysis with Draeger® Tubes", 4th ed.; Draeger: Pittsburgh, August 1979.

National Draeger, Inc., Bulletin No. ND-105; Draeger: Pittsburgh, January 1, 1982.

National Draeger, Inc., Measurements with Draeger® Tubes in the Threshold Limit

Value Range, Draeger® Gas Analysis Product Information, Leaflet 4340.3e; Draeger Pittsburgh, December 1981.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$13-22 (list).

See also: "Organic Vapors," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples
Laboratory Method

See "Fumigants," Appendix B.

Soil/Sediment Samples
Laboratory Method

METHOD SUMMARY:

Phosphine in waterlogged soils can be measured by gas chromatography (GC) with helium-ionization detection. The soil is air-dried and crushed, then incubated in sealed reaction vessels. The gases evolved are analyzed by GC with helium ionization detection. Some phosphine may remain sorbed on the soil after incubation, instead of escaping to the atmosphere being analyzed.

INTERFERENCES:

Sorption of the phosphine on the soil may affect results.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information is not furnished.

REFERENCE:

Burford, J. R.; Bremner, J. M. "Is Phosphate Reduced to Phosphine in Water-

logged Soils ?" Soil Biol. Biochem., 4(4), 1972, pp. 489-495.

COST INFORMATION:

Cost information has not been obtained.

Water Samples Laboratory Method

METHOD SUMMARY:

Phosphine in water samples can be measured by gas chromatography with flame photometric detection (GC/FPD) in the phosphorus mode. A thermionic detector or a microcoulometric detector may be employed as an alternative, if the phosphine is present in sufficient quantity.

INTERFERENCES:

Large concentrations of sulfur gases or vapors can interfere, unless preventive measures are taken.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision information is furnished. The limit of detection is 0.5, 500, or 2 micrograms of analyte per liter, respectively, using FPD, microcoulometric, or thermionic detection.

REFERENCE:

Berck, B.; Westlake, W. E.; Gunther, F. A. "Microdetermination of Phosphine by Gas-Liquid Chromatography with Microcoulometric, Thermionic, and Flame Photometric Detection," J. Agric. Food Chem., 18(1), 1970, pp. 143-147.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$120 (list).

PHOSPHORIC ACID (CAS Number 7664-38-2)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples
Laboratory Method

METHOD SUMMARY:

Analysis for phosphoric acid in air can be performed colorimetrically. A known volume of air is passed through a membrane filter and the analyte is removed from the filter by hot-water leaching. The resulting solution is reacted with sodium molybdate and hydrazine sulfate to form a blue (molybdenum blue) complex. The absorbance is read at 830 nm on a spectrophotometer.

INTERFERENCES:

Airborne particulate salts of phosphoric acid will interfere with this determination.

QUALITY CONTROL:

Percent recovery must be determined for the analytical procedure and results should be corrected for recoveries of less than 95 percent. Duplicate determinations should agree within 5 percent, and a method blank must be analyzed with each sample or set of samples.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 0.5 to 1.9 milligrams per cubic meter for a 90-liter sample of air. A variation of this method is

classified as operational by NIOSH over the range of 0.2 to 4 milligrams per cubic meter for the same sample size. Precision and accuracy information is furnished.

REFERENCES:

- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 216; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.
- U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set M, Method S333; PB-265 029 (NTIS), U.S. DHEW: Cincinnati, December 1976.
- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, Method S333; Publication No. 77-157-C, U.S DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$30-35 (list).

See also: "Inorganic Acids," Appendix B.
See also: "Phosphorus," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Phosphorus," Appendix B.

Water Samples Laboratory Method

See "Phosphorus," Appendix B. See "Weak Acids," Appendix B.

PHOSPHORUS (CAS Number 7723-14-0)

RQ: 0.454 kg

SAFETY INFORMATION: Flammable solid; inflames on contact with air. Potentially

chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

METHOD SUMMARY:

White phosphorus (also called yellow phosphorus) in air samples may be measured by gas chromatography. A known volume of air is passed through a tube containing Tenax GC resin to collect the phosphorus vapor, and the phosphorus is desorbed into xylene. Alternatively, the air sample may be passed through a midget impinger containing xylene. In either case, the xylene solution is analyzed by gas chromatography with flame photometric detection (GC/FPD).

INTERFERENCES:

Phosphorus compounds having retention times close to that of phosphorus will interfere.

QUALITY CONTROL:

Desorption efficiency must be determined over the range of interest when Tenax GC is used for sample collection, and a method blank should be processed with every 10 samples. The desorption efficiency and the results of the method-blank analysis should be used in calculation of results.

EPA/TECHNICAL STATUS:

The Tenax GC collection method is approved by the National Institute for Occupational Safety and Health (NIOSH), and has been validated over the range of 0.056 to 0.244 milligrams per cubic meter, using a 12-liter sample. The method is capable of measurement of much lower levels if the desorption efficiency is adequate. A second method utilizing Tenax GC collection is proposed by NIOSH for use over the range of 0.01 to 1.00 milligrams per cubic meter in a 20-liter air sample. A method employing collection in an impinger is proposed by NIOSH for use over the range of 0.005 to 5.0 milligrams per cubic meter in a 50-liter air sample. Precision and accuracy information is furnished.

REFERENCES:

- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, Method S334; Publication No. 78-175, U.S. DHEW: Cincinnati, August 1978.
- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 242; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.
- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 257; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$110-130 (list), plus sampling costs.

See also: "Phosphorus," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Phosphorus," Appendix B.

Water Samples Laboratory Method

See "Phosphorus," Appendix B.

PHOSPHORUS OXYCHLORIDE (CAS Number 10025-87-3)

RQ: 454 kg

SAFETY INFORMATION: Reacts violently with water or moisture to yield

hydrogen chloride.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

Reacts with water.

Water Samples Field Method

Reacts with water.

See "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples
Laboratory Method

See "Phosphorus," Appendix B.

Soil/Sediment Samples
Laboratory Method

Reacts with water.

See "Phosphorus," Appendix B.

Water Samples Laboratory Method

Reacts with water.

See "Phosphorus," Appendix B.

PHOSPHORUS PENTASULFIDE (CAS Number 1314-80-3)

RQ: 45.4 kg

SAFETY INFORMATION: Forms hydrogen sulfide on contact with water.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

Reacts with water.

See "Sulfides," Appendix B.

Water Samples Field Method

Reacts with water.

See "Aromatics and Other Compounds," Appendix B.

See "Fluorides and Other Compounds, Appendix B.

See "Halides, Cyanides, and Other Compounds," Appendix B.

See "Sulfides," Appendix B.

Air Samples Laboratory Method

See "Phosphorus," Appendix B.

See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment Samples
Laboratory Method

Reacts with water.

See "Phosphorus," Appendix B.

See "Sulfides," Appendix B.

Water Samples Laboratory Method

Reacts with water.

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See "Phosphorus," Appendix B. See "Sulfides," Appendix B.

PHOSPHORUS TRICHLORIDE (CAS Number 7719-12-2)

RQ: 454 kg

SAFETY INFORMATION: Reacts violently with water to produce hydrochloric and

phosphorus acids.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

Reacts with water.

Water Samples Field Method

Reacts with water.

See "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples Laboratory Method

See "Chlorides," Appendix B. See "Phosphorus," Appendix B.

Soil/Sediment Samples Laboratory Method

Reacts with water.

See "Phosphorus," Appendix B.

Water Samples Laboratory Method

Reacts with water.

See "Chlorides," Appendix B. See "Phosphorus," Appendix B.

POTASSIUM ARSENATE (CAS Number 7784-41-0)

RQ: 454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Arsenic," Appendix B.

Water Samples Field Method

See "Potassium," Appendix B.

Air Samples
Laboratory Method

See "Arsenic," Appendix B. See "Potassium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Arsenic," Appendix B. See "Potassium," Appendix B.

Water Samples
Laboratory Method

See "Arsenic," Appendix B. See "Potassium," Appendix B.

POTASSIUM ARSENITE (CAS Number 10124-50-2)

RQ: 454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Arsenic," Appendix B.

Water Samples Field Method

See "Potassium," Appendix B.

Air Samples Laboratory Method

See "Arsenic," Appendix B. See "Potassium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Arsenic," Appendix B. See "Potassium," Appendix B.

Water Samples
Laboratory Method

See "Arsenic," Appendix B. See "Potassium," Appendix B.

POTASSIUM CHROMATE (CAS Number 7789-00-6)

RQ: 454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

The user should obtain additional toxicity/hazard CAUTION:

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Chromium," Appendix B.

Soil/Sediment Samples Field Method

See "Chromium," Appendix B.

Water Samples Field Method

See "Chromates," Appendix B.

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

See "Potassium," Appendix B.

Air Samples Laboratory Method

See "Chromic Acid and Chromates," Appendix B.

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

See "Potassium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

See "Potassium," Appendix B.

Water Samples Laboratory Method

See "Chromium," Appendix B.

See "Chromium, Hexavalent," Appendix B.

See "Potassium," Appendix B.

POTASSIUM CYANIDE (CAS Number 151-50-8)

RQ: 4.54 kg

SAFETY INFORMATION: Exhibits toxicity (oral). Produces toxic hydrogen cyanide

upon contact acids.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Cyanides," Appendix B.

Soil/Sediment Samples Field Method

See "Cyanides," Appendix B.

