ANNOTATED BIBLIOGRAPHY OF ANALYTICAL METHODS FOR CERCLA HAZARDOUS SUBSTANCES

by

H. B. Kerfoot, J. L. Engels, D. F. Arnold, and A. R. Bujold
Lockheed Engineering and Management Services Company
Las Vegas, Nevada 89114

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Project Officer

Werner F. Beckert
Quality Assurance Division
Environmental Monitoring Systems Laboratory
Las Vegas, Nevada 89114

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
LAS VEGAS, NEVADA 89114

NITROGEN DIOXIDE AND NITRIC OXIDE

Air Samples
Laboratory Method 2

Compound	CAS Number
Nitric Oxide	10102-43-9
Nitrogen Dioxide	10102-44-0

METHOD SUMMARY:

Analysis of air samples for the above oxides of nitrogen can be performed by chromic acid oxidation and reaction with an azo-dye-forming reagent, followed by colorimetry. A known volume of air (up to 2.4 liters) is passed through a chromic acid oxidizer, nitric oxide is converted to nitrogen dioxide by chromic acid, and the resulting nitrogen dioxide and the nitrogen dioxide originally present are absorbed in an aqueous solution of sulfanilic acid, acetic acid, acetone, and N-(1-naphthyl)-ethylenediamine dihydrochloride. The red-violet color produced is measured spectrophotometrically at 550 nanometers. This method does not identify any particular compound, but measures the total amount of nitrogen dioxide present after oxidation of the sample by chromic acid.

INTERFERENCES:

Any significant interferences due to sulfur dioxide should be eliminated by the oxidation step. The addition of acetone to the color reagent retards the fading of color from the presence of sulfur dioxide. A five-fold ratio of ozone to nitrogen dioxide causes a slight orange tint in the solution.

QUALITY CONTROL:

No quality control procedures are provided.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The method is applicable to total nitric oxide/nitrogen dioxide concentrations in the range 4 to 10,000 micrograms per cubic meter. Precision information is furnished.

REFERENCE:

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.03, D3608-77; ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost per sample for analyses by this method is approximately $30-40, plus sampling costs.

NITROGEN DIOXIDE AND NITRIC OXIDE

Air Samples
Laboratory Method 3

Compound	CAS Number
Nitric Oxide	10102-43-9
Nitrogen Dioxide	10102-44-0

METHOD SUMMARY:

Nitric oxide and nitrogen dioxide levels in air samples can be determined by chemiluminescence. For measurement of only nitric oxide, the gas being sampled is blended with ozone in a flow reactor, and the intensity of emitted light from the reaction of nitric oxide with ozone is measured. To measure total nitric oxide and nitrogen dioxide, the gas is diverted through a converter that reduces nitrogen dioxide to nitric oxide before being admitted to the flow reactor. The concentration of nitrogen dioxide can be obtained by subtraction.

INTERFERENCES:

Possible interferences from hydrocarbons are eliminated by use of a red sharp-cut off filter. Any compounds converted to nitric oxide in the converter are potential interferences; ammonia does not interfere when the converter temperature is below 300°C. Other organic nitrogen compounds may decompose to liberate nitric oxide in the converter.

QUALITY CONTROL:

No quality control procedures are provided.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The method is applicable over the following ranges: nitric oxide, 10 micrograms to 30 milligrams per cubic meter; nitrogen dioxide, 20 micrograms to 50 milligrams per cubic meter. Precision and accuracy information is furnished.

REFERENCE:

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.03, D3824-79; ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost information has not been obtained.

PHOSPHORUS

Air Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Aluminum Phosphide	20859-73-8	Sodium Phosphate, Tribasic	7601-54-9
O-O-Diethyl S-Methyl Dithiophosphate	3288-58-2		7785-84-4
Diethyl-p-nitrophenyl Phosphate	311-45-5		10101-89-0
Diisopropyl Fluorophosphate	55-91-4		10361-89-4
Hexaethyl Tetraphosphate	757-58-4		7758-29-4
Lead Phosphate	7446-27-7		10124-56-8
Octamethylpyrophosphoramide	152-16-9	Tetraethyldithiopyrophosphate	3689-24-5
Phosphoric Acid	7664-38-2	Tetraethyl Pyrophosphate	107-49-3
Phosphorus	7723-14-0	Tris(2,3-Dibromopropyl) Phosphate	126-72-7
Phosphorus Oxychloride	10025-87-3	Zinc Phosphide	1314-84-7
Phosphorus Pentasulfide	1314-80-3		
Phosphorus Trichloride	7719-12-2		
Sodium Phosphate, Dibasic	7558-79-4		
	10039-32-4		
	10028-24-7		
	10140-65-5		

METHOD SUMMARY:

Analysis for the above phosphorus-containing compounds in air may be performed by inductively coupled plasma atomic emission spectroscopic (ICP) analysis for phosphorus. A known volume of air is drawn through a cellulose ester membrane filter. The filter is ashed with a mixture of nitric and perchloric acids and the digest analyzed by ICP. This method does not identify any specific phosphorus-containing compound, but measures the total phosphorus content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks, spikes, and standards are prepared and 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 5 to 2000 micrograms phosphorus per cubic meter for a 500-liter sample. Limited 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 351; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

COST INFORMATION:

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

PHOSPHORUS

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Aluminum Phosphide	20859-73-8	Phosphoric Acid	7664-38-2
Chlorpyrifos	2921-88-2	Phosphorus	7723-14-0
Coumaphos	56-72-4	Phosphorus Oxychloride	10025-87-3
Diazinon	333-41-5	Phosphorus Pentasulfide	1314-80-3
Dichlorvos	62-73-7	Phosphorus Trichloride	7719-12-2
O-O-Diethyl S-Methyl Dithiophosphate	3288-58-2	Sodium Phosphate, Dibasic	7558-79-4
			10039-32-4
Diethyl-p-nitrophenyl Phosphate	311-45-5		10028-24-7
			10140-65-5
Diisopropyl Fluorophosphate	55-91-4	Sodium Phosphate, Tribasic	7601-54-9
Dimethoate	60-51-5		7785-84-4
Disulfoton	298-04-4		10101-89-0
Ethion	563-12-2		10361-89-4
Hexaethyl Tetraphosphate	757-58-4		7758-29-4
Lead Phosphate	7446-27-7		10124-56-8
Malathion	121-75-5	Tetraethyldithiopyrophosphate	3689-24-5
Methyl Parathion	298-00-0		
Mevinphos	7786-34-7	Tetraethyl Pyrophosphate	107-49-3
Naled	300-76-5	Tris(2,3-Dibromopropyl) Phosphate	126-72-7
Octamethylpyrophosphoramide	152-16-9		
Parathion	56-38-2	Zinc Phosphide	1314-84-7
Phorate	298-02-2		

METHOD SUMMARY:

Total recoverable phosphorus and organic phosphorus, resulting from the compounds listed above, in sediment samples can be measured colorimetrically. The sediment is ignited at 550°C for 2 hours, after which the residue is acid-digested. The orthophosphate concentration in the acid extract is a measure of the total phosphorus content; the inorganic phosphorus is determined by digestion without ignition; the organic phosphorus is estimated by taking the difference. The color development is based on the reaction of ammonium molybdate with orthophosphate, followed by reduction with ascorbic acid to form a blue molybdenum complex. Either manual or automated procedures may be used. Antimony tartrate may be added to facilitate color development when following the automated procedure. These methods measure all forms of phosphorus except polyphosphates and minerals containing tightly bound phosphorus. They do not identify the specific phosphorus-containing compounds present, but measure the total phosphorus content of the sample.

INTERFERENCES:

Vanadium, zirconium, germanium, sulfide, tannins, lignins, hexavalent chromium, arsenic, and silica can interfere, but the effects are reduced by sample dilution. To prevent microbiological transformations that can result in

changes of concentration of available phosphate, samples should be drained of excess water immediately after collection and stored at 5°C or lower in the dark.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The analytical range is 100 to 2000 micrograms per gram of sample. The range may be extended upwards by diluting the sample. Precision and accuracy information is furnished.

REFERENCES:

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, Method D515-82; ASTM: Philadelphia, 1983, pp. 575-587.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, Method D4183-82; ASTM: Philadelphia, 1983, pp. 712-717.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Methods 424C, 424D; APHA: Washington, 1980.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70 (list).

PHOSPHORUS

Water Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Aluminum Phosphide	20859-73-8	Sodium Phosphate,	7601-54-9
O-O-Diethyl S-Methyl	3288-58-2	Tribasic	7785-84-4
Dithiophosphate			10101-89-0
Diethyl-p-nitrophenyl	311-45-5		10361-89-4
Phosphate			7758-29-4
Diisopropyl Fluorophosphate	55-91-4		10124-56-8
Hexaethyl Tetraphosphate	757-58-4	Tetraethyldithiopyro-	3689-24-5
Lead Phosphate	7446-27-7	phosphate	
Octamethylpyrophosphoramide	152-16-9	Tetraethyl Pyrophosphate	107-49-3
Phosphoric Acid	7664-38-2	Tris(2,3-Dibromopropyl)	126-72-7
Phosphorus	7723-14-0	Phosphate	
Phosphorus Oxychloride	10025-87-3	Zinc Phosphide	1314-84-7
Phosphorus Pentasulfide	1314-80-3		
Phosphorus Trichloride	7719-12-2		
Sodium Phosphate, Dibasic	7558-79-4		
	10039-32-4		
	10028-24-7		
	10140-65-5		

METHOD SUMMARY:

Analysis of water samples for the above phosphorus-containing compounds and phosphorus ions can be performed by automated colorimetric analysis for phosphorus. A 25-milliliter sample is digested with sulfuric acid, mercuric sulfate, and potassium sulfate, heated for several hours in a block digester, a molybdate/antimony solution is added, and the resulting solution is analyzed colorimetrically. This method does not differentiate between various phosphorus-containing compounds, but gives the total amount of phosphorus present in the sample.

INTERFERENCES:

Sample containers should be made of plastic or of borosilicate glass. Samples should be preserved by acidification and refrigeration at 4°C if the analysis cannot be performed on the day of collection.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is EPA-approved for the National Pollutant Discharge Elimination System (NPDES) of the Clean Water Act. Precision and accuracy information is provided.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 365.4; EPA-600/4-79-020, U.S. EPA: Cincinnati, March 1979.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Methods 424C, 424D; APHA: Washington, 1980.

American Society for Testing and Materials, 1983 Annual ASTM Book of Standards, Vol. 11.01, Method D515-82(D); ASTM: Philadelphia, 1983.

Alberta Environmental Centre, Methods Manual for Chemical Analysis of Water and Wastes, "Phosphorus, Total;" Publication No. AECV81-M1, Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $45-50 (list).

POTASSIUM

Air Samples
Laboratory Method

Compound	CAS Number
Antimony Potassium Tartrate	28300-74-5
Potassium Arsenate	7784-41-0
Potassium Arsenite	10124-50-2
Potassium Chromate	7789-00-6
Potassium Cyanide	151-50-8
Potassium Dichromate	7778-50-9
Potassium Hydroxide	1310-58-3
Potassium Permanganate	7722-64-7
Potassium Silver Cyanide	506-61-6
Zirconium Potassium Fluoride	16923-95-8

METHOD SUMMARY:

Analysis of air samples for the above potassium compounds can be performed by atomic absorption spectrometric (AA) analysis for potassium. Samples are collected on cellulose ester membrane filters. The filter and the particulates are digested with nitric acid, the digest diluted and analyzed by AA. This method does not differentiate between potassium-containing compounds, but measures the total potassium 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 for use over the range of 4.2 to 84 micrograms potassium per cubic meter in a 240-liter sample of air. Precision information is furnished.

REFERENCES:

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

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption;" HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

COST INFORMATION:

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

POTASSIUM

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Antimony Potassium Tartrate	28300-74-5
Potassium Arsenate	7784-41-0
Potassium Arsenite	10124-50-2
Potassium Chromate	7789-00-6
Potassium Cyanide	151-50-8
Potassium Dichromate	7778-50-9
Potassium Hydroxide	1310-58-3
Potassium Permanganate	7722-64-7
Potassium Silver Cyanide	506-61-6
Zirconium Potassium Fluoride	16923-95-8

METHOD SUMMARY:

Analysis of soil/sediment samples for the above potassium compounds can be performed by atomic emission spectrometric analysis for potassium. A 100-milligram sample is digested with nitric acid, perchloric acid, hydrogen fluoride, and boric acid, and analyzed. Samples should be stored in polyurethane bags and frozen immediately after collection. This method does not differentiate between potassium-containing compounds, but measures the total potassium content of the sample.

INTERFERENCES:

Standard and sample compositions should be closely matched. Solvent extraction techniques may be necessary. Cesium should be added to samples and standards to overcome ionization interference.

QUALITY CONTROL:

Quality control procedures are not provided.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The detection limit of the method is approximately 100 micrograms per gram. Precision and accuracy information is furnished.

REFERENCE:

Environment Canada, Analytical Methods Manual, NAQUADAT No. 19050; Environment Canada, Inland Waters Directorate, Water Quality Branch: Ottawa, Canada, 1975.

COST INFORMATION:

The cost per sample for analysis by this method is approximately $20-30 (list).

POTASSIUM

Water Samples
Laboratory Method 1

Compound	CAS Number
Antimony Potassium Tartrate	28300-74-5
Potassium Arsenate	7784-41-0
Potassium Arsenite	10124-50-2
Potassium Chromate	7789-00-6
Potassium Cyanide	151-50-8
Potassium Dichromate	7778-50-9
Potassium Hydroxide	1310-58-3
Potassium Permanganate	7722-64-7
Potassium Silver Cyanide	506-61-6
Zirconium Potassium Fluoride	16923-95-8

METHOD SUMMARY:

Analysis of water samples for the above potassium compounds can be performed by atomic absorption (AA) spectrometric analysis for potassium. When analysis is not to be performed soon after sample collection, samples should be preserved by acidification with nitric acid. This method does not differentiate between various potassium compounds, but measures the total amount of potassium present in a 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 each time a sample or set of samples is analyzed.

EPA/TECHNICAL STATUS:

This method is approved for the National Pollutant Discharge Elimination System (NPDES). Precision and accuracy information is furnished. The detection limit of the method is 5 micrograms per liter, the sensitivity 40 micrograms per liter, and the optimum concentration range is 0.1 to 2 milligrams per liter (APHA). Precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 258.1; EPA-600/4-79-020, U.S. EPA: Cincinnati, March 1979.

America Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 322A; APHA: Washington, 1980.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption;" HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

American Society for Testing and Materials, 1983 ASTM Book of Standards, Vol. 11.01, Method D4192-82; ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $10-20 (list).

POTASSIUM

Water Samples
Laboratory Method 2

Compound	CAS Number
Antimony Potassium Tartrate	28300-74-5
Potassium Arsenate	7784-41-0
Potassium Arsenite	10124-50-2
Potassium Chromate	7789-00-6
Potassium Cyanide	151-50-8
Potassium Dichromate	7778-50-9
Potassium Hydroxide	1310-58-3
Potassium Permanganate	7722-64-7
Potassium Silver Cyanide	506-61-6
Zirconium Potassium Fluoride	16923-95-8

METHOD SUMMARY:

Analysis of water samples for the above potassium compounds can be performed by flame photometric, colorimetric, or atomic absorption (AA) analysis for potassium. In the colorimetric analysis, a 10-milliliter sample is reacted with sodium cobaltinitrite and potassium dichromate in an acidic solution. The excess dichromate is proportional to the amount of potassium originally present and is determined colorimetrically with a spectrophotometer. Lower-level samples should be analyzed by flame photometry. These methods do not identify any specific potassium compound, but measure the total potassium content of the sample.

INTERFERENCES:

When using flame photometry, samples containing particulate matter should be filtered before analysis. Calcium, magnesium, and sodium may interfere. When using colorimetry, the final colored solution may require filtration if it is turbid. Colorimetric analysis should not be performed when ammonium ions are present in the sample.

QUALITY CONTROL:

Samples should be stored in plastic containers. When performing colorimetric analysis, a series of solutions of known potassium concentration must be carried through the procedure with each set of samples. Using any method, standards should be analyzed each time a set of samples is analyzed.

EPA/TECHNICAL STATUS:

These methods are approved for the National Pollutant Discharge Elimination System (NPDES). Precision and accuracy information is furnished. Concentrations of potassium above 0.1 milligram per liter can be detected by flame photometry (APHA), and the optimum concentration range for the flame photometic method is 1 to 100 milligrams per liter (ASTM).

REFERENCES:

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 14th ed., Method 322B; APHA: Washington, 1975.

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 258.1; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, Method D4192-82; ASTM: Philadelphia, 1983.

Alberta Environmental Centre, Methods Manual for the Chemical Analysis of Water and Wastes, "Potassium, Dissolved;" Publication No. AECV81-M1, Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

COST INFORMATION:

Cost per sample for analysis by these methods is approximately $20-40 (list).

SELENIUM

Air Samples
Laboratory Method 1

Compound	CAS Number
Selenious Acid	7783-00-8
Selenium	7782-49-2
Selenium Dioxide	7746-08-4
Selenium Disulfide	7488-56-4
Selenourea	630-10-4
Sodium Selenite	10102-18-8
	7782-82-3
Thallium(I) Selenide	12039-52-0

METHOD SUMMARY:

Analysis for the above selenium compounds in air may be performed by filtration of air and analysis of the particulates on the filter for selenium by X-ray fluorescence spectrometry. The method is non-destructive, so that further analyses may be performed on samples. The sample-collection time may be up to 8 hours, but analysis time is approximately 1 minute. The method measures total selenium present in the sample and does not differentiate between chemical compounds or selenium valence states.

INTERFERENCES:

Polyvinyl chloride (PVC) filters are unacceptable for sampling, and glass-fiber filters are acceptable for use only if they are of the highest purity. Cellulose membrane, cellulose fiber, polycarbonate membrane, and fluorocarbon membrane filters are acceptable.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision information is given. The method is sensitive to amounts of selenium above 40 micrograms.

REFERENCES:

Rhodes, J. R.; Stout, J. A.; 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.

SELENIUM

Air Samples
Laboratory Method 2

Compound	CAS Number
Selenious Acid	7783-00-8
Selenium	7782-49-2
Selenium Dioxide	7746-08-4
Selenium Disulfide	7488-56-4
Selenourea	630-10-4
Sodium Selenite	10102-18-8
	7782-82-3
Thallium(I) Selenide	12039-52-0

METHOD SUMMARY:

Air samples may be analyzed for selenium and the above selenium compounds by atomic absorption spectrometric analysis (AA) for selenium. The analysis is performed by filtration of a known volume of air through a cellulose ester membrane filter, followed by acid digestion of the filter and the deposited material and AA analysis of the resulting solution. This method measures the total selenium content of the air sampled and does not identify any specific selenium compounds.

INTERFERENCES:

Flame interferences may be minimized by use of background correction and an argon-hydrogen/air flame.

QUALITY CONTROL:

Percent recovery of spiked samples must be determined and, if below 95 percent, results should be corrected accordingly. A method blank should be processed with each set of 10 or fewer samples. 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 has been validated over the range of 0.1 to 0.5 milligrams per cubic meter in a 360-liter sample. Precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set M, Method S190; 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 S190; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.

U.S. Department of Health and Human Services, NIOSH Manual of Analytical Methods, Vol. 7, Method S190; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

COST INFORMATION:

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

SELENIUM

Air Samples
Laboratory Method 3

Compound	CAS Number
Selenious Acid	7783-00-8
Selenium	7782-49-2
Selenium Dioxide	7746-08-4
Selenium Disulfide	7488-56-4
Selenourea	630-10-4
Sodium Selenite	10102-18-8
	7782-82-3
Thallium (I) Selenide	12039-52-0

METHOD SUMMARY:

Analysis for the above selenium-containing compounds in air may be performed by inductively coupled plasma atomic emission spectroscopic (ICP) analysis for selenium. A known volume of air is drawn through a cellulose ester membrane filter. The filter is ashed with a mixture of nitric and perchloric acids followed by analysis by ICP. The method does not identify any specific selenium-containing compound, but measures the total selenium content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks, spikes, and standards are prepared and 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 5 to 2000 micrograms selenium per cubic meter for a 500-liter sample. Limited 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 351; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

COST INFORMATION:

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

SELENIUM

Air Samples
Laboratory Method 4

Compound	CAS Number
Selenious Acid	7783-00-8
Selenium	7782-49-2
Selenium Dioxide	7746-08-4
Selenium Disulfide	7488-56-4
Selenourea	630-10-4
Sodium Selenite	10102-18-8
	7782-82-3
Thallium(I) Selenide	12039-52-0

METHOD SUMMARY:

The above particulate selenium compounds in air samples can be measured by reaction-kinetic analysis. The analyte is collected from a known volume of air on a membrane filter. The filter and particulates are wet-ashed with a mixture of nitric and sulfuric acid and the resulting solution is neutralized with sodium hydroxide. The selenite in the sample solution is reduced to elemental selenium by sulfite and sulfide. The elemental selenium catalyzes the sulfide reduction of methylene blue to the colorless leuco form in alkaline solution. The selenium concentration is proportional to the rate of decolorization of the methylene blue, which is measured spectrophotometrically. This method does not identify any specific selenium compounds present, but measures the total selenium content of the sample.

INTERFERENCES:

Copper(II), silver, antimony, and bismuth ions may interfere, but addition of ethylenediamine tetracetic acid (EDTA) should mask these ions.

QUALITY CONTROL:

A filter blank should be processed with the samples. Filter standards should also be processed to determine recovery. The value of the blank and the recovery should be used in the calculation of the results.

EPA/TECHNICAL STATUS:

This method is classifed as proposed by the National Institute for Occupational Safety and Health (NIOSH) for use over the range of 0.6 to 10 micrograms per cubic meter of air, with a 90-liter sample. Limited precision and accuracy information is furnished.

REFERENCE:

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, Vol. 1, Method P&CAM 181; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost information has not been obtained.

SELENIUM

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Selenious Acid	7783-00-8
Selenium	7782-49-2
Selenium Dioxide	7746-08-4
Selenium Disulfide	7488-56-4
Selenourea	630-10-4
Sodium Selenite	10102-18-8
	7782-82-3
Thallium(I) Selenide	12039-52-0

METHOD SUMMARY:

Analysis of soil/sediment samples for the above selenium compounds can be performed by atomic spectrometric analysis for selenium. A 2-gram sample is digested with nitric acid and hydrogen peroxide and analyzed by atomic spectrometry. Low selenium concentrations are determined by furnace atomic absorption spectrometry (AA). Inductively coupled plasma (ICP) spectrometry is used to analyze higher-concentration samples. These methods do not identify any specific selenium-containing compounds, but measure the total selenium content of the sample.

INTERFERENCES:

Chlorides and sulfates in the sample may interfere. Nickel is added to control sulfate interference. For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods.

QUALITY CONTROL:

A reference standard should be analyzed routinely, 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 analyses under the Clean Water Act. 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 270.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, Methods 3010, 3020, 3040, 3050, 6010, 7740; SW-846, U.S. EPA: Washington, 1982.

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

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Selenium," pp. IV-88 to IV-98; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption;" HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

COST INFORMATION:

Cost per sample for analysis by furnace AA is approximately $28-60; by ICP, it is approximately $20.

SELENIUM

Water Samples
Laboratory Method

Compound	CAS Number
Selenious Acid	7783-00-8
Selenium	7782-49-2
Selenium Dioxide	7746-08-4
Selenium Disulfide	7488-56-4
Selenourea	630-10-4
Sodium Selenite	10102-18-8
	7782-82-3
Thallium(I) Selenide	12039-52-0

METHOD SUMMARY:

Analysis of water samples for the above selenium compounds can be performed by atomic spectrometric analysis for selenium. A 100-milliliter sample is digested with nitric acid and hydrogen peroxide and analyzed by atomic spectrometry. Low selenium concentrations are determined by furnace or gaseous hydride atomic absorption spectrometry (AA). Inductively coupled plasma (ICP) spectrometry is used to analyze higher-concentration samples. These methods do not identify any specific selenium-containing compounds, but measure the total selenium content of the sample.

INTERFERENCES:

Chlorides and sulfates in the sample may interfere, and nickel can be added to control sulfate interference. For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods.

QUALITY CONTROL:

A reference standard should be analyzed routinely 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 Clean Water Act analyses. The hydride method is applicable to concentrations from 0.2 to 5 micrograms per liter (Alberta Environment). 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, Methods 270.2, 270.3; EPA-600/4-79-020, U.S. EPA: Cincinnati, March 1979.

U.S. Environmental Protection Agency, Technical Additions to Methods for Chemical Analysis of Water and Wastes, Method 200.7; EPA-600/4-82-055, U.S. EPA: Cincinnati, December 1982.

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

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3010, 3020, 3040, 3050, 6010, 7740; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Selenium," pp. IV-88 to IV-98; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption;" HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 303E; APHA: Washington, 1983.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, Method D3859-79; ASTM: Philadelphia, 1983.

Alberta Environmental Centre, Methods Manual for the Chemical Analysis of Water and Wastes, "Selenium, Total;" Publication No. AECV81-M1, Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

COST INFORMATION:

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

SILVER

Air Samples
Laboratory Method 1

Compound	CAS Number
Potassium Silver Cyanide	506-61-6
Silver	7440-22-4
Silver Cyanide	506-64-9
Silver Nitrate	7761-88-8

METHOD SUMMARY:

Analysis for silver and the above silver compounds in air can be performed by high-volume filtration with a glass-fiber filter, followed by acid digestion of the filter with particles and analysis of the resulting solution by atomic absorption spectrometry (AA). This method is not specific for particulate silver metal or any silver-containing compound, but measures all silver and silver compounds retained by the filter.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

A check standard should be analyzed each time a sample or set of samples is processed.

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 21 to 170 micrograms per cubic meter in a 240-liter sample of air. 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, 1977.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III. Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

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

SILVER

Air Samples
Laboratory Method 2

Compound	CAS Number
Potassium Silver Cyanide	506-61-6
Silver	7440-22-4
Silver Cyanide	506-64-9
Silver Nitrate	7761-88-8

METHOD SUMMARY:

Analysis for silver and the above silver-containing compounds in air may be performed by inductively coupled plasma atomic emission spectroscopic (ICP) analysis for silver. A known volume of air is drawn through a cellulose ester membrane filter. The filter is ashed with a mixture of nitric and perchloric acids and the digest analyzed by ICP. The method does not identify any specific silver-containing compound, but measures the total silver content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks, spikes, and standards are prepared and 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 5 to 2000 micrograms silver per cubic meter, using a 500-liter sample. Limited 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 351; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

COST INFORMATION:

Cost per sample for analysis by this methods is approximately $10 (list), plus sampling costs.

Note: NIOSH Method S182 (filter collection, acid digestion, analyses by DC plasma emission spectrometry [DCP]) is applicable. (Reference provided by reviewer.)

SILVER

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Potassium Silver Cyanide	506-61-6
Silver	7440-22-4
Silver Cyanide	506-64-9
Silver Nitrate	7761-88-8

METHOD SUMMARY:

Analysis for the above silver compounds in soil/sediment samples can be performed by inductively coupled plasma (ICP) or atomic absorption (AA) spectrometric analysis for silver. A 2-gram sample is digested with nitric acid and hydrogen peroxide and analyzed by atomic spectrometry. For low concentrations, ICP is used. Flame AA is used for higher concentrations. Analysis for silver in oily samples may be performed by using ICP after dilution of the sample with methyl isobutyl ketone (MIBK) or xylene, using standards miscible with these solvents.

INTERFERENCES:

If adsorption to container walls or the formation of silver chloride is suspected, ammonium hydroxide and cyanogen iodide should be added. The summarized methods measure the total silver content of the sample and do not identify specific silver compounds.

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. Standards should be analyzed at least daily.

EPA/TECHNICAL STATUS:

These methods are in use in the CERCLA program and are approved for use in Resource Conservation and Recovery Act (RCRA) and Clean Water Act analyses. 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 272.1; EPA-600/4-79-020, U.S. EPA; Cincinnati, 1979.

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

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-124; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3010, 3020, 3040, 3050, 6010, 7740; SW-846, U.S. EPA: Washington, 1982.

COST INFORMATION:

Cost per sample for analysis by flame AA is approximately $20-30 (list); by ICP, it is approximately $20 (list).

SILVER

Water Samples
Laboratory Method

Compound	CAS Number
Potassium Silver Cyanide	506-61-6
Silver	7440-22-4
Silver Cyanide	506-64-9
Silver Nitrate	7761-88-8

METHOD SUMMARY:

Atomic spectrometric analysis for silver may be used to analyze water samples for the above silver compounds. A 100-milliliter sample is digested with nitric acid and hydrogen peroxide, and the digest analyzed by atomic spectrometry. Low silver concentrations are determined by furnace atomic absorption spectrometry (AA) or by chelation-extraction AA. The digested sample is extracted with ammonium pyrrolidinedithiocarbamate in methyl isobutyl ketone and the extract analyzed by flame AA. Inductively coupled plasma spectrometry (ICP) or flame AA is used to analyze higher-concentration samples. These methods do not identify any specific silver-containing compounds, but measure the total silver content of the sample.

INTERFERENCES:

Chlorides and sulfates in the sample may interfere. Nickel can be added to control sulfate interference. For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods.

QUALITY CONTROL:

A reference standard should be analyzed routinely, 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 EPA-approved for Resource Conservation and Recovery Act (RCRA) and Clean Water Act analyses. The detection limit is 10 microgams per liter for direct flame AA, the sensitivity is 60 micrograms per liter, and the optimum concentration range is 0.1 to 4 milligrams per liter (APHA). The chelation-extraction AA method is applicable in the range from 1 to 10 micrograms silver per liter (ASTM). 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, Methods 272.1, 272.2; EPA-600/4-79-020, U.S. EPA: Washington, 1979.

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

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

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3010, 3020, 3040, 3050, 6010, 7740; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-124; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Methods 303A, 303B; APHA: Washington, 1980.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, Methods D3866(A), D3866(B), D3866(C); ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost per sample for analysis by furnace AA is approximately $18-50 (list): by ICP, it is approximately $10 (list).

SODIUM

Air Samples
Laboratory Method 1

Compound	CAS Number	Compound	CAS Number
Sodium Arsenate	7631-89-2	Sodium Methylate	124-41-4
Sodium Arsenite	7784-46-5	Sodium Nitrite	7632-00-0
Sodium Azide	26628-22-8	Sodium Phosphate, Dibasic	7558-79-4
Sodium Bifluoride	1333-83-1		10039-32-4
Sodium Bisulfite	7631-90-5		10028-24-7
Sodium Chromate	7775-11-3		10140-65-5
Sodium Cyanide	143-33-9	Sodium Phosphate, Tribasic	7601-54-9
Sodium Dichromate	10588-01-9		7785-84-4
Sodium Dodecylbenzene	25155-30-0		10101-89-0
Sulfonate			10361-89-4
Sodium Fluoride	7681-49-4		7758-29-4
Sodium Hydrosulfide	16721-80-5		10124-56-8
Sodium Hydroxide	1310-73-2	Sodium Selenite	10102-18-8
Sodium Hypochlorite	7681-52-9		7782-82-3
	10022-70-5		

METHOD SUMMARY:

Analysis of air samples for the above sodium compounds can be performed by atomic absorption (AA) spectrometric analysis for sodium. Samples are collected with cellulose ester membrane filters. The filter and the particulates are digested with nitric acid, the digest is diluted and analyzed by AA. This method does not differentiate between sodium-containing compounds, but measures the total sodium content of the sample.

INTERFERENCES:

Cesium should be added to samples and standards to control ionization interference. The method of standard addition 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. Precision information is furnished.

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 2.1 to 42 micrograms per cubic meter in a 240-liter sample of air. Precision information is furnished.

REFERENCE:

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1; Method 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.

SODIUM

Air Samples
Laboratory Method 2

Compound	CAS Number	Compound	CAS Number
Sodium Arsenate	7631-89-2	Sodium Methylate	124-41-4
Sodium Arsenite	7784-46-5	Sodium Nitrite	7632-00-0
Sodium Azide	26628-22-8	Sodium Phosphate, Dibasic	7558-79-4
Sodium Bifluoride	1333-83-1		10039-32-4
Sodium Bisulfite	7631-90-5		10028-24-7
Sodium Chromate	7775-11-3		10140-65-5
Sodium Cyanide	143-33-9	Sodium Phosphate, Tribasic	7601-54-9
Sodium Dichromate	10588-01-9		7785-84-4
Sodium Dodecylbenzene	25155-30-0		10101-89-0
Sulfonate			10361-89-4
Sodium Fluoride	7681-49-4		7758-29-4
Sodium Hydrosulfide	16721-80-5		10124-56-8
Sodium Hydroxide	1310-73-2	Sodium Selenite	10102-18-8
Sodium Hypochlorite	7681-52-9		7782-82-3
	10022-70-5		

METHOD SUMMARY:

Analysis for sodium ions from the above compounds in air may be performed by inductively coupled plasma atomic emission spectroscopic (ICP) analysis for sodium. A known volume of air is drawn through a cellulose ester membrane filter. The filter and the particulates are ashed with a mixture of nitric and perchloric acids and the digest analyzed by ICP. The method does not identify any sodium-containing compound, but measures the total sodium content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks, spikes, and standards are prepared and 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 5 to 2000 micrograms sodium per cubic meter for a 500-liter sample. Limited 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 351; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

COST INFORMATION:

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

SODIUM

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Sodium	7440-23-5	Sodium Methylate	124-41-4
Sodium Arsenate	7631-89-2	Sodium Nitrite	7632-00-0
Sodium Arsenite	7784-46-5	Sodium Phosphate, Dibasic	7558-79-4
Sodium Azide	26628-22-8		10039-32-4
Sodium Bifluoride	1333-83-1		10028-24-7
Sodium Bisulfite	7631-90-5		10140-65-5
Sodium Chromate	7775-11-3	Sodium Phosphate, Tribasic	7601-54-9
Sodium Cyanide	143-33-9		7785-84-4
Sodium Dichromate	10588-01-9		10101-89-0
Sodium Dodecylbenzene Sulfonate	25155-30-0		10361-89-4
			7758-29-4
Sodium Fluoride	7681-49-4		10124-56-8
Sodium Hydrosulfide	16721-80-5	Sodium Selenite	10102-18-8
Sodium Hydroxide	1310-73-2		7782-82-3
Sodium Hypochlorite	7681-52-9		
	10022-70-5		

METHOD SUMMARY:

Atomic emission spectrometric analysis for sodium ions may be used to analyze soil/sediment samples for the above sodium compounds. A 100-milligram sample is digested with nitric acid, perchloric acid, hydrofluoric acid, and boric acid, and analyzed. Samples should be stored in polyurethane bags and frozen immediately after collection. This method does not differentiate between sodium-containing compounds, but measures the total sodium content of the sample.

INTERFERENCES:

Standard and sample compositions should be closely matched. Solvent extraction techniques may be necessary. Cesium should be added to samples and standards to overcome ionization interference.

QUALITY CONTROL:

Quality control procedures are not provided.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The detection limit of the method is approximately 100 milligrams of sodium per kilogram. Precision and accuracy information is furnished.

REFERENCES:

Environment Canada, Analytical Methods Manual, NAQUADAT No. 19050; Environment Canada, Inland Waters Directorate, Water Quality Branch: Ottawa, Canada, 1975.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption;" HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

Alberta Environmental Centre, Methods Manual for the Chemical Analysis of Water and Wastes, "Sodium, Dissolved;" Publication No. AECV81-M1, Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $20-30 (list).

SODIUM

Water Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Sodium	7440-23-5	Sodium Methylate	124-41-4
Sodium Arsenate	7631-89-2	Sodium Nitrite	7632-00-0
Sodium Arsenite	7784-46-5	Sodium Phosphate, Dibasic	7558-79-4
Sodium Azide	26628-22-8		10039-32-4
Sodium Bifluoride	1333-83-1		10028-24-7
Sodium Bisulfite	7631-90-5		10140-65-5
Sodium Chromate	7775-11-3	Sodium Phosphate, Tribasic	7601-54-9
Sodium Cyanide	143-33-9		7785-84-4
Sodium Dichromate	10588-01-9		10101-89-0
Sodium Dodecylbenzene Sulfonate	25155-30-0		10361-89-4
			7758-29-4
Sodium Fluoride	7681-49-4		10124-56-8
Sodium Hydrosulfide	16721-80-5	Sodium Selenite	10102-18-8
Sodium Hydroxide	1310-73-2		7782-82-3
Sodium Hypochlorite	7681-52-9		
	10022-70-5		

METHOD SUMMARY:

Analysis of water samples for sodium ions from the above compounds can be performed by flame photometric or atomic absorption (AA) spectrometric analysis for sodium. Samples should be stored in polyethylene bottles and preserved by acidification with nitric acid. This method does not identify specific sodium compounds, but measures the total amount of sodium present in a sample.

INTERFERENCES:

When using flame AA, it may be necessary to add potassium to both standards and samples to suppress ionization. When using flame photometry, samples containing particulate matter should be filtered before analysis. Relatively large amounts of potassium, calcium, chloride, sulfate, and bicarbonate can interfere. Standards and samples should contain the same amounts of interfering substances.

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 each time a sample or set of samples is analyzed.

EPA/TECHNICAL STATUS:

These methods are approved for the National Pollutant Discharge Elimination System (NPDES). Precision and accuracy information is furnished. For flame photometry, the minimum detectable concentration is 0.1 milligrams sodium

per liter (APHA), and the optimum concentration range is 1 to 100 milligrams per liter (ASTM). For AA analysis the procedure is applicable over the range of 0.2 to 3 milligrams sodium per liter (ASTM).

REFERENCES:

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 273.1; EPA-600/4-79-020, U.S. EPA: Cincinnati, March 1979.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption;" HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III., Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 14th ed.; APHA: Washington, 1975.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, Method D4191-82; ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost per sample for analysis by flame AA is approximately $10-20 (list).

STRONTIUM

Air Samples
Laboratory Method

Compounds	CAS Number
Strontium Chromate	7789-06-2
Strontium Sulfide	1314-96-1

METHOD SUMMARY:

Atomic absorption spectrometric (AA) analysis for strontium can be used for the analysis of air for the above strontium compounds. Samples are collected by passage of a known volume of air through a cellulose ester membrane filter. The filter and the particulates are digested with nitric acid, and the resulting solution is diluted and analyzed by AA. This method does not identify any specific strontium-containing compound, but measures the total strontium content of the sample.

INTERFERENCES:

Cesium should be added to samples and standards to control ionization interference, and lanthanum should be added as a releasing agent. The method of standard additions and/or background correction 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 for Occupational Safety and Health (NIOSH) for use over the range of 21 to 210 micrograms strontium per cubic meter for a 240-liter air sample. Precision information is furnished.

REFERENCES:

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.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption;" HASL-300 Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III., Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

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

STRONTIUM

Soil/Sediment Samples
Laboratory Method

Compounds	CAS Number
Strontium Chromate	7789-06-2
Strontium Sulfide	1314-96-1

METHOD SUMMARY:

Analysis of soil/sediment samples for the above strontium compounds can be performed by atomic absorption (AA) spectrometric analysis for strontium. A 100-milligram sample is digested with nitric acid, perchloric acid, hydrogen fluoride, and boric acid, and analyzed. Samples should be stored in polyurethane bags and frozen immediately after collection. This method does not identify any specific strontium-containing compounds, but measures the total strontium content of the sample.

INTERFERENCES:

Standard and sample compositions should be closely matched. Sodium should be added to both to overcome ionization interference. Solvent extraction techniques may be necessary to improve the detection limit.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information is furnished. The detection limit of this method is approximately 20 milligrams per kilogram.

REFERENCES:

Environment Canada, Analytical Methods Manual, NAQUADAT No. 38050; Environment Canada, Inland Waters Directorate, Water Quality Branch: Ottawa, Canada, 1975.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $20-30 (list).

STRONTIUM

Water Samples
Laboratory Method

Compounds	CAS Number
Strontium Chromate	7789-06-2
Strontium Sulfide	1314-96-1

METHOD SUMMARY:

Analysis of water samples for the above strontium compounds can be performed by atomic absorption (AA) spectrometric analysis for strontium. The sample is filtered through a 0.45-micron membrane filter, a lanthanum chloride/potassium chloride solution is added, and the resulting solution is analyzed for strontium by AA. This method measures the total dissolved strontium content of the sample, and does not identify any specific strontium-containing compounds.

QUALITY CONTROL:

No quality control procedures are given.

INTERFERENCES:

Chemical interferences by silicon, aluminum, and phosphate are controlled by the addition of lanthanum chloride. Ionization suppression can be controlled by the addition of potassium chloride.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The method is applicable over the range of 0.1 to 5 milligrams strontium per liter. For brackish water, sea water, and brines, the method is applicable to the range 5 to 2000 milligrams per liter. Precision and accuracy information is furnished.

REFERENCES:

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, Method D3920-80; ASTM: Philadelphia, 1983.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02 D3352074; ASTM: Philadelphia, 1983.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption;" HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $10-20 (list).

SULFATES (PARTICULATE)

Air Samples
Laboratory Method

Compound	CAS Number
Aluminum Sulfate	10043-01-3
Cupric Sulfate	7758-98-7
Cupric Sulfate, Ammoniated	10380-29-7
Ferric Sulfate	10028-22-5
Ferrous Ammonium Sulfate	10045-89-3
Ferrous Sulfate	7720-78-7
	7782-63-0
Mercuric Sulfate	7783-35-9
Nickel Sulfate	7786-81-4
Thallium(I) Sulfate	7446-18-6
Vanadyl Sulfate	27774-13-6
Zinc Sulfate	7733-02-0

METHOD SUMMARY:

Analysis of air samples for the above sulfates can be performed by colorimetric analysis for sulfate ion. A known volume of air is passed through a glass-fiber filter, using a high-volume sampler. Sulfate is extracted with hot water. The resulting solution is allowed to react with an acidic color reagent containing methylthymol blue and barium chloride, producing barium sulfate. The pH of the mixture is adjusted to 12 to 13, and the remaining barium is complexed with the methylthymol blue. The uncomplexed methylthymol blue, which is equimolar to the sulfate removed as barium sulfate, is determined by measuring its absorbance at 460 nm. This method is intended to be used with an automated system. This method does not identify the specific sulfate-containing compounds present, but measures the total sulfate content of the sample.

INTERFERENCES:

Interfering cations are removed by passing the sample through an ion-exchange column.

QUALITY CONTROL:

A baseline should be determined with all reagents, feeding distilled water through the sample line.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Single-laboratory precision and accuracy information is furnished.

REFERENCE:

Alberta Environmental Centre, Methods Manual for Chemical Analysis of Atmospheric Pollutants, Method No. 25556; Alberta Environmental Centre: Alberta, Canada, 1981.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $45-60 (list).

SULFATES

Water Samples
Laboratory Method 1

Compound	CAS Number
Aluminum Sulfate	10043-01-3
Cupric Sulfate	7758-98-7
Cupric Sulfate, Ammoniated	10380-29-7
Ferric Sulfate	10028-22-5
Ferrous Ammonium Sulfate	10045-89-3
Ferrous Sulfate	7720-78-7
	7782-63-0
Mercuric Sulfate	7783-35-9
Nickel Sulfate	7786-81-4
Thallium(I) Sulfate	7446-18-6
Vanadyl Sulfate	27774-13-6
Zinc Sulfate	7733-02-0

METHOD SUMMARY:

Analysis of water samples for the above sulfates can be performed by determination of the sulfate ion concentration by gravimetric, turbidimetric, or volumetric procedures. The gravimetric procedure is performed by addition of barium chloride to the sample and gravimetric measurement of the precipitated barium sulfate. The turbidimetric procedure involves addition of barium chloride and glycerin to form a barium sulfate suspension and turbidimetric analysis of it. The volumetric procedure involves titration of sulfate ion in an acidic alcoholic solution with barium chloride, using thorin as the indicator. For brackish water, sea water, and brines, the turbidimetric method, with adjustment of the ionic strength to 2 molal, is used.

INTERFERENCES:

Both cations and anions can cause errors in barium sulfate precipitation; potassium, iron, aluminum, phosphate, fluoride, and nitrate are common interferences. Most metal ions interfere seriously with the volumetric determination because of metal-thorin complex formation. Cation interferences may be removed by use of cation-exchange resin. The effects of sulfite interferences can be eliminated in calculations. Sulfides are precipitated as zinc sulfide. Chromate and dichromate ions are removed by treatment with hydrogen peroxide to form trivalent chromium, followed by removal with cation-exchange resin.

QUALITY CONTROL:

No quality control procedures are provided.

EPA/TECHNICAL STATUS:

These methods are not EPA-approved. The gravimetric method is applicable to samples containing 20 to 100 milligrams sulfate ion per liter. The turbidimetric and volumetric methods are useful in the range of 10 to 100 and 5 to 1000 milligrams sulfate ion per liter, respectively. Precision and accuracy information is furnished.

REFERENCES:

American Society for Testing and Material, 1983 Annual Book of ASTM Standards, Vol 11.01, Methods D516-82(A), D516-92(B), D516-82(C); ASTM: Philadelphia, 1983.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, D4130-82; ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost per sample for analysis by the volumetric method is approximately $20-30 (list).

SULFATES

Water Samples
Laboratory Method 2

Compound	CAS Number
Aluminum Sulfate	10043-01-3
Cupric Sulfate	7758-98-7
Cupric Sulfate, Ammoniated	10380-29-7
Ferric Sulfate	10028-22-5
Ferrous Ammonium Sulfate	10045-89-3
Ferrous Sulfate	7720-78-7
	7782-63-0
Mercuric Sulfate	7783-35-9
Nickel Sulfate	7786-81-4
Thallium(I) Sulfate	7446-18-6
Vanadyl Sulfate	27774-13-6
Zinc Sulfate	7733-02-0

METHOD SUMMARY:

Analysis of water samples for the above sulfates can be performed by precipitation and colorimetry. A sample is treated with barium chloride to precipitate barium sulfate, and methylthymol blue indicator is added to form a blue complex with any unprecipitated barium. The intensity of the color of the barium-methylthymol blue complex is measured and compared with that of standards. This method does not identify any specific sulfate salts, but measures the total dissolved sulfate concentration of the sample.

INTERFERENCES:

Cations, such as calcium, aluminum, and iron, that complex methylthymol blue, are removed by passage of the sample through an ion-exchange column.

QUALITY CONTROL:

Duplicate and fortified samples should be processed as part of a quality assurance program.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The method is suitable for use over the range 5 to 300 milligrams sulfate per liter. Precision and accuracy information is furnished.

REFERENCE:

Alberta Environmental Centre, Methods Manual for the Chemical Analysis of Water and Wastes, "Sulphate, Dissolved;" Publication No. AECV81-M1, Alberta Environmental Centre, Vegreville, Alberta, Canada, 1981.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $30-35 (list).

SULFIDES

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Ammonium Sulfide	12135-76-1
Arsenic Disulfide	1303-32-8
Arsenic Trisulfide	1303-33-9
Hydrogen Sulfide	7783-06-4
Lead Sulfide	1314-87-0
Phosphorus Pentasulfide	1314-80-3
Selenium Disulfide	7488-56-4
Sodium Hydrosulfide	16721-80-5
Strontium Sulfide	1314-96-1

METHOD SUMMARY:

The determination of the above sulfides in a 1-gram soil/sediment sample can be performed by colorimetric analysis for sulfide ions. The hydrogen sulfide is distilled from the acidified solution into a trap containing cadmium nitrate. When zinc acetate, dimethyl-p-diphenylenediamine, and ferric chloride are added to the distillate, methylene blue is formed. The intensity of the resulting color is measured spectrophotometrically at 670 nm. This method measures the total sulfide concentration in the sample and does not identify specific sulfide compounds.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

One distilled water blank, one spiked sample, and one duplicate should be analyzed with each set of ten samples.

EPA/TECHNICAL STATUS:

This method is in use in the CERCLA program. No precision or 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, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, Method 9030; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Methods, "Methods for the Determination of Sulfide," pp. IV-136 to IV-155; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $38-60 (list).

SULFIDES

Water Samples
Laboratory Method

Compound	CAS Number
Ammonium Sulfide	12135-76-1
Arsenic Trisulfide	1303-33-9
Hydrogen Sulfide	7783-06-4
Phosphorus Pentasulfide	1314-80-3
Sodium Hydrosulfide	16721-80-5
Strontium Sulfide	1314-96-1

METHOD SUMMARY:

Analysis of water samples for sulfide ions from the above compounds can be performed by titrimetric or colorimetric determination of sulfide ions. In the titrimetric analysis, a 200-milliliter sample is analyzed by the addition of excess iodine and back-titration with sodium thiosulfate or phenylarsine oxide, using starch as an indicator. For colorimetric analysis, a 7.5-milliliter sample is combined with dimethyl-p-phenylenediamine and ferric chloride and analyzed colorimetrically. Both methods measure the total sulfide content of the sample and do not identify specific sulfide-containing compounds.

INTERFERENCES:

Samples must be taken with a minimum of aeration, and all water used must be free of dissolved oxygen. If the titrimetric analysis is not to be started immediately, the samples must be preserved with zinc acetate. Samples containing volatile iodine-consuming substances and reduced sulfur compounds may require background correction for titrimetric analysis; acid-insoluble sulfides and sulfide concentrations below 1 milligram/liter cannot be determined by this method. For colorimetric analysis, all water used should contain no dissolved oxygen; acid-insoluble sulfides cannot be determined by this method. Color and turbidity in the samples may interfere with colorimetry.

QUALITY CONTROL:

A blank should be processed with each set of samples, and a check standard should be analyzed with each 10 samples. Ten percent of the samples should be processed in duplicate, and fortified samples or standard reference materials should be processed periodically.

EPA/TECHNICAL STATUS:

Both methods are in use in the CERCLA program and are approved for use in Clean Water Act analyses. The titrimetric method is EPA-approved for use under the Resource Conservation and Recovery Act (RCRA). The colorimetric method is suitable over the range 0.02 to 20 milligrams sulfide per liter (Alberta Environment). 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, Methods 376.1, 376.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, Method 9030; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Sulfide," pp. IV-136 to IV-155; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Alberta Environmental Centre, Methods Manual for the Chemical Analysis of Water and Wastes, "Sulphide, Total;" Publication No. AECV81-M1, Alberta Environmental Centre, Vegreville, Alberta, Canada, 1981.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Methods 427C, 427D; APHA: Washington, 1980.

COST INFORMATION:

Cost per sample for analysis by these methods is approximately $20-30 (list).

SULFITES

Water Samples
Laboratory Method

Compound	CAS Number
Ammonium Bisulfite	10192-30-0
Ammonium Sulfite	10196-04-0
Sodium Bisulfite	7361-90-5
Zinc Hydrosulfite	7779-86-4

METHOD SUMMARY:

Analysis of water samples for the above sulfite compounds may be performed by titration of sulfite ions. A 50-milliliter sample is mixed with 3 to 4 drops of phenolphthalein indicator and approximately 1 gram of starch indicator to discharge the red color produced by phenolphthalein. The resulting solution is titrated with a potassium iodide-iodate reagent. This method determines the amount of sulfite ion present, and does not differentiate between various sulfite-containing compounds.

INTERFERENCES:

Contact of the sample with air must be minimized, and the temperature must be kept below 50°C. Other oxidizable substances, nitrite, copper, and heavy metals may interfere.

QUALITY CONTROL:

A reagent blank must be analyzed periodically.

EPA/TECHNICAL STATUS:

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

REFERENCES:

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 377.1: EPA-600/4-79-020, U.S. EPA: Washington, 1979.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 428; APHA: Washington, 1980.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, D1339-78; ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $10-20 (list).

SULFUR COMPOUNDS (PARTICULATE)

Air Samples
Laboratory Method

Compound	CAS Number
Ammonium Sulfamate	7773-06-0
Ammonium Sulfide	12135-76-1
Ammonium Sulfite	10196-04-0
Ammonium Thiosulfate	7783-18-8
Arsenic Disulfide	1303-32-8
Arsenic Trisulfide	1303-33-9
Chromic Sulfate	10101-53-8
Cupric Sulfate	7758-98-7
Cupric Sulfate, Ammoniated	10380-29-7
Ferric Sulfate	10028-22-5
Ferrous Ammonium Sulfate	10045-89-3
Ferrous Sulfate	7720-78-7
	7782-63-0
Phosphorus Pentasulfide	1314-80-3
Selenium Disulfide	7488-56-4
Sodium Hydrosulfide	16721-80-5
Strontium Sulfide	1314-96-1
Thallium Sulfate	7446-18-6
Vanadyl Sulfate	27774-13-6
Zinc Hydrosulfite	7779-86-4
Zinc Sulfate	7733-02-0
Zirconium Sulfate	14644-61-2

METHOD SUMMARY:

Analysis for the above particulate sulfur compounds in air can be performed by filtration of air and analysis of the filter media for sulfur by X-ray fluorescence spectrometry. The technique is non-destructive, so further analyses can be carried out on the material retained by the filter if desired. This method measures the total amount of sulfur in the sample and does not identify any specific sulfur-containing compound.

INTERFERENCES:

Some sulfide or hydrosulfide compounds can react with water during sampling under humid conditions to form hydrogen sulfide, which may be lost from the filter medium. Sulfates, sulfamates, sulfites, and thiosulfates will not be lost under these conditions.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

The method is not EPA-approved. Precision information is furnished. This method can detect concentrations of sulfur greater than 0.01 to 50 micrograms per cubic meter of air.

REFERENCES:

Rhodes, J. R.; Stout, J. A.; 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.

THALLIUM

Air Samples
Laboratory Method 1

Compound	CAS Number
Thallic Oxide	1314-32-5
Thallium	7440-28-0
Thallium(I) Acetate	563-68-8
Thallium(I) Carbonate	6533-73-9
Thallium(I) Chloride	7791-12-0
Thallium(I) Nitrate	10102-45-1
Thallium(I) Selenide	12039-52-0
Thallium(I) Sulfate	7446-18-6

METHOD SUMMARY:

Analysis for the above soluble thallium compounds in air samples can be performed by high-volume filtration of air followed by atomic absorption (AA) analysis for thallium. A known volume of air is passed through a cellulose ester membrane filter, the filter is wet-ashed, and the resulting solution is analyzed by AA for thallium. This method measures the total thallium content of the air sampled and does not identify the specific thallium compounds present.

INTERFERENCES:

The presence of insoluble thallium compounds will interfere.

QUALITY CONTROL:

The analyte recovery should be determined in duplicate and, if less than 95 percent, results should be corrected appropriately. Duplicate determinations should agree within 5 percent.

EPA/TECHNICAL STATUS:

Method S306 is approved by the National Institute of Occupational Safety and Health (NIOSH), and has been validated over the range of 0.034 to 0.15 milligrams thallium per cubic meter for a 540-liter sample. Precision and accuracy information is furnished. NIOSH method P&CAM 173, classified as operational, is for use over the range of 0.2 to 0.84 milligrams thallium per cubic meter in a 240-liter sample. Precision information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set U, Method S306; PB-262 405 (NTIS), U.S. DHEW: Cincinnati, December 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.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

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

THALLIUM

Air Samples
Laboratory Method 2

Compound	CAS Number
Thallic Oxide	1314-32-5
Thallium	7440-28-0
Thallium(I) Acetate	563-68-8
Thallium(I) Carbonate	6533-73-9
Thallium(I) Chloride	7791-12-0
Thallium(I) Nitrate	10102-45-1
Thallium(I) Selenide	12039-52-0
Thallium(I) Sulfate	7446-18-6

METHOD SUMMARY:

Analysis for the above thallium-containing compounds in air may be performed by inductively coupled plasma atomic emission spectroscopic (ICP) analysis for thallium. A known volume of air is drawn through a cellulose ester membrane filter. The filter is ashed with a mixture of nitric and perchloric acids followed by analysis of the digest by ICP. The method does not identify any specific thallium-containing compound, but measures the total thallium content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks, spikes, and standards are prepared and 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 5 to 2000 micrograms thallium per cubic meter for a 500-liter sample. Limited 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 351; Publication No. 82-100, U.S. DHHS: Cinncinnati, August 1981.

COST INFORMATION:

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

THALLIUM

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Thallic Oxide	1314-32-5
Thallium	7440-28-0
Thallium(I) Acetate	563-68-8
Thallium(I) Carbonate	6533-73-9
Thallium(I) Chloride	7791-12-0
Thallium(I) Nitrate	10102-45-1
Thallium(I) Selenide	12039-52-0
Thallium(I) Sulfate	7446-18-6

METHOD SUMMARY:

Analysis of soil/sediment samples for the above thallium compounds can be performed by atomic spectrometric analysis for thallium. A 2-gram sample is digested with nitric acid and hydrogen peroxide and analyzed by atomic spectrometry. For low concentrations, furnace atomic absorption spectrometry (AA) is used, while flame AA is used for higher-concentration samples. These methods do not identify any specific thallium-containing compounds, but measure the total thallium content of the sample.

INTERFERENCES:

For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods.

QUALITY CONTROL:

A reference standard should be analyzed routinely, 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 analyses for the Clean Water Act. 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, Methods 279.1, 279.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 7480, 7481; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

Cost per sample for analysis by furnace AA is approximately $28-60 (list); by flame AA, it is approximately $20-30 (list).

