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OPERABLE UNIT 3 WORK PLAN ADDENDUM
VOLUME 4 OF 4 ATTACHMENT D.I
JUNE 1992

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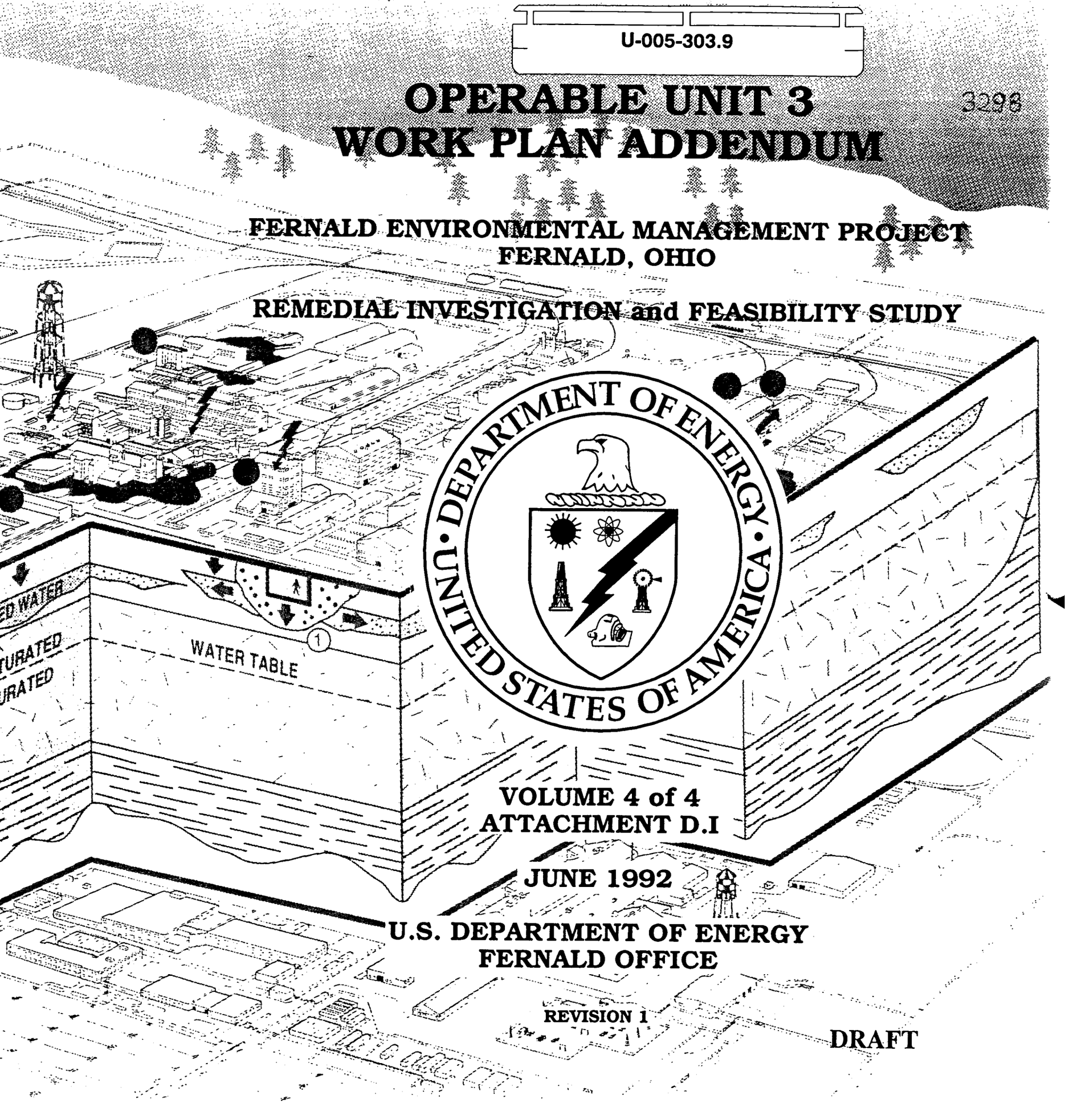
OPERABLE UNIT 3
WORK PLAN ADDENDUM

FERNALD ENVIRONMENTAL MANAGEMENT PROJECT
FERNALD, OHIO

REMEDIAL INVESTIGATION and FEASIBILITY STUDY



VOLUME 4 of 4
ATTACHMENT D.I

JUNE 1992

U.S. DEPARTMENT OF ENERGY
FERNALD OFFICE

REVISION 1

DRAFT

OPERABLE UNIT 3
WORK PLAN ADDENDUM

3298

FERNALD ENVIRONMENTAL MANAGEMENT PROJECT
FERNALD, OHIO

REMEDIAL INVESTIGATION AND FEASIBILITY STUDY

VOLUME 4 OF 4
ATTACHMENT D.I

JUNE 1,1992

U.S DEPARTMENT OF ENERGY
FERNALD OFFICE

DRAFT

ATTACHMENT D.I:

3298

ABSTRACTS OF PROCEDURES FOR THE OPERABLE UNIT 3 FIELD PROGRAM

Attachment D.I to the Sampling and Analysis Plan (SAP) contains 237 abstracts of the procedures (methods) which support the plan. The actual procedures are contained in the Sitewide CERCLA Quality Assurance Project Plan (SCQ).

Attachment D.I is subdivided into four abstract sections:

D.I.1 Field Procedures, Health and Safety

D.I.2 Field Procedures, Instrumental
- Radiological
- Chemical
- Physical

D.I.3 Field Procedures, Sampling

D.I.4 Laboratory Analytical Procedures
- Organic Compounds, RCRA Characteristics, and Metals
- Conventional Parameters
- Radiometric
- Geotechnical Parameters

Each abstract provides selected general information about the method including the method or procedure number, associated title, scope and application statement, and a method summary description.

This submittal contains abstracts of all the methods which are judged needed to support the SAP. Of the 237 procedures, 127 are contained in the SCQ. The remaining 110 procedures will be completed and added to the SCQ over the next six months, which will allow sampling and analysis to proceed on schedule.

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* Procedures shown in *italics* are being prepared [i.e., procedures are not currently contained in Sitewide CERCLA Quality Assurance Project Plan (SCQ)]

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ATTACHMENT D.I:

ABSTRACTS OF PROCEDURES FOR THE OPERABLE UNIT 3 FIELD PROGRAM

D.I.1 FIELD PROCEDURES, HEALTH AND SAFETY

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OPERATION OF HEAT STRESS MONITOR (PROCEDURE 707)

1.0 Scope and Application

This procedure establishes the responsibilities and methods for the use and care of a Heat Stress Monitor. This equipment is used to monitor the effective temperature of an environment for the protection of workers against heat related illness.

2.0 Method Summary

A Heat Stress monitor measures the collective total of environmental and physical work factors (light/heavy workload, impervious clothing, etc.) that constitute the total heat load imposed on the body. This monitoring ensures that the level of total heat stress on a worker stays below limits that are accompanied by an increased risk of illness.

HEAT STRESS (PROCEDURE 909)

1.0 Scope and Application

This procedure establishes the responsibilities and methods used for the control of workers exposed to hot work environments.

2.0 Method Summary

Exposure of workers to hot environments puts a worker under an environmental stress. Requirements for heat stress monitoring and the determination of worker work/rest cycles are discussed. Controls for minimizing stress through the use of proper clothing and the following of proper care and work regimens are discussed.

MONITORING OF PERSONNEL FOR RADIOACTIVE CONTAMINATION (PROCEDURE 900)

1.0 Scope and Application

This procedure establishes the responsibilities and methods for the monitoring of personnel for radioactive contamination.

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2.0 Method Summary

All personnel exiting an exclusion zone where radioactive contaminants are suspected are required to monitor themselves with a beta/gamma survey instrument and an alpha survey instrument. This monitoring is performed to establish the absence or presence of radioactive contamination on the person or his clothing.

ENTRANCE AND EXIT REQUIREMENTS FOR EXCLUSION AREAS (PROCEDURE 901)

1.0 Scope and Application

This procedure establishes the responsibilities and methods used for the control of personnel access to exclusion areas. Control of personnel access to an exclusion area is required to minimize worker exposure to hazards within the work zone.

2.0 Method Summary

Requirements for the entry and exit of exclusion zones are discussed. This includes monitoring requirements and sign-in and sign-out requirements.

SITE IDENTIFICATION AND CONTROL (PROCEDURE 906)

1.0 Scope and Application

This procedure establishes the responsibilities and methods used for the identification and control of personnel work areas.

2.0 Method Summary

All work areas are identified through the use of caution tape for an exclusion area and traffic cones for a support zone. Posting requirements are discussed for the different work areas.

HANDLING, USE, AND STORAGE OF CHEMICALS IN THE FIELD (PROCEDURE 907)

1.0 Scope and Application

This procedure establishes the responsibilities and methods used for the control of chemicals in the field.

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2.0 Method Summary

Chemical exposure to workers in the field is to be minimized, particularly to chemicals that have hazardous properties. Storage, use and handling requirements are all part of a hazardous chemical control program.

COLD STRESS (PROCEDURE 909)

1.0 Scope and Application

This procedure establishes the responsibilities and methods used for the control of workers exposure to cold work environments.

2.0 Method Summary

Exposure of workers to cold environments can place a worker under an environmental stress. Requirements for cold stress monitoring and the determination of proper work/rest cycles are discussed. Controls for minimizing stress through the use of proper clothing and the following of proper care and work regimens are discussed.

CONFINED SPACE ENTRY (PROCEDURE 910)

1.0 Scope and Application

This procedure establishes the responsibilities, methods and precautions which must be used when work is to be conducted in a confined space.

2.0 Method Summary

Training, monitoring, entry requirements, prevention of unauthorized entry, rescue, protection measures, and duty to other employee requirements are discussed for personnel entry into a confined space.

Attachment D.1.2
Instrumental

D.I.2 FIELD PROCEDURES, INSTRUMENTAL*

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OPERATION OF LUDLUM MODEL 12 (PROCEDURE 605a)

1.0 Scope and Application

This procedure establishes responsibilities and methods for the use and care of a Model 12 Survey Meter. This meter can be used with a beta/gamma sensitive probe or an alpha scintillation probe for the detection of surface contamination on equipment or personnel.

2.0 Method Summary

The Model 12 Survey Meter, when used with a scintillation probe, is capable of monitoring surface contamination resulting from alpha emitting radionuclides. The probe cannot be used to identify various alpha emitting radionuclides, but can only be used to establish the presence of alpha contamination.

OPERATION OF LUDLUM MODEL 12 (PROCEDURE 605b)

1.0 Scope and Application

This procedure establishes responsibilities and methods for the use and care of a Model 12 Survey Meter. This meter can be used with a beta/gamma sensitive probe or an alpha scintillation probe for the detection of surface contamination on equipment or personnel.

2.0 Method Summary

The Model 12 Survey Meter, when used with a pancake GM (geiger muller) probe, is capable of monitoring surface contamination resulting from beta emitting radionuclides. The probe cannot be used to identify various beta emitting radionuclides, but can only be used to establish the presence of beta contamination.

*** Includes instrumental field procedures for radiological, chemical, and physical parameters.**

OPERATION OF LUDLUM MODEL 19 MICRO R METER (PROCEDURE 606)

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1.0 Scope and Application

This procedure establishes responsibilities and methods for the use and care of a Model 19 Micro R Meter. This instrument measures gamma flux and provides a readout of micro R per hour.

2.0 Method Summary

The Model 19 is a survey instrument that can be used to measure the gamma fields associated with surface contamination and sources. The instrument is normally used as a field survey meter in areas where the ambient gamma flux results in an exposure rate less than 5 mR/hr.

OPERATION OF EBERLINE ESP WITH NAI PROBE (PROCEDURE 608)

1.0 Scope and Application

This procedure establishes responsibilities and methods for the use and care of the Eberline ESP ratemeter with a NaI probe. This system can be used for measuring gamma emission from surface contamination in buildings and/or structures.

2.0 Method Summary

The ESP monitor with a NaI detector allows for direct monitoring of surface contamination in a survey mode with data output in a countrate mode. In a count mode the detector can be placed in a standard geometry and allowed to count for a fixed time interval. Total counts can be measured during the counting interval.

OPERATION OF REUTER STOKES MODEL RSS-111 PRESSURIZED ION CHAMBER (PIC) (PROCEDURE 609)

1.0 Scope and Application

This procedure establishes responsibilities and methods for the use and care of a Model RSS-111 Pressurized Ion Chamber (PIC). This instrument measures and records gamma exposure rates and provides a readout of micro R per hour.

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2.0 Method Summary

The PIC is a monitoring system that can be used to determine the low-level gamma-ray exposure rates such as those due to fallout and natural background. The instrument is normally used as a secondary standard instrument for determining external gamma exposure rates at a height of one meter above the soil surface.

OPERATION OF THE CANBERRA MODEL 2400 ALPHA/BETA/GAMMA GAS FLOW PROPORTIONAL COUNTER (PROCEDURE 610)

1.0 Scope and Application

This procedure establishes responsibilities and methods for the use and care of the Canberra Model 2400 Alpha/Beta/Gamma gas flow proportional counter. The Alpha/Beta/Gamma system is designed specifically to measure very low activity levels of alpha, beta, and gamma radiation in environmental samples.

2.0 Method Summary

The Alpha/Beta/Gamma system is a dual gas proportional detector assembly which is used to capture radioactive emissions from the sample. The system consists of a large area guard detector and a thin window sample detector. The guard detector is used to detect extraneous radiation (background) which typically comes from cosmic radiation, lead x-rays, or gamma radiation from the sample. The sample detector captures the electronic pulses.

OPERATION OF THE THOMSON AND NEILSON RADON WORKING LEVEL (WL) METER (PROCEDURE 611)

1.0 Scope and Application

This procedure establishes responsibilities and methods for the use and care of the RADON WL METER from Thomson and Nielsen Electronics. This instrument is designed for measurement of radon working level in indoor environments such as residential and commercial buildings.

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2.0 Method Summary

The RADON WL METER is a hand-held instrument which operates on the principle that air containing radionuclides is sampled using a small, continuously operating pump and the radon daughter products are deposited on a filter. As alpha particles emitted from the filter are counted, the total alpha count is displayed on a digital liquid crystal display. The flow rate of the air and the total alpha count in a sampling period determine the radon working level.

OPERATION OF EXOTOX PORTABLE GAS MONITOR (PROCEDURE 704)

1.0 Scope and Application

This procedure establishes the responsibilities and methods for the use and care of a Exotox Model 50 or 75. This meter can be used to detect the presence of flammable atmospheres, oxygen concentrations, carbon monoxide concentrations, and other gases.

2.0 Method Summary

An Exotox can be used to measure ambient air in a building and/or confined space to determine if the air is hazardous to workers entering the environment. The existing capabilities allow for the detection of percent oxygen, amount of hydrogen sulfide, carbon monoxide in ppm, lower explosive level of flammable gases, relative humidity and ambient temperature.

OPERATION OF A PHOTOIONIZATION DETECTOR (PROCEDURE 705)

1.0 Scope and Application

This procedure establishes the responsibilities and methods for the use and care of a HNU Photoionization Detector. This meter can be used to detect the presence of organic vapors that may be hazardous to worker health.

2.0 Method Summary

A HNU Photoionization Detector is used to monitor the breathing zone, ambient air, or confined space air for organic vapors. Nominally the detector used will detect the presence of gasses having a photoionization potential above 10.2 eV. Instrument response is normalized to isobutylene and reads out in parts per million.

OPERATION OF DRAGER TUBES (PROCEDURE 706)

3298

1.0 Scope and Application

This procedure establishes the responsibilities and methods for the use and care of Drager Tube equipment. This equipment is capable of quantifying concentrations of airborne chemicals and can measure the CO content of a worker's blood.

2.0 Method Summary

Drager Tube equipment consists of a hand pump and selected chemical indicator tubes. When exposed to a specific airborne chemical the indicator material will change color in proportion to the concentration of the contaminant. This equipment is simple to use but relies on the user having a pre-knowledge of what chemical contaminant(s) or general category of chemical (i.e., ketones, aromatic hydrocarbons, alcohols, etc.) may be present.

OPERATION OF ELF FIELD STRENGTH METER (PROCEDURE 709)

1.0 Scope and Application

This procedure establishes the responsibilities and methods for the use and care of an ELF Field Strength Meter. This meter is used to determine magnetic and electrical field strength around electrical transmission lines, power substations, electrical generating equipment, and other equipment that may generate magnetic or electrical fields.

2.0 Method Summary

An ELF Field Strength Meter can be used to measure electrical and magnetic fields in the work environment. These fields can be generated by equipment or the result of electrical power transmission.

OPERATION OF A QUEST NOISE DOSIMETER (PROCEDURE 714)

1.0 Scope and Application

This procedure establishes the responsibilities and methods for the use and care of a Quest Noise Dosimeter. This equipment measures cumulative exposure to noise.

2.0 Method Summary

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A Quest Noise Dosimeter is a measuring device which integrates, over a work period, the sound levels, measured in decibels, experienced in a work environment. The meter measures and accumulates noise and produces a continuous reading of the percentage of the OSHA allowable exposure to which the worker has been subjected. If the allowable level has been exceeded, the worker is placed into a Hearing Conservation Program.

OPERATION OF A TAYLOR SLING PSYCHROMETER (PROCEDURE 715)

1.0 Scope and Application

This procedure establishes the responsibilities and methods for the use and care of a Taylor Sling Psychrometer. This equipment is used to determine relative humidity through obtaining the temperature from a dry-bulb and wet-bulb thermometer.

2.0 Method Summary

The values obtained are used for ambient temperature measurements and for affecting factors during compliance air sampling.

OPERATION OF A TYPE 2 SOUND LEVEL METER (PROCEDURE 718)

1.0 Scope and Application

This procedure establishes the responsibilities and methods for the use and care of a Type 2 Sound Level Meter. This equipment is used to determine if noise levels exceed permissible levels.

2.0 Method Summary

A Type 2 Sound Level Meter is used to measure continuous as well as impact or impulsive noise levels from equipment operation or the work environment. The levels obtained determine whether the exposure is within permissible limits set by 29 CFR 1926.52, Occupational Noise Exposure.

OPERATION OF THE ORGANIC VAPOR ANALYZER (OVA) (PROCEDURE 720)

1.0 Scope and Application

This procedure establishes the responsibilities and methods for the use and care of the OVA. This equipment is used to monitor trace quantities of organic materials and toxic organic vapors in air.

2.0 Method Summary

The OVA measures total organic vapor concentration by producing a response to an unknown sample which is related to a gas of known composition to which the instrument has been previously calibrated. The instrument is a portable field survey instrument used to identify air contaminants in areas where a toxic atmosphere may exist.

USE OF pH METER (PROCEDURE 721)

1.0 Scope and Application

This procedure establishes the responsibilities and methods for the use and care of a pH meter. This meter is used for the determination of the pH value of a liquid or solid/aqueous mixture matrix. A pH meter must be accurate, reproducible to 0.1 pH unit with a range of 0 to 14, and equipped with a temperature-compensation adjustment. The pH meter consists of a potentiometer, a glass electrode, a reference electrode, and a temperature-compensating device. Instrument failure may occur within the potentiometer or the electrode probes if not properly maintained.

2.0 Method Summary

The basic principle of using a pH meter is to measure the activity of the hydrogen ions by potentiometric measurement using a standard hydrogen electrode and a reference electrode. The electromotive force produced in the glass electrode system varies linearly with pH. By calibrating the instrument with solutions of known pH values, a sample's pH may be determined to increments of 0.001 units (instrument specific).

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POLYCHLORINATED BIPHENYLS (PCBs) DETERMINATION IN SOILS AND OILS BY DEXSIL® SCREENING KITS (PROCEDURE 722)

1.0 Scope and Application

This procedure applies to the screening of soil and oil matrices for PCBs. The method determines the level of chlorides present from PCB compounds by comparing the final color of a treated sample extract to a colormetric chart on each Dexsil® kit. Each screening test is performed in ten minutes or less.

2.0 Method Summary

A known amount of oil and soil is mixed with an extraction solvent. The soil mixture is allowed to settle and passed through a filter to remove inorganic chlorides. Sodium and a catalyst are then added to the extraction solvent for both oil and soil samples to strip the chlorides from the PCB molecule forming sodium chloride. A buffer solution in water is added to the extraction solvent which neutralizes the excess sodium and extracts the sodium chloride into the water. The chlorine in the water layer reacts with a known amount of mercuric nitrate and indicator solution to emit a clear to purple color indicating the level of PCBs present.

DETERMINATION OF HUMIDITY USING A HYGROMETER (PROCEDURE 723)

USE OF VWR SCIENTIFIC HYGROMETER

1.0 Scope and Application

This procedure establishes the responsibilities and methods for the use and care of a VWR Scientific digital hygrometer. This instrument can be used to detect the presence of humidity in air.

2.0 Method Summary

The instrument is calibrated at the factory to a National Institute of Standards and Technology-traceable calibration standard. The instrument's upper switch is placed to the ON position and the lower switch is placed to the %RH position. The instrument is allowed to read the air for up to 3.5 minutes or until stabilization is met. The measuring relative humidity range is from 10.0 to 95.0%.

USE OF A CONDUCTIVITY METER (PROCEDURE 724)

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1.0 Scope and Application

Conductivity is the numerical expression for the ability of an aqueous solution to carry an electric current. This ability depends on the presence of ions, their concentration, mobility, valence, and relative concentrations, and on the temperature of measurement. This procedure applies to the determination of conductivity of water or a soil extract. Conductivity is measured using an instrument consisting of a source of alternating current, a Wheatstone bridge, a null indicator, and a conductivity cell. The conductivity of soil is derived from extracting a known amount of soil with a known volume of water.

2.0 Method Summary

The conductivity meter is standardized with one or two aqueous solutions of known conductivity by immersing the conductivity cell electrode into the standard solution until the reading stabilizes. The meter is adjusted until the calibrated level is displayed for each standard. The probe is then inserted in an aqueous sample or soil extract to obtain the conductivity value. The meter range selector may need to be adjusted to obtain a conductivity reading on scale.

Attachment D.1.3
Sampling

D.I.3 FIELD PROCEDURES, SAMPLING

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SURFACE-WATER SAMPLING (PROCEDURE K.4.3)

1.0 Scope and Application

This method describes procedures applicable to the collection of water samples from basins and other areas. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the basins and other areas. Grab sampling and composite sampling are the two techniques that are used for collecting surface-water samples.

2.0 Method Summary

2.1 Grab samples are collected in clean sample containers using the appropriate preservatives approved for specific parameters of interest. If not collected directly from a body of water, non-volatile samples are obtained using a peristaltic pump or Kemerer sampler. Sample containers are filled as specified per parameter.

2.2 Composite samples can be collected manually or with automatic sampling equipment. Collection procedures for composite samples, if needed, will be included in PSPs.

SAMPLING FOR WATER AND AQUEOUS LIQUIDS FROM DRUMS (PROCEDURE K.4.3.4)

1.0 Scope and Application

This method describes the collection of water and aqueous liquid samples from drums, primary safety considerations, and techniques for proper compositing.

2.0 Method Summary

Specialized equipment is necessary for collecting water and aqueous liquids from drums due to the limited access and because long term storage sometimes causes stratification of the contents of the drums. Zone sampling devices are capable of collecting a sample from a predetermined level, can be constructed of a non-sparking material, and are small enough to pass through a bung hole without scraping the sides. Glass thieving rods can be used to characterize a column of the contents of drums by observation and can be used to composite a liquid sample directly into the appropriate sample bottle. Thieving rods are

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pre-cleaned, disposable, and relatively inexpensive. Zone sampling devices can be totally disassembled for thorough decontamination between sampling events.

WASTE WATER SAMPLING (PROCEDURE K.4.4)

1.0 Scope and Application

This method is applicable to the collection of waste water samples from basins and other areas as specified in PSPs. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the wastewater. The PSPs also specify the preservation and security of samples being delivered to the FEMP analytical section.

2.0 Method Summary

Most waste water samples are collected with automatic sampling equipment; however, grab samples are required for oil, grease and cyanide. Samples from sumps are collected by the grab sampling method. Grab samples are collected by lowering the sample container into the applicable tank or effluent stream.