Water Samples Field Method

See "Cyanides," Appendix B.

See "Halides, Cyanides, and Other Compounds," Appendix B.

See "Potassium," Appendix B.

Air Samples Laboratory Method

See "Cyanides," Appendix B. See "Potassium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Cyanides," Appendix B.

See "Potassium," Appendix B.

Water Samples Laboratory Method

See "Cyanides," Appendix B.

See "Potassium," Appendix B.

POTASSIUM DICHROMATE (CAS Number 7778-50-9)

RQ: 454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Chromium, Appendix B.

Soil/Sediment Samples Field Method

See "Chromium," Appendix B.

Water Samples Field Method

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

See "Potassium," Appendix B.

Air Samples Laboratory Method

See "Chromic Acid and Chromates," Appendix B.

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

See "Potassium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

See "Potassium," Appendix B.

Water Samples Laboratory Method

See "Chromium," Appendix B.

See "Chromium, Hexavalent," Appendix B.

See "Potassium," Appendix B.

POTASSIUM HYDROXIDE (CAS Number 1310-58-3)

RQ: 454 kg

SAFETY INFORMATION: Generates heat in contact with water. Generates flammable

hydrogen gas in contact with some metals.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Hydroxides," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Potassium," Appendix B.

Air Samples
Laboratory Method

See "Potassium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Potassium," Appendix B.

Water Samples Laboratory Method

See "Potassium," Appendix B.

POTASSIUM PERMANGANATE (CAS Number 7722-64-7)

RQ: 45.4 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

information before handling this substance

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

Analysis for potassium permanganate in air can be performed by filtration of air, followed by X-ray fluorescence analysis of the filter for manganese, using a portable X-ray fluorescence spectrometer and a radioisotope excitation source. Sampling time may be up to 8 hours, but analysis may be performed in approximately 1 minute. This method is non-destructive, so that further analysis of the filtered material may be undertaken following this measurement. The method is not specific for potassium permanganate, but measures the total manganese content of the sample.

INTERFERENCES:

Polyvinyl chloride filters are unacceptable for sampling, and silica quartz filter materials contain silicon, aluminum, and phosphorus, which interfere in the analysis. Cellulose membrane or fiber filters and polycarbonate or fluorocarbon membrane filters are acceptable, while glass fiber filters are satisfactory for sampling only if of the highest purity.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is not EPA-approved. Precision information is furnished. The method is sensitive to amounts of manganese over 280 micrograms, or approximately 0.1 to 10.0 micrograms per cubic meter, depending on the volume of air sampled.

REFERENCES:

Rhodes, J. R.; Stout, J. C.; Schindler, J. S.; Piorek, S. "Portable X-ray Survey Meters for in Situ Trace Element Monitoring of Air Particulates," In <u>Toxic Materials in the Atmosphere</u>, STP-786; American Society for Testing and <u>Materials</u>: Philadelphia, 1981, pp. 70-82.

Rhodes, J. R.; Pradzynski, A. H.; Hunter, C. B.; Payne, J. S.; Lindgren, J. L. "Energy Dispersive X-ray Fluorescence Analysis of Air Particulates in Texas," Environ. Sci. Technol., 6(10), 1972, pp. 922-927.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$30, plus sampling costs.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Potassium," Appendix B.

Air Samples
Laboratory Method 1

METHOD SUMMARY:

Analysis for potassium permanganate in air can be performed by filtration of air, followed by X-ray fluorescence analysis of the filter for manganese. Sampling time may be up to 8 hours but analysis may be performed in approximately 1 minute. This method is non-destructive, so that further analysis of the filtered material may be undertaken following this measurement. The method is not specific for potassium permanganate, but measures the total manganese content of the sample.

INTERFERENCES:

Polyvinyl chloride filters are unacceptable for sampling, and silica quartz filter materials contain silicon, aluminum, and phosphorus which interfere. Cellulose membrane or fiber filters and polycarbonate or fluorocarbon membrane filters are acceptable, while glass fiber filters are satisfactory for sampling only if of the highest purity.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision information is furnished. The method is sensitive to amounts of manganese over 280 micrograms, or approximately 0.1 to 10.0 microgram per cubic meter, depending on the volume of air sampled.

Rhodes, J. R.; Stout, J. C.; Schindler, J. S.; Piorek, S. "Portable X-ray Survey Meters for in Situ Trace Element Monitoring of Air Particulates," In <u>Toxic Materials in the Atmosphere</u>, STP-786; American Society for Testing and <u>Materials</u>: Philadelphia, 1981, pp. 70-82.

Rhodes, J. R.; Pradzynski, A. H.; Hunter, C. B.; Payne, J. S.; Lindgren, J. L. "Energy Dispersive X-ray Fluorescence Analysis of Air Particulates in Texas," Environ. Sci. Technol., 6(10), 1972, pp. 922-927.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$30 (list), \$20 (bid), plus sampling costs.

See also: "Potassium," Appendix B.

Air Samples Laboratory Method 2

METHOD SUMMARY:

Analysis for potassium permanganate in air samples may be performed by flame atomic absorption (AA) spectrometric determination of manganese. A known volume of air is drawn through a membrane or glass fiber filter. The filter with the collected particulates is digested with nitric or nitric and hydrofluoric acids, and an aliquot of the resulting solution is aspirated into the AA instrument. This method does not specifically identify potassium permanganate, but measures the total manganese content of the sample.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

A blank should be processed with every $10\ \text{samples}$. Standard solutions should be analyzed in duplicate.

EPA/TECHNICAL STATUS:

This method is classified as operational by the National Institute of Occupational Safety and Health (NIOSH) for use over the range of 21 to 125 micrograms manganese per cubic meter in a 240-liter air sample. Precision information is furnished. Alberta Environment has determined the limit of detection to be 0.01 micrograms manganese per cubic meter of air.

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 173; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

Alberta Environment, Methods Manual for Chemical Analysis of Atmospheric Pollutants, Methods 23535, 23542, Alberta Environmental Centre, Vegreville, Alberta, Canada, 1981.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$10-20 (list), plus sampling costs.

Soil/Sediment Samples Laboratory Method

METHOD SUMMARY:

Analysis for potassium permanganate in soil/sediment samples can be performed by atomic spectrometric analysis for manganese. A 2-gram sample is digested with nitric acid and hydrogen peroxide and analyzed by inductively coupled plasma (ICP) or flame atomic absorption spectrometry (AA). Flame AA is not recommended for low levels of manganese. Analysis for manganese in oil may be performed by ICP after sample dilution with methyl isobutyl ketone (MIBK) or xylene, using standards miscible with these solvents. This method is not specific for potassium permanganate, but measures the total manganese content of the sample.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods. Standards should be analyzed at least daily and a reference standard should be run weekly. At least 10 percent of the analyses should be duplicates and/or standards.

EPA/TECHNICAL STATUS:

These methods are in use in the CERCLA program, and are required for use in Resource Conservation and Recovery Act (RCRA) and Clean Water Act analyses. Precision and accuracy information is furnished.

Exhibit D, Chemical Analytical Services for Low and Medium Concentration Inorganics in All Media, Invitation for Bid (Solicitation Number WA 82-A072), U.S. EPA: Washington, April 23, 1982.

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 243.1, EPA-600/4-79-020, U.S. EPA: Cincinnati, March 1979.

Federal Register, 44(233), December 3, 1979, pp. 69559 - 69564.

Fassel, V. A.; Peterson, C. A.; Abercrombie, F. N.; Kniseley, R. N. "Simultaneous Determination of Wear Metals in Lubricating Oils by Inductively-Coupled Plasma Atomic Emission Spectrometry" Anal. Chem., 48(3), 1976, p. 516.

COST INFORMATION:

Cost per sample for analysis by flame AA is approximately \$20-30 (list); by ICP, it is approximately \$20 (list).

See also: "Potassium," Appendix B.

Water Samples
Laboratory Method

METHOD SUMMARY:

Analysis for potassium permanganate in water samples may be performed by atomic spectrometric analysis for manganese. A 100-milliliter sample is digested with nitric acid and hydrogen peroxide and analyzed by inductively coupled plasma (ICP) or flame atomic absorption spectrometry (AA). Flame AA is not recommended for low levels of manganese. This method is not specific for potassium permanganate, but measures the total manganese content of the sample.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods. A reference standard should be analyzed weekly and at least 10 percent of the analyses should be duplicates and/or standards.

EPA/TECHNICAL STATUS:

These methods are in use in the CERCLA program, and are required for Resource Conservation and Recovery Act (RCRA) and National Pollutant Discharge Elimination System (NPDES) analyses. Precision and accuracy information is furnished.

Exhibit D, Chemical Analytical Services for Low and Medium Concentration Inorganics in All Media, Invitation for Bid (Solicitation Number WA 82-A072), U.S. EPA: Cincinnati, April 23, 1982.

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 243.1, EPA-606/479-020, U.S. EPA: Cincinnati, March 1979.

Federal Register, 44(233), December 3, 1979, pp. 69559-69564.

COST INFORMATION:

Cost per sample for analysis by flame AA is approximately \$10-20\$ (list); by ICP, it is approximately \$10\$ (list).

See also: "Potassium," Appendix B.

POTASSIUM SILVER CYANIDE (CAS Number 506-61-6)

RQ: 4.54 kg

Strong oxidizer; can readily cause fires. SAFETY INFORMATION:

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Field Method

Soil/Sediment Samples Field Method

See "Cyanides," Appendix B.