THALLIUM

Water Samples
Laboratory Method

Compound	CAS Number
Thallic Oxide	1314-32-5
Thallium	7440-28-0
Thallium (I) Acetate	563-68-8
Thallium (I) Carbonate	6533-73-9
Thallium (I) Chloride	7791-12-0
Thallium (I) Nitrate	10102-45-1
Thallium (I) Selenide	12039-52-0
Thallium (I) Sulfate	7446-18-6

METHOD SUMMARY:

Atomic spectrometric analysis for thallium can be used to measure the above thallium compounds in water samples. A 100-milliliter sample is digested with nitric acid and hydrogen peroxide and analyzed by atomic spectrometry. For low concentrations, furnace atomic absorption spectrometry (AA) is used, while flame AA is used for higher-concentration samples. These methods do not identify any specific thallium-containing compounds, but measure the total thallium content of the sample.

INTERFERENCES:

For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods.

QUALITY CONTROL:

A reference standard should be analyzed routinely, 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 use under the Clean Water Act. 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, Methods 279.1, 279.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

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

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, Methods 7840, 7841; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption; HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

Fassel, V. A.; Peterson, C. A.; Abercrombie, F. N.; Kniseley, R. N. "Simultaneous Determination of Wear Metals in Lubricatng Oils by Inductively-Coupled Plasma Atomic Emission Spectrometry," Anal. Chem., 48(3), 1976, pp. 516-519.

COST INFORMATION:

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

THIOCYANATES

Water Samples
Laboratory Method

Compound	CAS Number
Ammonium Thiocyanate	1762-95-4
Lead Thiocyanate	592-87-0
Mercuric Thiocyanate	592-85-8

METHOD SUMMARY:

Analysis of water samples for the above thiocyanate-containing compounds can be performed by colorimetric analysis for thiocyanate ions. The sample is reacted with ferric nitrate at an acidic pH, and the intensity of the color of the resulting blood-red iron-thiocyanate complex is measured at 460 nanometers. This method does not identify any particular thiocyanate-containing compound, but measures the total thiocyanate content of the sample.

INTERFERENCES:

Various interfering organic compounds can be removed by adsorption on macroreticular resin prior to analysis. Hexavalent chromium interference is removed by adjustment of the pH to 2 and addition of ferrous sulfate. Raising the pH to 9 and filtration removes iron(III) and chromium(III). Reducing agents are destroyed by addition of hydrogen peroxide. If the sample will be used for cyanide determination, cadmium or lead salt should be added to remove sulfide before the pH is raised.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The method is applicable over the range 0.1 to 2 milligrams thiocyanate ion per liter, and higher concentrations can be measured by suitable sample dilution. Precision and accuracy information is furnished.

REFERENCE:

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, D4193-82; ASTM: Philadelphia, 1983.

COST INFORMATION:

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

URANIUM

Water Samples
Laboratory Method 1

Compound	CAS Number
Uranyl Acetate	541-09-3
Uranyl Nitrate	10102-06-4
	36478-76-9

METHOD SUMMARY:

Analysis of water samples for the above uranium compounds can be performed by X-ray fluorescence spectrometry. The water sample is filtered, acidified, and treated with ultraviolet radiation to decrease subsequent filtering time and improve recovery of the analyte. The uranium is coprecipitated with an iron dibenzyldithiocarbamate carrier complex, and the precipitate is collected by vacuum filtration. The precipitate on the filter is analyzed by wavelength-dispersive X-ray fluorescence spectrometry. This method does not identify the specific uranium-containing compounds present, but measures the total uranium content of the sample.

INTERFERENCES:

The following ions cause no significant interferences at levels below 0.1 grams per liter: chloride, magnesium, calcium, sodium, potassium, and sulfate.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Limited precision and accuracy information is furnished. This method is suitable for concentrations of uranium over 0.4 micrograms per liter.

REFERENCE:

Caravajal, G.; Mahan, K.; Leyden, D. "The Determination of Uranium in Natural Waters at PPB Levels by Thin-Film X-ray Fluorescence Spectrometry after Coprecipitation with an Iron Dibenzyldithiocarbamate Carrier Complex" Anal. Chim. Acta., 135, 1982, pp. 205-214.

COST INFORMATION:

No cost information has been obtained.

URANIUM

Water Samples
Laboratory Method 2

Compound	CAS Number
Uranyl Acetate	541-09-3
Uranyl Nitrate	10102-06-4
	36478-76-9

METHOD SUMMARY:

Analysis of water samples for the above uranium compounds can be accomplished by fluorescence spectrometric analysis for uranium. A nitrogen laser at 337 nm is used as an excitation source. A chelating agent, Fluran®, is used to increase fluorescence yield and the solution is buffered to the proper pH. The method is applicable to continuous monitoring.

INTERFERENCES:

Fluorescence from organic compounds is eliminated by use of time- or wavelength-discrimination techniques. High levels of carbonate and phosphate ions interfere, but chloride and sulfate do not.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved for any regulatory program. No precision and accuracy information is furnished.

REFERENCE:

Campen, W.; Bächmann, K. "Laser-Induced Fluorescence for the Direct Determination of Small Concentrations of Uranium in Water" Mikrochimica Acta Wein, II, 1979, pp. 159-170.

COST INFORMATION:

No cost information has been obtained.

VANADIUM

Air Samples
Laboratory Method 1

Compound	CAS Number
Ammonium Vanadate	7803-55-6
Vanadium Pentoxide	1314-62-1
Vanadyl Sulfate	27774-13-6

METHOD SUMMARY:

Analysis for the above particulate vanadium compounds in air samples may be performed by flame atomic absorption (AA) spectrometric determination of vanadium. 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 analyzed by AA, applying either direct aspiration or cupferron/butyl acetate extraction techniques. This method does not identify any specific vanadium compounds, but measures the total vanadium present in the sample.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

A blank should be processed with every 10 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 420 to 6300 micrograms per cubic meter, using a 240-liter air sample. Precision information is furnished. APHA has determined the detection limit to be 0.2 micrograms per cubic meter of air, using a 2000-cubic-meter sample.

REFERENCES:

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.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption; HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

Alberta Environment, Methods Manual for Chemical Analysis of Atmospheric Pollutants, Methods 23535, 23545; Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

American Public Health Service, Methods of Air Sampling and Analysis, 2nd ed., Method 321; APHA: Washington, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $20 (list), or $55-65 if the extraction technique is used, plus sampling costs.

VANADIUM

Air Samples
Laboratory Method 2

Compound	CAS Number
Ammonium Vanadate	7803-55-6
Vanadium Pentoxide	1314-62-1
Vanadyl Sulfate	27774-13-6

METHOD SUMMARY:

Analysis for the above vanadium-containing compounds in air may be performed by inductively coupled plasma atomic emission spectrometric (ICP) analysis for vanadium. A known volume of air is drawn through a cellulose ester membrane filter. The filter is ashed with a mixture of nitric and perchloric acids and the digest analyzed by ICP. The method does not identify any specific vanadium-containing compound, but measures the total vanadium content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks, spikes, and standards are prepared and 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 5 to 2000 micrograms vanadium per cubic meter, using a 500-liter sample. Limited 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 351; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

COST INFORMATION:

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

VANADIUM

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Ammonium Vanadate	7803-55-6
Vanadium Pentoxide	1314-62-1
Vanadyl Sulfate	27774-13-6

METHOD SUMMARY:

Analysis of soil/sediment samples for the above vanadium compounds can be performed by atomic spectrometric analysis for vanadium. A 2-gram sample is digested with nitric acid and hydrogen peroxide and the digest analyzed by inductively coupled plasma (ICP) or flame atomic absorption (AA) spectrometry. Analysis for vanadium in oily samples may be performed using ICP after sample dilution with methyl isobutyl ketone (MIBK) or xylene, using standards miscible with these solvents. These methods measure total vanadium present in the sample and do not differentiate between compounds of vanadium.

INTERFERENCES:

The addition of aluminum (1000 milligrams per liter) to both standards and samples will control interferences caused by high concentrations of aluminum and titanium in the sample. For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods.

QUALITY CONTROL:

A reference standard should be analyzed routinely, 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 use under the Clean Water Act. 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 286.1; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

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

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-125; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, Methods 7840, 7841; SW-846, U.S. EPA: Washington, 1982.

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, pp. 516-519.

COST INFORMATION:

Cost per sample for analysis by flame AA is approximately $20-30 (list); by ICP, it is approximately $20 (list).

VANADIUM

Water Samples
Laboratory Method 1

Compound	CAS Number
Ammonium Vanadate	7803-55-6
Vanadium Pentoxide	1314-62-1
Vanadyl Sulfate	27774-13-6

METHOD SUMMARY:

Analysis of water samples for the above vanadium compounds can be performed by atomic spectrometric analysis for vanadium. A 100-milliliter sample is digested with nitric acid and hydrogen peroxide and the digest analyzed by inductively coupled plasma (ICP) atomic spectrometry or by flame or furnace atomic absorption spectrometry (AA). These methods do not differentiate between various vanadium-containing compounds, but measure the total vanadium content of the sample.

INTERFERENCES:

The addition of aluminum (1000 milligrams per liter) to both standards and samples will control interferences caused by high concentrations of aluminum and titanium in the sample. For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods.

QUALITY CONTROL:

A reference standard should be analyzed routinely, 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 EPA-approved for analyses under the Clean Water Act and the Resource Conservation and Recovery Act (RCRA). No precision or 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, Methods 286.1, 286.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

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

U.S. Environmental Protection Agency, Technical Additions to Methods for Chemical Analysis of Water and Wastes, Method 200.7; EPA-600/4-82-055, U.S. EPA: Cincinnati, December 1982.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, Methods 7840, 7841; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-125; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption;" HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 303C; APHA: Washington, 1980.

COST INFORMATION:

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

VANADIUM

Water Samples
Laboratory Method 2

Compound	CAS Number
Ammonium Vanadate	7803-55-6
Vanadium Pentoxide	1314-62-1
Vanadyl Sulfate	27774-13-6

METHOD SUMMARY:

Analysis of water samples for the above vanadium-containing compounds can be performed by reaction-kinetic analysis for vanadium. Measurement of the rate of the oxidation of gallic acid by the persulfate ion in acid solution, which is proportional to the vanadium concentration, is performed by monitoring the absorbance of the solution at 415 nanometers on a spectrophotometer. The concentration of vanadium is quantified by use of a calibration curve. This method does not identify any specific vanadium-containing compound, but measures the total vanadium content of the sample.

INTERFERENCES:

Copper, iron, and, to a lesser degree, chromium, cobalt, molybdenum, nickel, silver, and uranium interfere. Chloride and traces of bromide and iodide interfere seriously, and these anions can be complexed with mercuric ion.

QUALITY CONTROL:

No quality control procedures are provided.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The method is applicable in the range from 1 to 100 micrograms vanadium per liter. Precision and accuracy information is furnished.

REFERENCES:

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, D3373-80; ASTM: Philadelphia, 1983.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 327B; APHA: Washington, 1980.

COST INFORMATION:

No cost information has been obtained.

ZINC

Air Samples
Laboratory Method 1

Compound	CAS Number
Zinc	7440-66-6
Zinc Acetate	557-34-6
Zinc Ammonium Chloride	14639-97-5
	14639-98-6
	52628-25-8
Zinc Borate	1332-07-6
Zinc Bromide	7699-45-8
Zinc Carbonate	3486-35-9
Zinc Chloride	7646-85-7
Zinc Cyanide	557-21-1
Zinc Fluoride	7783-49-5
Zinc Formate	557-41-5
Zinc Hydrosulfite	7779-86-4
Zinc Nitrate	7779-88-6
Zinc Phenolsulfonate	127-82-2
Zinc Phosphide	1314-84-7
Zinc Silicofluoride	16871-71-9
Zinc Sulfate	7733-02-0

METHOD SUMMARY:

Analysis of air samples for the above zinc compounds can be performed by filtration of air and analysis of the filter deposit by X-ray fluorescence spectrometry. The method is non-destructive, so that further analyses may be performed on samples. The sample-collection time may be up to 8 hours, but analysis time is approximately 1 minute. The method measures total zinc present in the sample and does not identify any specific zinc compounds.

INTERFERENCES:

Polyvinyl chloride (PVC) filters are unacceptable for sampling, and glass-fiber filters are acceptable for use only if they are of the highest purity. Cellulose membrane, cellulose fiber, polycarbonate membrane, and fluorocarbon membrane filters are acceptable.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision information is given. The method should be applicable to concentrations above 0.001 micrograms of zinc per cubic meter, depending on sample size, when at least 40 micrograms of zinc are collected.

REFERENCES:

Rhodes, J. R.; Stout, J. A.; 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 $30 (list), $20 (bid), plus sampling costs.

ZINC

Air Samples
Laboratory Method 2

Compound	CAS Number
Zinc	7440-66-6
Zinc Acetate	557-34-6
Zinc Ammonium Chloride	14639-97-5
	14639-98-6
	52628-25-8
Zinc Borate	1332-07-6
Zinc Bromide	7699-45-8
Zinc Carbonate	3486-35-9
Zinc Chloride	7646-85-7
Zinc Cyanide	557-21-1
Zinc Fluoride	7783-49-5
Zinc Formate	557-41-5
Zinc Hydrosulfite	7779-86-4
Zinc Nitrate	7779-88-6
Zinc Phenolsulfonate	127-82-2
Zinc Phosphide	1314-84-7
Zinc Silicofluoride	16871-71-9
Zinc Sulfate	7733-02-0

METHOD SUMMARY:

Analysis for the above zinc-containing compounds in air may be performed by inductively coupled plasma (ICP) or atomic absorption (AA) spectroscopic analysis for zinc. A known volume of air is drawn through a cellulose ester membrane filter. The filter is ashed with a mixture of nitric and perchloric acids and the digest analyzed by ICP or AA spectrometric methods. The method does not identify any specific zinc-containing compound, but measures the total zinc content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks, spikes, and standards are prepared and processed with the samples.

EPA/TECHNICAL STATUS:

The AA method is not EPA-approved. The ICP method has been proposed by the National Institute for Occupational Safety and Health (NIOSH) for use over the range of 5 to 2000 micrograms zinc per cubic meter for a 500-liter sample. Limited precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health and Human Services, NIOSH Manual of Analytical Methods, Vol. 7, Method P&CAM 351: Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

American Public Health Association, Methods of Air Sampling and Analysis, 2nd ed., Method 322; APHA: Washington, 1977.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

Cost per sample for analysis by flame AA is approximately $10-20 (list), plus sampling costs; by ICP, it is approximately $10 (list), plus sampling costs.

ZINC

Air Samples
Laboratory Method 3

Compound	CAS Number
Zinc	7440-66-6
Zinc Acetate	557-34-6
Zinc Ammonium Chloride	14639-97-5
	14639-98-6
	52628-25-8
Zinc Borate	1332-07-6
Zinc Bromide	7699-45-8
Zinc Carbonate	3486-35-9
Zinc Chloride	7646-85-7
Zinc Cyanide	557-21-1
Zinc Fluoride	7783-49-5
Zinc Formate	557-41-5
Zinc Hydrosulfite	7779-86-4
Zinc Nitrate	7779-88-6
Zinc Phenolsulfonate	127-82-2
Zinc Phosphide	1314-84-7
Zinc Silicofluoride	16871-71-9
Zinc Sulfate	7733-02-0

METHOD SUMMARY:

Analysis for the above particulate zinc compounds in air samples may be performed by flame atomic absorption (AA) spectrometric determination of zinc. 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 solution containing the analyte is analyzed by AA, applying either direct aspiration or cupferron/butyl acetate extraction techniques. This method does not identify any specific zinc compounds, but measures the total zinc present in the sample.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

A blank should be processed with every 10 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 4.2 to 42 micrograms per cubic meter in a 240-liter air sample. Precision information

is furnished. Alberta Environment has determined the detection limit to be 0.01 micrograms per cubic meter of air.

REFERENCES:

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, 23545; 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.

ZINC

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Zinc	7440-66-6
Zinc Acetate	557-34-6
Zinc Ammonium Chloride	14639-97-5
	14639-98-6
	52628-25-8
Zinc Borate	1332-07-6
Zinc Bromide	7699-45-8
Zinc Carbonate	3486-35-9
Zinc Chloride	7646-85-7
Zinc Cyanide	557-21-1
Zinc Fluoride	7783-49-5
Zinc Formate	557-41-5
Zinc Hydrosulfite	7779-86-4
Zinc Nitrate	7779-88-6
Zinc Phenolsulfonate	127-82-2
Zinc Phosphide	1314-84-7
Zinc Silicofluoride	16871-71-9
Zinc Sulfate	7733-02-0

METHOD SUMMARY:

Analysis of soil/sediment samples for the above zinc compounds can be performed by atomic spectrometric analysis for zinc. A 2-gram solid sample is digested with nitric acid and hydrogen peroxide and analyzed by inductively coupled plasma (ICP) or flame atomic absorption spectrometry (AA). Analysis for zinc in oily samples may be performed by ICP after sample dilution with methyl isobutyl ketone (MIBK) or xylene, using standards miscible with these solvents. These methods do not identify any specific zinc-containing compounds, but measure the total zinc content of the sample.

INTERFERENCES:

High levels of silicon in the sample may cause interference when using AA. The use of polypropylene sample container caps is recommended to avoid possible contamination by sample container cap liners. For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods.

QUALITY CONTROL:

A reference standard should be analyzed routinely, 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 EPA-approved for analyses under the Clean Water Act. 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 286.1; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

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

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, Methods 7950, 7951; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy." pp. IV-99 to IV-125; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

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, pp. 516-519.

COST INFORMATION:

Cost per sample for analysis by flame AA is approximately $20-30 (list); by ICP, it is approximately $20 (list).

ZINC

Water Samples
Laboratory Method 1

Compound	CAS Number
Zinc	7440-66-6
Zinc Acetate	557-34-6
Zinc Ammonium Chloride	14639-97-5
	14639-98-6
	52628-25-8
Zinc Borate	1332-07-6
Zinc Bromide	7699-45-8
Zinc Carbonate	3486-35-9
Zinc Chloride	7646-85-7
Zinc Cyanide	557-21-1
Zinc Fluoride	7783-49-5
Zinc Formate	557-41-5
Zinc Hydrosulfite	7779-86-4
Zinc Nitrate	7779-88-6
Zinc Phenolsulfonate	127-82-2
Zinc Phosphide	1314-84-7
Zinc Silicofluoride	16871-71-9
Zinc Sulfate	7733-02-0

METHOD SUMMARY:

Analysis of water samples for zinc ions in the above compounds can be performed by atomic spectrometric analysis for zinc. A 100-milliliter sample is digested with hydrochloric/nitric acid or with nitric acid and hydrogen peroxide and the digest is analyzed by inductively coupled plasma (ICP) or flame atomic absorption (AA) spectrometry. Alternatively, the digested sample can be chelated with pyrrolidinedithiocarbamic acid, the resulting complex extracted into chloroform or methyl isobutyl ketone, and the solvent evaporated. The residue is then dissolved in nitric acid and the resulting solution analyzed by AA. These methods do not identify any specific zinc-containing compounds, but measure the total zinc content of the sample.

INTERFERENCES:

High levels of silicon in the sample may cause interference when using AA. The use of polypropylene sample container caps is recommended to avoid possible contamination by sample container cap liner material. For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods.

QUALITY CONTROL:

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 use in Clean Water Act analyses. Precision and accuracy information is furnished. The detection limit of the direct AA method is 0.005 milligrams per liter, the sensitivity 0.02 milligrams per liter and the optimum concentration range 0.05 to 2 milligrams per liter (APHA). The chelation-extraction/AA procedure is applicable over the range from 1 to 200 microgams zinc 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, Method 289.1; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

U.S. Environmental Protection Agency, Technical Additions to Methods for Chemical Analysis of Water and Wastes, Method 200.7, EPA-600/4-82-055, U.S. EPA: Cincinnati, 1982.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, Methods 7950, 7951; SW-846, U.S. EPA: Washington, 1982.

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

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-125; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption;" HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 328A; APHA: Washington, 1980.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, D1691(C), D1691(D); ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost per sample for analysis by flame AA is approximately $10-20 (list), or approximately $65 (list) with the chelation step; by ICP, it is approximately $10 (list).

ZINC

Water Samples
Laboratory Method 2

Compound	CAS Number
Zinc	7440-66-6
Zinc Acetate	557-34-6
Zinc Ammonium Chloride	14639-97-5
	14639-98-6
	52628-25-8
Zinc Borate	1332-07-6
Zinc Bromide	7699-45-8
Zinc Carbonate	3486-35-9
Zinc Chloride	7646-85-7
Zinc Cyanide	557-21-1
Zinc Fluoride	7783-49-5
Zinc Formate	557-41-5
Zinc Hydrosulfite	7779-86-4
Zinc Nitrate	7779-88-6
Zinc Phenolsulfonate	127-82-2
Zinc Phosphide	1314-84-7
Zinc Silicofluoride	16871-71-9
Zinc Sulfate	7733-02-0

METHOD SUMMARY:

Analysis of water samples for zinc ions in the above compounds can be performed by colorimetric analysis for zinc. A sample is digested and treated with Zincon® at pH 9, and the absorbance at 620 nanometers is measured. Alternatively, the sample can be treated with dithizone and the resulting zinc-dithizone complex extracted into carbon tetrachloride. The absorbance of the zinc-dithizone complex at 535 nanometers is measured. These methods do not identify any specific zinc-containing compound, but measure the total zinc content of the sample.

INTERFERENCES:

Cadmium(II), aluminum, and manganese(II) interfere with the Zincon® method Interference from bismuth, cadmium, cobalt, copper, gold, lead, mercury, nickel palladium, silver, and stannous tin at low levels are eliminated by complexing with sodium thiosulfate in the dithizone procedure. Ferric iron, residual chlorine, and other oxidizing agents convert dithizone to a yellow-brown color. Contamination from glassware containing zinc oxide can be a major problem. Dithizone and dithizone complexes are rapidly decomposed in strong light.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

These methods are not EPA-approved. The Zincon® procedure is applicable to 0.002 to 5 milligrams zinc per liter, while the dithizone method is applicable to levels above 0.1 milligrams zinc per liter. Precision and accuracy information is furnished.

REFERENCES:

American Public Health Association, Standard Methods for the Examination of Water and Wastewater. 15th ed., Methods 328B, 328C, 328D; APHA: Washington, 1980.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, D1691A, D1691B; ASTM: Philadelphia, 1983.

COST INFORMATION:

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

ZIRCONIUM

Air Samples
Laboratory Method

Compound	CAS Number
Zirconium Nitrate	13746-89-9
Zirconium Potassium Fluoride	16923-95-8
Zirconium Sulfate	14644-61-2
Zirconium Tetrachloride	10026-11-6

METHOD SUMMARY:

Atomic absorption (AA) spectrometric analysis for zirconium can be used for the measurement of the above zirconium compounds in air samples. The analysis is performed by cellulose ester membrane filtration of a known volume of air, followed by acid digestion of the filter with the collected particulates, and AA analysis of the resulting solution. This method does not identify any specific zirconium compounds, but measures the total particulate zirconium content of the sample.

INTERFERENCES:

Some experimental data suggest that the method does not work for zirconium sulfate and that the presence of sulfate interferes. Addition of ammonium fluoride is recommended to increase sensitivity.

QUALITY CONTROL:

A method blank should be processed with each set of 10 samples. Recovery should be determined and, if less than 95 percent, results should be adjusted 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 range of 2 to 9 milligrams zirconium per cubic meter for a 720-liter air sample. Precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set M, Method S185; 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 S185; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost information has not been obtained.

ARSENIC

Air Samples
Laboratory Method 6

Compound	CAS Number	Compound	CAS Number
Arsenic	7440-38-2	Calcium Arsenite	52740-16-6
Arsenic Acid	1327-52-2	Cupric Acetoarsenite	12002-03-8
	7778-39-4	Lead Arsenate	7784-40-9
Arsenic Disulfide	1303-32-8		7645-25-2
Arsenic Pentoxide	1303-28-2		10102-48-4
Arsenic Trichloride	7784-34-1	Potassium Arsenate	7784-41-0
Arsenic Trioxide	1327-53-3	Potassium Arsenite	10124-50-2
Arsenic Trisulfide	1303-33-9	Sodium Arsenate	7631-89-2
Cacodylic Acid	75-60-5	Sodium Arsenite	7784-46-5
Calcium Arsenate	7778-44-1		

METHOD SUMMARY:

The above inorganic arsenic compounds in air samples can be measured by graphite-furnace atomic absorption spectrometry. A known volume of air is drawn through a sodium bicarbonate-impregnated cellulose ester filter and cellulose backup pad. The filter and pad pair are digested with a nitric acid-peroxide solution and nickel nitrate is added to modify the matrix. The solution is analyzed by graphite-furnace atomic absorption spectrometry (AA). This method does not identify any particular arsenic-containing compounds, but measures the total arsenic content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks and standards are analyzed 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 0.67 to 32.2 micrograms per cubic meter, using a 400-liter sample. Precision and accuracy information is furnished.

REFERENCE:

U.S. Department of Health and Human Services, NIOSH Manual of Analytical Methods, Vol. 7, P&CAM 346; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $28-75, plus sampling costs.

ARSENIC

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Arsenic	7440-38-2	Calcium Arsenite	52740-16-6
Arsenic Acid	1327-52-2	Cupric Acetoarsenite	12002-03-8
	7778-39-4	Dichlorophenylarsine	696-28-6
Arsenic Disulfide	1303-32-8	Diethylarsine	692-42-2
Arsenic Pentoxide	1303-28-2	Lead Arsenate	7784-40-9
Arsenic Trichloride	7784-34-1		7645-25-2
Arsenic Trioxide	1327-53-3		10102-48-4
Arsenic Trisulfide	1303-33-9	Potassium Arsenate	7784-41-0
Cacodylic Acid	75-60-5	Potassium Arsenite	10124-50-2
Calcium Arsenate	7778-44-1	Sodium Arsenate	7631-89-2
		Sodium Arsenite	7784-46-5

METHOD SUMMARY:

Analysis of soil/sediment samples for arsenic and the above arsenic compounds can be performed by atomic spectrometric analysis for arsenic. A 2-gram sample is digested with nitric acid and hydrogen peroxide and analyzed by atomic spectrometry. For low concentrations, furnace atomic absorption spectrometry (AA) is used, while inductively coupled plasma (ICP) spectrometry is used for higher-concentration samples. This method measures the total arsenic content of the sample, but does not identify specific arsenic compounds.

INTERFERENCES:

No specific 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 approved for the Resource Conservation and Recovery Act (RCRA) and the Clean Water Act. 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 206.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, March 1979.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Method 3010, 3020, 6010, 7060, 7061; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Arsenic," pp. IV-77 to IV-87; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

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, pp. 516-519.

COST INFORMATION:

Cost per sample for analysis by AA is approximately $38 (list); by ICP, it is approximately $20 (list).

ARSENIC

Water Samples
Laboratory Method 1

Compound	CAS Number	Compound	CAS Number
Arsenic	7440-38-2	Calcium Arsenite	52740-16-6
Arsenic Acid	1327-52-2	Cupric Acetoarsenite	12002-03-8
	7778-39-4	Dichlorophenylarsine	696-28-6
Arsenic Disulfide	1303-32-8	Diethylarsine	692-42-2
Arsenic Pentoxide	1303-28-2	Lead Arsenate	7784-40-9
Arsenic Trichloride	7784-34-1		7645-25-2
Arsenic Trioxide	1327-53-3		10102-48-4
Arsenic Trisulfide	1303-33-9	Potassium Arsenate	7784-41-0
Cacodylic Acid	75-60-5	Potassium Arsenite	10124-50-2
Calcium Arsenate	7778-44-1	Sodium Arsenate	7631-89-2
		Sodium Arsenite	7784-46-5

METHOD SUMMARY:

Analysis for arsenic and the above arsenic compounds in water can be performed by atomic spectrometric analysis for arsenic. A 100-milliliter sample is digested with nitric acid and hydrogen peroxide and analyzed by atomic spectrometry. For low concentrations, furnace atomic absorption spectrometry (AA) is used, while inductively coupled plasma (ICP) spectrometry is used for higher-concentration samples. These methods measure total arsenic, and do not identify specific arsenic compounds.

INTERFERENCES:

No specific 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 approved for use in analysis for the Resource Conservation and Recovery Act (RCRA) and the Clean Water Act. 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 206.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

U.S. Environmental Protection Agency, Technical Additions to Methods for Chemical Analysis of Water and Wastes, Method 200.7; EPA-600/4-82-005, U.S. EPA: Cincinnati, 1982.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3010, 3020, 6010, 7060, 7061; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals Using Inductively-Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-125; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

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, pp. 516-519.

COST INFORMATION:

Cost per sample for this analysis by AA is approximately $18-50 (list), and $10 by ICP.

ARSENIC

Water Samples
Laboratory Method 2

Compound	CAS Number	Compound	CAS Number
Arsenic	7440-38-2	Calcium Arsenite	52740-16-6
Arsenic Acid	1327-52-2	Cupric Acetoarsenite	12002-03-8
	7778-39-4	Dichlorophenylarsine	696-28-6
Arsenic Disulfide	1303-32-8	Diethylarsine	692-42-2
Arsenic Pentoxide	1303-28-2	Lead Arsenate	7784-40-9
Arsenic Trichloride	7784-34-1		7645-25-2
Arsenic Trioxide	1327-53-3		10102-48-4
Arsenic Trisulfide	1303-33-9	Potassium Arsenate	7784-41-0
Cacodylic Acid	75-60-5	Potassium Arsenite	10124-50-2
Calcium Arsenate	7778-44-1	Sodium Arsenate	7631-89-2
		Sodium Arsenite	7784-46-5

METHOD SUMMARY:

Atomic absorption spectrometry (AA) may be used for the analysis of arsenic and the above arsenic compounds in water. A 25-milliliter sample is combined with potassium iodide and the arsenic in the sample is reduced by stannous chloride and converted to arsine with zinc metal. The arsine gas is then analyzed by AA. Samples should be acidifed at the time of collection. This method does not differentiate among arsenic-containing compounds, but measures the total arsenic content of the sample.

INTERFERENCES:

High concentrations of antimony, chromium, cobalt, copper, iron, mercury, molybdenum, nickel, silver, and tin will interfere. Organic matter in the sample must be oxidized before analysis. Organic arsenic compounds must be converted to inorganic compounds. Samples containing industrial waste should be spiked to establish adequate recovery.

QUALITY CONTROL:

At least one duplicate sample should be analyzed every 10 samples. A reference standard should be analyzed periodically.

EPA/TECHNICAL STATUS:

This method is approved for the National Pollutant Discharge Elimination System (NPDES) and the Safe Drinking Water Act (SDWA). The method will determine arsenic concentrations of 2 micrograms arsenic per liter or greater and the optimum concentration range is 2 to 20 micrograms arsenic per liter. Precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 206.3; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, " Methods for the Determination of Arsenic," pp. IV-77 to IV-87; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed.; APHA: Washington, 1980, pp. 160-163, 173-177.

American Society for Testing and Materials, 1983 ASTM Book of Standards, Vol. 11.01, Method D 2972-78; ASTM: Philadelphia, 1983.

Alberta Environmental Centre, Methods Manual for Chemical Analysis of Water and Wastes, "Arsenic, Total:" Publication No. AECV81-M1, Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $18-50 (list).

ARSENIC

Water Samples
Laboratory Method 3

Compound	CAS Number	Compound	CAS Number
Arsenic	7440-38-2	Calcium Arsenite	52740-16-6
Arsenic Acid	1327-52-2	Cupric Acetoarsenite	12002-03-8
	7778-39-4	Dichlorophenylarsine	696-28-6
Arsenic Disulfide	1303-32-8	Diethylarsine	692-42-2
Arsenic Pentoxide	1303-28-2	Lead Arsenate	7784-40-9
Arsenic Trichloride	7784-34-1		7645-25-2
Arsenic Trioxide	1327-53-3		10102-48-4
Arsenic Trisulfide	1303-33-9	Potassium Arsenate	7784-41-0
Cacodylic Acid	75-60-5	Potassium Arsenite	10124-50-2
Calcium Arsenate	7778-44-1	Sodium Arsenate	7631-89-2
		Sodium Arsenite	7784-46-5

METHOD SUMMARY:

Arsenic and the above arsenic compounds in water samples can be measured spectrophotometrically. The arsenic is reduced to arsine in acid solution in a hydrogen generator. The arsine is passed through a scrubber to remove sulfide, and absorbed in a solution of silver diethyldithiocarbamate dissolved in pyridine or chloroform. The resultant red complex is measured in a spectrophotometer at 535 nm. This method does not identify any specific arsenic compounds, but measures the total arsenic content of the sample.

INTERFERENCES:

High concentrations of chromium, cobalt, copper, mercury, molybdenum, nickel, and silver can interfere in the generation of arsine. Antimony salts in the sample can form stibine, which interferes by yielding a red color with maximum absorbance at 510 nm.

QUALITY CONTROL:

The original calibration curve should be checked daily by analysis of a reagent blank and a standard.

EPA/TECHNICAL STATUS:

This method is approved for the National Pollutant Discharge Elimination System (NPDES) and the Safe Water Drinking Act (SWDA). Precision and accuracy information is furnished. The method is suitable for concentrations of arsenic at or above 10 micrograms per liter.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Waste, Method 206.4; EPA-600/4-79-020, U.S. EPA: Cincinnati, March 1979.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 307B; APHA: Washington, D.C., 1980.

American Society for Testing and Materials, 1983 ASTM Book of Standards, Vol. 11.01, Method D 2972-78; ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost information has not been obtained.

ARSINES

Air Samples
Laboratory Method

Compound	CAS Number
Dichlorophenylarsine	696-28-6
Diethylarsine	692-42-2

METHOD SUMMARY:

Atomic absorption spectrometry may be used to analyze air samples for the above arsines. Analysis is performed by passage of a known volume of air through a charcoal-tube sampling device, followed by desorption with dilute nitric acid and graphite-furnace atomic absorption (AA) analysis of the resulting solution. Nickel can be added to the solution to enhance the sensitivity. This method does not differentiate among various arsine-containing compounds.

INTERFERENCES:

High humidity severely decreases the capacity of the sampling apparatus. Interference by particulate arsenic compounds may be prevented by prefiltration of air during sampling.

QUALITY CONTROL:

Samples should be analyzed in duplicate, and 50 percent of the analyses should be standards, analyzed in duplicate if nickel is not added to the sample. If the nickel addition is made, a method blank and a standard should be run with every 10 samples.

EPA/TECHNICAL STATUS:

The method without using nickel is approved by the National Institute for Occupational Safety and Health (NIOSH), and has been validated over the range of 0.09 to 0.4 milligrams per cubic meter using a 10-liter sample. The method using nickel is proposed by NIOSH for use over the range of 1 to 10 micrograms per cubic meter. Precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set J, Method S229; PB-263 959 (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 S229; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $28-60 (list), plus sampling costs.

BERYLLIUM

Air Samples
Laboratory Method 1

Compound	CAS Number
Beryllium	7440-41-7
Beryllium Chloride	7787-47-5
Beryllium Fluoride	7787-49-7
Beryllium Nitrate	13597-99-4
	7787-55-5

METHOD SUMMARY:

Analysis for the above particulate beryllium compounds in air can be performed by atomic spectrometry. A known volume of air is passed through a cellulose membrane filter, the filter is digested, and the resulting solution is analyzed by graphite-furnace background-corrected atomic absorption (AA) spectrometry or by flame AA spectrometry. This method measures the total non-volatile beryllium content of the sample and does not identify specific beryllium compounds.

INTERFERENCES:

No specific interferences are reported for the furnace AA method, while high concentration of aluminum, silicon, and magnesium will interfere with the flame method unless controlled by the addition of oxine (8-hydroxyquinoline) to the standards and samples.

QUALITY CONTROL:

A method blank should be processed with each set of 10 samples, and the analytical method recovery must be determined over the concentration range of interest. The method blank and percent recovery results should be used in the calculation of beryllium concentrations.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 2.7 to 11.8 micrograms beryllium per cubic meter for the furnace AA method and 0.015 to 4.0 micrograms per cubic meter for the flame AA method. Precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, Method P&CAM 121; 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 173; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set N, Method S339; PB-258 433 (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 S339; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

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

BERYLLIUM

Air Samples
Laboratory Method 2

Compound	CAS Number
Beryllium	7440-41-7
Beryllium Chloride	7787-47-5
Beryllium Fluoride	7787-49-7
Beryllium Nitrate	13597-99-4
	7787-55-5

METHOD SUMMARY:

Beryllium and the above beryllium compounds in air samples can be measured with arc emission spectrography. The sample is collected by cellulose membrane filtration, by an impinger containing 10-percent nitric acid solution, or by an electrostatic precipitator. The filter or impinger sample is ashed in nitric acid and the residue is converted to the chloride form by evaporation with 1:1 hydrochloric acid. The residue is dissolved in hydrochloric acid and water, concentrated, added to an electrode, and an AC arc is used to excite the beryllium in the sample. The spectrum is recorded photographically and percent transmittance is measured at 234.86 nanometers. This method does not identify any specific beryllium-containing compounds, but measures the total beryllium content of the sample.

INTERFERENCES:

High concentrations of iron in the sample will interfere.

QUALITY CONTROL:

A method blank and standards should be processed along with the samples.

EPA/TECHNICAL STATUS:

This is an operational method according to the National Institute for Occupational Safety and Health (NIOSH), for use over the range of 0.001 to 0.05 micrograms beryllium on the electrode. No precision or accuracy information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, Method P&CAM 123; 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 123; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost information has not been obtained.

BERYLLIUM

Air Samples
Laboratory Method 3

Compound	CAS Number
Beryllium	7440-41-7
Beryllium Chloride	7787-47-5
Beryllium Fluoride	7787-49-7
Beryllium Nitrate	13597-99-4
	7787-55-5

METHOD SUMMARY:

Analysis for beryllium and the above beryllium-containing compounds in air may be performed by inductively coupled plasma atomic emission spectroscopic (ICP) analysis for beryllium. A known volume of air is drawn through a mixed cellulose filter. The filter is ashed with a mixture of nitric and perchloric acids followed by analysis of the resulting solution by ICP. This method does not identify any specific beryllium-containing compounds, but measures the total beryllium content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks, spikes, and standards are prepared and 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 5 to 2000 micrograms beryllium per cubic meter, using a 500-liter sample. Limited 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 351; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

COST INFORMATION:

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

BERYLLIUM

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Beryllium	7440-41-7
Beryllium Chloride	7787-47-5
Beryllium Fluoride	7787-49-7
Beryllium Nitrate	13597-99-4
	7787-55-5

METHOD SUMMARY:

Analysis of soil/sediment samples for the above beryllium compounds can be performed by atomic spectrometric analysis for beryllium. A 2-gram sample is digested with nitric acid and hydrogen peroxide and analyzed by atomic spectrometry. For low concentrations, furnace atomic absorption spectrometry (AA) is used, while inductively coupled plasma spectrometry (ICP) or flame AA is used for higher-concentration samples. This method does not differentiate between the various beryllium compounds.

INTERFERENCES:

No specific 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 approved for analyses for the Clean Water Act. 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 210.1; EPA-600/4-79-020, U.S. EPA: Cincinnati, March 1979.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3010, 3020, 6010, 7090, 7091; SW-846, U.S. EPA: Washington, 1982.

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

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-125; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

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, pp. 516-519.

COST INFORMATION:

Cost per sample for analysis by AA is approximately $38 (list); by ICP, it is approximately $20 (list).

BERYLLIUM

Water Samples
Laboratory Method

Compound	CAS Number
Beryllium	7440-41-7
Beryllium Chloride	7787-47-5
Beryllium Fluoride	7787-49-7
Beryllium Nitrate	13597-99-4
	7787-55-5

METHOD SUMMARY:

Water samples can be analyzed for the above beryllium compounds by atomic spectrometric analysis for beryllium. A 100-milliliter sample is digested with nitric acid and hydrogen peroxide and analyzed by atomic spectrometry. For low concentrations, furnace atomic absorption spectrometry (AA) is used, while inductively coupled plasma spectrometry (ICP) or flame spectrometry can be used for higher-concentration samples. These methods do not identify any particular beryllium compound, but measure the total beryllium content of the sample.

INTERFERENCES:

Aluminum concentrations above 500 micrograms per liter can interfere. Sodium and silicon concentrations above 1 milligram per liter depress the beryllium absorbance. Ionization suppression by addition of sufficient calcium chloride to standards and samples to yield a final calcium concentration of 900 milligrams per liter may be used.

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 approved for use in analyses for the Clean Water Act. Precision and accuracy information is furnished. The limits of detection are 25 micrograms beryllium per liter for flame AA, 0.3 micrograms per liter for ICP, and 0.2 micrograms per liter for furnace AA.

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 210.1, 210.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

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

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3010, 3020, 6010, 7090, 7091; SW-846, U.S. EPA: Washington, 1982.

U.S. Environmental Protection Agency, Technical Additions to Methods for Chemical Analysis of Water and Wastes, Method 200.7, EPA-600/4-82-055, U.S. EPA: Cincinnati, December 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-125; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

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, pp. 516-519.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 303C; APHA: Washington, 1980.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, D 3645-78; ASTM: Philadelphia, 1983.

COST INFORMATION:

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

CADMIUM

Air Samples
Laboratory Method 1

Compound	CAS Number
Cadmium	7440-43-9
Cadmium Acetate	543-90-8
Cadmium Bromide	7789-42-6
Cadmium Chloride	10108-64-2

METHOD SUMMARY:

Atomic spectrometry may be used for the analysis of the above cadmium compounds in air samples. The analysis is performed by membrane-filtration of a known volume of air, followed by acid digestion of the sampling medium, solubilization with hydrochloric acid, and atomic absorption (AA) spectrometric analysis of the resulting solution at 228.8 nm, either by direct aspiration or by an ammonium pyrrolidine dithiocarbamate/methyl isobutyl ketone (APDC/MIBK) solvent extraction procedure. This method measures the total cadmium content of the sample and does not identify specific cadmium compounds.

INTERFERENCES:

Silicon has been reported to interfere with the analysis.

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, results corrected accordingly. Duplicate determinations should agree within 5 percent.

EPA/TECHNICAL STATUS:

Method S312 is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 0.12 to 0.98 milligrams per cubic meter using a 25-liter sample. Method P&CAM 151 is an operational method for use over the range of 0.01 to 0.2 milligrams per cubic meter. Method P&CAM 173 is an operational method for use over the range of 4.2 to 84 micrograms per cubic meter using a 240-liter air sample. The APHA method is applicable in an optimum range of 0.05 to 20.0 micrograms of cadmium per milliliter of solution. An atmospheric concentration of 0.005 micrograms particulate cadmium per cubic meter can be detected using a 2000-cubic meter air sample. Environment Canada uses the solvent extraction procedure to obtain a detection limit of 0.001 micrograms per cubic meter of air. Precision and accuraccy information is furnished.

REFERENCES:

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.

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, Method S312; Publication No 77-157-C, U.S. DHEW: Cincinnati, 1977.

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, Method P&CAM 151; Publication No. 75-121, U.S. DHEW: Cincinnati, 1974.

U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set W, Method S312; PB-262 406 (NTIS), U.S. DHEW: Cincinnati, December 1976.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method D-10, "Trace Metal Analysis"; HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

American Public Health Association, Methods of Air Sampling and Analysis, 2nd ed., Method 311; APHA: Washington, 1977.

Alberta Environment, Methods Manual for Chemical Analysis of Atmospheric Pollutants, Methods 23535, 23536; 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.

CADMIUM

Air Samples
Laboratory Method 2

Compound	CAS Number
Cadmium	7440-43-9
Cadmium Acetate	543-90-8
Cadmium Bromide	7789-42-6
Cadmium Chloride	10108-64-2

METHOD SUMMARY:

Analysis for the above cadmium compounds in air may be performed by filtration of air and analysis of the filter medium by X-ray fluorescence spectrometry. The method is non-destructive, so that further analyses may be performed on samples after this analysis. The method is sensitive to amounts of cadmium above 40 micrograms, with a precision of 50 percent at that level. The sample-collection time may be up to 8 hours, but analysis time is only approximately 1 minute. The method measures total cadmium present in the sample, and does not differentiate between cadmium compounds or valence states.

INTERFERENCES:

Polyvinyl chloride (PVC) filters are unacceptable for sampling, and glass-fiber filters are acceptable for use only if they are of the highest purity. Cellulose membrane, cellulose fiber, polycarbonate membrane, and fluorocarbon membrane filters are acceptable.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The method should be applicable to 0.01 to 1.0 microgram of cadmium per cubic meter, depending on sample size. Precision information is furnished.

REFERENCES:

Rhodes, J. R.; Stout, J. A.; 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.

CADMIUM

Air Samples
Laboratory Method 3

Compound	CAS Number
Cadmium	7440-43-9
Cadmium Acetate	543-90-8
Cadmium Bromide	7789-42-6
Cadmium Chloride	10108-64-2

METHOD SUMMARY:

Analysis for cadmium and the above cadmium-containing compounds in air may be performed by inductively coupled plasma spectroscopy (ICP) analysis for cadmium. A known volume of air is drawn through a cellulose ester membrane filter. The filter is ashed with a mixture of nitric and perchloric acids, followed by ICP analysis. This method does not identify any specific cadmium-containing compounds, but measures the total cadmium content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks, spikes, and standards are prepared and 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 5 to 2000 micrograms cadmium per cubic meter for a 500-liter sample. Limited 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 351; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

COST INFORMATION:

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

CADMIUM

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Cadmium	7440-43-9
Cadmium Acetate	543-90-8
Cadmium Bromide	7789-42-6
Cadmium Chloride	10108-64-2

METHOD SUMMARY:

Analysis of soil/sediment samples for cadmium and the above cadmium compounds can be performed by atomic spectrometric analysis for cadmium. A 2-gram sample is digested with nitric acid and hydrogen peroxide and analyzed by atomic spectrometry. For low concentrations, furnace atomic absorption spectrometry (AA) is used, while inductively coupled plasma spectrometry (ICP) or flame AA is used for higher-concentration samples. These methods will not differentiate between various cadmium compounds.

INTERFERENCES:

No specific 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 approved for use in Resource Conservation and Recovery Act (RCRA) and Clean Water Act analyses. 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, Methods 213.1, 213.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

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

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

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3020, 6010, 7130, 7131; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48, "Methods for the Determination of Trace Metals Using Inductively-Coupled Plasma Atomic Emission Spectrometry," pp. IV-99 to IV-125; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

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, pp. 516-519.

COST INFORMATION:

Cost per sample for analysis by AA is approximately $25-50 (list); by ICP, it is approximately $20 (list).

CADMIUM

Water Samples
Laboratory Method

Compound	CAS Number
Cadmium	7440-43-9
Cadmium Acetate	543-90-8
Cadmium Bromide	7789-42-6
Cadmium Chloride	10108-64-2

METHOD SUMMARY

Analysis of water samples for the above cadmium compounds can be performed by atomic spectrometric analysis for cadmium. A 100-milliliter sample is digested with nitric acid and hydrogen peroxide and analyzed by atomic spectrometry. For low concentrations, furnace atomic absorption spectrometry (AA) is used, while inductively coupled plasma spectrometry (ICP) or flame AA is used for higher-concentration samples. This method measures cadmium, and does not identify specific cadmium compounds.

INTERFERENCES:

No specific 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 approved for Resource Conservation and Recovery Act (RCRA) and Clean Water Act analyses. Precision and accuracy information is furnished. Limits of detection are 5 micrograms cadmium per liter for flame AA, 0.1 micrograms per liter for furnace AA, and 4 micrograms per liter for ICP. APHA reports a detection limit of 2 micrograms per liter, a sensitivity of 25 micrograms per liter, and an optimum concentration range of 50 to 200 micrograms per liter for flame AA.

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 213.1, 213.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

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

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

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3010, 3020, 6010, 7130, 7131; SW-846, U.S. EPA: Washington, 1982.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method D-10, "Trace Metal Analysis," pp. D-10-1 to D-10-2; HASL-300, Environmental Measurements Laboratory, U.S. DOE, New York, 1983.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 303A; APHA: Washington, 1980.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, D 3557-78; ASTM: Philadelphia, 1983.

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, pp. 516-519.

COST INFORMATION:

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

CALCIUM

Air Samples
Laboratory Method 1

Compound	CAS Number
Calcium Arsenate	7778-44-1
Calcium Arsenite	52740-16-6
Calcium Carbide	75-20-7
Calcium Chromate	13765-19-0
Calcium Cyanide	592-01-8
Calcium Dodecyl-benzene Sulfonate	26264-06-2
Calcium Hypochlorite	7778-54-3

METHOD SUMMARY:

Analysis for the above calcium compounds in air may be performed by filtration of air and analysis of the filter medium for calcium by X-ray fluorescence spectrometry. The method measures total calcium present in the sample, and does not differentiate between chemical compounds or calcium valence states. The method is non-destructive, so that further analyses may be performed on samples after this analysis. The sample-collection time may be up to 8 hours, but analysis time is only approximately 1 minute.

INTERFERENCES:

Polyvinyl chloride (PVC) filters are unacceptable for sampling and glass-fiber filters are acceptable for use only if they are of the highest purity. Cellulose membrane, cellulose fiber, polycarbonate membrane, and fluorocarbon membrane filters are acceptable.

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 above 40 micrograms of calcium. The method should be applicable to 0.01 to 1.0 microgram of calcium per cubic meter, depending on sample size.

REFERENCES:

Rhodes, J. R.; Stout, J. A.; 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.

CALCIUM

Air Samples
Laboratory Method 2

Compound	CAS Number
Calcium Arsenate	7778-44-1
Calcium Arsenite	52740-16-6
Calcium Carbide	75-20-7
Calcium Chromate	13765-19-0
Calcium Cyanide	592-01-8
Calcium Dodecyl-benzene Sulfonate	26264-06-2
Calcium Hypochlorite	7778-54-3

METHOD SUMMARY:

Analysis for the above calcium-containing compounds in air may be performed by inductively coupled plasma atomic emission spectroscopic (ICP) analysis for calcium. A known volume of air is drawn through a cellulose ester membrane filter. The filter is ashed with a mixture of nitric and perchloric acids followed by analysis by ICP. The method does not identify any specific calcium-containing compounds, but measures the total calcium content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks, spikes, and standards are prepared and 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 5 to 2000 micrograms calcium per cubic meter, using a 500-liter sample. Limited precision and accuracy information is furnished.

REFERENCE:

U.S. Department of Health and Human Services, NIOSH Manaul of Analytical Methods, Vol. 7, Method P&CAM 351; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

COST INFORMATION:

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

CALCIUM

Air Samples
Laboratory Method 3

Compound	CAS Number
Calcium Arsenate	7778-44-1
Calcium Arsenite	52740-16-6
Calcium Carbide	75-20-7
Calcium Chromate	13765-19-0
Calcium Cyanide	592-01-8
Calcium Dodecyl-benzene Sulfonate	26264-06-2
Calcium Hypochlorite	7778-54-3

METHOD SUMMARY:

Analysis for the above particulate calcium compounds in air samples may be performed by flame atomic absorption (AA) spectrometric determination of calcium. A known volume of air is drawn through a membrane filter. The filter with the collected particulates is digested with nitric acid, and the resulting solution is analyzed by AA. This method does not identify any specific calcium-containing compounds, but measures the total calcium content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

A blank should be processed with every 10 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 4.2 to 210 micrograms per cubic meter, using a 240-liter air sample. Precision information is furnished.

REFERENCES:

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.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption;" HASL-300, Environmental Measurements Laboratory, U.S. DOE, New York, 1983.

COST INFORMATION:

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

CALCIUM

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Calcium Arsenate	7778-44-1
Calcium Arsenite	52740-16-6
Calcium Carbide	75-20-7
Calcium Chromate	13765-19-0
Calcium Cyanide	592-01-8
Calcium Dodecyl-benzene Sulfonate	26264-06-2
Calcium Hypochlorite	7778-54-3

METHOD SUMMARY:

Analysis of soil/sediment samples for the above calcium compounds can be performed by atomic spectrometric analysis for calcium. Samples of 100 milligrams are digested with nitric acid, perchloric acid, hydrogen fluoride, and boric acid, mixed with a lanthanum chloride solution, and analyzed by atomic absorption spectrometry. Samples should be stored in polyurethane bags and frozen immediately after collection. This method does not differentiate among calcium-containing samples, but measures the total calcium content of the sample.

INTERFERENCES:

Standard and sample compositions should be closely matched, and solvent extraction techniques may be necessary. An excess of sodium should be added to the sample to reduce ionization interferences.

QUALITY CONTROL:

Quality control procedures are not provided.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information is furnished. The detection limit is 10 milligrams of calcium per kilogram of sample (Environment Canada).

REFERENCES:

Environment Canada, Analytical Methods Manual, NAQUADAT No. 20050; Environment Canada, Inland Waters Directorate, Water Quality Branch: Ottawa, Canada, 1975.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $20-60 (list).

CALCIUM

Water Samples
Laboratory Method

Compound	CAS Number
Calcium Arsenate	7778-44-1
Calcium Arsenite	52740-16-6
Calcium Carbide	75-20-7
Calcium Chromate	13765-19-0
Calcium Cyanide	592-01-8
Calcium Dodecyl-benzene Sulfonate	26264-06-2
Calcium Hypochlorite	7778-54-3

METHOD SUMMARY:

Analysis of water samples for the above calcium compounds can be performed by atomic spectrometric or titrimetric analysis for calcium. In titrimetric analysis, a 50-milliliter sample is made basic with sodium hydroxide, mixed with indicator, and titrated with a disodium ethylenediamine tetraacetate (EDTA) solution. Alternatively, calcium in the sample can be precipitated quantitatively at pH 5 with oxalate, the precipitated calcium oxalate dissolved in acid, and the resulting solution titrated with potassium permanganate. In AA analysis, samples with a high level of organic substances must be digested before analysis. When analysis is not to be performed soon after sample collection, samples should be preserved by acidification with nitric acid. These methods do not differentiate among calcium compounds, but measure the total amount of calcium present in a sample.

INTERFERENCES:

When using flame AA, lanthanum should be added to the samples and standards to prevent possible interferences from phosphate, sulfate, and aluminum. High concentrations of magnesium will cause the calcium determination to be low. A large amount of alkali should be added to the sample and standards to control ionization interference. In the precipitation/titration analysis, use of an excess of oxalate overcomes interference by magnesium. Strontium oxalate may precipitate with calcium and require a correction of results. Silica interference may be removed by dehydration. Aluminum, iron, and manganese can be precipitated by ammonium hydroxide after treatment with persulfate. Phosphate should be removed by precipitation as the ferric salt, and suspended matter should be removed by centrifugation or filtration. When using EDTA titrimetry, strontium and barium may interfere, and alkalinity in excess of 30 milligrams per liter may cause an indistinct endpoint. Raising the pH to 12 to 13 will reduce or eliminate magnesium interferences in the EDTA titration.

QUALITY CONTROL:

When using AA, standards should be analyzed each time a sample or set of samples is analyzed. For each matrix analyzed, it is necessary to determine if

matrix effects and/or interferences require standard addition or serial dilution methods. Quality control information is not given for the titrimetric method.

EPA/TECHNICAL STATUS:

These methods are approved for the National Pollutant Discharge Elimination Service (NPDES). Precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Methods 215.1, 215.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption;" HASL-300, Environmental Measurements Laboratory, U.S. DOE, New York, 1983.

American Public Health Association, Standard Methods for the Examination of Water and Wastes, 15th ed. Method 303A, 311A, 311B, 311C; APHA: Washington, 1980.

American Society for Testing and Materials, 1983 ASTM Book of Standards, Vol. 11.01, Methods D 511-82B and D 511-82C; ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost per sample for analysis by AA is approximately $10-20 (list); by titration, it is approximately $20 (list).

CHLORIDES

Air Samples
Laboratory Method 1

Compound	CAS Number
Ammonium Chloride	12125-02-9
Antimony Trichloride	10025-81-9
Antimony Pentachloride	7647-18-9
Beryllium Chloride	7787-47-5
Cadmium Chloride	10108-64-2
Chromous Chloride	10049-05-5
Cupric Chloride	7447-39-4
Cyanogen Chloride	506-77-4
Ferric Chloride	7705-08-0
Ferrous Chloride	7758-94-3
Hydrochloric Acid	7647-01-0
Lead Chloride	7758-95-4
Nickel Chloride	7718-54-9
	37211-05-5
Phosphorus Trichloride	7719-12-2
Thallium(I) Chloride	7791-12-0
Zinc Chloride	7646-85-7

METHOD SUMMARY:

The above chlorides in air samples may be measured with an ion selective electrode. A known volume of air is passed through a midget impinger containing acetate buffer. The chloride collected is determined potentiometrically, using a chloride ion electrode. This method does not identify the specific chloride-containing compounds present, but measures the total chloride content of the sample.

INTERFERENCES:

Sulfide ion can interfere, as well as bromide, thiosulfate, and ammonium ions.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is classified as operational by the National Institute for Occupational Safety and Health (NIOSH) over the range of 0.1 to 1750 milligrams of chloride ion per cubic meter, using a 200-liter sample. Limited precision and accuracy information is provided.

REFERENCE:

U.S. Deparment of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 115; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $10-19 (list), plus sampling costs.