ORGANIC LIQUID SAMPLING (PROCEDURE K.4.4.1)

1.0 Scope and Application

This procedure is applicable to the collection of organic liquids, especially those in discrete layers overlying aqueous layers. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the liquid.

2.0 Method Summary

The sampling equipment is decontaminated using procedure K.11 prior to use. Skimming procedures are employed to obtain a discrete organic phase sample. A sufficient amount of sample is collected and placed into a shipping container. Samples requiring refrigeration are stored and shipped in an ice chest.

COLLECTION OF QA/QC SAMPLES (PROCEDURE K.4.5)

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1.0 Scope and Application

This method is applicable to the collection of QA/QC samples in accordance with the specified ASL and project-specific DQOs for aqueous samples.

2.0 Method Summary

2.1 Trip blanks are prepared in a controlled environment and accompany each shipping container of aqueous samples analyzed for VOCs unless specifically omitted in PSPs. De-ionized water is poured into the sample container specified for the parameter of interest.

2.2 Rinsate samples are decontamination samples that are obtained by pouring organic-free, de-ionized water over the equipment in the equipment in the container specified for the parameter of interest.

2.3 Duplicate and split samples are collected in the same manner as actual samples with the exception that samples are composite and distributed evenly between containers and while filling. Samples are not composited for VOC analysis.

2.4 Container blanks are empty containers that are submitted to the laboratory to check for constituents of concern.

2.5 Matrix spike/matrix duplicates are collected at triple sample volume every twentieth sample or every sampling round whichever is more frequent, and are handled in the same manner as other samples.

SAMPLING FOR ORGANIC RESIDUES IN TANKS, DUCTS, PIPES (PROCEDURE K.4.7.1)

1.0 Scope and Application

This procedure establishes the guidelines for collecting organic residues from vessels.

2.0 Method Summary

2.1 Sampling organic residues in tanks requires several different techniques, each depending on the consistency of the material as well as the accessibility of the vessel and its contents. In all cases, the sampling equipment will be decontaminated using procedure K.11 prior to use.

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2.2 This procedure defines the applications and limitations of the various sampling devices available and gives guidance to the appropriate selections.

SAMPLING FOR INORGANIC RESIDUES IN TANKS, DUCTS, PIPES (PROCEDURE K.4.7.3)

1.0 Scope and Application

This procedure establishes the guidelines for collecting inorganic residues from vessels.

2.0 Method Summary

2.1 Techniques specific to the consistency of the residue as well as the accessibility of the vessel and its contents are presented. In all cases, the sampling equipment will be decontaminated using procedure K.11 prior to use.

2.2 This procedure defines the applications and limitations of the various sampling devices available and gives guidance to the appropriate selections. Provisions for sample storage and preservations are analyte specific.

WATER AND AQUEOUS LIQUIDS FROM PRESSURIZED PIPES (PROCEDURE K.4.8.1)

1.0 Scope and Application

This procedure is applicable to the collection of water and/or aqueous liquids from pressurized pipes. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the water or aqueous liquid.

2.0 Method Summary

2.1 Instructions for establishing sample points are presented. Grab Sampling or automatic sampling procedures are employed to obtain samples.

2.2 Sampling containers are filled as specified by parameter. A sufficient amount of sample will be collected and placed into a suitable shipping container if more than on-site analysis is required.

INORGANIC LIQUID SAMPLES FROM PRESSURIZED PIPES (PROCEDURE K.4.8.2)

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1.0 Scope and Application

This method is applicable to the collection of inorganic liquid samples from pressurized pipes. The samples are collected to characterize radiological, organic, or inorganic contaminants present in the inorganic liquid.

2.0 Method Summary

2.1 Instructions for establishing sample points are presented. Grab sampling or automatic sampling methods are employed to obtain samples.

2.2 Sample containers are filled as specified by parameter. A sufficient amount of sample is collected and placed in an appropriate shipping package if off-site analysis is required.

ORGANIC LIQUIDS FROM PRESSURIZED PIPES (PROCEDURE K.4.8.4)

1.0 Scope and Application

This procedure is applicable to the collection of organic liquids from pressurized pipes. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the organic liquid.

2.0 Method Summary

2.1 Instructions for establishing sample points are presented. Grab sampling or automatic sampling procedures are employed to obtain samples.

2.2 Sampling containers are filled as specified by parameter. A sufficient amount of sample is collected and placed into a suitable shipping container if more than on-site analysis is required.

WATER AND AQUEOUS LIQUIDS FROM UNPRESSURIZED PIPES (PROCEDURE K.4.9.1)

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1.0 Scope and Application

This procedure is applicable to the collection of water and/or aqueous liquids from unpressurized pipes. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the water or aqueous liquid.

2.0 Method Summary

2.1 Instructions for establishing sample points are presented. Grab Sampling or automatic sampling procedures are employed to obtain samples.

2.2 Sampling containers are filled as specified by parameter. A sufficient amount of sample is collected and placed into a suitable shipping container if more than on-site analysis is required.

INORGANIC LIQUID SAMPLES FROM UNPRESSURIZED PIPES (PROCEDURE K.4.9.2)

1.0 Scope and Application

This method is applicable to the collection of inorganic liquid samples from unpressurized pipes. The samples are collected to characterize radiological, organic, or contaminants present in the inorganic liquid.

2.0 Method Summary

2.1 Instructions for establishing sample points are presented. Grab sampling or automatic sampling methods are employed to obtain samples.

2.2 Sample containers are filled as specified by parameter. A sufficient amount of sample is collected and placed in an appropriate shipping package if off-site analysis is required.

INORGANIC LIQUIDS FROM DUCTS (PROCEDURE K.4.9.3)

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1.0 Scope and Application

This method is applicable to the collection of inorganic liquids from ducts. The samples are collected to characterize radiological, organic, and inorganic contaminants.

2.0 Method Summary

2.1 Instructions for establishing sample points are presented, preferably utilizing existing duct accesses. Grab sampling methods are employed to obtain samples.

2.2 Sample containers are filled as specified by parameter. A sufficient amount of sample is collected and placed in an appropriate shipping package if off-site analysis is required.

ORGANIC LIQUIDS FROM UNPRESSURIZED PIPES (PROCEDURE K.4.9.4)

1.0 Scope and Application

This procedure is applicable to the collection of organic liquids from unpressurized pipes. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the organic liquid.

2.0 Method Summary

2.1 Instructions for establishing sample points are presented. Grab sampling or automatic sampling procedures are employed to obtain samples.

2.2 Sampling containers are filled as specified by parameter. A sufficient amount of sample is collected and placed into a suitable shipping container if more than on-site analysis is required.

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COLLECTION OF ORGANIC, INORGANIC AND AQUEOUS LIQUID GRABBED SAMPLES FROM PIPING, UTILITIES, AND EQUIPMENT (PROCEDURE K.4.9.5)

1.0 Scope and Application

This procedure describes the techniques and defines the sampling equipment that is the most appropriate for collecting liquid grab samples from piping, utilities, and equipment.

2.0 Method Summary

2.1 Grab samples are used to characterize the sampling media at a particular instance in time or process event. Safe access to the media is a part of the sampling program and is very instrumental in the determination of the sampling equipment being used. Mechanisms may be needed to help compensate for a sample to be representative of the entire waste stream or process effluent.

2.2 For piping samples, the chemical and physical nature of the material and the amount of pressure to be expected are determined. The condition of the tap or valve is checked and isolated from any potential pressure or reactive source. Sample is collected into a compatible, secondary container and then transferred to a sample bottle unless analyzing for any volatile organic compounds.

2.3 Sampling points from utilities can vary from weirs, flumes and manholes to open channels and treatment facilities. Measurements should be taken in order to document the volume and/or the conditions at the time of the sample collection. Any adverse conditions of the media or sample point are noted which may have a bearing on the analytical outcome.

2.4 Sampling from equipment requires that an operator or other personnel are familiar with the equipment that can assure the sampling team that the equipment has been immobilized and stable for the work to be conducted. Appropriate tools are used to gain access to the sample point(s) and any electrical sources are to have appropriate lockout/tagout procedure completed.

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COLLECTION OF ORGANIC, INORGANIC AND AQUEOUS LIQUID TIMED SAMPLES FROM PIPING, UTILITIES, AND EQUIPMENT (PROCEDURE K.4.9.6)

1.0 Scope and Application

This procedure discusses the sampling procedures, applications, and equipment necessary for the collection of composite liquid samples over timed intervals from piping, utilities and equipment.

2.0 Method Summary

2.1 Timed composite samples are comprised of a varying number of discrete samples collected at equally timed intervals during a sampling event or program. It is necessary to have some specialized equipment for measuring volumes, flow and time.

2.2 Samplers equipped with peristaltic pumps are programmed to collect specified volume aliquots over a predetermined period. Auto samplers require DC power source or AC with a converter. The microprocessor is programmed to show the following:

- Volumetric determination
- Sequential time
- Composite time
- Suction line size
- Suction line length
- Suction head

2.3 The pump is fitted with inert silicone or natural rubber pump tubing. The appropriate intake strainer is installed on the suction line and is placed in the waste stream, process line or piping tap.

2.4 Automated samplers may used be in a manual mode or grab samples can be collected and combined to form composite samples using conventional collection techniques. Individual aliquots are collected using stainless steel, teflon, or other inert material devices. Simple devices such as bailers, buckets and zone sampling devices are most desirable due to ease in decontamination to prevent cross-contamination.

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COLLECTION OF ORGANIC, INORGANIC AND AQUEOUS LIQUID PROPORTIONAL SAMPLES FROM PIPING, UTILITIES, AND EQUIPMENT (PROCEDURE K.4.9.7)

1.0 Scope and Application

This procedure describes the sampling and measuring procedures for collecting proportional composite samples of organic, inorganic, and aqueous liquids from piping, utilities, and equipment.

2.0 Method Summary

2.1 Proportional composite samples are collected either by flow proportion or by process proportion during the compositing period. The periods are usually by time-varying/constant volume or time-constant/varying volume. Constant volume procedures are typical with automatic sampling devices while varying volumes involve manual methods.

2.2 Samplers equipped with peristaltic pumps are programmed to collect calibrated volume aliquots at a point determined by either a flow or volume and composited into a common receiver. The microprocessor is programmed to show the directions in the following order:

- Volumetric determination
- Proportional flow
- Composite time
- Suction line size
- Suction line length
- Suction head (ft. elev.)

2.3 The sampler must also be supported by an analog device capable of sending information concerning volumes of material passing through the sample point. Flow meters may by a Doppler type device or a manual meter installed in a controlled weir, flume, or other v-notch apparatus used to measure the volume of material being sampled.

2.4 Manual proportional sampling is usually done only if the process is routinely stopped at given points or if the data does not require extreme accuracy. Collection devices are typically simple such as buckets or dippers; however, any documentation concerning the sampling activity should by of greater detail in order to reproduce the sampling event.

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WATER AND AQUEOUS LIQUID SAMPLES FROM DUCTS (PROCEDURE K.4.9.8)

1.0 Scope and Application

This method is applicable to the collection of water and/or aqueous liquid samples from ducts. The samples are collected to characterize radiological, organic, or inorganic contaminants present in the water or aqueous liquid.

2.0 Method Summary

2.1 Instructions for establishing sample points are presented, preferably utilizing existing duct accesses. Grab sampling methods are employed to obtain samples.

2.2 Sample containers are filled as specified by parameter. A sufficient amount of sample is collected and placed in an appropriate shipping package if off-site analysis is required.

ORGANIC LIQUIDS FROM DUCTS (PROCEDURE K.4.9.9)

1.0 Scope and Application

This method is applicable to the collection of organic liquids from ducts. The samples are collected to characterize radiological, organic, or inorganic contaminants.

2.0 Method Summary

2.1 Instructions for establishing sample points are presented, preferably utilizing existing duct accesses. Grab sampling methods are employed to obtain samples.

2.2 Sample containers are filled as specified by parameter. A sufficient amount of sample is collected and placed in an appropriate shipping package if off-site analysis is required.

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SURFACE SOIL SAMPLING (PROCEDURE K.5.1)

1.0 Scope and Application

This method is applicable to the collection of surface soil samples from bulk materials and basins. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the bulk materials and basins.

2.0 Method Summary

2.1 Decontaminated sampling equipment is used to collect samples, preferably at a designated staging area. Area to be sampled is free from grass and other vegetation. Samples are collected with a trowel, scoop or coring device relative to material being sampled and to parameters of interest and placed in specified containers.

2.2 Composite samples are divided into relatively even quarters, mixed by opposite quarters together and then mixed with resulting halves together. Procedure is repeated and the sample is then placed in appropriate containers.

SALTS AND POWDERS FROM UNPRESSURIZED PIPES AND DUCTS (PROCEDURE K.5.1.1)

1.0 Scope and Application

This procedure is applicable to the collection of salts and powders from unpressurized pipes or ducts. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the salts or powders.

2.0 Method Summary

2.1 Samples are collected using a scoop, spatula, or scapula and transferred to clean sample containers of the appropriate size. The quantity of sample collected will be determined by the test parameters specified.

2.2 Samples will be collected manually with protective apparel being determined by the potential hazards of the pipe or duct location and contents.

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SLUDGE SAMPLES FROM UNPRESSURIZED PIPES (PROCEDURE K.5.1.3)

1.0 Scope and Application

This procedure is applicable to the collection of sludge from unpressurized pipes. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the sludge.

2.0 Method Summary

2.1 Samples are collected using a scoop, spatula, or scapula and transferred to clean sample containers of the appropriate size. The quantity of sample collected will be determined by the test parameters specified.

2.2 Samples will be collected manually with protective apparel being determined by the potential hazards of the pipe location and contents.

SLUDGE SAMPLES FROM DUCTS (PROCEDURE K.5.1.4)

1.0 Scope and Application

This procedure is applicable to the collection of sludge from ducts. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the sludge.

2.0 Method Summary

2.1 Samples are collected using a scoop, spatula, or scapula and transferred to clean sample containers of the appropriate size. The quantity of sample collected will be determined by the test parameters specified.

2.2 Samples will be collected manually with protective apparel being determined by the potential hazards of the duct location and contents.

SLUDGE SAMPLES FROM PROCESS EQUIPMENT (PROCEDURE K.5.1.5)

1.0 Scope and Application

This procedure is applicable to the collection of sludge from Process Equipment. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the sludge.

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2.0 Method Summary

2.1 Samples are collected using a scoop, spatula, or scapula and transferred to clean sample containers of the appropriate size. The quantity of sample collected will be determined by the test parameters specified.

2.2 Samples will be collected manually with protective apparel being determined by the potential hazards of the location and contents of the process equipment.

SEDIMENT SAMPLING (PROCEDURE K.5.2)

1.0 Scope and Application

This method is applicable to the collection of sediment samples from bulk materials and basins. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the sediment. Parameter specific requirements are identified.

2.0 Method Summary

2.1 Decontaminated sampling equipment is used to collect samples. A sufficient amount of sample is collected and transferred to a stainless-steel pan for mixing prior to placing sample in a container. Samples requiring refrigeration are stored in an ice chest in the field.

2.2 Composite samples are divided in approximately even quarters, mixed by opposite quarters together and then mixed with resulting halves together. The procedure is repeated and sample is then placed in appropriate containers.

COLLECTION OF UNCONSOLIDATED SUBSURFACE SAMPLES (PROCEDURE K.5.3.1)

1.0 Scope and Application

This method describes the procedures for collecting unconsolidated subsurface samples from bulk materials at depth. The samples are collected to characterize radiological, organic and inorganic contaminants present in the unconsolidated subsurface. Parameter specific sampling procedures are described.

2.0 Method Summary

2.1 Decontaminated sampling equipment is used to collect undisturbed samples.

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Equipment is selected for a drilling method based on project objectives and specified requirements. Samples are collected at specified intervals using a replaceable Shelby tube sampler. Samples are left in tube and ends are sealed for analysis at an approved laboratory. Split-tube or split-spoon samplers may be used for sample collection when undisturbed samples are not required.

2.2 Split spoon samples are pushed or driven down the borehole and is removed and screened for VOCs and radionuclids using appropriate devices. Sample is described and removed from sampler and transferred to appropriate container for shipment to the laboratory.

QA/QC SAMPLES FOR SOIL AND SEDIMENT ANALYSES (PROCEDURE K.5.4)

1.0 Scope and Application

This method describes the basic requirements for QA/QC samples for soil and sediment analysis.

2.0 Method Summary

2.1 Trip blanks are prepared in a controlled environment and accompany each shipping container of soil and sediment samples analyzed for VOCs unless specifically omitted in PSPs. De-ionized water is poured into the sample container specified for the parameter of interest.

2.2 Rinsate samples are decontamination samples that are obtained by pouring organic-free, de-ionized water over the equipment in the container specified for the parameter of interest.

2.3 Duplicate and split samples are collected in the same manner as actual samples with the exception that samples are composited and distributed evenly between containers while filling. Samples are not composited for VOC analysis.

2.4 Container blanks are empty containers that are submitted to the laboratory to check for constituents of concern.

2.5 Matrix spike/matrix spike duplicates are collected at triple sample volume every twentieth sample or every sampling round, whichever is more frequent, and are handled in the same manner as other samples.

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DRUM SAMPLING (PROCEDURE K.5.5)

1.0 Scope and Application

This method describes the collection of samples from drums, specifying sampling, analytical and regulatory requirements.

2.0 Method Summary

Specific drum sampling equipment is required depending on the density or viscosity and chemical nature of the drum's contents. Drum samples are collected from the bottom to the top and boring locations on the top of the surface are random. Solid samples are collected using either a grain sampler or pipe sampler. Drums containing liquid waste are sampled using Composite Liquid Waste Samplers. Samples are placed in appropriate containers for transportation to a laboratory. Specific sampling procedures for VOC analysis are identified.

SOLID SAMPLES FROM TANKS (PROCEDURE K.5.5.7)

1.0 Scope and Application

This procedure describes the collection of bulk or solid samples from tanks or tank-like vessels.

2.0 Method Summary

Sampling solids in tanks requires specialized techniques and equipment or equipment combinations. The primary methods described in this procedure are selected based on the consistency of the matrix, logistics or accessibility to the tank and the analytical objectives. The sampling devices may be operated in principle by driving, augering, vibrating or by using combined techniques. Driving a tube or inserting a collection device directly into the media is the simplest and sometimes the most desirable method, but the resulting sample may not be representative of the contents of the tank. This method may alter the physical characteristics of the sample by compressing it. Augering may be desirable in materials that have a soil-like consistency that allow several depths of a sample to be collected without cave-in of the wall of the sample hole. Techniques may necessitate the use of several sizes of augers if multiple levels are to be taken. Special devices are used to reduce sample compaction by vibrating the sample tube through the matrix to the desired level. These devices are more expensive than most of the other devices; however, under certain conditions, vibration coring equipment may be the only alternative that preserves the integrity of the sample.

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SOLID SAMPLES FROM DRUMS (PROCEDURE K.5.5.8)

1.0 Scope and Application

This procedure describes the collection of solids and/or powders from drums.

2.0 Method Summary

Procedures are described for sampling solids and/or powders from drums that are representative of the contents of the entire drum. Thieving tubes and triers are very practical and desirable if the consistency supports the use of these devices. Most drums containing solids, powders and thick sludges are open head types and allow easy access. Particular safety procedures must be followed during the opening of the drums and lid removal. The sampling devices should be constructed of stainless steel or other specified material that is compatible with the sampling media and the analytical criterion. By using a laboratory spatula, scoop, or trowel, a grab sample can be collected from the top of the drum if the contents are known to be homogeneous. The most appropriate method for drum sampling is one that is compatible to the matrix and analytical methods being utilized. The sampling devices may be operated in principle by driving, augering, vibrating or by using combined techniques. Driving a tube or inserting a collection device directly into the media is the simplest and sometimes the most desirable method, but the resulting sample may not be representative of the contents of the drum. This method may alter the physical characteristics of the sample by compressing it. Vibrating devices may be used to reduce sample compaction by vibrating the sample tube through the matrix to the desired level. These devices are more expensive than most of the other devices; however, under certain conditions, vibration type devices may be the only alternative that preserves the integrity of the sample. When sampling powders, other methods may be necessary to keep the sample within the sampling tube.

SAMPLING WATER FROM TANKS (PROCEDURE K.5.6.1)

1.0 Scope and Application

This procedure is applicable to the collection of water samples in tanks [if high concentrations of inorganic solutes (salts, acids, bases) are anticipated, procedure K.5.6.2 should be used instead of K.5.6.1]. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the liquid.

2.0 Method Summary

2.1 Instructions for accessing tank contents are presented. Grab sampling or automatic sampling procedures are employed to obtain samples. The sampling

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equipment will be decontaminated using procedure K.11 prior to use.

2.2 Sample containers are filled as specified by parameter. A sufficient amount of sample is collected and placed into a shipping container if more than on-site analysis is required.

SAMPLING INORGANIC LIQUID FROM TANKS (PROCEDURE K.5.6.2)

1.0 Scope and Application

This procedure is applicable to the collection of water samples in tanks in which high concentrations of inorganic solutes (salts, acids, bases) are anticipated. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the liquid.

2.0 Method Summary

2.1 Instructions for accessing tanks thought to contain high concentrations of inorganic solutes are presented. Grab sampling or automatic sampling procedures suitable for potentially corrosive environments are employed to obtain samples. The sampling equipment will be decontaminated using procedure K.11 prior to and after use.

2.2 Sample containers are filled as specified by parameter. A sufficient amount of sample is collected and placed into a shipping container if more than on-site analysis is required.

SAMPLING ORGANIC LIQUIDS FROM TANKS (PROCEDURE K.5.6.4)

1.0 Scope and Application

This procedure is applicable to the collection of organic liquids contained in tanks, either in bulk or in discrete layers overlying aqueous layers. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the liquid.

2.0 Method Summary

2.1 The sampling equipment will be decontaminated using procedure K.11 prior to use. Bulk sampling or skimming procedures are employed to obtain a discrete organic phase sample.

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2.2 Sample containers are filled as specified by parameter. A sufficient amount of sample is collected and placed into a shipping container. Samples requiring refrigeration are stored and shipped in an ice chest.

RADON SAMPLING (PROCEDURE K.6.2)

1.0 Scope and Application

This method is applicable to radon gas sampling in buildings. Measurements of radon flux density using a passive charcoal collector are described.

2.0 Method Summary

Obtaining representative radon flux measurements of long term average emissions requires that measurements are not collected within 24 hours after a rainfall, when ambient temperatures are below two degrees centigrade or when the ground is frozen. A charcoal collector is specifically prepared and placed on surface for measurement. After the collector is exposed for 24 hours, it is removed and placed in an approved container. The sample is prepared and sent to laboratory as soon as possible to minimize loss of measurable activity.

GENERAL AREA AIR SAMPLES (PROCEDURE K.6.3)

1.0 Scope and Summary

This method describes the procedures to measure levels of airborne radioactive material applicable to buildings to properly characterize areas in accordance with DOE Orders and determine respiratory protection requirements.

2.0 Method Summary

The amount of activity is determined prior to sample collection. Flow rate and start time of sample collection are recorded. After samples are collected, the end time is recorded. The filter media is counted. Continuous air monitors equipped with strip charts produce useable CERCLA data for tracking ambient airborne levels of radioactive contaminants.

PHOTO-IONIZATION DETECTOR (PROCEDURE K.6.4.1)

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1.0 Scope and Application

This method describes the procedure for the use of the photo-ionization detector for monitoring the work zone for organic and certain inorganic vapors and for screening samples for organic analysis.

2.0 Method Summary

Instrument is calibrated with a known standard on a daily basis. The response of the instrument to an organic vapor source is verified prior to each use. The instrument is zeroed by operation in a clean area. Background readings are measured at the site prior to the start of work.

FILTER PUMPS (PROCEDURE K.6.4.5)

1.0 Scope and Application

This method is applicable to the collection of air particulate samples. Pump filters are quantified for asbestos analysis or particles of interest.

2.0 Method Summary

The filter manufacturer, filter size, installation and removal time, beginning and ending flow rate and length of pump operation are recorded when a filter pump is used.

WIPE SAMPLING (PROCEDURE K.7.1)

1.0 Scope and Application

This procedure is applicable to the collection of wipe samples to determine contamination levels on hard, smooth surfaces such as floors, walls, metal, and glass. The samples are collected to characterize radiological, organic, and inorganic contaminants present on the surface.