Water Samples Field Method

See "Cyanides," Appendix B. See "Potassium," Appendix B.

See "Silver," Appendix B.

Air Samples Laboratory Method

See "Cyanides," Appendix B. See "Potassium," Appendix B.

See "Silver," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Cyanides," Appendix B.

See "Potassium," Appendix B.

See "Silver," Appendix B.

Water Samples Laboratory Method

See "Cyanides," Appendix B.

See "Potassium," Appendix B.

See "Silver," Appendix B.

SELENIOUS ACID (CAS Number 7783-00-8)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Selenium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Selenium," Appendix B.

Air Samples Laboratory Method

See "Selenium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Selenium," Appendix B.

Water Samples Laboratory Method

SELENIUM (CAS Number 7782-49-2)

RQ: 0.454 kg

SAFETY INFORMATION: Exhibits toxicity (inhalation). Potentially chronically

toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Selenium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Selenium," Appendix B.

Air Samples
Laboratory Method

See "Selenium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Selenium," Appendix B.

Water Samples Laboratory Method

SELENIUM DIOXIDE (CAS Number 7446-08-4)

RQ: 454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Selenium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Selenium," Appendix B.

Air Samples Laboratory Method

See "Selenium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Selenium," Appendix B.

Water Samples Laboratory Method

SELENIUM DISULFIDE (CAS Number 7488-56-4)

RQ: 0.454 kg

SAFETY INFORMATION: Forms toxic hydrogen sulfide in contact with acid. Poten-

tially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Selenium," Appendix B.

Soil/Sediment Samples Field Method

See "Sulfides," Appendix B.

Water Samples Field Method

Insoluble in water.

Air Samples
Laboratory Method

See "Selenium," Appendix B.

See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Selenium," Appendix B.

See "Sulfides," Appendix B.

Water Samples
Laboratory Method

Insoluble in water.

SELENOUREA (CAS Number 630-10-4)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Selenium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

See "Selenium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Selenium," Appendix B.

Water Samples
Laboratory Method

RQ: 454 kg

SAFETY INFORMATION: Potentially chronically toxic.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

See "Silver," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Silver," Appendix B.

Water Samples
Laboratory Method

See "Silver," Appendix B.

SILVER CYANIDE (CAS Number 506-64-9)

RQ: 0.454 kg

SAFETY INFORMATION: Exhibits chronic toxicity.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Cyanides," Appendix B.

Water Samples Field Method

See "Cyanides," Appendix B. See "Silver," Appendix B.

Air Samples Laboratory Method

See "Cyanides," Appendix B. See "Silver," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Cyanides," Appendix B. See "Silver," Appendix B.

Water Samples Laboratory Method

See "Cyanides," Appendix B. See "Silver," Appendix B.

SILVER NITRATE (CAS Number 7761-88-8)

RQ: 0.454 kg

SAFETY INFORMATION: Exhibits chronic toxicity.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Nitrate Ions," Appendix B. See "Silver," Appendix B.

Air Samples Laboratory Method

See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

See "Silver," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

See "Silver," Appendix B.

Water Samples Laboratory Method

See "Nitrates," Appendix B. See "Silver," Appendix B.

SODIUM (CAS Number 7740-23-5)

RQ: 4.54 kg

SAFETY INFORMATION: Inflames upon contact with water.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

Reacts with water.

Water Samples Field Method

Reacts with water.

Air Samples Laboratory Method

See "Sodium," Appendix B.

Soil/Sediment Samples
Laboratory Method

Reacts with water.

See "Sodium," Appendix B.

Water Samples Laboratory Method

Reacts with water.

SODIUM ARSENATE (CAS Number 7631-89-2)

RQ: 454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Arsenic," Appendix B.

Water Samples Field Method

Air Samples Laboratory Method

See "Arsenic," Appendix B. See "Sodium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Arsenic," Appendix B. See "Sodium," Appendix B.

Water Samples
Laboratory Method

See "Arsenic," Appendix B. See "Sodium," Appendix B.

SODIUM ARSENITE (CAS Number 7784-46-5)

RQ: 454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic. CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Arsenic," Appendix B.

Water Samples Field Method

Air Samples Laboratory Method

See "Arsenic," Appendix B. See "Sodium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Arsenic," Appendix B. See "Sodium," Appendix B.

Water Samples Laboratory Method

See "Arsenic," Appendix B. See "Sodium," Appendix B.

SODIUM AZIDE (CAS Number 26628-22-8)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

METHOD SUMMARY:

Analysis for sodium azide in air can be performed by high-volume filtration of air, followed by dissolution of particulates in deionized water and analysis of the resulting solution for azide ion by ion chromatography.

INTERFERENCES:

Use of an alkaline sodium carbonate eluant may eliminate interferences due to the presence of phosphate ions. The presence of bromide ions in the sample interferes with accurate determination of azide concentration.

QUALITY CONTROL:

The air filter should collect greater than 99.96 percent of particulates larger than 3 micrometers in diameter.

EPA/TECHNICAL STATUS:

This method has not been extensively evaluated and precision and accuracy information is not furnished.

Westwood, L. C.; Stokes, E. L. In <u>Ion Chromatographic Analysis of Environmental Pollutants</u>, Vol. 2; J. D. Mulik and E. G. Sawicki, Eds.; Ann Arbor Science: Ann Arbor, 1979, pp. 141-156.

See also: "Sodium," Appendix B.

COST INFORMATION:

The cost per sample for method is approximately \$75-80 (list), plus sampling costs.

Soil/Sediment Samples
Laboratory Method

See "Sodium, " Appendix B.

Water Samples Laboratory Method

SODIUM BIFLUORIDE (CAS Number 1333-83-1)

RQ: 45.4 kg

SAFETY INFORMATION: Reacts with water to form hydrofluoric acid.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

See "Fluorides," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Fluorides and Other Compounds," Appendix B.

Air Samples Laboratory Method

See "Fluorides," Appendix B. See "Sodium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Fluorides," Appendix B. See "Sodium," Appendix B.

Water Samples Laboratory Method

See "Fluorides," Appendix B. See "Sodium," Appendix B.

SODIUM BISULFITE (CAS Number 7631-90-5)

RQ: 2270 kg SAFETY INFORMATION:

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

See "Sodium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Sodium," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B.

See "Sodium," Appendix B.

See "Sulfites," Appendix B.

SODIUM CHROMATE (CAS Number 7775-11-3)

RQ: 454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

sing this analytical process

Air Samples Field Method

See "Chromium," Appendix B.

Soil/Sediment Samples Field Method

See "Chromium," Appendix B.

Water Samples Field Method

See "Chromates," Appendix B.

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

Air Samples
Laboratory Method

See "Chromic Acid and Chromates," Appendix B.

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

See "Sodium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

See "Sodium," Appendix B.

Water Samples
Laboratory Method

See "Chromium," Appendix B.

See "Chromium, Hexavalent," Appendix B.

SODIUM CYANIDE (CAS Number 143-33-9)

RQ: 4.54 kg

Exhibits toxicity (oral). Reacts with acid to form very SAFETY INFORMATION:

toxic hydrogen cyanide.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Cyanides," Appendix B.

Soil/Sediment Samples Field Method

See "Cyanides," Appendix B.

Water Samples Field Method

See "Cyanides," Appendix B.

See "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples Laboratory Method

See "Cyanides," Appendix B. See "Sodium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Cyanides," Appendix B.

See "Sodium," Appendix B.

Water Samples Laboratory Method

See "Cyanides," Appendix B.

SODIUM DICHROMATE (CAS Number 10588-01-9)

RO: 454 kg

SAFETY INFORMATION: Potentially carcinogenic. Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Chromium," Appendix B.

Soil/Sediment Samples Field Method

See "Chromium," Appendix B.

Water Samples Field Method

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

Air Samples Laboratory Method

See "Chromic Acid and Chromates," Appendix B.

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

See "Sodium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

See "Sodium," Appendix B.

Water Samples Laboratory Method

See "Chromium," Appendix B.

See "Chromium, Hexavalent," Appendix B.

SODIUM DODECYLBENZENE SULFONATE (CAS Number 25155-30-0)

RQ: 454 kg SAFETY INFORMATION:

Air Samples Field Method

Soil/Sediment Samples Field Method

Water Samples Field Method

See "Anionic Detergents," Appendix B. See "Sodium," Appendix B.

Air Samples Laboratory Method

See "Sodium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Sodium," Appendix B.

Water Samples
Laboratory Method

See "Alkyl Benzene Sulfonates," Appendix B. See "Sodium," Appendix B.

SODIUM FLUORIDE (CAS Number 7681-49-4)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Fluorides," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Fluorides and Other Compounds," Appendix B.

Air Samples Laboratory Method

See "Fluorides," Appendix B.

See "Fluorides and Hydrogen Fluoride," Appendix B.

See "Sodium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Sodium," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B.

See "Fluorides," Appendix B.

SODIUM HYDROSULFIDE (CAS Number 16721-80-5)

RQ: 2270 kg

SAFETY INFORMATION: Evolves toxic hydrogen sulfide on contact with acids.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Sulfides," Appendix B.

Water Samples Field Method

See "Halides, Cyanides, and Other Compounds," Appendix B. See "Sulfides," Appendix B.

Air Samples Laboratory Method

See "Sodium," Appendix B.