CHLORIDES

Air Samples
Laboratory Method 2

Compound	CAS Number
Ammonium Chloride	12125-02-9
Antimony Trichloride	10025-91-9
Antimony Pentachloride	7647-18-9
Beryllium Chloride	7787-47-5
Cadmium Chloride	10108-64-2
Chromous Chloride	10049-05-5
Cupric Chloride	7447-39-4
Cyanogen Chloride	506-77-4
Ferric Chloride	7705-08-0
Ferrous Chloride	7758-94-3
Hydrochloric Acid	7647-01-0
Lead Chloride	7758-95-4
Nickel Chloride	7718-54-9
	37211-05-5
Phosphorus Trichloride	7719-12-2
Thallium(I) Chloride	7791-12-0
Zinc Chloride	7646-85-7

METHOD SUMMARY:

Air samples can be analyzed for the above chloride compounds by passage of a known volume of air through fritted glass absorbers, impingers, filters, or an electrostatic precipitator. The sample is then dissolved and titrated with mercuric nitrate in the presence of a mixed diphenylcarbazone-bromphenol blue indicator. Mercuric ions react with chloride to form $HgCl_2$, and then the excess mercuric ions react with the diphenylcarbazone to form a blue complex. Fritted glass absorbers and impingers are used to sample gaseous chlorides, while filters, impingers, or electrostatic precipitators are used for particulate chlorides. This method does not identify any particular chloride salt, but measures the total inorganic chloride content of the sample.

INTERFERENCES:

Heavy metals can change the color of the solution at the endpoint and can be removed by a cation exchanger. The sample must contain less than 2.5 milligrams of ferric ion plus chromate ion. The presence of sulfides or sulfites requires treatment with hydrogen peroxide. Iodide, bromide, cyanate, and thiocyanate are titrated and reported as chloride.

QUALITY CONTROL:

A reagent blank should be processed with each sample.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. This method is applicable over the range of 0.07 to 1.5 milligrams of chloride per cubic meter, using a 30-liter air sample. Precision and accuracy information is furnished.

REFERENCE:

American Public Health Association, Methods of Air Sampling and Analysis, 2nd ed., Method 202; APHA: Washington, 1977.

COST INFORMATION:

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

CHLORIDES

Water Samples
Laboratory Method

Compound	CAS Number
Ammonium Chloride	12125-02-9
Antimony Trichloride	10025-91-9
Antimony Pentachloride	7647-18-9
Beryllium Chloride	7787-47-5
Cadmium Chloride	10108-64-2
Chromous Chloride	10049-05-5
Cupric Chloride	7447-39-4
Cyanogen Chloride	506-77-4
Ferric Chloride	7705-08-0
Ferrous Chloride	7758-94-3
Hydrochloric Acid	7647-01-0
Lead Chloride	7758-95-4
Nickel Chloride	7718-54-9
	37211-05-5
Phosphorus Trichloride	7719-12-2
Thallium(I) Chloride	7791-12-0
Zinc Chloride	7646-85-7

METHOD SUMMARY:

Analysis for the above chloride salts in water can be performed by analysis for chloride. The analysis can be performed titrimetrically or colorimetrically. Titrimetry can be done using a silver nitrate titrant and potassium chromate indicator, a mercuric nitrate titrant and diphenylcarbazone indicator, or a silver nitrate titrant and a silver/silver chloride electrode and millivolt meter as an indicator. The colorimetric method involves addition of ferric nitrate/mercuric thiocyanate to the sample, and the intensity of the color of the blood-red ferric thiocyanate, formed by reaction with the thiocyanate displaced by chloride, is measured. This method does not identify any particular chloride salt, but measures the total chloride content of the sample.

INTERFERENCES:

Bromide and iodide register as equivalent chloride concentrations in all three titrimetric methods. Cyanide does the same in the silver nitrate/potassium chromate titrimetric method, and sulfide, thiosulfate and sulfite ions interfere, but can be removed by treatment with hydrogen peroxide. In the mercuric nitrate titrimetric method, chromate, ferric, and sulfite ions above 10 milligrams per liter interfere. In the silver nitrate/potentiometric method ferricyanide causes high results, chromate and dichromate interfere and should be reduced to chromic ions, and ferric ions interfere if present in an amount substantially above the chloride concentration. In the colorimetric method, turbid samples should be filtered before analysis.

QUALITY CONTROL:

Duplicate and fortified samples should be processed in accordance with a quality assurance program.

EPA/TECHNICAL STATUS:

The mercuric nitrate titrimetric method and the colorimetric method are EPA-approved for use under the Clean Water Act. The methods are suitable for analysis of relatively clean samples in the range from 0.15 to 10 milligrams chloride in a 50-milliliter sample (APHA). Precision and accuracy information is furnished.

REFERENCES:

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 407; APHA: Washington, 1980.

Alberta Environmental Centre, Methods Manual for the Chemical Analysis of Water and Wastes, "Chloride, Dissolved;" Publication No. AECV81-M1, Alberta Environmental Centre, Vegreville, Alberta, Canada, 1981.

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Methods 325.1, 325.2, 325.3; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

COST INFORMATION:

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

CHROMIC ACID AND CHROMATES

Air Samples
Laboratory Method

Compound	CAS Number
Ammonium Chromate	7788-98-9
Ammonium Dichromate	7789-09-5
Calcium Chromate	13765-19-0
Chromic Acid	11115-74-5
Lithium Chromate	14307-35-8
Potassium Chromate	7789-00-6
Potassium Dichromate	7778-50-9
Sodium Chromate	7775-11-3
Sodium Dichromate	10588-01-9
Strontium Chromate	7789-06-2

METHOD SUMMARY:

Air samples may be analyzed for the above hexavalent chromium compounds by colorimetry or by atomic spectrometry. The colorimetric analysis is performed by passing a known volume of air through a polyvinyl chloride filter, leaching the analyte from the filter with dilute sulfuric acid, and analyzing the resulting solution for Cr(VI) with a spectrophotometer. After using the same filtration procedure for sample collection, atomic spectrometric analysis is performed by extraction of Cr(VI) from the filter with hexane/methyl isobutyl ketone and ammonium pyrrolidinedithiocarbamate. The hexane solution is then analyzed for chromium by atomic absorption spectrometry. These methods do not identify any particular Cr(VI)-containing compound, but measure the total Cr(VI) content of the sample.

INTERFERENCES:

Many heavy metals, such as iron, copper, nickel, and vanadium, can interfere.

QUALITY CONTROL:

For the colorimetric method, a method blank must be processed with each set of 10 samples and the collection efficiency of the filtration system must be determined and used in calculations.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH) and validated over the range of 0.052 to 0.204 milligrams chromium(VI) per cubic meter for a 22.5-liter air sample. Precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set O, Method S317; PB-262 402 (NTIS), U.S. DHEW: Cincinnati, 1976.

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, Method S317; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.

American Public Health Association, Methods of Air Sampling and Analysis, 2nd ed., Method 807; APHA: Washington, 1977.

COST INFORMATION:

Cost per sample for analysis by the colorimetric method is approximately $20-30, plus sampling costs; for analysis by AA, it is approximately $20-50 (list), plus sampling costs.

CHROMIUM

Air Samples
Laboratory Method 1

Compound	CAS Number
Ammonium Chromate	7788-98-9
Ammonium Dichromate	7789-09-5
Calcium Chromate	13765-19-0
Chromic Acetate	1066-30-4
Chromic Acid	11115-74-5
Chromic Sulfate	10101-53-8
Chromium	7440-47-3
Chromous Chloride	10049-05-5
Lithium Chromate	14307-35-8
Potassium Chromate	7789-00-6
Potassium Dichromate	7778-50-9
Sodium Chromate	7775-11-3
Sodium Dichromate	10588-01-9
Strontium Chromate	7789-06-2

METHOD SUMMARY:

Analysis for the above chromium compounds in air may be performed by filtration of air and analysis of the filter and its contents for chromium by X-ray fluorescence spectrometry. The method is non-destructive, so that further analyses may be performed on samples after this analysis. The sample-collection time may be up to 8 hours, but analysis time is approximately 1 minute.

INTERFERENCES:

Polyvinyl chloride (PVC) filters are unacceptable for sampling, and glass-fiber filters are acceptable for use only if they are of the highest purity. Cellulose membrane, cellulose fiber, polycarbonate membrane, and fluorocarbon membrane filters are acceptable. The method measures total chromium present in the sample, and does not differentiate between chemical compounds or chromium valence states.

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 chromium above 7 micrograms and is applicable to 0.01 to 1.0 microgram of chromium per cubic meter, depending on sample size.

REFERENCES:

Rhodes, J. R.; Stout, J. A.; 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), or $20 (bid) plus sampling costs.

CHROMIUM

Air Samples
Laboratory Method 2

Compound	CAS Number
Ammonium Chromate	7788-98-9
Ammonium Dichromate	7789-09-5
Calcium Chromate	13765-19-0
Chromic Acetate	1066-30-4
Chromic Acid	11115-74-5
Chromic Sulfate	10101-53-8
Chromium	7440-47-3
Chromous Chloride	10049-05-5
Lithium Chromate	14307-35-8
Potassium Chromate	7789-00-6
Potassium Dichromate	7778-50-9
Sodium Chromate	7775-11-3
Sodium Dichromate	10588-01-9
Strontium Chromate	7789-06-2

METHOD SUMMARY:

Analysis for chromium and the above soluble chromium compounds in air samples can be performed by filtration of a known volume of air through a cellulose membrane filter, followed by acid digestion of the filter and its contents and analysis of the resulting solution for chromium by atomic absorption (AA) spectrometry. This method measures the total nitric acid-soluble chromium content of the sample and does not identify specific chromium compounds or valence states.

INTERFERENCES:

Iron and nickel may interfere. Some chromium compounds may not be totally dissolved by this procedure.

QUALITY CONTROL:

A method blank must be processed with each set of 10 samples and results calculated using the method blank results.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH) and validated over the range of 0.493 to 1.830 milligrams chromium per cubic meter, using a 90-liter sample. Measurement of lower concentrations can be made by use of smaller solution volumes, longer sampling times, or scale expansion. A method classified as operational by NIOSH is for use over the range of 21 to 210 micrograms of chromium per cubic meter of air, using a 240-liter sample. The detection limit stated by Alberta Environment is

0.03 micrograms of chromium per cubic meter. The APHA states that a concentration of 0.005 micrograms chromium per cubic meter can be detected, using a 2000-cubic meter sample. Precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set 0, Methods S323, S352; PB-262 402 (NTIS), U.S. DHEW: Cincinnati, November 1976.

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, Methods S323, S352; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, Method P&CAM 152; 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 173; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Alberta Environment, Methods Manual for Chemical Analyses of Atmospheric Pollutants, Methods 23535, 23537; Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

American Public Health Association, Methods of Air Sampling and Analysis, 2nd ed., Method 312; APHA: Washington, 1977.

COST INFORMATION:

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

CHROMIUM

Air Samples
Laboratory Method 3

Compound	CAS Number
Ammonium Chromate	7788-98-9
Ammonium Dichromate	7789-09-5
Calcium Chromate	13765-19-0
Chromic Acetate	1066-30-4
Chromic Acid	11115-74-5
Chromic Sulfate	10101-53-8
Chromium	7440-47-3
Chromous Chloride	10049-05-5
Lithium Chromate	14307-35-8
Potassium Chromate	7789-00-6
Potassium Dichromate	7778-50-9
Sodium Chromate	7775-11-3
Sodium Dichromate	10588-01-9
Strontium Chromate	7789-06-2

METHOD SUMMARY:

Analysis for the above chromium-containing compounds in air may be performed by inductively coupled plasma atomic emission spectroscopic (ICP) analysis for chromium. A known volume of air is drawn through a cellulose ester membrane filter. The filter is ashed with a mixture of nitric and perchloric acid followed by analysis by ICP. This method does not identify any specific chromium-containing compounds, but measures the total chromium content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks, spikes, and standards are prepared and 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 5 to 2000 micrograms chromium per cubic meter for a 500-liter sample. Limited precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health and Human Services, NIOSH Manual of Analytical Methods, Vol. 7, Method P&CAM 351; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy" pp. IV-99 to IV-125; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

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

CHROMIUM

Air Samples
Laboratory Method 4

Compound	CAS Number
Ammonium Chromate	7788-98-9
Ammonium Dichromate	7789-09-5
Calcium Chromate	13765-19-0
Chromic Acid	11115-74-5
Lithium Chromate	14307-35-8
Potassium Chromate	7789-00-6
Potassium Dichromate	7778-50-9
Sodium Chromate	7775-11-3
Sodium Dichromate	10588-01-9

METHOD SUMMARY:

Particulate chromium compounds in air samples can be measured by reaction kinetic analysis. The analyte is collected from a known volume of air on a polyvinyl chloride filter. Chromium(VI) is extracted from the filter into deionized distilled water by ultrasonic agitation and separated from interfering ions with a cation exchange resin. The chromium(VI) is determined by the kinetics of a catalytic reaction. The chromium(VI) catalyzes the oxidation by hydrogen peroxide of o-dianisidine to its semiquinone in an aqueous-ethanolic solution near pH 4. The rate of production of the semiquinone is proportional to the concentration of chromium(VI) and is measured spectrophotometrically as a function of time. This method does not identify any specific chromium-containing compounds present, but measures the total chromium(VI) content of the sample.

INTERFERENCES:

The ion exchange treatment step eliminates interfering ions.

QUALITY CONTROL:

A filter blank should be processed with the samples. Filters exposed to standard air samples should also be processed to determine recovery. The value of the blank and the recovery should be used in calculation of results.

EPA/TECHNICAL STATUS:

This method is classifed as proposed by the National Institute for Occupational Safety and Health (NIOSH) for use over the range of 0.01 to 0.25 milligrams chromium per cubic meter of air, using a 100-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 182; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost information has not been obtained.

CHROMIUM

Air Samples
Laboratory Method 5

Compound	CAS Number
Ammonium Chromate	7788-98-9
Ammonium Dichromate	7789-09-5
Calcium Chromate	13765-19-0
Chromic Acetate	1066-30-4
Chromic Acid	11115-74-5
Chromic Sulfate	10101-53-8
Chromium	7740-47-3
Chromous Chloride	10049-05-5
Lithium Chromate	14307-35-8
Potassium Chromate	7789-00-6
Potassium Dichromate	7778-50-9
Sodium Chromate	7775-11-3
Sodium Dichromate	10588-01-9
Strontium Chromate	7789-06-2

METHOD SUMMARY:

Analysis of air for chromium and the above chromium-containing compounds can be performed by colorimetry. A known volume of air is passed through silver metal membrane filters for soluble particulate chromium and compounds, while insoluble chromium particulates are collected on cellulose ester-type membrane filters. The collected samples are oxidized to transform all chromium present to the hexavalent state, which is then reacted with s-diphenylcarbazide. The intensity of the color of the resulting complex is measured with a spectrophotometer. This method does not identify any specific chromium-containing compound, but measures the total hexavalent and/or trivalent chromium content or the total bivalent and metallic chromium content of the sample. The method requires a special fusion step for determination of insoluble chromium in particulates.

INTERFERENCES:

Iron and vanadium react with s-diphenylcarbazide to produce yellow-brown colors, and mercury produces a violet precipitate very slowly, which can be avoided by the addition of sodium chloride. Extraction at pH 4 with 8-quinolinol can be used to remove large amounts of iron, molybdenum, copper, and vanadium. Nitrates are detrimental to color stability, and interferences from iron can be reduced by addition of phosphate buffer before the color reagent.

QUALITY CONTROL:

No quality control procedures are provided.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The method is applicable to chromium concentrations of 0.02 milligrams and greater per cubic meter, using a 150-liter sample. Limited precision and accuracy information is furnished.

REFERENCE:

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.03, Method D 3586-77; ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost information has not been obtained.

CHROMIUM

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Ammonium Chromate	7788-98-9
Ammonium Dichromate	7789-09-5
Calcium Chromate	13765-19-0
Chromic Acetate	1066-30-4
Chromic Acid	11115-74-5
Chromic Sulfate	10101-53-8
Chromium	7740-47-3
Chromous Chloride	10049-05-5
Lithium Chromate	14307-35-8
Potassium Chromate	7789-00-6
Potassium Dichromate	7778-50-9
Sodium Chromate	7775-11-3
Sodium Dichromate	10588-01-9
Strontium Chromate	7789-06-2

METHOD SUMMARY:

Analysis of soil/sediment samples for the above chromium compounds can be performed by atomic spectrometric analysis for chromium. A 2-gram solid sample is digested with nitric acid and hydrogen peroxide, and analyzed by inductively coupled plasma (ICP) or flame atomic absorption spectrometry (AA). Analysis for chromium in oily samples may be performed by ICP, after diluting the sample with methyl isobutyl ketone (MIBK) or xylene, using standards miscible with these solvents. These methods measure only the total chromium content of samples and do not differentiate between various compounds and/or oxidation states.

INTERFERENCES:

Iron, nickel, and other metals in the sample may cause interferences when using flame atomic absorption spectrometry.

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 analyzed 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 approved for Resource Conservation and Recovery Act (RCRA) and Clean Water Act analyses. Limited 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: Cincinnati, April 23, 1982.

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 218.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

U.S. Environmental Protection Agency, Technical Additions to Methods for Chemical Analysis of Water and Wastes, Method 200.7; EPA-600/4-82-055, U.S. EPA: Cincinnati, December 1982.

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

U.S. Environmental Protection Agency, Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3010, 3020, 3040, 3050, 6010, 7190, 7191; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-125; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

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, pp. 516-519.

COST INFORMATION:

Cost per sample for analysis by AA is approximately $20-30 (list); by ICP, it is approximately $20 (list).

CHROMIUM

Water Samples
Laboratory Method

Compound	CAS Number
Ammonium Chromate	7788-98-9
Ammonium Dichromate	7789-09-5
Calcium Chromate	13765-19-0
Chromic Acetate	1066-30-4
Chromic Acid	11115-74-5
Chromic Sulfate	10101-53-8
Chromium	7740-47-3
Chromous Chloride	10049-05-5
Lithium Chromate	14307-35-8
Potassium Chromate	7789-00-6
Potassium Dichromate	7778-50-9
Sodium Chromate	7775-11-3
Sodium Dichromate	10588-01-9
Strontium Chromate	7789-06-2

METHOD SUMMARY:

Analysis for the above chromium compounds in water samples may be performed by atomic spectrometric analysis for chromium. A 100-milliliter sample is digested with nitric acid and hydrogen peroxide and analyzed by atomic spectrometry. For low concentrations, either furnace atomic absorption spectrometry (AA) or extraction of the sample at pH 3 to 9 with ammonium pyrrolidinedithiocarbamate and methyl isobutyl ketone and AA analysis of the extract is used, while inductively coupled plasma (ICP) spectrometry or flame AA can be used for higher-concentration samples. This method measures total chromium concentration and does not identify specific chromium-containing compounds or chromium oxidation states.

INTERFERENCES:

Iron, nickel, and cobalt at 100 micrograms per liter and magnesium at 30 milligrams per liter cause a negative interference in flame AA, which can be eliminated by additon of 8-hydroxyquinoline to samples.

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 analysis should be duplicates and/or standards.

EPA/TECHNICAL STATUS:

These methods are in use in the CERCLA program and are approved for Resource Conservation and Recovery Act (RCRA) and Clean Water Act analyses.

Precision and accuracy information is furnished. The detection limit is 0.05 milligrams chromium per liter for flame AA analysis, 21 micrograms chromium per liter for furnace AA, and 7 micrograms chromium per liter for ICP (instrumental detection limit).

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 218.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

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

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3010, 3020, 6010, 7190, 7191; SW-846, U.S. EPA: Washington, 1982.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Methods 303A, 303B; APHA: Washington, 1980.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, Method D1687; ASTM: Philadelphia, 1983.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-125; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

Cost per sample for analysis by furnace AA is approximately $18-50 (list): by ICP, approximately $10 (list); by extraction/AA, approximately $30-80 (list); and by flame AA, approximately $10-20 (list).

CHROMIUM, HEXAVALENT

Air Samples
Laboratory Method

Compound	CAS Number
Ammonium Chromate	7788-98-9
Ammonium Dichromate	7789-09-5
Calcium Chromate	13765-19-0
Chromic Acid	11115-74-5
Lithium Chromate	14307-35-8
Potassium Chromate	7789-00-6
Potassium Dichromate	7778-50-9
Sodium Chromate	7775-11-3
Sodium Dichromate	10588-01-9
Strontium Chromate	7789-06-2

METHOD SUMMARY:

The above hexavalent chromium compounds in air samples may be measured by colorimetry. A known volume of air is drawn through a polyvinyl chloride filter. The filter is extracted with hot 3-percent sodium carbonate/2-percent sodium hydroxide solution to dissolve all Cr(VI) and to protect it from reduction to Cr(III). The extract is acidified with sulfuric acid, sym-diphenylcarbazide is added, and the absorbance of the resulting solution at 540 nanometers is measured. This method does not identify any specific chromium compound, but measures the total hexavalent chromium content of the sample.

INTERFERENCES:

High concentrations of iron, vanadium, mercury, or nitrate in the sample can interfere.

QUALITY CONTROL:

One method blank is processed along with every 10 samples, and its value is used as a correction in the final calculation.

EPA/TECHNICAL STATUS:

This method has been proposed by the National Institute for Occupational Safety and Health (NIOSH) for use over the range of 0.5 to 10 micrograms per cubic meter, using a 600-liter 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 319; Publication No. 80-125, U.S. DHHS: Cincinnati, August 1980.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $20-50 (list), plus sampling costs.

CHROMIUM, HEXAVALENT

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Ammonium Chromate	7788-98-9
Ammonium Dichromate	7789-09-5
Calcium Chromate	13765-19-0
Chromic Acid	11115-74-5
Lithium Chromate	14307-35-8
Potassium Chromate	7789-00-6
Potassium Dichromate	7778-50-9
Sodium Chromate	7775-11-3
Sodium Dichromate	10588-01-9
Strontium Chromate	7789-06-2

METHOD SUMMARY:

Soil/sediment samples may be analyzed for the above hexavalent chromium compounds by coprecipitation and atomic absorption spectrometry (AA), by colorimetry, or by chelation-extraction and atomic spectrometry. The sample must be digested before analysis. For analysis by coprecipitation/AA, the hexavalent chromium in a sample is coprecipitated as lead chromate, and the lead chromate is separated from the supernatant. It is reduced, redissolved, and measured as trivalent chromium using either flame or furnace AA. For analysis by colorimetry, the dissolved hexavalent chromium is reacted with diphenylcarbazide and the concentration of the product is measured spectrophotometrically. For analysis by the chelation-extraction method, the hexavalent chromium in the sample is chelated and extracted with methyl isobutyl ketone (MIBK), and the extract is analyzed by flame AA.

INTERFERENCES:

Samples containing high concentrations of sulfate and chloride should be diluted before coprecipitation/AA analysis. High concentrations of molybdenum, vanadium, iron, and mercury in the sample may interfere with colorimetric analysis. High concentrations of other metals in the sample may interfere with chelation-extraction/AA procedures. In all cases, the samples should be stored at 4°C and the analysis should be carried out as soon as possible.

QUALITY CONTROL:

For every sample matrix analyzed, it must be determined if a reducing condition or chemical interference is present by analysis of a fortified sample. Recovery must be 85 to 115 percent for colorimetry. The method of standard additions may be necessary. Spikes, blanks, duplicates, and check standards must be analyzed approximately every 10 to 15 samples.

EPA/TECHNICAL STATUS:

These methods are approved for the Resource Conservation and Recovery Act (RCRA, 40 CFR, Part 261, Identification and Listing of Hazardous Wastes). Precision or accuracy information is not furnished.

REFERENCE:

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 7195, 7196, 7197; SW-846, U.S. EPA: Washington, 1982.

COST INFORMATION:

Cost per sample for analysis by chelation-extraction/AA is approximately $30-65 (list); by colorimetry, approximately $20-25 (list); and by coprecipitation/AA, approximately $20-70 (list).

CHROMIUM, HEXAVALENT

Water Samples
Laboratory Method

Compound	CAS Number
Ammonium Chromate	7788-98-9
Ammonium Dichromate	7789-09-5
Calcium Chromate	13765-19-0
Chromic Acetate	1066-30-4
Chromic Acid	11115-74-5
Chromic Sulfate	10101-53-8
Lithium Chromate	14307-35-8
Potassium Chromate	7789-00-6
Potassium Dichromate	7778-50-9
Sodium Chromate	7775-11-3
Sodium Dichromate	10588-01-9
Strontium Chromate	7789-06-2

METHOD SUMMARY:

Water samples may be analyzed for the above hexavalent chromium compounds by coprecipitation and atomic absorption spectrometry (AA), by colorimetry, or by chelation-extraction and AA. The sample must be digested before analysis. For analysis by coprecipitation/AA, the hexavalent chromium in a sample is coprecipitated as lead chromate and the lead chromate is separated from the supernatant. It is reduced, redissolved, and measured as trivalent chromium using either flame or furnace AA. For analysis by colorimetry, the dissolved hexavalent chromium sample is reacted with diphenylcarbazide and the product concentration is measured spectrophotometrically. For analysis by the chelation-extraction method, the hexavalent chromium in a 100-milliliter sample is chelated and extracted with methyl isobutyl ketone (MIBK), and the extract is analyzed by flame AA.

INTERFERENCES:

Samples containing high concentrations of sulfate and chloride should be diluted before coprecipitation/AA analysis. High concentrations of molybdenum, vanadium, iron, and mercury in the sample may interfere with colorimetric analysis. High concentrations of other metals in the sample may interfere with chelation-extraction/AA procedures. In all cases, the samples should be stored at 4°C and the analysis should be carried out as soon as possible.

QUALITY CONTROL:

For every sample matrix analyzed, it must be determined if a reducing condition or chemical interference is present by analysis of a fortified sample. Recovery must be 85 to 115 percent for colorimetry. The method of standard additions may be necessary. Spikes, blanks, duplicates, and check standards must be analyzed approximately every 10 to 15 samples.

EPA/TECHNICAL STATUS:

These methods are approved for the Resource Conservation and Recovery Act (RCRA, 40 CFR, Part 261, Identification and Listing of Hazardous Wastes). Precision and accuracy information is furnished. The colorimetric method is applicable over the range from 0.01 to 0.4 milligrams chromium per liter. Limited precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 7195, 7196, 7197; SW-846, U.S. EPA: Washington, 1982.

U.S. Environmental Protection Agency, Technical Additions to Methods for Chemical Analysis of Water and Wastes, Method 218.5; EPA-600/4-82-055, U.S. EPA: Cincinnati, December 1982.

Alberta Environmental Centre, Methods Manual for Chemical Analysis of Water and Wastes, "Chromium, Hexavalent, Dissolved;" Publication No. AECV81-M1, Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 3128; APHA: Washington, 1980.

COST INFORMATION:

Cost per sample for analysis by chelation extraction/AA is approximately $30-65 (list); by colorimetry, approximately $20-25 (list) and by coprecipitation/AA, approximately $20-70 (list).

CHROMIUM, TRIVALENT

Water Samples
Laboratory Method

Compound	CAS Number
Chromic Acetate	1066-30-4
Chromic Sulfate	10101-53-8

METHOD SUMMARY:

A colorimetric method may be used to analyze water samples for the above trivalent chromium compounds, which are decomposition products of hexavalent chromium compounds after reaction with organic molecules or other easily oxidized substances. A 150-milliliter sample is mixed with hydrochloric acid and ion exchange resin to remove any hexavalent chromium present. The remaining chromium is oxidized, concentrated, acidified, then treated with sym-diphenylcarbazide color reagent. The intensity of the developed color is measured by spectrophotometer or filter photometer and is proportional to the original amount of trivalent chromium in the sample. This method does not differentiate between trivalent chromium-containing compounds, but measures the total trivalent chromium content of the sample.

INTERFERENCES:

This method should not be used if organic matter in the sample cannot be easily oxidized or is present in greater concentrations than 100 milligrams per liter. Precipitable hydroxides and large concentrations of iron may interfere. Large amounts of phosphate or alkali earth metals may cause the loss of chromium.

QUALITY CONTROL:

No quality control procedures are provided.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information is furnished. The method will determine trivalent chromium concentrations of 0.5 to 5.0 milligrams per liter.

REFERENCE:

American Society for Testing and Materials, Annual Book of ASTM Methods, Volume 11.01, Method D 1687; ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $50-55 (list).

COBALT

Air Samples
Laboratory Method 1

Compound	CAS Number
Cobaltous Bromide	7789-43-7
Cobaltous Formate	544-18-3
Cobaltous Sulfamate	14017-41-5

METHOD SUMMARY:

Analysis for the above particulate cobalt compounds in air samples may be performed by flame atomic absorption (AA) spectrometric determination of cobalt. 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 solution containing the analyte is aspirated into the AA, either by direct aspiration, or after filter digestion, the resulting solution is extracted, and the resulting extract is then analyzed by AA. This method does not identify any specific cobalt-containing compound, but measures the total cobalt content of the sample.

INTERFERENCES:

Some cobalt compounds may not be dissolved completely by this procedure.

QUALITY CONTROL:

A blank should be processed with every 10 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 2.1 to 210 micrograms of cobalt per cubic meter, using a 240-liter air sample. Precision information is furnished. The detection limit stated by Alberta Environment is 0.001 micrograms of cobalt per cubic meter.

REFERENCES:

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, 1977.

Alberta Environment Methods Manual for Chemical Analysis of Atmospheric Pollutants, Methods 23535, 23538, Alberta Environmental Centre: Vegreville, Alberta, Ontario, 1981.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption;" HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

COST INFORMATION:

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

COBALT

Air Samples
Laboratory Method 2

Compound	CAS Number
Cobaltous Bromide	7789-43-7
Cobaltous Formate	544-18-3
Cobaltous Sulfamate	14017-41-5

METHOD SUMMARY:

Analysis for the above cobalt-containing compounds in air may be performed by inductively coupled plasma atomic emission spectroscopic (ICP) analysis for cobalt. A known volume of air is drawn through a cellulose ester membrane filter. The filter is ashed with a mixture of nitric and perchloric acids followed by analysis by ICP. This method does not identify any specific cobalt-containing compound, but measures the total cobalt content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks, spikes, and standards are prepared and 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 5 to 2000 micrograms cobalt per cubic meter, using a 500-liter sample. Limited precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health and Human Services, NIOSH Manual of Analytical Methods, Vol. 7, Method P&CAM 351; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals by Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-125: EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

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

COBALT

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Cobaltous Bromide	7789-43-7
Cobaltous Formate	544-18-3
Cobaltous Sulfamate	14017-41-5

METHOD SUMMARY:

Analysis of soil/sediment samples for the above cobalt compounds can be performed by atomic absorption spectrometry (AA). Samples of 100 milligrams are digested with perchloric acid, nitric acid, hydrofluoric acid, and boric acid, and analyzed for cobalt by AA. Samples should be stored in polyurethane bags and frozen immediately after collection. This method does not identify any specific cobalt compound, but measures the total cobalt content of the sample.

INTERFERENCES:

Chemical interferences in the sample may require solvent extraction techniques; standard and sample compositions should be closely matched.

QUALITY CONTROL:

No quality control procedures are provided.

EPA/TECHNICAL STATUS:

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

REFERENCES:

Environment Canada, Analytical Methods Manual; NAQUADAT No. 27050, Environment Canada, Inland Waters Directorate, Water Quality Branch: Ottawa, Canada, 1975.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $20-30 (list).

COBALT

Water Samples
Laboratory Method

Compound	CAS Number
Cobaltous Bromide	7789-43-7
Cobaltous Formate	544-18-3
Cobaltous Sulfamate	14017-41-5

METHOD SUMMARY:

Analysis of water samples for the above cobalt compounds can be performed by atomic spectrometric analysis for cobalt. A 100-milliliter sample is digested with nitric acid and hydrogen peroxide and analyzed by atomic spectrometry. For low concentrations, either furnace atomic absorption spectrometry (AA) or extraction of the sample at pH 2 to 10 with ammonium pyrrolidine dithiocarbamate and methyl isobutyl ketone followed by AA analysis of the extract is used, while inductively coupled plasma spectrometry (ICP) can be used for higher-concentration samples. This method measures the total cobalt content of the sample and does not differentiate among various cobalt-containing compounds.

INTERFERENCE:

No specific 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 approved for Clean Water Act analyses. Precision and accuracy information is furnished. The detection limit for flame AA is 30 micrograms cobalt per liter, the sensitivity is 0.2 milligrams per liter, and the optimum concentration range is 0.5 to 10 milligrams per liter (APHA). The chelation-extraction method is applicable in the range 10 to 1000 micrograms of cobalt 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, Method 219.1; EPA-600/4-79-020, U.S. EPA: Cincinnati, March 1979.

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

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, " Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-124; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Methods 303A, 303B, APHA: Washington, 1980.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, Method D 3558-82; ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost per sample for analysis by AA is approximately $18-60 (list); by ICP, it is approximately $10 (list).

COPPER

Air Samples
Laboratory Method 1

Compound	CAS Number
Copper	7440-50-8
Copper Cyanides	544-92-3
Cupric Acetate	142-71-2
Cupric Acetoarsenite	12002-03-8
Cupric Chloride	7447-39-4
Cupric Nitrate	3251-23-8
Cupric Oxalate	5893-66-3
Cupric Sulfate	7758-98-7
Cupric Sulfate, Ammoniated	10380-29-7
Cupric Tartrate	815-82-7

METHOD SUMMARY:

Atomic absorption spectrometry may be used to analyze air samples for dusts and mists containing the above copper compounds. A known volume of air is drawn through a cellulose ester membrane, followed by acid digestion of the sampling media and atomic absorption (AA) spectrometric analysis of the resulting solution. This method measures the total copper content of the sample and does not identify specific copper compounds.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

The percent recovery of the analysis must be determined for each sample or set of samples processed.

EPA/TECHNICAL STATUS:

This method is approved for National Institute for Occupational Safety and Health (NIOSH) compliance analyses and has been validated over the range of 0.5 to 2 milligrams of copper per cubic meter. Precision and accuracy information is furnished. A method classified by NIOSH as operational is for use over the range of 21 to 210 micrograms of copper per cubic meter of air, using a 240-liter sample. The limit of detection is 0.01 micrograms per cubic meter of air (Alberta Environment). An atmospheric concentration of 0.005 micrograms per cubic meter can be detected for a minimum sample volume of 2000 cubic meters (APHA).

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set M, Method S186; PB-265 029 (NTIS), U.S DHEW: Cincinnati, 1976.

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, Method S186; 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. 1, Method P&CAM 173; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals"; DOE: New York, 1983.

Alberta Environment, Methods Manual for Chemical Analyses of Atmospheric Pollutants, Method 23535, 23539; Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

American Public Health Association, Methods of Air Sampling and Analysis, 2nd ed., Method 313; APHA: Washington, 1977.

COST INFORMATION:

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

COPPER

Air Samples
Laboratory Method 2

Compound	CAS Number
Copper	7440-50-8
Copper Cyanides	544-92-3
Cupric Acetate	142-71-2
Cupric Acetoarsenite	12002-03-8
Cupric Chloride	7447-39-4
Cupric Nitrate	3251-23-8
Cupric Oxalate	5893-66-3
Cupric Sulfate	7758-98-7
Cupric Sulfate, Ammoniated	10380-29-7
Cupric Tartrate	815-82-7

METHOD SUMMARY:

Analysis for the above copper compounds in air may be performed by filtration of air and analysis of the filter medium for copper by X-ray fluorescence spectrometry. The method is non-destructive, so that further analyses may be performed on samples. The sample-collection time may be up to 8 hours, but analysis time is approximately 1 minute. The method measures total copper present in the sample, and does not differentiate between chemical compounds or copper valence states.

INTERFERENCES:

Polyvinyl chloride (PVC) filters are unacceptable for sampling, and glass-fiber filters are acceptable for use only if they are of the highest purity. Cellulose membrane, cellulose fiber, polycarbonate membrane, and fluorocarbon membrane filters are acceptable.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The method is sensitive to amounts of copper above 35 micrograms and is applicable to 0.01 to 1.0 microgram of copper per cubic meter, depending on sample size. Precision information is furnished.

REFERENCES:

Rhodes, J. R.; Stout, J. A.; 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.

COPPER

Air Samples
Laboratory Method 3

Compound	CAS Number
Copper	7440-50-8
Copper Cyanide	544-92-3
Cupric Acetate	142-71-2
Cupric Acetoarsenite	12002-03-8
Cupric Chloride	7447-39-4
Cupric Nitrate	3251-23-8
Cupric Oxalate	5893-66-3
Cupric Sulfate	7758-98-7
Cupric Sulfate, Ammoniated	10380-29-7
Cupric Tartrate	815-82-7

METHOD SUMMARY:

Analysis for the above copper-containing compounds in air may be performed by inductively coupled plasma atomic emission spectroscopic (ICP) analysis for copper. A known volume of air is drawn through a filter, the filter is ashed with a mixture of nitric and perchloric acids, and the resulting solution is analyzed by ICP. This method does not identify any specific copper-containing compound, but measures the total copper content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks, spikes, and standards are prepared and 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 5 to 2000 micrograms copper per cubic meter, using a 500-liter sample. Limited precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health and Human Services, NIOSH Manual of Analytical Methods, Vol. 7, Method P&CAM 351; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-124; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

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

COPPER

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Copper	7440-50-8
Copper Cyanides	544-92-3
Cupric Acetate	142-71-2
Cupric Acetoarsenite	12002-03-8
Cupric Chloride	7447-39-4
Cupric Nitrate	3251-23-8
Cupric Oxalate	5893-66-3
Cupric Sulfate	7758-98-7
Cupric Sulfate, Ammoniated	10380-29-7
Cupric Tartrate	815-82-7

METHOD SUMMARY:

Analysis of soil/sediment samples for the above copper compounds can be performed by atomic spectrometric analysis for copper. A 2-gram sample is digested with nitric acid and hydrogen peroxide and analyzed by inductively coupled plasma (ICP) or flame atomic absorption (AA) spectrometry. Flame AA spectrometry is not recommended for low levels of copper. These methods do not identify specific copper-containing compounds, but measure the total copper content of the sample.

INTERFERENCES:

For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods.

QUALITY CONTROL:

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 Clean Water Act analyses. 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 219.1; EPA-600/4-79-020, U.S. EPA: Cincinnati, March 1979.

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

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3010, 3020, 3050, 6010, 7210, 7211; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-124; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

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, pp. 516-519.

COST INFORMATION:

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

COPPER

Water Samples
Laboratory Method

Compound	CAS Number
Copper	7440-50-8
Copper Cyanides	544-92-3
Cupric Acetate	142-71-2
Cupric Acetoarsenite	12002-03-8
Cupric Chloride	7447-39-4
Cupric Nitrate	3251-23-8
Cupric Oxalate	5893-66-3
Cupric Sulfate	7758-98-7
Cupric Sulfate, Ammoniated	10380-29-7
Cupric Tartrate	815-82-7

METHOD SUMMARY:

Analysis of water samples for the above copper compounds can be performed by atomic spectrometric analysis for copper. A 100-milliliter sample is digested with nitric acid and hydrogen peroxide and analyzed by flame-atomization atomic absorption spectrometry (AA) or inductively coupled plasma spectrometry (ICP). Furnace atomization, rather than flame, AA is recommended for low levels of copper. For low levels, chelation/extraction with ammonium pyrrolidine dithiocarbamate in chloroform followed by digestion and AA analysis can also be used. These methods do not identify specific copper-containing compounds, but measure the total copper content of the sample.

INTERFERENCES:

For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods.

QUALITY CONTROL:

A reference standard should be analzyed weekly. Standards should be analyzed at least daily, 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 Clean Water Act and Resource Conservation and Recovery Act analyses. Precision and accuracy information is furnished. The detection limit is 0.02 milligrams per liter, the sensitivity is 0.12 milligrams per liter, and the optimum concentration range is 0.3 to 10 milligrams per liter for flame AA (APHA). For the chelation/extraction AA method, the optimum concentration range is 2 to 500 micrograms 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 220.1, 200.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

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

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

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3010, 3020, 3050, 6010, 7210, 7211; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-124; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, J. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption"; HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 303A; APHA: Washington, 1980.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, D1688-82; ASTM: Philadelphia, 1983.

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, pp. 516-519.

COST INFORMATION:

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

CYANIDES

Air Samples
Laboratory Method

Compound	CAS Number
Barium Cyanide	542-62-1
Calcium Cyanide	592-01-8
Copper Cyanide	544-92-3
Cyanide	57-12-5
Cyanogen	460-19-5
Cyanogen Bromide	506-68-3
Cyanogen Chloride	506-77-4
Mercuric Cyanide	592-04-1
Nickel Cyanide	557-19-7
Potassium Cyanide	151-50-8
Potassium Silver Cyanide	506-61-6
Silver Cyanide	506-64-9
Sodium Cyanide	143-33-9
Zinc Cyanide	557-21-1

METHOD SUMMARY:

Analysis for the above cyanides in air can be performed by passage of a known volume of air through a cellulose membrane filter and an impinger charged with 0.1-normal sodium hydroxide (in series), extraction of the filter with sodium hydroxide and analysis of the two resulting solutions with a cyanide ion-specific electrode and a pH/millivolt meter. This method does not identify any specific cyanide compound, but measures the total cyanide content of the air sample.

INTERFERENCES:

Sulfide ions irreversibly poison the electrode and may be removed by addition of a small amount of cadmium carbonate to the solution. High concentrations of other ions that form insoluble silver salts will also cause the electrode to malfunction, as will the presence of cations that form cyano-complexes. Gaseous hydrogen cyanide is an interference.

QUALITY CONTROL:

Sample recovery should be determined and a method blank should be processed with each set of 10 or fewer samples. Results of method-blank analyses and sample recovery determinations should be used in the calculation of the results.

EPA/TECHNICAL STATUS:

Method S250 is approved by the National Institute for Occupational Safety and Health (NIOSH), and has been validated over the range of 2.6 to 9.7 milligrams cyanide per cubic meter, using a 90-liter air sample. Method P&CAM

116 is classified as operational by NIOSH for use over the range of 0.013 to 13 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. 1, Method P&CAM 116; 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 S250; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.

American Public Health Service, Methods of Air Sampling and Analysis, 2nd ed., Method 808; APHA: Washington, 1977.

U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set R, Method S250; PB-262 403 (NTIS), U.S. DHEW: Cincinnati, December 1976.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $35 (list), plus sampling costs.

CYANIDES

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Barium Cyanide	542-62-1
Calcium Cyanide	592-01-8
Copper Cyanide	544-92-3
Cyanide	57-12-5
Cyanogen	460-19-5
Cyanogen Bromide	506-68-3
Cyanogen Chloride	506-77-4
Hydrogen Cyanide	74-90-8
Mercuric Cyanide	592-04-1
Nickel Cyanide	557-19-7
Potassium Cyanide	151-50-8
Potassium Silver Cyanide	506-61-6
Silver Cyanide	506-64-9
Sodium Cyanide	143-33-9
Zinc Cyanide	557-21-1

METHOD SUMMARY:

Determination of the above cyanides in soil/sediment samples can be done colorimetrically or by titrimetry. The cyanide in a 1-gram soil/sediment sample is distilled from an acidic solution into sodium hydroxide. The cyanide in the distillate is determined by either conversion to cyanogen chloride and colorimetry or by titration with silver nitrate. Chlorination preceeding distillation allows determination of total cyanide, including iron complexes. This method does not identify any specific cyanide-containing compounds, but measures the total cyanide content of the sample.

INTERFERENCES:

Samples should be preserved with sodium hydroxide after collection and stored at 4°C. All standards and samples must have the same salt content.

QUALITY CONTROL:

A blank, duplicate, spiked sample, and a reference standard must be distilled and analyzed with each 10 samples.

EPA/TECHNICAL STATUS:

This method is in use in the CERCLA program and is approved for use under the Resource Conservation and Recovery Act. No precision or 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, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Method 9010: SW-846, U.S. EPA: Washington, 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $43-63 (list).

CYANIDES

Water Samples
Laboratory Method

Compound	CAS Number
Barium Cyanide	542-62-1
Calcium Cyanide	592-01-8
Copper Cyanide	544-92-3
Cyanide	57-12-5
Cyanogen	460-19-5
Cyanogen Chloride	506-77-4
Hydrogen Cyanide	74-90-8
Mercuric Cyanide	592-04-1
Nickel Cyanide	557-19-7
Potassium Cyanide	151-50-8
Potassium Silver Cyanide	506-61-6
Silver Cyanide	506-64-9
Sodium Cyanide	143-33-9
Zinc Cyanide	557-21-1

METHOD SUMMARY:

Analysis of water samples for the above cyanides can be performed by colorimetry, titrimetry, or potentiometry. The cyanide in the sample can be released and converted to cyanogen chloride, which is determined colorimetrically. Alternatively, the acidified sample can be heated and hydrocyanic acid distilled into sodium hydroxide, and the resulting solution analyzed titrimetrically, colorimetrically, or potentiometrically with a cyanide-specific electrode. Titration is used for higher-level concentrations and colorimetry or potentiometry is used for lower levels. These methods do not identify specific cyanide-containing compounds, but measure the total cyanide content of the sample.

INTERFERENCES:

In some instances, thiocyanates in the sample will cause a positive interference. Titration cannot be used with samples containing fatty acids. In all methods, samples must be preserved with sodium hydroxide at the time of collection and stored at 4°C. Sulfides and oxidizing agents in the sample require the addition of cadmium carbonate and ascorbic acid, respectively.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

These methods are in use in the CERCLA program and are approved for use in Clean Water Act and Resource Conservation and Recovery Act analyses. Precision and accuracy information is furnished. The cyanogen chloride method has a

limit of detection of approximately 1 milligram cyanide per liter. The distillation-only method has limits of detection of 1.0, 0.03, and 0.03 milligrams cyanide per liter, respectively, depending on whether measurements are by titrimetry, colorimetry, or ion-selective electrode.

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 335.2, 335.3; EPA-600/4-79-020, U.S. EPA: Cincinnati, March 1979.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, Method D2036-82, ASTM: Philadelphia, 1983.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Method 9010; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Cyanide," pp. IV-126 to IV-135; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Alberta Environmental Centre, Methods Manual for Chemical Analysis of Water and Wastes, "Cyanide;" Publication No. AECV81-M1, Alberta Environmental Centre, Vegreville, Alberta, Canada, 1981.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Methods 412A, 412B, 412C, 412D, 412E: APHA: Washington, 1980.

COST INFORMATION:

Cost per sample for analysis by these methods is approximately $28-40 (list).

FLUORIDES

Air Samples
Laboratory Method 1

Compound	CAS Number
Antimony Trifluoride	7783-56-4
Beryllium Fluoride	7787-49-7
Ferric Fluoride	7783-50-8
Lead Fluoride	7783-46-2
Sodium Fluoride	7681-49-4
Zinc Fluoride	7783-49-5

METHOD SUMMARY:

Analysis for the above water-soluble particulate fluoride compounds in air can be performed by filtration and use of an ion-selective electrode. After high-volume glass-fiber filtration of air for 24 hours, the fluoride is extracted from the filter and the fluoride concentration in the extract is determined with a fluoride-specific electrode and an expanded-scale pH/millivolt meter. Five standard fluoride solutions should be used to calibrate the instrument. This method does not identify the fluoride-containing compounds present, but measures the total water-soluble particulate fluoride content of the sample.

INTERFERENCES:

Polyvalent cations such as Si(IV), Fe(III), and Al(III) can interfere. A Total Ionic Strength Activity Buffer (TISAB) is used to eliminate these interferences.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved, but is used by Environment Canada. No precision or accuracy information is furnished. The method is sensitive to concentrations of fluoride above 10 nanograms per cubic meter of air.

REFERENCES:

Alberta Environment, Methods Manual for Chemical Analysis of Atmospheric Pollutants, Method 23030; Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.03, Methods D3269-79 (11) and D3270-80; ASTM: Philadelphia, 1983.

COST INFORMATION:

The cost per sample for analysis by this method is approximately $25-44 (list), plus sampling costs.

FLUORIDES

Air Samples
Laboratory Method 2

Compound	CAS Number
Ammonium Bifluoride	1341-49-7
Ammonium Fluoride	12125-01-8
Beryllium Fluoride	7787-49-7
Ferric Fluoride	7783-50-8
Hydrogen Fluoride	7664-39-3
Lead Fluoride	7783-46-2
Sodium Bifluoride	1333-83-1
Sodium Fluoride	7681-49-4
Zinc Fluoride	7783-49-5

METHOD SUMMARY:

Analysis of air samples for the above fluorides can be performed spectrophotometrically. The sample is collected in a bubbler containing hot (70°C) sulfuric acid, which dissolves most interferences and releases the fluoride. The fluoride is then trapped in a solution of 0.05-percent sulfamic acid. Any nitrate present will react with the sulfamic acid and be converted to nitrogen gas. Alizarin complexone, lanthanum nitrate buffer, acetone, and distilled water are added, in that order, to the fluoride solution. The absorbance is measured at 618 nm on a spectrophotometer, and the concentration is obtained from a standard curve. This method does not identify the specific fluoride-containing compounds present, but measures the total fluoride content of the sample.

INTERFERENCES:

When the sampling procedure described is followed, all likely interferences are removed.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision or accuracy information is not given.

REFERENCES:

"Spectrophotometric Determination of Atmospheric Fluorides" In Environmental Pollutants-Selected Analytical Methods (SCOPE 6); W. Gallay, H. Egan, J. L. Monkman, R. Jruhaut, P. W. West, G. Widmark, Eds.; Ann Arbor Science: Ann Arbor, 1975, pp. 231-235.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.03, Method D3269 (10); ASTM: Philadelphia, 1983.

COST INFORMATION:

No cost information has been obtained.

FLUORIDES

Air Samples
Laboratory Method 3

Compound	CAS Number
Ammonium Bifluoride	1341-49-7
Ammonium Fluoride	12125-01-8
Beryllium Fluoride	7787-49-7
Ferric Fluoride	7783-50-8
Hydrogen Fluoride	7664-39-3
Lead Fluoride	7783-46-2
Sodium Bifluoride	1333-83-1
Sodium Fluoride	7681-49-4
Zinc Fluoride	7783-49-5

METHOD SUMMARY:

The above fluoride compounds in air samples may be measured using an ion-specific electrode. A known volume of air is drawn through midget impingers containing 0.1-molar sodium hydroxide. The samples are diluted 1:1 with Total Ionic Strength Activity Buffer (TISAB) followed by analysis using the fluoride-ion specific electrode. An alternative sampling and preparation procedure employs a device containing a membrane filter to collect particulate fluorides and an alkali-impregnated cellulose pad for gaseous fluorides. The filter and particulates are made alkaline and ashed, the residue fused, and the melt dissolved; the gaseous fluoride is extracted from the cellulose pad with water. The solutions are treated with TISAB and analyzed.

INTERFERENCES:

Hydroxide ion is the only significant interference; however, the addition of TISAB eliminates the problem. Large amounts of polyvalent metal ions such as aluminum may result in low readings.

QUALITY CONTROL:

A method blank and standards are processed along with the samples.

EPA/TECHNICAL STATUS:

The method with impinger sampling is an operational one as defined by the National Institute for Occupational Health and Safety (NIOSH), with a lower limit of detection of 0.009 milligrams of fluoride per cubic meter of air. The method with filtration sampling is defined as proposed by NIOSH, with a lower limit of 0.005 milligrams per cubic meter of air. No precision or accuracy information is furnished.

REFERENCES:

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 212; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 413; APHA: Washington, 1977.

American Society for Testing and Materials, ASTM 1983 Annual Book of Standards, Vol. 11.03, D3269-72 (11), D3269-80; ASTM: Philadelphia, 1983.

COST INFORMATION:

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

FLUORIDES

Water Samples
Laboratory Method 1

Compound	CAS Number
Ammonium Bifluoride	1341-49-7
Ammonium Fluoride	12125-01-8
Ammonium Silicofluoride	16919-19-0
Antimony Trifluoride	7783-56-4
Beryllium Fluoride	7787-49-7
Ferric Fluoride	7783-50-8
Hydrogen Fluoride	7664-39-3
Lead Fluoride	7783-46-2
Sodium Bifluoride	1333-83-1
Sodium Fluoride	7681-49-4
Zinc Fluoride	7783-49-5
Zinc Silicofluoride	16871-71-9
Zirconium Potassium Fluoride	16923-95-8

METHOD SUMMARY:

Automated colorimetry may be used to analyze water samples for the above fluoride compounds. The sample is distilled, the distillate is reacted with alizarin fluorine blue-lanthanum reagent, and the resulting blue complex is analyzed colorimetrically with an automated spectrophotometric analysis system. The method does not identify any specific fluoride compound, but determines the total fluoride content of the sample.

INTERFERENCES:

Interference by aluminum may be removed by addition of 8-hydroxyquinoline and extraction with chloroform before analysis.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is EPA-approved for use in Clean Water Act analyses. This method is applicable to fluoride concentrations of approximately 0.05 to 1.5 milligrams per liter. No precision or accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 340.3; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 413E; APHA: Washington, 1980.

COST INFORMATION:

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

FLUORIDES

Water Samples
Laboratory Method 2

Compound	CAS Number
Ammonium Bifluoride	1341-49-7
Ammonium Fluoride	12125-01-8
Ammonium Silicofluoride	16919-19-0
Antimony Trifluoride	7783-56-4
Beryllium Fluoride	7787-49-7
Ferric Fluoride	7783-50-8
Hydrogen Fluoride	7664-39-3
Lead Fluoride	7783-46-2
Sodium Bifluoride	1333-83-1
Sodium Fluoride	7681-49-4
Zinc Fluoride	7783-49-5
Zinc Silicofluoride	16871-71-9
Zirconium Potassium Fluoride	16923-95-8

METHOD SUMMARY:

Potentiometry may be used to analyze water samples for the above fluorides. A 50-milliliter sample is distilled as is and the distillate is analyzed with a fluoride-selective electrode and a pH/mV meter. This method does not identify any particular fluoride-containing compound, but measures the total fluoride content of the sample.

INTERFERENCES:

A pH 5 buffer containing a strong chelating agent should be added to prevent possible interferences for silicon, iron, aluminum, and pH.

QUALITY CONTROL:

Blanks and standards should be analyzed periodically.

EPA/TECHNICAL STATUS:

This method is EPA-approved for use in Clean Water Act analyses and will detect fluoride concentrations of approximately 0.1 to 1000 milligrams per liter. Limited precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 340.2; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 413; APHA: Washington, 1977.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, D1179-80; ASTM: Philadelphia, 1983.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, D3868-79; ASTM: Philadelphia, 1983.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.03, D3269 (11); ASTM: Philadelphia, 1983.

Alberta Environmental Centre, Methods Manual for Chemical Analysis of Water and Wastes, "Fluoride, Dissolved;" Publication No. AECV81-M1, Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $28-38 (list).

FLUORIDES

Water Samples
Laboratory Method 3

Compound	CAS Number
Ammonium Bifluoride	1341-49-7
Ammonium Fluoride	12125-01-8
Ammonium Silicofluoride	16919-19-0
Antimony Trifluoride	7783-56-4
Beryllium Fluoride	7787-49-7
Ferric Fluoride	7783-50-8
Hydrogen Fluoride	7664-39-3
Lead Fluoride	7783-46-2
Sodium Bifluoride	1333-83-1
Sodium Fluoride	7681-49-4
Zinc Fluoride	7783-49-5
Zinc Silicofluoride	16871-71-9
Zirconium Potassium Fluoride	16923-95-8

METHOD SUMMARY:

Spectrophotometry may be used to analyze water samples for the above fluorides. A 300-milliliter sample is distilled, after pretreatment for chlorine residues, and the distillate is reacted with zirconyl-SPADNS dye. The resultant color loss is measured spectrophotometrically. This method does not identify any particular fluoride-containing compound, but measures the total fluoride content of the sample.

INTERFERENCES:

The SPADNS reagent is more tolerant of interfering materials than other accepted fluoride reagents. No specific interferences are reported.

QUALITY CONTROL:

Blanks and standards must be analyzed routinely.

EPA/TECHNICAL STATUS:

This method is EPA-approved for use in Clean Water Act analyses and is applicable to samples containing approximately 0.1 to 2.5 milligrams of fluoride per liter. Precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 340.1; EPA-600/4-79-020, U.S. EPA: Cincinnati, March 1979.

American Public Health Association, Methods of Air Sampling and Analysis, 2nd ed., Methods 809, 810; APHA: Washington, 1977.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, Method D 1179-80; ASTM: Philadelphia, 1983.

COST INFORMATION:

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

HYDRAZINES

Air Samples
Laboratory Method

Compound	CAS Number
1,1-Dimethylhydrazine	57-14-7
Hydrazine	302-01-2
Methylhydrazine	60-34-4

METHOD SUMMARY:

The above hydrazines in air samples may be measured by gas chromatography. A known volume of air is drawn through a tube containing sulfuric acid-coated silica gel to trap the hydrazine compounds. The analytes are desorbed with distilled water and treated with a reagent containing sodium acetate and furfural. The resulting derivatives are extracted into ethyl acetate and analyzed by gas chromatography with flame ionization detection (GC/FID).

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

A blank should be 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 ranges of 0.002 to 60 milligrams hydrazine, 0.09 to 90 milligrams methylhydrazine, and 0.04 to 120 milligrams 1,1-dimethylhydrazine per cubic meter, based on a 96-liter air sample. 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 248; Publication No. 77-157-A, U.S. DHEW: Washington, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $85-100 (list).

INORGANIC ACIDS

Air Samples
Laboratory Method

Compound	CAS Number
Hydrochloric Acid	7647-01-0
Nitric Acid	7697-37-2
Phosphoric Acid	7664-38-2
Sulfuric Acid	7664-93-9

METHOD SUMMARY:

Inorganic acids in air, including hydrochloric, nitric, phosphoric, and sulfuric acids, may be measured using ion chromatography. A known volume of air is drawn through a silica gel tube, followed by analyte desorption in a heated dilute sodium bicarbonate/sodium carbonate solution. Ion chromatography is used for the analyses of the resulting solutions.

INTERFERENCES:

Possible interferences include sulfur dioxide for sulfuric acid, nitrogen dioxide for nitric acid, and chlorine or hypochlorite ion for hydrochloric acid.