2.0 Method Summary

2.1 Wipe samples are an effective means for collecting a specimen of ambient contaminants deposited or settled out on surfaces as a result of an intermittent

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or continuous release. Samples will be taken over a specified area of the surface. Contamination will be expressed as activity or mass per unit area.

2.2 Wipe samples will be taken on a sampling medium (the wipe) appropriate for each analyte as tabulated in this procedure. Once the wipe has been used, it is placed in the appropriate shipping container, or if appropriate, analyzed promptly on site.

INSTRUMENT SURVEYS OF EQUIPMENT - CONCRETE, METAL, GLASS, FABRIC/FIBERS (PROCEDURE K.7.2)

1.0 Scope and Application

This method is applicable to the performance of direct instrument surveys on equipment to verify contamination/radiation levels. Parameter specific requirements are identified.

2.0 Method Summary

2.1 Calibrated hand-held contamination survey instruments are utilized to determine total contamination levels (alpha and/or beta-gamma) on equipment. Radiation survey instruments are also used to determine radiation fields in the evaluation areas.

2.2 Results are documented on a standard survey logsheet. Total contamination levels may be compared to removable contamination levels to distinguish between "removable" and "fixed" contamination.

PAINT CHIP SAMPLING (PROCEDURE K.8.1)

1.0 Scope and Application

This method describes the procedures for collecting paint chip samples. The samples, which are non-biased, are collected at sample locations specified in PSPs. Collection processes are similar for each ASL.

2.0 Method Summary

Clean plastic sheeting is placed on the ground, concrete or floor surface within a sampling area. A decontaminated putty knife or paint scraper is used to remove loose paint material from a host surface. Paint chip samples are placed in a stainless-steel pan or tray and transferred to an appropriate sample container.

WOOD SAMPLING (PROCEDURE K.8.2)

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1.0 Scope and Application

This method is applicable to procedures for collecting wood samples. The samples are non-biased and are collected at sample locations specified in PSPs. Collection process are similar for each ASL.

2.0 Method Summary

Clean plastic sheeting is placed on a surface within a sample area. A rotary drill with a decontaminated wood bit is used to obtain the wood cuttings. The cuttings are placed in a clean stainless-steel tray or pan and transferred to an appropriate sample container.

CONCRETE SAMPLING (PROCEDURE K.8.3)

1.0 Scope and Application

This method describes the procedures for the collection of concrete samples. Samples are non-biased and collection processes are similar for each ASL. Sample locations are described in PSPs.

2.0 Method Summary

Clean plastic sheeting is placed on the ground, concrete, or floor surface within a sampling area. A rotary drill equipped with a decontaminated coring bit is used to collect the concrete cuttings. The cuttings are placed in a stainless-steel pan or tray. The cuttings are transferred to an appropriate sample container.

ASPHALT CORE SAMPLING (PROCEDURE K.8.3.1)

1.0 Scope and Application

This procedure is applicable to the collection of core samples to determine contamination levels in asphalt from buildings, pads, roads, and parking lots. The samples are non-biased and are collected at locations specified in the PSPs. The samples are collected to characterize radiological, organic, and inorganic contaminants that may be present within (at depth) the solid material.

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2.0 Method Summary

2.1 The sampling equipment including the drill and coring bit, stainless scoops, spoons, and pans or trays will be decontaminated using procedure number K.11 prior to use. The core samples will normally be collected using a Milwaukee or equivalent rotary drill with a coring bit. Cuttings and core are transferred to stainless pans and then placed in the appropriate shipping container. Sample containers are filled as specified by parameter.

2.2 In order to completely characterize the core, slices will be removed from the core at designated intervals to determine the vertical and horizontal contaminant distribution.

TRANSITE CORE SAMPLING (PROCEDURE K.8.3.2)

1.0 Scope and Application

This procedure is applicable to the collection of core samples to determine contamination levels in Transite. The samples are collected to characterize radiological, organic, and inorganic contaminants that may be present within (at depth) the solid material. The samples are non-biased and are collected at locations specified in the PSPs.

2.0 Method Summary

2.1 Sampling crews will be adequately protected against asbestos inhalation and other potential hazards in accordance with the Health and Safety Plan. The sampling equipment including the drill and coring bit, stainless scoops, spoons, and pans or trays will be decontaminated using procedure number K.11 prior to use. Sample containers are filled as specified by parameter. The core samples will normally be collected using a Milwaukee or equivalent rotary drill with a coring bit. Cuttings and core are transferred to stainless pans and then placed in the appropriate shipping container.

2.2 In order to completely characterize the core, slices will be removed from the core at designated intervals to determine the vertical and horizontal contaminant distribution.

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DRYWALL CHUCK SAMPLING (PROCEDURE K.8.3.3)

1.0 Scope and Application

This procedure is applicable to the collection of chip or chunk samples to determine contamination levels on drywall. The samples are collected to characterize radiological, organic, and inorganic contaminants present within the solid material.

2.0 Method Summary

2.1 The sampling equipment will be decontaminated using procedure K.11 prior to use. The chip, chunk, or piece samples will be collected by a variety of ways including scrapers, putty knives, small drill bits, hammers, and chisels. Pieces of samples material will be collected in stainless steel pans or trays and sufficient quantities will be transferred to the appropriate sample/shipping container. Sample containers will be filled as specified per parameter.

2.2 In order to completely characterize the sampled material, a piece or pieces will be collected that represent the entire cross section. The pieces will then be ground up at the laboratory for complete analyses.

STRUCTURAL STEEL CORES FROM BUILDINGS, PADS, AND RAILROADS (PROCEDURE K.8.3.4)

1.0 Scope and Application

This method is applicable to the collection of steel samples from structures. Parameter specific requirements are identified.

2.0 Method Summary

2.1 Decontaminated sampling equipment is used to collect samples. Representative and sufficient samples are taken from the structure to accommodate the intended analytical procedures

2.2 Samples are to be collected utilizing a rotary drill and bit or a reciprocating saw or other devices such as a portable band saw, metal planer, or a coring saw relative to the configuration of the item being sampled and to the parameters of interest. Shavings, turnings, and filings are collected and placed in appropriate containers as required per analyte.

MASONRY CORE SAMPLING (PROCEDURE K.8.3.5)

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1.0 Scope and Application

This procedure is applicable to the collection of core samples to determine contamination levels in masonry, particularly masonry composed of porous or semi-porous material. The samples are non-biased and are collected at locations specified in the PSPs. The samples are collected to characterize radiological, organic, and inorganic contaminants that may be present within (at depth) the stone and/or mortar.

2.0 Method Summary

2.1 The sampling equipment will be decontaminated using procedure K.11 prior to use. The core samples will normally be collected using a Milwaukee or equivalent rotary drill with a coring bit. Cuttings and core are transferred to stainless pans and then placed in the appropriate shipping container. Sample containers are filled as specified by parameter.

2.2 In order to completely characterize the core, slices will be removed from the core at designated intervals to determine the vertical and horizontal contaminant distribution.

FABRIC AND FIBER SAMPLING (PROCEDURE K.8.3.8)

1.0 Scope and Application

This procedure is applicable to the collection of fabric and fiber samples to determine contamination levels entrained in the material. The samples are collected to characterize radiological, organic, and inorganic contaminants.

2.0 Method Summary

The sampling equipment will be decontaminated using procedure K.11 prior to use. Stainless steel shears and tweezers will be used to detach representative portions of the fiber or fabric. Pieces of samples material will be collected in stainless steel pans or trays and sufficient quantities will be transferred to the appropriate sample/shipping container. Sample containers will be filled as specified per parameter.

DUST COLLECTOR RESIDUE SAMPLING (PROCEDURE K.8.4)

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1.0 Scope and Application

This method describes the procedures for the collection of dust collector residue samples. The samples are non-biased and are collected at sample locations specified in PSPs. Collection processes are similar for each ASL.

2.0 Method Summary

Clean plastic sheeting is placed on a surface within the sampling area. A decontaminated stainless-steel scoop is used to collect the samples. The dust residue is transferred to an appropriate sample container.

DEBRIS SAMPLING: SALT, POWDER, DUST, AND HETEROGENEOUS WASTE (PROCEDURE K.8.4.1)

1.0 Scope and Application

This procedure is applicable to the collection of debris samples to determine contamination levels in loose, solid materials such as residues, powders, dusts, and salts. The samples are collected to characterize radiological, organic, and inorganic contaminants present in the debris.

2.0 Method Summary

2.1 The sampling equipment will be decontaminated prior to use. The sample normally will be collected by using a stainless steel scoop although other devices may be required depending upon the total volume of the debris. The bulk sample will be transferred to an appropriate sample container for shipment.

2.2 Grab sampling is expected to be the standard technique for sampling debris. Composite sampling of debris is expected to be infrequent.

WASTE PILE SAMPLING - ASPHALT (PROCEDURE K.8.5.1)

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1.0 Scope and Application

This method is applicable to the collection of asphalt samples from waste piles. Parameter specific requirements are identified.

2.0 Method Summary

2.1 Decontaminated sampling equipment (K.11) is used to collect samples. Representative and sufficient samples are taken from the waste pile and a sketch of the sample location is documented.

2.2 Samples are to be collected utilizing a scoop or trowel. Compacted asphalt may require the use of a hammer and chisel to obtain sufficient sample quantities. Samples are placed in container(s) appropriate for analyte(s).

WASTE PILE SAMPLING - TRANSITE (PROCEDURE K.8.5.2)

1.0 Scope and Application

This method is applicable to the collection of transite samples from waste piles. Parameter specific requirements are identified.

2.0 Method Summary

2.1 Decontaminated sampling equipment (K.11) is used to collect samples. Representative and sufficient samples are taken from the waste pile and a sketch of the sample location is documented.

2.2 Samples are to be collected utilizing a hand or electric saw. Precautions are taken for working with asbestos containing materials (ACM). Samples are placed in an appropriate container.

WASTE PILE SAMPLING - DRYWALL (PROCEDURE K.8.5.3)

1.0 Scope and Application

This method is applicable to the collection of drywall samples from waste piles. Parameter specific requirements are identified.

2.0 Method Summary 3298

2.1 Decontaminated sampling equipment is used to collect samples. Representative and sufficient samples are taken from the waste pile and a sketch of the sample location is documented.

2.2 Samples are to be collected utilizing a hand saw (drywall saw), a scoop or trowel if the drywall is crumbled, or other device relative to the parameters of interest. Samples are placed in container(s) appropriate for analyte(s).

WASTE PILE SAMPLING - METAL (PROCEDURE K.8.5.4)

1.0 Scope and Application

This method is applicable to the collection of metal samples waste piles. Parameter specific requirements are identified.

2.0 Method Summary

2.1 Decontaminated sampling equipment is used to collect samples. Representative and sufficient samples are taken from the waste pile and a sketch of the sample location is documented.

2.2 Samples are to be collected utilizing a reciprocating saw or other devices such as a portable band saw, metal planer, or a coring saw relative to the metal being sampled and to the parameters of interest. Samples are placed in appropriate containers as required per analyte.

WASTE PILE SAMPLING - GLASS (PROCEDURE K.8.5.5)

1.0 Scope and Application

This method is applicable to the collection of glass samples from waste piles. Parameter specific requirements are identified.

2.0 Method Summary

2.1 Decontaminated sampling equipment is used to collect samples. Representative and sufficient samples are taken from the waste pile and a sketch of the sample location is documented.

2.2 Samples are to be collected utilizing a scoop or trowel. Glass may be size reduced utilizing an impact method to obtain appropriate sample size. Samples are placed in container(s) appropriate for analyte(s).

WASTE PILE SAMPLING - MASONRY (PROCEDURE K.8.5.6)

1.0 Scope and Application

This procedure is applicable to the collection of samples to determine contamination levels in discarded pieces of masonry, particularly masonry composed of porous or semi-porous material.

2.0 Method Summary

2.1 Decontaminated sampling equipment (K.11) is used to collect samples. Representative and sufficient samples are taken from the waste pile and a sketch of the sample location is documented.

2.2 The sampling equipment will be decontaminated using procedure K.11 prior to use. Samples from larger pieces will normally be collected using a Milwaukee or equivalent rotary drill with a coring bit. Samples from smaller pieces will be obtained with a geologist's hammer or other device as provided. Cuttings and core samples are transferred to stainless pans and then placed in the appropriate shipping container. Sample containers are filled as specified by parameter.

WASTE PILE SAMPLING - FABRIC/FIBERS (PROCEDURE K.8.5.8)

1.0 Scope and Application

This method is applicable to the collection of fabric/fiber samples (e.g., fiberglass, process filter media, discarded cloth) from waste piles. Parameter specific requirements are identified.

2.0 Method Summary

2.1 Decontaminated sampling equipment is used to collect samples. Representative and sufficient samples are taken from the waste pile and a sketch of the sample locations is documented.

2.2 Samples are to be collected utilizing shears or a knife for the intact materials. A scoop or trowel should be used for disintegrated materials. Samples are placed in container(s) appropriate for analyte(s).

SOLID SAMPLES FROM BOXES, OVERPACKS, ROLL-OFFS (PROCEDURE K.8.6.1)

1.0 Scope and Application

This procedure establishes the guidelines for collecting solid samples from boxes, overpacks, roll-offs and other containers which may be used for material consolidation or short term storage.

2.0 Method Summary

Gaining access to the stored material is a consideration in the selection of appropriate sampling equipment. Since the containers are usually large and not designed for sampling access, precautions should be taken to prevent tip-over and falling from ladders, platforms or lifts. Materials that are not homogeneous or are loaded into the containers at different occasions must be systematically sampled for adequate representation. A core can be collected from top to bottom if the consistency of the material allows. Grab samples can be obtained and composited if materials are loaded in lifts or by event. A combination of sampling devices may be required if some settling or stratification has taken place. Casing the sample point can be accomplished by using pipe, if layer inter-mingling needs to be avoided. Containers that do not appear secure or are susceptible to leaking when disturbed should be wrapped, bermed with an area capable of containing all of the material, or overpacked prior to sampling. Large containers should not be moved for sample characterization unless they are specifically designed for transporting the contained material.

FABRIC AND FIBER SAMPLES FROM BOXES, OVERPACKS, ROLL-OFFS (PROCEDURE K.8.6.8)

1.0 Scope and Application

This procedure establishes the guidelines for collecting solid samples of fabric and fiber from boxes, overpacks, roll-offs and other containers.

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2.0 Method Summary

Provisions are made to maintain adequate control over any materials that may become airborne when sampling fabric, especially fibers. Sampling for the actual fibers instead of contamination on the fibers requires specialized equipment such as air pumps, HEPA (high efficiency particulate air) filters, and containers for specific fibers. Fabric samples may be cut from the original pieces using cutting devices which have been appropriately decontaminated. If the fabrics are composite materials, it may be necessary to separate the materials under controlled conditions away from the field. Since sharp cutting devices may be used, safety precautions and appropriate equipment should be designed into the program prior to sampling. Chain mail gloves or gauntlets should be worn by the sampling personnel. The roll-offs or other containers should not be entered by the sampling personnel without emergency egress equipment and standby safety support personnel. A grab sample can be obtained and composited if the contained materials were loaded in lifts or by event. The outside of each container should be monitored for temperature changes during activities that could potentially change the conditions inside the container. Fire suppression equipment should be readily available as layers of fabric are exposed to the air.

SAMPLE COLLECTION FORMS (PROCEDURE K.9)

1.0 Scope and Application

This method describes the information that is required to complete sample collection forms.

2.0 Method Summary

Sample collection forms are generated for each sample, and include specific information about the sample collected. The forms also contain a description of the sample containers, analytical parameters, preservation methods, sampling team members and types and identification numbers of equipment used.

FIELD STORAGE AND SHIPMENT OF SAMPLES (PROCEDURE K.10)

1.0 Scope and Application

This method identifies the procedures and requirements for the field storage and shipment of environmental, hazardous and radioactive samples.

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2.0 Method Summary

2.1 The field storage of environmental samples is handled in prescribed methods to ensure sample integrity. Samples are preserved according to specific constituent requirements and are shipped, maintaining chain-of-custody requirements.

2.2 The packing and transporting of hazardous waste samples are conducted according to prescribed federal regulations.

2.3 Radioactive samples are packaged, labeled and shipped according to applicable federal regulations that are dependent upon the quantity and specific activity of the sample.

DECONTAMINATION (PROCEDURE K.11)

1.0 Scope and Application

This method identifies the procedures for the decontamination of sampling equipment at specified central decontamination areas.

2.0 Method Summary

2.1 Cleaning materials for decontamination are based on U.S. EPA guidance documents. Variations from specified materials will be recorded on the daily field log and the samples potentially affected will be noted.

2.2 General equipment cleaning procedures are designated by three levels of decontamination, depending on constituent contamination and whether sampling equipment comes into contact with the sampled media. Specific decontamination procedures are followed for particular sampling equipment.

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D.I.4 LABORATORY ANALYTICAL PROCEDURES*

GENERAL LABORATORY REQUIREMENTS (PROCEDURE FM-GEN-0010)

1.0 Purpose

This procedure defines the format to be followed for each method and generally discusses data package deliverables and quality control sample requirements. In an integrated program to generate quality data, some activities can be classified as management related (quality assurance) and others as functional (quality control). The material herein is an overview of the Fernald Environmental Management Program (FEMP) program that laboratories generating analytical data must meet.

2.0 Method Format

The methods will typically contain 15 points. Sections that do not pertain to the specific method will be identified as "not applicable." Other sections may contain generic text common to all analytical methods, as discussed below.

2.1 Scope and Application

This section will consist of introductory comments explaining the method (e.g., specific target analytes) and the applicability of the method to specific sample matrixes. If there is more than one method for determining a specific target compound in the manual, then it will be necessary to define when the specific method should be used instead of another method or technique.

2.2 Method Summary

This section will contain a summary or outline of how the method is performed.

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*Includes analytical procedures for organic compounds, RCRA characteristics, metals, conventional parameters, radiometric, and geotechnical parameters.

VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/ MASS SPECTROMETRY (PROCEDURE FM-ORG-0010)

Working Linear Range: Analyte- and matrix-dependent
Reporting Limit: Analyte- and matrix-dependent
Matrix: Water, sediment, soil, and waste

1.0 Scope and Application

This analytical method is designed to analyze water, sediment, and soil. The method target compound lists and the corresponding practical quantitation limits are summarized in Tables 1 and 2.

2.0 Method Summary

2.1 Water Samples

2.1.1 A sample aliquot is diluted with water. An inert gas is bubbled through a 5-mL aliquot of the diluted sample contained in a specifically designed purging chamber at ambient temperature. Purgeables are efficiently transferred from aqueous phase to vapor phase. The vapor is swept through a sorbent column where purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables, which are then detected with a mass spectrometer.

2.2 Soil/Sediment Samples

2.2.1 Low Level: An inert gas is bubbled through a mixture of a 5-gram sample and reagent water contained in a specially designed purging chamber at elevated temperatures. Purgeables are efficiently transferred from aqueous phase to vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables, which are then detected with a mass spectrometer.

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Table 1
Target Compound List (TCL) and Practical Quantitation Limits

Parameter	Water (µg/L)	Low Soil (µg/kg)	Medium Soil (µg/kg)
Chloromethane	10	10	1200
Bromomethane	10	10	1200
Vinyl Chloride	10	10	1200
Chloroethane	10	10	1200
Methylene chloride	10	10	1200
Acetone	50	50	6000
Carbon disulfide	50	50	6000
1,1-Dichloroethene	10	10	1200
1,1-Dichloroethane	10	10	1200
1,2-Dichloroethene (total)	10	10	1200
Chloroform	10	10	1200
1,2-Dichloroethane	10	10	1200
1,1,2-Trichloroethane	10	10	1200
Benzene	10	10	1200
cis-1,3-Dichloropropene	10	10	1200
Bromoform	10	10	1200
2-Hexanone	50	50	6000
2-Butanone	50	50	6000
1,1,1-Trichloroethane	10	10	1200
Carbon tetrachloride	10	10	1200
Bromodichloromethane	10	10	1200
1,2-Dichloropropane	10	10	1200
trans-1,3-Dichloropropene	10	10	1200
Trichloroethene	10	10	1200
Dibromochloromethane	10	10	1200
4-Methyl-2-pentanone	50	50	6000
Tetrachloroethene	10	10	1200
Toluene	10	10	1200
Chlorobenzene	10	10	1200
Ethyl benzene	10	10	1200
Styrene	10	10	1200
Total Xylenes	10	10	1200

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Table 2
Supplemental Target Compound List (STL) and Practical Quantitation Limits

Parameter	Practical Quantitation Limits: Water (µg/L)	Low Soil (µg/kg)	Medium Soil (µg/kg)
Acetonitrile	200	200	24000
Acrolein	100	100	In review
Acrylonitrile	100	100	In review
Butanol	In review	In review	In review
2-Chloroethyl vinyl ether	10	10	1200
3-Chloropropene	In review	In review	In review
1,2-Dibromoethane	10	10	1200
1,3-Dibromo-3-chloropropane	In review	In review	In review
Dichlorodifluoromethane	20	20	2400
Dioxane	500	500	In review
Ethyl methacrylate	50	50	6000
Methacrylonitrile	50	50	6000
Methyl iodide	In review	In review	In review
Methyl methacrylate	50	50	6000
Methyl bromide	In review	In review	In review
1,1,1,2-Tetrachloroethane	In review	In review	In review
Trichlorofluoromethane	10	10	1200
1,2,3-Trichloropropane	10	10	1200

2.2.2 Medium Level: A measured amount of soil is extracted with methanol, and a portion of the extract is diluted to 5 mL with reagent water. An inert gas is bubbled through the solution in a specifically designed purging chamber at ambient temperature. The purgeables are effectively transferred from aqueous phase to vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb purgeables onto a GC column. The gas chromatograph is temperature programmed to separate the purgeables, which are then detected with a mass spectrometer.

PREPARATION OF ORGANIC MATERIALS FOR VOC ANALYSIS (PROCEDURE FM-ORG-0012)

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Working Linear Range:	Matrix and analyte dependent
Reporting Limit:	Matrix and analyte dependent
Reporting Units:	Parts per million (mass)
Matrix:	Wood, bulk solids with high organic content

1.0 Scope and Application

This is a method to prepare bulk organic samples with low expected yields for VOC analysis by either FM-ORG-0010 or FM-ORG-0050.

2.0 Method Summary

2.1 The sample is cooled to -20°C and then finely divided by mechanical grinding. The equipment used to grind the sample is held at -20°C to maintain sample temperature during the process.

2.2 The cooled sample is transferred directly to a purging chamber, water is added, and volatile organics are purged and collected under heat.

2.3 Analysis proceeds as described in FM-ORG-0010 or in FM-ORG-0050.

AROMATIC VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/ PHOTOIONIZATION DETECTOR (PROCEDURE FM-ORG-0020)

Working Linear Range: Matrix and analyte specific
Reporting Limit: Matrix dependent
Reporting Units: Water, µg/L; solids (dry weight), µg/kg
Matrix: Water, soil, sediment, or sludge

1.0 Scope and Application

The method is used to determine the concentration of various aromatic volatile organic compounds. Target compounds and the practical quantitation limits (PQLs) are summarized in Table 1.

Table 1
Practical Quantitation Limits (PQLs)[a] for Aromatic Volatile Organics

Compound	Practical Quantitation Limits Water (µg/L)	Practical Quantitation Limits Soil[b] (µg/kg)
Benzene	1	1
Chlorobenzene	1	1
1,4-Dichlorobenzene	5	5
1,3-Dichlorobenzene	5	5
1,2-Dichlorobenzene	5	5
Ethylbenzene	1	1
Toluene	1	1
Xylenes	5	5

[a]Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable.
[b]PQLs listed for soil/sediment samples are based on wet weight. Normally, data is reported on a dry weight basis; therefore, PQLs will be higher, based on the percent moisture in each sample.

2.0 Method Summary

2.1 The method describes chromatographic conditions for the detection of aromatic volatile compounds. Samples will be analyzed using a purge-and-trap method, although the method also covers direct injection. The gas chromatograph uses a temperature program to separate the organic compounds. Detection is achieved by a photoionization detector.

2.2 If interferences are encountered, the method specifies an optional gas chromatographic column that may be helpful in resolving the analytes from the interferences and for confirmation of analytes.