See "Sulfides," Appendix B, Part II, Section A.

See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Sodium," Appendix B. See "Sulfides," Appendix B.

Water Samples Laboratory Method

See "Sodium," Appendix B. See "Sulfides," Appendix B.

SODIUM HYDROXIDE (CAS Number 1310-73-2)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Hydroxides," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples
Laboratory Method

METHOD SUMMARY:

Sodium hydroxide in air samples can be measured by titration. A known volume of air is drawn through a polytetrafluoroethylene filter. The sodium hydroxide is extracted with a known amount of excess hydrochloric acid, and the solution is purged with nitrogen. The excess hydrochloric acid is titrated under nitrogen with a standard solution of sodium hydroxide and the amount of sodium hydroxide collected is calculated from the difference between the amount of acid added and the amount remaining after extraction of the filter. An alternative sampling procedure is collection of the sodium hydroxide in a fritted glass bubbler containing excess hydrochloric acid. These methods are not specific for sodium hydroxide, but measure the total alkalinity in particulate form when a filter is used for sampling, or total alkalinity in all phases when a bubbler is used.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

A method blank should be processed with each set of samples. Results of the method-blank analysis should be used in calculations. A recovery factor should be determined when a filter is used for sampling.

EPA/TECHNICAL STATUS:

The filter sampling method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 0.76 to 3.9 milligrams per cubic meter, using a 360-liter sample. The detection limit is estimated to be 77 micrograms per cubic meter. The bubbler-sampling method is proposed by NIOSH for the range of 1 to 4 milligrams per cubic meter in a 420-liter sample. Precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, Method S381; Publication No. 78-175, U.S. DHEW: Cincinnati, August 1978.

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 241; Publication No. 77-157-A, U.S. DHEW: 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$30 (list), plus sampling costs.

See also: "Sodium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Sodium," Appendix B.

Water Samples Laboratory Method

SODIUM HYPOCHLORITE (CAS Numbers 7681-52-9, 10022-70-5)

RQ: 45.4 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Halides, Cyanides, and Other Compounds," Appendix B.

Air Samples
Laboratory Method

See "Sodium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Sodium," Appendix B.

Water Samples Laboratory Method

SODIUM METHYLATE (CAS Number 124-41-4)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

Reacts with water.

Water Samples Field Method

Reacts with water.

Air Samples Laboratory Method

See "Sodium," Appendix B.

Soil/Sediment Samples
Laboratory Method

Reacts with water-

See "Sodium," Appendix B.

Water Samples Laboratory Method

Reacts with water.

SODIUM NITRITE (CAS Number 7632-00-0)

RQ: 45.4 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples
Laboratory Method

See "Sodium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Sodium," Appendix B.

Water Samples Laboratory Method

SODIUM PHOSPHATE, DIBASIC (CAS Numbers 7558-79-4, 10039-32-4, 10028-24-7, 10140-65-5)

RQ: 2270 kg SAFETY INFORMATION:

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

See "Phosphorus," Appendix B. See "Sodium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Phosphorus," Appendix B. See "Sodium," Appendix B.

Water Samples Laboratory Method

See "Phosphorus," Appendix B. See "Sodium," Appendix B.

SODIUM PHOSPHATE, TRIBASIC (CAS Numbers 7601-54-9, 7785-84-4, 10101-89-0, 10361-89-4, 7758-29-4, 10124-56-8)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: T

The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

<u>Air Samples</u> Laboratory Method

See "Phosphorus," Appendix B. See "Sodium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Phosphorus," Appendix B. See "Sodium," Appendix B.

Water Samples Laboratory Method

See "Phosphorus," Appendix B. See "Sodium," Appendix B.

SODIUM SELENITE (CAS Numbers 10102-18-8, 7782-82-3)

RQ: 454 kg

SAFETY INFORMATION: Exhibits toxicity (oral). Potentially chronically toxic.

Y: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Selenium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Selenium," Appendix B.

Air Samples Laboratory Method

See "Selenium," Appendix B. See "Sodium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Selenium," Appendix B. See "Sodium," Appendix B.

Water Samples Laboratory Method

See "Selenium," Appendix B. See "Sodium," Appendix B.

STRONTIUM CHROMATE (CAS Number 7789-06-2)

RQ: 454 kg

SAFETY INFORMATION: Potentially carcinogenic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

See "Chromic Acid and Strontium Chromate," Appendix B. See "Chromium," Appendix B.

Soil/Sediment Samples Field Method

See "Chromium," Appendix B.

Water Samples Field Method

See "Chromates," Appendix B. See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

Air Samples Laboratory Method

See "Chromic Acid and Chromates," Appendix B.

See "Chromium," Appendix B.

See "Heavy Metals and Compounds (Particulate)," Appendix B.

See "Strontium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Chromium," Appendix B.

See "Hexavalent Chromium," Appendix B.

See "Strontium," Appendix B.

Water Samples Laboratory Method

See "Chromium," Appendix B.

See "Chromium, Hexavalent," Appendix B.

See "Strontium," Appendix B.

STRONTIUM SULFIDE (CAS Number 1314-96-1)

RQ: 45.4 kg

Reacts to form toxic H_2S upon contact with acids. SAFETY INFORMATION:

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

See "Sulfides," Appendix B.

Water Samples Field Method

See "Sulfides," Appendix B.

Air Samples Laboratory Method

See "Strontium," Appendix B.

See "Sulfur Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Strontium," Appendix B. See "Sulfides," Appendix B.

Water Samples Laboratory Method

See "Strontium," Appendix B. See "Sulfides," Appendix B.

SULFURIC ACID (CAS Number 7664-93-9)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

Sulfuric acid in air samples can be measured by titration. The sample is passed through filter paper (Whatman No. 4 or equivalent). The filter paper is macerated in distilled water and the resulting solution is titrated with 0.002 N sodium hydroxide, using a pH meter to determine the endpoint.

INTERFERENCES:

Since matter normally present as dust is neutral, and gases such as sulfur dioxide, hydrogen chloride, oxides of nitrogen, and ammonia pass through the filter without affecting its pH, the residual acidity measured equals the acidity of the sulfuric acid aerosol collected. This method will measure any acidic particulate or aerosol compounds in the sample and does not identify sulfuric acid.

QUALITY CONTROL:

The pH of the filter paper batch must have a consistency of 0.03 pH unit, and a deviation of no more than 0.10 pH unit from that of the distilled water used in the measurement.

SENSITIVITY:

This method is sensitive to concentration levels of sulfuric acid greater than 10 nanograms per cubic meter.

REFERENCE:

Mader, P. P.; Hamming, W. J.; Bellin, A. "Determination of Small Amounts of Sulfuric Acid in the Atmosphere" Anal. Chem., 22(9), 1950, pp. 1181-1183.

COST INFORMATION:

Cost information has not been obtained.

Note: NIOSH Method 267 is applicable, (reference furnished by reviewer).

Soil/Sediment Samples Field Method

Water Samples Field Method

Air Samples Laboratory Method

METHOD SUMMARY:

Analysis for sulfuric acid in air can be performed by volatilization, prefiltration, and derivatization of the sulfuric acid in air samples, followed by ion chromatographic analysis of the decomposition products of the derivative for sulfate ions. The method is sensitive to concentration levels of sulfuric acid above approximately 0.5 micrograms per cubic meter. Samples are stable on the collection medium for some time. This method is specific for sulfuric acid and does not give positive results for ammonium sulfate, sulfur dioxide, or other sulfur compounds.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved and has not been extensively evaluated. Precision and accuracy information is not furnished.

REFERENCE:

Mason, D. W.; Miller, H. C. In <u>Ion Chromatographic Analysis of Environmental Pollutants</u>, Vol. 2; J. D. Mulik and E. Sawicki, Eds.; Ann Arbor Science: Ann Arbor, 1979, p. 193.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$108-120 (list), plus sampling costs.

Air Samples
Laboratory Method 2

METHOD SUMMARY:

Sulfuric acid in air samples can be measured by titration. A known volume of air is drawn through a cellulose membrane filter. The analyte is extracted with distilled water and isopropyl alcohol followed by pH adjustment with dilute perchloric acid. The resulting solution is titrated with 0.005 M barium perchlorate using Thorin as the indicator. The endpoint is reached when the solution color abruptly changes from yellow to apricot.

INTERFERENCES:

Metal ion interferences can be eliminated by passing the solution through cation exchange resin. When concentrations of phosphate ions are high the phosphate ions can be removed by precipitation with magnesium carbonate.

QUALITY CONTROL:

A method blank is analyzed for every 10 samples obtained.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 0.6 to 2.6 milligrams per cubic meter in a 180-liter sample. Precision and accuracy information is furnished.

REFERENCES:

- U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set L, Method S174; PB-250 159 (NTIS), U.S. DHEW: Cincinnati, January 1976.
- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, Method S174; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost per sample by this method is approximately \$20-45 (list), plus sampling costs.

Air Samples Laboratory Method 3

METHOD SUMMARY:

Sulfuric acid mist in air samples may be measured by turbidimetry. The sulfuric acid is collected in a midget impinger containing distilled water. The sulfate in the solution is precipitated as barium sulfate and the turbidity of the suspension is measured at 420 nm on a spectrophotometer.

INTERFERENCES:

Large concentrations of colored or suspended matter or organic materials may interfere. Silica may interfere, and if sulfate salts are present as particulates in the sample air, they will be reported as sulfuric acid.