QUALITY CONTROL:

Method blanks and standards are analyzed with the samples.

EPA/TECHNICAL STATUS:

This method is classified as operational by the National Institute for Occupational Safety and Health (NIOSH) for use over the range (in milligrams per cubic meter) of 0.14 to 14 for hydrochloric acid, using a 15-liter sample, 1.0 to 10 for nitric acid, and 0.5 to 2 for phosphoric acid and sulfuric acid, using a 48-liter sample. Precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health and Human Services, NIOSH Manual of Analytical Methods, Vol. 7, Method P&CAM 339; Publication No. 82-100, U.S. DHHS: August 1981.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-11, "Determination of Anions by Ion Chromatography;" HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

COST INFORMTION:

Cost per sample for analysis by this method is approximately $80 (list).

IODIDES AND BROMIDES

Water Samples
Laboratory Method

Compound	CAS Number
Antimony Tribromide	7789-61-9
Cadmium Bromide	7789-42-6
Lead Iodide	10101-63-0
Zinc Bromide	7699-45-8

METHOD SUMMARY:

Analysis of water samples for iodide and/or bromide ions from the above compounds can be performed by determination of bromide or iodide ion concentrations. The iodide ion concentration can be determined volumetrically, by oxidation of iodide to iodate with bromine, reduction of the iodate with iodine, and titration of the iodine with sodium thiosulfate. The combined iodide and bromide ion concentration can be determined volumetrically, by oxidation to iodate and bromate with hypochlorite, reduction of the resulting iodate and bromate with iodide to form iodine, and titration of the resulting iodine with sodium thiosulfate. Alternatively, iodide in the sample can be oxidized with nitrous acid and extracted into carbon tetrachloride. The intensity of the iodine color at 517 nanometers is then measured colorimetrically. Bromide ions can be oxidized to bromine with chromium trioxide, and the resulting bromine is extracted into carbon tetrachloride and measured spectrophotometrically at 417 nanometers. Additionally, an iodide-selective electrode and millivolt meter can be used. These methods do not identify any specific bromide- or iodide-containing compound, but measure the total iodide and/or bromide ion content of the sample.

INTERFERENCES:

The volumetric method suffers interferences from iron, manganese, and organics. Iron and manganese can be removed by precipitation and filtration, and traces of residual iron can be masked by the addition of fluoride. Interferences from hydrocarbons in the colorimetric determination of bromide can be removed by a pre-extraction with carbon tetrachloride. In the colorimetric iodide determination, chloride and bromide ions interfere, and interferences from sulfide ions can be removed by buffering the sample at a pH below 6. These methods are designed for brackish water, sea water, and brines.

QUALITY CONTROL:

No quality control procedures are provided.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The volumetric method is suitable for concentrations from 0.2 to 2000 milligrams iodide per liter and 5 to 6500 milligrams bromide per liter. The colorimetric methods are applicable to the

range 0.2 to 2000 milligrams iodide per liter and 40 to 6500 milligrams bromide per liter, while the iodide-selective electrode method is applicable to concentrations from 1 to 2000 milligrams of iodide per liter. Precision and accuracy information is furnished.

REFERENCE:

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, D3869-79; ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost per sample for analysis by the titrimetric methods are approximately $20-50 (list); by the ion-selective electrode method, approximately $10-19 (list).

IRON

Air Samples
Laboratory Method 1

Compound	CAS Number
Ferric Ammonium Citrate	1185-57-5
Ferric Ammonium Oxalate	2944-67-4
	55488-87-4
Ferric Chloride	7705-08-0
Ferric Dextran	9004-66-4
Ferric Fluoride	7783-50-8
Ferric Nitrate	10421-48-4
Ferric Sulfate	10028-22-5
Ferrous Ammonium Sulfate	10045-89-3
Ferrous Chloride	7758-94-3
Ferrous Sulfate	7720-78-7
	7782-63-0

METHOD SUMMARY:

Analysis for the above particulate iron compounds in air can be performed by filtration of air and analysis of the filter medium for iron by X-ray fluorescence spectrometry. The method is non-destructive, so that further analyses may be performed on samples. The sample-collection time may be up to 8 hours, but analysis time is approximately 1 minute. The method measures total iron present in the sample, and does not differentiate between chemical compounds or iron valence states.

INTERFERENCES:

Polyvinyl chloride (PVC) filters are unacceptable for sampling, and glass-fiber filters are acceptable for use only if they are of the highest purity. Cellulose membrane, cellulose fiber, polycarbonate membrane, and fluorocarbon membrane filters are acceptable.

QUALITY CONTROL:

Attenuation corrections must be included in the calculation of results.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision information is furnished. The method should be applicable to 0.01 to 1.0 micrograms of iron per cubic meter, depending on sample size.

REFERENCES:

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-09, "Trace Metals by X-ray Fluorescence;" HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

Rhodes, J. R.; Stout, J. A.; 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.

IRON

Air Samples
Laboratory Method 2

Compound	CAS Number
Ferric Ammonium Citrate	1185-57-5
Ferric Ammonium Oxalate	2944-67-4
	55488-87-4
Ferric Chloride	7705-08-0
Ferric Dextran	9004-66-4
Ferric Fluoride	7783-50-8
Ferric Nitrate	10421-48-4
Ferric Sulfate	10028-22-5
Ferrous Ammonium Sulfate	10045-89-3
Ferrous Chloride	7758-94-3
Ferrous Sulfate	7720-78-7
	7782-63-0

METHOD SUMMARY:

Analysis for the above particulate iron compounds in air samples may be performed by flame atomic absorption (AA) spectrometric determination of iron. 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 acid, and an aliquot of the resulting solution is aspirated into the AA spectrometer. This method does not identify any specific iron compound, but measures the total iron content of the sample.

INTERFERENCES:

High nickel and silica concentrations may interfere.

QUALITY CONTROL:

A blank should be processed with every 10 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 210 micrograms of iron per cubic meter in a 240-liter air sample. Precision information is furnished. The limit of detection determined by Alberta Environment is 0.05 micrograms iron per cubic meter of air.

REFERENCES:

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.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption;" HASL-300, Environmental Measurements Laboratory, U.S. DOE, New York, 1983.

Alberta Environment, Methods Manual for Chemical Analysis of Atmospheric Pollutants, Methods 23535, 23540; 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.

IRON

Air Samples
Laboratory Method 3

Compound	CAS Number
Ferric Ammonium Citrate	1185-57-5
Ferric Ammonium Oxalate	2944-67-4
	55488-87-4
Ferric Chloride	7705-08-0
Ferric Dextran	9004-66-4
Ferric Fluoride	7783-50-8
Ferric Nitrate	10421-48-4
Ferric Sulfate	10028-22-5
Ferrous Ammonium Sulfate	10045-89-3
Ferrous Chloride	7758-94-3
Ferrous Sulfate	7720-78-7
	7782-63-0

METHOD SUMMARY:

Analysis for the above iron-containing compounds in air may be performed by inductively coupled plasma atomic emission spectroscopy (ICP). A known volume of air is drawn through a cellulose ester membrane filter. The filter is ashed with a mixture of nitric and perchloric acids followed by analysis by ICP. This method does not identify any specific iron-containing compound, but measures the total iron content of the sample.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks, spikes, and standards are prepared and processed with the samples.

EPA/TECHNICAL STATUS:

This method has been proposed by the National Institute of Occupational Safety and Health (NIOSH) for use over the range of 5 to 2000 micrograms of iron per cubic meter for a 500-liter sample. Limited precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health and Services, NIOSH Manual of Analytical Methods, Vol. 7, Method P&CAM 351; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-124; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

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

IRON

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Ferric Ammonium Citrate	1185-57-5
Ferric Ammonium Oxalate	2944-67-4
	55488-87-4
Ferric Chloride	7705-08-0
Ferric Dextran	9004-66-4
Ferric Fluoride	7783-50-8
Ferric Nitrate	10421-48-4
Ferric Sulfate	10028-22-5
Ferrous Ammonium Sulfate	10045-89-3
Ferrous Chloride	7758-94-3
Ferrous Sulfate	7720-78-7
	7782-63-0

METHOD SUMMARY:

Analysis of soil/sediment samples for the above iron compounds can be performed by atomic spectrometric analysis for iron. 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). Direct AA is not recommended for low levels of iron. Alternatively, the metal can be chelated with pyrrolidinedithiocarbamic acid, the resulting complex extracted with chloroform, and the extract analyzed by AA. Analysis for iron in oily samples may be performed by ICP after sample dilution with methyl isobutyl ketone (MIBK) or xylene, using standards miscible with these solvents. These methods do not identify any specific iron-containing compounds, but measure the total iron content of the sample.

INTERFERENCES:

For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods.

QUALITY CONTROL:

Standards should be analyzed at least daily and a reference standard should be analyzed weekly. At least 10 percent of the analyses should be duplicates and/or standards.

EPA/TECHNICAL STATUS:

The ICP and direct AA methods are in use in the CERCLA program and are approved for use in Clean Water Act analyses. 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 236.1; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

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

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Methods for the Determination of Trace Metals Using Inductively Coupled Plasma Atomic Emission Spectroscopy," pp. IV-99 to IV-124; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Fassell, 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., 43(3), 1976, pp. 516-519.

American Public Health Association, Standard Methods for the Examination of Water and Wastes, 15th ed., Methods 303A, 303B; APHA: Washington, 1980.

COST INFORMATION:

Cost per sample for analysis by flame AA is approximately $20-30 (list); by ICP, it is approximately $20 (list).

IRON

Water Samples
Laboratory Method 1

Compound	CAS Number
Ferric Ammonium Citrate	1185-57-5
Ferric Ammonium Oxalate	2944-67-4
	55488-87-4
Ferric Chloride	7705-08-0
Ferric Fluoride	7783-50-8
Ferric Nitrate	10421-48-4
Ferric Sulfate	10028-22-5
Ferrous Ammonium Sulfate	10045-89-3
Ferrous Chloride	7758-94-3
Ferrous Sulfate	7720-78-7
	7782-63-0

METHOD SUMMARY:

Analysis of water samples for the above iron compounds can be performed by colorimetric or atomic spectrometric analysis for iron. Colorimetric analysis is performed by addition of orthophenanthroline and measurement of the absorbance at 510 nanometers, or by addition of bathophenanthroline to a sample previously reduced with hydroxylamine hydrochloride, and measurement of the absorbance at 533 nanometers. For atomic spectrometric analysis, 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). Furnace rather than flame AA is recommended for low levels of iron. Alternatively, the metal can be chelated with pyrrolidinedithiocarbamic acid, the resulting complex extracted with chloroform, and the extract analyzed by AA. These methods do not identify any specific iron-containing compound, but measure the total iron content of the sample.

INTERFERENCES:

For each matrix analyzed, it is necessary to determine if matrix effects and/or interferences require standard addition or serial dilution methods when performing direct atomic spectrometric analyses. Background correction may be necessary to determine low levels of iron in some samples. Interferences may be removed by extracting iron from a hydrochloric acid solution with diisopropyl ether and then extracting the ether with water to obtain an interference-free water solution.

QUALITY CONTROL:

A reference standard should be analyzed weekly, and at least 10 percent of the analyses should be duplicate samples and/or standards.

EPA/TECHNICAL STATUS:

These methods are in use in the CERCLA program and are approved for Clean Water Act analyses. Precision and accuracy information is furnished. For direct flame AA analysis the detection limit is 20 micrograms per liter, the sensitivity 120 micrograms per liter, and the optimum concentration range 0.3 to 10 milligrams per liter (APHA). The chelation-extraction AA procedure is applicable over the range of 5 to 500 micrograms per liter (ASTM). The bathophenanthroline colorimetric method is suitable for the determination of low levels of total iron (200 micrograms per liter and below), while the orthophenanthroline colorimetric method can be used for the determination of total, dissolved, and ferrous iron over the range of 0.05 to 3 milligrams per liter.

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 236.1; EPA-600/4-79-020, U.S. EPA: Cincinnati, 1979.

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

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

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Methods 303A, 303B; APHA: Washington, 1980.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.01, Methods D1068(A), D1068(B), D1068(C), D1068(D): 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); by colorimetric methods, approximately $20 (list).

IRON

Water Samples
Laboratory Method 2

Compound	CAS Number
Ferric Ammonium Citrate	1185-57-5
Ferric Ammonium Oxalate	2944-67-4
	55488-87-4
Ferric Chloride	7705-08-0
Ferric Fluoride	7783-50-8
Ferric Nitrate	10421-48-4
Ferric Sulfate	10028-22-5
Ferrous Ammonium Sulfate	10045-89-3
Ferrous Chloride	7758-94-3
Ferrous Sulfate	7720-78-7
	7782-63-0

METHOD SUMMARY:

Analysis of water samples for the above iron-containing compounds can be performed by spectrophotometric analysis for iron. A 50-milliliter sample is treated with hydroxylamine hydrochloride, and the ferrous iron originally present or formed by the hydroxylamine is reacted with 2,4,6-tripyridyl-s-triazine (TPTZ). The intensity of the color of the iron-TPTZ complex formed is measured and compared with that of standards. This method measures the total iron content of the sample and is not specific for any particular iron-containing compound.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

Duplicate and fortified samples should be processed in accordance with a quality control program.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The method is applicable to samples in the range of 0.05 to 2 milligrams iron per liter. Precision and accuracy information is furnished.

REFERENCE:

Alberta Environmental Centre, Methods Manual for Chemical Analysis of Water and Wastes, "Iron;" Publication No. AECV81-M1, Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $15-25 (list).

LEAD

Air Samples
Laboratory Method 1

Compound	CAS Number
Lead	7439-92-1
Lead Acetate	301-04-2
Lead Arsenate	7784-40-9
	7645-25-2
	10102-48-4
Lead Chloride	7758-95-4
Lead Fluoborate	13814-96-5
Lead Fluoride	7783-46-2
Lead Iodide	10101-63-0
Lead Nitrate	10099-74-8
Lead Phosphate	7446-27-7
Lead Stearate	7428-48-0
	1072-35-1
	56189-09-4
Lead Subacetate	1335-32-6
Lead Sulfate	15739-80-7
	7446-14-2
Lead Sulfide	1314-87-0
Lead Thiocyanate	592-87-0

METHOD SUMMARY:

Atomic absorption spectrometric analysis (AA) for lead may be used for the determination of the above particulate lead compounds in air samples. A known volume of air is passed through a cellulose-membrane or glass-fiber filter. The filter and collected particulates are wet-ashed and the resulting solution is then analyzed by flame or furnace AA. This method does not identify any specific lead-containing compound, but measures the total lead content of the sample.

INTERFERENCES:

Acetate, carbonate, fluoride, iodide, and phosphate ions suppress absorbance if at concentrations ten times that of lead. The presence of EDTA (0.1 molar) will overcome this interference. Calcium and high levels of sulfate ions may interfere. High levels of organic compounds in the sample may necessitate the use of background correction techniques. Since flameless AA is more prone to matrix effects than flame methods, the method of standard additions should be employed when flameless AA is used.

QUALITY CONTROL:

One blank filter should be analyzed with each set of ten or fewer samples, and corrections for the blank must be made for each sample. At least five standards should be used for calibration.

EPA/TECHNICAL STATUS:

Method S341 has been validated over the range of 0.128 to 0.399 milligrams lead per cubic meter in 180 liters of air and is approved by the National Institute for Occupational Safety and Health (NIOSH). Method P&CAM 155 is a NIOSH operational method for use over the range of 0.01 to 4 milligams per cubic meter in 100 liters of air. Method P&CAM 173 is a NIOSH operational method for use over the range of 42 to 840 micrograms per cubic meter, using a 240-liter sample. Method P&CAM 214 is proposed by NIOSH for use over the range of 0.02 to 0.4 milligrams per cubic meter, using a 5-liter sample of air. The APHA method uses flame AA and has a detection limit of 10 micrograms per cubic meter for a 100-liter air sample and an optimum concentration range of up to approximately 1 milligram per cubic meter. Alberta Environment has determined that the limit of detection is 0.01 micrograms per cubic meter. Precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set 0, Method S341; PB-262 402 (NTIS), U.S. DHEW: Cincinnati, 1976.

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, Method P&CAM 155; 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 173; 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 S341; 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. 1, Method P&CAM 214; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

U.S. Department of Health and Human Services, NIOSH Manual of Analytical Methods, Vol. 7, Method S341; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Elemental Analysis by Atomic Absorption Spectrometry," pp. IV-2 to IV-48; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-04, "Trace Metals by Atomic Absorption;" HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

Alberta Environment, Method Manual for Chemical Analysis of Atmospheric Pollutants, Methods 23535, 23541; Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

American Public Health Association, Methods of Air Sampling and Analysis, 2nd ed., Method 813; APHA: Washington, 1977.

COST INFORMATION:

Cost per sample for analysis by flame AA is approximately $10-20, plus sampling costs; by furnace AA, it is approximately $18-50, plus sampling costs.

LEAD

Air Samples
Laboratory Method 2

Compound	CAS Number
Lead	7439-92-1
Lead Acetate	301-04-2
Lead Arsenate	7784-40-9
	7645-25-2
	10102-48-4
Lead Chloride	7758-95-4
Lead Fluoborate	13814-96-5
Lead Fluoride	7783-46-2
Lead Iodide	10101-63-0
Lead Nitrate	10099-74-8
Lead Phosphate	7446-27-7
Lead Stearate	7428-48-0
	1072-35-1
	56189-09-4
Lead Subacetate	1335-32-6
Lead Sulfate	15739-80-7
	7446-14-2
Lead Sulfide	1314-87-0
Lead Thiocyanate	592-87-0

METHOD SUMMARY:

Analysis for the above particulate lead compounds in air may be performed by filtration of air and analysis of the filter deposit for lead by X-ray fluorescence spectrometry. The method is non-destructive, so that further analyses may be performed on samples. The method is sensitive to amounts of lead above 4 micrograms with a precision of 60 percent at that level. The sample-collection time may be up to 8 hours, but analysis time is only approximately 1 minute. The method measures total lead present in the sample, and does not differentiate between chemical compounds or lead valence states.

INTERFERENCES:

Polyvinyl chloride (PVC) filters are unacceptable for sampling, and glass-fiber filters are acceptable for use only if they are of the highest purity. Cellulose membrane, cellulose fiber, polycarbonate membrane, and fluorocarbon membrane filters are acceptable.

QUALITY CONTROL:

No quality control procedures are given.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $10-20 (list).

Note: NIOSH Method 351 (filter collection, acid digestion, analysis by ICP) is applicable. (Reference supplied by reviewer.)

B. ORGANIC SUBSTANCES

ACETATES

Air Samples
Laboratory Method

Compound	CAS Number
iso-Amyl Acetate	123-92-2
sec-Amyl Acetate	626-38-0
Butyl Acetate	123-86-4
iso-Butyl Acetate	110-19-0
sec-Butyl Acetate	105-46-4
tert-Butyl Acetate	540-88-5

METHOD SUMMARY:

Analysis of air samples for the above acetate esters can be performed by passage of the a known volume of air through a charcoal-tube filter, followed by desorption of analytes with carbon disulfide and analysis of the resulting solution by gas chromatography with flame ionization detection (GC/FID). The method is capable of measuring much smaller amounts if the desorption efficiency is adequate.

INTERFERENCES

High humidity severely decreases the capacity of the sampling apparatus.

QUALITY CONTROL:

The desorption efficiency must be determined over the range of analysis, and a method blank should be processed with each 10 samples. The desorption efficiency and blank results 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 following ranges (milligrams per cubic meter): 208 to 874 (iso-amyl acetate), 349 to 1460 (sec-amyl acetate), 306 to 1280 (iso-butyl acetate), 478 to 2005 (sec-butyl acetate), 424 to 1780 (tert-butyl acetate), 352 to 1475 (butyl acetate), using a 10-liter air sample. Precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set D, Methods S31, S32, S44, S45, S46, S47; PB-245 935, U.S. DHEW: Washington, 1975.

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, Methods S31, S32, S44, S45, S46, S47; Publication No. 77-157-B, U.S. DHEW: Washington, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70-90 (list), plus sampling costs.

ACID AND BASE/NEUTRAL EXTRACTABLE ORGANICS

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Acenaphthene	83-32-9	Diethyl Phthalate	84-66-2
Acenaphthylene	208-96-8	2,4-Dimethylphenol	105-67-9
Aldrin	309-00-2	Dimethyl Phthalate	131-11-3
Anthracene	120-12-7	4,6-Dinitro-o-cresol and salts	543-52-1
Benzidine	92-87-5	2,4-Dinitrophenol	51-28-5
Benzo[a]anthracene	56-55-3	2,4-Dinitrotoluene	121-14-2
Benzo[b]fluoranthene	205-99-2	2,6-Dinitrotoluene	606-20-2
Benzo[k]fluoranthene	207-08-9	Di-n-Octyl Phthalate	117-84-0
Benzo[ghi]perylene	191-24-2	1,2-Diphenylhydrazine	122-66-7
Benzo[a]pyrene	50-32-8	Endosulfan Sulfate	1031-07-8
α-BHC	319-85-7	Endrin	72-20-8
β-BHC	319-86-8	Endrin Aldehyde	7421-93-4
Bis(2-chloroethoxy)-methane	111-91-4	Fluoranthene	206-44-0
Bis(2-chloroethyl)-ether	111-44-4	Fluorene	86-73-7
Bis(2-chloroisopropyl)-ether	108-60-1	Heptachlor	76-44-8
4-Bromophenyl Phenyl Ether	101-55-3	Heptachlor Epoxide	1024-57-3
Chrysene	218-01-9	Hexachlorobenzene	118-74-1
Butyl Benzyl Phthalate	85-68-7	Hexachlorobutadiene	87-68-3
Chlordane	57-74-9	Hexachloroethane	67-72-1
p-Chloro-m-cresol	59-50-7	Indeno[1,2,3,-cd]pyrene	193-39-5
2-Chloronaphthalene	91-58-7	Isophorone	78-59-1
2-Chlorophenol	95-57-8	Naphthalene	91-20-3
4-Chlorophenyl Phenyl Ether	7005-72-3	p-Nitroaniline	100-01-6
DDD	72-54-8	Nitrobenzene	98-95-3
DDE	72-55-9	2-Nitrophenol	88-75-3
DDT	50-29-3	4-Nitrophenol	100-02-7
Dibenz[a,h]anthracene	53-70-3	N-Nitrosodimethylamine	62-75-9
Di-n-butyl Phthalate	84-74-2	N-Nitrosodiphenylamine	86-30-6
1,2-Dichlorobenzene	95-50-1	N-Nitrosodi-n-propylamine	621-64-7
1,3-Dichlorobenzene	541-73-1	Pentachlorophenol	87-86-5
1,4-Dichlorobenzene	106-46-7	Phenanthrene	85-01-8
3,3'-Dichlorobenzidine	91-94-1	Phenol	108-95-2
2,4-Dichlorophenol	121-14-2	Polychlorinated Biphenyls	1336-36-3
Dieldrin	60-57-1	Pyrene	129-00-0
		Toxaphene	8001-35-2
		1,2,4-Trichlorobenzene	120-82-1
		2,4,6-Trichlorophenol	88-06-2

METHOD SUMMARY:

Simultaneous analysis for the above organic compounds in soil/sediment samples may be performed by extraction and determination by gas chromatography/mass spectrometry (GC/MS). A 1-gram portion of sample is mixed with surrogates

and anhydrous sodium sulfate. The mixture is sonified with methylene chloride and the supernatant is screened by gas chromatography with flame ionization detection (GC/FID), in order to determine if extraction by the low-level method is necessary. The extract is concentrated and a portion is analyzed by GC/MS with a fused-silica capillary colum (FSCC). If screening yields negative or low results, a 30-gram sample mixed with surrogates and anhydrous sodium sulfate is extracted with methylene chloride/acetone and the extract is concentrated. A gel permeation chromatography (GPC) cleanup is optional. Acid and most base/neutral analytes may be determined simultaneously since the soil/sediment samples are extracted without pH adjustment.

QUALITY CONTROL:

The concentrate is analyzed using multiple internal standards, with quantitative determination based on the internal standard specified in the protocol. Mass abundance calibrations, internal standard responses, and retention times must be monitored daily. The ratios of the response factors of deuterated compounds to those of non-deuterated compounds must be between 0.8 and 1.2. The percent relative standard deviation for calibration check compounds must be less than 30 percent. A system performance check must be performed to assure that minimum average response factors are met before the calibration curve is used. The same check must be performed each 12 hours to assure the quality of the analytical system. A matrix spike and matrix-spike duplicate must be performed once for each set of samples or once for every 20 samples of similar concentration and/or sample matrix, whichever is greater.

INTERFERENCES:

Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing equipment. These materials must be routinely demonstrated to be free from laboratory interferences by processing and analyzing reagent blanks. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences may vary considerably from source to source.

EPA/TECHNICAL STATUS:

This method is in use in the CERCLA Contractor Laboratory Program and is based on methods approved for use under the Clean Water Act and the Resource Conservation and Recovery Act (RCRA). Precision and accuracy information is furnished (EPA Methods 624, 625). The method detection limit is 1 to 10 micrograms per liter, except for diethylphthalate (22), benzidine (44), 3,3'-dichlorobenzidine, polychlorinated biphenyls (30 to 40), 2,4-dinitrophenol (42), and 2-methyl-4,6,-dinitrophenol (24).

REFERENCES:

U.S. Environmental Protection Agency, Invitation for Bid, "Organics Analysis, Multi-Media, Multi-Concentration, GC/MS with Medium-Level GC Screen," Solicitation Number WA84A-266; U.S. EPA: Washington, June 15, 1984.

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, J. Longbottom; J. Lichtenburg, Eds., Methods 624, 625; EPA-600/4-82-057, U.S. EPA: Cincinnati, 1982.

Plumb, R. H., Characterization of Hazardous Waste Sites: A Methods Manual, Vol. II. Available Laboratory Analytical Methods, pp. III-93 to III-133, EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 8250, 8270; SW-846, U.S. EPA: Washington, 1982.

U.S. Geological Survey, Methods for Determination of Organic Substances in Water and Fluvial Sediments, Book 5, Laboratory Analysis, Chapter A4; Open-File Report 82-1004, USGS: Denver, 1983.

COST INFORMATION:

Cost per sample for the GC screen is approximately $70-90 (list), $60 (bid); GC/MS analysis is approximately $450-495 (list), $230 (bid).

ACID-EXTRACTABLE ORGANIC COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number
p-Chloro-m-Cresol	59-50-7
2-Chlorophenol	95-57-8
2,4-Dichlorophenol	120-83-2
2,4-Dimethylphenol	105-67-9
4,6-Dinitro-o-cresol and salts	534-52-1
2,4-Dinitrophenol	51-28-5
2-Nitrophenol	88-75-5
4-Nitrophenol	100-02-7
Pentachlorophenol	87-86-5
Phenol	108-95-2
2,4,6-Trichlorophenol	88-06-2

METHOD SUMMARY:

Analysis for the above acid-extractable organic compounds may be performed by gas chromatography/mass spectrometry (GC/MS). A 1-liter sample is fortified with surrogates and extracted with methylene chloride at a pH below 2. A portion of the extract is screened for organic compounds by gas chromatography with flame ionization detection (GC/FID) to determine whether sample dilution prior to extraction is required. If no dilution is required, then the extract is dried, concentrated, and analyzed by GC/MS. If the screening reveals that it is a medium-level sample, a 50-milliliter aliquot of the original sample is diluted to 1 liter, surrogates are added, and the resulting solution is extracted and analyzed as described. GC/MS analysis may be performed using a fused silica capillary column (FSCC).

QUALITY CONTROL:

The concentrate is analyzed using multiple internal standards with and the quantitative determinations are based on the internal standard specified in the protocol. Mass abundance calibrations, internal standard responses, and retention times must be monitored daily. The ratios of the response factors of deuterated compounds to those of non-deuterated compounds must be between 0.8 and 1.2. The percent relative standard deviation for calibration check compounds must be less than 30 percent. A system performance check must be performed to assure that minimum average response factors are met before the calibration curve is used. The same check must be performed each 12 hours to assure the quality of the analytical system. A matrix spike and matrix-spike duplicate must be performed once for each set of samples or once for every 20 samples of similar concentration and/or sample matrix, whichever is greater.

INTERFERENCES:

Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing equipment. These materials must be routinely

demonstrated to be free from laboratory interferences by processing and analyzing reagent blanks. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences may vary considerably from source to source.

EPA/TECHNICAL STATUS:

This method is currently in use in the CERCLA program. Precision and accuracy information is furnished. The method detection limit is 1 to 4 micrograms per liter, except for 2,4-dinitrophenol (42 micrograms) and 4,6-dinitro-o-cresol (26 micrograms).

REFERENCES:

U.S. Environmental Protection Agency, Invitation for Bid, "Organics Analysis, Multi-Media, Multi-Concentration, GC/MS with Medium Level GC Screen," Solicitation Number WA84A-266; U.S. EPA: Washington, June 15, 1984.

"Base/Neutrals and Acids - Method 625," Methods for Organic Chemical Analysis for Municipal and Industrial Wastewater, Longbottom, J; Lichtenburg, J.; Eds., EPA-600/4-82-057, U.S. EPA, 1983.

Method 8250, 8270, Test Methods for the Evaluation of Solid Wastes, 2nd ed., U.S. EPA, SW-846, July 16, 1982.

Sauter, A. D.; Betowski, L. D.; Smith, T. R.; Strickler, V. A; Beimer, R. G.; Colby, B. N.; Wilkenson, J. E. "Fused Silica Capillary Column GC/MS for the Analysis of Priority Pollutants," J. High Resolut. Chromatogr. Chromatogr. Comm., 4, 1981, pp. 366-384.

COST INFORMATION:

Cost per sample for the GC screen is approximately $70-90 (list); $60 (bid); the GC/MS analysis is approximately $225-250 (list).

ALIPHATIC ALDEHYDES

Air Samples
Laboratory Method

Compound	CAS Number
Acetaldehyde	75-07-0
Chloral	75-87-6
Chloroacetaldehyde	107-20-0
Crotonaldehyde	4170-30-3
	123-73-9
Glycidylaldehyde	765-34-4

METHOD SUMMARY:

Analysis for the above low-molecular-weight aldehydes in air can be performed by gas chromatography. The aldehydes are collected in bisulfite-filled impingers by sampling for 1 hour at 2 liters per minute. The bisulfite solution is then analyzed by gas chromatography with flame ionization detection (GC/FID).

INTERFERENCES:

Alcohols, esters, and ketones may be partially collected and the corresponding peaks may appear on the chromatogram.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished.

REFERENCE:

American Public Health Association, Methods of Air Sampling and Analysis, 2nd ed., M. Katz, Ed.; APHA: Washington, 1977, p. 300.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $60-90 (list), plus sampling costs.

ALIPHATIC AMINES

Air Samples
Laboratory Method

Compound	CAS Number
Butylamine	109-73-9
Diethylamine	109-89-7
Dimethylamine	124-40-3
Triethylamine	121-44-8

METHOD SUMMARY:

Gas chromatography may be used for the analysis of the above aliphatic amines in air samples. The analysis is performed by passage of a known volume of air through a silica-gel sampling device. The silica gel and adsorbed amines are treated with sulfuric acid, a portion of the solution is made alkaline with excess sodium hydroxide, and an aliquot of the alkaline solution is analyzed 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 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 proposed by the National Institute for Occupational Safety and Health (NIOSH), for use over the range of 1 to 2,400 milligrams per cubic meter, for a 10-liter sample of air. 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 221; Publication No. 77-157-A, U.S. DHEW: Washington, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $80-90 (list), plus sampling costs.

ALIPHATIC AMINES

Water Samples
Laboratory Method

Compound	CAS Number
Butylamine	109-73-9
iso-Butylamine	78-81-9
sec-Butylamine	513-49-5
	13952-84-6
tert-Butylamine	75-64-9
Diethylamine	109-89-7
Dimethylamine	124-40-3
Monoethylamine	75-04-7
n-Propylamine	107-10-8
Trimethylamine	75-50-3

METHOD SUMMARY:

Analysis of water samples for the above amines can be performed by gas chromatography (GC). A 1.5-microliter sample is injected directly into a gas chromatograph with nitrogen-sensitive detection.

INTERFERENCES:

Water may interfere. This can be eliminated by lowering the GC column temperature. The addition of ammonia to solutions before analysis suppresses adsorption of amines on glass surfaces and on the column packing. Other interferences are not reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished. The method can be used to detect amine concentrations as low as 0.03 parts per million for short-chain amines and 0.1 part per million for other amines.

REFERENCE:

Dalene, M.; Mathiasson, L.; Jönsson, J. A. "Trace Analysis of Free Amines by Gas-Liquid Chromatography," J. Chromatogr., 207, 1981, pp. 37-46.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $60 (list).

ALIPHATIC COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number
Acetaldehyde	75-07-0
Acetic Acid	64-19-7
Acetone	67-64-1
iso-Butyl Alcohol	78-83-1
n-Butyl Alcohol	71-36-3
Ethyl Ether	60-39-7
Methanol	67-56-1
Methyl Ethyl Ketone	78-93-3
Methyl Isobuty Ketone	108-10-1
Propionic Acid	79-09-4

METHOD SUMMARY:

The above aliphatic compounds in water samples can be measured by steam carrier gas chromatography with flame ionization detection (SCGC/FID). The sample is analyzed directly, without any pretreatment, producing a chromatogram with a signal-to-noise ratio large enough to permit quantitative analyses.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision or accuracy information is not furnished. The limit of detection is approximately 20 to 60 micrograms per liter.

REFERENCE:

Urano, K.; Ogura, K.; Wada, H. "Direct Analytical Method for Aliphtic Compounds in Water by Steam Carrier Gas Chromatography," Water Res., 15, 1981, pp. 225-231.

COST INFORMATION:

No cost information has been obtained.

ALKALOIDS

Water Samples
Laboratory Method

Compound	CAS Number
Reserpine	50-55-5
Strychnine	57-24-9

METHOD SUMMARY:

Analysis for the above alkaloids in water samples can be performed by high-performance liquid chromatography (HPLC). A sample is extracted with chloroform/methanol or diethyl ether/methanol mixture, and the extract is analyzed by HPLC with ultraviolet absorbance detection.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished. The limit of detection for this method is 20 nanograms injected on the column.

REFERENCE:

Verpoorte, R.; Svendsen, A. B. "High-speed Liquid Chromatography of Alkaloids. I," J. Chromatogr., 100, 1974, pp. 227-230.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $145 (list).

ALKYL BENZENE SULFONATES

Water Samples
Laboratory Method 1

Compound	CAS Number
Calcium Dodecylbenzene Sulfonate	26264-06-2
Dodecylbenzenesulfonic Acid	27176-87-0
Isopropanolamine Dodecylbenzene Sulfonate	42504-46-1
Sodium Dodecylbenzene Sulfonate	25155-30-0
Triethanolamine Dodecylbenzene Sulfonate	27323-41-7

METHOD SUMMARY:

Analysis for the above alkyl benzene sulfonates in water can be performed by addition of an acidic methylene blue solution to an acidified 100-milliliter sample, followed by serial extraction with chloroform. The chloroform extract is then analyzed spectrophotometrically. This method measures methylene-blue active substances, but does not specifically identify the methylene-blue active substance(s) being measured. For such information, methods such as infrared spectrometry or mass spectrometry should be used.

INTERFERENCES:

If potential interferences are present in the sample, pretreatment to remove them is required. This includes boiling the acidified sample to near dryness, addition of aqueous alkali solution and boiling, and finally extraction with 1-methyl heptylamine. Possible interferences include phosphorodithioates (e.g., organophosphate pesticides), benzene sulfonates, thiocyanates, picric acid, and alkyl sulfonates.

QUALITY CONTROL:

All glassware used must be free of scratches and etch marks, and should be preconditioned by rinsing with chloroform. A blank should be processed with each sample or set of samples. A quality control standard, near the midrange of the calibration curve, should be analyzed with each set of samples and the calibration factor should fall within the 95-percent confidence interval.

EPA/TECHNICAL STATUS:

This method is EPA-approved for the Clean Water Act. Precision information is furnished. The method is applicable over the range from 0.03 to 3.5 milligrams per liter, using a 100-milliliter sample (ASTM). The minimum detectable quantity of methylene-blue active substance(s) using this method is 10 micrograms.

REFERENCES:

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 512A; APHA: Washington, 1980.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Volume 11.02, D 2330-82; ASTM: Philadelphia, 1983, pp. 85-97.

Alberta Environmental Centre, Methods Manual for the Chemical Analysis of Water and Wastes, "Methylene Blue Active Substances;" Publication No. AECV81-M1, Alberta Environmental Centre, Vegreville, Alberta, Canada, 1981.

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 425.1; EPA 600/4-79-020, U.S. EPA: Cincinnati, 1979.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $60-100 (list).

ALKYL BENZENE SULFONATES

Water Samples
Laboratory Method 2

Compound	CAS Number
Calcium Dodecylbenzene Sulfonate	26264-06-2
Dodecylbenzenesulfonic Acid	27176-87-0
Isopropanolamine Dodecylbenzene Sulfonate	42504-46-1
Sodium Dodecylbenzene Sulfonate	25155-30-0
Triethanolamine Dodecylbenzene Sulfonate	27323-41-7

METHOD SUMMARY:

Analysis of water samples for the above alkyl benzene sulfonates can be performed using infrared spectrometry. A sample containing 10 to 25 milligrams of surfactant is treated with activated carbon, the adsorbed surfactant is desorbed with an alkaline benzene/methanol solution, the surfactants are hydrolyzed, and the resulting acids are converted to amine salts. The infrared absorptions due to the substituted benzene ring (9900 nm or 1010 cm^{-1}) and the sulfonate group (9600 nm or 1041 cm^{-1}) are measured. Quantification is made by use of a calibration curve. This method measures the total amount of the above alkyl benzene sulfonates in the sample, but cannot distinguish between linear alkylbenzene sulfonates; it does distinguish alkylbenzene sulfonates from other sulfonates.

INTERFERENCES:

Most natural interferences are removed in sample preparation. No specific interferences are listed.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished. The method is suitable for samples containing at least 0.25 milligrams methylene-blue active substance per liter (USGS).

REFERENCES:

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed.; Method 512B; APHA: Washington, 1980.

U.S. Geological Service, Methods for the Determination of Organic Substances in Water and Fluvial Sediments, Book 5, "Laboratory Analysis," Chapter A3; USGS: Denver, Open-File Report 82-1004, 1983.

COST INFORMATION:

Cost per sample for analyses by this method is approximately $75-85.

AMINES

Water Samples
Laboratory Method

Compound	CAS Number
Butylamine	109-73-9
Dipropylamine	142-84-7
Triethylamine	121-44-8

METHOD SUMMARY:

Analysis of water samples for the above alkylamines can be performed by ion-pair chromatography. The amines are separated with naphthalene-2-sulfonate as a counter-ion using high-performance liquid chromatography (HPLC) with ultra-violet absorbance detection.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision or accuracy information is not supplied.

REFERENCE:

Crommen, J.; Fransson, B.; Schill, G. "Ion-Pair Chromatography in the Low Concentration Range by Use of Highly Absorbing Counter Ions," J. Chromatogr., 142, 1977, pp. 283-297.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $125 (list).

AMINES AND ALCOHOLS

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Aniline	62-53-3
n-Butyl Alcohol	71-36-3
Butylamine	109-73-9
sec-Butylamine	513-49-5
	13952-84-6
Phenol	108-95-2
Pyridine	110-86-1
Quinoline	91-22-5
Thiophenol	108-98-5
Triethylamine	121-44-8

METHOD SUMMARY:

The above compounds may be measured in soil/sediment samples by extraction, derivatization, and F-19 nuclear magnetic resonance (NMR). A sample is extracted with dimethylformamide, treated with trifluoromethanesulfonyl chloride, and the products analyzed by F-19 NMR. The method gives NMR shifts for each derivative, but no quantitative results are given.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished.

REFERENCE:

Shue, F. F.; Yen, T. F. "Trifluoromethanesulfonyl Chloride for Identification of Oxygen, Nitrogen, and Sulfur Functional Groups by Fluorine-19 Nuclear Magnetic Resonance Spectrometry," Anal. Chem., 54, 1982, pp. 1641-1642.

COST INFORMATION:

No cost information has been obtained.

AMINES AND OTHER NITROGEN COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number
Ammonium Fluoride	12125-01-8
Ammonium Oxalate	6009-70-7
	5972-73-6
	14258-49-2
Aniline	62-53-3
Benzidine	92-87-5
Butylamine	109-73-9
iso-Butylamine	78-81-9
p-Chloroaniline	106-47-8
Ethylenediamine	107-15-3

METHOD SUMMARY:

Analysis of water samples for the above amines and other nitrogen compounds can be performed by spectrophotometry. Chloranil is added to a pH 9 buffered sample and the intensity of the color developed due to the formation of a molecular or charge-transfer complex is monitored. Thirty minutes should be allowed for color development at 65°C.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Some precision and accuracy information is furnished. The working range of the method is approximately 1 milligram per liter to 100 milligrams per liter.

REFERENCE:

Al-Ghabsha, T. S.; Rahim, S. A. "Spectrophotometric Determination of Microgram Amounts of Amines with Chloranil," Anal. Chim. Acta., 85, 1976, pp. 189-194.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $20-35 (list).

AROMATIC AMINES

Air Samples
Laboratory Method

Compound	CAS Number
Aniline	62-53-3
p-Nitroaniline	100-01-6

METHOD SUMMARY:

Gas chromatography may be used for the analysis for the above aromatic amines in air. A known volume of air is drawn through a tube containing silica gel. The gel is treated with ethanol, and an aliquot of the resulting solution is injected into a gas chromatograph equipped with a flame ionization detector (GC/FID).

INTERFERENCES:

High humidity severely decreases the capacity of the sampling apparatus.

QUALITY CONTROL:

A method blank and standards are analyzed with the samples.

EPA/TECHNICAL STATUS:

This is an operational method as set forth by the National Institute for Occupational Safety and Health (NIOSH) for use over the range of 0.01 to 14 milligrams per sample. Precision and accuracy information is furnished.

REFERENCE:

U.S. Department of Health, Education and Welfare, NIOSH Manual of Analytical Methods, Method P&CAM 168; Publication No. 75-121, U.S. DHEW: Cincinnati, 1974.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70-90 (list), , , plus sampling costs.

AROMATIC AMINES

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Benzidine	92-87-5
3,3'-Dichlorobenzidine	91-94-1
3,3'-Dimethoxybenzidine	119-90-4
4,4'-Methylenebis-(2-Chloroaniline)	101-14-4

METHOD SUMMARY:

Analysis of soil/sediment samples for the above aromatic amines can be performed by high-performance liquid chromatography with electrochemical detection (HPLC/EC) and pre-column concentration. A 1- to 10-gram soil sample is slurried in 100 to 1,000 milliliters water, and 20 milliliters of the resulting solution are passed through a preconcentration column. The concentrated sample is then flushed into the HPLC/EC and analyzed.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

A representative sample of the solids left after slurrying should be processed to ascertain that extraction has been complete.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information is furnished. The method can detect concentrations above approximately 1 nanogram per gram.

REFERENCE:

Rice J.; Kissinger, P. "Liquid Chromatography with Precolumn Sample Preconcentration and Electrochemical Detection: Determination of Aromatic Amines in Environmental Samples," Environ. Sci. Technol., 16(5), 1982, pp. 263-268.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $130-155 (list).

AROMATIC AMINES

Water Samples
Laboratory Method 1

Compound	CAS Number
Benzidine	92-87-5
3,3'-Dichlorobenzidine	91-94-1
3,3'-Dimethoxybenzidine	119-90-4
4,4'-Methylenebis-(2-Chloroaniline)	101-14-4

METHOD SUMMARY:

Analysis of water samples for the above aromatic amines can be performed by high-performance liquid chromatography with electrochemical detection (HPLC/EC). A 1- to 10-milliliter sample is filtered, concentrated on a preconcentration column, and analyzed by reversed-phase HPLC/EC.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

A representative sample of the residue trapped in sample filtration should be analyzed to ascertain that no analyte is adsorbed on the filtered material.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information is furnished. The method is sensitive to low nanogram-per-liter concentrations.

REFERENCE:

Rice, J. R.; Kissinger, P. T. "Liquid Chromatography with Precolumn Sample Preconcentration and Electrochemical Detection: Determination of Aromatic Amines in Environmental Samples," Environ. Sci. Technol., 16(5), 1982, pp. 263-268.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $130-155 (list).

AROMATIC AMINES

Water Samples
Laboratory Method 2

Compound	CAS Number
4-Aminopyridine	504-24-5
Aniline	62-53-3
p-Nitroaniline	100-01-6
5-Nitro-o-Toluidine	99-55-8
o-Toluidine Hydrochloride	636-21-5

METHOD SUMMARY:

Analysis of water samples for the above aromatic amines can be performed by diazotization and colorimetric measurement. A 35-milliliter water sample is acidified with hydrochloric acid, sodium nitrite solution is added, and the solution is allowed to stand for 5 minutes. A color reagent, either 8-amino-1-hydroxy-naphthalene-3,6-disulfoni acid or N-(1-naphthyl)ethylenediamine, is added to produce a cherry-red or purplish-red color, respectively, and the intensity of the color is measured spectrophotometrically.

INTERFERENCES:

No interferences are reported. All glassware, including absorption cells, should be cleaned frequently with hydrochloric acid.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

The method is not EPA-approved. No precision or accuracy information is furnished. The method is sensitive to levels above approximately 0.1 milligrams per liter in reagent-water samples.

REFERENCE:

Norwitz, G.; Keliher, P. N. "Spectrophotometric Determination of Aromatic Amines by the Diazotization-Coupling Technique with 8-Amino-1-hydroxynaphthalene-3,6-disulfonic acid and N-(1-Naphthyl)ethylenediamine as the Coupling Agents," Anal. Chem., 54(4), 1982, pp. 807-809.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $24-30 (list).

AROMATIC PRIMARY AMINES

Air Samples
Laboratory Method

Compound	CAS Number
Aniline	62-53-3
3,3'-Dichlorobenzidine	91-94-1
3,3'-Dimethoxybenzidine	119-90-4
4,4'-Methylenebis (2-Chloroaniline)	101-14-4
1-Naphthylamine	134-32-7
p-Nitroaniline	100-01-6

METHOD SUMMARY:

The above aromatic primary amines in air samples can be measured by gas chromatography (GC). A 1-liter air sample is drawn through a bubbler containing 0.1 M hydrochloric acid. The resulting solution is then diluted with more 0.1 M hydrochloric, if necessary. Disodium tetraborate, heptafluorobutyryl chloride, and cyclohexane are added. After shaking the mixture, an aliquot of the cyclohexane fraction is analyzed by GC.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

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

REFERENCE:

Meddle, D. W.; Smith, A. F. "Field Method for the Determination of Aromatic Primary Amines in Air. Part I. Generation of Standard Atmospheres of Amines," Analyst, 106, 1981, pp. 1082-1087, 1094.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $105-150 (list), plus sampling costs.

AROMATIC VOLATILE ORGANIC COMPOUNDS

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Benzene	71-43-2
Chlorobenzene	108-90-7
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
Ethylbenzene	100-41-4
Toluene	108-88-3
Xylene	1330-20-7
m-Xylene	108-38-3
o-Xylene	95-47-6
p-Xylene	106-42-3

METHOD SUMMARY:

Analysis for the above volatile aromatic organic compounds in soil/sediment samples can be accomplished by gas chromatography. The sample is dispersed in polyethylene glycol (PEG) or distilled-in-glass methanol to dissolve the volatile organic compounds. A portion of this solution is combined with water, purged with an inert gas onto a trap, and the trap back-flushed into a temperature-programmed gas chromatograph with a photoionization detector (GC/PID). Either internal or external standards are used for calibration.

INTERFERENCES:

Samples should be collected in glass containers, sealed, and refrigerated immediately. Contamination from the plumbing head of the trap and gas chromatograph from previous injections and diffusion of volatile compounds through the sample container seal can affect analytical results.

QUALITY CONTROL:

The plumbing head of the trap and gas chromatograph must be demonstrated to be free of interferences by analysis of a method blank each time a set of samples is processed or a change in reagents occurs. Field replicates, laboratory replicates, and fortified samples must be analyzed to demonstrate the precision and accuracy of the method. In cases of doubt concerning the identity of a peak in the chromatogram, confirmatory techniques, such as gas chromatography/mass spectrometry (GC/MS) or second-column confirmation, should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved for Resource Conservation and Recovery Act analyses (40 CFR, Part 261, Identification and Listing of Hazardous Wastes). Precision and accuracy information is furnished.

REFERENCE:

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 5030, 5020, 8020; SW-846, U.S. EPA: Washington, July 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $100-115 (list); GC/MS confirmation is approximately $150-200 (list); second-column confirmation is approximately $100-115 (list).

AROMATIC VOLATILE ORGANIC COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number
Benzene	71-43-2
Chlorobenzene	108-90-7
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
Ethylbenzene	100-41-4
Toluene	108-88-3
Xylene	1330-20-7
m-Xylene	108-38-3
o-Xylene	95-47-6
p-Xylene	106-42-3

METHOD SUMMARY:

Analysis of water samples for the above volatile aromatic organic compounds can be performed by gas chromatography. A water sample is purged with an inert gas onto a trap, and the trap is backflushed into a temperature-programmed gas chromatograph with a photoionization detector (GC/PID). Either internal or external standards are used for calibration.

INTERFERENCES:

Contamination from the plumbing head of the trap and the gas chromatograph and diffusion of volatile compounds through the sample container seal can affect analytical results.

QUALITY CONTROL:

Samples should be collected in glass containers, sealed and refrigerated immediately. The plumbing head of the trap and the gas chromatograph must be demonstrated to be free of interferences by the analysis of a method blank each time a set of samples is processed or a change in reagents occurs. Field replicates, laboratory replicates, and fortified samples must be analyzed to demonstrate the precision and accuracy of the method. In cases of doubt concerning the identity of a peak in the chromatogram, confirmatory techniques, such as gas chromatography/mass spectrometry (GC/MS) or second-column confirmation, should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved for Resource Conservation and Recovery Act (RCRA) analyses (40 CFR, Part 261, Identification and Listing of Hazardous Wastes). No precision or accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 5030, 8020; SW-846, U.S. EPA: Washington, July 1982.

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, J. E. Longbottom; J. J. Lichtenberg; Eds.; Method 602; EPA-600/4-82-057, U.S. EPA: Washington, July 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $100-115 (list); GC/MS confirmation is approximately $150-200 (list); second column confirmation is approximately $80 (list).

O-ARYL CARBAMATE PESTICIDES

Water Samples
Laboratory Method

Compound	CAS Number
Carbaryl	63-25-2
Mexacarbate	315-18-4

METHOD SUMMARY:

Analysis for the above O-aryl carbamate pesticides in water samples can be performed by a thin-layer chromatographic method. A 1-liter water sample is extracted with dichloromethane, and the extract is dried, concentrated, and cleaned up by Florisil® column chromatography. Fractions from the eluate are concentrated, portions are separated by thin-layer chromatography, and the carbamates are visualized by reaction with 2,6-dibromoquinone chlorimide. Semi-quantitative measurement is made by visual comparison of the color intensity with the color intensity of standards, and confirmation is established by observation of color changes with pH.

INTERFERENCES:

Interferences by phenols may be overcome by the use of a different solvent system.

QUALITY CONTROL:

Quality control charts should be used, and duplicate, spiked, and performance evaluation samples should be analyzed regularly.

EPA/TECHNICAL STATUS:

This method is EPA-approved for National Pollutant Discharge Elimination System (NPDES) analyses. Precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol, and Pesticides in Water and Wastewater, U.S. EPA: Washington, September 1978.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Supplement, "Selected Analytical Methods Approved and Cited by the U.S. EPA," APHA: Washington, 1981, pp. S60, S73.

COST INFORMATION:

Cost information has not been obtained.

BASE/NEUTRAL-EXTRACTABLE ORGANIC COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Acenaphthene	83-32-9	Dichloroethyl Ether	111-44-4
Acenaphthylene	208-96-8	Dieldrin	60-57-1
Aldrin	309-00-2	Diethyl Phthalate	84-66-2
Anthracene	120-12-7	Dimethyl Phthalate	131-11-3
Benzidine	92-87-5	2,4-Dinitrotoluene	121-14-2
Benzo[a]anthracene	56-55-3	2,6-Dinitrotoluene	606-20-2
Benzo[b]fluoranthene	205-99-2	Di-n-octyl Phthalate	117-84-0
Benzo[k]fluoranthene	207-08-9	1,2-Diphenylhydrazine	122-66-7
Benzo[a]pyrene	50-32-8	Endosulfan Sulfate	1031-07-8
Benzo[ghi]perylene	191-24-2	Endrin	
α-BHC	319-85-7	Endrin Aldehyde	7421-93-4
β-BHC	319-86-8	Fluoranthene	206-44-0
Bis(2-chloroethoxy)methane	111-91-1	Fluorene	86-73-7
Bis(2-chloroethyl)ether	111-44-4	Heptachlor	76-44-8
Bis(2-chloroisopropyl)ether	108-60-1	Heptachlor Epoxide	1024-57-3
4-Bromophenyl Phenyl Ether	101-55-3	Hexachlorobenzene	118-74-1
Butyl Benzyl Phthalate	85-68-7	Hexachlorobutadiene	87-68-3
Chlordane	57-74-9	Hexachloroethane	67-72-1
2-Chloronaphthalene	91-58-7	Indeno[1,2,3-cd]pyrene	193-39-5
4-Chlorophenyl Phenyl Ether	7005-72-3	Isophorone	78-59-1
Chrysene	218-01-9	Naphthalene	91-20-3
4,4'-DDD	72-54-8	p-Nitroaniline	100-01-6
DDE		Nitrobenzene	98-95-3
4,4'-DDT	50-29-3	N-Nitrosodimethylamine	62-75-9
Dibenz[a,h]anthracene	53-70-3	N-Nitrosodiphenylamine	86-30-6
Di-n-butyl Phthalate	84-74-2	N-Nitrosodi-n-propylamine	621-64-7
1,2-Dichlorobenzene	95-50-1	Phenanthrene	85-01-8
1,3-Dichlorobenzene	541-73-1	Polychlorinated Biphenyls	110-86-1
1,4-Dichlorobenzene	106-46-7	Pyrene	129-00-0
3,3'-Dichlorobenzidine	91-94-1	Toxaphene	8001-35-2
		1,2,4-Trichlorobenzene	120-82-1

METHOD SUMMARY:

Analysis for the above organic compounds in water samples can be performed by extraction followed by gas chromatography/mass spectrometry (GC/MS). A 1-milliliter sample is transferred into a vial and the pH is adjusted to 11. The sample is extracted with methylene chloride and the methylene chloride extract is screened for organic compounds by gas chromatography with a flame ionization detector (GC/FID), in order to determine whether dilution will be required for gas chromatographic/mass spectrometric (GC/MS) analysis. This procedure is designed to screen at 100 milligrams per liter, in the absence of interfering compounds. After dilution (as appropriate), samples that give positive results in screening are analyzed by GC/MS. For samples that give negative results in the GC/FID screening, a 1- to 2-liter sample is extracted

at pH 11 with methylene chloride. The extract is dried and concentrated to approximately 1 milliliter. For GC/MS analysis using a fused-silica capillary column (FSCC), both the pH 11 extract and a pH 2 extract may be combined for simultaneous chromatographic analysis of both acid and base/neutral extractable organic compounds. The concentrate is analyzed using GC/MS with multiple internal standards, and quantitative determinations are based on the response factor of the nearest internal standard.

INTERFERENCES:

If emulsions form during the extraction step, a continuous-extraction technique should be used.

QUALITY CONTROL:

Instrument performance is checked by minimum detection limit criteria and ongoing monitoring of relative response factors, relative retention times, internal standard response factors, stability of a 3-point calibration curve, and performance of the gas chromatographic system. Specifications for instrument tuning are given. Analytical performance is monitored by use of surrogates, duplicate analyses, and fortified samples, and criteria for surrogate recoveries are listed. The analytical system must be demonstrated to be free of interferences before analysis of any samples.

EPA/TECHNICAL STATUS:

This method is currently in use in the CERCLA program and is EPA-approved for use in Clean Water Act analyses and analyses under the Resource Conservation and Recovery Act (RCRA). Precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, GC Screen, GC/MS Analysis of Organic Compounds, Invitation for Bid (Solicitation Number WA 82-A154); Exhibit D, U.S. EPA: Washington, July 16, 1982.

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis for Municipal and Industrial Wastewater, J. E. Longbottom; J. J. Lichtenberg; Eds.; Method 625; EPA-600/4-82-057, U.S. EPA: Cincinnati, 1982.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 8250, 8270; SW-846, U.S. EPA: Washington, 1982.

Sauter, A. D.; Betowski, L. D.; Smith, T. R.; Strickler, V. A.; Beimer, R. G.; Colby, B. N.; Wilkinson, J. E. "Fused Silica Capillary Column GC/MS for the Analysis of Priority Pollutants," J. High Resolut. Chromatogr. Chromatogr. Comm., 4, 1981, pp. 366-384.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Analysis of Methylene Chloride Extracts of Aqueous Samples for Base/Neutral Compounds," pp. III-112 to III-117; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Geological Survey, Methods for the Determination of Organic Substances in Water and Fluvial Sediments, Book 5, "Laboratory Analysis," Chapter A3; USGS: Denver, Open-File Report 82-1004, 1983.

COST INFORMATION:

Cost per sample for the GC screen is approximately $70-90 (list), $60 (bid); the GC/MS analysis is approximately $275-415 (list), $230 (bid).