CHLORINATED PESTICIDES/PCBS BY GAS CHROMATOGRAPHY (PROCEDURE FM-ORG-0030)

Working Linear Range:	To be determined. Analyte- and matrix-dependent
Reporting Limits:	Required reporting limits are listed in Table 1
Reporting Units:	Liquids, µg/L; solids, µg/kg
Matrix:	Waters, soils, sludges, and wastes

1.0 Scope and Application

1.1 This method describes procedures to analyze water, soil, sludges, and wastes for chlorinated pesticides/PCBs. The method is applicable to water, soils, sludges, and wastes. The extraction procedure to be used must be appropriate for sample matrix.

1.2 Target compounds and the corresponding practical quantitation limits (PQLs) for this method are listed in Table 1.

2.0 Method Summary

A measured amount of sample is extracted with methylene chloride. Appropriate cleanup procedures are employed to remove or minimize interferences from the matrix. The extract is then dried and exchanged to hexane during concentration to a volume of 10 mL or less. The extract is separated by gas chromatography, and the parameters of interest are measured with an electron capture detector (ECD) or Hall electrolytic conductivity detector (HECD). Confirmation of tentatively identified parameters is required. This may be accomplished by using a second confirmation GC column or GC/MS techniques.

Table 1
Target Analytes and Required Reporting Limits

Compound	Water/Matrix (µg/L)	Soil/Sediment (µg/kg)
Aldrin	0.01	8.0
a-BHC	0.01	8.0
b-BHC	0.02	8.0
d-BHC	0.01	8.0
g-BHC	0.01	8.0
Chlordane	0.1	80
4,4′-DDD	0.02	16
4,4′-DDE	0.02	16
4,4′-DDT	0.02	16
Dieldrin	0.02	16
Endosulfan I	0.02	8.0
Endosulfan II	0.02	16
Endosulfan Sulfate	0.02	16
Endrin	0.02	16
Endrin Aldehyde	0.02	16
Endrin Ketone	0.02	16
Heptachlor	0.01	8.0
Heptachlor Epoxide	0.01	8.0
Methoxychlor	0.5	160
Toxaphene	0.5	160
PCB 1016	0.8	80
PCB 1221	2	80
PCB 1232	2	80
PCB 1242	0.8	80
PCB 1248	0.4	80
PCB 1254	0.2	160
PCB 1260	0.2	160

PREPARATION OF BULK MATERIALS FOR PCB/PESTICIDE ANALYSIS (PROCEDURE FM-ORG-0032)

Working Linear Range:	Matrix and analyte dependent
Reporting Limit:	Matrix and analyte dependent
Reporting Units:	Liquids, µg/L; solids, µg/kg
Matrix:	Bulk solids with high organic content, other solids

1.0 Scope and Application

1.1 This is a method to prepare bulk samples for Pesticide and PCB analysis by FM-ORG-0030.

1.2 The method covers the determination of organic compounds that may be partitioned by using methylene chloride and are amenable to analysis by GC/MS.

2.0 Method Summary

2.1 The sample is cooled to 0°C or below, then finely divided by mechanical grinding. The equipment used to grind the sample is held at 0°C to maintain sample temperature during the process.

2.2 A measured amount of sample is extracted with methylene chloride. Appropriate cleanup procedures are employed to remove or minimize interferences from the matrix. The extract is then dried and concentrated to a volume of 10 mL or less by using hexane.

2.3 Analysis proceeds as described in FM-ORG-0030.

ORGANOPHOSPHORUS PESTICIDES BY GAS CHROMATOGRAPHY (PROCEDURE FM-ORG-0040)

Working Linear Range: Analyte- and matrix-dependent
Reporting Limit: Analyte-dependent
Reporting Units: Water, µg/L; solids, µg/kg
Matrix: Water, soil, sediment and waste

1.0 Scope and Application

This method is applicable to water, wastewater, sediment, and soil samples. The method target compound list and practical quantitation limits (PQLs) are summarized in Table 1.

Table 1. Organophosphorus Pesticides Target Compound List and Practical Quantitation Limits (PQLs)[a]

Compounds (including alternate names)	PQL Water (µg/L)	PQL Soil/Sediment (µg/kg)[b]
Azinphos methyl (Guthion)	1	50
Bolstar (Sulprofos)	1	50
Chloropyrifos	1	50
Conumaphos (Co-Ral)	1	50
Demeton, O & S	2	100
Diazinon	1	50
Dichlorvos (DDVP)	1	50
Ethoprop	1	50
Fensulfothion (Daasanit)	2	100
Fenthion	1	50
Parathion, methyl	1	50
Phorate (Thimet)	1	50
Ronnel (Trolene)	1	50
Stirophos (Rabon)	10	500
Tokuthn (Prothiofos)	1	50
Trichloronate (Agritox)	1	50

[a] Sample PQLs are matrix-dependent.
[b] PQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis; therefore, PQLs will be higher, based on percent moisture in each sample.

2.0 Method Summary

A measured aliquot of a water sample at native pH is extracted with methylene chloride using the separatory funnel technique. A measured aliquot of soil/sediment is dried with sodium sulfate and extracted with 50:50 methylene chloride:acetone using sonication. The extract is dried and concentrated, and the solvent is exchanged into hexane.

2.1 Typically, no cleanup efforts of organophosphorus pesticides are made before analysis. If peak detection and identification are prevented because of interferences, cleanup may be considered. Before using any cleanup procedure, the analyst must process a standard through the procedure to validate elution pattern and absence of interference from reagents.

2.2 A gas chromatograph (GC) with a flame photometric detector (FPD) in the phosphorus mode or a thermionic bead detector (TID) or nitrogen phosphorus detector (NPD) is used in the capillary mode for purposes of examining multicomponent, multilevel organic residues extracted from water and soil/ sediment matrixes. A digital computer is used to acquire and process the associated data, based on available multi-internal standard techniques. Chemical components are qualitatively identified by relative retention times (RRT) and quantified by relative response factors (RRF), both values being calculated relative to appropriate internal standards.

2.3 Before extracts are analyzed, an initial calibration must be completed that demonstrates lack of instrument contamination and active degradation and adequate resolution and identification of analytes. Continuing calibration standards must be injected and evaluated for criteria compliance after every group of 10 or 20 extract injections (depending on analytical support level [ASL]) and at the end of the analytical sequence. Internal standards are used to determine RRTs and RRFs.

2.4 Target compounds tentatively identified by the primary analysis must be confirmed by at least one qualitative technique, either a second gas chromatograph analysis using a dissimilar analytical column or by GC/MS (if the concentration of the target parameter is above GC/MS detection limits). Confirmation analysis **must** be qualitative and **may** be quantitative.

A variety of problems may be encountered during the course of sample analysis. If data are acquired using automation without the immediate attendance of an analyst, calibration problems may require partial or complete reanalysis of the affected extracts. If extracts are not screened before primary analysis, targets may exceed the instrument calibration range. These extracts must be diluted and reanalyzed. Matrix interference will sometimes persist even after all optional cleanups are applied. Poor surrogate recovery can seldom be completely rectified by reextraction since the holding time for the raw sample has usually

expired before the surrogate recovery is measured (if enough raw sample is available for reextraction).

2.5 The laboratory prepares a case narrative and submits it with the sample data package. The analyst documents all problems and observations as samples are processed. The documentation is then used to qualify noncompliances in the data or analytical problems.

VOLATILE ORGANIC CARBONS BY GAS CHROMATOGRAPHY, PURGE AND TRAP (PROCEDURE FM-ORG-0050)

Working Linear Range:	1 to 200 µg/L
Reporting Limit:	Matrix dependent
Reporting Units:	µg/L or µg/kg dry weight
Matrix:	Water, soil, sediment or sludge

1.0 Scope and Application

This method describes procedures to analyze water, soil, and sediment samples for selected volatile organic compounds by using purge-trap-desorb gas chromatographic techniques. The target compound list and method detection limits are summarized in Table 1.

2.0 Method Summary

2.1 Water samples are analyzed directly by bubbling an inert gas through a sample contained in a specially designed purging chamber at ambient temperature.

2.2 Soil, sediment, and sludge samples are analyzed by one of two methods depending on the concentration of volatile components in the matrix. For low-level solid samples, the matrix is suspended in water and the suspension purged in a heated sparging chamber. Medium- to high-level solid samples are extracted with methanol. An aliquot of the methanol extract is spiked into reagent water and analyzed according to procedures described for water samples.

2.3 The volatile components from the different matrixes are thus transferred efficiently from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column (analytical trap) where the volatile components are stripped from the purge gas. After purging is completed, the sorbent column is heated and backflushed with an inert gas directed into a gas chromato-graph. The volatile components are transferred to a gas chromatographic column that is temperature programmed to separate the compounds. The gas chromatograph is equipped with a photoionization detector (PID) connected in series with a Hall electrolytic conductivity detector (HECD). The PID can detect aromatic compounds, and the HECD can detect the halogenated compounds.

Table 1
Target Compound List and Method Detection Limits

Compound	MDL (µg/L) 5-mL Sample	MDL (µg/L) 25-mL Sample
Chloromethane	1.0	0.5
Bromomethane	1.0	0.5
Dichlorodifluoromethane	1.0	0.5
Vinyl chloride	1.0	0.5
Chloroethane	1.0	0.5
Methylene chloride	1.0	0.5
Trichlorofluoromethane	1.0	0.5
1,1-Dichloroethene	1.0	0.5
1,1-Dichloroethane	1.0	0.5
trans-1,2-Dichloroethene	1.0	0.5
Chloroform	1.0	0.5
1,2-Dichloroethane	1.0	0.5
1,1,1-Trichloroethane	1.0	0.5
Carbon tetrachloride	1.0	0.5
Bromodichloromethane	1.0	0.5
1,2-Dichloropropane	1.0	0.5
cis-1,3-Dichloropropene	1.0	0.5
Trichloroethene	1.0	0.5
Dibromochloromethane	1.0	0.5
1,1,2-Tetrachloroethane	1.0	0.5
trans-1,3-Dichloropropene	1.0	0.5
Bromoform	1.0	0.5
1,1,2,2-Tetrachloroethane	1.0	0.5
Tetrachloroethene	1.0	0.5
2-Chloroethyl vinyl ether	1.0	0.5
1,2-Dibromo-3-chloropropane (DBCP)	1.0	0.5
1,1,2-Tetrachloropropane	1.0	0.5
Methylene bromide	1.0	0.5
1,2-Dibromoethane (Ethylene dibromide)	1.0	0.5
Benzene	1.0	0.5
Chlorobenzene	1.0	0.5
Toluene	1.0	0.5
Ethylbenzene	1.0	0.5
1,3-Dichlorobenzene	1.0	0.5
1,2-Dichlorobenzene	1.0	0.5
1,4-Dichlorobenzene	1.0	0.5
Xylenes (total)	1.0	0.5
Styrene	1.0	0.5

PREPARATION OF ORGANIC MATERIALS FOR VOC ANALYSIS (PROCEDURE FM-ORG-0052)

Working Linear Range:	Matrix and analyte dependent
Reporting Limit:	Matrix and analyte dependent
Reporting Units:	Parts per million (mass)
Matrix:	Wood, bulk solids with high organic content

1.0 Scope and Application

This is a method to prepare bulk organic samples with medium to high expected yields for VOC analysis by either FM-ORG-0010 or FM-ORG-0050.

2.0 Method Summary

2.1 The sample is cooled to -20°C and then finely divided by mechanical grinding. The equipment used to grind the sample is held at -20°C to maintain sample temperature during the process.

2.2 The samples are extracted with methanol at room temperature. An aliquot of the methanol extract is added to reagent water.

2.3 Analysis for VOCs in water proceeds as described in FM-ORG-0010 or in FM-ORG-0050.

SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/ MASS SPECTROMETRY (PROCEDURE FM-ORG-0060)

Working Linear Range:	Detection limit to 200 ppb
Reporting Limit:	Analyte- and matrix-dependent
Reporting Units:	µg/L or µg/kg
Matrix:	Water, soils, sediments and wastes

1.0 Scope and Application

1.1 The method is based on the use of gas chromatography (GC) in combination with mass spectrometry (MS) to identify and quantitate organic compounds in a sample. The components are separated in a GC column and identified and quantified by an MS detector.

1.2 Identification is based on the ionization of a compound into charged fragments using an electron beam. The fragmentation (called mass spectrum) pattern is unique to each compound. Since the MS cannot usually distinguish between isomers with the same mass spectra, the GC retention times provide a means for determining the identity of isomers.

1.3 Quantification is accomplished by comparing the area integration of a particular mass ion (called characteristic ion) in a compound's mass spectrum to that of a standard of known concentration.

1.4 Summarized in Table 1 are the target compounds and the corresponding practical quantitation limits.

2.0 Method Summary

2.1 The method is used for the determination of semivolatile organic compounds in waters, soils, sediments, and wastes.

2.2 The method covers the determination of organic compounds that may be partitioned into methylene chloride and are amenable to GC/MS. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons, pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols including nitrophenols.

Table 1
Practical Quantitation Limits
for Semivolatile Compounds[a]

Compound	Water (µg/L)	Soil/Sediment (µg/kg)
Acenaphthene	10	330
Acenaphthylene	10	330
Acetophenone	10	330
Aniline	20	660
Anthracene	10	330
4-Aminobiphenyl	50	2,400
Benzidine	50	2,400
Benzoic acid	50	2,400
Benzo(a)anthracene	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(g,h,i)perylene	10	330
Benzo(a)pyrene	10	330
Benzyl alcohol	20	660
Bis (2-chloroethoxy)methane	10	330
Bis (2-chloroethyl)ether	10	330
Bis (2-chloroisopropyl)ether	10	330
Bis (2-ethylhexyl)phthalate	10	330
4-Bromophenyl phenyl ether	10	330

Table 1
Practical Quantitation Limits
for Semivolatile Compounds[a]

Compound	Water (µg/L)	Soil/Sediment (µg/kg)	
Benzyl butyl phthalate	10	330	1
4-Chloroaniline	20	660	2
1-Chloronaphthalene	10	330	3
2-Chloronaphthalene	10	330	4
4-Chloro-3-methylphenol	20	660	5
2-Chlorophenol	10	330	6
4-Chlorophenyl phenyl ether	10	330	7
Chrysene	10	330	8
Dibenzo(a,h)anthracene	10	330	9
Dibenzofuran	10	330	10
Di-n-butylphthalate	10	330	11
1,3-Dichlorobenzene	10	330	12
1,4-Dichlorobenzene	10	330	13
1,2-Dichlorobenzene	10	330	14
3,3′-Dichlorobenzidine	20	330	15
2,4-Dichlorophenol	10	330	16
2,6-Dichlorophenol	10	330	17
Diethylphthalate	10	330	18
p-Dimethylaminoazobenzene	50	2,400	19
7,12-Dimethylbenz(a)anthracene	10	330	20
2,4-Dimethylphenol	10	330	21

Table 1
Practical Quantitation Limits
for Semivolatile Compounds[a]

Compound	Water (µg/L)	Soil/Sediment (µg/kg)	
Dimethylphthalate	10	330	1
4,6-Dinitro-2-methylphenol	50	330	2
2,4-Dinitrophenol	50	330	3
2,4-Dinitrotoluene	10	330	4
2,6-Dinitrotoluene	10	330	5
Diphenylamine	20	660	6
Di-n-octylphthalate	10	330	7
Ethyl methanesulfonate	10	330	8
Fluoranthene	10	330	9
Fluorene	10	330	10
Hexachlorobenzene	10	330	11
Hexachlorobutadiene	10	330	12
Hexachlorocyclopentadiene	10	330	13
Hexachloroethane	10	330	14
Indeno(1,2,3-cd)pyrene	10	330	15
Isophorone	10	330	16
3-Methylcholanthrene	10	330	17
Methyl methanesulfonate	10	330	18
2-Methylnaphthalene	10	330	19
2-Methylphenol (o-cresol)	10	330	20
4-Methylphenol (p-cresol)	10	330	21

Table 1
Practical Quantitation Limits
for Semivolatile Compounds[a]

Compound	Water (µg/L)	Soil/Sediment (µg/kg)	
Naphthalene	10	330	1
1-Naphthylamine	50	2,400	2
2-Naphthylamine	50	2,400	3
2-Nitroaniline	50	2,400	4
3-Nitroaniline	50	2,400	5
4-Nitroaniline	50	2,400	6
Nitrobenzene	10	330	7
2-Nitrophenol	10	330	8
4-Nitrophenol	50	2,400	9
N-Nitroso-di-n-butylamine	10	330	10
N-Nitrosodimethylamine	10	330	11
N-Nitrosodiphenylamine	10	330	12
N-Nitrosodi-n-propylamine	10	330	13
N-Nitrosopiperidine	10	330	14
Pentachlorobenzene	50	2,400	15
Pentachloronitrobenzene	10	2,400	16
Pentachlorophenol	50	2,400	17
Phenacetin	10	330	18
Phenanthrene	10	330	19
Phenol	10	330	20
2-Picoline	50	2,400	21

Table 1
Practical Quantitation Limits
for Semivolatile Compounds[a]

Compound	Water (µg/L)	Soil/Sediment (µg/kg)
Pyrene	10	330
1,2,4,5-Tetrachlorobenzene	50	2,400
2,3,4,6-Tetrachlorophenol	10	330
1,2,4-Trichlorobenzene	10	330
2,4,5-Trichlorophenol	50	2,400
2,4,6-Trichlorophenol	10	330

[a]Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance.

[b]PQLs listed for soil/sediment are based on wet weight. Normally data are reported on a dry weight basis; therefore, PQLs will be higher, based on the percent of moisture in each sample.

2.3 Problems have been associated with some compounds covered by the method. Dichlorobenzidine and 4-chloroaniline are subject to oxidative losses during solvent concentration, and chromatography is poor. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described. N-nitrosodiphenylamine decomposes in the GC inlet forming diphenylamine and consequently cannot be separated from diphenylamine native to the sample. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

2.4 Pesticides and PCBs should be analyzed by GC procedures provided for the specific pesticide class.

2.5 The method may be used for confirmation of chlorinated pesticides and PCBs identified by gas chromatography using electron capture detection (ECD) if concentrations permit.

PREPARATION OF BULK MATERIALS FOR SVOC ANALYSIS (PROCEDURE FM-ORG-0062)

Working Linear Range:	Matrix and analyte dependent
Reporting Limit:	Matrix and analyte dependent
Reporting Units:	Parts per million (mass)
Matrices:	Wood, bulk solids with high organic content, other solids

1.0 Scope and Application

1.1 This is a method to prepare bulk samples for SVOC analysis by FM-ORG-0060.

1.2 The method covers the determination of organic compounds that may be partitioned by using methylene chloride and are amenable to analysis by GC/MS.

2.0 Method Summary

2.1 The sample is cooled to 0°C or below, then finely divided by mechanical grinding. The equipment used to grind the sample is held at 0°C to maintain sample temperature during the process.

2.2 The cooled sample is transferred directly to an extraction vessel and SVOCs are extracted with methylene chloride.

2.3 Analysis proceeds as described in FM-ORG-0060.

HERBICIDES (PROCEDURE FM-ORG-0070)

Working Linear Range:	Matrix- and analyte-specific
Reporting Limit:	To be determined
Reporting Units:	Water, µg/L; soil (dry weight), µg/kg
Matrix:	Water, soil, waste, sediment, or sludge

1.0 Scope and Application

1.1 This is a gas chromatographic method applicable to the determination of the chlorinated acid herbicides listed in Table 1 in water, soil waste, sediment, and sludge.

1.2 The method detection limits for each target compound are summarized in Table 1. Because the method is matrix-dependent, practical quantitation limits are summarized in Table 2.

Table 1. Chromatographic Detection Limits for Chlorinated Herbicides

Compound	Method Detection Limit (µg/L)
2,4-D	1.2
2,4-DB	0.91
2,4,5-T	0.20
2,4,5-TP (Silvex)	0.17
Dalapon	5.8
Dicamba	0.27
Dichloroprop	0.65
Dinoseb	0.07
MCPA	249.0
MCPP	192.0

Table 2. Determination of Practical Quantitation Limits (PQL) for Various Matrices[a]

Matrix	Factor[b]
Ground Water	10
Low-level soil by sonication with GPC cleanup	670
High-level soil and sludges by sonication	10,000
Non-water miscible waste	100,000

[a] Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

[b] PQL = [Method detection limit (Table 1)] × [Factor (Table 2)]. For nonaqueous samples, the factor is on a wet weight basis.

2.0 Method Summary

2.1 The method includes extraction, esterification, and gas chromatographic conditions for analysis of chlorinated acid herbicides. The esters are hydrolyzed with potassium hydroxide, and extraneous organic material is removed by a solvent wash. After acidification, the acids are extracted with solvent and converted to their methyl esters using diazomethane as the derivetizing agent. The extract is cleaned by passing it through a micro-absorption column. After excess reagent is removed, the esters are analyzed by gas chromatography employing an electron capture detector. The results are reported as acid equivalents.

2.2 The sensitivity of the method usually depends on the level of interferences rather than on instrumental limitations.

VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY--DRINKING WATER (PROCEDURE FM-ORG-0100)

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Working Linear Range: Analyte- and matrix-dependent
Reporting Limit: Analyte- and matrix-dependent
Reporting Units: Liquids, µg/L

1.0 Scope and Application

1.1 This is a general-purpose method for identification and simultaneous measurement of purgeable volatile organic compounds in finished drinking water. The method is applicable to a wide range of organic compounds, including the four trihalomethane disinfection byproducts, that have sufficiently high volatility and low water solubility to be removed efficiently from water samples with purge-and-trap procedures. The following compounds (Table 1) can be determined by the method:

Table 1
Practical Quantitation Limits for Volatile Organics[a]

Compound	PQL (µg/L)
Benzene	5.0
Bromobenzene	25.0
Bromochloromethane	25.0
Bromodichloromethane	5.0
Bromoform	5.0
Bromomethane	5.0
n-Butylbenzene	25.0
sec-Butylbenzene	25.0
tert-Butylbenzene	25.0
Carbon tetrachloride	5.0
Chlorobenzene	5.0
Chloroethane	5.0
Chloroform	5.0
Chloromethane	5.0
2-Chlorotoluene	5.0
4-Chlorotoluene	5.0
1,2-Dibromo-3-chloropropane	5.0

Table 1
Practical Quantitation Limits for Volatile Organics[a]
(Continued)

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Compound	PQL (µg/L)
1,2-Dibromoethane	5.0
Dibromomethane	5.0
1,2-Dichlorobenzene	5.0
1,3-Dichlorobenzene	5.0
1,4-Dichlorobenzene	5.0
Dichlorofluoromethane	5.0
1,1-Dichloroethane	5.0
1,2-dichloroethane	5.0
1,1-Dichloroethene	5.0
cis-1,2-dichloroethene	5.0
trans-1,2-dichloroethene	5.0
1,2-Dichloropropane	5.0
1,3-Dichloropropane	5.0
2,2-Dichloropropane	5.0
1,1-Dichloropropene	5.0
Ethylbenzene	5.0
Hexachlorobutadiene	5.0
Isopropylbenzene	25.0
4-Isopropyltoluene	25.0
Methylene chloride	25.0
Naphthalene	5.0
n-Propylbenzene	5.0
Styrene	5.0
1,1,1,2-Tetrachloroethane	5.0
1,1,2,2-Tetrachloroethane	25.0
Tetrachloroethene	5.0
Toluene	5.0
1,2,3-Trichlorobenzene	5.0
1,2,4-Trichlorobenzene	5.0
1,1,1-Trichloroethane	5.0
1,1,2-Trichloroethane	5.0
Trichloroethene	5.0
Trichlorofluoromethane	5.0
1,2,3-Trichloropropane	5.0
1,2,4-Trimethylbenzene	5.0

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Table 1
Practical Quantitation Limits for Volatile Organics[a]
(Continued)

Compound	PQL (µg/L)
1,3,5-Trimethylbenzene	5.0
Vinyl chloride	5.0
o-Xylene	5.0
m-Xylene	5.0
p-Xylene	5.0

[a]Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

1.2 Method Detection Limits (MDLs) are chemical- and instrument-dependent and can range from 0.02 to 0.35 µg/L. The applicable concentration range of the method is primarily column-dependent and ranges from 0.02 to 200 µg/L for the wide-bore, thick-film columns. Narrow-bore, thin-film columns may have a loading capacity limit that will lower the range from 0.02 to 20 µg/L. Analytes that are inefficiently purged from water will not be detected when present at low concentrations, but they can be measured with acceptable accuracy and precision when present at higher concentrations. Practical quantitation limits are included in Table 1.