QUALITY CONTROL:

One out of every 10 samples should be processed without addition of the barium reagent for use as a blank.

EPA/TECHNICAL STATUS:

This method is proposed by the National Institute of Occupational Safety and Health (NIOSH) for use over the range of 0.1 to 4.0 milligrams per cubic meter of air in a 10-liter sample. Limited precision and accuracy information is furnished.

REFERENCE:

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 187; Publication No. 77-157-A, U.S. DHEW, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$30-35 (list), plus sampling costs.

Note: NIOSH Method 267 is applicable (reference furnished by reviewer).

See also: "Inorganic Acids," Appendix B.

Soil/Sediment Samples
Laboratory Method

Water Samples Laboratory Method

SULFUR MONOCHLORIDE (CAS Number 12771-08-3)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

Sulfur monochloride can be measured by titration. The sample is collected in silver nitrate contained in a fritted bubbler, and acidified with nitric acid. Sodium chloride is added and the sample is titrated with 0.1 N silver nitrate. (This method is described by Jacobs, M. B. The Analytical Toxicology of Industrial Inorganic Poisons; Interscience: New York, 1967, p. 568, and summarized in the reference given below.)

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is sensitive to concentration levels of approximately 5 milligrams of sulfur monochloride per cubic meter. It should be considered a source method.

REFERENCE:

Quantitative Analysis of Gaseous Pollutants, W. E. Ruch, Ed.; Ann Arbor-Humphrey Science: Ann Arbor, 1970, p. 218.

COST INFORMATION:

Cost information has not been obtained.

Soil/Sediment Samples Field Method

Reacts with water.

Water Samples
Field Method
Reacts with water.

See "Aromatics and Other Compounds," Appendix B.

Air Samples
Laboratory Method

Soil/Sediment Samples
Laboratory Method

Reacts with water.

Water Samples Laboratory Method

Reacts with water.

TETRAETHYL LEAD (CAS Number 78-00-2)

RO: 45.4

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Organic Vapors," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

METHOD SUMMARY:

Analysis for tetraethyl lead in air can be performed by passage of 120 liters of air through XAD-2 resin, desorbing the tetraethyl lead with pentane, and analyzing the resulting solution by gas chromatography with photoionization detection (GC/PID).

INTERFERENCES:

Compounds that coelute with tetraethyl lead can interfere. In such cases, separation conditions should be changed.

QUALITY CONTROL:

The desorption efficiency must be determined over the concentration range of interest. Sampling apparatus must be calibrated before use. With each batch of ten samples a method blank should be processed. Results should be corrected to reflect the desorption efficiency.

EPA/TECHNICAL STATUS:

This method has been reviewed and evaluated by the National Institute for Occupational Safety and Health (NIOSH) and has been validated over the range of 45 to 200 micrograms tetraethyl lead per cubic meter in a 120-liter sample of air. The sensitivity of the method is estimated to be at least 0.1 microgram per milliliter. Precision and accuracy information is furnished.

REFERENCE:

U.S. Department of Health, Education, and Welfare, Ten NIOSH Analytical Methods, Set 3, Method S383, PB275-834 (NTIS), U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$100-135 (list), plus sampling costs.

Soil/Sediment Samples Laboratory Method

Water Samples Laboratory Method

METHOD SUMMARY:

Tetraethyl lead in water samples can be determined by gas chromatography, using an atomic absorption spectrometer as the detector (GC/AA). A 200-milliliter water sample is extracted with hexane, and an aliquot of the solvent containing the analyte is injected directly into the injection port of the chromatograph.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

The method of standard addition is used.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Accuracy information is furnished.

REFERENCE:

Chau, Y. K.; Wong, P. T. S.; Bengert, G. A.; Kramar, O. "Determination of Tetraalkyllead Compounds in Water, Sediment, and Fish Samples," Anal. Chem., 51(2), 1979, pp. 186-188.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$220-300 (list).

THALLIC OXIDE (CAS Number 1314-32-5)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

See "Thallium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Thallium," Appendix B.

Water Samples Laboratory Method

See "Thallium," Appendix B.

THALLIUM (CAS Number 7440-28-0)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

 $\frac{\texttt{Soil/Sediment Samples}}{\texttt{Field Method}}$

Reacts with water.

Water Samples Field Method

Reacts with water.

Air Samples Laboratory Method

See "Thallium," Appendix B.

Soil/Sediment Samples
Laboratory Method

Reacts with water.

See "Thallium," Appendix B.

Water Samples Laboratory Method

Reacts with water.

See "Thallium," Appendix B.

THALLIUM(I)ACETATE (CAS Number 563-68-8)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially chronically toxic.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples
Laboratory Method

See "Thallium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Thallium," Appendix B.

Water Samples
Laboratory Method

See "Acid Anions," Appendix B. See "Thallium," Appendix B.

THALLIUM(I)CARBONATE (CAS Number 6533-73-9)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

See "Nitrates," Appendix B. See "Thallium Compounds," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Thallium," Appendix B.

Water Samples Laboratory Method

See "Thallium," Appendix B.

THALLIUM(I)CHLORIDE (CAS Number 7791-12-0)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

<u>Air Samples</u> Laboratory Method

See "Chlorides," Appendix B. See "Thallium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Thallium," Appendix B.

Water Samples Laboratory Method

See "Chloride," Appendix B. See "Thallium," Appendix B.

THALLIUM(I)NITRATE (CAS Number 10102-45-1)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Nitrate Ions," Appendix B.

Air Samples Laboratory Method

See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

See "Thallium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

See "Thallium," Appendix B.

Water Samples Laboratory Method

See "Nitrates," Appendix B. See "Thallium," Appendix B.

THALLIUM(I)SELENIDE (CAS Number 12039-52-0)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Selenium," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

Insoluble in water.

Air Samples
Laboratory Method

See "Selenium," Appendix B. See "Thallium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Selenium," Appendix B. See "Thallium," Appendix B.

Water Samples Laboratory Method

See "Selenium," Appendix B. See "Thallium," Appendix B.

THALLIUM(I)SULFATE (CAS Number 7446-18-6)

RO: 454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

See "Nitrates and Sulfates," Appendix B. See "Sulfates (Particulate)," Appendix B.

See "Sulfur Compounds (Particulate)," Appendix B.

See "Thallium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Nitrates and Sulfates," Appendix B.

See "Thallium," Appendix B.

Water Samples Laboratory Method

See "Thallium," Appendix B.

See "Sulfates," Appendix B.

URANYL ACETATE (CAS Number 541-09-3)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

Soil/Sediment Samples
Laboratory Method

Water Samples Laboratory Method

See "Uranium," Appendix B.

URANYL NITRATE (CAS Numbers 10102-06-4, 36478-76-9)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Nitrate Ions," Appendix B.

Air Samples Laboratory Method

See "Nitrates," Appendix B. See "Nitrates and Sulfates," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Nitrates," Appendix B. See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Nitrates," Appendix B. See "Uranium," Appendix B.

VANADIUM PENTOXIDE (CAS Number 1314-62-1)

RQ: 454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

METHOD SUMMARY:

Analysis for particulate vanadium pentoxide in air can be performed by membrane-filtration of 25 liters of air, followed by alkaline digestion of the sampling medium and analysis of the resulting solution for vanadium by atomic absorption spectrometry (AA) using graphite-furnace atomization. The method is specific for vanadium oxides, not vanadium metal.

INTERFERENCES:

There are no reported interferences.

QUALITY CONTROL:

A method blank should be processed with each set of 10 or fewer samples. The percent recovery of spiked samples should be determined and, if it is less than 95 percent, results should be corrected accordingly. Duplicate analyses should agree within 5 percent.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the ranges of 0.2 to 0.9 milligrams per cubic meter in a 25-liter air sample and 0.06 to 0.29 milligrams per cubic meter in a 22.5-liter air sample. Precision and accuracy information is furnished.

REFERENCES:

- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, Method S391; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.
- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, Method S388; Publication No. 78-175, U.S. DHEW: Cincinnati, 1978.
- U.S. Department of Health, Education, and Welfare, <u>Ten NIOSH Analytical Methods</u>, Set 1, Method S391; PB-271 712 (NTIS), U.S. DHEW: Cincinnati, 1977.
- U.S. Department of Health, Education, and Welfare, <u>Ten NIOSH Analytical Methods</u>, <u>Set 2</u>, Method S388; PB-271 464 (NTIS), U.S. DHEW: Cincinnati, July 1977.

COST INFORMATION:

Cost per sample for analysis by furnace AA is approximately \$18-50 (list), plus sampling costs.

Note: NIOSH Mehod P&CAM 290 (analysis by graphite furnace AA) is applicable. (Reference furnished by reviewer).

See also: "Vanadium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Vanadium," Appendix B.

Water Samples
Laboratory Method

See "Vanadium," Appendix B.

VANADYL SULFATE (CAS Number 27774-13-6)

RQ: 454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

See "Nitrates and Sulfates," Appendix B.

See "Sulfates (Particulate), "Appendix B.

See "Sulfur Compounds (Particulate)," Appendix B.

See "Vanadium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Nitrates and Sulfates," Appendix B.

See "Vanadium," Appendix B.

Water Samples Laboratory Method

See "Sulfates," Appendix B. See "Vanadium," Appendix B.

ZINC (CAS Number 7440-66-6)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Zinc," Appendix B.