BASIC ORGANIC COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number
Aniline	62-53-3
p-Chloroaniline	106-47-8
1-Naphthylamine	134-32-7
2-Picoline	109-06-8
Pyridine	110-86-1
Quinoline	91-22-5

METHOD SUMMARY:

The above basic organic compounds in water samples can be determined by gas chromatography with flame ionization detection (GC/FID). The sample is passed through a cation-exchange resin to collect the organic bases, and the resin is washed to remove the sorbed neutral and acidic compounds. The eluate is concentrated by evaporation of solvent. An aliquot is injected into a GC equipped with both a packed column and a fused-silica capillary column.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished.

REFERENCE:

Kaczvinsky, J. R., Jr.; Saitoh, K.; Fritz, J. S. "Cation-Exchange Concentration of Basic Organic Compounds from Aqueous Solution," Anal. Chem. 55(8), 1983, pp. 1210-1215.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $105-120.

BENZAL CHLORIDE, BENZOTRICHLORIDE, AND BENZYL CHLORIDE

Air Samples
Laboratory Method

Compound	CAS Number
Benzal Chloride	98-87-3
Benzotrichloride	98-07-7
Benzyl Chloride	100-44-7

METHOD SUMMARY:

Benzal chloride, benzotrichloride, and benzyl chloride in air samples can be measured by gas chromatography with flame ionization detection (GC/FID). The air is passed through a tube packed with Tenax-GC to adsorb the analytes, which are desorbed with carbon tetrachloride. An aliquot is injected into the GC/FID.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

The method is not EPA-approved. Precision or accuracy information is not furnished.

REFERENCE:

Matsushita, H.; Kanno, S. "A Gas Chromatographic Determination of Benzotrichloride and Related Compounds in the Work Environment," Ind. Health, 17, 1979, pp. 199-206.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70-90 (list), plus sampling costs.

BENZIDINE AND CONGENERS

Air Samples
Laboratory Method

Compound	CAS Number
Benzidine	92-87-5
3,3'-Dichlorobenzidine	75-27-4
3,3'-Dimethoxybenzidine	119-90-4
3,3'-Dimethylbenzidine	119-93-7

METHOD SUMMARY:

Benzidine and the above congeners in particulate air samples can be measured by high-performance liquid chromatography with electrochemical detection (HPLC/EC). Samples are collected on glass-fiber filters using a high-volume air sampler. The filter is then extracted twice with chloroform. After combining the extracts, the amines in the resulting solution are extracted into 1M sulfuric acid, the acid solution is neutralized, and the amines are re-extracted into chloroform. After a series of dilutions with methanol and concentration steps, the solution is buffered and then analyzed by HPLC/EC.

INTERFERENCES:

3,3'-Dimethylbenzidine and 3,3'-dimethoxybenzidine are not resolved chromatographically.

QUALITY CONTROL:

Blank filters, spiked filters, and blind duplicate samples should be analyzed with the rest of the samples.

EPA/TECHNICAL STATUS:

The method is not EPA-approved. Precision and accuracy information is furnished. The detection limit is approximately 1 nanogram per cubic meter.

REFERENCE:

Riggin, R. M.; Howard, C. C.; Scott, D. R.; Hedgecoke, R. L. "Determination of Benzidine Related Congeners, and Pigments in Atmospheric Particulate Matter," J. Chromatogr. Sci., 21, 1983, pp. 321-325.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $15 (list), plus sampling costs.

BENZIDINE AND CONGENERS

Water Samples
Laboratory Method

Compound	CAS Number
Benzidine	92-87-5
3,3'-Dimethoxybenzidine	119-90-4
3,3'-Dimethylbenzidine	119-93-7

METHOD SUMMARY:

Benzidine and the above congeners in water samples can be measured by spectrophotofluorimetry (SPF). Wastewater samples are extracted with benzene, the extract is cleaned up on an alumina column, the eluate is evaporated just to dryness, and the residue is dissolved in methanol. The free amine in the methanol solution is determined by SPF.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information are furnished. The limit of detection is approximately 10 micrograms per liter.

REFERENCE:

Bowman, M. C.; King. J. R.; Holder, C. L. "Benzidine and Congeners: Analytical Chemical Properties and Trace Analysis in Five Substrates," Intern. J. Environ. Anal. Chem., 4, 1976, pp. 205-223.

COST INFORMATION:

Cost information has not been obtained.

CARBAMATE AND UREA PESTICIDES

Water Samples
Laboratory Method

Compound	CAS Number
Carbaryl	63-25-2
Carbofuran	1563-66-2
Diuron	330-54-1
Methomyl	16752-77-5
Mexacarbate	315-18-4

METHOD SUMMARY:

High-performance liquid chromatography (HPLC) can be used to determine the above pesticides in water samples. A 1-liter sample is solvent-extracted with methylene chloride and the extract is dried, concentrated, and analyzed by HPLC with UV detection.

INTERFERENCES:

A Florisil® column cleanup procedure may be used when interferences are present.

QUALITY CONTROL:

Laboratory reagent blanks must be analyzed to determine that reagents and apparatus are free from interferences. Standard solutions must be checked frequently for signs of degradation. The accuracy and precision of measurements must be validated before analysis. At least 10 percent of the analyses must be spiked samples, and duplicate samples should be analyzed periodically.

EPA/TECHNICAL STATUS:

This method is EPA-approved. Precision and accuracy information is furnished. The method is sensitive to levels of analyte above two micrograms per liter (USGS).

REFERENCES:

Pressley, T.; Longbottom, J. The Determination of Carbamate and Urea Pesticides in Industrial and Municipal Wastewater, Method 632; U.S. EPA: Cincinnati, January 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Determination of Carbamate and Urea Pesticides in Industrial and Municipal Wastewater," pp. III-268 to III-275; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Geological Survey, Methods for the Determination of Organic Substances in Water and Fluvial Sediments, Book 5, "Laboratory Analysis," Chapter A3; USGS: Denver, Open-File Report 82-1004, 1983.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $155 to 180 (list).

CARBAMATE PESTICIDES

Air Samples
Laboratory Method

Compound	CAS Number
Carbaryl	63-25-2
Carbofuran	1563-66-2

METHOD SUMMARY:

Gas chromatography can be used for the analysis of air samples for the above carbamate pesticides. A known volume of air is passed through a glass-fiber, polyurethane foam, or composite filter, the sampling medium is Soxhlet-extracted with hexane/diethyl ether, and the resulting solution is analyzed with a gas chromatograph with a nitrogen-selective eletrolytic conductivity detector or by chemical derivatization with alpha-bromo-2,3,4,5,6-pentafluorotoluene and analysis with a gas chromatograph with an electron-capture detector (GC/ECD).

INTERFERENCES:

No interferences are listed.

QUALITY CONTROL:

Control limits for recovery must be established and recovery monitored. Results for sets of samples processed with a fortified sample that gives results that do not fall within control limits must be labeled as suspect.

EPA/TECHNICAL STATUS:

This method is not EPA-approved for use in any regulatory program. Precision and accuracy information is furnished.

REFERENCES:

Sherma, J.; Beroza, M. Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples, Sections 8A and 8B; EPA-600/8-80-038, U.S. Environmental Protection Agency: Research Triangle Park, North Carolina, 1980.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Determination of Carbamate Pesticides in Air," pp. III-293 to III-299; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $125-145 (list), plus sampling costs.

CARBAMATE PESTICIDES

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Carbofuran	1563-66-2
Mexacarbate	315-18-4

METHOD SUMMARY:

Analysis of soil/sediment samples for the above carbamate pesticides can be performed by gas chromatography with electrolytic conductivity detection (GC/ECD). A 50-gram sample is extracted with an acetone/aqueous sodium chloride/aqueous sodium hydrogen carbonate solution and the extract is partitioned with benzene. The solution is concentrated, cleaned up on a deactivated Florisil column and the pesticides in the column eluate are determined by GC/ECD.

INTERFERENCES:

No specific interferences are listed.

QUALITY CONTROL:

Recovery should be determined at 0.1 and/or 1.0 milligram-per-kilogram fortification levels.

EPA/TECHNICAL STATUS:

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

REFERENCE:

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Determination of Carbamate Pesticides in Soil," pp. III-276 to III-280; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $190 (list).

CARBOXYLIC ACIDS

Water Samples
Laboratory Method

Compound	CAS Number
Acetic Acid	64-19-7
Acrylic Acid	79-10-7
Fumaric Acid	110-17-8
Maleic Acid	110-16-7

METHOD SUMMARY:

Water samples can be analyzed for the above carboxylic acids by ion exchange chromatography. The sample is analyzed by monitoring the eluates with a UV detector at 210 nm, without pretreatment other than degassing.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

The method is not EPA-approved. No precison or accuracy information is furnished.

REFERENCE:

Richards, M. "Separation of Mono- and Dicarboxylic Acids by Liquid Chromatography," J. Chromatogr., 115, 1975, pp. 259-261.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70 (list).

CHLORINATED AND OTHER ORGANIC CONTAMINANTS

Water Samples
Laboratory Method

Compound	CAS Number
Bis(2-Chloroethyl)ether	111-44-4
Chlorodibromomethane	124-48-1
1,4-Dichlorobenzene	106-46-7
Hexachlorobenzene	118-74-1
Hexachlorobutadiene	87-68-3
Pentachlorobenzene	608-93-5
Styrene	100-42-5
1,2,4-Trichlorobenzene	120-82-1

METHOD SUMMARY:

The above chlorinated organic contaminants in water samples can be measured by gas chromatography/mass spectrometry (GC/MS). A one-liter sample is passed through a closed-loop stripping system to purge the organics, which are collected in carbon disulfide. A 2-microliter aliquot of the extract is injected into a gas chromatograph equipped with a fused-silica capillary column. Quantification is based on the ion current area of a single selected mass ion of the mass spectrum.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

Procedural blanks are analyzed and results are corrected for contaminants in the blanks.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information is furnished. This method is suitable for concentrations of analyte at the nanogram-per-liter level.

REFERENCE:

Coleman, W. E.; Munch, J. W.; Slater, R. W.; Melton, R. G.; Kopfler, F. C. "Optimization of Purging Efficiency and Quantification of Organic Contaminants from Water Using a 1-liter Closed-Loop-Stripping Apparatus and Computerized Capillary Column GC/MS," Environ. Sci. Technol., 17(10), 1983, pp. 571-576.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $250-325 (list).

CHLORINATED BENZENES

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Hexachlorobenzene	118-74-1
Pentachloronitrobenzene	82-68-8

METHOD SUMMARY:

Hexachlorobenzene and pentachloronitrobenzene in soil samples can be determined by gas chromatography with electron capture detection (GC/ECD). The sample is mixed with water and shaken with toluene and isopropanol. The solvent layer is separated from water and is washed twice with an aqueous sodium sulfate solution, centrifuged, shaken with Nuchar-Attaclay, and filtered. The filtrate is diluted, if necessary, and analyzed by GC/ECD. Compound identification can be verified by gas chromatography/mass spectrometry (GC/MS).

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy informtion is furnished for the compounds listed.

REFERENCE:

de Vos, R. H.; ten Noever de Brauw, M. C.; Olthof, P. D. A. "Residues of Pentachloronitrobenzene and Related Compounds in Greenhouse Soils," Bull. Environ. Contam. Toxicol., 11, 1974, pp. 567-571.

COST INFORMATION:

Cost per sample for the GC/ECD analysis is approximately $120-165 (list).

CHLORINATED BENZENES

Air Samples
Laboratory Method

Compound	CAS Number
Pentachlorobenzene	608-93-5
Pentachloronitrobenzene	82-68-8

METHOD SUMMARY:

Analysis for the above airborne chlorinated benzenes can be performed by passage of 1000 liters of air through a polyurethane-foam sampling tube, followed by Soxhlet extraction of the foam and analysis of the extract by gas chromatography with electron capture detection (GC/ECD).

INTERFERENCES:

Phthalate esters and polychlorinated naphthalenes can interfere. Use of non-TFE plastics must be avoided.

QUALITY CONTROL:

When concentration levels are high enough, confirmation of peak identification is performed by gas chromatography/mass spectrometry (GC/MS). Solvents, reagents, glassware, and polyurethane plugs must be shown to be free of interfering artifacts. A field blank must be analyzed with each set of samples, and both a quality control standard and a method blank should be analyzed with each set of 5 to 15 samples.

EPA/TECHNICAL STATUS:

This method has been evaluated by the Environmental Protection Agency, but is not approved for any specific regulatory program. No precision or accuracy information is furnished. This method is applicable to a range from 1 nanogram per cubic meter to 1000 nanograms per cubic meter.

REFERENCE:

U.S. Environmental Protection Agency, Guidelines for Air Monitoring at Hazardous Waste Sites, Assignment 26, Appendix B; Contract 68-02-3168; U.S. EPA: Research Triangle Park, North Carolina, May 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $115 (list), plus sampling costs.

CHLORINATED ETHERS AND OTHER COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Bis(2-chloroethyl)ether	111-44-4	Isophorone	78-59-1
Bromoform	75-25-2	Naphthalene	91-20-3
Chlorobenzene	108-90-7	N-Nitrosodimethylamine	62-75-9
Chlorodibromomethane	124-48-1	N-Nitroso-di-*n*-propylamine	621-64-7
2-Chloroethyl Vinyl Ether	110-75-8	Phenol	108-95-2
Chloroform	67-66-3	1,1,2,2-Tetrachloroethane	79-34-5
2-Chlorophenol	95-57-8	Tetrachloroethylene	127-18-4
1,2-Dichlorobenzene	95-50-1	Toluene	108-88-3
1,4-Dichlorobenzene	106-46-7	1,2,4-Trichlorobenzene	120-82-1
1,1-Dichloroethane	75-34-3	1,1,1-Trichloroethane	71-55-6
1,2-Dichloroethane	107-06-2	1,1,2-Trichloroethane	79-00-5
1,3-Dichloropropene	542-75-6	Trichloroethylene	79-01-6
Ethylbenzene	100-41-4	*m*-Xylene	108-38-3
Hexachlorobutadiene	87-68-3	*o*-Xylene	95-47-6
Hexachloroethane	67-72-1	*p*-Xylene	106-42-3

METHOD SUMMARY:

Gas chromatography may be used to analyze water samples for the above chlorinated ethers and other compounds. A 120-milliliter water sample is extracted with methylene chloride. The extract is condensed, the solvent is exchanged for hexane, and the sample is analyzed by gas chromatography with electron capture detection (GC/ECD), flame ionization detection (GC/FID), or Hall electrolytic conductivity detection (HECD).

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

Triplicate samples should be analyzed. Standard solutions should be extracted and analyzed. When the identification of a peak is in doubt, GC/MS should be used for confirmation.

EPA/TECHNICAL STATUS:

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

REFERENCE:

Otson, R.; Williams D. T. "Evaluation of a Liquid-Liquid Extraction Technique for Water Pollutants," J. Chromatogr., 212, 1981, pp. 187-197.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $110-215 (list), plus sampling costs.

CHLORINATED HYDROCARBONS

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Benzotrichloride	98-07-7
Benzyl Chloride	100-44-7
α-BHC	319-86-8
β-BHC	319-84-5
γ-BHC	319-85-7
2-Chloronaphthalene	91-58-7
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
Hexachlorobenzene	118-74-1
Hexachlorobutadiene	87-68-3
Hexachlorocyclopentadiene	77-47-4
Hexachloroethane	67-72-1
Lindane	59-89-9
1,2,4,5-Tetrachlorobenzene	95-94-3
1,2,4-Trichlorobenzene	120-82-1

METHOD SUMMARY:

Analysis for the above chlorinated hydrocarbons in soil/sediment samples may be performed by extraction and gas chromatography. A soil/sediment sample is extracted at neutral pH with dichloromethane, using either Soxhlet extraction or sonication, and the extract is dried, solvent-exchanged into hexane, concentrated, and analyzed by gas chromatography with electron capture detection (GC/ECD) using either internal or external standards.

INTERFERENCES:

Contact with plastics must be avoided during collection, storage, and processing of samples. Florisil® column cleanup may be required to eliminate interferences.

QUALITY CONTROL:

An aliquot of each sample must be spiked with standards to determine recovery and limits of detection. The analytical system must be demonstrated to be free of interferences by analysis of a method blank with each set of samples or change in reagents. Field and laboratory replicates and duplicate samples should be analyzed to ascertain the precision and accuracy of the method. In cases where the identification of a peak in the chromatogram is in question, confirmatory techniques, such as gas chromatography/mass spectrometry (GC/MS) or second-column confirmation, should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved for Resource Conservation and Recovery Act analyses (RCRA). No precision or accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater; J. J. Longbottom; J. J. Lichtenberg; Eds.; Method 612; EPA-600/4-82-059, U.S. EPA: Cincinnati, 1982.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3540, 3550, 8120; SW-846, U.S. EPA: Washington, 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $135-150 (list); the cost of a Florisil® column cleanup is approximately $35 (list).

CHLORINATED HYDROCARBONS

Water Samples
Laboratory Method

Compound	CAS Number
Benzotrichloride	98-07-7
Benzyl Chloride	100-44-7
α-BHC	319-86-8
β-BHC	319-84-5
γ-BHC	319-85-7
2-Chloronaphthalene	91-58-7
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
Hexachlorobenzene	118-74-1
Hexachlorobutadiene	87-68-3
Hexachlorocyclopentadiene	77-47-4
Hexachloroethane	67-72-1
Lindane	59-89-9
1,2,4,5-Tetrachlorobenzene	95-94-3
1,2,4-Trichlorobenzene	120-82-1

METHOD SUMMARY:

Analysis for the above chlorinated hydrocarbons in water can be performed by extraction and gas chromatography. A sample of water is extracted with dichloromethane and the extract is dried, solvent-exchanged into hexane, concentrated, and analyzed by gas chromatography with electron capture detection (GC/ECD), using either internal or external standards.

INTERFERENCES:

Contact with plastics must be avoided in collection, storage, and processing of samples. Florisil®-column cleanup may be required to eliminate interferences.

QUALITY CONTROL:

An aliquot of each sample must be spiked with standards to determine recovery and limits of detection. The analytical system must be demonstrated to be free of interferences by the analysis of a method-blank with each set of samples or change in reagents. Field and laboratory replicates and duplicate samples should be analyzed to ascertain the precision and accuracy of the method. In cases where identification of a peak in the chromatogram is in question, confirmatory techniques, such as gas chromatography/mass spectrometry (GC/MS) or second-column confirmation, should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved for Resource Conservation and Recovery Act analyses (40 CFR, Part 261, Identification and Listing of Hazardous Wastes) and for analyses for the Clean Water Act. Precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, J. J. Longbottom; J. J. Lichtenburg; Eds., Method 612; EPA-600/4-82-059, U.S. EPA: Cincinnati, 1982.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3510, 3520, 8120; SW-846, U.S. EPA: Washington, July 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $120-170 (list); GC/MS confirmation is approximately $150-200 (list); second-column confirmation is approximately $80 (list).

CHLORINATED PHENOXY ACID HERBICIDES

Soil/Sediment Samples
Laboratory Method 1

Compound	CAS Number
2,4-D Acid	94-75-7
2,4-D Esters	94-11-1
	94-79-1
	94-80-4
	1320-18-9
	1928-38-7
	1928-61-6
	1929-73-3
	2971-38-2
	25168-26-7
	53467-11-1
Dicamba	1918-00-9
Dinoseb	88-85-7
2,4,5-T	93-76-5
2,4,5-T Amines	2008-46-0
2,4,5-T Esters	93-79-8
	2545-59-7
	61792-07-2
	1928-47-8
	25168-15-4
2,4,5-TP Acid	93-72-1
2,4,5-TP Acid Esters	32534-95-5
2,4,5-T Salts	13560-99-1

METHOD SUMMARY:

Analysis for the above chlorinated herbicides in soil/sediment samples may be performed by extraction and gas chromatography. A soil/sediment sample equivalent to a 50-gram dry mass is acidified and extracted with acetone followed by diethyl ether. The combined extracts are heated with aqueous potassium hydroxide, and the aqueous solution is washed with diethyl ether. The aqueous solution is then acidified and extracted with diethyl ether. The diethyl ether extract is dried, concentrated, and treated with diazomethane, and the solution of methyl esters is analyzed by gas chromatography with an electron capture detector (GC/ECD), using either internal or external standards for quantification.

INTERFERENCES:

Samples must be collected in glass containers, and all contact with plastics should be avoided during sample collection, storage, and processing. Organic acids and phenols can interfere, and all glassware and glass wool should be acid-washed, and all sodium sulfate should be acidified before use.

QUALITY CONTROL:

The analytical system must be demonstrated to be free of interferences by analysis of a method blank with each set of samples and/or change in reagents. Laboratory and field replicates should be processed to ascertain the precision of sampling and analysis, and fortified samples should be analyzed to validate the accuracy of the analysis. Where doubt exists concerning the identification of a peak in the chromatogram, confirmatory techniques, such as gas chromatography/mass spectrometry (GC/MS) and second-column confirmation, should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved for Resource Conservation and Recovery Act sample analyses (40 CFR, Part 261, Identification and Listing of Hazardous Wastes). No precision or accuracy information is furnished.

REFERENCES:

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, D3478-79; ASTM: Philadelphia, 1983, pp. 151-157.

American Society for Testing and Materials, ASTM Standards on Chromatography, D-3478-79; ASTM: Philadelphia, 1981, pp. 440-447.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Method 8150; SW-846, U.S. EPA: Washington, July 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $155-230 (list).

CHLORINATED PHENOXY ACID HERBICIDES

Soil/Sediment Samples
Laboratory Method 2

Compound	CAS Number
2,4-D Acid	94-75-7
2,4-D Esters	94-11-1
	94-79-1
	94-80-4
	1320-18-9
	1928-38-7
	1928-61-6
	1929-73-3
	2971-38-2
	25168-26-7
	53467-11-1
Dicamba	1918-00-9
Dinoseb	88-85-7
2,4,5-T	93-76-5
2,4,5-T Amines	2008-46-0
2,4,5-T Esters	93-79-8
	2545-59-7
	61792-07-2
	1928-47-8
	25168-15-4
2,4,5-TP Acid	93-72-1
2,4,5-TP Acid Esters	32534-95-5
2,4,5-T Salts	13560-99-1

METHOD SUMMARY:

Analysis of soil/sediment samples for the above chlorinated phenoxy acid herbicides can be performed by extraction, derivatization, and analysis by gas chromatography with electron capture detection (GC/ECD). A 25-gram sample is acidified and extracted with acetone/hexane, the extract is dried, solvent-exchanged into benzene, treated with boron trifluoride/methanol, and cleaned up by silica column chromatography. The resulting solution is analyzed by GC/ECD.

INTERFERENCES:

No interferences are listed.

QUALITY CONTROL:

No quality control procedures are provided.

EPA/TECHNICAL STATUS:

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

REFERENCES:

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Analysis of Sediment Samples for Chlorinated Phenoxy Acid Acid Herbicides by Acetone-Hexane Extraction," pp. III-318 to III-321; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Geological Survey, Methods for the Determination of Organic Substances in Water and Fluvial Sediments, Book 5, "Laboratory Analysis," Chapter A3; USGS: Denver , Open-File Report, 82-1004, 1983.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $170-200 (list).

CHLORINATED PHENOXY ACID HERBICIDES

Soil/Sediment Samples
Laboratory Method 3

Compound	CAS Number
2,4-D Acid	94-75-7
2,4-D Esters	94-11-1
	94-79-1
	94-80-4
	1320-18-9
	1928-38-7
	1928-61-6
	1929-73-3
	2971-38-2
	25168-26-7
	53467-11-1
Dicamba	1918-00-9
Dinoseb	88-85-7
2,4,5-T	93-76-5
2,4,5-T Amines	2008-46-0
2,4,5-T Esters	93-79-8
	2545-59-7
	61792-07-2
	1928-47-8
	25168-15-4
2,4,5-TP Acid	93-72-1
2,4,5-TP Acid Esters	32534-95-5
2,4,5-T Salts	13560-99-1

METHOD SUMMARY:

Analysis of soil/sediment samples for the above chlorinated phenoxy acid herbicides can be performed by high-performance liquid chromatography (HPLC). A 20-gram sample is acidified and extracted with dichloromethane, the dichloromethane is evaporated, and the residue is hydrolyzed with methanolic potassium hydroxide. The resulting solution is extracted with methylene chloride to remove organic interferences, the solution is acidified, the solvent is evaporated, the residue is dissolved in methanol, and the resulting solution is cleaned up by reversed-phase column chromtography. The resulting solution is then analyzed by high performance liquid chromatography (HPLC) with UV detection at 285 nanometers.

INTERFERENCES:

No interferences are listed.

QUALITY CONTROL:

No quality control procedures are provided.

EPA/TECHNICAL STATUS:

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

REFERENCE:

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Analysis of Sediment Samples for Chlorinated Phenoxy Acid Acid Herbicides by Acetone-Hexane Extraction," pp. III-318 to III-321; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $210 (list).

CHLORINATED PHENOXY ACID HERBICIDES

Water Samples
Laboratory Method 1

Compound	CAS Number
2,4-D Acid	94-75-7
2,4-D Esters	94-11-1
	94-79-1
	94-80-4
	1320-18-9
	1928-38-7
	1928-61-6
	1929-73-3
	2971-38-2
	25168-26-7
	53467-11-1
Dicamba	1918-00-9
Dinoseb	88-85-7
2,4,5-T	93-76-5
2,4,5-T Amines	2008-46-0
2,4,5-T Esters	93-79-8
	2545-59-7
	61792-07-2
	1928-47-8
	25168-15-4
2,4,5-TP Acid	93-72-1
2,4,5-TP Acid Esters	32534-95-5
2,4,5-T Salts	13560-99-1

METHOD SUMMARY:

Analysis of water samples for the above chlorinated herbicides can be performed by extraction and chromatographic analysis. A water sample is extracted at pH 2 with diethyl ether, the extract is heated with aqueous potassium hydroxide, and the aqueous solution is washed with diethyl ether, acidified, and extracted with diethyl ether. The diethyl ether extract is dried, concentrated, and treated with diazomethane. The resulting solution of methyl esters is analyzed by gas chromatography with an electron capture detector (GC/ECD), using either internal or external standards for quantification.

INTERFERENCES:

Grab samples must be collected in glass containers, and all contact with plastics should be avoided in sample collection, storage, and processing. Organic acids and phenols can interfere, and all glassware and glass wool should be acid-washed and all sodium sulfate acidified before use.

QUALITY CONTROL:

The analytical system must be demonstrated to be free of interferences by analysis of a method blank with each set of samples and/or change in reagents. Laboratory and field replicates should be processed to ascertain the precision of sampling and analysis, and fortified samples should be analyzed to validate the accuracy of the analysis. Where doubt exists concerning the identification of a peak in the chromatogram, confirmatory techniques, such as gas chromatography/mass spectrometry (GC/MS) and second-column confirmation, should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved for Resource Conservation and Recovery Act (RCRA) sample analyses. Precision and accuracy information is furnished. Detection limits (ASTM, 1983) are 20 nanograms per liter for 2,4-D acid and 5 nanograms per liter for 2,4,5-T and 2,4,5-TP acid.

REFERENCES:

American Society for Testing and Materials, ASTM Standards on Chromatography, D-3478-79; ASTM: Philadelphia, 1981, pp. 440-447.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, ASTM: Philadelphia, 1983, pp. 151-157.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Method 8150; SW-846, U.S. EPA: Washington, July 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $155-230 (list).

CHLORINATED PHENOXY ACID HERBICIDES

Water Samples
Laboratory Method 2

Compound	CAS Number
2,4-D Acid	94-75-7
2,4-D Esters	94-11-1
	94-79-1
	94-80-4
	1320-18-9
	1928-38-7
	1928-61-6
	1929-73-3
	2971-38-2
	25168-26-7
	53467-11-1
Dicamba	1918-00-9
Dinoseb	88-85-7
2,4,5-T	93-76-5
2,4,5-T Amines	2008-46-0
2,4,5-T Esters	93-79-8
	2545-59-7
	61792-07-2
	1928-47-8
	25168-15-4
2,4,5-TP Acid	93-72-1
2,4,5-TP Acid Esters	32534-95-5
2,4,5-T Salts	13560-99-1

METHOD SUMMARY:

Analysis of water samples for the above chlorinated phenoxy acid herbicides can be performed by gas chromatography. A 1-liter sample is acidified and extracted with chloroform; the extract is dried, concentrated, solvent-exchanged into methanol, and the herbicide residues are converted to methyl esters with a boron trifluoride/methanol reagent. The methyl esters are extracted into benzene, the extract is cleaned up with a Florisil column, and the resulting solution is analyzed by gas chromatography with electron capture detection (GC/ECD).

INTERFERENCES:

No interferences are listed.

QUALITY CONTROL:

No quality control procedures are provided.

EPA/TECHNICAL STATUS:

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

REFERENCES:

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Analysis of Sediment Samples for Chlorinated Phenoxy Acid Acid Herbicides by Acetone-Hexane Extraction," pp. III-318 to III-321; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Geological Survey, Methods for the Determination of Organic Substances in Water and Fluvial Sediments, Book 5, "Laboratory Analysis," Chapter A3; USGS: Denver, Open-File Report 82-1004, 1983.

Environment Canada, Analytical Methods Manual, Inland Waters Directorate, Water Quality Branch, Ottowa, Ontario, Canada, 1973.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $170-200 (list).

CHLORINATED PESTICIDES

Air Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Aldicarb	116-06-3	α-Endosulfan	959-98-8
Aldrin	309-00-2	β-Endosulfan	33213-65-9
α-BHC	319-84-6	Endrin	72-20-8
β-BHC	319-85-7	Endrin Aldehyde	7421-93-4
γ-BHC	319-86-8	Heptachlor	76-44-8
Carbaryl	63-25-2	Heptachlor Epoxide	1024-57-3
Carbofuran	1563-66-2	Hexachlorobenzene	118-74-1
Chlordane	57-74-9	Lindane	58-89-9
DDE	72-55-9	Malathion	121-75-5
DDT	50-29-3	Methomyl	16752-77-5
Diazinon	333-41-5	Methyl Parathion	298-00-0
Dieldrin	60-57-1	Parathion	56-38-2
Endosulfan	115-29-7	Toxaphene	8001-35-2

METHOD SUMMARY:

Analysis of air samples for the above pesticides can be performed by gas chromatography. The pesticides are collected on polyurethane foam by sampling air with a calibrated sampling device; the polyurethane foam is Soxhlet-extracted and the extract is cleaned up, concentrated, and analyzed by gas chromatography. Organochlorine pesticides are measured by gas chromatography with electron-capture detection (GC/ECD), organophosphorus pesticides by gas chromatography with flame photometric detection (GC/FPD), and carbamate pesticides by either gas chromatography with Hall electrolytic conductivity detection (GC/HECD) in the N-mode, or derivatization and GC/ECD. Confirmation of peak identification can be performed by gas chromatography/mass spectrometry (GC/MS).

INTERFERENCES:

Extracts to be analyzed for organochlorine pesticides should be cleaned up by alumina column chromatography; the presence of polychlorinated biphenyls (PCBs) may require cleanup by silicic acid column chromatography. Analysis for organophosphorus pesticides must be performed without column chromatographic cleanup, and extracts to be analyzed for carbamate pesticides should be cleaned up by silica-gel column chromatography.

QUALITY CONTROL:

The collection efficiency and desorption efficiency of the sampling device must be demonstrated by multiple tests before sampling begins. The sampling system must be calibrated before use.

EPA/TECHNICAL STATUS:

This method is not EPA-approved, but has undergone EPA evaluation. No precision or accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples; EPA-600/8-80-038, U.S. EPA: Research Triangle Park, North Carolina, 1980.

U.S. Environmental Protection Agency, Guidelines for Air Monitoring at Hazardous Waste Sites, Assignment 26, Appendix B; Contract 68-02-3168; U.S. EPA: Research Triangle Park, North Carolina, May 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $135-220 (list), plus sampling costs.

CHLORINATED PHENOLS

Air Samples
Laboratory Method

Compound	CAS Number
2,4-Dichlorophenol	120-83-2
Pentachlorophenol	87-86-5
2,4,5-Trichlorophenol	95-95-4
2,4,6-Trichlorophenol	88-06-2

METHOD SUMMARY:

Analysis of an air sample for the above airborne chlorinated phenols is performed by passage of 1000 liters of air through a polyurethane-foam sampling tube, followed by Soxhlet extraction of the foam, and analysis of the extract by gas chromatography with electron capture detection (GC/ECD). When concentration levels are high, confirmation of peak identification can be performed by gas chromatography/mass spectrometry (GC/MS).

INTERFERENCES:

Phthalate esters and polychlorinated naphthalenes can interfere, and use of non-TFE plastics must be avoided.

QUALITY CONTROL:

Solvents, reagents, glassware, and polyurethane plugs must be shown to be free of interfering artifacts. A field blank must be analyzed with each set of samples, and both a quality control standard and a method blank should be analyzed with each set of 5 to 15 samples.

EPA/TECHNICAL STATUS:

This method has been evaluated by the Environmental Protection Agency, but is not approved for any specific regulatory program. No precision or accuracy information is furnished. This method is applicable to concentrations of 1 to 1000 nanograms per cubic meter.

REFERENCE:

U.S. Environmental Protection Agency, Guidelines for Air Monitoring at Hazardous Waste Sites, Assignment 26, Appendix B; Contract 68-02-3168, U.S. EPA, U.S. EPA: Research Triangle Park, North Carolina, May 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $115 (list); the cost of GC/MS confirmation is approximately $150-200 (list). Sampling costs are additional.

CHLORINATED PHENOLS

Water Samples
Laboratory Method

Compound	CAS Number
2,4-D Acid	94-75-7
Hexachlorophene	70-70-4
Pentachlorophenol	87-86-5
2,4,5-T-Acid	93-76-5
2,3,4,6-Tetrachlorophenol	58-90-2
2,4,6-Trichlorophenol	25167-82-2

METHOD SUMMARY:

The above chlorinated phenols in water samples can be measured by gas chromatography with electron capture detection (GC/ECD). The sample is passed through a strongly basic anion-exchange resin bed, then the phenols are extracted with benzene (containing an internal standard), and derivatized into the corresponding methyl ethers. The extract, containing the methyl ethers, is analyzed by GC/ECD and the results are compared to standards.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The detection limits for the different substances range from 0.001-0.1 parts per billion in one liter of water. No precision or accuracy information is furnished.

REFERENCES:

Renberg, L. "Ion Exchange Technique for the Determination of Chlorinated Phenols and Phenoxy Acids in Organic Tissue, Soil, and Water," Anal. Chem., 46(3), 1974, pp. 459-461.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $155-180 (list).

CHLOROBENZENES

Air Samples
Laboratory Method

Compound	CAS Number	Range (mg/m^3)
Pentachlorobenzene	608-93-5	0.008-22
1,2,4,5-Tetrachlorobenzene	95-95-4	0.003-31
1,2,4-Trichlorobenzene	120-82-1	0.002-100

METHOD SUMMARY:

Gas chromatography can be used for the analysis of the above chlorobenzenes in air. A known volume of air is drawn through a 2-stage sampler consisting of a Teflon fiber mat filter followed by a tube containing 2 sections of Amberlite XAD-2 resin sorbent. The analyte from the filter and 2 sorbent sections is desorbed separately with hexane. The resulting solutions are analyzed with a gas chromatograph with electron capture detection (GC/ECD).

INTERFERENCES:

Separation of 1,2,3,5-tetrachlorobenzene and 1,2,4,5-tetrachlorobenzene is not possible using this method.

QUALITY CONTROL:

Method blanks and standards must be analyzed with the samples.

EPA/TECHNICAL STATUS:

This method has been accepted by the National Institute for Occupational Safety and Health (NIOSH) for use over the ranges listed above (in a 10- to 12-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 343; Publication No. 82-100, U.S. DHHS: Cincinnati, August 1981.

COST INFORMATION:

Cost per sample for analyses by this method is approximately $90 (list), plus sampling costs.

β-CHLOROETHERS

Air Samples
Laboratory Method

Compound	CAS Number
Bis(2-chloroethoxy) Methane	111-91-1
Bis(2-chloroethyl) Ether	111-44-4
Bis(2-chloroisopropyl) Ether	108-60-1
2-Chloroethyl Vinyl Ether	110-75-8

METHOD SUMMARY:

The above β-chloroethers in air samples can be measured by gas chromatography/mass spectrometry (GC/MS). The samples are collected by drawing air through two tubes packed with Tenax GC, which are both desorbed with methanol. An aliquot of the resulting solution is then analyzed by GC/MS.

INTERFERENCES:

Chloroethyl vinyl ether and chloroethyl ethyl ether have the same retention time and thus are not separatable by use of the GC/MS.

QUALITY CONTROL:

Tubes spiked in the field and control tubes are handled in the same manner as samples.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information is furnished. The limit of detection is 0.7 micrograms per cubic meter.

REFERENCE:

Sherman, P. L.; Kemmer, A. M.; Metacalfe, L.; Toy, H. D. Environmental Monitoring Near Industrial Sites: β-Chloroethers; EPA-560/6-78-003, U.S. Environmental Protection Agency: Washington, June 1978.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $250-335 (list), plus sampling costs.

CRESOLS

Air Samples
Laboratory Method

Compound	CAS Number
Cresol	1319-77-3
m-Cresol	108-39-4
o-Cresol	95-48-7
p-Cresol	106-44-5

METHOD SUMMARY:

Analysis of air samples for the above cresols can be performed by passage of a known volume of air through a silica-gel tube trap, followed by desorption with acetone 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:

A method blank must be processed and the desorption efficiency calculated for each set of samples. The results for the samples must be corrected accordingly.

EPA/TECHNICAL STATUS:

This method is approved by the National Institute for Occupational Safety and Health (NIOSH), and validated over the range of 10 to 42 milligrams per cubic meter for a 20-liter air sample. Precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set L, Method S167; 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 S167; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70-90 (list), plus sampling costs.

DINITROBENZENES

Air Samples
Laboratory Method

Compounds	CAS Number
Dinitrobenzene (mixed)	25154-54-5
m-Dinitrobenzene	99-65-0
o-Dinitrobenzene	528-29-0
p-Dinitrobenzene	100-25-4

METHOD SUMMARY:

The above dinitrobenzenes in air samples may be measured by high-performance liquid chromatography (HPLC). A known volume of air is drawn through a mixed cellulose ester membrane filter connected in series to a midget bubbler containing 10 milliliters of ethylene glycol. After collection of the sample, the filter is placed in the bubbler flask and 5 milliliters of methanol are added. The resulting solution is analyzed by HPLC with UV detection.

INTERFERENCES:

Any compound that has the same retention time as dinitrobenzene will interfere.

QUALITY CONTROL:

Since no internal standard is used in this method, standard solutions must be analyzed at the same time as the samples. A correction for the blank must be made 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 0.42 to 2.4 milligrams per cubic meter, using a 90-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 S214; Publication No. 78-175, U.S. DHEW: Cincinnati, 1978.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $100-110 (list), plus sampling costs.

DINITROTOLUENES

Air Samples
Laboratory Method

Compounds	CAS Number
Dinitrotoluene	25321-14-6
2,4-Dinitrotoluene	121-14-2
2,6-Dinitrotoluene	606-20-2

METHOD SUMMARY:

Analysis of air for the above dinitrotoluenes can be performed by sampling with silica gel tubes, desorption with carbon tetrachloride, and gas chromatographic (FID) analysis. The method differentiates between dinitrotoluene isomers.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

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

REFERENCE:

Hunt, R. I.; Neubauer, N. R.; Picone, R. F. "An Improved Procedure for Sampling and Analysis of Dinitrotoluene Vapor Concentrations in Workplace Air," Am. Ind. Hyg. Assoc. J., 41(8), 1980, pp. 592-594.

COST INFORMATION:

Cost per sample for this analysis is approximately $70-90 (list), plus sampling costs.

DIURON AND PYRETHRINS

Air Samples
Laboratory Method

Compounds	CAS Number
Diuron	330-54-1
Pyrethrins	121-29-9
	121-21-1

METHOD SUMMARY:

Diuron and pyrethrins in air can be determined by high-performance liquid chromatography (HPLC). The sample is collected in a low-volume system using polyurethane foam as a trapping medium. The foam is Soxhlet extracted with 5-percent ethyl ether in hexane, the extract is concentrated in a rotary evaporator, and an aliquot is analyzed by reversed-phase HPLC with UV detection.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information for the analyses is furnished.

REFERENCE:

Lewis, R. G.; MacLeod, K. E. "Portable Sampler for Pesticides and Semivolatile Industrial Organic Chemicals in Air," Anal Chem., 54, 1982, pp. 310-315.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $165 (list), plus sampling costs.

EXPLOSIVES

Water Samples
Laboratory Method

Compounds	CAS Number
Dinitrotoluene	25321-14-6
Nitroglycerine	55-63-0

METHOD SUMMARY:

The above explosives in water samples can be measured by single-sweep polarography. The substances are extracted from the water into a suitable solvent, which is then evaporated. The analyte is determined by polarography with an appropriate supporting electrolytic solution, either directly or by the standard addition method.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished. The method is for use over the range of 5 micrograms to 5 milligrams of analyte per liter.

REFERENCE:

Whitnack, G. C. "Single-Sweep Polarographic Techniques Useful in Micropollution Studies of Ground and Surface Waters," Anal. Chem., 47(4), 1975, pp. 618-621.

COST INFORMATION:

Cost information has not been obtained.

FUMIGANTS

Air Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Acrylonitrile	107-13-1	Hydrogen Cyanide	74-90-8
Bis(2-Chloroethyl) Ether	111-44-4	Methyl Bromide	74-83-9
Bromoform	75-25-2	Methyl Chloride	74-87-3
Carbon Disulfide	75-15-0	Methylene Chloride	75-09-2
Carbon Tetrachloride	56-23-5	Phosphine	7803-51-2
Chlorobenzene	108-90-7	Propylene Dichloride	78-87-5
Chloroform	67-66-3	Tetrachloroethylene	127-18-4
1,2-Dichlorobenzene	95-50-1	1,1,1-Trichloroethane	71-55-6
1,4-Dichloroenzene	106-46-7	Trichloroethylene	79-01-6
Ethylene Dibromide	106-93-4		

METHOD SUMMARY:

Gas chromatography may be used to analyze air for the above fumigants. A bubbler, charged with 1-butanol, n-pentane, or m-xylene is used to collect the analyte, and the resulting solution is analyzed by gas chromatography with flame ionization detection (GC/FID).

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

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

REFERENCE:

Berck, B. "Determination of Fumigant Gases by Gas Chromatography," J. Agric. Food Chem., 13(4), 1965, pp. 373-377.

COST INFORMATION:

Cost per sample of analysis by this method is approximately $60-90 (list), plus sampling costs.

GASEOUS CONTAMINANTS

Air Samples
Laboratory Method

Compound	CAS Number
Acetonitrile	75-05-8
Benzene	71-43-2
Cyclohexane	110-82-7
1,2-Dichlorobenzene	95-50-1
Dichlorodifluoromethane	75-71-8
1,2-Dichloroethane	107-06-2
Furan	10-00-9
Methyl Ethyl Ketone	78-93-3
Vinyl Chloride	75-01-4

METHOD SUMMARY:

The above gaseous contaminants in air samples can be determined by long-path laser absorption spectrometry. Measurements are performed using a 50-centimeter cell with sodium chloride windows. The response of the sample to the laser is received by a pyroelectric detector, amplified, and the data treated by computer.

INTERFERENCES:

The number and absorption characteristics of other substances in the sample affect the determination of each particular substance.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Limited precision and accuracy information is furnished. The detection limit varies with the composition of the sample, but is generally in the parts-per-million range.

REFERENCES:

Green, B. D.; Steinfeld, J. I. "Monitoring Complex Trace-Gas Mixtures by Long-Path Laser Absorption Spectrometry," Proc. Soc. Photo-Opt. Instrum. Eng., 99, 1977, pp. 32-38.

Green, B. D.; Steinfeld, J. I. "Laser Absorption Spectroscopy: Method for Monitoring Complex Trace Gas Mixtures," Environ. Sci. Technol., 10(2), 1976, pp. 1134-1138.

COST INFORMATION:

No cost information has been obtained.

HALOGENATED, AROMATIC, AND OTHER COMPOUNDS

Air Samples
Laboratory Method

Compound	CAS Number	Compounds	CAS Number
Acetaldehyde	75-07-0	1,2-Dichlorobenzene	95-50-1
Acetic Acid	64-19-7	1,4-Dichlorobenzene	106-46-7
Acetic Anhydride	108-24-7	1,1-Dichloroethane	75-34-3
Acetone	67-64-1	1,2-Dichloroethane	107-06-2
Acetonitrile	75-05-8	1,1-Dichloroethylene	156-60-5
Acetophenone	98-86-2	1,2-Dichloroethylene	
Acrolein	107-02-8	1,1-Dimethylhydrazine	57-14-7
Acrylonitrile	107-13-1	Epichlorohydrin	106-89-8
Allyl Alcohol	107-18-6	Ethyl Acetate	141-78-6
Allyl Chloride	107-05-1	Ethyl Acrylate	140-88-5
Aniline	62-53-3	Toluene	108-88-3
Benzene	71-43-2	1,2,4-Trichlorobenzene	120-82-1
Benzoic Acid	65-85-0	1,1,1-Trichloroethane	71-55-6
Bis(2-Chloroethyl) Ether	111-44-4	1,1,2-Trichloroethane	79-00-5
Butyl Acetate	123-86-4	Trichloroethylene	79-01-6
n-Butyl Alcohol	71-36-3	Vinyl Chloride	75-01-4
Carbon Tetrachloride	56-23-5	Xylene	1330-20-7
Chlorobenzene	108-90-7	m-Xylene	108-38-3
Chloroethane	75-00-3	o-Xylene	95-47-6
Chloroform	67-66-3	p-Xylene	106-42-3
Cumene			

METHOD SUMMARY:

Analysis for the above halogenated and aromatic compounds in air can be performed by passage of 25 to 80 liters of air through a Tenax© sorbent tube, followed by thermal desorption of the analytes into a temperature-programmed gas chromatograph with mass spectrometric detection (GC/MS).

INTERFERENCES:

Sorbent contaminants represent the primary source of interferences, and sorbent plugs should be pre-extracted and packaged to prevent contamination both before and after sampling. The sorbent should be Soxhlet-extracted with methanol, and then with pentane, for 24 hours, and dried under helium before use. Decomposition of analyte due to ultraviolet radiation has been reported.

QUALITY CONTROL:

Analysis of duplicate samples should agree within 20 percent. One method blank must be processed with each set of 20 or fewer samples, and a blank sampling cartridge should be analyzed after each analysis of a sample with heavy organic loading. If method blanks show any peaks, the results must be adjusted accordingly. A field blank should be analyzed with each set of 20 or fewer samples, and a laboratory control standard must be processed with each

set of 20 or fewer samples and give results within 20 percent of the actual concentration.

EPA/TECHNICAL STATUS:

This method is not EPA-approved for any regulatory program, but has been evaluated by the EPA. No precision or accuracy information is furnished. The method is applicable from a level of 2 micrograms per cubic meter. Breakthrough volumes are reported by Brown and Purnell.

REFERENCES:

U.S. Environmental Protection Agency, Guidelines for Air Monitoring at Hazardous Wastes Sites, Assignment No. 26, Appendix A; Contract 68-02-3168, U.S. EPA: Research Triangle Park, North Carolina, 1982.

Schlitt, H.; Knoeppel, H.; Versino, B.; Peil, A.; Schauenburg, H.; Visers, H. In Sampling and Analysis of Toxic Organics in the Atmosphere; ASTM STP 721, American Society for Testing and Materials: Philadelphia, 1980, pp. 22-35.

Brooks J.; West, D. Portable Miniature Sampler for Potential Airborne Carcinogens in Microenvironments, Phase 1: Development; EPA-600/2-80-026, U.S. EPA, 1980.

Pellizzari, E. D.; Carpenter, B. H.; Bunch, J. E.; Sawicki, E. "Collection and Analysis of Trace Organic Vapor Pollutants in Ambient Atmospheres," Environ. Sci. Technol., 9(6), 1975, pp. 556-560.

Brown, R. H.; Purnell, C. J. "Collection and Analyses of Trace Organic Vapor Pollutants in Ambient Atmospheres," J. Chromatogr. 178, 1979, pp. 79-90.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $250-325 (list), plus sampling costs.

HALOGENATED COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number
Bromoform	75-25-2
Carbon Tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chlorodibromomethane	124-48-1
Chloroform	67-66-3
Dichlorobromomethane	75-27-4
Methylene Chloride	75-09-2
Methyl Iodide	74-88-4
1,1,2,2-Tetrachloroethane	79-34-5
Trichloroethylene	79-01-6

METHOD SUMMARY:

The above halogenated compounds in water samples can be measured by gas chromatography (GC). The analytes are collected from the water by a purge and trap apparatus, then desorbed directly into the GC instrument. The effluent from the GC column is split to be monitored by both a flame ionization detector (FID) and a microwave emission detector, which is element-selective and can distinguish the different halogens.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished. Limits of detection range from 54 to 280 nanograms of analyte per liter of water.

REFERENCE:

Quimby, B. D.; Delaney, M. F.; Uden, P. C.; Barnes, R. M. "Determination of Trihalomethanes in Drinking Water by Gas Chromatography with a Microwave Plasma Emission Detector," Anal. Chem., 51(7), 1979, pp. 875-880.

COST INFORMATION:

Cost information has not been obtained.

HALOGENATED VAPORS

Air Samples
Laboratory Method

Compound	CAS Number
Carbon Tetrachloride	56-23-5
Trichloromonofluoromethane	75-69-4

METHOD SUMMARY:

Analysis of air for carbon tetrachloride and trichloromonofluoromethane can be performed by gas chromatography. A sample is pressurized, the analytes are condensed with liquid nitrogen, and the sample is evacuated into a gas chromatograph with electron capture detection (GC/ECD). Calibration is by the exponential dilution method.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are provided.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The lower limit of detection without preconcentration is 10 microliters per cubic meter for carbon tetrachloride and 2 microliters per cubic meter for trichloromonofluoromethane. No precision or accuracy information is furnished.

REFERENCE:

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-07, "Chromatographic Determination of SF_6, CCl_3F, CCl_4, and N_2O," HASL-300, Environmental Measurement Laboratory, U.S. DOE: New York, 1983.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70, plus sampling costs.

HALOGENATED VOLATILE ORGANIC COMPOUNDS

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number	Estimated Detection Limit (μg/L) (SW-846, EPA)
Benzyl Chloride	100-44-7	0.20
Bromoform	75-25-2	-
Chloral	75-87-6	-
Chloroacetaldehyde	107-20-0	-
Chlorobenzene	108-90-7	0.25
Chloroethane	75-00-3	0.52
2-Chloroethyl Vinyl Ether	110-75-8	0.13
Chloroform	67-66-3	0.05
Chloromethyl Methyl Ether	107-30-2	-
Dichlorobenzene (mixed)	25321-22-6	0.24
1,2-Dichlorobenzene	95-50-1	0.15
1,3-Dichlorobenzene	541-73-1	0.32
1,4-Dichlorobenzene	106-46-7	0.24
Dichlorobromomethane	75-27-4	0.10
Dichlorodifluoromethane	75-71-8	1.81
1,1-Dichloroethane	75-34-3	0.07
1,2-Dichloroethane	107-06-2	0.03
1,1-Dichloroethylene	75-35-4	0.13
1,2-trans-Dichloroethylene	156-60-5	0.10
1,2-Dichloropropane	78-87-5	0.04
1,3-cis-Dichloropropene	26952-23-8	0.20
1,3-trans-Dichloropropene	542-75-6	0.34
Methyl Bromide	74-83-9	1.18
Methyl Chloride	74-87-3	0.08
Methylene Bromide	74-95-3	-
Methylene Chloride	75-09-2	0.25
1,1,1,2-Tetrachloroethane	630-20-6	-
1,1,2,2-Tetrachloroethane	79-34-5	0.03
Tetrachloroethylene	127-18-4	0.03
1,1,1-Trichloroethane	71-55-6	0.03
1,1,2-Trichloroethane	79-00-5	0.02
Trichloroethylene	79-01-6	0.12
Trichloromonofluoromethane	75-69-4	-
Vinyl Chloride	75-01-4	0.18

METHOD SUMMARY:

Analysis of soil/sediment samples for the above halogenated compounds can be performed by gas chromatography. A solid sample, dispersed in methanol or polyethylene glycol, is purged with an inert gas onto a sorbent trap, and the trap is heated and backflushed into a gas chromatograph equipped with an electron capture detector (GC/ECD). Either internal or external standards may be used for quantification.

INTERFERENCES:

Sample contact with plastics during collection, storage, or processing must be avoided.

QUALITY CONTROL:

The analytical system must be demonstrated to be free of interferences through analysis of a method blank each time a set of samples is processed or a change in reagents occurs. Field replicates, laboratory replicates, and fortified samples must be analyzed to demonstrate the precision and accuracy of the method. In cases of doubt regarding the identity of a peak in the chromatogram, confirmatory techniques such as gas chromatography/mass spectrometry (GC/MS) or second-column confirmation should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved for use under the Resource Conservation and Recovery Act (RCRA). Precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, J. E. Longbottom; J. J. Lichtenberg, Eds., Method 601; EPA-600/4-82-059, U.S. EPA: Cincinnati, 1982.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 8010, 5020, 5030; SW-846, U.S. EPA: Washington, July 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $80-145 (list).

HALOGENATED VOLATILE ORGANIC COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number	Method Detection Limit (μg/L) (SW-846, EPA)
Benzyl Chloride	100-44-7	0.20
Bromoform	75-25-2	-
Chloral	75-87-6	-
Chloroacetaldehyde	107-20-0	-
Chlorobenzene	108-90-7	0.25
Chloroethane	75-00-3	0.52
2-Chloroethyl Vinyl Ether	110-75-8	0.13
Chloroform	67-66-3	0.05
Chloromethyl Methyl Ether	107-30-2	-
Dichlorobenzene (mixed)	25321-22-6	0.24
1,2-Dichlorobenzene	95-50-1	0.15
1,3-Dichlorobenzene	541-73-1	0.32
1,4-Dichlorobenzene	106-46-7	0.24
Dichlorobromomethane	75-27-4	0.10
Dichlorodifluoromethane	75-71-8	1.81
1,1-Dichloroethane	75-34-3	0.07
1,2-Dichloroethane	107-06-2	0.03
1,1-Dichloroethylene	75-35-4	0.13
1,2-trans-Dichloroethylene	156-60-5	0.10
1,2-Dichloropropane	78-87-5	0.04
1,3-cis-Dichloropropene	26952-23-8	0.20
1,3-trans-Dichloropropene	542-75-6	0.34
Methyl Bromide	74-83-9	1.18
Methyl Chloride	74-87-3	0.08
Methylene Bromide	74-95-3	-
Methylene Chloride	75-09-2	0.25
1,1,1,2-Tetrachloroethane	630-20-6	-
1,1,2,2-Tetrachloroethane	79-34-5	0.03
Tetrachloroethylene	127-18-4	0.03
1,1,1-Trichloroethane	71-55-6	0.03
1,1,2-Trichloroethane	79-00-5	0.02
Trichloroethylene	79-01-6	0.12
Trichloromonofluoromethane	75-69-4	-
Vinyl Chloride	75-01-4	0.18

METHOD SUMMARY:

Analysis of water samples for the above volatile organic compounds can be performed by gas chromatography. A water sample is purged with an inert gas onto a trap and the trap is heated and backflushed into a gas chromatograph with an electron capture detector (GC/ECD), using either internal or external standards for quantification. Alternatively, the sample can be extracted with isooctane and the resulting solution analyzed by GC/ECD.

INTERFERENCES:

Contact of the samples with plastics during collection, storage, or processing must be avoided. The analytical system must be demonstrated to be free of interferences by the analysis of a method blank each time a set of samples is processed or a change in reagents occurs.

QUALITY CONTROL:

Field replicates, laboratory replicates, and fortified samples must be analyzed to determine the precision and accuracy of the method. In cases of doubt regarding the identity of a peak on the chromatogram, confirmatory techniques such as gas chromatography/mass spectrometry (GC/MS) or second-column confirmation should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved for use under the Federal Water Pollution Control Act and the Resource Conservation and Recovery Act (40 CFR, Part 261, Identification and Listing of Hazardous Waste). Precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 8010, 5030; SW-846; U.S. EPA: Washington, July 1982.

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, J. E. Longbottom; J. J. Lichtenberg; Eds., Method 601; EPA-600/4-82-059, U.S. EPA: Cincinnati, 1982.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, D3973-80; ASTM: Philadelphia, 1983.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $115 (list).