1.3 Analytes not separated chromatographically but having different mass spectra and noninterfering quantitation ions can be identified and measured in the same calibration mixture or water sample. Analytes that have very similar mass spectra cannot be individually identified and measured in the same calibration mixture or water sample unless they have different chromatographic retention times. Co-eluting compounds with very similar mass spectra, typically many structural isomers, must be reported as an isomeric group or pair. Two of the three xylenes and two of the three dichlorobenzenes are examples of structural isomers that may not be resolved on the capillary column and, if not, must be reported as isomeric pairs.

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2.0 Method Summary

Volatile organic compounds and surrogates with low water solubility are extracted (purged) from the sample matrix by bubbling an inert gas such as helium through the aqueous sample. Purged sample components are then trapped in a stainless steel tube containing suitable sorbent materials such as coconut charcoal, Tenax, or silica gel. When purging is complete, the sorbent tube is heated and backflushed with helium to desorb the trapped sample components onto a capillary gas chromatography (GC) column interfaced to a mass spectrometer (MS). The GC is temperature programmed to produce separation between the method analytes by their differing boiling points and sorption capacity, which are then detected with the MS. Compounds eluting from the GC column into the MS are identified by comparing their measured mass spectra and chromatographic retention times to standard reference spectra and retention times in the system's database. Reference spectra and retention times for method analytes are obtained by the initial measurement of calibration standards under the same system conditions used for analyzing the samples. The concentration of each component identified is measured by relating the MS response of the quantitation ion produced by that compound to the MS response of the quantitation ion produced by a compound used as an internal standard. Surrogate analytes, whose concentrations are known in every sample, are measured with the same internal standard calibration procedure to monitor purging efficiency or possible matrix interference.

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TOXICITY CHARACTERISTIC LEACHING PROCEDURE (PROCEDURE FM-MISC-0010)

Working Linear Range: Method- and analyte-specific
Reporting Limit: See list in Section 1.2
Reporting Units: mg/L
Matrix: Liquid or solid

1.0 Scope and Application

1.1 The TCLP is an analytical method designed to simulate the climatic leaching action expected to occur in landfills. Ideally, it determines the mobility of both organic and inorganic target analytes present in liquid, solid, and multiphasic wastes. The target analyte list includes 25 organic compounds (volatiles, semivolatiles, pesticides, and herbicides) and 8 metals.

1.2 Rather than defining a method detection limit and working range, the TCLP rule invokes a regulatory limit, when exceeded causes the waste to be deemed hazardous. For the target analytes of this operating procedure, the regulatory levels at which the waste is deemed hazardous are:

Target Analyte	Limit (mg/L)
Arsenic	5.0
Barium	100.0
Benzene	1.0
Cadmium	1
Carbon Tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
o-Cresol	200.0 *
m-Cresol	200.0 *
p-Cresol	200.0 *
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
1,1-Dichloroethene	0.7

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Target Analyte	Limit (mg/L)
2,4-Dinitrotoluene	0.13 **
Heptachlor (and its hydroxide)	0.008
Endrin	0.02
Hexachlorobenzene	0.5 **
Hexachloro-1,3-butadiene	0.5
Hexachloroethane	3.0
Lead	5
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0 **
Selenium	1
Silver	5
Tetrachloroethene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl Chloride	0.2

*If o-, m-, and p-cresol concentrations cannot be differentiated, then total cresol concentration is used. The regulatory level for total cresol is 200 mg/L.

**Quantitation limit is greater than the regulatory level. The quantitation limit therefore becomes the regulatory level.

1.3 If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high levels that even after accounting for dilutions from the other fractions of the extract the concentration would be above the regulatory threshold for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

1.4 If an analysis of extract obtained using a bottle extractor shows that the concentration of any regulated volatile contaminant exceeds the regulatory threshold for that compound, then the waste is hazardous and extraction using zero headspace extraction (ZHE) is not necessary. However, the extract from a bottle extractor cannot be used to demonstrate that the concentration of volatile compounds is below the regulatory threshold.

1.5 The analytical methods must have a method detection limit less than the regulatory limit.

1.6 If a total analysis of the waste demonstrates that individual contaminants are not present in the waste, or that they are present at such low concentrations that the appropriate regulatory thresholds could not possibly be exceeded, the TCLP need not be run.

2.0 Method Summary

Two extraction methods are detailed in this procedure: one for volatiles compounds only, the other for the remaining target compounds. The overall process is broken down into four major steps:

- Determining percent solids
- Identifying the appropriate extraction solution
- Extracting the sample
- Analyzing the sample for target compounds

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CORROSIVITY (PROCEDURE FM-MISC-0020)

Working Linear Range:	None
Reporting Limit:	None
Reporting Units:	None
Matrix:	Liquid

1.0 Scope and Application

1.1 The corrosivity characteristic, as defined in 40 CFR Part 261.22, is designed to identify wastes that might pose a hazard to human health or the environment because of their ability to:

- Mobilize toxic metals if discharged into a landfill environment
- Corrode handling, storage, transportation, and management equipment
- Destroy human or animal tissue in the event of inadvertent contact

1.2 A solid waste is corrosive if a representative sample of the waste has either of the following properties:

- It is aqueous and has a pH of < 2 or ≥ 12.5 as determined by a pH meter using SW-846 Method 9040.
- It is a liquid and corrodes steel (SAE 1020) at a rate greater than 6.35 mm (0.250 inch) per year at a test temperature of 55°C (130°F) as determined by SW-846 Method 1110.

1.3 If the waste is an aqueous liquid, then the pH can be measured using Method No. FM-CON-0110. However, if the waste is not aqueous in nature, then corrosivity must be evaluated using the Coupon Corrosivity Test as presented in SW-846 Method No. FM-CON-1110 and discussed below.

1.4 Rather than defining a method detection limit and working range, corrosivity is defined as a regulatory limit that, when exceeded, causes a liquid waste to be deemed hazardous. To designate a waste as corrosive towards steel (SAE7 1020), it must corrode steel at a rate greater than 6.35 mm (0.250 inch) per year at a test temperature of 55°C (130°F).

2.0 Method Summary

Corrosivity can be evaluated in two ways for a waste of interest. If the waste is an aqueous liquid, then the pH of the solution can be measured using Method No. FM-CON-0110. If it is not aqueous, then a preweighed steel coupon is immersed in the waste in a closed vessel at 55° ± 1°C for 24 hours and the loss of steel to corrosion is measured. If the rate of corrosion is greater than 6.35 mm per year, the waste is deemed corrosive.

IGNITABILITY (PROCEDURE FM-MISC-0030)

Working Linear Range:	None
Reporting Limit:	None
Reporting Units:	°C (or °F)
Matrix:	Liquid

1.0 Scope and Application

1.1 The ignitability characteristic is used to identify wastes that either present fire hazards under routine storage, disposal, and transportation or are capable of severely exacerbating a fire once started. A waste is ignitable if a representative sample has any of the following properties:

- It is a liquid, other than an aqueous solution, and has a flash point less than 60°C (140°F) and also contains less than 24% alcohol by volume, as determined by SW-846 Method 1010.
- It is not a liquid and is capable, under standard temperature and pressure, of causing fire through friction, absorption of moisture, or spontaneous chemical changes and, when ignited, burns so vigorously and persistently that it creates a hazard.
- It is an ignitable compressed gas.
- It is an oxidizer.

1.2 Flash point measures the tendency of the sample to form and flammable mixture with air under controlled laboratory conditions. Flash point can indicate the possible presence of highly volatile and flammable materials in a relatively nonvolatile or inflammable material.

1.3 Method 1010 is the Pensky-Martens closed-cup test for determining the flash point of liquids. Rather than defining a method detection limit and working range, ignitability is defined as a regulatory limit that, when exceeded, causes the waste to be deemed hazardous. To designate a waste as ignitable it must have a flash point less than 60°C (140°F).

2.0 Method Summary

A sample of the liquid waste is heated at a slow, constant rate with continual stirring. A small flame is directed at the test cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which the test flame ignites the vapor above the liquid sample.

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REACTIVITY (PROCEDURE FM-MISC-0040)

Working Linear Range:	None
Reporting Limit:	None
Reporting Units:	mg/kg
Matrix:	Liquid or solid
Holding Time:	None

Introduction

A solid waste is considered to be reactive if it exhibits any of the following characteristics:

- It is normally unstable and readily undergoes violent change without detonation.
- It reacts violently with water.
- It forms potentially explosive mixtures with water.
- When mixed with water, it generates toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.
- It is a cyanide- or sulfide-bearing waste that, when exposed to pH conditions between 2 and 12.5, can generate toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.
- Is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement.
- It is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure.
- It is a forbidden explosive, as defined in 49 CFR 173.51, .53, or .88.
- A solid waste that exhibits the characteristic of reactivity, but is not listed as a hazardous waste in Subpart D, and has the EPA Hazardous Waste Number of D003.

Of the nine characteristics listed above, only one (level of releasable cyanide or sulfide) can be quantified. Currently the EPA regulatory limit that, when exceeded, causes the waste to be deemed hazardous is:

- Total releasable cyanide: 250 mg HCN/kg waste
- Total releasable sulfide: 500 mg H_2S/kg waste

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This operating procedure discusses separately the determination of releasable cyanide and sulfide.

1.0 Determination of Releasable Hydrogen Cyanide

1.1 Scope and Application

The method is applicable to all wastes, with the condition that the waste is not explosive when combined with acids. The method provides a way to estimate the specific rate of release of hydrogen cyanide upon contact with an aqueous acid. The procedure measures only the hydrogen cyanide evolved under the test conditions, and it is not intended to measure other forms of cyanide. Rather than defining a method detection limit and working range, reactivity is defined as a regulatory limit that, when exceeded, causes the waste to be deemed hazardous. To designate a waste as reactive, it must have a total releasable cyanide concentration greater than 250 mg CN/kg waste.

1.2 Method Summary

1.2.1 The method is applicable to all wastes, with the condition that the waste is not explosive when combined with acids. The method provides a way to estimate the specific rate of release of hydrocyanic acid upon contact with an aqueous acid. The procedure measures only the hydrocyanic acid evolved under the test conditions, and it is not intended to measure other forms of cyanide. In summary, an aliquot of the waste is acidified to pH 2 in a closed system. Any gas generated by the waste is swept into a scrubber, and the releasable cyanide is measured titrametrically using SW-846 Method 9010A (November 1990).

1.2.2 As discussed above, rather than defining a method detection limit and working range, reactivity is defined as a regulatory limit that, when exceeded, causes the waste to be deemed hazardous. To designate a waste as reactive, it must have a total releasable cyanide concentration greater than 250 mg HCN/kg waste.

2.0 Determination of Releasable Hydrogen Sulfide

2.1 Scope and Application

The method is applicable to all wastes, with the condition that the waste is not explosive when combined with acids. The method provides a way to estimate the specific rate of release of hydrogen sulfide upon contact with an aqueous acid. The procedure measures only the hydrogen sulfide evolved under the test conditions, and it is not intended to measure

other forms of sulfide. Rather than defining a method detection limit and working range, reactivity is defined as a regulatory limit that, when exceeded, causes the waste to be deemed hazardous. To designate a waste as reactive, it must have a total releasable sulfide concentration greater than 500 mg H_2S/kg waste.

2.2 Method Summary

An aliquot of waste is acidified to pH 2 in a closed system. Any gas generated by the waste is swept into a scrubber, and the releasable sulfide is quantified titrametrically.

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METALS BY GRAPHITE FURNACE ATOMIC ABSORPTION (PROCEDURE FM-INO-0010)

Working Linear Range:	Metal-dependent; see specific metal
Reporting Limits:	Metal-dependent; see specific metal
Reporting Units:	µg/L for water; mg/kg for solids
Matrix:	Water, soil, sediment, and waste

1.0 Scope and Application

1.1 Metals in solution may be readily determined by several techniques, including atomic absorption spectroscopy. The graphite furnace technique often offers the advantage of lower detection limits than the conventional flame and the inductively coupled plasma techniques. Another advantage of the technique is that it requires only a small volume for analysis. It is, however, subject to more interferences than the flame technique and requires longer analysis time.

1.2 The graphite furnace technique is applicable to the analysis of metals in aqueous and nonaqueous samples following digestion. It is recommended for determinations that require detection limits that cannot be achieved by other methods. Estimated detection limits are listed in Table 1. Detection limits are dependent on the spectrometer and the sample matrix.

2.0 Method Summary

The sample is subjected to an acid digestion procedure to ensure that the metals are in solution (the digestion procedure may be done using microwave energy). The digestate is then analyzed by graphite furnace atomic absorption spectroscopy and the concentration determined using a calibration curve. Most metals may require the addition of matrix modifiers to sample during the sample injection process.

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Table 1
Recommended Wavelengths and Estimated Detection Limits for Graphite Furnace Analysis

Element	Wavelength (nm)	Estimated Detection Limit–Waters (µg/L IDL)	Required Reporting Limit–Waters (µg/L CRDL)	Required Reporting Limit–Soils (mg/kg)
Antimony	217.6	3	60	12
Arsenic	193.7	1	10	2
Beryllium	234.9	0.2	5	1
Cadmium	228.8	0.1	5	1
Chromium	357.9	1	10	2
Cobalt	240.7	1	*	*
Lead	283.3	1	3	0.6
Selenium	196.0	2	5	1
Silver	328.1	0.2	10	2
Thallium	276.8	1	10	2
Vanadium	318.4	4	*	*

* Not available.

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METALS IN BULK SOLIDS BY ATOMIC ABSORPTION: PREPARATORY METHOD (PROCEDURE FM-INO-0012)

Working Linear Range:	N/A
Reporting Limit:	N/A
Reporting Units:	N/A
Matrix:	Bulk solids with high organic content, earthen solids

1.0 Scope and Application

This method is applicable to the preparation of bulk solids for the analysis of metals by method FM-INO-0010 or by method FM-INO-0020.

2.0 Method Summary

2.1 Samples are ground to approximately 16 mesh or less and ashed to remove organics.

2.2 The residue is homogenized and an aliquot is acid-digested on a steam bath or hot plate. The digestate is then analyzed per FM-INO-0010 or FM-INO-0020.

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METALS BY ATOMIC ABSORPTION SPECTROMETRY, FLAME TECHNIQUE (PROCEDURE FM-INO-0020)

Working Linear Range:	Metal-dependent; see specific metal
Reporting Limit:	Metal-dependent; see specific metal
Reporting Units:	Water, µg/L; solids, mg/kg
Matrix:	Water, soil, sediment, and waste

1.0 Scope and Application

1.1 Scope and Application: The method is applicable to a large number of metals in drinking, surface, and saline waters and domestic and industrial wastes. Drinking water free of particulate matter may be analyzed directly, but groundwater, other aqueous samples, TCLP extracts, industrial wastes, soils, sludges, sediments, and other solid wastes require digestion before analysis.

1.2 Detection limits, sensitivity, and optimum ranges for analysis will vary with sample matrix and the model of atomic absorption system used (and method of background correction). Table 1 lists typical detection limits for clean samples. Detection limits may be improved by sample concentration or by solvent extraction techniques.

1.3 Table 1 lists metals that may be analyzed using the flame technique. Where the flame technique does not offer detection low enough to meet the required reporting limits, other techniques must be used.

2.0 Method Summary

An aliquot of sample is acid-digested on a steam bath or hot plate, or using microwave energy. The volume of the digestate is adjusted. The digestate is then aspirated into the flame and the absorbance measured at a characteristic wavelength. The concentration of the sample is determined using a calibration curve.

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Table 1
Detection and Reporting Limits
Flame Atomic Absorption

Metal	Wavelength (nm)	Detection Limits (Water)(µg/L)	Required Reporting Limits (Water)(µg/L)	Required Reporting Limits (Soil)(mg/kg)
Aluminum	309.3	100	200	40
Antimony	217.6	200	60	12
Barium	553.6	100	200	40
Beryllium	234.9	5	5	1
Cadmium	228.8	5	5	1
Calcium	422.7	10	5,000	1,000
Chromium	357.9	50	10	2
Cobalt	240.7	50	50	10
Copper	324.7	20	25	5
Iron	248.3	30	100	20
Lead	283.3	100	3	0.6
Magnesium	285.2	1	5,000	1,000
Manganese	279.5	10	15	3
Nickel	232.0	40	40	8
Potassium	766.5	10	5,000	1,000
Silver	328.1	10	10	2
Sodium	589.6	2	5,000	1,000
Thallium	276.8	100	10	2
Vanadium	318.4	200	50	10
Zinc	213.9	5	20	4

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METALS BY INDUCTIVELY COUPLED PLASMA (PROCEDURE FM-INO-0030)

Working Linear Range:	Matrix- and analyte-dependent
Reporting Limits:	Metal-dependent; see specific metal
Reporting Units:	Water, µg/L; solids, mg/kg
Matrix:	Water, soil, sediment, and waste

1.0 Scope and Application

Dissolved elements are determined in filtered and acidified samples. Total elements are determined after appropriate digestion procedures are performed. Table 1 lists elements with recommended wavelengths and typical estimated instrument detection limits using conventional pneumatic nebulization. Actual method detection limits are instrument dependent and may vary with sample matrix. The required reporting limits are listed in Table 1. The list includes all metals that may be analyzed by ICP. Certain metals, however, have regulatory limits that are often lower than the levels that may be detected by ICP. They normally include arsenic, lead, selenium, and thallium. When low detection limits are required, more sensitive techniques, such as graphite furnace, may have to be considered as the method of choice.

2.0 Method Summary

2.1 Water samples are prepared by digesting an aliquot of the sample with nitric acid and hydrochloric acid to put the metals in solution. After volume adjustment, the digestate is analyzed by ICP. Filtration of the digestate may be necessary.

2.2 A representative aliquot of soil samples is digested in nitric acid and hydrogen peroxide, and the digestate is refluxed with hydrochloric acid. After volume adjustment, the digestate is analyzed by ICP. The digestate must be filtered. A separate aliquot is used for moisture determination if results are to be reported on a dry weight basis.

2.3 Both water and soil samples may be digested with nitric acid using microwave energy. The volume of digestate is adjusted, and analysis by ICP is performed.

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Table 1
Recommended Wavelengths and Detection Limits*

Analyte	Wavelength (nm)	Estimated Detection Limit Waters (µg/L)	Required Reporting Limit for Waters (µg/L)	Required Reporting Limit for Soils (mg/kg)
Aluminum	308.215	45	200	40
Antimony	206.833	32	60	12
Arsenic	193.696	53	10	2
Barium	455.403	2	200	40
Beryllium	313.042	0.3	5	1
Boron	249.773	0.3	1,000	200
Cadmium	226.502	4	5	1
Calcium	317.933	10	5,000	1,000
Chromium	267.716	7	10	2
Cobalt	228.616	7	50	10
Copper	324.754	6	25	5
Iron	259.940	7	100	20
Lead	220.353	42	3	0.6
Lithium	670.70	5	5,000	1,000
Magnesium	279.079	30	5,000	1,000
Manganese	257.610	2	15	3
Molybdenum	202.030	8	10	2
Nickel	231.604	15	40	8
Potassium	766.491	263	5,000	1,000
Selenium	196.026	75	5	1
Silver	328.068	7	10	2
Sodium	588.995	29	5,000	1,000
Thallium	190.864	40	10	2
Vanadium	292.402	8	50	10
Zinc	213.856	2	20	4

*The wavelengths listed are recommended because of their sensitivity and overall acceptance. Others may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. The use of alternative wavelengths should be reported (in nm) with the sample data. The actual method detection limits are instrument dependent and may change as the sample matrix varies.

MERCURY ANALYSIS BY COLD VAPOR (ATOMIC ABSORPTION SPECTROMETRY) (PROCEDURE FM-INO-0040)

Working Linear Range:	Matrix- and analyte-specific
Reporting Limit:	0.2 µg/L; 0.04 mg/kg
Reporting Units:	Water, µg/L; Solids, mg/kg
Matrix:	Water, soil, sediment, and waste

1.0 Scope and Application

1.1 The method is based on the absorption of radiation at 253.7 nm by mercury vapor. Mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorption is measured as a function of the mercury concentration.

1.2 The method is applicable to the determination of organic and inorganic mercury in water samples, aqueous wastes, mobility-procedure extracts, soils, and solid wastes.

1.3 An automated procedure has been included for the analysis of water samples. The procedure suffers from interference effects from high chloride in the sample. Heavy precipitate formation in some wastewaters and effluents upon the addition of concentrated sulfuric acid has also been reported. When these problems are encountered, the manual method should be used.

2.0 Method Summary

Organic and inorganic forms of mercury in the sample are oxidized to the mercuric state by potassium permanganate and potassium persulfate. Excess oxidizers are reduced using a sodium-chloride-hydroxylamine hydrochloride solution. The mercuric ions are then reduced to the elemental state by adding stannous chloride. The mercury vapor is aerated and swept into an absorption cell. The absorbance reading is measured and compared with those of standard solutions.

CYANIDE (TOTAL)--TITRIMETRIC (PROCEDURE FM-CON-0010)

Working Linear Range: Greater than 1.0 mg/L, infinite with dilution
Reporting Limit: 1.0 mg/L
Reporting Units: Water, mg/L; Solids, mg/kg
Matrix: Water, wastes, soil, and sediment

1.0 Scope and Application

1.1 The method is applicable to the determination of cyanide in drinking, ground, surface, and saline waters; domestic and industrial aqueous wastes; soils; and sediments.

1.2 The titration procedure using silver nitrate with p-dimethylaminobenzylrhodanine indicator for measuring concentrations of cyanide exceeding 1 mg/L (0.25 mg/250 mL of absorbing liquid).

2.0 Method Summary

2.1 Cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution (NaOH).

2.2 The titrimetric measurement uses a standard solution of silver nitrate to titrate cyanide in the presence of a silver sensitive indicator.

CYANIDE (TOTAL)--SPECTROPHOTOMETRIC, SEMIAUTOMATIC (PROCEDURE FM-CON-0015)

Working Linear Range: 0.005 to 0.200 mg/L
Reporting Limit: 0.005 mg/L
Reporting Units: Water, mg/L; Solids, mg/kg
Matrix: Water, wastes, soils, and sediments

1.0 Scope and Application

1.1 The method is applicable to the determination of cyanide in drinking, ground, surface, and saline waters; domestic and industrial aqueous wastes; soils; and sediments.

1.2 The working range of the semiautomated spectrophotometric method is 0.005 to 0.200 mg/L. Higher level samples must be diluted to fall within the working range.

2.0 Method Summary

2.1 Cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution (NaOH). The cyanide ion in the absorbing solution is then determined colorimetrically.

2.2 Cyanide is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The absorbance is read at 578 nm for pyridine-barbituric acid.

SOIL pH (PROCEDURE FM-CON-0020)

Working Linear Range:	0 to 14
Reporting Limit:	N/A
Reporting Units:	pH units
Matrix:	Soil, sediment, or sludge
Holding Time:	Analyze immediately

1.0 Scope and Application

This is an electrometric procedure that can be applied to measuring pH in calcareous and noncalcareous soils.

2.0 Method Summary

The soil sample is mixed either with ASTM Type II water or with a calcium chloride ($CaCl_2$) solution, depending whether the soil is considered to be calcareous or noncalcareous. The pH of the solution is then measured with a pH meter.

NITROGEN, NITRATE-NITRITE (COLORIMETRIC, AUTOMATED, CADMIUM REDUCTION) (PROCEDURE FM-CON-0030)

Working Linear Range: 0.05 to 10.0 mg/L nitrate-nitrite as N
Reporting Limit: 0.05 mg/L as N
Reporting Units: mg/L as N
Matrix: Water and wastes

1.0 Scope and Application

This method is applicable to the determination of nitrite only or of nitrite and nitrate combined, in fresh and saline waters and in domestic and industrial aqueous wastes. The applicable range of the method is 0.05 to 10.0 mg/L as N. The range may be extended with sample dilution.