Water Samples Field Method

See "Zinc," Appendix B.

Air Samples Laboratory Method

See "Zinc," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Zinc," Appendix B.

Water Samples Laboratory Method

See "Zinc," Appendix B.

RQ: 454 kg

SAFETY INFORMATION: Potentially chronically toxic.

Air Samples Field Method

See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Zinc," Appendix B.

Water Samples Field Method

See "Zinc," Appendix B.

Air Samples Laboratory Method

See "Zinc," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Zinc," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B. See "Zinc," Appendix B.

ZINC AMMONIUM CHLORIDE (CAS Numbers 52628-25-8, 14639-97-5, 14639-98-6)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

Air Samples Field Method

See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Zinc," Appendix B.

Water Samples Field Method

See "Ammonia," Appendix B. See "Halides, Cyanides, and Other Compounds," Appendix B.

See "Volatile Species," Appendix B.

See "Zinc," Appendix B.

Air Samples Laboratory Method

See "Ammonia," Appendix B. See "Zinc," Appendix B.

> Soil/Sediment Samples Laboratory Method

See "Ammonia," Appendix B. See "Zinc," Appendix B.

> Water Samples Laboratory Method

See "Ammonia," Appendix B. See "Zinc," Appendix B.

RQ: 454 kg

SAFETY INFORMATION: Potentially chronically toxic.

Air Samples Field Method

See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Zinc," Appendix B.

Water Samples Field Method

See "Zinc," Appendix B.

Air Samples Laboratory Method

See "Zinc," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Zinc," Appendix B.

Water Samples Laboratory Method

See "Zinc," Appendix B.

ZINC BROMIDE (CAS Number 7699-45-8)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

Air Samples Field Method

See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Zinc," Appendix B.

Water Samples Field Method

See "Halides, Cyanides, and Other Compounds," Appendix B. See "Zinc," Appendix B.

Air Samples Laboratory Method

See "Zinc," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Zinc," Appendix B.

Water Samples Laboratory Method

See "Zinc," Appendix B.
See "Iodides and Bromides," Appendix B.

ZINC CARBONATE (CAS Number 3486-35-9)

RQ: 454 kg

SAFETY INFORMATION: Potentially chronically toxic.

Air Samples Field Method

See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Zinc," Appendix B.

Water Samples Field Method

See "Halides, Cyanides, and Other Compounds," Appendix B. See "Zinc," Appendix B.

Air Samples
Laboratory Method

See "Zinc," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Zinc," Appendix B.

Water Samples Laboratory Method

See "Zinc," Appendix B.

ZINC CHLORIDE (CAS Number 7646-85-7)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Zinc," Appendix B.

Water Samples Field Method

See "Halides, Cyanides, and Other Compounds," Appendix B. See "Zinc," Appendix B.

Air Samples Laboratory Method

See "Chlorides," Appendix B. See "Zinc," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Zinc," Appendix B.

Water Samples Laboratory Method

See "Chlorides," Appendix B. See "Zinc," Appendix B.

ZINC CYANIDE (CAS Number 557-21-1)

RQ: 4.54 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Cyanides," Appendix B. See "Zinc," Appendix B.

Water Samples Field Method

See "Cyanides," Appendix B.

See "Halides, Cyanides, and Other Compounds," Appendix B.

See "Zinc," Appendix B.

Air Samples
Laboratory Method

See "Cyanides," Appendix B. See "Zinc," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Cyanides," Appendix B. See "Zinc," Appendix B.

Water Samples Laboratory Method

See "Cyanides," Appendix B. See "Zinc," Appendix B.

ZINC FLUORIDE (CAS Number 7783-49-5)

RQ: 454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Fluorides," Appendix B. See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Zinc," Appendix B.

Water Samples Field Method

See "Fluorides and Other Compounds," Appendix B. See "Zinc," Appendix B.

Air Samples
Laboratory Method

See "Fluorides," Appendix B.

See "Fluorides and Hydrogen Fluoride," Appendix B.

See "Zinc," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Zinc," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B.

See "Fluorides," Appendix B.

See "Zinc," Appendix B.

ZINC FORMATE (CAS Number 557-41-5)

RQ: 454 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Zinc," Appendix B.

Water Samples Field Method

See "Zinc," Appendix B.

Air Samples Laboratory Method

See "Zinc," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Zinc," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B. See "Zinc," Appendix B.

ZINC HYDROSULFITE (CAS Number 7779-86-4)

RQ: 454 kg

SAFETY INFORMATION: Potentially chronically toxic.

Air Samples Field Method

See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Zinc," Appendix B.

Water Samples Field Method

See "Sulfite Ions," Appendix B. See "Zinc," Appendix B.

Air Samples Laboratory Method

See "Sulfur Compounds (Particulate)," Appendix B. See "Zinc," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Zinc," Appendix B.

Water Samples Laboratory Method

See "Sulfites," Appendix B. See "Zinc," Appendix B.

ZINC NITRATE (CAS Number 7779-88-6)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Zinc," Appendix B.

Water Samples Field Method

See "Nitrate Ions," Appendix B. See "Zinc," Appendix B.

Air Samples Laboratory Method

See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

See "Zinc," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Nitrates," Appendix B.

See "Nitrates and Sulfates," Appendix B.

See "Zinc," Appendix B.

Water Samples
Laboratory Method

See "Nitrates," Appendix B. See "Zinc," Appendix B.

ZINC PHENOLSULFONATE (CAS Number 127-82-2)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Phenolic Compounds," Appendix B. See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Phenolic Compounds," Appendix B. See "Zinc," Appendix B.

Water Samples Field Method

See "Zinc," Appendix B.

Air Samples Laboratory Method

See "Zinc," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Zinc," Appendix B.

Water Samples
Laboratory Method

See "Zinc," Appendix B.

ZINC PHOSPHIDE (CAS Number 1314-84-7)

RQ: 454 kg

SAFETY INFORMATION: Potentially chronically toxic. Forms toxic phosphine upon

contact with water.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

Reacts with water.

See "Zinc," Appendix B.

Water Samples Field Method

Reacts with water.

See "Zinc," Appendix B.

Air Samples Laboratory Method

See "Phosphorus," Appendix B. See "Zinc," Appendix B.

Soil/Sediment Samples
Laboratory Method

Reacts with water.

See "Phosphorus," Appendix B. See "Zinc," Appendix B.

Water Samples Laboratory Method

Reacts with water.

See "Phosphorus," Appendix B. See "Zinc," Appendix B.

ZINC SILICOFLUORIDE (CAS Number 16871-71-9)

RQ: 2270 kg

SAFETY INFORMATION: Potentially chronically toxic.

Air Samples Field Method

See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Zinc," Appendix B.

Water Samples Field Method

See "Zinc," Appendix B.

Air Samples Laboratory Method

See "Zinc," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Zinc," Appendix B.

Water Samples Laboratory Method

See "Fluorides," Appendix B. See "Zinc," Appendix B.

ZINC SULFATE (CAS Number 7733-02-0)

RQ: 454 kg

SAFETY INFORMATION: Potentially chronically toxic.

Air Samples Field Method

See "Zinc and Compounds (Particulate)," Appendix B.

Soil/Sediment Samples Field Method

See "Zinc," Appendix B.

Water Samples Field Method

See "Zinc," Appendix B.

Air Samples Laboratory Method

See "Nitrates and Sulfates," Appendix B.

See "Sulfates (Particulate)," Appendix B.

See "Sulfur Compounds (Particulate)," Appendix B.

See "Zinc," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Nitrates and Sulfates," Appendix B. See "Zinc," Appendix B.

Water Samples Laboratory Method

See "Sulfates," Appendix B. See "Zinc," Appendix B.

ZIRCONIUM NITRATE (CAS Number 13746-89-9)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Nitrate Ions," Appendix B. See "Zirconium," Appendix B.

Air Samples Laboratory Method

See "Nitrates," Appendix B. See "Nitrates and Sulfates," Appendix B. See "Zirconium," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Nitrates," Appendix B. See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

See "Nitrates," Appendix B.

ZIRCONIUM POTASSIUM FLUORIDE (CAS Number 16923-95-8)

RQ: 454 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Fluorides," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Fluorides and Other Compounds," Appendix B.

See "Potassium," Appendix B.

See "Zirconium," Appendix B.

Air Samples Laboratory Method

See "Potassium," Appendix B. See "Zirconium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Potassium," Appendix B.

Water Samples Laboratory Method

See "Acid Anions," Appendix B. See "Fluorides," Appendix B. See "Potassium," Appendix B.

ZIRCONIUM SULFATE (CAS Number 14644-61-2)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

See "Zirconium," Appendix B.

Air Samples Laboratory Method

See "Nitrates and Sulfates," Appendix B.

See "Sulfur Compounds (Particulate)," Appendix B.

See "Zirconium," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Nitrates and Sulfates," Appendix B.

Water Samples Laboratory Method

ZIRCONIUM TETRACHLORIDE (CAS Number 10026-11-6)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

Reacts with water.

Water Samples Field Method

Reacts with water.

See "Zirconium," Appendix B.

Air Samples
Laboratory Method
Reacts with water.

See "Zirconium," Appendix B.

Soil/Sediment Samples
Laboratory Method

Reacts with water.

Water Samples Laboratory Method

Reacts with water.