HAZARDOUS ORGANIC EMISSIONS

Air Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Acetaldehyde	75-07-0	Ethylene Dibromide	106-93-4
Acetone		Ethyl Ether	60-29-7
Acetophenone	98-86-2	Glycidylaldehyde	765-34-3
Acrolein	107-02-8	Hexachlorobutadiene	87-68-3
Allyl Chloride	107-05-1	Hexachloroethane	67-72-1
Aniline	62-53-3	Methanol	90-80-5
Bis(Chloromethyl) Ether	542-88-1	Methyl Bromide	74-83-9
Bis(2-Chloroisopropyl) Ether	108-60-1	Methyl Chloride	74-87-3
		Methylene Bromide	74-95-3
Bromoform	75-25-2	Methyl Chloride	75-09-2
iso-Butylamine	78-81-9	Methyl Methacrylate	80-62-6
Butyric Acid	107-92-6	Nitric Oxide	10102-43-9
Carbon Tetrachloride	56-23-5	Nitrogen Dioxide	10102-44-0
Chlorobenzene	108-90-7	N-Nitrosodiethylamine	55-18-5
Chloroform	67-66-3	N-Nitrosodimethylamine	62-75-9
1,2-Dichlorobenzene	95-50-1	Pentachloroethane	76-01-7
1,3-Dichlorobenzene	541-73-1	Phenol	108-95-2
1,2-Dichloroethane	107-06-2	Phosgene	75-44-5
1,1-Dichloroethylene	75-34-5	Propylene Oxide	75-56-9
1,2-Dichloropropane	78-87-5	Pyridine	110-86-1
Dichloropropene	26952-23-8	1,1,1,2-Tetrachloroethane	630-20-6
1,2:3,4-Diepoxybutane	1464-53-5	1,1,2,2-Tetrachloroethane	79-34-5
Diethyl Phthalate	84-66-2	Tetrachloroethylene	127-18-4
Dimethylamine	124-40-3	Toluene	108-88-3
Dimethyl Phthalate	131-11-3	1,1,1-Trichloroethane	71-55-6
Epichlorohydrin	106-89-8	Trichloroethylene	79-01-6
Ethyl Acetate	141-78-6	Vinyl Chloride	75-01-4
Ethylbenzene	100-41-4		

METHOD SUMMARY:

Analysis of air for the above organic compounds can be performed by gas chromatography/mass spectrometry (GC/MS). Air is pumped through a Tenax® cartridge, the cartridge is desorbed into a liquid nitrogen trap, and the sample is released into the GC/MS instrument by rapidly heating the trap.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

With each batch of samples, two or three sampling cartridges should be processed as blanks.

EPA/TECHNICAL STATUS:

Sampling precision and accuracy information for some compounds is given. Breakthrough volumes are given or estimated for some compounds.

REFERENCE:

Krost, K.; Pellizzari, E. D.; Walburn, S. G.; Hubbard, S. A. "Collection and Analysis of Hazardous Organic Emissions," Anal. Chem., 54(4), 1982, pp. 810-817.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $250-325 (list), plus sampling costs.

HAZARDOUS COMPOUNDS

Air Samples
Laboratory Method

Compound	CAS Number
2-Acetylaminofluorene	53-96-3
Benzidine	92-87-5
3,3'-Dichlorobenzidine	91-94-1
Diethylstilbestrol	56-53-1
3,3'-Dimethoxybenzidine	119-90-4
3,3'-Dimethylbenzidine	119-93-7
1-Naphthylamine	134-32-7
2-Naphthylamine	91-59-8

METHOD SUMMARY:

The above hazardous compounds in air samples can be measured by gas chromatography with electron capture detection (GC/ECD). The sample is collected on an air filter. The procedure for removal of the compounds from the filter is not specified. The sample is partitioned between an aqueous acid solution and benzene. The two phases are treated separately by pH adjustments and additonal extraction steps to eventually produce three fractions; a phenolic fraction containing estrogens (diethylstilbestrol), a neutral fraction containing acetylaminofluorene hydrolyzed to 2-aminofluorene, and a basic fraction containing the amines. The residues are converted to their pentafluoropropionyl derivatiives before GC/ECD analysis.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information is furnished. The limit of detection is not specified, but the method is designed for analysis at the sub- to low- parts-per-billion range.

REFERENCE:

Bowman, M. C. "Trace Analysis: A Requirement for Toxicological Research with Carcinogens and Hazardous Substances," J. Assoc. Off. Anal. Chem., 61(5), 1978, pp. 1253-1262.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $150-240 (list) (est.).

HAZARDOUS COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number
2-Acetylaminofluorene	53-96-3
Benzidine	92-87-5
3,3'-Dichlorobenzidine	91-94-1
Diethylstilbestrol	56-53-1
3,3'-Dimethoxybenzidine	119-90-4
3,3'-Dimethylbenzidine	119-93-7
1-Naphthylamine	134-32-7
2-Naphthylamine	91-59-8

METHOD SUMMARY:

The above hazardous compounds in water samples can be measured by gas chromatography with electron capture detection (GC/ECD). The sample is acidified and then extracted with benzene. The two phases are treated separately by pH adjustments and additional extraction steps to eventually produce three fractions: a phenolic fraction containing estrogens (diethylstilbestrol), a neutral fraction containing acetylaminofluorene hydrolyzed to 2-aminofluorene, and a basic fraction containing the amines. The residues are converted to their pentafluoropropionyl derivatives before the GC/ECD analysis.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information is furnished. The limit of detection is not specified, but the method is designed for analysis at the sub- to low- parts-per-billion range.

REFERENCE:

Bowman, M. C. "Trace Analysis: A Requirement for Toxicological Research with Carcinogens and Hazardous Substances," J. Assoc. Off. Anal. Chem., 61(5), 1978, pp. 1253-1262.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $150-240 (list) (est.).

HERBICIDES

Air Samples
Laboratory Method

Compound	CAS Number
2,4-D Esters	94-11-1
	94-79-1
	94-80-4
	1320-18-9
	1928-38-7
	1928-61-6
	1929-73-3
	25168-26-7
	53467-11-1
	2971-38-2
2,4,5-T Esters	93-79-8
	2545-59-7
	61792-07-2
	1928-47-8
	25168-15-4

METHOD SUMMARY:

Analysis for the above airborne herbicides is performed by passage of 1000 liters of air through a polyurethane-foam sampling tube, followed by Soxhlet extraction of the foam, and analysis of the extract by gas chromatography with electron capture detection (GC/ECD). When concentration levels are high, confirmation of peak identification is performed by gas chromatography/mass spectrometry (GC/MS).

INTERFERENCES:

Phthalate esters and polychlorinated naphthalenes can interfere, and use of non-TFE plastics must be avoided.

QUALITY CONTROL:

Solvents, reagents, glassware, and polyurethane plugs must be shown to be free of interfering artifacts. A field blank must be analyzed with each set of samples, and both a quality control standard and a method blank should be analyzed with each set of 5 to 15 samples.

EPA/TECHNICAL STATUS:

This method has been evaluated by the Environmental Protection Agency, but is not approved for any specific regulatory program. No precision or accuracy information is furnished. This method is applicable to a range from 1 nanogram to 1000 nanograms per cubic meter.

REFERENCE:

U.S. Environmental Protection Agency, Guidelines for Air Monitoring at Hazardous Waste Sites, Assignment 26, Appendix B; Contract 68-02-3168, U.S. EPA: Research Triangle Park, North Carolina, May 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $115 (list); the cost of GC/MS confirmation is approximately $150-200 (list). Sampling costs are additional.

HYDROCARBON ODORANTS

Air Samples
Laboratory Method

Compound	CAS Number
Benzene	71-43-2
Ethylbenzene	100-41-4
Isoprene	78-79-5
Toluene	108-88-3
m-Xylene	108-38-3
o-Xylene	95-47-6
p-Xylene	106-42-3

METHOD SUMMARY:

The above hydrocarbon odorants in air samples can be measured by gas chromatography (GC). The sample is collected by cold trapping with liquid nitrogen and undergoes a pre-column concentration step before analysis.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision information is furnished. The detection limits are 0.1 to 5.5 micrograms per cubic meter.

REFERENCE:

Hoshika, Y.; Nihei, Y.; Muto, G. "Pattern Display for Characterization of Trace Amounts of Odorants Discharged from Nine Odour Sources," Analyst, 106, 1981, pp. 1187-1202.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $105-120 (list).

LOW-MOLECULAR-WEIGHT ALIPHATIC AMINES

Air Samples
Laboratory Method

Compound	CAS Number
Butylamine	109-73-9
iso-Butylamine	78-81-9
sec-Butylamine	513-49-5
	13852-84-6
tert-Butylamine	75-64-9
Diethylamine	109-89-7
Dimethylamine	124-40-3
Trimethylamine	75-50-3

METHOD SUMMARY:

The above low-molecular-weight aliphatic amines in air samples can be measured by gas chromatography with nitrogen-phosphorus detection (GC/NPD). A known volume of air is passed through a prepared SEP-PAK C_{18} cartridge to collect the analyte which is then desorbed with methanol:water (1:1). The pH of the eluant is adjusted, the solution is brought to volume, and an aliquot is analyzed by GC/NPD.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precison and accuracy information is furnished. The limit of detection is 9 micrograms per cubic meter.

REFERENCE:

Kuwata, K.; Akiyama, E.; Yamazaki, Y.; Yamasaki, H.; Kuge, Y.; Kiso, Y. "Trace Determination of Low Molecular Weight Aliphatic Amines in Air by Gas Chromatography," Anal. Chem., 55, 1983, pp. 2199-2201.

COST INFORMATION:

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

LOW-MOLECULAR-WEIGHT HALOGENATED HYDROCARBONS

Water Samples
Laboratory Method

Compound	CAS Number
Bromoform	75-25-2
Chlorodibromomethane	124-48-1
Chloroform	67-66-3
Tetrachloroethylene	127-18-4
1,1,1-Trichloroethane	71-55-6

METHOD SUMMARY:

Gas chromatography may be used to measure the above low-molecular-weight halogenated hydrocarbons in water samples. A 5-milliliter sample is extracted with 1 milliliter of isooctane, and an aliquot of the organic phase is analyzed in a gas chromatograph with an electron capture detector (GC/ECD).

INTERFERENCES:

Volatile compounds that are extractable and responsive to electron capture detection may interfere, as may impurities in the extracting solvent.

QUALITY CONTROL:

Blanks and standards should be analyzed with the samples.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The applicable concentration range for trihalomethanes is 1 to 200 micrograms per liter. Precision and accuracy information is furnished.

REFERENCE:

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, D 3973-80; ASTM: Philadelphia, 1983, pp. 145-150.

COST INFORMATION:

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

MERCAPTANS

Air Samples
Laboratory Method

Compound	CAS Number
Methyl Mercaptan	74-93-1
Thiophenol	108-98-5

METHOD SUMMARY:

Analysis of air samples for the above mercaptans can be performed by colorimetry. A sample is taken, either manually or automatically, by passage of a known volume of air through a bubbler charged with a mercuric acetate/acetic acid solution. The resulting solution is treated with an acidic solution of ferric chloride and N,N-dimethyl-*p*-phenylenediamine hydrochloride. After 30 minutes the absorbance of the resulting solution at 500 nanometers is measured with a spectrophotometer. This method measures the total mercaptan content of the sample, and does not identify any particular compound.

INTERFERENCES:

Other sulfur-containing compounds, including hydrogen sulfide, can give positive results. The mercuric acetate used must be free of mercurous ion or turbidity will result.

QUALITY CONTROL:

No quality control procedures are provided.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The method is suitable for measurement of atmospheric concentrations of mercaptans below 195 micrograms per cubic meter. The minimum detectable amount of methyl mercaptan is 0.04 micrograms per milliliter of absorbing solution. The minimum detectable concentration is 1.95 micrograms methyl mercaptan per cubic meter, using a 240-liter sample. No precision or accuracy information is furnished.

REFERENCES:

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.03, D2913-75; ASTM: Philadelphia, 1983.

COST INFORMATION:

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

METHYLAMINES

Air Samples
Laboratory Method

Compound	CAS Number
Dimethylamine	124-40-3
Monomethylamine	74-89-5
Trimethylamine	75-50-3

METHOD SUMMARY:

The above methylamines in air samples can be measured by collection of the sample on an activated charcoal trap and analysis by gas chromatography with alkali flame ionization detection (GC/AFID).

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method has been evaluated by a single laboratory and is not EPA-approved. No precision or accuracy information is furnished. The method is suitable for concentration levels of analyte greater than approximately 10 micrograms per cubic meter.

REFERENCE:

Fuselli, S.; Benedetti, G.; Mastrangeli, R. "Determination of Methylamines in Air Using Activated Charcoal Traps and Gas Chromatographic Analysis with an Alkali Flame Detector (AFID)," Atmos. Environm., 16(12), 1982, pp. 2943-2946.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $120-130 (list), plus sampling costs.

N-METHYLCARBAMATES

Water Samples
Laboratory Method

Compound	CAS Number
Carbaryl	63-25-2
Carbofuran	1563-66-2
Mercaptodimethur	2032-65-7

METHOD SUMMARY:

Analysis of water samples for the above N-methylcarbamate pesticides can be performed by derivatization and gas chromatography with electron-capture detection (GC/ECD). The sample is acidified to pH 3 to 4, and the pesticides are extracted into methylene chloride. The extract is washed with potassium carbonate, dried, and concentrated. Ten-percent methanolic potassium hydroxide is used for hydrolysis, and the pentafluorobenzyl bromide derivatives are prepared. The derivatives are cleaned up and separated by silica-gel micro-column chromatography, by eluting the column three times, each time with a different eluant. The eluates are analyzed by GC/ECD.

INTERFERENCES:

Carbamate phenols are removed in the potassium carbonate wash step. Determination of mexacarbate and aminocarb is not possible with this method.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved for any regulatory program. Precision and accuracy information for the overall analysis and the cleanup step is furnished.

REFERENCE:

Coburn, J. A.; Ripley, B. D.; Chau, A. S. Y. "Analysis of Pesticide Residues by Chemical Derivatization. II. N-Methylcarbamates in Natural Water and Soils," J. Assoc. Off. Anal. Chem., 59(1), 1976, pp. 188-196.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $160-185 (list).

NAPHTHYLAMINES

Air Samples
Laboratory Method

Compound	CAS Number
1-Naphthylamine	134-32-7
2-Naphthylamine	91-59-8

METHOD SUMMARY:

The above naphthylamines in air samples may be measured by gas chromatography. A three-stage sampler consisting of a high-efficiency glass fiber filter and two beds of silica gel is used to collect the naphthylamine. The analyte is desorbed with a solution of acetic acid in 2-propanol, and an aliquot of this solution is injected into a gas chromatograph with flame ionization detection (GC/FID).

INTERFERENCES:

The retention time of 1-nitronaphthalene is between those of 1- and 2-naphthylamine and interferes with their determinations.

QUALITY CONTROL:

A method blank should be run with 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 4 to 70 micrograms per cubic meter for a 50-liter sample. The detection limit is 0.01 micrograms per sample. Precision information is furnished.

REFERENCE:

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, Method P&CAM 264; Publication No. 78-175, U.S.DHEW: Cincinnati, 1978.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70-90 (list) plus sampling costs.

NITRILES

Water Samples
Laboratory Method

Compound	CAS Number
Acetonitrile	75-05-8
Acrylonitrile	107-13-1
Benzonitrile	100-47-0

METHOD SUMMARY:

Analysis for the above nitriles in water samples can be performed by direct injection of 3 to 5 microliters of the sample into a gas chromatograph with a flame ionization detector (GC/FID), utilizing either temperature-programmed or isothermal operation.

INTERFERENCES:

Samples should be quick-frozen if not analyzed immediately, and sample pH should be adjusted to 7 at the time of collection.

QUALITY CONTROL:

Standard solutions should be regarded as unstable and stored in a freezer.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. It has undergone inter-laboratory evaluation and extensive review, and shows a precision of 3 to 10 percent over the range of 10 to 60 milligrams per liter. The detection limit is approximately 1 milligram per liter.

REFERENCE:

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, D 3371-79; ASTM: Philadelphia, 1983, pp. 162-164.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $60-90 (list).

NITROAROMATIC COMPOUNDS AND BENZONITRILE

Air Samples
Laboratory Method

Compound	CAS Number
Benzonitrile	100-47-0
p-Dinitrobenzene	100-25-4
Nitrobenzene	98-95-3

METHOD SUMMARY:

The above nitroaromatic compounds in air samples can be measured by gas chromatography. Sensitivites for specific compounds vary widely with the type of detector used; the electron capture detector is quite effective for nitrobenzene and *p*-dinitrobenzene. Analysis of benzonitrile requires a flame ionization detector.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

A reference standard must be run just before and after each group of samples.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished.

REFERENCE:

Mitchell, J.; Deveraux, H.D. "Determination of Traces of Organic Compounds in the Atmosphere: Role of Detectors in Gas Chromatography," Anal. Chim. Acta., 100, 1978, pp. 45-52.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $60-90 (list) with the FID, and approximately $80-100 (list) with the ECD, plus sampling costs.

NITROAROMATICS

Water Samples
Laboratory Method

Compound	CAS Number
Dinitrobenzenes, Mixed	25154-54-5
m-Dinitrobenzene	99-65-0
o-Dinitrobenzene	528-29-0
p-Dinitrobenzene	100-25-4
Dinitrotoluene	25321-14-6
2,4-Dinitrotoluene	121-14-2
2,6-Dinitrotoluene	606-20-2
Nitrotoluene	1321-12-6
5-Nitro-o-Toluidine	99-55-8
Toluene	108-88-3
sym-Trinitrobenzene	99-35-4

METHOD SUMMARY:

Water samples may be analyzed for the above nitroaromatics by gas chromatography with flame ionization detection (GC/FID). A 20-milliliter sample is extracted with diethyl ether, the extract is dried, concentrated, and analyzed using an internal standard. Identification of components is confirmed by gas chromatography/mass spectrometry (GC/MS).

INTERFERENCES:

No specific interferences are reported. Complex wastewater matrices require the use of fused-silica capillary column GC/FID.

QUALITY CONTROL:

The GC/FID should be calibrated twice daily, precision should be determined by analysis of multiple samples, and accuracy measured by analysis of fortified samples.

EPA/TECHNICAL STATUS:

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

REFERENCE:

Spanggord, R. J.; Gibson, B. W.; Keck, R. G.; Thomas, D. W. "Effluent Analysis of Wastewater Generated in the Manufacture of 2,4,6-Trinitrotoluene. 1. Characterization Study," Environ. Sci. Technol., 16(4), 1982, pp. 229-232.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $110-155 (list), excluding the GC/MS confirmation.

NITROAROMATICS AND CYCLIC KETONES

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Dinitrobenzenes, Mixed	25154-54-5
Dinitrotoluene	25321-14-6
2,4-Dinitrotoluene	121-14-2
2,6-Dinitrotoluene	606-20-2
Isophorone	78-59-1
1,4-Naphthoquinone	130-15-4
Nitrobenzene	98-95-3

METHOD SUMMARY:

Analysis of soil/sediment samples for the above nitroaromatics and cyclic ketones can be performed by gas chromatography. A soil/sediment sample is extracted with methylene chloride at neutral pH, using Soxhlet extraction or sonication, and the extract is dried, concentrated, and analyzed by gas chromatography with electron capture detection (GC/ECD) or flame ionization detection (GC/FID), using either internal or external standards for quantification.

INTERFERENCES:

The use of plastics in collection, storage, or processing of samples must be avoided.

QUALITY CONTROL:

An aliquot of each sample must be spiked to determine the detection limits and recovery, and a method blank should be analyzed with each set of samples or change in reagents. Field and laboratory replicates and duplicate samples should be analyzed to demonstrate the sensitivity and accuracy of the method. When doubt exists about the identification of a peak in the chromatogram, confirmatory methods, such as gas chromatography/mass spectrometry (GC/MS) or second-column confirmation, should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved for the Resource Conservation and Recovery Act (RCRA). No precision or accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, J. E. Longbottom; J. J. Lichtenberg, Eds., "Nitroaromatics and Isophorone" Method 609; EPA-600/4-82-057, U.S. EPA: Cincinnati, 1982.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3540, 3550, 8090; SW-846, U.S. EPA: Washington, July 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $120-130 (list).

NITROAROMATICS AND CYCLIC KETONES

Water Samples
Laboratory Method

Compound	CAS Number
Dinitrobenzene, Mixed	25154-54-5
1,4-Naphthoquinone	130-15-4

METHOD SUMMARY:

Analysis for the above nitroaromatics and cyclic ketones in water samples can be performed by gas chromatography. A water sample is extracted with methylene chloride, and the extract is dried, concentrated, and analyzed by gas chromatography with flame ionization detection (GC/FID) or with electron capture detection (GC/ECD).

INTERFERENCES:

Use of plastics during collection, storage, or processing of samples must be avoided.

QUALITY CONTROL:

An aliquot of each sample must be spiked to determine the detection limits and recovery, and a method blank should be analyzed with each set of samples or change in reagents. Field and laboratory replicates and duplicate samples should be analyzed to demonstrate the sensitivity and accuracy of the method. When doubt exists about the identification of a peak in the chromatogram, confirmatory methods, such as gas chromatography/mass spectrometry (GC/MS) or second-column confirmation, should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved for the Resource Conservation and Recovery Act (40 CFR, Part 261, Identification and Listing of Hazardous Wastes). No precision or accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, J. E. Longbottom; J. J. Lichtenberg, Eds., Method 609; EPA-600/4-82-057, U.S. EPA: Cicinnati, 1982.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3540, 3550, 8090; SW-846, U.S. EPA: Washington, July 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $110-160 (list).

NITROGENOUS DRUGS

No Matrix Given
Laboratory Method

Compound	CAS Number
Methapyrilene	91-80-5
Nicotine	54-11-5
Phenacetin	62-44-2

METHOD SUMMARY:

Analysis for the above nitrogenous drugs can be performed by gas chromatography with both flame-ionization and nitrogen-selective rubidium silicate bead alkali flame ionization detectors. The drugs are chromatographed with a caffeine internal standard on a 3-percent OV-17 column with a temperature program of 100°C to 250°C at 4° per minute.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are furnished.

EPA/TECHNICAL STATUS:

This method is not EPA-approved for use in any regulatory program. No precision or accuracy information is given.

REFERENCE:

Baker, J. K. "Identification and Chemical Classification of Drugs Based on the Relative Response of a Nitrogen Selective Detector and a Flame Ionization Detector in Gas Chromatographic Analysis," Anal. Chem., 49(7), 1977, pp. 906-908.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $180 (list), plus sample preparation.

NITROPHENOLS

Water Samples
Laboratory Method

Compound	CAS Number
Nitrophenol, Mixed	25154-55-6
2-Nitrophenol	88-75-5
3-Nitrophenol	554-84-7
4-Nitrophenol	100-02-7

METHOD SUMMARY:

The above nitrophenols in water samples can be measured by high-performance liquid chromatography (HPLC). The samples are adjusted to pH 2, and the nitrophenols are collected on mixed XAD-4/8 macroreticular resins. After extraction, concentration, and isolation from interferences, the nitrophenols are analyzed by HPLC with a UV detector.

INTERFERENCES:

The sample extract is isolated from interfering neutral hydrophobic organic compounds by retention on cesium silicate and elution with methanol.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

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

REFERENCE:

Graham, J. A. In Trace Analysis Vol. I, J. F. Lawrence, Ed.; Academic Press: New York, 1981.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $205-240 (list).

N-NITROSAMINES

Air Samples
Laboratory Method

Compound	CAS Number
N-Nitrosodi-N-butylamine	924-16-3
N-Nitrosodiethylamine	55-18-5
N-Nitrosodimethylamine	62-75-9
N-Nitrosodiphenylamine	86-30-6
N-Nitrosodi-N-propylamine	621-64-7
N-Nitrosopiperidine	100-75-4
N-Nitrosopyrrolidine	930-55-2

METHOD SUMMARY:

The above N-nitrosamines in air samples can be measured by gas chromatography/mass spectrometry (GC/MS). The samples are collected with trapping concentrators and cartridges. The cartridges are then pre-eluted to remove interfering compounds. After the nitrosamines are eluted they are analyzed by GC/MS using selective ion monitoring.

INTERFERENCES:

Interfering compounds trapped on the cartridges during sampling are removed by the pre-elution step.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished.

REFERENCE:

Marano, R. S.; Updegrove, W. S.; Machen, R.C. "Determination of Trace Levels of Nitrosamines in Air by Gas Chromatography/Low-Resolution Mass Spectrometry," Anal. Chem., 54, 1982, pp. 1947-1951.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $250-325 (list), plus sampling costs.

N-NITROSAMINES

Water Samples
Laboratory Method 1

Compound	CAS Number
N-Nitrosodiethylamine	55-18-5
N-Nitrosodimethylamine	62-75-9
N-Nitrosopiperidine	100-75-4
N-Nitrosopyrrolidine	930-55-2

METHOD SUMMARY:

The above N-nitrosamines in water samples can be measured on a gas chromatograph interfaced with a Thermal Energy Analyzer (GC-TEA). The sample is passed through a carbonaceous adsorbent. The nitrosamines are eluted with dichloromethane and analyzed by GC-TEA. This method was designed for the analysis of tap water.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished.

REFERENCE:

Kimoto, W.; Dooley, C. J.; Carré, J.; Fiddler, W. "Nitrosamines in Tap Water After Concentration by a Carbonaceous Adsorbent," Water Res., 15(9), 1981, pp. 1099-1106.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $150-210 (list).

N-NITROSAMINES

Water Samples
Laboratory Method 2

Compound	CAS Number
N-Nitrosodi-n-butylamine	924-16-3
N-Nitrosodiethylamine	55-18-5
N-Nitrosodimethylamine	62-75-9
N-Nitrosopiperidine	100-75-4
N-Nitrosodi-n-propylamine	621-64-7

METHOD SUMMARY:

The above N-nitrosamines in water samples can be measured on a gas chromatography with an N-nitroso compound-specific thermal energy analysis detector (GC/TEA). The samples are collected in brown bottles and stored at 4°C. The sample is extracted three times with methylene chloride, and the extracts are combined and dried, concentrated, and analyzed by GC/TEA or HPLC/TEA. A high-performance liquid chromatograph (HPLC) equipped with TEA detection may also be used for the analysis.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Accuracy information is furnished. The detection limit for N-nitrosodimethylamine and N-nitrosodiethylamine is about 0.002 micrograms per liter.

REFERENCES:

Fine, D. H.; Rounbehler, D. P. "N-Nitroso Compounds in Water" In Identification and Analysis of Organic Pollutants in Water, Ch. 17; L. H. Keith, Ed.; Ann Arbor Science: Ann Arbor, 1976.

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, J. E. Longbottom, J. J. Lichtenburg, Eds.; Method 607; U.S. EPA: Cincinnati, 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $150-295 (list).

NITROSO COMPOUNDS

No Matrix Given
Laboratory Method

Compound	CAS Number
N-Nitroso-N-Ethylurea	759-73-9
N-Nitroso-N-Methylurea	684-93-5
N-Nitroso-N-Methylurethane	615-53-2

METHOD SUMMARY:

High-pressure liquid chromatography (HPLC) may be used to measure the above nitroso compounds. The sample must first be extracted and concentrated. The extract is hydrolyzed in dilute hydrochloric acid to give nitrite. The nitrite is reacted with naphthylethylenediamine to give a colored dye, which is analyzed by HPLC. The extraction and concentration steps are not necessary when large amounts of nitroso compounds are present. This method does not identify any specific compound, but is specific to all compounds which hydrolyze in dilute hydrochloric acid to yield nitrite. This method is capable of sensitivities of 0.5 nanomoles of injected nitrosamides and may be used for analysis for nitriles, nitrosamides, nitrocarbamates, and alkylnitrites.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

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

REFERENCES:

Singer, G. M.; Singer S. S.; Schmidt, D. G. "A Nitrosamide-Specific Detector for use with High-Pressure Liquid Chromatography," J. Chromatogr., 133, 1977, pp. 59-66.

Heyns, K.; Röper H. "Analysis of N-Nitroso Compounds. II. Separation and Quantitative Determination of N-Nitroso-N-alkylureas and N-Nitroso-N-alkylurethans by High-Speed High-Pressure Liquid Chromatography," J. Chromatogr., 93, 1974, pp. 429-439, (German).

COST INFORMATION:

Cost per sample for analysis by this method is approximately $125-215 (list).

N-NITROSO COMPOUNDS

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
N-Nitrosodi-n-butylamine	924-16-3
N-Nitrosodiethylamine	55-18-5
N-Nitrosodiphenylamine	86-30-6
N-Nitrosodi-n-butylamine	924-16-3
N-Nitrosodi-n-propylamine	621-64-7
N-Nitrosopiperidine	100-75-4
N-Nitrosopyrrolidine	930-55-2

METHOD SUMMARY:

The above N-nitroso compounds in soil/sediment samples can be measured by chemiluminescence. The sample is extracted with methylene chloride, the extract is filtered and then partitioned with water for back-extraction of nitrites. The extract is dried and concentrated. Analysis is by hydrobromic acid-catalyzed denitrosation and subsequent detection of the evolved nitric oxide by its chemiluminescent reaction with ozone. This method is not specific for a particular N-nitroso compound, but measures the total N-nitroso group content of the sample.

INTERFERENCES:

N-Alkylnitrites interfere if not removed. If water is present in the extract during analysis, the detector signal will be severely reduced.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information is furnished. The limit of detection is, on the average, 100 nanograms of N-nitroso compound per kilogram of soil.

REFERENCE:

Drescher, G. S.; Frank, C. W. "Estimation of Extractable N-Nitroso Compounds at the Parts-per-Billion Level," Anal. Chem., 50(14), 1978, pp. 2118-2121.

COST INFORMATION:

Cost information has not been obtained.

NON-HALOGENATED VOLATILE ORGANIC COMPOUNDS

Soil/Sediment Samples
Laboratory Method

Compounds	CAS Number
Acetonitrile	75-05-8
Acrolein	107-02-8
Acrylamide	79-06-1
Acrylonitrile	107-13-1
Carbon Disulfide	75-15-0
Methyl Ethyl Ketone	78-93-3
Methyl Isobutyl Ketone	108-10-1
Paraldehyde	123-63-7

METHOD SUMMARY:

Analysis for the above volatile organic compounds in soil/sediment samples can be performed by gas chromatography. A soil/sediment sample is dispersed in methanol or polyethylene glycol, the solution is purged with an inert gas onto a trap, and the trap is backflushed into a temperature-programmed gas chromatograph with flame ionization detection (GC/FID). Either internal or external standards are used for quantification.

INTERFERENCES:

Samples should be collected in glass containers and immediately sealed and refrigerated.

QUALITY CONTROL:

The analytical system must be demonstrated to be free of interferences by analysis of a method blank each time a set of samples is processed or a change in reagents occurs. Field replicates, laboratory replicates, and fortified samples must be analyzed to demonstrate the precision and accuracy of the method. In cases of doubt concerning the identity of a peak in the chromatogram, confirmatory techniques, such as gas chromatography/mass spectrometry (GC/MS) or second-column confirmation, should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved for Resource Conservation and Recovery Act (RCRA) sample analyses. Limited precision and accuracy information is furnished (Method 603).

REFERENCES:

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 5020, 5030, 8015, 8030; SW-846, U.S. EPA: Washington, July 1982.

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, J. E. Longbottom, J. J. Lichtenberg, Eds., Method 603; EPA-600/4-82-057, U.S. EPA: Cincinnati, 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70-125 (list).

NON-HALOGENATED VOLATILE ORGANIC COMPOUNDS

Water Samples
Laboratory Method

Compounds	CAS Number
Acetonitrile	75-05-8
Acrolein	107-02-8
Acrylamide	79-06-1
Acrylonitrile	107-13-1
Carbon Disulfide	75-15-0
Methyl Ethyl Ketone	78-93-3
Methyl Isobutyl Ketone	108-10-1
Paraldehyde	123-63-7

METHOD SUMMARY:

Analysis of water samples for the above volatile organic compounds can be performed by gas chromatography. A water sample is purged with an inert gas onto a Tenax® trap, and the trap is backflushed into a temperature-programmed gas chromatograph with a flame ionization detector (GC/FID). Either internal or external standards are used for calibration.

INTERFERENCES:

Samples should be collected in glass containers, sealed, and refrigerated immediately.

QUALITY CONTROL:

The analytical system must be demonstrated to be free of interferences by the analysis of a method blank each time a set of samples is processed or a change in reagents occurs. Field replicates, laboratory replicates, and fortified samples must be analyzed to demonstrate the precision and accuracy of the method. In cases of doubt concerning the identity of a peak in the chromatogram, confirmatory techniques, such as gas chromatography/mass spectrometry (GC/MS) or second-column confirmation, should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved for Clean Water Act and Resource Conservation and Recovery Act (RCRA) sample analyses. Precision and accuracy information is furnished (Method 603). The method detection limit for acrolein is 0.6 µg per liter and for acrylonitrile is 0.5 µg per liter.

REFERENCES:

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 5020, 5030, 8015, 8030; SW-846, U.S. EPA: Washington, July 1982.

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, J. E. Longbottom; J. J. Lichtenberg, Eds., Method 603; EPA-600/4-82-057, U.S. EPA: Cincinnati, 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70-115 (list).

NON-VOLATILE ORGANIC COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number
Benzyl Chloride	100-44-7
Captan	133-06-2
Chlorpyrifos	2921-88-2
Diazinon	333-41-9
Dichlobenil	1194-65-6
Dichlone	117-80-6
Dichlorovos	62-73-7
2,4-Dimethylphenol	105-67-9
o-Dinitrobenzene	528-29-0
p-Dinitrobenzene	100-25-4
Disulfoton	298-04-4
Ethion	563-12-2
Kelthane	115-32-2
Kepone	143-50-0
Malathion	121-75-5
Methoxychlor	72-43-5
Methyl Parathion	298-00-0
Mevinphos	7786-24-7
Nitrotoluene	1321-12-6
Parathion	56-38-2
Propargite	2312-35-8
Quinoline	91-22-5
Resorcinol	108-46-3
Trichlorfon	52-68-6

METHOD SUMMARY:

Analysis of water samples for the above non-volatile organic compounds can be performed by gas chromatography/mass spectrometry (GC/MS). A 1.0-liter sample is serially extracted at acidic and basic pH. The base/neutral extract is obtained by extraction of the sample with methylene chloride at pH 11. The remaining aqueous solution is extracted with methylene chloride at pH 2 to obtain the acidic extract. The two fractions are each concentrated in a Kuderna-Danish apparatus and analyzed by GC/MS.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

Fortified samples should be analyzed to determine recovery and measurement precision.

EPA/TECHNICAL STATUS:

This method is not EPA-approved, but is a modification of EPA Method 625, which is approved. This method will detect concentrations as low as approximately 0.2 milligrams per liter. Precision information is furnished.

REFERENCE:

Spingarn, N. E.; Northington, D. J.; Pressely, T. "Analysis of Non-Volatile Organic Hazardous Substances by GC/MS," J. Chromatogr. Sci., 20, December 1982, pp. 571-574.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $320-490 (list).

ORGANIC ACIDS

Water Samples
Laboratory Method 1

Compound	CAS Number
Acetic Acid	64-19-7
Butyric Acid	107-92-6
Formic Acid	64-18-6
Propionic Acid	79-09-4

METHOD SUMMARY:

The above organic acids of low molecular mass (i.e., C_1 to C_4) can be determined in water by gas chromatography. The acids are converted to their benzyl esters and injected into a gas chromatograph equipped with a flame ionization detector (GC/FID). This method is used over the range of 1.0 to 2.5 milligrams of acid per liter in a 10-milliliter water sample.

INTERFERENCES:

High concentrations of acids other than those to be determined will interfere.

QUALITY CONTROL:

Standards are analyzed along with the samples.

EPA/TECHNICAL STATUS:

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

REFERENCE:

Bethge, P. O.; Lindström, K. "Determination of Organic Acids of Low Relative Molecular Mass (C_1 to C_4) in Dilute Aqueous Solutions," Analyst, 99(2), 1974, pp. 137-142.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $90 (list), plus sampling costs.

ORGANIC ACIDS

Water Samples
Laboratory Method 2

Compound	CAS Number
Acetic Acid	64-19-7
Benzoic Acid	65-85-0
Ethylenediamine-tetraacetic Acid	60-00-4
Phenol	108-95-2
Phthalic Anhydride	85-44-9

METHOD SUMMARY:

Analysis of water samples for the above organic acids can be performed by ion chromatography. Separation is achieved by use of a cross-linked dextran geltype cation exchanger and a tetramethylammonium chloride eluent, and detection is by use of a flow-cell-equipped spectrophotometer or by derivatization and determination as the hydroxamic acid. Aromatic acids are detected directly, while ethylenediaminetetraacetic acid is detected as the cobalt complex, and aliphatic acids are derivatized.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved for any regulatory program. It has been used to separate aqueous standard solutions. No precision or accuracy information is furnished.

REFERENCE:

Tokunaga, Y.; Waki, H.; Ohashi, S. "Elution Behavior of Organic Acids in Donnan Exclusion Chromatography," J. Liq. Chromatogr., 6(2), 1983, pp. 271-280.

COST INFORMATION:

No cost information has been obtained.

ORGANIC COMPOUNDS

Water Samples
Laboratory

Compound	CAS Number	Compound	CAS Number
Acenaphthene	83-32-9	Furan	110-00-9
Acetaldehyde	75-07-0	Methacrylonitrile	126-98-7
Acetone	67-64-1	Methyl Ethyl Ketone	78-93-3
Acetonitrile	75-05-8	Methyl Isobutyl Ketone	108-10-1
Aniline	62-53-3	Naphthalene	91-20-3
Benzene	71-43-2	Phenol	85-01-8
Benzonitrile	100-47-0	Propanenitrile	107-12-0
Butyric Acid	107-92-6	Pyridine	110-86-1
Cresol	1319-77-3	Quinoline	91-22-5
m-Cresol	108-39-4	Styrene	100-42-5
o-Cresol	95-48-7	Toluene	108-88-3
p-Cresol	106-44-5	Xylene	1330-20-7
2,4-Dimethylphenol	105-67-9	m-Xylene	108-38-3
Ethylbenzene	100-41-4	o-Xylene	95-47-6
Fluorene	86-73-7	p-Xylene	106-42-3

METHOD SUMMARY:

Analysis of water samples for the above organic compounds can be performed by gas chromatography/mass spectrometry (GC/MS). A sample is purged with inert gas through a Tenax® trap and the trap is thermally desorbed into a gas chromatograph/mass spectrometer (GC/MS) for characterization and measurement of the volatile components of the sample. The sample is then extracted with Freon TF, and the extract is concentrated, fractionated, and the fractions analyzed by GC/MS. Gas chromatography/Fourier transform infrared spectrometry (GC/FTIR) can be used to aid in identification of compounds as needed.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

Blanks should be used to monitor materials used in purification. All equipment and reagents should be demonstrated to be free of interferences before being used in this method. The GC/MS system must be calibrated daily, and a twelve-component reference compound solution is used to evaluate the performance of the GC/MS each day.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The lower limit of detection is 0.1 microgram per liter. Precision information is furnished.

REFERENCE:

Pellizzari, E. D.; Castillo, N. P.; Willis, S.; Smith, D.; Bursey, J. T., "Identification of Organic Components in Aqueous Effluents from Energy-Related Processes," Measurement of Organic Pollutants in Water and Wastewater, C. E. Van Hall, Ed.; STP 686, American Society for Testing and Materials: Philadelphia, 1979, pp. 256-274.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $250-325 (list).

ORGANIC VAPORS

Air Samples
Laboratory Method

Compound	CAS Number
iso-Butyl Alcohol	78-83-1
Chloroform	67-66-3
Ethyl Acrylate	140-88-5
Ethylbenzene	100-41-4
Ethyl Ether	60-29-7
Methylene Chloride	75-09-2
Pyridine	110-86-1
Tetraethyl Lead	78-00-2
1,1,2-Trichloroethane	79-00-5

METHOD SUMMARY:

Analysis for the above organic vapors in air can be performed by gas chromatography. Air samples are collected by aspirating 2 to 50 liters of air through activated charcoal sampling tubes, followed by desorption of the analytes with carbon disulfide (ethyl acetate for ethyl ether and methylene chloride for pyridine) and analysis of the resulting solution by gas chromatography with flame ionization detection (GC/FID).

INTERFERENCES:

High humidity can greatly reduce the adsorptive capacity of the charcoal. Peak superimposition can be overcome by changes in gas chromatography columns or operating conditions. No specific interferences are reported.

QUALITY CONTROL:

Desorption efficiency must be determined for each set of samples and must be above 75 percent. Field blanks must be processed with each set of samples. Calculations should include the desorption efficiency. The collecting system must be tested for breakthrough with each sample, and if breakthrough has occurred, results should not be used.

EPA/TECHNICAL STATUS:

This method has been approved by the National Institute for Occupational Safety and Health (NIOSH), and has been validated by that agency. Precision and accuracy information is furnished.

REFERENCE:

American Society for Testing and Materials, ASTM Standards on Chromatography, D 3686-78, D 3687-78; ASTM: Philadelphia, 1981, pp. 537-555.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70-90 (list), plus sampling costs.

ORGANOCHLORINE AND ORGANOPHOSPHORUS PESTICIDES

Water Samples
Laboratory Method

Compounds	CAS Number	Compound	CAS Number
Aldrin	309-00-2	Heptachlor	76-44-8
α-BHC	319-84-6	Heptachlor Epoxide	1024-57-3
β-BHC	319-85-7	Hexachlorobenzene	118-74-1
δ-BHC	319-86-8	Hexachlorohexahydro-endo, endo-dimethanonaphthalene	465-73-6
Captan	133-06-2	Kelthane	115-32-2
Chlordane	57-74-9	Kepone	143-50-0
Chlorpyrifos	2921-88-2	Lindane	58-89-9
DDD	72-54-8	Malathion	121-75-5
DDE	72-55-9	Methoxychlor	72-43-5
DDT	50-29-3	Methyl Parathion	298-00-0
2,4-D Esters	94-11-1	Mevinphos	7786-34-7
	94-79-1	Naled	300-76-5
	94-80-4	Pentachloronitrobenzene	82-68-8
	1320-18-9	Phorate	298-02-2
	1928-38-7	Polychlorinated Biphenyls	11097-69-1
	1928-61-6		11096-82-5
	1929-73-3		1336-36-3
	2971-38-2		12674-11-2
	25168-26-7		11104-28-2
	53467-11-1		11141-16-5
Diazinon	333-41-5		53469-21-9
Dieldrin	60-57-1		12672-29-6
Dimethoate	60-51-5	2,4,5-T Esters	93-79-8
Disulfoton	298-04-4		2545-59-7
α-Endosulfan	959-98-8		61792-07-2
β-Endosulfan	33213-65-9		1928-47-8
Endrin	72-20-8		25168-15-4
		Toxaphene	8001-35-2

METHOD SUMMARY:

Gas chromatography may be used for the analysis of water samples for the above organochlorine and organophosphorus pesticides. A 500-milliliter sample is extracted with methylene chloride and the extract is dried, concentrated, separated into fractions on a deactivated silica-gel column, and the fractions analyzed by gas chromatography, using an electron capture detector (GC/ECD) for organochlorine compounds and a flame photometric detector (GC/FPD) for organophosphorus compounds.

INTERFERENCES:

Organochlorine pesticide-containing samples must be refrigerated at 2° to 4°C and analyzed within 1 week. Organophosphorus pesticide-containing samples must be frozen immediately after collection and analyzed within 4 days.

QUALITY CONTROL:

Reagent blanks and standards should be analyzed with each sample or set of samples. Further confirmatory techniques, such as gas chromatography/mass spectrometry or thin-layer chromatography, should be used when uncertainty exists concerning the identity of a compound.

EPA/TECHNICAL STATUS:

This method has been evaluated in an EPA study, but is not EPA-approved for any regulatory program. Precision and accuracy information is furnished.

REFERENCES:

American Society for Testing and Materials, ASTM Standards on Chromatography, D 3086-79; ASTM: Philadelphia, 1981, pp. 326-349.

U.S. Environmental Protection Agency, Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples, Section 10, EPA-600/8-80-038, U.S. EPA: Cincinnati, June 1980.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $150-215 (list).

ORGANOCHLORINE PESTICIDES

Water Samples
Laboratory Method

Compounds	CAS Number	Compound	CAS Number
Aldrin	309-00-2	Dieldrin	60-57-1
α-BHC	319-84-6	Endosulfan	115-29-7
β-BHC	319-85-7	Endrin	72-20-8
δ-BHC	319-86-8	Heptachlor	76-44-8
Captan	133-06-2	Heptachlor Epoxide	1024-57-3
Chlordane	57-74-9	Lindane	58-89-9
DDD	72-54-8	Methoxychlor	72-43-5
DDE	72-55-9	Pentachloronitrobenzene	82-68-8
DDT	50-29-3	Toxaphene	8001-35-2

METHOD SUMMARY:

The above organochlorine pesticides in water samples may be measured by gas chromatography. The sample is extracted with methylene chloride or ethyl ether/hexane (15/85), interferences are removed if necessary, and the extract is analyzed by gas chromatography with electron capture, microcoulometric, or electrolytic conductivity detection.

INTERFERENCES:

Reagents and glassware can cause discrete artifacts or elevated baselines. Polychlorinated aromatics, phthalate esters, and organophosphate pesticides may interfere. Interferences can be minimized by such treatments as liquid-liquid partitioning, macro and micro chromatography, and thin-layer chromatography.

QUALITY CONTROL:

When the volume of sample permits, one set of duplicates and one spiked sample should also be analyzed as a quality control check.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The method sensitivity limit (ASTM) ranges from 1 to 10 nanograms per liter for single-component pesticides, and 50 to 1000 nanograms per liter for multi-component pesticides, using a 1-liter sample. Precision and accuracy information is furnished.

REFERENCES:

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, D 3086-79; ASTM: Philadelphia, 1983, pp. 178-201.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Analysis of Water Samples for Organochlorine Pesticides/Polychlorinated Biphenyls," EPA-600/54-84-038, pp. III-186-III-201; U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $130-175 (list).

ORGANOCHLORINE PESTICIDES AND PCB's

Soil/Sediment Samples
Laboratory Method 1

Compound	CAS Number	Detection Limit (μg/L)*
Aldrin	309-00-2	0.004
α-BHC	319-84-6	0.004
β-BHC	319-85-7	0.006
δ-BHC	319-86-8	0.004
Chlordane	57-74-9	0.014
DDD	72-54-8	0.012
DDE	72-55-9	0.004
DDT	50-29-3	0.012
Dieldrin	60-57-1	0.002
Endosulfan	115-29-7	--
α-Endosulfan	959-98-8	0.014
β-Endosulfan	33213-65-9	0.004
Endosulfan Sulfate	1031-07-8	0.066
Endrin	72-20-8	0.006
Endrin Aldehyde	7421-93-4	0.023
Heptachlor	76-44-8	0.004
Heptachlor Epoxide	1024-57-3	0.083
Kepone	143-50-0	--
Lindane	58-89-9	0.009
Methoxychlor	72-43-5	0.176
Polychlorinated	1336-36-3	--
Biphenyls	12674-11-2	--
	11104-28-2	--
	11141-16-5	--
	53469-21-9	--
	12672-29-6	--
	11097-69-1	--
	11096-82-5	--
Toxaphene	8001-35-2	0.24

METHOD SUMMARY:

Analysis of soil/sediment samples for the above organochlorine pesticides and PCB's can be performed by gas chromatography. A soil/sediment sample is extracted at neutral pH with dichloromethane, hexanelacetone (1/1), or other designated solvent mixture and the extract is dried, cleaned up on a Florisil® column using appropriate elution mixtures, solvent-exchanged into hexane if necessary, concentrated, and analyzed by gas chromatography with electron-capture detection (GC/ECD). Quantification is performed using either external or internal standards, and an aliquot of each sample is spiked to determine recovery and detection limits.

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* From Method 8080 (U.S. EPA); μg/liter of liquid extract.

INTERFERENCES:

Phthalate esters interfere, so that all contact with non-Teflon® plastics should be avoided. Elemental sulfur interferes and can be removed by treatment with mercury or copper.

QUALITY CONTROL:

The gas chromatographic system must be demonstrated to be free of interferences by analysis of a method blank with each set of samples or change in reagents. Laboratory replicates, field replicates, and duplicate samples should be processed and analyzed to ascertain the precision and accuracy of the method. Where doubt exists concerning the identification of a peak in the chromatogram, confirmatory techniques, such as gas chromatography/mass spectrometry (GC/MS) or second-column confirmation, should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved under the Clean Water Act and the Resource Conservation and Recovery Act (RCRA) and is in use in the CERCLA program. Precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Invitation for Bid, "Organics Analysis, Multi-Media, Multi-Concentration, GC/MS with GC Screen," Solicitation Number WA84A-266; U.S. EPA; Washington, June 15, 1984.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3510, 3520, 8080; SW-846, U.S. EPA: Washington, July 1982.

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 608; EPA-600/4-82-057, U.S. EPA: Washington, 1982.

U.S. Environmental Protection Agency, Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol, and Pesticides in Water and Wastewater, U.S. EPA: Cincinnati, 1978, pp. 7-24.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Analysis of Sediment Samples for Polychlorinated Biphenyls," pp. III-202 to III-210; EPA-600/84-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

ASTM STP 573, American Society for Testing and Materials, Philadelphia, 1975, p. 206.

COST INFORMATION:

Cost per sample for the GC screen is approximately $90-150 (list), $60 (bid); the cost of GC/MS confirmation is approximately $150-200 (list), $137-177 (bid); the cost of second-column confirmation is approximately $80 (list).

ORGANOCHLORINE PESTICIDES AND PCB's

Soil/Sediment Samples
Laboratory Method 2

Compound	CAS Number
Aldrin	309-00-2
DDE	72-55-9
DDT	50-29-3
Heptachlor	76-44-8
Hexachlorobenzene	118-74-1
Hexachlorohexahydro-endo, endo-dimethano-naphthalene	465-73-6
Polychlorinated Biphenyls (PCB's)	1336-36-3
	12674-11-2
	53469-21-9
	11104-28-2
	12672-29-6
	11141-16-5
	11097-69-1
	11096-82-5

METHOD SUMMARY:

The above organochlorine pesticides and polychlorinated biphenyls in soil/sediment samples can be measured by gas chromatography with electron-capture detection (GC/ECD). The sample, with water added, is extracted with acetone, concentrated, partitioned into water, extracted with petroleum ether, then dried and concentrated. The extract is added to an alumina column, which is eluted with petroleum ether. The concentrated eluate is added to a silica gel column, which is also eluted with petroleum ether. After concentration and removal of sulfur, the sample is analyzed by capillary column GC/ECD. When different eluents are used in the columns, additional organochlorine pesticides can be determined.

INTERFERENCES:

PCB's can interfere with analysis for heptachlor, aldrin, and DDT.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information is furnished. The limit of detection is 0.01 milligrams of analyte per kilogram of soil/sediment, on a dry-weight basis.

REFERENCE:

Wegman, R. C. C.; Hofstee, A. W. M. "Determination of Organochlorines in River Sediment by Capillary Gas Chromatography," Water Res., 16, 1982, pp. 1265-1272.

COST INFORMATION:

Cost per sample for this analysis is approximately $180-200 (list).

ORGANOCHLORINE PESTICIDES AND PCB's

Water Samples
Laboratory Method 1

Compound	CAS Number	Detection Limit (μg/L)*
Aldrin	309-00-2	0.004
α-BHC	319-84-6	0.003
β-BHC	319-85-7	0.006
δ-BHC	319-86-8	0.009
Chlordane	57-74-9	0.011
DDD	72-54-8	0.004
DDE	72-55-9	0.004
DDT	50-29-3	0.004
Dieldrin	60-57-1	0.002
Endosulfan	115-29-7	0.014
α-Endosulfan	959-98-8	0.014
β-Endosulfan	33213-65-9	0.004
Endosulfan Sulfate	1031-07-8	0.066
Endrin	72-20-8	0.006
Endrin Aldehyde	7421-93-4	0.023
Heptachlor	76-44-8	0.003
Heptachlor Epoxide	1024-57-3	0.083
Hexachlorobenzene	118-74-1	--
Lindane	58-89-9	0.004
Polychlorinated	1336-36-3	--
Biphenyls	12674-11-2	--
	11104-28-2	--
	11141-16-5	--
	53469-21-9	--
	12672-29-6	--
	11097-69-1	--
	11096-82-5	--
Toxaphene	8001-35-2	--

METHOD SUMMARY:

Analysis for the above organochlorine pesticides and polychlorinated biphenyls in water samples can be performed by gas chromatography. A 1-liter portion of sample is mixed with surrogates and extracted with methylene chloride at a pH of 5 to 9, using a separatory funnel or a continuous extractor. The extract is dried, solvent-exchanged to hexane, and concentrated. Cleanup techniques are optional. A portion of the extract is screened by gas chromatography with an electron capture detector (GC/ECD). The presence of pesticides is qualitatively confirmed by a second column or by gas chromatography/mass spectrometry (GC/MS). If the screening results are positive, a 50-milliliter portion of the sample is diluted to 1 liter prior to extraction. The extract

* From EPA Method 608

is then analyzed as described above. Quantification is accomplished on a packed column, whereas confirmation can be on either a packed column or a capillary column.

INTERFERENCES:

Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing equipment. These materials must be routinely demonstrated to be free from laboratory interferences by analyzing reagent blanks. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences may vary considerably from source to source.

QUALITY CONTROL:

Pesticides are analyzed by a two-column confirmatory technique. The analytes must be quantified by packed column GC. The extract is analyzed using external standards with at least 3 concentrations. All single-peak pesticides may be in one calibration mixture provided that a 25-percent or greater resolution can be achieved between each compound. All multicomponent standards must be in separate calibration solutions except Aroclors 1016/1260. Both columns used for analysis must meet the same quality control requirements: Prior to sample analysis, analyte retention time windows must be established for compound identification. DDT and endrin degradation must not exceed 5 percent and 10 percent, respectively. DDT must elute at greater than 12 minutes when injected onto the packed column. The retention time shift for dibutyl chlorendate during 8 hours of analysis must not exceed 2 percent relative difference (RPD) for packed column analysis and 0.3 percent for FSCC analysis.

EPA/TECHNICAL STATUS:

This method is in use in the CERCLA program and is approved for use under the Resource Conservation Recovery Act and the Clean Water Act. Precision and accuracy information is furnished (ASTM STP 573 and D 3086-79 and EPA Methods 608 and 8080).

REFERENCES:

U.S. Environmental Protection Agency, Invitation for Bid, "Organics Analysis, Multi-Media, Multi-Concentration, GC/MS with Medium Level GC Screen," Solicitation Number WA84A-266; U.S. EPA: Washington, DC, June 15, 1984.

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, J. E. Longbottom, J. J. Lichtenberg, Eds., Method 608; EPA-600/4-82-057, U.S. EPA: Cincinnati, 1982.

Pressley, T. A.; Longbottom, J. E. "The Determination of Organohalide Pesticides and PCBs in Industrial and Municipal Wastewater," Method 617; EPA-600/4-82-006, U.S. EPA: Cincinnati, 1982, 35 pp.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 2nd ed., Methods 3510, 3520, 8080; SW-846, U.S. EPA Washington, 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $125-150 (list).

ORGANOPHOSPHORUS AND CARBAMATE INSECTICIDES

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Carbofuran	1563-66-2
Dichlorvos	62-73-7
Methomyl	16752-77-5
Trichlorfon	52-68-6

METHOD SUMMARY:

Analysis for the above compounds in soil/sediment samples can be performed by gas chromatography. The analyte is extracted from the soil, cooled to precipitate water and waxes, and analyzed by GC with an alkali flame ionization detector (GC/AFID). On-column acetylation with acetic anhydride is used for trichlorfon. Mass spectrometry (MS) is used to confirm GC results.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished.

REFERENCE:

Holland, P. T. "Routine Methods for Analysis of Organophosphorus and Carbamate Insecticides in Soil and Ryegrass," Pestic. Sci., 8, 1977, pp. 354-358.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $125-170 (list); the cost of GC/MS confirmation is approximately $150-200 (list).

ORGANOPHOSPHORUS COMPOUNDS

Air Samples
Laboratory Method

Compound	CAS Number
Chlorpyrifos	2921-88-2
Diazinon	333-41-5
Dichlorvos	62-73-7
Malathion	121-75-5
Methyl Parathion	298-00-0
Parathion	56-38-2

METHOD SUMMARY:

Analysis for the above airborne organophosphorus compounds can be performed by passage of 1000 liters of air through a polyurethane-foam sampling tube, followed by Soxhlet extraction of the foam, and analysis of the extract by gas chromatography with electron capture detection (GC/ECD). When concentration levels are high enough, confirmation of peak identification can be performed by gas chromatography/mass spectrometry (GC/MS).

INTERFERENCES:

Phthalate esters and polychlorinated naphthalenes can interfere, and use of non-TFE plastics must be avoided.

QUALITY CONTROL:

Solvents, reagents, glassware, and polyurethane plugs must be shown to be free of interfering artifacts. A field blank must be analyzed with each set of samples, and both a quality control standard and a method blank should be analyzed with each set of 5 to 15 samples.

EPA/TECHNICAL STATUS:

This method has been evaluated by the Environmental Protection Agency, but is not approved for any specific regulatory program. The method is applicable to a range from 50 nanograms per cubic meter to 1000 nanograms per cubic meter. No precision or accuracy information is furnished.

REFERENCE:

U.S. Environmental Protection Agency, Guidelines for Air Monitoring at Hazardous Waste Sites, Assignment 26, Appendix B, Contract 68-02-3168; U.S. EPA: Research Triangle Park, North Carolina, May 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $115 (list); the cost for GC/MS confirmation is approximately $150-200 (list). Sampling costs are additional.

ORGANOPHOSPHORUS PESTICIDES

Air Samples
Laboratory Method

Compound	CAS Number
Chlorpyrifos	2921-88-2
Coumaphos	56-72-4
Diazinon	333-41-5
Dichlorvos	62-73-7
Disulfoton	298-04-4
Guthion	86-50-0
Malathion	121-75-5
Methyl Parathion	298-00-0
Mevinphos	7786-34-7
Naled	300-76-5
Phorate	298-02-2

METHOD SUMMARY:

Analysis of air for the above organophosphorus pesticides can be performed by highvolume air sampling, followed by Soxhlet extraction of the analyte from the sampling medium and concentration and analysis of the resulting solution by gas chromatography with flame photometric detection (GC/FPD).