2.0 Method Summary

A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. The nitrite (that originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye that is measured colorimetrically. Separate nitrate and nitrite values, rather than combined, are readily obtained by carrying out the procedure first with, then without, the copper-cadmium reduction step.

NITROGEN, NITRATE-NITRITE (SPECTROPHOTOMETRIC, CADMIUM REDUCTION) (PROCEDURE FM-CON-0040)

Working Linear Range: 0.01 to 1.0 mg/L nitrate-nitrite as N
Reporting Limits: 0.01 mg/L as N
Reporting Units: mg/L as N
Matrix: Water and wastes

1.0 Scope and Application

The method is applicable to the determination of nitrite only or nitrite and nitrate combined in drinking, surface, and saline waters and domestic and industrial aqueous wastes. The applicable range of the method is 0.01 to 1.0 mg/L as N. The range may be extended with sample dilution.

2.0 Summary of Method

A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. The nitrite (that originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye that is measured spectrophotometrically. Separate rather than combined nitrate-nitrite values are readily obtained by carrying out the procedure first with, then without, the copper-cadmium reduction step.

NITRATE/NITRITE BY FLOW INJECTION ANALYSIS (PROCEDURE FM-CON-0042)

Working Linear Range:	Infinite with dilution
Reporting Limit:	Varies by matrix
Reporting Units:	Water, mg/L; solids, mg/Kg
Matrix:	Water, soil, sediment, non-soil solids

1.0 Scope and Application

This method is used to determine the concentration of nitrate/nitrite ions concentration by automated flow injection analyzer. This method is applicable for an aqueous matrix with no suspended solids and can be extended for use with other matrices in conjunction with FM-CON-0086 which serves to dissolve the nitrate/nitrite anions in an aqueous matrix.

2.0 Method Summary

Nitrate is quantitatively reduced to nitrite by treatment of the sample with a copperizd cadmium metal column. The nitrite (reduced nitrate plus original nitrite) is then determined by diazotizing with sulfanilamide, followed by coupling with N-(1-naphthyl) ethylenediamine-dihydrochloride. The resulting water-soluble dye is magenta-colored and its absorption is measured at 520 nm. Nitrite alone can be determined by removing the cadmium reduction column.

CONDUCTIVITY (PROCEDURE FM-CON-0050)

Working Linear Range: NA
Reporting Limit: µmhos/cm
Reporting Units: µmhos/cm
Matrix: Water

1.0 Scope and Application

This method is applicable to drinking, ground, surface, and saline waters and domestic and industrial aqueous wastes.

2.0 Method Summary

2.1 Sample conductivity is measured using a self-contained conductivity meter (Wheatstone bridge-type, or equivalent).

2.2 Ideally, samples are analyzed at 25°C. If not, temperature corrections are made and results reported at 25°C.

TOTAL KJELDAHL NITROGEN (PROCEDURE FM-CON-0060)

Working Linear Range: 0.1 to 100 mg/L (matrix-dependent)
Reporting Limit: 0.1 mg/L
Reporting Units: mg/L
Matrix: Water and wastes

1.0 Scope and Application

1.1 The method covers the determination of total Kjeldahl nitrogen (TKN) in drinking, surface, and saline waters and domestic and industrial aqueous wastes. The procedure converts nitrogen components of biological origin such as amino acids, proteins and peptides to ammonia, but may not convert nitrogenous compounds of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, semicarbazones, and some refractory tertiary amines.

1.2 Two alternatives are listed for the determination of ammonia after distillation: the titrimetric method, which is applicable to concentrations above 1 mg N/liter; or the Nesslerization method, which is applicable to concentrations below 1 mg N/liter.

1.3 The method is described for macro and micro glassware systems.

2.0 Method Summary

The sample is heated in the presence of concentrated H_2SO_4, K_2SO_4, and $HgSO_4$ and evaporated until SO_3 fumes are obtained and the solution becomes colorless or pale yellow. The residue is cooled, diluted, treated, and made alkaline with a hydroxide-thiosulfate solution. The ammonia is distilled and determined after distillation by Nesslerization or titration.

TOTAL ORGANIC CARBON (PROCEDURE FM-CON-0080)

Working Linear Range: Instrument-dependent
Reporting Limits: 1 mg/L
Reporting Units: mg/L
Matrix: Water and wastes

1.0 Scope and Application

The method is applicable to the measurement of organic carbon in drinking, ground, surface, and saline waters and domestic and industrial aqueous wastes.

2.0 Method Summary

2.1 Total carbon is defined as all the carbon present in a sample and comprises total organic and inorganic carbon. Each carbon analyzer is different, and each instrument handles total inorganic carbon differently.

2.1.1 Total Inorganic Carbon (TIC): Refers to carbon that is converted to carbon dioxide after acidification of a sample. TIC includes all dissolved carbon dioxide, bicarbonate, and carbonate species and is reported in terms of total mass of carbon per unit of sample (e.g., mg C/L).

2.1.2 Total Organic Carbon (TOC): Refers to all carbon atoms bonded in organic molecules that are converted to carbon dioxide by oxidation after inorganic carbon has been removed or subtracted.

2.2 Organic carbon is measured using a carbonaceous analyzer. Organic carbon in a sample is converted to carbon dioxide (CO_2) by either catalytic combustion or wet chemical oxidation, depending on the analytical instrument chosen. The CO_2 formed is then either measured directly by an infrared detector or converted to methane (CH_4) and measured by a flame ionization detector. The amount of CO_2 or CH_4 in a sample is directly proportional to the concentration of carbonaceous material in the sample.

2.3 Carbonaceous analyzers are capable of measuring all forms of carbon in a sample. However, because of various properties of carbon-containing compounds in liquid samples, sample preparation and choice of analytical instruments will determine which forms of carbon are actually measured. The forms of carbon that can be measured by the method are:

- Soluble, nonvolatile organic carbon: e.g., natural sugars
- Soluble, volatile organic carbon: e.g., mercaptans, alkanes, low molecular weight alcohols
- Insoluble, partially volatile carbon: e.g., low molecular weight oils
- Insoluble, particulate carbonaceous materials: e.g., cellulose fibers
- Soluble or insoluble carbonaceous materials adsorbed or entrapped on insoluble inorganic suspended matter: e.g., oily matter adsorbed on silt particles

2.4 This is a quantitative analysis that qualitatively detects all organic carbon rather than a specific form or compound. The working range of the analysis is dependent upon the TOC analyzer and the range of working calibration standards used.

ANALYSIS OF TOTAL ORGANIC CARBON IN SOILS, SLUDGES, OR NON-SOIL SOLIDS MATRIX (PROCEDURE FM-CON-0082)

Working Linear Range:	Infinite with dilution
Reporting Limit:	Varies by matrix
Reporting Units:	mg/Kg
Matrix:	Soils, sludges, sediments, non-soil solids

1.0 Scope and Application

This method is applicable for non-aqueous matrix samples in conjunction with (1) FM-CON-0086 which serves to dissolve water-soluble analytes in an aqueous matrix; or (2) the procedure described in the Dohrmann Xertex DC-80 Total Organic Carbon Analyzer Systems manual for solid samples using the auxiliary high-temperature combustion furnace.

2.0 Method Summary

TOC is measured by high-temperature combustion, followed by non-dispersive infrared detection.

ANALYSIS OF TOTAL ORGANIC CARBON (PROCEDURE FM-CON-0085)

Working Linear Range: Infinite with dilution
Reporting Limit: Varies by matrix
Reporting Units: Water, mg/L
Matrix: Water

1.0 Scope and Application

This method is applicable for an aqueous matrix with no suspended solids and can be extended for use with other matricies in conjunction with: (1) FM-CON-0082 which is a detailed SOP for performing TOC analyses on solid matrix samples; (2) FM-CON-0086 which serves to dissolve water-soluble analytes in an aqueous matrix; or (3) the procedure described in the Dohrmann Xertex DC-80 Total Organic Carbon Analyzer Systems manual for solid samples using the auxiliary high-temperature combustion furnace.

2.0 Method Summary

TOC is measured by UV-promoted persulfate oxidation or by high-temperature combustion, followed by non-dispersive infrared detection. External sparging is used to remove inorganic carbon.

DISSOLUTION OF ANIONS FROM SOLID MATERIALS (PROCEDURE FM-CON-0086)

Working Linear Range:	Infinite with dilution
Reporting Limit:	Varies by matrix and by test
Reporting Units:	mg/Kg
Matrix:	Soil, sediment, and non-soil solids

1.0 Scope and Application

This extraction procedure is to extract soluble anions from soil or other solid material for the analysis of anions or to perform other analyses and is applicable to a large number of solid matrices and to analytical testing procedures that may not provide for the analysis of solid matrices.

2.0 Method Summary

2.1 This method is based on "Methods for Chemical Analysis of Water and Wastes," USEPA 600/4-79-020, March, 1983. The procedure referenced is:

Method 300.0: The Determination of Inorganic Anions in Water by Ion Chromatography.

2.2 This method is based on "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," USEPA SW 846, 3rd edition. The procedure referenced is:

Method 9045: Soil pH

2.3 An amount of ASTM Type II deionized water equal to ten times the dry weight of solid material taken as an aliquot is added to the sample. This slurry is mixed together for ten minutes using a magnetic stirring device and sonicated for 15 minutes. The resulting slurry is filtered before analysis using a 0.45 µm membrane filter. Other liquid to solid ratios may be employed, but the ratios actually used must be documented.

ALKALINITY (TITRIMETRIC) (PROCEDURE FM-CON-0090)

Working Linear Range: All concentration ranges
Reporting Limit: 1.0 mg/L
Reporting Units: mg/L as $CaCO_3$
Matrix: Water and wastes

1.0 Scope and Application

1.1 The method is applicable to drinking, surface, and saline waters and domestic and industrial aqueous wastes.

1.2 The method is suitable for all concentration ranges of alkalinity. Appropriate aliquots should be used to avoid a titration volume greater than 50 mL.

2.0 Method Summary

An unaltered sample is titrated with a standard acid solution to a pH of 4.5 for total alkalinity. The endpoint may be determined electrometrically or with a color indicator. The sample must not be filtered, diluted, or altered in any way.

ALKALINITY (COLORIMETRIC) (PROCEDURE FM-CON-0100)

Working Linear Range: 10 to 200 mg/L as $CaCO_3$
Reporting Limit: 10 mg/L as $CaCO_3$
Reporting Units: mg/L as $CaCO_3$
Matrix: Water and wastes

1.0 Scope and Application

1.1 This automated method is applicable to drinking, surface, and saline waters and domestic and industrial aqueous wastes. The applicable range is 10 to 200 mg/L as $CaCO_3$.

1.2 The method is not approved for NPDES monitoring for samples containing turbidity or color.

2.0 Method Summary

Methyl orange is used as the indicator in the method because its pH range is in the same range as the equivalence point for total alkalinity and because it has a distinct color change that can be easily measured. The methyl orange is dissolved in a weak buffer at a pH of 3.1, just below the equivalence point, so that any addition of alkalinity causes a loss of color directly proportional to the amount of alkalinity.

pH (ELECTROMETRIC) (PROCEDURE FM-CON-0110)

Working Linear Range: 0 to 14
Reporting Limit: N/A
Reporting Units: pH units
Matrix: Water and wastes

1.0 Scope and Application

This method is applicable to drinking, surface, ground, and saline waters and domestic and industrial aqueous wastes.

2.0 Method Summary

The pH of a sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode.

CHLORIDE (COLORIMETRIC, AUTOMATED FERRICYANIDE) (PROCEDURE FM-CON-0120)

Working Linear Range: 1.0 to 200 mg/L
Reporting Limit: 1.0 mg/L
Reporting Units: mg/L
Matrix: Water and wastes

1.0 Scope and Application

This automated method is applicable to drinking, surface, saline, and ground waters and domestic and industrial wastes. The applicable range is 1 to 200 mg Cl^- per liter of sample. The range can be extended by diluting the sample.

2.0 Method Summary

Thiocyanate ion (SCN) is liberated from mercuric thiocyanate through sequestration of mercury by chloride ion to form un-ionized mercuric chloride. In the presence of ferric ion, the liberated SCN forms highly colored ferric thiocyanate in a concentration proportional to the original chloride concentration.

SULFIDE (PROCEDURE FM-CON-0130)

Working Linear Range: Infinite with dilution
Reporting Limit: 1 mg/L
Reporting Units: mg/L
Matrix: Water

1.0 Scope and Application

1.1 The method applies to the measurement of total sulfides in drinking, surface, saline, and ground waters. The applicable range is infinite with proper dilutions.

1.2 Acid-insoluble sulfides are not measured by this test. (Copper sulfide is the only common sulfide in this class.)

2.0 Method Summary

Excess iodine is added to a water sample that has been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is back-titrated with sodium thiosulfate.

ANALYSIS OF TOTAL SULFIDE IN WATER, SOIL, AND SEDIMENT MEDIA (PROCEDURE FM-CON-0134)

Working Linear Range:	Infinite with dilution
Reporting Limit:	Varies by matrix
Reporting Units:	Water, mg/L, µg/L; Solids, mg/Kg, µg/Kg
Matrix:	Water, soil, sediment, non-soil solids

1.0 Scope and Application

This method is used to determine the concentration of total sulfide and is applicable to a large number of matricies. All matricies require acidic reflux-distillation into an aqueous caustic scrubber solution before analysis. An iodometric titration analysis is performed on an aliquot of the distillate; an excess of iodine solution is added to the distillate aliquot and back-titrated with a standardized thiosulfate solution.

2.0 Method Summary

2.1 This method is based on "Methods for Chemical Analysis of Water and Wastes," USEPA SW 846, 3rd edition. The procedure referenced is:

Method 376.1: Sulfide, Titrimetric, Iodine

2.2 This method is based on "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," USEPA SW 846, 3rd edition. The procedure referenced is:

Method 9012: Sulfide

2.3 The sample is refluxed and distilled with acid to release sulfide as hydrogen sulfide (H_2S), which is absorbed in a scrubber containing NaOH and is determined titrimetrically by titration of an aliquot of the distillate. A known volume of iodine solution is added to the aliquot so there is an excess of iodine stoichiometrically compared to the amount of sulfide in the aliquot. The aliquot is then titrated with standardized thiosulfate solution to a colorless endpoint, using a starch indicator.

AMMONIA (PROCEDURE FM-CON-0140)

Working Linear Range: 1.0 to 25 mg NH_3-N/L
Reporting Limit: 1.0 mg NH_3-N/L
Reporting Units: mg NH_3-N/L
Matrix: Water

1.0 Scope and Application

The method is applicable to measurement of ammonia in surface, saline, and ground waters and domestic and industrial aqueous wastes.

2.0 Method Summary

The sample is buffered at a pH of 9.5 with a borate buffer to decrease hydrolysis of cyanates and organic nitrogen compounds, and is then distilled into a solution of boric acid. The concentration of ammonia in the distillate is determined titrametrically with standard sulfuric acid using a mixed indicator.

HEXAVALENT CHROMIUM (CR^{6+}) (PROCEDURE FM-CON-0150)

Working Linear Range: 10 to 250 µg/L
Reporting Limit: 10 µg/L
Report Units: µg/L
Matrix: Water

1.0 Scope and Application

The method covers the determination of dissolved hexavalent chromium in drinking, surface, and saline waters. It may be applicable to some domestic and industrial aqueous wastes.

2.0 Method Summary

Only hexavalent chromium is measured using the method. Hexavalent chromium in a sample is determined colorimetrically by reaction with diphenylcarbazide in an acid solution, which produces a violet color of unknown composition.

TEMPERATURE (PROCEDURE FM-CON-0160)

Working Linear Range:	Thermometer-dependent
Reporting Limit:	Thermometer-dependent
Reporting Unit:	°C
Matrix:	Water and waste

1.0 Scope and Application

This method is applicable to drinking, surface, and saline waters and domestic and industrial aqueous wastes.

2.0 Method Summary

Temperature measurements may be made with any good grade of mercury-filled or dial type centigrade thermometer or a thermistor. Measurement device should be routinely checked against a precision thermometer certified by the NIST. At a minimum, the thermometer should have a scale marked for every 0.1°C, with markings etched on the capillary glass. The thermometer should have a minimal thermal capacity to permit rapid equilibration. Temperature measurements are often made in conjunction with other methods.

CHLORIDE (TITRIMETRIC, MERCURIC NITRATE) (PROCEDURE FM-CON-0170)

Working Linear Range: All concentration ranges
Reporting Limit: 1.0 mg/L
Reporting Units: mg/L
Matrix: Water and wastes

1.0 Scope and Application

1.1 The method is applicable to drinking, surface, saline, and ground waters and domestic and industrial aqueous wastes.

1.2 The method is suitable for all concentration ranges of chloride content. To avoid large titration volumes, a sample aliquot containing not more than 10 to 20 mg Cl^- per 50 mL should be used.

1.3 Automated titration may be used.

2.0 Method Summary

An acidified sample is titrated with mercuric nitrate in the presence of mixed diphenylcarbazone-bromophenol blue indicator. The end point of the titration is the formation of the blue-violet mercury diphenylcarbazone complex.

OIL AND GREASE--INFRARED (PROCEDURE FM-CON-0180)

Working Range:	1.0 to 1,000 mg/L
Reporting Limit:	1.0 mg/L
Reporting Unit:	mg/L
Matrix:	Water and waste

1.0 Scope and Application

1.1 The method addresses the measurement of 1,1,2-trichloro-1,2,2-trifluoroethane (fluorocarbon-113) extractable matter from surface and saline waters and industrial and domestic aqueous wastes. It is applicable to the determination of hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases, and related matter.

1.2 Although the extraction procedure for the method is identical to that of gravimetric oil and grease, infrared detection permits the measurement of many relatively volatile hydrocarbons. Thus, the lighter petroleum distillates, with the exception of gasoline, may be measured with greater accuracy.

2.0 Method Summary

The sample is acidified to a low pH (< 2) and extracted with fluorocarbon-113. The concentration of oil and grease is determined by comparison of the infrared absorbance of the sample extract with standards.

OIL AND GREASE IN SLUDGE (PROCEDURE FM-CON-0182)

Working Linear Range: Gravimetric, infinite
Reporting Limit: 5.0 to 1000 mg/kg
Reporting Units: mg/Kg
Matrix: Sludge

1.0 Scope and Application

1.1 This method is applicable to the extraction and measurement of nonvolatile hydrocarbons from sludge samples.

1.2 This method is not applicable to the measurement of hydrocarbons that volatilize at temperatures below 70°C.

2.0 Method Summary

2.1 Sludge samples are mixed with distilled water under constant agitation to form a slurry. The slurry is acidified to low pH (<2).

2.2 Oils and greases are extracted into 1,1,2-trichloro-1,2,2-trifluoroethane (fluorocarbon-113). Sonication is used to promote phase separation if necessary.

2.3 A second extraction is performed on the aqueous phase and the two extractant fractions are combined.

2.4 The extractant is filtered to remove suspended solids, and the filtered fluorocarbon-113 is evaporated in a tared vessel. The residue is weighed to determine the weight of oil and grease

OIL AND GREASE (GRAVIMETRIC ONLY) (PROCEDURE FM-CON-0185)

Working Linear Range: Gravimetric: infinite
Reporting Limit: 5.0 to 1,000 mg/L
Reporting Unit: mg/L
Matrix: Water and wastes

1.0 Scope and Application

1.1 The method includes the measurement of 1,1,2-trichloro-1,2,2-trifluoroethane (fluorocarbon-113) extractable matter from surface and saline waters, and industrial and domestic aqueous wastes. It is applicable to the determination of relatively nonvolatile hydrocarbons, vegetable oils, animal fats, soaps, greases, and related matter.

1.2 The method is not applicable to measurement of light hydrocarbons that volatilize at temperatures below 70°C. Petroleum fuels from gasoline through No. 2 fuel oils are completely or partially lost in the solvent removal operation.

1.3 Some crude oils and heavy fuel oils contain a significant percentage of residue-type materials that are not soluble in fluorocarbon-113. Accordingly, recoveries of these materials will be low.

2.0 Method Summary

The sample is acidified to a low pH (< 2) and extracted with fluorocarbon-113. Oil and grease is determined by evaporating the solvent from the extract and weighing the residue.

PERCENT SOLIDS (MOISTURE) (PROCEDURE FM-CON-0190)

Working Linear Range: 0.1 to 100%
Reporting Limit: 0.1%
Reporting Units: %
Matrix: Sludge, soil, or sediment

1.0 Scope and Application

The method applies to the determination of the solids (or moisture) content in sludge, soil, or sediment samples.

2.0 Method Summary

An aliquot of sample is placed in a dried, preweighed evaporating dish, and the sample is weighed. The sample is then dried at 103° to 105°C and weighed, and the percentage of solids is determined.

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TOTAL PETROLEUM HYDROCARBONS—INFRARED (PROCEDURE FM-CON-0200)

Working Linear Range: 0.2 to 1,000 mg/L
Reporting Limit: Water, 0.2 mg/L
Reporting Units: Water, mg/L
Matrix: Water and waste

1.0 Scope and Application

1.1 The method is applicable for analysis of surface, saline, and ground waters for total recoverable hydrocarbons extracted by 1,1,2-trichloro,1,2,2-trifluoroethane (fluorocarbon-113) and analyzed by infrared spectrometry.

1.2 Infrared detection permits the measurement of many relatively volatile hydrocarbons, with the exception of gasoline.

2.0 Method Summary

The sample is acidified to a low pH (< 2) and serially extracted with fluorocarbon-113 in a separatory funnel. Interferences are removed with silica gel adsorbent. Infrared analysis of the extract is performed by direct comparison with standards.

TOTAL PETROLEUM HYDROCARBONS IN SEDIMENTS AND SLUDGE - INFRARED (PROCEDURE FM-CON-0202)

Working Linear Range: 0.2 to 1,000 mg/Kg
Reporting Limit: To be determined
Reporting Units: mg/Kg
Matrix: Soil, sediment, sludge

1.0 Scope and Application

1.1 This method is applicable to the extraction and measurement of petroleum hydrocarbons from soil, sediment, and sludge samples.

1.2 Infrared detection permits the measurement of many volatile hydrocarbons, with the exception of gasoline.

2.0 Method Summary

2.1 Sludge samples are mixed with distilled water under agitation to form a slurry. The slurry is acidified to low pH (<2).

2.2 Hydrocarbons are extracted into 1,1,2-trichloro-1,2,2-trifluoroethane (fluorocarbon-113) in a separatory funnel.

2.3 A second extraction is performed on the aqueous phase and the two extractant fractions are combined.

2.4 The extractant is filtered to remove suspended solids, and interferences are removed with silical gel adsorbent.

2.5 Infrared analysis of the extract is performed via direct comparison with standards.

TOTAL DISSOLVED SOLIDS (PROCEDURE FM-CON-0210)

Working Linear Range: 10 to 20,000 mg/L
Reporting Limits: 10 mg/L
Reporting Unit: mg/L
Matrix: Water

1.0 Scope and Application

The method measures filterable residue (total dissolved solids) in a water matrix. Filterable residue is defined as solids capable of passing through a glass fiber filter and dried to constant weight at 180°C.

2.0 Method Summary

A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180°C.

PHOSPHORUS ANALYSIS—SINGLE REAGENT METHOD (PROCEDURE FM-CON-0220)

Working Linear Range: 0.01 to 0.5 mg/L
Reporting Limit: 0.05 mg/L
Reporting Units: mg/L
Matrix: Water

1.0 Scope and Application

1.1 The method covers the determination of total phosphorus, orthophosphate, and total organic phosphorus in surface and ground waters. Phosphorus occurs in natural waters and in wastewaters almost solely as phosphates. These are classified as orthophosphates (PO_4), acid-hydrolyzable phosphates (pyro-, meta-, and other polyphosphates), and organically bound phosphates. Phosphates occur in solutions, in particles or detritus, or in the bodies of aquatic organisms.

1.2 Many forms of phosphorus can be quantified using analytical techniques; however, the method is concerned only with the measurement of total phosphorus, orthophosphate, and total organic phosphorus.

1.2.1 Total phosphorus is all phosphorus present in the sample, regardless of form, and will be measured using the persulfate digestion procedure.

1.2.2 Total orthophosphate is a measure of all the inorganic phosphorus [$(PO_4)^{-3}$] and is determined directly through colorimetry without digestion.