Part II: Organic Compounds

ACENAPHTHENE (CAS Number 83-32-9)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially carcinogenic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

See "Polycyclic Aromatic Hydrocarbons," Appendix B.

Soil/Sediment Samples
Laboratory Method

See "Acid and Base/Neutral Extractable Organics," Appendix B. See "Polycyclic Aromatic Hydrocarbons," Appendix B.

Water Samples Laboratory Method

See "Base/Neutral Extractable Organic Compounds," Appendix B.

See "Organic Compounds," Appendix B.

See "Polycyclic Aromatic Hydrocarbons," Appendix B.

See "Trace Organic Contaminants," Appendix B.

ACENAPHTHYLENE (CAS Number 208-96-8)

RQ: 0.454 kg

SAFETY INFORMATION: Potentially carcinogenic.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

Soil/Sediment Samples Laboratory Method

See "Acid and Base/Neutral Extractable Organics," Appendix B. See "Polycyclic Aromatic Hydrocarbons," Appendix B.

Water Samples
Laboratory Method

See "Base/Neutral Extractable Organic Compounds," Appendix B. See "Polycyclic Aromatic Hydrocarbons," Appendix B.

ACETALDEHYDE (CAS Number 75-07-0)

RQ: 454 kg

SAFETY INFORMATION: Flash point -38°F (-39°C).

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

METHOD SUMMARY:

Analysis for acetaldehyde in air may be performed by use of a detector tube. A 2-liter air sample is collected and analyzed with a hand-operated bellows pump and an acetaldehyde gas detector tube (Draeger® 67 26665, or equivalent). A positive result is indicated by a color change in the tube from orange to brownish-green.

INTERFERENCES:

Other aldehydes and easily oxidized organic compounds may also give positive results.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is sensitive to concentration levels of approximately 180 to 1800 milligrams per cubic meter.

REFERENCES:

National Draeger, Inc., <u>Draeger® Detector Tube Handbook</u>, "Air Investigations and Technical Gas Analysis with Draeger® Tubes", 4th ed.; Draeger: Pittsburgh, August 1979.

National Draeger, Inc., Bulletin No. ND-105; Draeger: Pittsburgh, January 1, 1982.

National Draeger, Inc., Measurements with Draeger® Tubes in the Threshold Limit Value Range, Draeger® Gas Analysis Product Information, Leaflet 4340.3e; Draeger: Pittsburgh, December 1981.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$13-20 (list), plus sampling costs.

Soil/Sediment Samples Field Method

Water Samples Field Method

METHOD SUMMARY:

Acetaldehyde in water samples can be measured colorimetrically. The Acetaldehyde reacts with propionaldehyde (3-pheny-12-quinoxalinyl)-hydrazone in 95 percent ethanol to form a red product. Reaction is complete in 20 minutes. The absorbance of the solution is read at 465 nm on a portable spectrometer.

INTERFERENCES:

Aromatic aldehydes, glucose, and ketones do not react.

QUALITY CONTROL

No quality control procedures are given.

SENSITIVITY

This method is suitable for acetaldehyde concentrations between approximately $2\ \text{and}\ 44\ \text{milligrams}$ per liter.

REFERENCE:

Tagami, S.; Nakamura, T.; Nakano, T.; Shiho, D. "Characteristic Detection and Determination of Aliphatic Aldehydes," Chem. Pharm. Bull., 23(4), 1975, pp. 891-894.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$35-50 (list).

See also: "Volatile Species," Appendix B.

Air Samples
Laboratory Method

METHOD SUMMARY:

Infrared absorption spectrometry can be used to determine the concentration of acetaldehyde in air samples. The absorbance at 8900 nm (1123 cm $^{-1}$) is measured, using a 10-meter pathlength cell. Air can be sampled in a Saran® or

Mylar® plastic bag: approximately 5 liters of the sample are drawn into an evacuated cell. Continuous monitoring of air flowing through the cell is possible if the spectrometer is set to one particular wavelength. Some vapors can be adsorbed on silica gel and quantitatively desorbed into the absorption cell later. Tentative qualitative identification of compounds can be made based on a full infrared absorption spectrum.

INTERFERENCES:

Compounds present in the sample that absorb infrared radiation at the wavelength of interest will interfere.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The sensitivity of the method, using a 10-meter pathlength cell, is 55 milligrams per cubic meter. No precision or accuracy information is furnished.

REFERENCE:

American Public Health Association, "Infrared Absorption Spectroscopy," In Methods of Air Sampling and Analysis, 2nd ed., M. Katz, Ed.; APHA: Washington, 1977, pp. 79-84.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$75-85 (list), plus sampling costs.

Note: NIOSH Method S345 (bubbler collection, derivatization with Girad T reagent, analysis by HPLC) is applicable. (Reference furnished by reviewer).

See also: "Aliphatic Aldehydes," Appendix B.

See also: "Halogenated, Aromatic and Other Compounds," Appendix B.

See also: "Hazardous Organic Emissions," Appendix B.

Soil/Sediment Samples Laboratory Method

Water Samples Laboratory Method

See "Aliphatic Compounds," Appendix B.

See "Organic Compounds," Appendix B.

See "Volatile Alcohols and Other Organic Compounds," Appendix B.

See "Volatile Carbonyl Compounds," Appendix B. See "Volatile Organics," Appendix B.

ACETIC ACID (CAS Number 64-19-7)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Acetic Acid and Acetic Anhydride," Appendix B. See "Volatile Organic Compounds," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples
Laboratory Method 1

METHOD SUMMARY:

Gas chromatography may be used for the determination of acetic acid in air samples. The analysis is performed by passage of a known volume of air through a charcoal-tube sampling device, followed by desorption of the analyte with formic acid, and analysis of the resulting solution by gas chromatography with flame ionization detection (GC/FID).

INTERFERENCES:

Any compound with the same retention time as acetic acid is an interferent.

QUALITY CONTROL:

Desorption efficiency must be determined over the range of interest, and a method blank should be processed with each 10 samples. The desorption efficiency and the results of the method-blank analysis should be used in the calculation of the results.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 12.5 to 50 milligrams per cubic meter, using a 173-liter sample. The method is capable of measurement of lower levels if the desorption efficiency is adequate. Precision and accuracy information is furnished.

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REFERENCE:

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, Method S169; Publication No. 78-175, U.S. DHEW: Cincinnati, August 1978.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$70-90 (list), plus sampling costs.

Air Samples Laboratory Method 2

METHOD SUMMARY:

Infrared absorption spectrometry can be used to determine the concentration of acetic acid in air samples. The absorbance at 8500 nm (1176 cm⁻¹) is measured, using a 10-meter pathlength cell. Air can be sampled in a Saran® or Mylar® plastic bag; approximately 5 liters of the sample are drawn into an evacuated cell. Continuous monitoring of air flowing through the cell is possible if the spectrometer is set to one particular wavelength. Some vapors can be adsorbed on silica gel and quantitatively desorbed into the absorption cell later. Tentative qualitative identification of compounds can be made based on a full infrared absorption spectrum.

INTERFERENCES:

Compounds present in the sample that absorb infrared radiation at the wavelength of interest will interfere.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The sensitivity of the method, using a 10-meter pathlength cell, is 12 milligrams per cubic meter. No precision or accuracy information is furnished.

REFERENCE:

American Public Health Association, "Infrared Absorption Spectroscopy" In Methods of Air Sampling and Analysis, 2nd ed., M. Katz, Ed.; APHA: Washington, 1977, pp. 79-84.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$75-85 (list), plus sampling costs.

"Halogenated, Aromatic and Other Compounds," Appendix B. See also:

"Volatile Organic Compounds," Appendix B. See also:

Soil/Sediment Samples Laboratory Method

Water Samples Laboratory Method

See "Acid Anions," Appendix B.
See "Aliphatic Compounds," Appendix B.
See "Carboxylic Acids," Appendix B.
See "Organic Acids," Appendix B.
See "Volatile Organic Acids," Appendix B.

ACETIC ANHYDRIDE (CAS Number 108-24-7)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Acetic Acid and Acetic Anhydride," Appendix B. See "Volatile Organic Compounds," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples Laboratory Method

METHOD SUMMARY:

Analysis for acetic anhydride in air is performed by collection of the acetic anhydride from 100 liters of air in a midget bubbler and colorimetric analysis of the resulting solution. The bubbler is charged with an alkaline hydroxylamine solution, and iron(III)chloride is used to develop color in the solution. This method is a source method for high concentrations of acetic anhydride.

INTERFERENCES:

Substances containing a carbonyl group (such as aldehydes, esters, acid chlorides, and ketones) interfere. The most likely potential interference is ketene because it is likely to coexist with acetic anhydride in air.

QUALITY CONTROL:

A method blank should be analyzed with each set of samples.

EPA/TECHNICAL STATUS:

The method is approved by the National Institute for Occupational Safety and Health and has been validated over the range of 9 to 37 milligrams per cubic meter. The method is capable of measurement of lower concentrations by

use of larger samples or a longer absorption-cell pathlength. Precision and accuracy information is furnished.

REFERENCES:

- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, Method S170; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.
- U.S. Department of Health, Education, and Welfare, NIOSH Analytical Method for Set L, Method S170, PB-250 159 (NTIS), U.S. DHEW: Washington, January 1976.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$20-25 (list), plus sampling costs.

See also: "Halogenated, Aromatic, and Other Compounds," Appendix B.