INTERFERENCES:

Elemental sulfur can interfere with the determination, but can be removed by the addition of copper powder.

QUALITY CONTROL:

Retention time efficiency of standards must be determined for each pesticide to be analyzed by at least 6 replicate tests.

EPA/TECHNICAL STATUS:

This method is not EPA-approved, but the gas chromatographic analysis applied to water and wastewater extracts is approved under NPDES. Precision and accuracy information is furnished.

REFERENCES:

Sherma, J.; Beroza, M. Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples, Section 8B; EPA-600/8-80-038, U.S. Environmental Protection Agency: Research Triangle Park, North Carolina, 1980.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Determination of Organophosphorus Pesticides in Air," EPA-600/S4-84-038, pp. III-247 to III-250; U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $165 (list), plus sampling costs.

ORGANOPHOSPHORUS PESTICIDES

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number	Estimated Detection Limit (µg/L)
Chlorpyrifos	2921-88-2	0.3*
Coumaphos	56-72-4	1.5*
Diazinon	333-41-5	0.012**
Dichlorvos	62-73-7	0.1*
Dimethoate	60-51-5	---
Disulfoton	298-04-4	0.20*
Ethion	563-12-2	---
Guthion	86-50-0	1.5*
Malathion	121-75-5	---
Mevinphos	7786-34-7	0.3*
Naled	300-76-5	---
Parathion	56-38-2	0.015**
Phorate	298-02-2	0.15*

METHOD SUMMARY:

Gas chromatography can be used to analyze soil/sediment samples for the above organophosphorus pesticides. A soil/sediment sample is extracted at neutral pH with dichloromethane or with hexane-isopropanol, and the extract is dried, concentrated, and analyzed by gas chromatography using a nitrogen/phosphorus detector (GC/NPD), a flame photometric detector (GC/FPD), or (as applicable) a halogenspecific detector (GC/HSD).

INTERFERENCES:

The use of Florisil® column or silica-gel column cleanup is not recommended for removal of interferences, but the use of GC/HSD for naled and dichlorvos, or of GC/FPD, minimizes the effects of the presence of many interferents. Elemental sulfur can interfere with GC/FPD.

QUALITY CONTROL:

Either internal or external standards may be used for quantification, and an aliquot of each sample must be spiked to determine recoveries and detection limits. The analytical system must be demonstrated to be free of interferences by the analysis of a method blank with each set of samples or change in reagents. Field laboratory replicates should be processed and analyzed to assess the precision of the sampling and analysis methods. Fortified samples should be

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* As cited in Method 622
** As cited in Method 614

analyzed to ascertain the accuracy of the analysis. In cases where uncertainty exists concerning the identification of a peak in the chromatogram, confirmatory techniques, such as gas chromatography/mass spectrometry (GC/MS) or second-column confirmation, should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved for the Resource Conservation and Recovery Act (RCRA) sample analyses. Precision and accuracy information is furnished.

REFERENCES:

Pressley, T. A.; Longbottom, J. E. "The Determination of Organophosphorous Pesticides in Industrial and Municipal Wastewater," Method 614; U.S. EPA: Cincinnati, 1982, 21 pp.

Pressley, T. A.; Longbottom, J. E. "The Determination of Organophosphorous Pesticides in Industrial and Municipal Wastewater," Method 622; U.S. EPA: Cincinnati, 1982, 35 pp.

U.S. Environmental Protection Agency, Test Methods for Evaluating of Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3540, 3550, 8140; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Determination of Organophosphorus Pesticides in Air," EPA-600/S4-84-038, pp. III-247 to III-250; U.S. Environmental Protection Agency: Las Vegas, 1984.

Tonan, J. A. Design and Development Branch, U.S. EPA, Toxicant Analysis Center, Private Communication to Edward Kantor, U.S. EPA-EMSL, Las Vegas, Nevada.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $165-210 (list).

ORGANOPHOSPHORUS PESTICIDES

Water Samples
Laboratory Method 1

Compound	CAS Number	Estimated Detection Limit (µg/L)
Chlorpyrifos	2921-88-2	0.3*
Coumaphos	56-72-4	1.5*
Diazinon	333-41-5	0.012**
Dichlorvos	62-73-7	0.1*
Dimethoate	60-51-5	---
Disulfoton	298-04-4	0.20*
Guthion	86-50-0	1.5*
Malathion	121-75-5	---
Mevinphos	7786-34-7	0.3*
Naled	300-76-5	---
Parathion	56-38-2	0.015**
Phorate	298-02-2	0.15*

METHOD SUMMARY:

Analysis for the above organophosphorus pesticides in water samples can be performed by gas chromatography. A water sample is extracted at neutral pH with methylene chloride, and the extract is dried, concentrated, and analyzed by gas chromatography using a nitrogen/phosphorus detector (GC/NPD), a flame photometric detector (GC/FPD), or (as applicable) a halogen-specific detector (GC/HSD).

INTERFERENCES:

The use of Florisil® column or silica-gel column cleanup is not recommended for removal of interferences, but the use of GC/HSD for naled and dichlorvos, or of GC/FPD, minimizes the effects of the presence of many interferents. Elemental sulfur can interfere with GC/FPD.

QUALITY CONTROL:

Either internal or external standards may be used for quantification, and an aliquot of each sample must be spiked to determine recoveries and detection limits. The analytical system must be demonstrated to be free of interferences by analysis of a method blank with each set of samples or with each change in reagents, and field and laboratory replicates should be processed and analyzed to assess the precision of the sampling and analysis methods. Fortified samples should be analyzed to ascertain the accuracy of the analysis. In cases where uncertainty exists concerning the identification of a peak in the chromatogram, confirmatory techniques, such as gas chromatography/mass spectrometry (GC/MS) or second-column confirmation, should be used.

*As cited in Method 622
**As cited in Method 614

EPA/TECHNICAL STATUS:

This method is EPA-approved for the Resource Conservation and Recovery Act (RCRA) and the Clean Water Act. Precision and accuracy information is furnished.

REFERENCES:

Pressley, T. A.; Longbottom, J. E., "The Determination of Organophosphorous Pesticides in Industrial and Municipal Wastewater," Method 614; U.S. EPA: Cincinnati, 1982, 21 pp.

Pressley, T. A.; Longbottom, J. E., "The Determination of Organophosphorous Pesticides in Industrial and Municipal Wastewater," Method 622; U.S. EPA: Cincinnati, 1982, 35 pp.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3510, 3520, 8140; SW-846; U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Determination of Organophosphorus Pesticides in Air," EPA-600/S4-84-038, pp. III-247 to III-250; U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $165-210 (list).

ORGANOPHOSPHORUS PESTICIDES

Water Samples
Laboratory Method 2

Compound	CAS Number
Coumaphos	56-72-4
Diazinon	333-41-5
Dichlorvos	62-73-7
O,O-Diethyl O-Pyrazinyl Phosphorothioate	297-97-2
Dimethoate	60-51-5
Disulfotos	298-04-4
Ethion	563-12-2
Mevinphos	7786-34-7
Parathion	56-38-2
Phorate	298-02-2
Trichlorfon	57-68-6

METHOD SUMMARY:

The above organophosphorus pesticides in water samples can be measured by gas chromatography (GC) with phosphorus-selective flame thermionic detection. In most instances, the sample is extracted with chloroform, and cleanup, if necessary, is performed by passing the solvent containing the analyte through a Nuchar® carbon column, and eluting with chloroform. Coumaphos and dichlorvos require an alternative cleanup procedure because they are retained on Nuchar. The eluate is solvent-exchanged with acetone or ethanol, and the resulting solution is analyzed by GC.

INTERFERENCES:

Volatile nitrogen-containing compounds may cause false peaks if they are present in sufficient concentration.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Accuracy information is furnished. The limit of detection is approximately 0.1 microgram per liter.

REFERENCE:

Askew, V.; Ruzicka, J. H.; Wheals, B. B. "A General Method for Determination of Organophosphorus Pesticide Residues in River Waters and Effluents by Gas, Thin-layer, and Gel Chromatography," Analyst, 94, 1969, pp. 275-283.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $195-235 (list).

PARTICULATE AROMATIC HYDROCARBONS

Air Samples
Laboratory Method

Compound	CAS Number
Anthracene	120-12-7
Benzo[a]anthracene	56-55-3
Benzene	71-43-2
Benzo[b]fluoranthene	205-99-2
Benzo[k]fluoranthene	207-08-9
Benzo[ghi]perylene	191-24-2
Benzo[a]pyrene	50-32-8
Chrysene	218-01-9
DDD	72-54-8
DDE	72-55-9
DDT	50-29-3
Dibenz[a,b]anthracene	53-70-3
Fluoranthene	206-44-0
Fluorene	86-73-7
Naphthalene	91-20-3
Phenanthrene	85-01-8
Polychlorinated Biphenyls (Aroclor 1260)	11096-82-5
Pyrene	129-00-0

METHOD SUMMARY:

The above particulate aromatic hydrocarbons in air samples may be measured by high-performance liquid chromatography (HPLC). The particulates are collected on a glass-fiber filter, then extracted ultrasonically in the presence of silica powder. The hydrocarbons in the extract are separated by HPLC on a column of Porasil II with a nonpolar solvent, and the absorbance is measured by a UV detector at 254 nm.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Replicates and a blank should be processed.

EPA/TECHNICAL STATUS:

This method is classified as proposed by the National Institute for Occupational Safety and Health (NIOSH). The minimum detection limit for benzo[a]pyrene is approximately 3.3 nanograms per cubic meter of air, using a 1500-cubic meter sample. 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 206; Publication No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $150 (list).

PESTICIDE RESIDUES

Air Samples
Laboratory Method

Compound	CAS Number
Aldrin	309-00-2
α-BHC	319-84-6
β-BHC	319-85-7
Carbaryl	63-25-2
Carbofuran	1563-66-2
DDD	72-54-8
DDE	72-55-9
DDT	50-29-3
Diazinon	333-41-9
Dieldrin	60-57-1
Endrin	72-20-8
Ethion	563-12-2
Heptachlor Epoxide	1024-57-3
Lindane	58-89-9
Malathion	121-75-5
Mercaptodimethur	2032-65-7
Methyl Parathion	298-00-0
Mexacarbate	315-18-4
Parathion	56-38-2

METHOD SUMMARY:

Gas chromatography may be used for the analysis of the above chlorinated, organophosphate, and N-methyl carbamate insecticides in air. A known volume of air is drawn through an impinger containing 100 milliliters of ethylene glycol, followed by extraction of the resulting solution with methylene chloride and fractionation of the extract on silica gel. The fraction containing chlorinated compounds is analyzed with a gas chromatograph equipped with an electron-capture detector (GC/ECD); the fraction containing phosphate compounds is analyzed with a gas chromatograph equipped with a flame photometric detector (GC/FPD). The carbamates are derivatized with pentafluoropropionic anhydride, and the products are analyzed with a gas chromatograph equipped with an electron capture detector (GC/ECD).

INTERFERENCES:

Lindane may elute in Fractions I and II, but can be resolved and quantified in both. Diazinon, if present in Fraction III, would co-elute with carbaryl under the conditions used. The derivatization step will effectively remove this interference.

QUALITY CONTROL:

Method blanks and standards are prepared and processed along with the samples.

EPA/TECHNICAL STATUS:

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

REFERENCE:

Sherma, J.; Shafik, T. M. "A Multiclass, Multiresidue Analytical Method for Determining Pesticide Residues in Air," Arch. Environ. Contam. Toxicol., 3(1), 1975, pp. 55-71.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $115-180 (list), plus sampling costs.

PESTICIDES

Water Samples
Laboratory Method 1

Compound	CAS Number
Aldrin	309-00-2
α-BHC	319-84-6
β-BHC	319-85-7
δ-BHC	319-86-8
Captan	133-06-2
DDE	72-55-9
DDT	50-29-3
Dichlorvos	62-73-7
Dieldrin	60-57-1
Parathion	56-38-2
Diethyl-p-nitrophenyl Phosphate	311-45-5
α-Endosulfan	959-98-8
β-Endosulfan	33213-65-9
Endrin	72-20-8
Ethion	563-12-2
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3
Lindane	58-59-9
Methoxychlor	72-43-5
Methyl Parathion	298-00-0
Parathion	56-38-2

METHOD SUMMARY:

The above pesticides in water samples can be measured by gas chromatography with electron capture detection (GC/ECD). For good quantitative performance, the column, OV-17, a phenyl methyl silicone, should be saturated with microgram quantities of the compounds to be separated.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished.

REFERENCE:

Leoni, V.; Puccetti, G. "Gas-Liquid Chromatography of Pesticides on OV-17 Stationary Phase," J. Chromatogr., 43, 1969, pp. 388-391.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70-100 (list).

PESTICIDES

Water Samples
Laboratory Method 2

Compound	CAS Number
Aldrin	309-00-2
DDE	72-55-9
Dieldrin	60-57-1
Endrin	72-20-8
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3
Hexachlorohexahydro-dimethanonaphthalene	465-73-6
Malathion	121-75-5
Methyl Parathion	298-00-0
Parathion	56-38-2

METHOD SUMMARY:

The above pesticides in water samples can be measured by gas chromatography with electron capture detection (GC/ECD). A hexane extract of the water sample is applied to an alumina column. An elution solvent is added in small increments, and each resulting eluate fraction is analyzed separately, allowing adequate resolution of the peaks in the chromatogram.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

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

REFERENCE:

Law, L. M.; Goerlitz, D. F. "Microcolumn Chromatographic Cleanup for the Analysis of Pesticides in Water," J. Assoc. Off. Anal. Chem., 53,(6), 1970, pp. 1276-1286.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $135-150 (list).

PHENOLIC COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number
2-Chlorophenol	95-57-8
Cresol	1319-77-3
m-Cresol	108-39-4
o-Cresol	95-48-7
p-Cresol	106-44-5
2,4-Dichlorophenol	120-83-2
2,6-Dichlorophenol	87-65-0
Phenol	108-95-2

METHOD SUMMARY:

Analysis for the above phenols, methylphenols (cresols), and mono- and dichlorophenols in water may be performed by direct injection of 1 to 3 microliters of the sample into a gas chromatograph with a flame ionization detector (GC/FID).

INTERFERENCES:

Suspended particulate matter may cause problems, and interferences by co-eluting compounds should be removed by an appropriate preliminary separation technique. In strongly alkaline samples, salt formation may be a problem, and pH adjustment may be required.

QUALITY CONTROL:

Three microliters of distilled water should be injected into the gas chromatograph between sample injections. Triplicate determinations of each sample should be performed.

EPA/TECHNICAL STATUS:

The method is applicable over the range of 1 to 80 milligrams per liter, and has undergone single-laboratory evaluation. The method is not EPA-approved. Single-operator precision information is available (ASTM).

REFERENCES:

Kirshen, N.A. "The Analysis of Phenols in Water by Direct Aqueous Injection," In Varian Instruments at Work, No. 66, October 1982.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Volume 11.02, D 2580-80; ASTM: Philadelphia, 1981, pp. 208-216.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $60-90 (list).

PHENOLICS

Air Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Ammonium Picrate	131-74-8	2,4-Dinitrophenol	51-28-5
p-Chloro-m-cresol	59-50-7	2,5-Dinitrophenol	329-71-5
2-Chlorophenol	95-57-8	2,6-Dinitrophenol	573-56-8
Cresol	1319-77-3	Nitrophenol, Mixed	25154-55-6
m-Cresol	108-39-4	m-Nitrophenol	554-84-7
o-Cresol	95-48-7	o-Nitrophenol	88-75-5
p-Cresol	106-44-5	p-Nitrophenol	100-02-7
2,4-Dichlorophenol	120-83-2	Pentachlorophenol	87-86-5
2,6-Dichlorophenol	87-65-0	Phenol	108-95-2
2,4-Dimethylphenol	105-67-9	2,3,4,6-Tetrachlorophenol	58-90-2
4,6-Dinitro-o-cresol	534-52-1	Trichlorophenol	25167-82-2
4,6-Dinitro-o-cyclohexyl-phenol	131-89-5	2,4,5-Trichlorophenol	95-95-4
		2,4,6-Trichlorophenol	88-06-2
Dinitrophenol	25550-58-7	Xylenol	1300-71-6

METHOD SUMMARY:

Analysis of air for the above phenolic compounds can be performed spectrophotometrically. The phenolics present in approximately 1000 liters of air are collected on a fiberglass filter with a high-volume sampler at 28 liters per minute. The filter medium should be wet with sodium hydroxide. The phenolics are Soxhlet-extracted from the filter medium with benzene, and the resulting solution is extracted with sodium hydroxide, acidified, and distilled. The phenolics present in the distillate are determined by spectrophotometry. This method does not identify the phenolic compounds present, but measures the total phenolic content of the sample.

INTERFERENCES:

Any color or turbidity in the sample that carries through the distillation step can interfere. Sulfur compounds and certain metal ions interfere. The addition of copper sulfate to the alkaline solution of phenols will stabilize the solution.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The distillation/colorimetric portion of the method is approved for use in Clean Water Act analyses. The method is sensitive to levels above approximately 80 micrograms per cubic meter for the recommended sample size. Precision and accuracy information is furnished.

REFERENCES:

American Public Health Association, "Tentative Method of Analysis for Phenols in the Atmosphere (Gas Chromatographic Method)," Methods of Air Sampling and Analysis, M. Katz, Ed., Method 121; APHA: Washington, 1972, pp. 328-332.

Mohler, E. F.; Jacob, L. N. "Determination of Phenolic-Type Compounds in Water and Industrial Waste Waters," Anal. Chem., 29(9), 1957, pp. 1369-1374.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $85-100 (list), plus sampling costs.

PHENOLICS

Water Samples
Laboratory Method

Compound	CAS Number
p-Chloro-m-cresol	59-50-7
2-Chlorophenol	95-57-8
Cresol	1319-77-3
m-Cresol	108-39-4
o-Cresol	95-48-7
2,4-Dichlorophenol	120-83-2
2,6-Dichlorophenol	87-65-0
2,5-Dinitrophenol	329-71-5
2,6-Dinitrophenol	573-56-8
2-Nitrophenol	88-75-5
3-Nitrophenol	554-84-7
Pentachlorophenol	87-86-5
Phenol	108-95-2
2,3,4,6-Tetrachlorophenol	58-90-2
Trichlorophenol	25167-82-2
2,4,5-Trichlorophenol	95-95-4
2,4,6-Trichlorophenol	88-06-2

METHOD SUMMARY:

The above phenolic compounds in water samples can be measured photometrically. After acid distillation, the phenolics in the sample are reacted with 4-amino-antipyrine at a pH of 10 $\pm$ 0.2 in the presence of potassium ferricyanide. The antipyrine dye may be extracted from the aqueous solution into chloroform and the absorbance measured at 460 nm, or the absorbance can be measured directly in the aqueous solution at 505, 510, or 520 nm. The extraction method provides 20-fold lower detection. The analysis may be performed manually or with a Technicon AutoAnalyzer (or equivalent). A similar procedure uses 3-methyl-2-benzothiolinone hydrazene hydrochloride (MBTH) as the color reagent and ceric ammonium sulfate as an oxidant. This procedure is performed at pH 4 and the color is measured at 520 or 490 nm. This method does not identify the specific phenolic compounds present, but measures the total phenolic content of the sample. The result should be considered as a minimum value.

INTERFERENCES:

Sulfur compounds, oxidizing agents, oil and tar may interfere. While phenol-decomposing bacteria, reducing substances, and strongly alkaline conditions may also interfere, provisions have been incorporated in the method to minimize their effects. Phenolic compounds that have a substituent in the *para* position usually do not produce color, unless the *para* substituent is a carboxyl, halogen, hydroxyl, methoxyl or sulfonic acid group.

QUALITY CONTROL:

A standard and a blank must be prepared and the results should be used in the calculations.

EPA/TECHNICAL STATUS:

These methods are approved for SDWA and NPDES analyses. Limits of detection are: for the 4-aminoantipyridine methods 5 micrograms per liter if the chloroform extraction is performed, 50 to 100 micrograms per liter if it is not, and 2 micrograms per liter for the automated method; for the MBTH methods, 2 micrograms per liter if the extraction is performed and 50 micrograms per liter if it is not. Precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and WAstes, Methods 420.1, 420.2, 420.3; EPA-600/4-79-020, U.S. EPA: Washington, 1979.

American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 15th ed., Method 510; APHA: Washington, 1981 pp. 508-513.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, D 1783-80, ASTM: Philadelphia, 1983 pp. 202-207.

U.S. Geological Survey, Methods for the Determination of Organic Substances in Water and Fluvial Sediments, Book 5, "Laboratory Analysis," Chapter A3; USGS: Denver, Open-File Report 82-1004, 1983.

Alberta Environmental Centre, Methods Manual for the Chemical Analysis of Water and Wastes, "Phenolics;" Publication No. AECV81-M1; Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

COST INFORMATION:

Cost information has not been obtained.

PHENOLS

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number	Detection Limit (µg/L)*
p-Chloro-m-cresol	59-50-7	0.36
2-Chlorophenol	95-57-8	0.31
2,4-Dichlorophenol	120-83-2	0.39
2,6-Dichlorophenol	87-65-0	--
2,4-Dimethylphenol	105-67-9	0.32
4,6-Dinitro-o-cresol		--
and salts	534-52-1	16.0
4,6-Dinitro-o-cyclohexyl-		--
phenol	131-89-5	--
Dinitrophenol	25550-58-7	13.0
2,4-Dinitrophenol	51-28-5	13.0
2-Nitrophenol	88-75-5	0.45
4-Nitrophenol	100-02-7	2.8
Pentachlorophenol	87-86-5	7.4
Phenol	108-95-2	0.14
2,3,4,6-Tetrachlorophenol	58-90-2	--
2,4,6-Trichlorophenol	88-06-2	0.64

METHOD SUMMARY:

Analysis of soil/sediment samples for the above phenols can be performed by gas chromatography. A soil/sediment sample is extracted at pH 2 with methylene chloride, and the extract is dried, concentrated, and analyzed by gas chromatography with either a flame ionization detector (GC/FID) or an electron capture detector (GC/ECD).

INTERFERENCES:

Use of plastics in collection, storage, or processing of samples must be avoided.

QUALITY CONTROL:

An aliquot of each sample must be spiked to determine the detection limits and recovery, and a method blank should be analyzed with each set of samples or change in reagents. Field and laboratory replicates and duplicate samples should be analyzed to demonstrate the sensitivity and accuracy of the method. When doubt exists about the identification of a peak in the chromatogram, confirmatory methods, such as gas chromatography/mass spectrometry (GC/MS) or second-column confirmation, should be used.

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* As listed in Method 604 (EPA)

EPA/TECHNICAL STATUS:

This method is EPA-approved for Resource Conservation and Recovery Act (40 CFR, Part 261, Identification and Listing of Hazardous Wastes) sample analyses. No precision or accuracy information is furnished.

REFERENCES:

Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, J. E. Longbottom, J. J. Lichtenberg, Eds., Method 604;" EPA-600/4-82-057, U.S. EPA: Cincinnati, 1982.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3540, 3550, 8040: SW-846, U.S. EPA: Washington, July 1982.

U.S. Environmental Protection Agency, Development and Application of Test Procedures for Specific Organic Toxic Substances in Wastewaters. Category 3-Phenols, Report for EPA Contract 68-03-2625.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $105-160 (list).

PHENOLS

Water Samples
Laboratory Method

Compound	CAS Number	Detection Limit (µg/L)*
p-Chloro-m-cresol	59-50-7	0.36
2-Chlorophenol	95-57-8	0.31
2,4-Dichlorophenol	120-83-2	0.39
2,6-Dichlorophenol	87-65-0	--
2,4-Dimethylphenol	105-67-9	0.32
4,6-Dinitro-o-cresol		--
and salts	534-52-1	16.0
4,6-Dinitro-o-cyclohexyl		
phenol	131-89-5	--
Dinitrophenol	25550-58-7	13.0
2,4-Dinitrophenol	51-28-5	13.0
2-Nitrophenol	88-75-5	0.45
4-Nitrophenol	100-02-7	2.8
Pentachlorophenol	87-86-5	7.4
Phenol	108-95-2	0.14
2,3,4,6-Tetrachlorophenol	58-90-2	--
2,4,6-Trichlorophenol	88-06-2	0.64

METHOD SUMMARY:

Analysis of water samples for the above phenols can be performed by gas chromatography. A water sample is extracted at pH 2 with methylene chloride and the extract is dried, concentrated, and analyzed by gas chromatography with either a flame ionization detector (GC/FID) or an electron capture detector (GC/ECD).

INTERFERENCES:

The use of plastics in collection, storage, or processing of samples must be avoided.

QUALITY CONTROL:

An aliquot of each sample must be spiked to determine the detection limits and recovery, and a method blank should be analyzed with each set of samples or change in reagents. Field and laboratory replicates and duplicate samples should be analyzed to demonstrate the sensitivity and accuracy of the method. When doubt exists about the identification of a peak in the chromatogram, confirmatory methods, such as gas chromatography/mass spectrometry (GC/MS) or second-column confirmation, should be used.

*As cited in Method 604.

EPA/TECHNICAL STATUS:

This method is EPA-approved for Resource Conservation and Recovery Act (40 CFR, Part 261, Identification and Listing of Hazardous Wastes) sample analyses. No precision or accuracy information is furnished.

REFERENCES:

Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, J. E. Longbottom, J. J. Lichtenberg, Eds., Method 604; EPA-600/4-82-057, U.S. EPA: Cinncinati, 1982.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3540, 3550, 8040; SW-846, U.S. EPA: Washington, July 1982.

U.S. Environmental Protection Agency, Development and Application of Test Procedures for Specific Organic Toxic Substances in Wastewaters. Category 3-Phenols, Report for EPA Contract 68-03-2625.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $105-160 (list).

PHENOLS AND OTHER COMPOUNDS

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Chlorambucil	305-03-3
Daunomycin	20830-81-3
Melphalan	148-82-3
Methomyl	16752-77-5
Mitomycin C	50-07-7
5-Nitro-o-toluidine	99-55-8
Reserpine	50-55-5
Streptozotocin	18883-66-4
Thiram	137-26-8
Warfarin	81-81-2

METHOD SUMMARY:

Analysis for the above phenols and other compounds may be performed by high-performance liquid chromatograpy (HPLC). The sample is extracted with diethyl ether and the extract is analyzed by solvent-programmed reversed-phase HPLC with UV detection at 254 nm.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

Method blanks should be processed with each set of samples or change in operating conditions or matrix. Field and laboratory replicates should be analyzed and fortified samples should be processed and analyzed.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precison or accuracy information is furnished.

REFERENCE:

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Method 8320; SW-846, U.S. EPA: Washington, 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $130-175 (list).

PHENOLS AND OTHER COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number
Chlorambucil	305-03-3
Daunomycin	20830-81-3
Melphalan	148-82-3
Methomyl	16752-77-5
Mitomycin C	50-07-7
5-Nitro-o-toluidine	99-55-8
Reserpine	50-55-5
Streptozotocin	18883-66-4
Thiram	137-26-8
Warfarin	81-81-2

METHOD SUMMARY:

Analysis for the above phenols and other compounds in water samples can be performed by high-performance liquid chromatograpy (HPLC). A water sample is extracted with diethyl ether, and the extract is analyzed by solvent-programmed reversephase HPLC with UV detection at 254 nm.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

A method blank should be processed with each set of samples or change in operating conditions or sample matrix. Fortified samples, field replicates, and laboratory replicates should be processed and analyzed.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precison or accuracy information is furnished.

REFERENCE:

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Method 8320; SW-846, U.S. EPA: Washington, 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $130-175 (list).

PHTHALATE ESTERS

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number	Detection Limit (µg/L)*
Bis(2-Ethylhexyl) Phthalate	117-81-7	2.0
Di-n-butyl Phthalate	84-74-2	0.36
Diethyl Phthalate	84-66-2	0.49
Dimethyl Phthalate	113-11-3	0.29
Di-n-octyl Phthalate	117-84-0	3.0

METHOD SUMMARY:

Analysis of soil/sediment samples for the above phthalate esters can be performed by gas chromatography. The sample is extracted at neutral pH with methylene chloride, the solvent exchanged, and the resulting solution is analyzed by gas chromatography (GC), using either an electron-capture detector (ECD) or a flame-ionization detector (FID).

INTERFERENCES:

Use of plastics in sampling or sample processing must be avoided.

QUALITY CONTROL:

A reagent blank should be processed each time a change in analytical conditions occurs or with each new set of samples. Field and laboratory replicates and fortified samples should be analyzed to validate the analytical results.

EPA/TECHNICAL STATUS:

This method is EPA-approved for use under the Clean Water Act and the Resource Conservation and Recovery Act (RCRA). Precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, J. E. Longbottom, and J. J. Lichtenberg, Eds., Method 606; EPA 600/4-82-057, U. S. EPA: Cincinnati, 1982.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Method 8060; SW-846; U.S. EPA: Washington, 1982.

* As cited in Method 606.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $90-145 (list).

PHTHALATE ESTERS

Water Samples
Laboratory Method

Compound	CAS Number	Detection Limit (µg/L)*
Bis(2-Ethylhexyl) Phthalate	117-81-7	2.0
Di-n-butyl Phthalate	84-74-2	0.36
Diethyl Phthalate	84-66-2	0.49
Dimethyl Phthalate	113-11-3	0.29
Di-n-octyl Phthalate	117-84-0	3.0

METHOD SUMMARY:

Analysis of water samples for the above phthalate esters can be performed by gas chromatography. The sample is extracted at neutral pH with methylene chloride, and the extract is analyzed by gas chromatography (GC), using either an electron-capture detector (ECD) or a flame-ionization detector (FID).

INTERFERENCES:

Use of plastics in sampling or sample processing must be avoided.

QUALITY CONTROL:

A reagent blank should be processed each time a change in analytical conditions occurs or with each new set of samples. Field and laboratory replicates and fortified samples should be analyzed to validate the analytical results.

EPA/TECHNICAL STATUS:

This method is EPA-approved for use under the Clean Water Act and the Resource Conservation and Recovery Act (RCRA). Precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, J. E. Longbottom, and J. J. Lichtenberg, Eds., Method 606; EPA 600/4-82-057, U. S. EPA: Cincinnati, 1982.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Method 8060; SW-846; U.S. EPA: Washington, 1982.

*As cited in Method 606.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $90-145 (list).

POLYCYCLIC AROMATIC HYDROCARBONS

Air Samples
Laboratory Method 1

Compound	CAS Number
Benzo[k]fluoranthene	207-08-9
Benzo[a]pyrene	50-32-8

METHOD SUMMARY:

Analysis of air for benzo[a]pyrene (BaP) and benzo[k]fluoranthene (BkF) can be performed by high-volume glass-fiber filtration of air for 24 hours, followed by cyclohexane Soxhlet extraction for 6 hours, extract concentration, and fluorimetric or high-performance liquid chromatographic (HPLC) determination of BaP and BkF.

INTERFERENCES:

BaP and BkF are separated from potentially interfering organic compounds by chromatography on alumina.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished. The detection limit for BaP and BkF is 0.01 nanograms per cubic meter of air.

REFERENCES:

Alberta Environmental Centre, Manual of Methods for the Analysis of Atmospheric Pollutants, Methods 21515, 21516; Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Analysis of Air for Polycyclic Aromatic Hydrocarbons," pp. III-414 to III-420; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

COST INFORMATION:

Cost per sample for analysis for this method is approximately $85-160 (list).

POLYCYCLIC AROMATIC HYDROCARBONS

Air Samples
Laboratory Method 2

Compound	CAS Number
Benzo[a]anthracene	56-55-3
Benzo[b]fluoranthene	205-99-2
Benzo[k]fluoranthene	207-08-9
Benzo[ghi]perylene	191-24-2
Benzo[a]pyrene	50-32-8
Chrysene	218-01-9
Dibenz[a,h]anthracene	53-70-3
7,12-Dimethylbenz[a]anthracene	57-97-6
Fluoranthene	206-44-0
3-Methylcholanthrene	56-49-5
Pyrene	129-00-0

METHOD SUMMARY:

Analysis of air for the above polycyclic aromatic hydrocarbons can be performed by high-volume glass-fiber filtration of approximately 200 cubic meters of air, followed by Soxhlet extraction of the filter with methylene chloride, solvent-exchange into isooctane, and analysis of the resulting solution by high-performance liquid chromatography (HPLC) with ultraviolet-absorbance or fluorescence detection, or by gas chromatography with flame-ionization detection (GC/FID), or with peak-trapping followed by ultraviolet spectrophotometric analysis of the individual peaks.

INTERFERENCES:

Extensive sample cleanup is usually not required, but the isooctane extract may be extracted with phosphoric acid in dimethylsulfoxide, and the resulting solution may be purified by column chromatography.

QUALITY CONTROL:

When doubt exists regarding the identification of peaks in the chromatogram, detection at two wavelengths or gas chromatography/mass spectrometry (GC/MS) can be used to confirm tentative identifications.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information is furnished. The method can measure levels above approximately 1 microgram per cubic meter.

REFERENCES:

Thomas, R. S.; Lao, R. C.; Wang, D. T.; Robinson, D.; Sakuma, T. In Carcinogenesis, Vol. 3: Polynuclear Aromatic Hydrocarbons; P. W. Jones and R. I. Freudenthal, Eds.; Raven Press: New York, 1978, pp. 9-19.

McElroy, F. C.; Searl, T. D.; Brown R. A. In Trace Organic Analysis: A New Frontier in Analytical Chemistry, National Bureau of Standards Special Publication 519, 1978, pp. 7-18.

Das, B. S.; Thomas, G. H. In Trace Organic Analysis: A New Frontier in Analytical Chemistry, National Bureau of Standards Special Publication 519, 1979, pp. 41-56.

COST INFORMATION:

Cost per sample for analysis by HPLC is approximately $155-175 (list), plus sampling costs; by GC/FID, approximately $115 (list), plus sampling costs. Cost information for the U.V. spectrometrty peak-trapping method has not been obtained.

POLYCYCLIC AROMATIC HYDROCARBONS

Air Samples
Laboratory Method 3

Compound	CAS Number
Anthracene	120-12-7
Fluoranthene	206-44-0
Phenanthrene	85-01-8
Pyrene	129-00-0

METHOD SUMMARY:

Analysis for the above airborne polycyclic aromatic hydrocarbons is performed by passage of 1000 liters of air through a polyurethane-foam sampling tube, followed by Soxhlet extraction of the foam and analysis of the extract by gas chromatography with electron capture detection (GC/ECD). When concentration levels are high enough, confirmation of peak identification can be performed by gas chromatography/mass spectrometry (GC/MS).

INTERFERENCES:

Phthalate esters and polychlorinated naphthalenes can interfere, and use of non-TFE plastics must be avoided.

QUALITY CONTROL:

Solvents, reagents, glassware, and polyurethane plugs must be shown to be free of interfering artifacts. A field blank must be analyzed with each set of samples, and both a quality control standard and a method blank should be analyzed with each set of 5 to 15 samples.

EPA/TECHNICAL STATUS:

This method has been evaluated by the Environmental Protection Agency, but is not approved for any specific regulatory program. This method is applicable to a range from 50 to 1000 nanograms per cubic meter. Precision and accuracy information is furnished.

REFERENCE:

U.S. Environmental Protection Agency, Guidelines for Air Monitoring at Hazardous Waste Sites, Appendix B, Assignment 26; Contract 68-02-3168, U.S. EPA: Research Triangle Park, North Carolina, May 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $115 (list): the cost for GC/MS confirmation is approximately $150-200 (list). Sampling costs are additional.

POLYCYLIC AROMATIC HYDROCARBONS

Air Samples
Laboratory Method 4

Compound	CAS Number
Anthracene	120-12-7
Benzo[a]anthracene	56-55-3
Benzo[k]fluoranthene	207-08-9
Benzo[ghi]perylene	191-24-2
Benzo[a]pyrene	50-32-8
Chrysene	218-01-9
Dibenz[a,h]anthracene	53-70-3
Fluoranthene	206-44-0
Fluorene	86-73-7
Phenanthrene	85-01-8
Pyrene	129-00-0

METHOD SUMMARY:

Analysis of air for the above polycyclic aromatic hydrocarbons can be performed by passage of approximately 200 cubic meters of air through a sampling train with a glass-fiber filter, followed by cyclohexane Soxhlet extraction of the filter, concentration of the extract and analysis by high-performance liquid chromatography (HPLC) with ultraviolet or fluorescence detection.

INTERFERENCES:

Use of two wavelengths for absorbance detectors can eliminate some interference and/or co-elution problems.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

The method is not EPA-approved. No precision or accuracy information is furnished.

REFERENCE:

Thomas, R. S.; Lao, R. C.; Wang, D. T.; Robinson, D.; Sakuma, T. In Carcinogenesis, Vol. 3: Polynuclear Aromatic Hydrocarbons; P. W. Jones and R. I. Freudenthal, Eds.; Raven Press: New York, 1978, pp. 9-19.

COST INFORMATION:

The cost per sample for analysis by this method is approximately $160-180 (list), plus sampling costs.

POLYCYCLIC AROMATIC HYDROCARBONS

Air Samples
Laboratory Method 5

Compound	CAS Number
Benz[c]acridine	225-51-4
Benzo[a]anthracene	56-55-3
Benzo[b]fluoranthene	205-99-2
Benzo[k]fluoranthene	207-08-9
Benzo[ghi]perylene	191-24-2
Benzo[a]pyrene	50-32-8
Chrysene	218-01-9
Fluoranthene	206-44-0
Pyrene	129-00-0

METHOD SUMMARY:

The above polycyclic aromatic hydrocarbons in air samples may be measured spectrophotometrically. The analytes are collected on a filter and extracted with benzene. Internal standards are added, the extract is concentrated, and a portion of the concentrate is injected into a gas chromatograph (GC). The polynuclear aromatic hydrocarbons and the internal standards are trapped individually as they elute from the GC column, and each compound is measured by UV spectrophotometry.

INTERFERENCES:

Any compound with similar GC retention time as one of the analytes or internal standards, and any compound with a similar UV absorption spectrum as one of the analytes will interfere.

QUALITY CONTROL:

A filter blank should be processed, and its value used in the calculation of the results. Also, appropriate corrections must be made based on the recovery of the internal standard.

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 2 to 1,000 micrograms of each compound per cubic meter of air, with a 0.5-liter sample. No precision or 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 183; Publicaton No. 77-157-A, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost information has not been obtained.

POLYCYCLIC AROMATIC HYDROCARBONS

Air Samples
Laboratory Method 6

Compound	CAS Number
Benzo[a]anthracene	56-55-3
Benzo[k]fluoranthene	207-08-9
Benzo[a]pyrene	50-32-8
Chrysene	218-01-9
Fluoranthene	206-44-0
Pyrene	129-00-0

METHOD SUMMARY:

The above polycyclic aromatic hydrocarbons in air samples may be measured spectrophotometrically. The analyte is collected on a filter and extracted with benzene. The hydrocarbons are separated on an alumina column with a n-pentanediethyl ether mixture as eluting solvent. The collected fractions are analyzed by UV spectrophotometry.

INTERFERENCES:

Any substance that hinders the separation or absorbs radiation at the same wavelengths as the analytes will interfere.

QUALITY CONTROL:

The recovery should be determined, and a blank should be processed with each group of samples. The recovery and the value of the blank should be used in the calculation of the results.

EPA/TECHNICAL STATUS:

This method has been classified as operational by the National Institute of Occupational Safety and Health (NIOSH) for use over the range of 0 to 3 milligrams of benzene-soluble polycyclic aromatic hydrocarbons per cubic meter. Precision or accuracy information is not furnished.

REFERENCE:

U.S. Department of Health, Education and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 184; Publicaton No.77-157-A, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $90-105 (list), plus sampling costs.

POLYCYCLIC AROMATIC HYDROCARBONS

Air Samples
Laboratory Method 7

Compound	CAS Number
Benzo[a]anthracene	56-55-3
Benzo[b]fluoranthene	205-99-2
Benzo[k]fluoranthene	207-08-9
Benzo[a]pyrene	50-32-8
Benzo[ghi]perylene	191-24-2
Benzo[a]pyrene	50-32-8
Chrysene	218-01-9
Dibenzo[a,h]anthracene	53-70-3
Dibenzo[a,i]pyrene	189-55-9
Fluoranthene	206-44-0
Indeno[1,2,3-cd]pyrene	193-39-5
Phenanthrene	85-01-8
Pyrene	129-00-0

METHOD SUMMARY:

The above polycyclic aromatic hydrocarbons in particulate air samples can be measured by liquid chromatography (LC). Samples are collected in a specially designed baghouse. An aliquot of the collected particulates is Soxhlet-extracted with methylene chloride. Sample cleanup involves concentration, solvent exchange to cyclohexane, liquid-liquid partition between cyclohexane and nitromethane, and a final concentration step. Analysis is by LC with fluorescence wavelength programming. Also described are a gas chromatographic method and a sequential (reversed-phase) LC method, with specific sample extraction and cleanup procedures for each.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

Internal standards are specified.

EPA/TECHNICAL STATUS:

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

REFERENCE:

May, W. E.; Wise, S. A. "Liquid Chromatographic Determination of Polycyclic Aromatic Hydrocarbons in Air Particulate Extracts," Anal. Chem., 56, 1984, pp. 225-232.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $195 (list), plus sampling costs.

POLYCYCLIC AROMATIC HYDROCARBONS

Air Samples
Laboratory Method 8

Compound	CAS Number
Acenaphthene	83-32-9
Anthracene	120-12-7
Benzo[a]pyrene	50-32-8
Fluorene	86-73-7

METHOD SUMMARY:

The above polycyclic aromatic hydrocarbons in particulate air samples can be measured by synchronous luminescence spectroscopy. The samples are collected either in high-volume samplers or on filter papers.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision or accuracy information is not furnished. The detection limit is approximately 7 micrograms of analyte per cubic meter of air.

REFERENCE:

Vo-Dinh, T. "New Luminescence Techniques Simplify Air Analysis," InTech, 28,(5), 1981, pp. 45-48.

COST INFORMATION:

Cost information has not been obtained.

POLYCYCLIC AROMATIC HYDROCARBONS

Soil/Sediment Samples
Laboratory Method 1

Compound	CAS Number
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo[a]anthracene	56-55-3
Benzo[b]fluoranthene	207-08-9
Benzo[k]fluoranthene	205-99-2
Benzo[a]pyrene	50-32-8
Benzo[ghi]perylene	191-24-2
Chrysene	218-01-9
Dibenzo[a,h]anthracene	53-70-3
Dibenzo[a,i]pyrene	189-55-9
Fluorene	86-73-7
Indeno[1,2,3-cd]pyrene	193-39-5
3-Methylcholanthrene	56-49-5
Phenanthrene	85-01-8
Pyrene	129-00-0

METHOD SUMMARY:

Analysis of soil/sediment samples for the above polycyclic aromatic hydrocarbons can be performed by gas chromatography. A soil/sediment sample is Soxhlet-extracted or sonicated at neutral pH with dichloromethane, and the extract is dried, concentrated, and analyzed by gas chromatography with flame ionization detection (GC/FID), using internal or external standards for quantification. Confirmation of compound identification can be accomplished by gas chromatography/mass spectrometry (GC/MS).

INTERFERENCES:

A silica-gel column cleanup procedure may be required to eliminate interferences, and some samples may require further cleanup procedures.

QUALITY CONTROL:

An aliquot of each sample should be spiked with standards and analyzed to determine recovery and sensitivity. The gas chromatographic system must be demonstrated to be free of interferences from previous sample injections by analysis of a method blank with each set of samples or each change in reagents. Field and laboratory replicates, fortified samples, and duplicate samples should be processed to ascertain the precision and accuracy of the method. In cases where doubt exists regarding the identification of peaks in the chromatogram, confirmatory techniques, such as gas chromatography/mass spectrometry (GC/MS) and secondcolumn confirmation, should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved for analyses under the Resource Conservation and Recovery Act (RCRA). No precision or accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 610; EPA 600/4-82-057, U.S. EPA: Washington, 1982.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3540, 3550, 8100, 8310; SW-846, U.S. EPA: Washington, 1982.

U.S. Department of Energy Environmental Measurements Laboratory, EML Procedures Manual, H. L. Volchok and G. de Planque, Eds., Method E-00-14, "Determination of Polynuclear Aromatic Hydrocarbons in Sediments;" HASL-300, Environmental Measurements Laboratory, U.S. DOE: New York, 1983.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $120 (list); the cost of a silica-gel column cleanup is approximately $25-35 (list).

POLYCYCLIC AROMATIC HYDROCARBONS

Soil/Sediment Samples
Laboratory Method 2

Compound	CAS Number
Acenaphthene	83-32-9
Anthracene	120-12-7
Dibenz[a,h]anthracene	53-70-3
7,12-Dimethyl-benz[a]anthracene	57-97-6
Fluorene	86-73-7
Naphthalene	91-20-3
Phenanthrene	85-01-8

METHOD SUMMARY:

Analysis for the above fluorescent organic compounds in soil/sediment samples can be performed by gas chromatography (GC) with fluorescence detection. A flame photometric detector is modified with quartz fiber optics to achieve the necessary instrumental configuraion.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished. The analytical technique has a sub-nanogram sensitivity and high selectivity. Limits of detection are provided.

REFERENCE:

Thomas, L. C.; Adams, A. K. "Detection of Fluorescent Compounds by Modified Flame Photometric Gas Chromatography Detectors," Anal. Chem., 54(14), 1982, pp. 2597-2599.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $135-180 (list).

POLYCYCLIC AROMATIC HYDROCARBONS

Soil/Sediment Samples
Laboratory Method 3

Compound	CAS Number
Anthracene	120-12-7
Benzo[a]anthracene	56-55-3
Benzo[a]pyrene	50-32-8
Chrysene	218-01-9
Fluoranthene	206-44-0
Fluorene	86-73-7
Phenanthrene	85-01-8
Pyrene	129-00-0

METHOD SUMMARY:

The above polycyclic aromatic hydrocarbons in sediment samples may be measured by gas chromatography. The wet sample is dewatered by washing with methanol and then extracted with methylene chloride/methanol (2:1) using a ball-mill tumbler or a shaker table. The extracts and methanol washes are combined and filtered into a separatory funnel. The methanol is partitioned into water. The methanol/water solution is extracted into methylene chloride. The extracts are filtered, then separated into saturated and unsaturated hydrocarbons by silica gel adsorption chromatography, and the resulting solutions are analyzed by gas chromatography with flame ionization detection (GC/FID) using a capillary column.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

Recovery internal standards should be added and the results used in calculations. A reagent blank, reagent spike, GC internal standard, and GC calibration standard should also be used.

EPA/TECHNICAL STATUS:

This method is not EPA-approved and has no status as an ASTM standard method. Individual hydrocarbons can be quantified in the parts-per-billion range. Precision information is furnished.

REFERENCE:

American Society for Testing and Materials, "Proposed Test Method for Hydrocarbons in Marine Sediments," 1983 Annual Book of ASTM Standards, Vol. 11.02, (no method number), ASTM: Philadelphia, 1983, pp. 963-969.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $150-195 (list).

POLYCYCLIC AROMATIC HYDROCARBONS

Soil/Sediment Samples
Laboratory Method 4

Compound	CAS Number	Method Detection Limit (µg/l)
Acenaphthene	83-32-9	1.8
Acenaphthylene	208-96-8	2.3
Anthracene	120-12-7	0.66
Benzo[a]anthracene	56-55-3	0.013
Benzo[b]fluoranthene	205-99-2	0.018
Benzo[k]fluoranthene	207-08-9	0.017
Benzo[ghi]perylene	191-24-2	0.076
Benzo[a]pyrene	50-32-8	0.023
Chrysene	218-01-9	0.15
Dibenz[a,h]anthracene	53-70-3	0.030
Fluoranthene	206-44-0	0.21
Fluorene	86-73-7	0.21
Indeno[1,2,3-cd] pyrene	193-39-5	0.043
Naphthalene	91-20-3	1.8
Phenanthrene	85-01-8	0.64
Pyrene	129-00-0	0.27

METHOD SUMMARY:

Analysis of soil/sediment samples for the above polycyclic aromatic hydrocarbon compounds (PAHs) can be performed by high-performance liquid chromatography (HPLC). A 10- to 100-gram sample is spiked with a radiolabeled PAH and refluxed with ethanolic potassium hydroxide. The resulting solution is centrifuged, the liquid is extracted with isooctane, and the isooctane solution is concentrated by evaporation. The solution is solvent-exchanged into toluene and the toluene solution is cleaned up by Florisil column chromatography, dimethyl sulfoxide partitioning and Sephadex LH-20 chromatography. The cleaned extract is then analyzed by HPLC with ultraviolet absorbance and fluorescence detection.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

Recovery of radiolabeled PAH compounds should be measured and sample results corrected accordingly.

EPA/TECHNICAL STATUS:

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

REFERENCES:

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Analysis of Sediment Samples for Polycyclic Aromatic Hydrocarbons," pp. III-395 to III-401: EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Dunn, B. P.; Armour R. J. "Sample Extraction and Purification for Determination of Polycyclic Aromatic Hydrocarbons by Reversed-Phase Chromatography" Anal. Chem., Vol. 52, No. 13 (1980), pp. 2027-2031.

COST INFORMATION:

Cost information has not been obtained.

POLYCYCLIC AROMATIC HYDROCARBONS

Water Samples
Laboratory Method 1

Compound	CAS Number	Method Detection Limit (μg/l)
Acenaphthene	83-32-9	1.8
Acenaphthylene	208-96-8	2.3
Anthracene	120-12-7	0.66
Benzo[a]anthracene	56-55-3	0.013
Benzo[b]fluoranthene	205-99-2	0.018
Benzo[k]fluoranthene	207-08-9	0.017
Benzo[ghi]perylene	191-24-2	0.076
Benzo[a]pyrene	50-32-8	0.023
Chrysene	218-01-9	0.15
Dibenz[a,h]anthracene	53-70-3	0.030
Fluoranthene	206-44-0	0.21
Fluorene	86-73-7	0.21
Indeno[1,2,3-cd] pyrene	193-39-5	0.043
Naphthalene	91-20-3	1.8
Phenanthrene	85-01-8	0.64
Pyrene	129-00-0	0.27

METHOD SUMMARY:

Analysis of water samples for the above polycyclic aromatic hydrocarbons can be performed by high-performance liquid chromatography (HPLC). A one-liter sample is extracted with methylene chloride, the extract solution is concentrated, and the solvent is exchanged into cyclohexane. An optional silica-gel cleanup procedure removes high levels of interferences. The solvent is then exchanged into acetonitrile and the resulting solution is analyzed by gradient reverse-phase HPLC with ultraviolet absorbance and fluorescent detection.

INTERFERENCES:

Other polycyclic aromatic compounds besides those listed may interfere.

QUALITY CONTROL:

A minimum of ten percent of all samples should be fortified and analyzed in duplicate.

EPA/TECHNICAL STATUS:

This method is EPA-approved for Clean Water Act analyses. Precision and accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 610; EPA 600/4-82-057, U.S. EPA: Cincinnati, 1982.

Plumb, R. H. Characterization of Hazardous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, " Analysis for Polycyclic Aromatic Hydrocarbons in Water Samples," pp. III-386 to III-394, EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

U.S. Geological Survey, Methods for the Determination of Organic Substanes in Water and Fluvial Sediment, Book 5, "Laboratory Analysis," Chpater A3; USGS: Denver, Open-File Report 82-1004, 1983.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $175-255 (list).

POLYCYLIC AROMATIC HYDROCARBONS

Water Samples
Laboratory Method 2

Compound	CAS Number
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo[a]anthracene	56-55-3
Benzo[b]fluoranthene	205-99-2
Benzo[k]fluoranthene	207-08-9
Benzo[ghi]perylene	191-24-2
Benzo[a]pyrene	50-32-8
Chrysene	218-01-9
Dibenz[a,h]anthracene	53-70-3
Dibenz[a,i]pyrene	189-55-9
Fluoranthene	206-44-0
Fluorene	86-73-7
Indeno[1,2,3-cd] pyrene	193-39-5
3-Methylcholanthrene	56-49-5
Phenanthrene	85-01-8
Pyrene	129-00-0

METHOD SUMMARY:

Water samples can be analyzed for the above polycyclic aromatic hydrocarbons by gas chromatography. A water sample is Soxhlet- or ultrasonically extracted at neutral pH with dichloromethane, and the extract is dried, concentrated and analyzed by gas chromatography with flame ionization detection (GC/FID), using internal or external standards.

INTERFERENCES:

A silica-gel column cleanup procedure may be required to eliminate interferences, and some samples may require further cleanup procedures.

QUALITY CONTROL:

An aliquot of each sample is spiked with standards and analyzed to determine recovery and sensitivity. The gas chromatographic system must be demonstrated to be free of interferences by the analysis of a method blank with each set of samples or each change in reagents. Field and laboratory replicates, fortified samples, and duplicate samples should be processed to ascertain the precision and accuracy of the method. In cases where some doubt exists regarding the identification of peaks in the chromatogram, confirmatory techniques, such as gas chromatography/mass spectrometry (GC/MS) and second-column confirmation, should be used.

EPA/TECHNICAL STATUS:

This method is EPA-approved for the Resource Conservation and Recovery Act (RCRA) sample analyses. No precision or accuracy information is furnished.

REFERENCE:

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Methods 3510, 3520, 8100, 8130; SW-846, U.S. EPA: Washington, July 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $110-120 (list); the cost of a silica-gel column cleanup is approximately $25-35 (list).

POLYCYCLIC AROMATIC HYDROCARBONS

Water Samples
Laboratory Method 3

Compound	CAS Number
Acenaphthene	83-32-9
Anthracene	120-12-7
Dibenz[a,h]anthracene	53-70-3
7,12-Dimethyl-benz[a]anthracene	57-97-6
Fluorene	86-73-7
Naphthalene	91-20-3
Phenanthrene	85-01-8

METHOD SUMMARY:

Analysis of water samples for the above polycyclic aromatic hydrocarbons can be performed by extraction and gas chromatography (GC) with fluorescence detection. A flame photometric detector is modified with quartz fiber optics to achieve the necessary instrumental configuration.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The analytical technique has a sub-nanogram sensitivity and high selectivity. No precision or accuracy information is furnished. Limits of detection are provided.

REFERENCE:

Thomas, L. C.; Adams, A. K. "Detection of Fluorescent Compounds by Modified Flame Photometric Gas Chromatography Detectors," Anal. Chem., 54(14), 1982, pp. 2597-2599.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $125-135 (list).

POLYCYCLIC AROMATIC HYDROCARBONS

Water Samples
Laboratory Method 4

Compound	CAS Number
Benzo[a]anthracene	56-55-3
Benzo[b]fluoranthene	205-99-2
Benzo[k]fluoranthene	207-08-9
Benzo[ghi]perylene	191-24-2
Benzo[a]pyrene	50-32-8
Chrysene	218-01-9
Dibenz[a,h]anthracene	53-70-3
7,12-Dimethylbenz[a]anthracene	57-97-6
Fluoranthene	206-44-0
3-Methylcholanthrene	56-49-5
Pyrene	129-00-0

METHOD SUMMARY:

Analysis of water for the above polycyclic aromatic hydrocarbons can be performed by extraction of a 3-liter sample with methylene chloride, solvent-exchange into isoocatane, and analysis of the resulting solution by high-performance liquid chromatography (HPLC) with ultraviolet absorbance and/or fluorescence detection, by gas chromatography with flame-ionization detection (GC/FID), or by gas chromatography with peak-trapping followed by ultraviolet spectrophotometric analysis of the individual peaks.

INTERFERENCES:

Extensive sample cleanup is usually not required, but the isooctane extract may be extracted with phosphoric acid in dimethylsulfoxide, and the resulting solution may be purified by column chromatography.

QUALITY CONTROL:

When doubt exists regarding the identification of peaks in the chromatogram, detection at two wavelengths or gas chromatography/mass spectrometry (GC/MS) can be used to confirm tentative identifications.

EPA/TECHNICAL STATUS:

The method is not EPA-approved. The method can measure levels above approximately 1 microgram per liter. Precision and accuracy information is furnished.

REFERENCES:

Thomas, R. S.; Lao, R. C.; Wang, D. T.; Robinson, D.; Sakuma, T. In Carcinogenesis, Vol. 3: Polynuclear Aromatic Hydrocarbons, P. W. Jones and R. I. Freudenthal, Eds.; Raven Press: New York, 1978, pp. 9-19.

McElroy, F. C.; Searl, T. D.; Brown R. A. In Trace Organic Analysis: A New Frontier in Analytical Chemistry, National Bureau of Standards Special Publication 519, 1978, pp. 7-18.

Das, B. S.; Thomas, G. H. In Trace Organic Analysis: A New Frontier in Analytical Chemistry, National Bureau of Standards Special Publication 519, 1978, pp. 41-56.

COST INFORMATION:

Cost per sample for analysis by the HPLC method is approximately $155-175 (list); by GC/FID, approximately $115 (list). Cost information for the UV spectrometry peak-trapping method has not been obtained.

PRIMARY AND SECONDARY AMINES

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number
Diethylamine	109-89-7
Dimethylamine	124-40-3
Dipropylamine	142-84-7
Ethylenediamine	107-15-3
Monoethylamine	75-04-7
Monomethylamine	74-89-2
n-Propylamine	107-10-8

METHOD SUMMARY:

Analysis of soil/sediment samples for the above primary and secondary amines can be performed by distillation of a mixture of a 10-gram sample, 70 milliliters of 50-percent sodium hydroxide, and 100 milliliters of water, followed by formation of the pentafluorobenzoyl derivatives of the amines in the distillate, and analysis of the resulting solution by gas chromatography with a nitrogen/phosphorus detector (GC/NPD). Analysis of the extract can also be performed by gas chromatography/mass spectrometry (GC/MS).