1.2.3 Total organic phosphorus is the inorganic and oxidizable organic phosphorus in the sample. It is calculated as total phosphorus minus hydrolyzable phosphorus and orthophosphate rather than measured directly. Hydrolyzable phosphorus must be determined to measure total organic phosphorus and is the phosphorus in the sample measured by the sulfuric acid hydrolysis procedure.

2.0 Method Summary

2.1 Phosphorus analyses usually consist of two steps:

- Conversion of the phosphorus form of interest to dissolved orthophosphate through hydrolysis or digestion
- Colorimetric determination of dissolved orthophosphate

2.2 Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phosphomolybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. Color intensity is proportional to the phosphorus concentration.

SURFACTANTS (MBAS) (PROCEDURE FM-CON-0250)

Working Linear Range:	Can be diluted for infinite range
Reporting Limit:	0.02 mg/L calculated as LAS
Reporting Units:	mg/L
Matrix:	Water

1.0 Scope and Application

The method is applicable to the measurement of all species of methylene blue active substances (MBAS) in surface, ground, and drinking waters. It cannot be used to measure specific species, and it is nonapplicable to saline waters.

2.0 Method Summary

The dye methylene blue in aqueous solutions reacts with anionic-type surface active materials to form a blue colored salt. The salt is extractable with chloroform, and the intensity of color produced is proportional to the concentration of MBAS.

PHENOLICS, TOTAL RECOVERABLE (COLORIMETRIC, AUTOMATED 4-AAP WITH DISTILLATION) (PROCEDURE FM-CON-0260)

Working Linear Range: 0.002 to 0.500 mg/L
Reporting Limit: 0.002 mg/L
Reporting Units: mg/L
Matrix: Water and waste

1.0 Scope and Application

1.1 The method is applicable to the analysis of drinking, ground, surface, and saline waters and domestic and industrial aqueous waste.

1.2 The method is capable of measuring phenolic materials from 2 to 500 µg/L in the aqueous phase using phenol as a standard. The working ranges are 2 to 200 µg/L and 10 to 500 µg/L, depending on the autoanalyzer instrument manifold used.

2.0 Method Summary

This automated method is based on the distillation of phenol and subsequent reaction of the distillate with alkaline ferricyanide ($K_3Fe(CN)_6$) and 4-aminoantipyrine (4-AAP) to form a red complex that is measured at 505 or 520 nm.

PHENOLICS, TOTAL RECOVERABLE (SPECTROPHOTOMETRIC, MANUAL 4-AAP WITH DISTILLATION) (PROCEDURE FM-CON-0270)

Working Linear Range: 1.0 to 250.0 µg/L
Reporting Limit: 5 µg/L
Reporting Units: µg/L
Matrix: Water and waste

1.0 Scope and Application

1.1 The method is applicable to the analysis of ground, drinking, surface, and saline waters and domestic and industrial aqueous waste.

1.2 The method is capable of measuring phenolic materials at the 5-µg/L level when the colored end product is extracted and concentrated in a solvent phase using phenol as a standard. It is also capable of measuring phenolic materials that contain more than 50 µg/L in the aqueous phase (without solvent extraction) using phenol as a standard.

1.3 It is not possible to use the method to differentiate between different kinds of phenols.

2.0 Method Summary

Phenolic materials react with 4-aminoantipyrine in the presence of potassium ferricyanide at a pH of 10 to form a stable, reddish-brown antipyrine dye. The amount of color produced is a function of the concentration of phenolic material.

SULFATE (COLORIMETRIC, AUTOMATED, METHYLTHYMOL BLUE) (PROCEDURE FM-CON-0280)

Working Linear Range: 1 to 300 mg/L
Reporting Limit: 1.0 mg/L
Reporting Units: mg/L
Matrix: Water and waste

1.0 Scope and Application

This automated method is applicable to drinking, surface, and ground waters and domestic and industrial aqueous waste.

2.0 Method Summary

Barium sulfate is formed by the reaction of the SO_4^{-2} with barium chloride ($BaCl_2$) at a low pH. At a high pH, excess barium reacts with methylthymol blue to produce a blue chelate. Uncomplexed methylthymol blue is gray. The amount of gray uncomplexed methylthymol blue indicates the concentration of sulfate.

SULFATE (TURBIDIMETRIC) (PROCEDURE FM-CON-0290)

Working Linear Range:	1.0 to 40.0 mg/L
Reporting Limit:	1.0 mg/L
Reporting Units:	mg/L
Matrix:	Water and wastes

1.0 Scope and Application

The method is applicable to drinking, surface, and ground waters and domestic and industrial aqueous wastes. It is suitable for all concentration ranges of sulfate (SO_4^{-2}); however, to obtain reliable readings, use a sample aliquot containing not more than 40 mg/L of SO_4^{-2}.

2.0 Method Summary

Sulfate ion is converted to a barium sulfate suspension under controlled conditions. The resulting turbidity is determined by a nephelometer, filter photometer, or spectrophotometer and compared with a curve prepared from standard sulfate solution.

FLUORIDE (PROCEDURE FM-CON-0300)

Working Linear Range: 0.1 to 1.4 mg/L F^-
Reporting Limit: 0.1 mg/L F^-
Reporting Units: mg/L F^-
Matrix: Water and wastes

1.0 Scope and Application

The method is applicable to the measurement of fluoride in ground, drinking, surface, and saline waters and domestic and industrial aqueous wastes.

2.0 Method Summary

The SPADNS colorimetric method is based on the reaction between fluoride and a zirconium-dye lake. Fluoride reacts with the dye lake, dissociating a portion of it into a colorless complex anion (ZrF_6^{2-}) and the dye. As the amount of fluoride increases, the color produced becomes progressively lighter.

TOTAL ORGANIC HALIDES (PROCEDURE FM-CON-0320)

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Working Linear Range: 5.0 to 1,000 µg/L
Reporting Limit: 5.0 µg/L
Reporting Units: µg/L
Matrix: Water

1.0 Scope and Application

1.1 The method is applicable to the measurement of total organic halides in ground, drinking, surface, and saline waters.

1.2 The presence of halogenated organic molecules is thought to be indicative of synthetic chemical contamination. There is no target compound list for this method; rather, the result is the sum of halogenated compounds present in the sample. Examples of halogenated compounds that contribute to the TOX result include:

- Trihalomethanes
- Organic solvents such as trichloroethylene and other halogenated alkanes and alkenes
- Chlorinated and brominated pesticides and herbicides
- Chlorinated aromatics such as hexachlorobenzene
- Polychlorinated biphenyls (PCBs)
- High molecular weight, partially chlorinated aquatic humic substances

1.3 The method measures only the total molar amount of dissolved organically bound halogen retained on the carbon adsorbent; it does not measure the molar amount of organically bound halogens on undissolved solids. The method yields no information about the structure or nature of the organic compounds to which the halogens are bound or about the individual halogens present. It is sensitive to organically bound chloride, bromide, and iodide but does not detect fluorinated organics.

2.0 Method Summary

2.1 The method comprises four major steps.

2.1.1 Dissolved organic material is separated from inorganic halides and concentrated from aqueous solution by adsorption onto activated carbon.

2.1.2 Inorganic halides present on the activated carbon are removed by competitive displacement by nitrate ions.

2.1.3 The activated carbon with adsorbed organic material is introduced into a furnace that pyrolyzes organic carbon to carbon dioxide and the bound halogens to hydrogen halides (HX).

2.1.4 The HX is transported in a carrier gas stream to a micro-coulometric titration cell where the amount of halide is quantified by measuring the current produced by silver-ion precipitation of the halides.

2.2 As mentioned above, there is no specific target analyte as this is a qualitative measure of dissolved halogenated organic material. The practical range of the determination is 5.0 to 1,000 µg/L.

COLOR (PROCEDURE FM-CON-0330)

Working Linear Range: With dilution, range is infinite.
Reporting Limit: 5 units
Reporting Units: Color units
Matrix: Water

1.0 Scope and Application

The method is applicable to the measurement of water color derived from naturally occurring materials such as vegetable residues (leaves, bark, roots, humus, and peat materials) in ground, surface, and drinking water samples. It is not applicable to color measurements on waters containing highly colored industrial wastes.

2.0 Method Summary

The water sample is placed in a Nessler tube and visually compared to color standards in similar tubes. If the color exceeds the standard range, the sample may be diluted. **Note:** The method is pH-dependent.

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OXIDATION-REDUCTION POTENTIAL (PROCEDURE FM-CON-0340)

Working Linear Range: Instrument-dependent
Reporting Limits: Instrument-dependent
Reporting Units: mV
Matrix: Water and wastes

1.0 Scope and Application

The method is applicable to drinking, surface, ground, and saline waters and domestic and industrial aqueous wastes.

2.0 Method Summary

Oxidation-reduction potential (ORP) is defined as the electromotive force developed by a platinum electrode immersed in a water sample relative to a standard reference electrode of known Eh. The obtained value is a crude estimate of the oxygen status of the sample. A positive value indicates that the water sample is in an oxidized state or that oxygen is present. A negative value would indicate an absence of oxygen or reducing conditions.

3298

ASBESTOS PARTICULATES IN AIR SAMPLES (PROCEDURE FM-CON-0440)

Working Linear Range: 100 to 1300 fibers/mm^2 filter area
Reporting Limit: 7 Fibers/mm^2 filter area
Reporting Units: Fibers/mm^2 or fibers/cc
Matrix: Air

1.0 Scope and Application

1.1 The phase contrast microscopy method (NIOSH 7400) is applicable to a range of 0.02 fibers per cubic centimeter (f/cc) of air (1920-liter [L] air sample) to 1.25 f/cc (400-L air sample). This method does not differentiate between asbestos, tremolite, anthophyllite, actinolite, or other fibers such as fibrous glass. Asbestos, tremolite, anthophyllite, and actinolite fibers less than 0.25 µm diameter will not be detected by this method. Any airborne fiber may interfere since all particles meeting the counting criteria are counted.

1.2 The transmission electron microscopy method (NIOSH 7402) is applicable to a range of 0.04 to 0.5 fibers/cc for a 1000-L air sample. This method is used to determine asbestos fibers in the optically visible range and is intended to compliment the results obtained by phase contrast microscopy. High concentrations of background dust interfere with fiber identification. Some amphibole and non-amphibole particles have elemental compositions similar to asbestos minerals and may interfere with the transmission electron microscopy.

2.0 Method Summary

2.1 Air sample filters are prepared for phase contrast microscopy analysis using the acetone/triacetin method or a method with an equivalent index of refraction and similar clarity. Fiber counts are made by only counting fibers equal to or longer than 5 micrometers and have a length-to-width ratio (aspect ratio) of 3:1 or greater. Each set of samples taken will include 10 percent blanks or a minimum of 2 blanks. The blank results shall be averaged and subtracted from the analytical results before reporting.

2.2 Air sample filters are prepared for transmission electron microscopy analysis using acetone method, the "hot block" clearing technique, or the DMF clearing technique. Fiber counts are made using the guideline of phase contrast microscopy. However, higher magnification is used to determine fiber dimensions and countability under the acceptable criteria. A minimum of 10 percent of the fibers, and at least 3 asbestos fibers are analyzed by energy-dispersive X-ray and selected area electron diffraction to confirm the presence of asbestos. Two field blanks per sample set shall be counted to document possible contamination of samples.

3298

ASBESTOS IN HETEROGENEOUS WASTE (PROCEDURE FM-CON-0442)

Working Linear Range:	1% to 100% asbestos
Reporting Limit:	≤1% of asbestos content of projected sample area
Reporting Units:	Percentage
Matrix:	Solid waste material

1.0 Scope and Application

The polarized light microscopy method (NIOSH 9002) is applicable to a range of 1-100 percent asbestos perceived by the analyst in comparison to standard area projections. This method is not applicable to samples containing large amounts of fine fibers below the resolution of the light microscope. Fibers with optical properties similar to asbestos minerals may give positive interferences. Asbestos fibers may not be detected if fibers are coated, fibers are finer than 0.3 µm, or the index of refraction of asbestos is altered by the heat acid treatment.

2.0 Method Summary

Collected waste samples are prepared for polarized light microscopy by placing a few drops of refractive index liquids on a small portion of a sample in a slide. The slide is scanned to identify any asbestos minerals using the optical properties of morphology, refractive indices, color, and dispersion staining characteristics.

3298

ISOTOPIC PLUTONIUM IN WATER BY ALPHA SPECTROMETRY (PROCEDURE FM-RAD-0010)

Working Linear Range: Greater than 0.02 pCi/L, infinite with dilution
Reporting Limit: ~0.02 pCi/L
Reporting Units: pCi/L
Matrix: Water

1.0 Scope and Application

1.1 This test method covers the determination of plutonium isotopes Pu-239/240 and Pu-238 in water by means of chemical separations and alpha spectrometry. The test method applies to soluble plutonium and to suspended particulate matter containing plutonium. In the latter situation, an acid dissolution step is required to assure that all of the plutonium dissolves. The nominal sensitivity may vary with each analysis but is approximately 0.02 pCi/L.

1.2 This analytical method applies to Analytical Support Levels C and D.

2.0 Method Summary

2.1 Plutonium-242 is added as a tracer before any chemical separations are performed. Iron is added to the water sample as iron (III), and the plutonium is coprecipitated with the iron as ferric hydroxide. After decantation and centrifugation, the ferric hydroxide precipitate containing the coprecipitated plutonium is dissolved, and the solution is adjusted to 8 M in HNO_3 for anion exchange separation. When the sample fails to dissolve because of the presence of insoluble residue, the residue is treated by a rigorous acid dissolution using concentrated nitric and hydrofluoric acids.

2.2 After an anion exchange separation, the plutonium is electrodeposited onto a stainless steel disk for counting by alpha pulse height analysis using a silicon surface barrier detector. From the recovery of the Pu-242 tracer, the absolute activities of Pu-238 and Pu-239/240 can be calculated.

ISOTOPIC PLUTONIUM IN ORGANIC SOLIDS (PROCEDURE FM-RAD-0011)

Working Linear Range: Infinite with dilution
Reporting Limit: To be determined
Reporting Units: pCi/g
Matrix: Organic and high-organic solids

1.0 Scope and Application

1.1 This test method covers the determination of plutonium isotopes Pu-239/240 and Pu-238 in dry-ashed residues of organic samples by chemical separations and alpha spectrometry. The nominal sensitivity may vary with each analysis but is approximately 0.02 pCi/L.

1.2 This analytical method applies to Analytical Support Levels C and D.

2.0 Method Summary

2.1 If necessary, the sample is granulated to increase the surface to volume ratio. Plutonium-242 is added as a tracer and the sample is dry ashed to remove organics. The residue is dissolved in concentrated nitric acid followed by concentrated hydrochloric acid. Any residue not dissolved is separated by filtration and further decomposed by hydrofluoric acid, dried, and dissolved in 8 M HNO3. The combined sample is adjusted to 8 M in HNO3 for anion exchange separation.

2.2 After an anion exchange separation, the plutonium is electrodeposited onto a stainless steel disk for counting by alpha pulse height analysis using a silicon surface barrier detector. From the recovery of the Pu-242 tracer, the absolute activities of Pu-238 and Pu-239/240 can be calculated.

ISOTOPIC PLUTONIUM IN INORGANIC SOLIDS (PROCEDURE FM-RAD-0012)

Working Linear Range: To be determined, probably .1 g to 100 g
Reporting Limit: To Be determined
Reporting Units: pCi/g
Matrix: Inorganic solids

1.0 Scope and Application

This method is applicable to the determination of Plutonium-238 (Pu-238) and Pu-239/240 in samples requiring alpha isotopic analyses in an inorganic solid matrix.

2.0 Method Summary

2.1 This procedure provides for the separation of plutonium alpha-emitting radionuclides by elution from anion/cation exchange resin columns. Separation is based on the differences in absorption and chemical properties of the radionuclides of interest.

2.2 An appropriate weight of sample is aliquoted for analysis. The appropriate yield tracers (Pu-236 or Pu-242) and carriers are added to the sample. As appropriate, the sample is dried and/or muffled in a furnace to ash. The aliquot is dissolved with a series of additions of nitric acid (HNO_3) and hydrofluoric acid (HF) and evaporation to dryness. Finally the sample is microwaved with HNO_3 and HF. This solution is converted to the nitrate form prior to loading on the ion exchange columns.

2.3 Thorium (Th), Pu, and neptunium are separated from americium, curium and uranium by use of a nitrate form anion column. From this column, Th is eluted as chloride followed by Pu with hydrochloric acid and an iodide solution.

2.4 The Pu fraction is mounted on filter paper by use of a neodymium coprecipitation technique. Filters are counted by using surface barrier detectors to determine alpha isotopic activities.

ISOTOPIC PLUTONIUM IN ORGANIC SOLIDS (PROCEDURE FM-RAD-0014)

Working Linear Range: To be determined, probably .1 g to 10 g
Reporting Limit: To be determined
Reporting Units: pCi/g
Matrix: Inorganic solids

1.0 Scope and Application

This method is applicable to the determination of Plutonium-238 (Pu-238), and Pu-239/240 in samples requiring alpha isotopic analyses in an organic solid matrix.

2.0 Method Summary

2.1 This procedure provides for the separation of plutonium alpha-emitting radionuclides by elution from anion/cation exchange resin columns. Separation is based on the differences in absorption and chemical properties of the radionuclides of interest.

2.2 An appropriate weight of sample is aliquoted for analysis. The appropriate yield tracers (Pu-236 or Pu-242) and carriers are added to the sample. As appropriate, the sample is dried and/or muffled in a furnace to ash. The exact muffling sequence and amount of sample must be determined on an individual matrix basis to prevent fires and/or explosions. The extent of removal of organic material will directly bear upon the recovery of Pu in the separation procedure. The aliquot is dissolved with a series of additions of nitric acid (HNO_3) and hydrofluoric acid (HF) and evaporation to dryness. Finally the sample is microwaved with HNO_3 and HF. This solution is converted to the nitrate form prior to loading on the ion exchange columns.

2.3 Thorium, Pu, and neptunium are separated from americium, curium and uranium by use of a nitrate form anion column. From this column, Th is eluted as chloride followed by Pu with hydrochloric acid and an iodide solution.

2.4 The Pu fraction is mounted on filter paper by use of a neodymium coprecipitation technique. Filters are counted by using surface barrier detectors to determine alpha isotopic activities.

ISOTOPIC PLUTONIUM IN PARTICULATE AIR SAMPLES (PROCEDURE FM-RAD-0016)

Working Linear Range:	To be determined
Reporting Limit:	To be determined by number of filters and volume
Reporting Units:	pCi/filter, pCi/mL, pCi/L, pCi/cc
Matrix:	Filter(s)

1.0 Scope and Application

This method is applicable to the determination of Plutonium-238 (Pu-238), and Pu-239/240 in samples requiring alpha isotopic analyses on one or more air particulate filter(s).

2.0 Method Summary

2.1 This procedure provides for the separation of plutonium alpha-emitting radionuclides by elution from anion/cation exchange resin columns. Separation is based on the differences in absorption and chemical properties of the radionuclides of interest.

2.2 One or more air particulate filter(s) are selected for analysis. The sample is placed in a beaker and muffled in a programmable furnace. If glass fiber filters were used, dissolve the residue in hydrofluoric acid and evaporate to dryness. Repeat several times. Dissolve this residue or the ash residue from the muffle furnace for regular filters in a series of HNO_3 additions. The appropriate yield tracers (Pu-236 or Pu-242) and carriers are added to the sample. This solution is converted to the nitrate form prior to loading on the ion exchange columns.

2.3 Thorium, Pu, and neptunium are separated from americium, curium, and uranium by use of a nitrate form anion column. From this column, Th is eluted as chloride followed by Pu with hydrochloric acid and an iodide solution.

2.4 The Pu fraction is mounted on filter paper by use of a neodymium coprecipitation technique. Filters are counted by using surface barrier detectors to determine alpha isotopic activities.

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PLUTONIUM-241 IN AQUEOUS MEDIA (PROCEDURE FM-RAD-0020)

Working Linear Range: With dilution .1 pCi/L to 10000 pCi/L
Reporting Limit: To be determined
Reporting Units: pCi/L
Matrix: Aqueous liquids, water, surface water, ground water

1.0 Scope and Application

This method is applicable to the determination of Pu-241 following alpha isotopic determination of Pu-238, and Pu-239/240 by Method FM-RAD-0030. The samples contain aqueous liquids or water. If alpha isotopic analysis is not needed, Method FM-RAD-0030 should be followed through the elution of the Pu fraction.

2.0 Method Summary

2.1 This procedure provides for the determination of plutonium-241 following the alpha isotopic determination of plutonium. The determination is based on dissolving the isotopic filter and measuring the plutonium beta by use of a liquid scintillation counting technique.

2.2 After final approval of the alpha isotopic determination of plutonium, the filter is dissolved. Place the filter in a beaker and ash in a muffle furnace for 8 hours at 525° C. Add nitric acid (HNO_3) to the ash and add perchloric acid. Evaporate to dryness and add HNO_3. Repeat the HNO_3 and evaporation steps several times. Finally dissolve in 1 mL of dilute HNO_3, dilute to 5 mL with water, and transfer to liquid scintillation cocktail for counting. An efficiency spike and Pu-241 standard should be prepared and counted with the sample or batch of samples.

2.3 If Method FM-RAD-0030 has been followed and isotopic determination is not required, evaporate the Pu fraction to dryness and follow the above steps following the muffling sequence.

3298

PLUTONIUM-241 IN INORGANIC SOLIDS (PROCEDURE FM-RAD-0022)

Working Linear Range: To be determined, probably .1 g to 100 g
Reporting Limit: To be determined
Reporting Units: pCi/g
Matrix: Inorganic solids

1.0 Scope and Application

This method is applicable to the determination of Plutonium-241 (Pu-241) following alpha isotopic determination of Pu-238 and Pu-239/240 by Method FM-RAD-0012. The samples are in an inorganic solid matrix. If alpha isotopic analysis is not needed, Method FM-RAD-0012 should be followed through the elution of the Pu fraction.

2.0 Method Summary

2.1 This procedure provides for the determination of Pu-241 following the alpha isotopic determination of plutonium. The determination is based on dissolving the isotopic filter and measuring the plutonium beta by use of a liquid scintillation counting technique.

2.2 After final approval of the alpha isotopic determination of plutonium, the filter is dissolved. Place the filter in a beaker and ash in a muffle furnace for eight hours at 525° C. Add nitric acid (HNO_3) to the ash and add perchloric acid. Evaporate to dryness and add HNO_3. Repeat the HNO_3 and evaporation steps several times. Finally dissolve in 1 mL of dilute HNO_3, dilute to 5 mL with water, and transfer to liquid scintillation cocktail for counting. An efficiency spike and Pu-241 standard should be prepared and counted with the sample or batch of samples.

2.3 If Method FM-RAD-0012 has been followed and isotopic determination is not required, evaporate the Pu fraction to dryness and follow the above steps following the muffling sequence.

PLUTONIUM-241 IN ORGANIC SOLIDS (PROCEDURE FM-RAD-0024)

Working Linear Range: To be determined, probably .1 g to 10 g
Reporting Limit: To be determined
Reporting Units: pCi/g
Matrix: Organic solids

1.0 Scope and Application

This method is applicable to the determination of Plutonium-241 (Pu-241) following alpha isotopic determination of Pu-238 and Pu-239/240 by Method FM-RAD-0014. The samples are in an organic solid matrix. If alpha isotopic analysis is not needed, Method FM-RAD-0014 should be followed through the elution of the Pu fraction.

2.0 Method Summary

2.1 This procedure provides for the determination of Pu-241 following the alpha isotopic determination of plutonium. The determination is based on dissolving the isotopic filter and measuring the plutonium beta by use of a liquid scintillation counting technique.

2.2 After final approval of the alpha isotopic determination of plutonium, the filter is dissolved. Place the filter in a beaker and ash in a muffle furnace for eight hours at 525° C. Add nitric acid (HNO_3) to the ash and add perchloric acid. Evaporate to dryness and add HNO_3. Repeat the HNO_3 and evaporation steps several times. Finally dissolve in 1 mL of dilute HNO_3, dilute to 5 mL with water, and transfer to liquid scintillation cocktail for counting. An efficiency spike and Pu-241 standard should be prepared and counted with the sample or batch of samples.