Soil/Sediment Samples
Laboratory Method

Water Samples
Laboratory Method

ACETONE (CAS Number 67-64-1)

RQ: 2270 kg

SAFETY INFORMATION: Flash point -4°F.

Air Samples Field Method

See "Acetone and Methyl Ketones," Appendix B. See "Volatile Organic Compounds," Appendix B. See "Organic Vapors," Appendix B.

Soil/Sediment Samples Field Method

See "Volatile Species," Appendix B.

Water Samples Field Method

See "Volatile Species," Appendix B.

Air Samples
Laboratory Method 1

METHOD SUMMARY:

Analysis for acetone in air is performed by passage of a known volume of air through a charcoal-tube sampling device, followed by desorption of the analyte with carbon disulfide and analysis of the resulting solution by gas chromatography with flame ionization detection (GC/FID).

INTERFERENCES:

High humidity severely decreases the capacity of the sampling apparatus.

QUALITY CONTROL:

Desorption efficiency must be determined over the range of interest, and a method blank should be processed with every 10 samples. The desorption efficiency and the results of the method-blank analysis should be used in the calculation of the results.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and has been validated over the range of 1200 to 4500 milligrams per cubic meter, using a 2-liter sample. Precision and accuracy information is furnished. The method is capable of measurement of much lower levels if the desorption efficiency is adequate.

REFERENCES:

- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, Method S1; Publication No. 77-157-B, U.S. DHEW:
- U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set A, Method S1; PB-245 850 (NTIS), U.S. DHEW: Washington, October 1975.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$70-90, plus sampling costs.

Air Samples Laboratory Method 2

METHOD SUMMARY:

Infrared absorption spectrometry can be used to determine the concentration of acetone in air samples. The absorbance at 8200 nm (1219 cm⁻¹) is measured, using a 10-meter pathlength cell. Air can be sampled in a Saran® or Mylar® plastic bag; approximately 5 liters of the sample are drawn into an evacuated cell. Continuous monitoring of air flowing through the cell is possible if the spectrometer is set to one particular wavelength. Some vapors can be adsorbed on silica gel and quantitatively desorbed into the absorption cell later. Tentative qualitative identification of compounds can be made based on a full infrared absorption spectrum.

INTERFERENCES:

Compounds present in the sample that absorb infrared radiation at the wavelength of interest will interfere.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The sensitivity of the method, using a 10-meter pathlength cell, is 12 milligrams per cubic meter. No precision or accuracy information is furnished.

REFERENCE:

American Public Health Association, "Infrared Absorption Spectroscopy." In Methods of Air Sampling and Analysis, 2nd ed., M. Katz, Ed.; APHA: Washington, 1977, pp. 79-84.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$75-85 (list), plus sampling costs.

"Halogenated, Aromatic, and Other Compounds," Appendix B. See also:

"Hazardous Organic Emissions," Appendix B.
"Volatile Carbonyl Compounds," Appendix B.
"Volatile Organic Compounds," Appendix B. See also: See also: See also: "Volatile Organic Solvents," Appendix B. See also:

Soil/Sediment Samples Laboratory Method

See "Volatile Organic Compounds," Appendix B.

Water Samples Laboratory Method

See "Aliphatic Compounds," Appendix B.

See "Organic Compounds," Appendix B.

See "Volatile Alcohols and Other Organic Compounds," Appendix B.

See "Volatile Carbonyl Compounds," Appendix B. See "Volatile Organic Compounds," Appendix B.

See "Volatile Organics," Appendix B.

ACETONE CYANOHYDRIN (CAS Number 75-86-5)

RQ: 4.54 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Volatile Organic Compounds," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

METHOD SUMMARY:

Acetone cyanohydrin can be detected in water samples by visible spectro-photometry. Copper sulfate solution and ammonia are added to a water sample, and the blue tetraamine copper complex is destroyed by acetone cyanohydrin. (This method is described by Légrádi, L. Mikrochim.Acta 4, 1974, pp. 759-764, and is summarized in the reference given below.)

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

SENSITIVITY:

This method is sensitive to concentration levels of acetone cyanohydrin higher than 50 micrograms.

REFERENCE:

Simons, J. R. <u>Bibliography on Hazardous Materials Analysis Methods</u>; EPA 600/2-81-094, U.S. Environmental Protection Agency: Cincinnati, 1981, p. 12.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$15-23 (list).

See also: "Halides, Cyanides, and Other Compounds," Appendix B.

<u>Air Samples</u> Laboratory Method

METHOD SUMMARY:

Gas chromatography may be used for the analysis of acetone cyanohydrin in air samples. A known volume of air is drawn through a tube containing Porapak QS®, followed by desorption with ethyl acetate. An aliquot of the resulting solution is injected into a gas chromatograph equipped with a nitrogen-phosphorus detector (GC/NPD).

INTERFERENCES:

Samples taken in humid atmospheres should be refrigerated immediately to protect the analyte from decomposition in the presence of water vapor.

QUALITY CONTROL:

Method blanks and standards are processed with the samples.

EPA/TECHNICAL STATUS:

This method has been proposed by the National Institute for Occupational Safety and Health (NIOSH) for use over the range of 0.33 to 16.7 milligrams per cubic meter for a 3-liter sample. Precision and accuracy information is furnished. This method should be applicable over a broader range of concentrations, provided that good desorption efficiency can be achieved.

REFERENCE:

U.S. Department of Health and Human Services, NIOSH Manual of Analytical Methods, Vol. 7, Method P&CAM 340; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$130 (list), plus sampling costs.

Soil/Sediment Samples
Laboratory Method

Water Samples Laboratory Method

See "Substituted Nitriles and Tetranitromethane," Appendix B.

ACETONITRILE (CAS Number 75-05-8)

RQ: 454 kg

SAFETY INFORMATION: Exhibits chronic toxicity. Flash point 42°F.

CAUTION: The user should obtain additional toxicity/hazard

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Organic Vapors," Appendix B. See "Volatile Organic Compounds," Appendix B.

Soil/Sediment Samples

Water Samples Field Method

Air Samples
Laboratory Method 1

METHOD SUMMARY:

Analysis for acetonitrile in air is performed by passage of a known volume of air through a charcoal-tube air sampler, followed by desorption of the analyte with benzene and gas chromatographic analysis with flame ionization detection (GC/FID) of the resulting solution.

INTERFERENCES:

High humidity severely decreases the efficiency of the sampling apparatus.

QUALITY CONTROL:

Desorption efficiency must be determined and results corrected accordingly. A method blank should be analyzed with each set of samples and the results used to correct the data for each sample.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 30 to 140 milligrams per cubic meter in a 10-liter sample. The method should be applicable over a broader range of concentrations, provided that good desorption efficiency can be achieved. Precision and accuracy information is furnished.

REFERENCES:

- U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set L, Method S165; PB-250 159 (NTIS), U.S. DHEW: Washington, January 1976.
- U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, Method S165; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

The cost per sample for analysis by this method is approximately \$70-90 (list), plus sampling costs.

Air Samples Laboratory Method 2

METHOD SUMMARY:

Infrared absorption spectrometry can be used to determine the concentration of acetonitrile in air samples. The absorbance at 9580 nm (1044 cm⁻¹) is measured, using a 10-meter pathlength cell. Air can be sampled in a Saran® or Mylar® plastic bag; approximately 5 liters of the sample as drawn into an evacuated cell. Continuous monitoring of air flowing through the cell is possible if the spectrometer is set to one particular wavelength. Some vapors can be adsorbed on silica gel and quantitatively desorbed into the absorption cell later. Tentative qualitative identification of compounds can be made based on a full infrared absorption spectrum.

INTERFERENCES:

Compounds present in the sample that absorb infrared radiation at the wavelength of interest will interfere.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The sensitivity of the method, using a 10-meter pathlength cell, is 160 milligrams per cubic meter. No precision or accuracy information is furnished.

REFERENCE:

American Public Health Association, "Infrared Absorption Spectroscopy." In Methods of Air Sampling and Analysis, 2nd ed., M. Katz, Ed.; APHA: Washington, 1977, pp. 79-84.

COST INFORMATION:

Cost per sample for analysis by this method is approximately \$75-85 (list), plus sampling costs.

See also: "Gaseous Contaminants," Appendix B.

See also: "Halogenated, Aromatic, and Other Compounds," Appendix B.

Soil/Sediment Samples Laboratory Method

See "Non-Halogenated Volatile Organic Compounds," Appendix B.

Water Samples Laboratory Method

See "Nitriles," Appendix B.

See "Non-Halogenated Volatile Organic Compounds," Appendix B.

See "Organic Compounds," Appendix B.

See "Volatile Alcohols and Other Organic Compounds," Appendix B.

ACETOPHENONE (CAS Number 98-86-2)

RQ: 2270 kg

SAFETY INFORMATION: CAUTION: The user should obtain additional toxicity/hazaro

information before handling this substance or

using this analytical procedure.

Air Samples Field Method

See "Volatile Organic Compounds," Appendix B.

Soil/Sediment Samples Field Method

> Water Samples Field Method

Air Samples
Laboratory Method

See "Halogenated, Aromatic, and Other Compounds," Appendix B. See "Hazardous Organic Emissions," Appendix B.

Soil/Sediment Samples
Laboratory Method

Water Samples
Laboratory Method

See "Trace Organic Contaminants," Appendix B.
See "Volatile Alcohols and Other Organic Compounds," Appendix B.