INTERFERENCES:

Excess derivatizing agent should be used, and chlorinated solvents can cause a reversible loss of sensitivity in the detector.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved and has not undergone interlaboratory evaluation. The method is sensitive to levels over 1 milligram per gram. No precision or accuracy information is furnished.

REFERENCE:

Ripley, B. D.; French, B. J.; Edgington, L. V. "Gas Chromatographic Determination of Primary and Secondary Amines as Pentafluorobenzamide Derivatives," J. Assoc. Off. Anal. Chem., 65(5), 1982, pp. 1066-1072.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $158-180 (list).

PRIMARY AND SECONDARY AMINES

Water Samples
Laboratory Method

Compound	CAS Number
Diethylamine	109-89-7
Dimethylamine	124-40-3
Dipropylamine	142-84-7
Ethylenediamine	107-15-3
Monoethylamine	75-04-7
Monomethylamine	74-89-2
n-Propylamine	107-10-8

METHOD SUMMARY:

Analysis of water samples for the above primary and secondary amines can be performed by alkaline distillation of a 100-milliliter sample, followed by formation of the pentafluorobenzoyl derivatives of the amines in the distillate, and analysis of the resulting solution by gas chromatography with a nitrogen/phosphorus detector (GC/NPD). Analysis of the extract can also be performed by gas chromatography/mass spectrometry (GC/MS).

INTERFERENCES:

Excess derivatizing agent should be used, and chlorinated solvents can cause a reversible loss of sensitivity in the detector.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved and has not undergone interlaboratory evaluation. The method is sensitive to levels over 1 milligram per liter of sample. No precision or accuracy information is furnished.

REFERENCE:

Ripley, B. D.; French, B. J.; Edgington, L. V. "Gas Chromatographic Determination of Primary and Secondary Amines as Pentafluorobenzamide Derivatives," J. Assoc. Off. Anal. Chem., 65(5), 1982, pp. 1066-1072.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $158-180 (list).

SUBSTITUTED NITRILES AND TETRANITROMETHANE

Water Samples
Laboratory Method

Compound	CAS Number
Acetone Cyanohydrin	75-86-5
Cyanogen Bromide	506-68-3
Propanedinitrile	109-77-3
Tetranitromethane	509-14-8

METHOD SUMMARY:

Analysis for the above compounds in water samples can be performed by chemiluminescence. A sample is made alkaline and treated with hydrogen peroxide and luminol. The resulting chemiluminescence is monitored by a spectrofluorimeter. This method does not identify any specific compound, but gives a positive response for any substance that is chemiluminescent after the above treatment.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished. Relative intensities of chemiluminescence are provided.

REFERENCE:

Yurow, H. W.; Sass, S. "Detection of Various α-Substituted Nitriles and Gem-Halonitroalkanes by Chemiluminescence," Anal. Chim. Acta., 77, 1975, pp. 324-326.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $55-95 (list).

SULFUR-CONTAINING GASES

Air Samples
Laboratory Method

Compound	CAS Number
Hydrogen Sulfide	7783-06-4
Methylmercaptan	74-93-1

METHOD SUMMARY:

The above sulfur-containing compounds in air samples can be measured by gas chromatography. A Teflon® column and a flame-photometric detector are used.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. This method is suitable for concentrations in the approximate range of 1.5 to 150 milligrams analyte per cubic meter. No precision or accuracy information is furnished.

REFERENCE:

de Souza, T. L. C.; Lane, D. C.; Bhatia, S. P. "Analysis of Sulfur-Containing Gases by Gas-Solid Chromatography on a Specially Treated Porapak QS Column Packing," Anal. Chem., 47, 1975, pp. 543-545.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $135 (list), plus sampling costs.

THIOIC ACID ESTER PESTICIDES

Water Samples
Laboratory Method

Compound	CAS Number
Diuron	330-54-1
Methomyl	16752-77-5

METHOD SUMMARY:

Analysis of water samples for diuron and methomyl can be performed by reverse-phase high-performance liquid chromatography (HPLC). A water sample is serially extracted with three 60-milliliter portions of methylene chloride, the extract is solvent-exchanged into one milliliter of acetonitrile, and the resulting acetonitrile solution is analyzed by gradient-elution HPLC with UV detection at 254 nm. Additional sample cleanup, for samples with solids or high levels of interferences, can be performed before analysis by use of a SEP-PAK® cartridge.

INTERFERENCES:

Methomyl undergoes hydrolysis at high pH; sample pH should be adjusted to 6 to 8 within four hours of sampling, and the samples should be kept refrigerated.

QUALITY CONTROL:

A minimum of ten percent of all analyses should be fortified samples.

EPA/TECHNICAL STATUS:

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

REFERENCES:

U.S. Environmental Protection Agency, Test Methods for Nonconventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Method 109; EPA 440/1-83/079-C, U.S. EPA: Cincinnati, 1983, PB 83176636.

E.I. duPont de Nemours & Co, Inc, "Determination of Houston Plant Pesticides, High Performance Liquid Chromatography (HPLC) Methods," Method W28.303(R); Houston, Texas, 29 April 1980.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $140-205 (list).

THIOUREAS AND OTHER COMPOUNDS

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
1-Acetyl-2-thiourea	591-08-2	N-Nitroso-N-methylurea	684-93-5
Azaserine	115-02-6	N-Phenylthiourea	103-85-5
1-(o-Chlorophenyl) thiourea	5344-82-1	Thioacetamide	62-55-5
Crotonaldehyde	123-73-9	Thiosemicarbazide	79-19-6
	4170-30-3	Thiourea	62-56-6
Diethylstilbestrol	56-53-1	Trypan Blue	72-57-1
Epinephrine	51-43-4		
Ethylenethiourea	96-45-7		
α-Naphthylthiourea	86-88-4		

METHOD SUMMARY:

Analysis of soil/sediment samples for the above thioureas and other compounds can be performed by extraction and high-performance liquid chromatography (HPLC). The sample is extracted with diethyl ether and analyzed by reverse-phase HPLC with UV detection at 254 nm, using an acetonitrile/water mobile phase.

INTERFERENCES:

No specific interferences are reported.

QUALITY CONTROL:

A method blank should be processed with each set of samples or each change in operating conditions or sample matrix. Field and laboratory replicates and fortified samples should be analyzed.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished.

REFERENCE:

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, 2nd ed., Method 8330; SW-846, U.S. EPA: Washington, 1982.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $130-205 (list).

TRACE ORGANIC CONTAMINANTS

Water Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Acenaphthene	83-32-9	1,4-Dichlorobenzene	106-46-7
Acetophenone	98-86-2	Diethyl Phthalate	84-66-2
Anthracene	120-12-7	Dimethyl Phthalate	131-11-3
Benzoic Acid	65-85-0	Ethylbenzene	100-41-4
Benzonitrile	100-47-0	Fluorene	86-73-7
Benzyl Chloride	100-44-7	Methyl Methacrylate	80-62-6
Bis(2-ethyhexyl) Phthalate	117-81-7	Naphthalene	91-20-3
		Nitrobenzene	98-95-3
Chlorobenzene	108-90-7	Phenol	108-95-2
2-Chlorophenol	95-57-8	Quinoline	91-22-5
o-Cresol	95-48-7	1,2,4,5-Tetrachlorobenzene	95-94-3
Cumene	98-82-8	2,4,6-Trichlorophenol	88-06-2
1,2-Dichlorobenzene	95-50-1		

METHOD SUMMARY:

Analysis of water samples for the above organic compounds can be performed by gas chromatography. The organic compounds can be concentrated by sorption on a small column of macroreticular resin and eluted; the eluate can then be concentrated and analyzed by gas chromatography with flame-ionization detection (GC/FID). A 1-liter sample is passed through a column of XAD resin at 30 to 50 milliliters per minute, and the resin is eluted with diethyl ether. The ether solution is dried, concentrated, and analyzed by GC/FID. Gas chromatography/mass spectrometry (GC/MS) can be used for identity confirmation.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

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

REFERENCE:

Junk, G. A.; Richard, J. J.; Grieser, M. D.; Witiak, D.; Witiak, J. L.; Arguello, M. D.; Vick, R.; Svec, H. J.: Fritz, J. S.; Calder, G. V. "Use of Macroreticular Resins in the Analysis of Water For Trace Organic Contaminants," J. Chromatogr., 99, 1974, pp. 745-762.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $110 (list); the cost of GC/MS confirmation is approximately $150-200 (list).

VOLATILE ALCOHOLS AND OTHER ORGANIC COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Acetaldehyde	75-07-0	1,4-Dioxane	123-91-1
Acetone	67-64-1	Epichlorohydrin	106-89-8
Acetonitrile	75-05-8	Ethyl Acetate	141-78-6
Acetophenone	98-86-2	Ethylbenzene	100-41-4
Acrylonitrile	107-13-1	Ethylene Oxide	75-21-8
Benzene	71-43-2	Isophorone	78-59-1
Bis(2-Chloroethyl) Ether	111-44-4	Methanol	67-56-1
Butyl Acetate	123-86-4	Methyl Ethyl Ketone	78-93-3
iso-Butyl Acetate	110-19-0	Methyl Isobutyl Ketone	108-10-1
sec-Butyl Acetate	105-46-4	Paraldehyde	123-63-7
tert-Butyl Acetate	540-88-5	2-Picoline	109-06-8
Crotonaldehyde	123-73-9	Propylene Oxide	75-56-9
	4170-30-3	Toluene	108-88-3
1,2-Dichloroethane	107-06-2	Vinyl Acetate	108-05-4
1,2-Dichloropropane	78-87-5	p-Xylene	106-42-3

METHOD SUMMARY:

Analysis of water samples for the above alcohols and other volatile compounds can be performed by gas chromatography with flame ionization detection (GC/FID). The method involves direct injection of 10 microliters of sample.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

The method may not be suitable for simultaneous analysis for organic compounds with similar functionalities, and the qualitative identification of peaks in the chromatogram should be confirmed by a multicolumn approach or other techniques.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The method has undergone interlaboratory evaluation for samples containing only alcohols under the American Society for Testing and Materials. Precision and accuracy information is furnished.

REFERENCES:

American Society for Testing and Materials, Annual Book of ASTM Standards, Part 31, Water; ASTM: Philadelphia, 1978, pp. 834-838, pp. 672-680.

American Society for Testing and Materials, 1983 Annual Book of ASTM Standards, Vol. 11.02, D 2908-74 (1980), D 3695-78; ASTM: Philadelphia, 1983, pp. 50-58, 241-248.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $60-80 (list).

VOLATILE CARBONYL COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number
Acetaldehyde	75-07-0
Acetone	67-64-1
Acrolein	107-02-8
Crotonaldehyde	123-73-9
	4170-30-3
Formaldehyde	50-00-0
Furfural	98-01-1
Methyl Ethyl Ketone	78-93-3
Methyl Isobutyl Ketone	108-10-1

METHOD SUMMARY:

Analysis for the above low-molecular-weight carbonyl compounds can be performed by derivatization and gas chromatography. A sample containing approximately 0.5 millimoles of carbonyl compounds is added to a solution of pentafluorophenylhydrazine in methanol and allowed to stand overnight. The resulting solution is then analyzed by gas chromatography with electron capture detection (GC/ECD).

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The method can detect amounts of derivatives of about 0.01 nanograms and above. No precision or accuracy information is furnished.

REFERENCE:

Hoshika, Y.; Muto, G. "Sensitive Gas Chromatographic Determination of Lower Aliphatic Carbonyl Compounds as their Pentafluorophenylhydrazones," J. Chromatogr., 152, 1978, pp. 224-227.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $90-105 (list).

VOLATILE CHLORINATED ALIPHATIC HYDROCARBONS

Water Samples
Laboratory Method

Compound	CAS Number
Carbon Tetrachloride	56-23-5
Chloroform	67-66-3
Hexachlorobutadiene	87-68-3
Hexachloroethane	67-72-1
Pentachloroethane	76-01-7
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethylene	127-18-4
1,1,1-Trichloroethane	71-55-6
Trichloroethylene	79-01-6

METHOD SUMMARY:

The above volatile chlorinated aliphatic hydrocarbons in water samples can be measured using gas chromatography with electron capture detection (GC/ECD). The water sample is extracted with n-pentane; the extract is dried with anhydrous sodium sulfate and analyzed by temperature-programmed GC/ECD.

INTERFERENCES:

Chlorobromomethanes and laboratory contaminants may interfere; however, the method provides instrumental conditions to minimize their effects.

QUALITY CONTROL:

Standard solutions should be freshly prepared each week.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision information is furnished. The method is suitable for concentrations of 0.01 to 10 micrograms per liter.

REFERENCE:

Deetman, A. A.; Demeulemeester, P.; Garcia, M.; Hauck, G.; Hollies, J. I.; Krockenberger, D.; Palin, D. E.; Prigge, H.; Rohrschneider, L.; Schmidhammer, L. "Standardization of Methods for the Determination of Traces of Some Volatile Chrorinated Aliphatic Hydrocarbons in Air and Water by Gas Chromatography" Anal. Chim. Acta., 82, 1976, pp. 1-17.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $100-145 (list).

VOLATILE ORGANIC ACIDS

Water Samples
Laboratory Method

Compound	CAS Number
Acetic Acid	64-19-7
Butyric Acid	107-92-6
iso-Butyric Acid	79-31-2
Propionic Acid	79-09-4

METHOD SUMMARY:

Analysis of water samples for the above volatile organic acids can be performed by gas chromatography. A 10-milliliter sample is mixed with solid metaphosphoric acid and centrifuged to remove suspended solids. The supernatant is then analyzed by gas chromatography with flame ionization detection (GC/FID).

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

Standard addition methods may be required. No other quality control procedures are given.

EPA/TECHNICAL STATUS:

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

REFERENCE:

Narkis, N.; Henfeld-Furie, S. "Direct Analytical Procedure for Determination of Volatile Organic Acids in Raw Municipal Wastewater," Water Research, 12, 1978, pp. 437-446.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70-80 (list).

VOLATILE ORGANIC COMPOUNDS

Air Samples
Laboratory Method 1

Compound	CAS Number
Acetic Acid	64-19-7
Acetone	67-64-1
Benzene	71-43-2
Butyl Acetate	123-86-4
Butyric Acid	107-92-6
iso-Butyric Acid	79-31-2
Ethyl Acetate	141-78-6
Methyl Ethyl Ketone	78-93-3
Propionic Acid	79-09-4
Styrene	100-42-5
Toluene	108-88-3
Xylene	1330-20-7
m-Xylene	108-38-3
o-Xylene	95-47-6
p-Xylene	106-42-3

METHOD SUMMARY:

Analysis for the above volatile organics in air is performed by passage of 0.75 to 2 liters of air through a Tenax® sampling cartridge, followed by thermal desorption of the analytes into a temperature-programmed gas chromatograph with flame ionization detection (GC/FID). Sampling cartridges should be conditioned at 200°C for at least 2 hours before use.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

In case of uncertainty about the identification of peaks, gas chromatography/mass spectrometry (GC/MS) or second-column techniques should be used for confirmation. A fortified sample should be analyzed and standards should be analyzed in triplicate.

EPA/TECHNICAL STATUS:

This method is used by Environment Canada, but is not EPA-approved. No precision or accuracy information is furnished. The detection limit for most of the compounds is 4 micrograms per cubic meter.

REFERENCES:

Alberta Environmental Centre, Methods Manual for Chemical Analysis of Atmospheric Pollutants; N.C. Das, Ed., Method 17070; Alberta Environmental Centre: Vegreville, Alberta, Canada, 1981.

Holzer, G.; Shanfield, H.; Zlatkis, A.; Bertsch, W.; Juarez, P.; Mayfield, H.; Liebich, H. M. "Collection and Analysis of Trace Organic Emissions from Natural Sources," J. Chromatogr., 142, 1977, pp. 755-764.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $60-80 (list), plus sampling costs.

VOLATILE ORGANIC COMPOUNDS

Air Samples
Laboratory Method 2

Compound	CAS Number	Compound	CAS Number
Acetic Acid	64-19-7	Methyl Isobutyl Ketone	108-10-1
Acetone	67-64-1	Pentachlorobenzene	608-93-5
Benzene	71-43-2	Propionic Acid	79-09-4
Butyl Acetate	123-86-4	Styrene	100-42-5
Butyric Acid	107-92-6	1,1,2,2-Tetrachloroethane	79-34-5
iso-Butyric Acid	79-31-2	Tetrachloroethylene	127-18-4
Carbon Tetrachloride	56-23-5	Toluene	108-88-3
Chlorobenzene	108-90-7	1,1,1-Trichloroethane	71-55-6
Chloroform	67-66-3	1,1,2-Trichloroethane	79-00-5
Dichlorobenzene (Mixed)	25321-22-6	Trichloroethylene	79-01-6
1,1-Dichloroethane	75-34-3	Vinyl Acetate	108-05-4
1,2-Dichloroethane	107-06-2	Vinyl Chloride	75-01-4
Dichloropropane	26638-19-7	Xylene	1330-20-7
1,3-Dichloropropene	542-75-6	m-Xylene	108-38-3
Ethyl Acetate	141-78-6	o-Xylene	95-47-6
Hexachlorobutadiene	87-68-3	p-Xylene	106-42-3
Methyl Ethyl Ketone	78-93-3		

METHOD SUMMARY:

Analysis of air for the above volatile organic compounds can be performed by gas chromatography/mass spectrometry (GC/MS). A known volume of air is sampled through a Tenax® GC cartridge, the cartridge is thermally desorbed, and the analytes are collected in a liquid-nitrogen-cooled nickel capillary trap and introduced into a gas chromatograph for analysis by GC/MS.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are provided.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. Precision and accuracy information is furnished. The estimated detection limits (nanograms per cubic meter) for the following compounds are: benzene, 100; carbon tetrachloride, 250; chlorobenzene, 2.10; chloroform, 200; 1,2-dichloroethane, 32; tetrachloroethylene, 2.5; 1,1,1-trichloroethane, 66; trichloroethylene, 10; and tetrachlorethylene 2.5.

REFERENCES:

Plumb, R. H. Characterization of Hazarodous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Analysis of Air Samples for Volatile Organics," pp. III-46 to III-55; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Pellizzari, E. D. "Analysis for Organic Vapor Emissions near Industrial and Chemical Waste Disposal Sites," Environ. Sci. Technol., 16(11), 1982, pp. 781-785.

Krost, K. J.; Pellizzari, E. D.; Walburn, S. G.; Hubbard, S. A. "Collection and Analysis of Hazardous Organic Emissions," Anal. Chem., 54(4), 1982, pp. 810-817.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $150-325 (list).

VOLATILE ORGANIC COMPOUNDS

Soil/Sediment Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Acetone	67-64-1	Ethylbenzene	100-41-4
Acrolein	107-02-8	Methyl Bromide	74-83-9
Acrylonitrile	107-13-1	Methyl Chloride	74-87-3
Benzene	71-43-2	Methylene Chlorine	75-09-2
Bromoform	75-25-2	Methyl Ethyl Ketone	78-93-3
Carbon Disulfide	75-15-0	Methyl Isobutyl Ketone	108-10-1
Carbon Tetrachloride	56-23-5	N-Nitrosodi-n-butylamine	924-16-3
Chlorobenzene	108-90-7	N-Nitrosodiethylamine	55-18-5
Chlorodibromomethane	124-48-1	N-Nitrosodimethylamine	62-75-9
Chloroethane	75-00-3	Styrene	100-42-5
2-Chloroethyl Vinyl Ether	110-75-8	1,1,1,2-Tetrachloroethane	630-20-6
Chloroform	67-66-3	1,1,2,2-Tetrachloroethane	79-34-5
Dichlorobromomethane	75-27-4	Tetrachloroethylene	127-18-4
Dichlorodifluoromethane	75-71-8	Toluene	108-88-3
1,1-Dichloroethane	75-34-3	1,1,1-Trichloroethane	71-55-6
1,2-Dichloroethane	107-06-2	1,1,2-Trichloroethane	79-00-5
1,1-Dichloroethylene	75-35-4	Trichloroethylene	79-01-6
1,2-trans-Dichloroethylene	156-60-5	Trichloromonofluoromethane	75-69-4
Dichloropropane	26638-19-7	Vinyl Acetate	108-05-4
1,2-Dichloropropane	78-87-5	Vinyl Chloride	75-01-4
1,3-Dichloropropene	542-75-6	o-Xylene	95-47-6

METHOD SUMMARY:

Analysis for the above volatile organic compounds in soil/sediment samples can be performed by gas chromatography. A 3-gram sample of solid waste is extracted with hexadecane and screened by gas chromatography with flame ionization detection (GC/FID). If screening results are positive, another measured portion of the sample is dispersed in tetraglyme. A portion of the tetraglyme mixture is combined with water in a specially designed purging chamber, and an inert gas is bubbled through the solution at ambient temperature to transfer the volatile organics onto a Tenax® sorbent. The sorbent is then heated and the desorbed compounds are backflushed into a gas chromatograph/mass spectrometer (GC/MS). If screening results are negative, a 10-gram sample is combined with 10 milliliters of organic-free water and purged at 55°C onto a Tenax® trap. The trap is heated and the desorbed compounds are backflushed into the GC/MS.

INTERFERENCES:

The tetraglyme must be free of low-molecular-weight impurities before use, and peroxide formation should be prevented during storage by the addition of 2,6-di-tert-butyl-4-methyl phenol. Peroxides may be removed from the tetraglyme by passage through an activated alumina column.

QUALITY CONTROL:

The analytical system must be demonstrated to be free of impurities. Use of tubing, thread sealant, or components made of plastics other than TFE (e.g., Teflon®) must be avoided. Instrumental performance is checked by minimum detection limit criteria, ongoing monitoring of relative response factors, relative retention times, internal standard response factors, stability of a 3-point calibration curve, and performance of the gas chromatograph. Specifications for instrument tuning are given. Analytical performance is monitored by use of surrogates, duplicate analyses, and fortified samples. Criteria for surrogate recoveries are given.

EPA/TECHNICAL STATUS:

This method is in use in the CERCLA program and is based on a method in use in the analyses of samples under the Clean Water Act. No precision or accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Methods of Organic Chemical Analysis of Municipal and Industrial Wastewater; J. E. Longbottom, J. J. Lichtenberg, Eds., Method 624; EPA 600/4-82-057, U.S. EPA: Cincinnati, 1982.

U.S. Environmental Protection Agency, GC Screen, GC/MS Analysis of Organic Compounds, Exhibit D; Invitation for Bid (Solicitation Number WA 82-A54), U.S. EPA: Washington, July 16, 1982.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, 2nd ed., Method 8240; SW-846, U.S. EPA: Washington, 1982.

Plumb, R. H. Characterization of Hazarodous Waste Sites: A Methods Manual, Vol. III, Available Laboratory Analytical Methods, "Analysis of Air Samples for Volatile Organics," pp. III-46 to III-55; EPA-600/S4-84-038, U.S. Environmental Protection Agency: Las Vegas, 1984.

Budde, W. L.; Eichelberger, J. W. An EPA Manual for Organics Analysis Using Gas Chromatography-Mass Spectrometry, pp. 26-35; EPA-600/8-79-006, U.S. Environmental Protection Agendy, Cincinnati, 1979.

COST INFORMATION:

Cost per sample for the GC screen is approximately $90-115 (list), $60 (bid); the GC/MS analysis is approximately $170-250 (list), $115 (bid).

VOLATILE ORGANIC COMPOUNDS

Water Samples
Laboratory Method

Compound	CAS Number	Compound	CAS Number
Acetaldehyde	75-07-0	1,2-trans-Dichloroethylene	156-60-5
Acetone	67-64-1	Dichloropropane	26638-19-7
Acrolein	107-02-8	1,2-Dichloropropane	78-87-5
Acrylonitrile	107-13-1	1,3-Dichloropropene	542-75-6
Allyl Alcohol	107-18-6	Ethylene Dibromide	106-93-4
Allyl Chloride	107-05-1	Ethylbenzene	100-41-4
Amyl Acetate	628-63-7	Furfural	98-01-1
Benzene	71-43-2	Isoprene	78-79-5
Benzonitrile	100-47-0	Methyl Bromide	74-83-9
Benzyl Chloride	100-44-7	Methyl Chloride	74-87-3
Bromoform	75-25-2	Methylene Chloride	75-09-2
Butyl Acetate	123-86-4	Methyl Ethyl Ketone	78-93-3
Carbon Disulfide	75-15-0	Methyl Methacrylate	80-62-6
Carbon Tetrachloride	56-23-5	Methyl Isobutyl Ketone	108-10-1
Chlorobenzene	108-90-7	N-Nitrosodi-n-butylamine	924-16-3
Chlorodibromomethane	124-48-1	N-Nitrosodiethylamine	55-18-5
Chloroethane	75-00-3	N-Nitrosodimethylamine	62-75-9
2-Chloroethyl Vinyl Ether	110-75-8	Styrene	100-42-5
Chloroform	67-66-3	1,1,1,2-Tetrachloroethane	630-20-6
Crotonaldehyde	123-73-9	1,1,2,2-Tetrachloroethane	79-34-5
	4170-30-3	Tetrachloroethylene	127-18-4
Cyclohexane	110-82-7	Toluene	108-88-3
Dichlorobromomethane	75-27-4	1,1,1-Trichloroethane	71-55-6
Dichlorodifluoromethane	75-71-8	1,1,2-Trichloroethane	79-00-5
1,1-Dichloroethane	75-34-3	Trichloroethylene	79-01-6
1,2-Dichloroethane	107-06-2	Trichloromonofluoromethane	75-69-4
1,1-Dichloroethylene	75-35-4	Vinyl Acetate	108-05-4
		Vinyl Chloride	75-01-4
		o-Xylene	95-47-6

METHOD SUMMARY:

Analyses for the above volatile organic compounds in water samples can be performed by gas chromatography/mass spectrometry (GC/MS). An inert gas is bubbled through a 5-milliliter sample contained in a specially-designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapors are swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatogaph is temperature-programmed to separate the purgeables which are then determined with a mass spectrometer.

INTERFERENCES:

Inpurities in the purge gas, organic compound out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from laboratory contaminants under the conditions of the analysis by analzying reagent blanks. Samples can be contaminated by diffusion of organics through the septum seal during storage and handling. Contamination can also occur whenever high-level and low-level samples are analyzed sequentially.

QUALITY CONTROL:

The vapor is analyzed using multiple internal standards with quantitative determinations based on the internal standards specified in the protocol. Mass abundance calibrations, internal standard responses, and retention times must be monitored daily. The ratio of the response factors of deuterated compounds to that of non-deuterated ones must be between 0.8 and 1.2. The percent relative standard deviation for calibration check compounds must be less than 30 percent. A system performance check must be performed to assure that minimum average response factors are met before the calibration curve is used. The same check must be performed each 12 hours to assure the quality of the analytical system. A matrix spike and matrix-spike duplicate must be performed once for each case of samples, once for every 20 samples of similar concentration and/or sample matrix, whichever is greater.

EPA/TECHNICAL STATUS:

This method is in use in the CERCLA program and is based on a method in the Clean Water Act. No precision or accuracy information is furnished.

REFERENCES:

U.S. Environmental Protection Agency, Invitation for Bid, "Organics Analysis, Multi-Media, Multi-Concentration, GC/MS with Medium Level GC Screen," Solicitation Number WA84A-266; U.S. EPA: Washington, June 15, 1982.

U.S. Environmental Protection Agency, Methods of Organic Chemical Analysis of Municipal and Industrial Wastewater, J. E. Longbottom, J. J. Lichtenberg, Eds., Method 624; EPA-600/4-82-057, U.S. EPA: Cincinnati, 1982.

COST INFORMATION:

Cost per sample for the GC screen is approximately $90-115 (list); $60 (bid); the cost of the GC/MS analysis is approximately $170-250 (list), $115 (bid).

VOLATILE ORGANICS

Water Samples
Laboratory Method 1

Compound	CAS Number
Acetone	67-64-1
Benzene	71-43-2
Bis(2-chloroethyl) Ether	111-44-4
n-Butyl Alcohol	71-36-4
Chloroform	67-66-3
1,4-Dioxane	123-91-1
Ethyl Ether	60-29-7
Tetrachloroethylene	127-18-4
Tetrahydrofuran	109-99-9
Toluene	108-88-3
Trichloroethylene	79-01-6
Xylene	1330-20-7

METHOD SUMMARY:

The above volatile organics in water samples can be measured by gas chromatography/mass spectrometry (GC/MS). Samples are stored in refrigerated 125-milliliter zero-headspace glass containers after collection. The volatile organics are stripped from a 100-milliliter sample in a stirred stripping flask. The headspace gases are purged with nitrogen, and the volatiles are removed from the gas stream by passage through an adsorbent column. The volatiles are thermally desorbed from the column material and flushed through a separate loop into the GC column for temperature-programmed analysis.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

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

REFERENCE:

De Walle, F. B.; Chian, E. S. K. "Detection of Trace Organics in Well Water Near a Solid Waste Landfill," J. Am. Water Works Assn., April 1981, pp. 206-211.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $250-325 (list).

VOLATILE ORGANICS

Water Samples
Laboratory Method 2

Compound	CAS Number
Acetaldehyde	75-07-0
Acetone	67-64-1
Benzene	71-43-2
n-Butyl Alcohol	71-36-4
Chloroform	67-66-3
Ethyl Acetate	141-78-6
Ethyl Ether	60-29-7
Methanol	67-56-1
Methylene Chloride	75-09-2
Methyl Ethyl Ketone	78-93-3
Toluene	108-88-3

METHOD SUMMARY:

The above volatile organics in water samples can be determined by gas chromatography with flame ionization detection (GC/FID). The sample is gas-stripped by passing helium across the water sample, and the organics are adsorbed onto a sorbent such as Tenax GC and then thermally desorbed into the GC instrument.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

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

REFERENCE:

Kuo, P. P. K.; Chian, E. S. K.; De Walle, F. B.; Kim, J. H. "Gas Stripping, Sorption, and Thermal Desorption Procedures for Preconcentrating Volatile Polar Water-Soluble Organics from Water Samples for Analysis by Gas Chromatography," Anal. Chem., 49(7), 1977, pp. 1023-1029.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $60-90 (list).

VOLATILE ORGANIC SOLVENTS

Air Samples
Laboratory Method 1

Compound	CAS Number	Detection Limit (mg/sample)
Acetone	67-64-1	---
Benzene	71-43-2	0.01
Carbon Tetrachloride	56-23-5	0.20
Chloroform	67-66-3	0.10
1,2-Dichloroethane	107-06-2	0.05
1,4-Dioxane	123-91-1	0.05
Methylene Chloride	75-09-2	0.05
Methyl Ethyl Ketone	78-93-3	0.01
Styrene	100-42-5	0.10
Tetrachloroethylene	127-18-4	0.06
Toluene	108-88-3	0.01
1,1,1-Trichloroethane	71-55-6	0.05
1,1,2-Trichloroethane	79-00-5	0.05
Trichloroethylene	79-01-6	0.05
Xylene	1330-20-7	0.02

METHOD SUMMARY:

Gas chromatography can be used for the analysis of air for the above volatile organic solvents. A known volume of air is drawn through a charcoal tube. The trapped compounds are desorbed with carbon disulfide and an aliquot of the resulting solution is injected into a gas chromatograph equipped with flame ionization detection (GC/FID).

INTERFERENCES:

High humidity severely decreases the capacity of the sampling apparatus. Any compound having the same retention time as the analyte will interfere. Second-column confirmation is recommended.

QUALITY CONTROL:

A method blank should be prepared along with samples.

EPA/TECHNICAL STATUS:

This method is used by the National Institute for Occupational Safety and Health (NIOSH). Detections limits are listed above. Limited 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.

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, Method P&CAM 127; 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 127; Publication No. 75-121, U.S. DHEW: Cincinnati, 1975.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70-90 (list), plus sampling costs.

VOLATILE ORGANIC SOLVENTS

Air Samples
Laboratory Method 2

Compound	CAS Number
Acetone	67-64-1
n-Butyl Alcohol	71-36-3
Ethyl Acetate	141-78-6
Methyl Ethyl Ketone	78-93-3
Methyl Isobutyl Ketone	108-10-1
Toluene	108-88-3

METHOD SUMMARY:

Gas chromatography is used to determine concentrations of the above volatile organic solvents in air. The airborne solvents are sorbed onto an activated charcoal sampling tube, and desorbed into a carbon disulfide-water two-phase mixture. Each phase is analyzed separately by gas chromatography with flame ionization detection. The carbon disulfide-water mixture should be refrigerated after collection to prevent losses.

INTERFERENCES:

An alternate gas chromatographic column is necessary if toluene is to be determined separately.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. The method is sensitive to concentrations of above approximately 0.3 milligrams per cubic meter. Limited precision and accuracy information is furnished.

REFERENCE:

Langvardt, P. W.; Melcher, R. G. "Simultaneous Determination of Polar and Non-Polar Solvents in Air Using a Two-Phase Desorption From Charcoal," Am. Ind. Hyg. Assoc. J., 40, 1979, pp. 1006-1012.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70-90 (list), plus sampling costs.

WEAK ACIDS

Water Samples
Laboratory Method

Compound	CAS Number
Benzoic Acid	65-85-0
Phenol	108-95-2
Phosphoric Acid	7664-38-2
Resorcinol	108-46-3

METHOD SUMMARY:

Weak-acid anions from the above compounds in water samples can be determined by catalytic thermometric titrimetry. To a sample containing 0.1 millimole of weak acid anion, 4 milliliters dimethyl sulfoxide and 2 milliliters acrylonitrile are added. The sample solution is then titrated with 0.5 M potassium hydroxide in 2-propanol, at a rate of 0.2 milliliters per minute. The endpoint is indicated by a sharp rise in temperature from the exothermic polymerization of acrylonitrile. This method does not identify any specific weak acid or anion, but indicates total sample content of substances that can initiate the polymerization of acrylonitrile.

INTERFERENCES:

No interferences are reported.

QUALITY CONTROL:

No quality control procedures are given.

EPA/TECHNICAL STATUS:

This method is not EPA-approved. No precision or accuracy information is furnished.

REFERENCE:

Greenhow, E. J.; Shafi, A. A. "The Determination of Weak Acids in Aqueous Solution by Catalytic Thermometric Titrimetry," Talanta, 23, 1976, pp. 73-76.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $20-45 (list).

XYLENES

Air Samples
Laboratory Method 1

Compound	CAS Number
Xylene	1330-20-7
m-Xylene	108-38-3
o-Xylene	95-47-6
p-Xylene	106-42-3

METHOD SUMMARY:

The above xylenes in air can be measured by gas chromatography. 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 carbon disulfide and analysis of the resulting solution by gas chromatography with flame ionization detection (GC/FID). The method is capable of measurement of much lower levels if the desorption efficiency is adequate.

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 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 and has been validated over the range of 220 to 870 milligrams per cubic meter in a 12-liter sample. Precision and accuracy information is furnished.

REFERENCES:

U.S. Department of Health, Education, and Welfare, NIOSH Analytical Methods for Set U, Method S318, PB-262 405 (NTIS), U.S. DHEW: Washington, December 1976.

U.S. Department of Health, Education, and Welfare, NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, Method S318; Publication No. 77-157-C, U.S. DHEW: Cincinnati, 1977.

COST INFORMATION:

Cost per sample for analysis by this method is approximately $70-90 (list), plus sampling costs.

XYLENES

Air Samples
Laboratory Method 2

Compound	CAS Number
Xylene	1330-20-7
m-Xylene	108-38-3
o-Xylene	95-47-6
p-Xylene	106-42-3

METHOD SUMMARY:

Infrared absorption spectrometry can be used to determine the concentration of the above xylenes in air samples. The absorbance at 13030, 13510, and/or 12580 nm (768, 740, and/or 795 cm^{-1}) for the m-, o-, and p- isomer, respectively, is measured, using a 10-meter path length 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 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 path-length cell, is 43 milligrams per cubic meter, or 10 parts per million for each of the isomers. No precision or accuracy information is furnished.

REFERENCE:

American Public Health Association, In Methods of Air Sampling and Analysis, 2nd ed., "Infrared Absorption Spectrometry," M. Katz, Ed. APHA: Washington, 1977.

COST INFORMATION:

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

APPENDIX C

Contents

CAUTION

The following information is presented in each volume containing the appendices to the report "Analytical Methods for CERCLA Hazardous Substances."

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 brief summaries of the information contained in the references being described. Efforts were purposely made to avoid speculation about applications of methods to analytes or circumstances not documented by a reference. There is an Index/Table of Contents following the Report text, 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 the 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 determining the cost figures cited, see the text of 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.

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Appendix C

The F and K Waste Streams under the Resource Conservation and Recovery Act of 1976 (RCRA)*

* From Federal Register, 48(102), 25 May 1983, pp. 23597-23601.

Use of this Appendix - Each specific waste stream has references listed to compounds of interest and to methods which are generally applicable to potential components of that stream. For references to specific compounds, the consensual name used in the text is furnished and a notation of "Appendix A" as the location of the reference is given. For references to multi-compound methods, the name of the method and the location, as "Appendix B", is provided. This listing is not complete or exhaustive, and individual waste streams will have different characteristics. The sources of information used to compile the contents of this Appendix were: Background Document, Resource Conservation and Recovery Act, Subtitle C - Identification and Listing of Hazardous Waste, U.S. EPA Office of Solid Waste, 14 November 1980, 853 pp. and The Condensed Chemical Dictionary, Eighth ed., Van Nostrand, NY, 1972.

Waste Streams	Reference
F001:	
The following spent halogenated solvents used in degreasing and sludges from the recovery of these solvents in degreasing operations:	
(a) Tetrachloroethylene	See Tetrachloroethylene, Appendix A
(b) Trichloroethylene	See Trichloroethylene, Appendix A
(c) Methylene Chloride	See Methylene Chloride, Appendix A
(d) 1,1,1-Trichloroethane	See 1,1,1-Trichloroethane, Appendix A
(e) Carbon Tetrachloride	See Carbon Tetrachloride, Appendix A
(f) Chlorinated Fluorocarbons	See Trichloromonofluoromethane, Appendix A See Dichlorodifluoromethane, Appendix A
F002:	
The following spent halogenated solvents and the still bottoms from the recovery of these solvents:	
(a) Tetrachloroethylene	See Tetrachloroethylene, Appendix A
(b) Methylene Chloride	See Methylene Chloride, Appendix A
(c) Trichloroethylene	See Trichloroethylene, Appendix A
(d) 1,1,1-Trichloroethane	See 1,1,1-Trichloroethane, Appendix A
(e) Chlorobenzene	See Chlorobenzene, Appendix A
(f) 1,1,2-Trichloro-1,2,2-trifluoroethane	
(g) o-Dichlorobenzene	See 1,2-Dichlorobenzene, Appendix A
(h) Trichlorofluoromethane	See Trichloromonofluoromethane, Appendix A
F003:	
The following spent non-halogenated solvents and the still bottoms from the recovery of these solvents:	
(a) Xylene	See Xylene, Appendix A
(b) Acetone	See Acetone, Appendix A
(c) Ethyl Acetate	See Ethyl Acetate, Appendix A
(d) Ethylbenzene	See Ethylbenzene, Appendix A
(e) Ethyl Ether	See Ethyl Ether, Appendix A
(f) Methyl Isobutyl Ketone	See Methyl Isobutyl Ketone, Appendix A
(g) n-Butyl Alcohol	See n-Butyl Alcohol, Appendix A
(h) Cyclohexanone	See Cyclohexanone, Appendix A
(i) Methanol	See Methanol, Appendix A

(continued)

Waste Streams	Reference
F004:	
The following spent non-halogenated solvents and the still bottoms from the recovery of these solvents:	
(a) Cresols/Cresylic Acid	See Cresol, Appendix A
(b) Nitrobenzene	See Nitrobenzene, Appendix A
F005:	
The following spent non-halogenated solvents and the still bottoms from the recovery of these solvents:	
(a) Toluene	See Toluene, Appendix A
(b) Methyl Ethyl Ketone	See Methyl Ethyl Ketone, Appendix A
(c) Carbon Disulfide	See Carbon Disulfide, Appendix A
(d) Isobutanol	See iso-Butyl Alcohol, Appendix A
(e) Pyridine	See Pyridine, Appendix A
F006:	
Wastewater treatment sludges from electroplating operations except from the following processes:	
(1) sulfuric acid anodizing of aluminum;	See Aluminum, Appendix B
(2) tin plating on carbon steel;	See Cadmium, Appendix B
(3) zinc plating (segregated basis) on carbon steel;	See Chromium, Appendix B See Cyanides, Appendix B
(4) aluminum or zinc-aluminum plating on carbon steel;	See Nickel, Appendix B
(5) cleaning/stripping associated with tin, zinc and aluminum plating on carbon steel; and	
(6) chemical etching and milling of aluminum.	
F007:	
Spent cyanide plating bath solutions from electroplating operations (except for precious metals electroplating spent cyanide plating bath solutions).	See Cyanides, Appendix B

(continued)

Waste Streams	Reference
F008:	
Plating bath sludges from the bottom of plating baths from electroplating operations where cyanides are used in the process (except for precious metals electroplating plating bath sludges).	See Cyanides, Appendix B
F009:	
Spent stripping and cleaning bath solutions from electroplating operations where cyanides are used in the process (except for precious metals electroplating spent stripping and cleaning bath solutions).	See Cyanides, Appendix B
F010:	
Quenching bath sludge from oil baths from metal heat treating operations where cyanides are used in the process (except for precious metals heat treating quenching batch sludges).	See Cyanides, Appendix B
F011:	
Spent cyanide solutions from salt bath pot cleaning from metal heat treating operations (except for precious metals heat treating spent cyanide solutions from salt bath pot cleaning).	See Cyanides, Appendix B
F012:	
Quenching wastewater treatment sludges from metal heat treating operations where cyanides are used in the process (except for precious metals heat treating quenching wastewater treatment sludges).	See Cyanides, Appendix B
F013:	
Wastewater treatment sludges from the chemical conversion coating of aluminum.	See Aluminum, Appendix B See Aluminum Sulfate, Appendix A

(continued)

Waste Streams	Reference
K001:	
Bottom sediment sludge from the treatment of wastewaters from wood preserving processes that use creosote and/or pentachlorophenol.	See Pentachlorophenol, Appendix A See Creosote, Appendix A See Phenols, Appendix B
K002:	
Wastewater treatment sludge from the production of chrome yellow and orange pigments.	See Chromium, Appendix B See Lead Chromate, Appendix A
K003:	
Wastewater treatment sludge from the production of molybdate orange pigments.	See Chromium, Appendix B See Lead Chromate, Appendix A
K004:	
Wastewater treatment sludge from the production of zinc yellow pigments.	See Chromium, Appendix B See Zinc, Appendix B
K005:	
Wastewater treatment sludge from the production of chrome green pigments.	See Chromium, Appendix B
K006:	
Wastewater treatment sludge from the production of chrome oxide green pigments (anhydrous and hydrated).	See Chromium, Appendix B
K007:	
Wastewater treatment sludge from the production of iron blue pigments.	See Chromium, Appendix B See Iron, Appendix B See Potassium Dichromate, Appendix A

(continued)

Waste Streams	Reference
K008:	
Oven residue from the production of chrome oxide green pigments.	See Chromium, Appendix B See Nitrates and Sulfates, Appendix B See Nitrate Ions, Appendix B
K009:	
Distillation bottoms from the production of acetaldehyde from ethylene.	See Acetaldehyde, Appendix A See Chloroform, Appendix A See Methyl Chloride, Appendix A See Formaldehyde, Appendix A See Formic Acid, Appendix A See Methylene Chloride, Appendix A See Paraldehyde, Appendix A
K010:	
Distillation side cuts from the production of acetaldehyde from ethylene.	See Acetaldehyde, Appendix A
K011:	
Bottom stream from the wastewater stripper in the production of acrylonitrile.	See Acetaldehyde, Appendix A See Acetonitrile, Appendix A See Acrylamide, Appendix A See Acrylonitrile, Appendix A See Chloroform, Appendix A See Chloromethane, Appendix A See Cyanides, Appendix B See Formaldehyde, Appendix A See Formic Acid, Appendix A See Methylene Chloride, Appendix A See Paraldehyde, Appendix A
K013:	
Bottom stream from the acetonitrile column in the production of acrylonitrile.	See Acetonitrile, Appendix A See Acrylamide, Appendix A See Acrylonitrile, Appendix A See Cyanides, Appendix B
K014:	
Bottoms from the acetonitrile purification column in the production of acrylonitrile.	See Acetonitrile, Appendix A See Cyanides, Appendix B See Acrylonitrile, Appendix A

(continued)

Waste Streams	Reference
K015:	
Still bottoms from the distillation of benzyl chloride.	See Benzotrichloride, Appendix A See Benzyl Chloride, Appendix A See Toluene, Appendix A See Chlorobenzene, Appendix A
K016:	
Heavy ends or distillation residues from the production of carbon tetrachloride.	See Carbon Tetrachloride, Appendix A See Hexachloroethane, Appendix A See Hexachlorobutadiene, Appendix A See Hexachlorobenzene, Appendix A See Tetrachloroethylene, Appendix A
K017:	
Heavy ends (still bottoms) from the purification column in the production of epichlorohydrin.	See Epichlorohydrin, Appendix A
K018:	
Heavy ends from the fractionation column in ethyl chloride production.	See Chloroethane, Appendix A See 1,2-Dichloroethane, Appendix A See Trichloroethylene, Appendix A
K019:	
Heavy ends from the distillation of ethylene dichloride in ethylene dichloride production.	See Carbon Tetrachloride, Appendix A See Chloroform, Appendix A See 1,1,2-Trichloroethane, Appendix A See 1,1,2,2-Tetrachloroethane, Appendix A See 1,1-Dichloroethylene, Appendix A See 1,1,1-Trichloroethane, Appendix A
K020:	
Heavy ends from the distillation of vinyl chloride in vinyl chloride monomer production.	See Carbon Tetrachloride, Appendix A See Chloroform, Appendix A See 1,1,2-Trichloroethane, Appendix A See 1,1,2,2-Tetrachloroethane, Appendix See 1,1-Dichloroethylene, Appendix A See 1,1,1-Trichloroethane, Appendix A See Vinyl Chloride, Appendix A

(continued)

Waste Streams	Reference
K021:	
Aqueous spent antimony catalyst waste from fluoromethanes production.	See Antimony, Appendix B See Chloroform, Appendix A See Carbon Tetrachloride, Appendix A
K022:	
Distillation bottom tars from the production of phenol/acetone from cumene.	See Phenol, Appendix A See Acetone, Appendix A See Cumene, Appendix A See Polycyclic Aromatic Hydrocarbons, Appendix B
K023:	
Distillation light ends from the production of phthalic anhydride from naphthalene.	See Vanadium Pentoxide, Appendix A See Phthalic Anhydride, Appendix A See Naphthalene, Appendix A See 1,4-Naphthoquinone, Appendix A
K024:	
Distillation bottoms from the production of phthalic anhydride from naphthalene.	See Vanadium Pentoxide, Appendix A See Phthalic Anhydride, Appendix A See Naphthalene, Appendix A See Phthalate Esters, Appendix B
K025:	
Distillation bottoms from the production of nitrobenzene by the nitration of benzene.	See Sulfuric Acid, Appendix A See Nitric Acid, Appendix A See Benzene, Appendix A See Nitrobenzene, Appendix A See m-Dinitrobenzene, Appendix A See 2,4-Dinitrotoluene, Appendix A
K026:	
Stripping still tails from the production of methyl ethyl pyridines.	See Paraldehyde, Appendix A See 2-Picoline, Appendix A See Pyridine, Appendix A

(continued)

Waste Streams	Reference
K027:	
Centrifuge and distillation residues from toluene diisocyanate production.	See Phosgene, Appendix A See Hydrochloric Acid, Appendix A See Toluenediamine, Appendix A See Toluene Diisocyanate, Appendix A
K028:	
Spent catalyst from the hydrochlorinator reactor in the production of 1,1,1-trichloroethane.	See Chloroform, Appendix A See 1,2-Dichloroethane, Appendix A See 1,1-Dichloroethylene, Appendix A See 1,1,1,2-Tetrachloroethane, Appendix A See 1,1,2,2-Tetrachloroethane, Appendix A See 1,1,1-Trichloroethane, Appendix A
K029:	
Waste from the product steam stripper in the production of 1,1,1-trichloroethane.	See Chloroform, Appendix A See 1,2-Dichloroethane, Appendix A See 1,1-Dichloroethylene, Appendix A See 1,1,1,2-Tetrachloroethane, Appendix A See 1,1,2,2-Tetrachloroethane, Appendix A See 1,1,1-Trichloroethane, Appendix A
K030:	
Column bottoms or heavy ends from the combined production of trichloroethylene and perchloroethylene.	See Chloroethane, Appendix A See Hexachloroethane, Appendix A See Hexachlorobenzene, Appendix A See 1,1,2,2-Tetrachlorobenzene, Appendix A
K031:	
By-product salts generated in the production of MSMA and cacodylic acid.	See Arsenic, Appendix B
K032:	
Wastewater treatment sludge from the production of chlordane.	See Chlordane, Appendix A See Hexachlorocyclopentadiene, Appendix A

(continued)

Waste Streams	Reference
K033:	
Wastewater and scrub water from the chlorination of cyclopentadiene in the production of chlordane.	See Chlordane, Appendix A See Hexachlorocyclopentadiene, Appendix A
K034:	
Filter solids from the filtration of hexachlorocyclopentadiene in the production of chlordane.	See Chlordane, Appendix A See Hexachlorocyclopentadiene, Appendix A See Heptachlor, Appendix A
K035:	
Wastewater treatment sludges generated in the production of creosote.	See Creosote, Appendix A See Phenol, Appendix A See Polycyclic Aromatic Hydrocarbons, Appendix B
K036:	
Still bottoms from toluene reclamation distillation in the production of disulfoton.	See Disulfoton, Appendix A See Hydrochloric Acid, Appendix A See Toluene, Appendix A
K037:	
Wastewater treatment sludges from the production of disulfoton.	See Disulfoton, Appendix A See Hydrochloric Acid, Appendix A
K038:	
Wastewater from the washing and stripping of phorate production.	See Disulfoton, Appendix A See Formaldehyde, Appendix A See Hydrochloric Acid, Appendix A See Phorate, Appendix A
K039:	
Filter cake from the filtration of diethylphosphorodithioic acid in the production of phorate.	See Disulfoton, Appendix A See Hydrochloric Acid, Appendix A See Phorate, Appendix A

(continued)

Waste Streams	Reference
K040:	
Wastewater treatment sludge from the production of phorate.	See Disulfoton, Appendix A See Hydrochloric Acid, Appendix A See Phorate, Appendix A
K041:	
Wastewater treatment sludge from the production of toxaphene.	See Toxaphene, Appendix A
K042:	
Heavy ends or distillation residues from the distillation of tetrachlorobenzene in the production of 2,4,5-T.	See o-Dichlorobenzene, Appendix A See Hexachlorobenzene, Appendix A See Tetrachlorobenzene, Appendix A
K043:	
2,6-Dichlorophenol waste from the production of 2,4-D.	See 2,4-Dichlorophenol, Appendix A See 2,6-Dichlorophenol, Appendix A See 2,4,6-Trichlorophenol, Appendix A
K044:	
Wastewater treatment sludges from the manufacturing and processing of explosives.	See sym-Trinitrobenzene, Appendix A See Lead, Appendix B
K045:	
Spent carbon from the treatment of wastewater containing explosives.	See sym-Trinitrobenzene, Appendix A See Lead, Appendix B
K046:	
Wastewater treatment sludges from the manufacturing, formulation and loading of lead-based initiating compounds.	See Lead, Appendix B
K047:	
Pink/red water from TNT operations.	See sym-Trinitrobenzene, Appendix A

(continued)

Waste Streams	Reference
K048:	
Dissolved air flotation (DAF) float from the petroleum refining industry.	See Chromium, Appendix B See Lead, Appendix B
K049:	
Slop oil emulsion solids from the petroleum refining industry.	See Chromium, Appendix B See Lead, Appendix B
K050:	
Heat exchanger bundle cleaning sludge from the petroleum refining industry.	See Chromium, Appendix B See Lead, Appendix B
K051:	
API separator sludge from the petroleum refining industry.	See Chromium, Appendix B See Lead, Appendix B
K052:	
Tank bottoms (leaded) from the petroleum refining industry.	See Chromium, Appendix B See Lead, Appendix B
K060:	
Ammonia still lime sludge from coking operations.	See Cyanides, Appendix B See Naphthalene, Appendix A See Phenolic Compounds, Appendix B See Phenolics, Appendix B See Polycyclic Aromatic Hydrocarbons, Appendix B
K061:	
Emission control dust/sludge from the primary production of steel in electric furnaces.	See Cadmium, Appendix B See Chromium, Appendix B See Lead, Appendix B

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Waste Streams	Reference
K062:	
Spent pickle liquor from steel finishing operations.	See Chromium, Appendix B See Sulfuric Acid, Appendix A See Hydrochloric Acid, Appendix A See Lead, Appendix B See Phosphoric Acid, Appendix A
K069:	
Emission control dust/sludge from secondary lead smelting.	See Cadmium, Appendix B See Chromium, Appendix B See Lead, Appendix B
K071:	
Brine purification muds from the mercury cell process in chlorine production, where separately prepurified brine is not used.	See Mercury, Appendix B
K073:	
Chlorinated hydrocarbon waste from the purification step of the diaphragm cell process using graphite anodes in chlorine production.	See Carbon Tetrachloride, Appendix A See Chloroform, Appendix A See Dichloroethylene, Appendix A See Hexachloroethane, Appendix A See 1,1,2,2-Tetrachloroethane, Appendix See Tetrachloroethene, Appendix A See Trichloroethane, Appendix A
K083:	
Distillation bottoms from aniline extraction.	See Aniline, Appendix A See Diphenylamine, Appendix A See Nitrobenzene, Appendix A See Chlorobenzene, Appendix A
K084:	
Wastewater treatment sludges generated during the production of veterinary pharmaceuticals from arsenic or organic-arsenic compounds.	See Arsenic, Appendix B

(continued)

Waste Streams	Reference
K085:	
Distillation or fractionation column bottoms from the production of chlorobenzenes.	See Benzene, Appendix A See Chlorine, Appendix A See Dichlorobenzene, Appendix A
K086:	
Solvent washes and sludges, caustic washes and sludges, or water washes and sludges from cleaning tubs and equipment used in the formulation of ink from pigments, driers, soaps, and stabilizers containing chromium and lead.	See Chromates, Appendix B See Chromium, Appendix B See *Hexavalent Chromium*, Appendix B See Lead, Appendix B
K087:	
Decanter tank tar sludge from coking operations.	See Phenol, Appendix A See Naphthalene, Appendix A
K093:	
Distillation light ends from the production of phthalic anhydride from ortho-xylene.	See Phthalate Esters, Appendix B See Phthalic Anhydride, Appendix A See o-Xylene, Appendix A
K094:	
Distillation bottoms from the production of phthalic anhydride from ortho-xylene.	See Phthalate Esters, Appendix B See Phthalic Anhydride, Appendix A See o-Xylene, Appendix A
K095:	
Distillation bottoms from the production of 1,1,1-trichloroethane.	See 1,1,1-Trichloroethane, Appendix A
K096:	
Heavy ends from the heavy ends column from the production of a,a,a-trichlorethane, 1,1,1.	See 1,1,1-Trichloroethane, Appendix A

(continued)

Waste Streams	Reference
K097:	
Vacuum stripper discharge from the chlordane chlorinator in the production of chlordane.	See Chlordane, Appendix A See Toxaphene, Appendix A
K098:	
Untreated process wastewater from the production of toxaphene.	See Toxaphene, Appendix A
K099:	
Untreated wastewater from the production of 2,4-D.	See 2,4-D, Appendix A
K100:	
Waste leaching solution from acid leaching of emission control dust/sludge from secondary lead smelting. (Components of this waste are identical with those of K069.)	See Lead, Appendix B See Cadmium, Appendix B See Chromium, Appendix B
K101:	
Distillation tar residues from the distillation of aniline-based compounds in the production of veterinary pharmaceuticals from arsenic or organo-arsenic compounds.	See Arsenic, Appendix B
K102:	
Residue from the use of activated carbon for decolorization in the production of veterinary pharmaceuticals from arsenic or organo-arsenic compounds.	See Aniline, Appendix A See Arsenic, Appendix B
K103:	
Process residues from aniline extraction from the production of aniline.	See Aniline, Appendix A See Nitrobenzene, Appendix A

(continued)

Waste Streams	Reference
K104:	
Combined wastewater streams generated from nitrobenzene/aniline chlorobenzenes.	See Ammonia, Appendix A See Aniline, Appendix A See Chlorobenzene, Appendix A See Nitrobenzene, Appendix A
K105:	
Separated aqueous stream from the reactor product washing step in the production of chlorobenzenes.	See Benzene, Appendix A See Chlorine, Appendix A See Chlorobenzene, Appendix A See Dichlorobenzene, Appendix A See Hexachlorobenzene, Appendix A See Pentachlorobenzene, Appendix A See Tetrachlorobenzene, Appendix A See Trichlorobenzene, Appendix A See 2,4,6-Trichlorophenol, Appendix A
K106:	
Wastewater treatment sludge from the mercury cell process in chlorine production.	See Mercury, Appendix B