2.3 If Method FM-RAD-0014 has been followed and isotopic determination is not required, evaporate the Pu fraction to dryness and follow the above steps following the muffling sequence.

3298

PLUTONIUM-241 IN PARTICULATE AIR SAMPLES (PROCEDURE FM-RAD-0026)

Working Linear Range: To be determined
Reporting Limit: To be determined by number of filters and volume
Reporting Units: pCi/filter, pCi/mL, pCi/L, pCi/cc
Matrix: Filter(s)

1.0 Scope and Application

This method is applicable to the determination of Plutonium-241 (Pu-241) following alpha isotopic determination of Pu-238 and Pu-239/240 by Method FM-RAD-0014. The samples are one or more air particulate filter(s). If alpha isotopic analysis is not needed, Method FM-RAD-0016 should be followed through the elution of the Pu fraction.

2.0 Method Summary

2.1 This procedure provides for the determination of Pu-241 following the alpha isotopic determination of plutonium. The determination is based on dissolving the isotopic filter and measuring the plutonium beta by use of a liquid scintillation counting technique.

2.2 After final approval of the alpha isotopic determination of plutonium, the filter is dissolved. Place the filter in a beaker and ash in a muffle furnace for eight hours at 525° C. Add nitric acid (HNO_3) to the ash and add perchloric acid. Evaporate to dryness and add HNO_3. Repeat the HNO_3 and evaporation steps several times. Finally dissolve in 1 mL of dilute HNO_3, dilute to 5 mL with water, and transfer to liquid scintillation cocktail for counting. An efficiency spike and Pu-241 standard should be prepared and counted with the sample or batch of samples.

2.3 If Method FM-RAD-0016 has been followed and isotopic determination is not required, evaporate the Pu fraction to dryness and follow the above steps following the muffling sequence.

CERENKOV SCREENING FOR HIGH ENERGY BETA-EMITTING NUCLIDES IN LIQUIDS (PROCEDURE FM-RAD-0027)

Working Linear Range:	Nuclide and Matrix Specific
Reporting Limit:	Nuclide and Matrix Specific
Reporting Units:	pCi/L
Matrix:	Translucent Liquids

1.0 Scope and Application

This method screens for high-energy beta-emitting nuclides in translucent liquids. Sufficiently low count rates demonstrate the absence of various hard beta emitters above release levels, and indicate that further analyses are not required.

2.0 Method Summary

2.1 A 20 ml aliquot of the liquid is placed directly a 20 ml plastic scintillation vial. Cerenkov radiation produced in the liquid by energetic beta particles is detected when the vial is counted in commercial liquid scintillation system. No reagents or sample preparation is required.

2.2 Detection efficiency is greater than 50% for Sr-90/Y-90.

3298

BETA-EMITTING NUCLIDES IN ORGANIC LIQUIDS (PROCEDURE FM-RAD-0028)

Working Linear Range: Nuclide and Matrix Specific
Reporting Limit: Nuclide and Matrix Specific
Reporting Units: pCi/L
Matrix: Organic Liquids

1.0 Scope and Application

1.1 This method is applicable to direct screening for beta-emitting nuclides in organic liquids compatible with liquid scintillators. Sufficiently low count rates in specified beta energy regions demonstrate the absence of various beta emitters above release levels, and indicate that further analyses are not required.

1.2 The compatibility of the matrix with the liquid scintillator is determined automatically by a quench indicating system.

2.0 Method Summary

2.1 An aliquot of the matrix is added directly to an "Environmentally Safe" commercial liquid scintillation solution in a 20 ml scintillation vial. The vial is counted in a commercial liquid scintillation system.

2.2 For best results, a counting system with alpha/beta pulse shape discrimination and energy spectrum display should be used.

3298

BETA-EMITTING NUCLIDES IN AQUEOUS LIQUIDS (PROCEDURE FM-RAD-0029)

Working Linear Range: Nuclide and Matrix Specific
Reporting Limit: Nuclide and Matrix Specific
Reporting Units: pCi/L
Matrix: Aqueous Liquids

1.0 Scope and Application

1.1 This method is applicable to screening for beta-emitting nuclides in aqueous liquids compatible with aqueous-accepting liquid scintillators. Sufficiently low count rates in specified beta energy regions demonstrate the absence of various beta emitters above release levels, and indicate that further analyses are not required.

1.2 The compatibility of the matrix with the liquid scintillator is determined automatically by a quench indicating system.

2.0 Method Summary

2.1 An aliquot of the matrix is added directly to an "Environmentally Safe" aqueous-accepting commercial liquid scintillation solution in a 20 ml scintillation vial. The vial is counted in a commercial liquid scintillation system.

2.2 For best results, a counting system with alpha/beta pulse shape discrimination and energy spectrum display should be used.

3298

SELECTED ALPHA EMITTING NUCLIDES IN WATER (PROCEDURE FM-RAD-0030)

Working Linear Range: With dilution: .1 pCi/L to 10000 pCi/L
Reporting Limit: To be determined
Reporting Units: pCi/L
Matrix: Water, aqueous liquids, ground water, surface water

1.0 Scope and Application

This method is applicable to the determination of uranium-233/234 (U-233/234), U-235/236, U-238, thorium-228 (Th-228), Th-230, Th-232, neptunium-237 (Np-237), plutonium-238 (Pu-238), Pu-239/240, americium-241 (Am-241), Am-243, curium-242 (Cm-242), and Cm-244 in samples requiring alpha isotopic analyses in an aqueous matrix with no suspended solids.

2.0 Method Summary

2.1 This procedure provides for the sequential separation of various alpha emitting radionuclides by elution from anion/cation exchange resin columns. Separation is based on the differences in absorption and chemical properties of the radionuclides of interest.

2.2 An appropriate amount of sample is aliquoted for analysis. The appropriate yield tracers and carriers are added to the sample. The alpha emitters are precipitated from solution by calcium phosphate. After precipitation is complete, the solid material is washed and the supernate is decanted. The precipitate is dissolved in nitric acid and the elements of interest are converted to the nitrate form prior to loading on the ion exchange column.

2.3 Th, Pu, and Np are separated from Am, Cm and U by use of a nitrate form anion column. From this column Th, is eluted as chloride followed by Pu with hydrochloric acid and an iodide solution. Np is finally removed by a chloride hydrofluoric acid elution. U is separated from the Am/Cm fraction by use of a chloride form of an anion resin. The U is eluted from this column as a dilute chloride solution. Individual fractions are processed to produce filters for counting.

2.4 Samples are mounted on filter paper by use of a neodymium coprecipitation technique. Filters are counted by using surface barrier detectors to determine alpha isotopic activities.

3298

SELECTED ALPHA EMITTING NUCLIDES IN INORGANIC SOLIDS (PROCEDURE FM-RAD-0032)

Working Linear Range: To be determined, probably .1 g to 100 g
Reporting Limit: To be determined
Reporting Units: pCi/g
Matrix: Inorganic solids

1.0 Scope and Application

This method is applicable to the determination of uranium-233/234 (U-233/234), U-235/236, U-238, thorium-228 (Th-228), Th-230, Th-232, neptunium-237 (Np-237), plutonium-238 (Pu-238), Pu-239/240, americium-241 (Am-241), Am-243, curium-242 (Cm-242), and Cm-244 in samples requiring alpha isotopic analyses in an inorganic solid matrix.

2.0 Method Summary

2.1 This procedure provides for the sequential separation of various alpha emitting radionuclides by elution from anion/cation exchange resin columns. Separation is based on the differences in absorption and chemical properties of the radionuclides of interest.

2.2 An appropriate weight of sample is aliquoted for analysis. The appropriate yield tracers and carriers are added to the sample. As appropriate, the sample is dried and or muffled in a furnace to ash. The aliquot is dissolved with a series of additions of nitric acid and hydrofluoric acid and evaporation to dryness. Finally the sample is microwaved with HNO_3 and HF. This solution is converted to the nitrate form prior to loading on the ion exchange columns.

2.3 Th, Pu, and Np are separated from Am, Cm and U by use of a nitrate form anion column. From this column, Th is eluted as chloride followed by Pu with hydrochloric acid and an iodide solution. Np is finally removed by a chloride hydrofluoric acid elution. U is separated from the Am/Cm fraction by use of a chloride form of an anion resin. The U is eluted from this column as a dilute chloride solution. Individual fractions are processed to produce filters for counting.

2.4 Samples are mounted on filter paper by use of a neodymium coprecipitation technique. Filters are counted by using surface barrier detectors to determine alpha isotopic activities.

SELECTED ALPHA EMITTING NUCLIDES IN ORGANIC SOLIDS (PROCEDURE FM-RAD-0034)

Working Linear Range: To be determined, probably .1 g to 10 g
Reporting Limit: To be determined
Reporting Units: pCi/g
Matrix: Organic solids

1.0 Scope and Application

This method is applicable to the determination of uranium-233/234 (U-233/234), U-235/236, U-238, thorium-228 (Th-228), Th-230, Th-232, neptunium-237 (Np-237), plutonium-238 (Pu-238), Pu-239/240, americium-241 (Am-241), Am-243, curium-242 (Cm-242), and Cm-244 in samples requiring alpha isotopic analyses in an organic solid matrix.

2.0 Method Summary

2.1 This procedure provides for the sequential separation of various alpha emitting radionuclides by elution from anion/cation exchange resin columns. Separation is based on the differences in absorption and chemical properties of the radionuclides of interest.

2.2 An appropriate weight of sample is aliquoted for analysis. The appropriate yield tracers and carriers are added to the sample. As appropriate, the sample is dried and/or muffled in a furnace to ash. The exact muffling sequence and amount of sample must be determined on an individual matrix basis to prevent fires and/or explosions. The extent of removal of organic material will directly bear upon the recovery of the radionuclides. The aliquot is dissolved with a series of additions of nitric acid (HNO_3) and hydrofluoric acid (HF) and evaporation to dryness. Finally the sample is microwaved with HNO_3 and HF. This solution is converted to the nitrate form prior to loading on the ion exchange columns.

2.3 Th, Pu, and Np are separated from Am,Cm and U by use of a nitrate form anion column. From this column Th is eluted as chloride, followed by the Pu with hydrochloric acid and an iodide solution. Np is finally removed by a chloride HF elution. U is separated from the Am/Cm fraction by use of a chloride form of an anion resin. The U is eluted from this column as a dilute chloride solution. Individual fractions are processed to produce filters for counting.

2.4 Samples are mounted on filter paper by use of a neodymium coprecipitation technique. Filters are counted by using surface barrier detectors to determine alpha isotopic activities.

SELECTED ALPHA EMITTING NUCLIDES IN AIR PARTICULATE SAMPLES (PROCEDURE FM-RAD-0036)

Working Linear Range: To be determined
Reporting Limit: To be determined by number of filters and volume
Reporting Units: pCi/filter, pCi/mL, pCi/L, pCi/cc
Matrix: Filter

1.0 Scope and Application

This method is applicable to the determination of uranium-233/234 (U-233/234), U-235/236, U-238, thorium-228 (Th-228), Th-230, Th-232, neptunium-237 (Np-237), plutonium-238 (Pu-238), Pu-239/240, americium-241 (Am-241), Am-243, curium-242 (Cm-242), and Cm-244 in samples requiring alpha isotopic analyses on one or more air particulate filter(s).

2.0 Method Summary

2.1 This procedure provides for the sequential separation of various alpha-emitting radionuclides by elution from anion/cation exchange resin columns. Separation is based on the differences in absorption and chemical properties of the radionuclides of interest.

2.2 One or more air particulate filter(s) are selected for analysis. The sample is placed in a beaker and muffled in a programmable furnace. If glass fiber filters are used, dissolve the residue in hydrofluoric acid (HF) and evaporate. Repeat several times. Dissolve the residue from either the HF step or the muffle step in a series of HNO_3 additions. The appropriate yield tracers and carriers are added. This solution is in the nitrate form and ready for loading on the ion exchange columns.

2.3 Th, Pu, and Np are separated from Am, Cm and U by use of a nitrate form anion column. From this column, Th is eluted as chloride followed by Pu with hydrochloric acid and an iodide solution. Np is finally removed by a chloride HF elution. U is separated from the Am/Cm fraction by use of a chloride form of an anion resin. The U is eluted from this column as a dilute chloride solution. Individual fractions are processed to produce filters for counting.

2.4 Samples are mounted on filter paper by use of a neodymium coprecipitation technique. Filters are counted by using surface barrier detectors to determine alpha isotopic activities.

RADIUM-228 IN WATER AND AIR FILTERS BY BETA COUNTING (PROCEDURE FM-RAD-0040)

Working Linear Range: Greater than 1 pCi/L, infinite with dilution
Reporting Limit: ~1 pCi/L Water; TBD for Air Filters
Reporting Units: Water, pCi/L; Air filters, pCi/air filter
Matricies: Water, air filters

1.0 Scope and Application

1.1 The method covers the determination of radium-228 (Ra-228) in water and air filters. The nominal sensitivity of the procedure may vary with each analysis but is approximately 1 pCi/L for water. The nominal sensitivity that may be obtained by this exact method for air filters requires additional performance data.

1.2 This analytical method applies to Analytical Support Levels C and D.

2.0 Method Summary

2.1 The radium in the sample is coprecipitated as radium-barium sulfate. The precipitate is dissolved in a pentasodium diethylenetriamine pentaacetate solution.

2.2 Radium-228, a weak beta emitter, decays to actinium-228 (Ac-228), which is allowed to ingrow for at least 36 hours. The Ac-228 is then extracted into di-2-ethylhexylphosphoric acid and back-extracted with nitric acid.

2.3 The Ac-228 is beta counted in a low background proportional counter and its measured activity is used to calculate the Ra-228 concentration of the original sample.

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RADIUM-226 IN WATER BY EMANATION/SCINTILLATION COUNTING (PROCEDURE FM-RAD-0050)

Working Linear Range: Greater than 0.1 pCi/L, infinite with dilution
Reporting Limit: ~0.1 pCi/L
Reporting Units: pCi/L
Matrix: Water

1.0 Scope and Application

1.1 The method is used for the determination of low-level radium-226 activity in water. The nominal sensitivity of the procedure may vary with each analysis but is approximately 0.1 pCi/L in water.

1.2 This analytical method applies to Analytical Support Levels C and D.

2.0 Method Summary

2.1 The method is based on the emanation and scintillation counting of the radon-222 (Rn-222) progeny of radium-226 (Ra-226). It is made specific for Ra-226 by allowing the shorter-lived radon progeny of other radium isotopes to decay before counting.

2.2 Radium is coprecipitated with barium sulfate and the resulting barium-radium sulfate is decomposed with phosphoric acid. The glassy melt product is dissolved to form soluble barium-radium salts. The salts are dissolved and the solution stored to allow the Rn-222 to ingrow. After the ingrowth period, the radon gas is purged from the solution, collected in a counting cell, and counted after a 4-hour wait. Radium-226 yield is determined by using a Ba-133 tracer.

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RADIUM-226 IN SOIL/SEDIMENT, MILK, AND AIR FILTERS BY EMANATION/SCINTILLATION COUNTING (PROCEDURE FM-RAD-0060)

Working Linear Range: Infinite with dilution
Reporting Limit: ~0.1 pCi/L Milk, ~0.04 pCi/g Solids, TBD Air Filters
Reporting Units: Milk, pCi/L; Solids, pCi/g; Air filters, pCi/air filter
Matricies: Milk, soil/sediment, air filters

1.0 Scope and Application

1.1 The method is used for the determination of low-level radium-226 activity in soil/sediment, milk, and air filters. The technique may be applied to almost any material that can be converted to a homogeneous solution. The nominal sensitivity of the procedure may vary with each analysis but is approximately 0.04 pCi/g for soil/sediment and 0.1 pCi/L for milk. The nominal sensitivity that may be obtained by this exact method for air filters needs performance data.

1.2 This analytical method applies to Analytical Support Levels C and D.

2.0 Method Summary

2.1 The method is based on the emanation and scintillation counting of the radon-222 (Rn-222) progeny of radium-226 (Ra-226). It is made specific for Ra-226 by allowing the shorter-lived radon progeny of other radium isotopes to decay before counting.

2.2 After sample pretreatment, most interferences are removed by successive fuming nitric acid separations. Radium is then coprecipitated with barium chromate and dissolved in perchloric acid and water. The solution is stored to allow the Rn-222 to ingrow. After the ingrowth period, the radon gas is purged from the solution, collected in a counting cell, and counted after a 4-hour wait. Radium-226 yield is determined by using a Ba-133 tracer.

2.3 The method differs for the sample matrices only in the sample preparation steps.

LEAD-210 IN WATER BY BETA COUNTING (PROCEDURE FM-RAD-0070)

Working Linear Range: Greater than 7 pCi/L, infinite with dilution
Reporting Limit: ~7 pCi/L
Reporting Units: Water, pCi/L
Matrix: Water

1.0 Scope and Application

1.1 The method is applicable to the analysis of Pb-210 in water. The nominal sensitivity of the procedure may vary with each analysis but is approximately 7 pCi/L for water.

1.2 The method applies to Analytical Support Levels C and D.

2.0 Method Summary

2.1 Bismuth-210 is counted to determine the amount of Pb-210 present in the sample.

2.2 Lead carrier is added to a water sample. The lead is separated as a lead bromide complex by solvent extraction with methyltricaprylammonium chloride in toluene. Bismuth carrier is added, and the sample preparation is stored to allow the ingrowth of Bi-210. The Bi-210 progeny is separated from the Pb-210 parent by precipitation as bismuth oxychloride and is measured using a low background beta counter. A simplified flow chart is shown in Figure 1. Chemical yields are determined by atomic absorption for lead and gravimetrically for bismuth. A calibration standard and reagent blank are processed concurrently with the samples.

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LEAD-210 IN INORGANIC SOLIDS (PROCEDURE FM-RAD-0072)

Working Linear Range: To be determined, probably .1 g to 100 g
Reporting Limit: To be determined
Reporting Units: pCi/g
Matrix: Inorganic solids

1.0 Scope and Application

This method is applicable to the determination of lead-210 (Pb-210) in samples requiring Pb-210 analysis in an inorganic matrix.

2.0 Method Summary

2.1 This procedure provides for the separation of Pb-210 beta emitting nuclides by elution from anion exchange resin columns. Separation is based on the differences in absorption and chemical properties of Pb-210 and contaminants.

2.2 An appropriate weight of sample is aliquoted for analysis. Stable lead carrier is added to the sample. As appropriate, the sample is dried and/or muffled to ash in a furnace. The exact muffling sequence and amount of sample must be determined on an individual matrix basis. The aliquot is dissolved with a series of additions of nitric acid (HNO_3) and hydrofluoric acid (HF) and evaporated to dryness. The sample is microwaved with HNO_3 and HF. The sample is converted to a chloride form prior to loading onto ion exchange columns.

2.3 The lead solution is evaporated to dryness and dissolved into water. Lead sulfate is precipitated and transferred to a preweighed planchet. The precipitate is dried to constant weight to determine chemical recovery, and bismuth-210 (Bi-210) is allowed to ingrow for 21 days.

2.4 The ingrown Bi-210/lead sulfate is counted by gas proportional counter to determine the Bi-210 and therefore the Pb-210 activity.

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LEAD-210 IN ORGANIC SOLIDS (PROCEDURE FM-RAD-0074)

Working Linear Range: To be determined, probably .1 g to 100 g
Reporting Limit: To be determined
Reporting Units: pCi/g
Matrix: Organic solids

1.0 Scope and Application

This method is applicable to the determination of lead-210 (Pb-210) in samples requiring Pb-210 analysis in an organic matrix.

2.0 Method Summary

2.1 This procedure provides for the separation of Pb-210 beta emitting nuclides by elution from anion exchange resin columns. Separation is based on the differences in absorption and chemical properties of Pb-210 and contaminants.

2.2 An appropriate weight of sample is aliquoted for analysis. Stable lead carrier is added to the sample. As appropriate, the sample is dried and/or muffled to ash in a furnace. The exact muffling sequence and amount of sample must be determined on an individual matrix basis to prevent fires and/or explosions. The extent of removal of organic material will directly affect the recovery of lead in the separation procedure. The aliquot is dissolved with a series of additions of nitric acid (HNO_3) and hydrofluoric acid (HF) and evaporated to dryness. The sample is microwaved with HNO_3 and HF. The sample is converted to a chloride form prior to loading onto ion exchange columns.

2.3 The lead solution is evaporated to dryness and dissolved into water. Lead sulfate is precipitated and transferred to a preweighed planchet. The precipitate is dried to constant weight to determine chemical recovery and bismuth-210 (Bi-210) is allowed to ingrow for 21 days.

2.4 The ingrown Bi-210/lead sulfate is counted by gas proportional counter to determine the Bi-210 and therefore the Pb-210 activity.

LEAD-210 IN AIR PARTICULATE FILTERS (PROCEDURE FM-RAD-0076) 3298

Working Linear Range: To be determined
Reporting Limit: To be determined by number of filters and volume
Reporting Units: pCi/filter, pCi/mL, pCi/L, pCi/cc
Matrix: Filter(s)

1.0 Scope and Application

This method is applicable to the determination of lead-210 (Pb-210) on one or more air particulate filter(s).

2.0 Method Summary

2.1 This procedure provides for the separation of Pb-210 by elution from anion/cation exchange resin columns. Separation is based on the differences in absorption and chemical properties of the Pb-210 radionuclide and detection is by beta counting.

2.2 One or more air particulate filter(s) are selected for analysis. Stable, standardized lead carrier is added to the sample. The sample is placed in a beaker and muffled in a programmable furnace. If glass fiber filters were used, dissolve the residue in HF and evaporate to dryness. Repeat several times. Dissolve this residue or the ash residue from the muffle furnace for regular filters in a series of nitric acid additions. This solution is converted to the chloride form prior to loading on the ion exchange columns.

2.3 The lead solution is evaporated to dryness and dissolved into water. Lead sulfate is precipitated and transferred to a preweighed planchet. The precipitate is dried to constant weight to determine chemical recovery, and bismuth-210 (Bi-210) is allowed to ingrow for 21 days.

2.4 The ingrown Bi-210/lead sulfate is counted by gas proportional counter to determine the Bi-210 and thus the Pb-210 activity.

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ISOTOPIC THORIUM IN MILK, VEGETATION, SOIL/SEDIMENT, WATER, AND AIR FILTERS BY ALPHA SPECTROMETRY (PROCEDURE FM-RAD-0080)

Working Linear Range: Infinite with dilution
Reporting Limit: To be determined
Reporting Units: Milk, water, pCi/L; Solids, pCi/g; Air filters, pCi/air filter
Matricies: Milk, vegetation, water, soil/sediment, air filters

1.0 Scope and Application

1.1 The method covers the measurement of thorium isotopes 228, 230, and 232 in milk, vegetation, soil/sediment, water, and air filters. The mass of the isotopes can be calculated from the measured activities using the specific activity factors for each isotope. The nominal sensitivity that may be obtained by this exact method for the media requires performance data. Note that the beta emitting Th-234 isotope is not covered by the method.

1.2 This analytical method applies to Analytical Support Levels C and D.

2.0 Method Summary

The sample is dissolved in acid or ashed, and the residue is dissolved in acid. Thorium is separated from uranium by organic extraction and purified by adsorption on anion exchange resin from nitric acid. It is stripped from the resin with HCl and electrodeposited onto a stainless steel disk. The disk is counted on a solid state detector (e.g., a silicon surface barrier) to determine the thorium isotopes by alpha spectrometry. The recovery of the thorium isotopes is measured using a Th-229 tracer.

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ISOTOPIC THORIUM IN ORGANIC LIQUIDS BY ALPHA SPECTROSCOPY (PROCEDURE FM-RAD-0082)

Working Linear Range: Infinite with dilution
Reporting Limit: To be determined
Reporting Units: pCi/L
Matrix: Organic liquids

1.0 Scope and Application

This procedure is applicable to alpha-emitting thorium isotopes dissolved in organic liquids immiscible with water.

2.0 Method Summary

2.1 A Th-229 tracer in an organic phase is added to the sample. Thorium isotopes are extracted into an aqueous phase and the extract is purified by adsorption on anion change resin, followed by stripping with HCl. The eluted solution is electrodeposited onto a metal disk.

2.2 The disk is counted on a solid state detector (e.g., a silicon surface barrier) to determine the thorium isotopes by alpha spectrometry. The recovery of the thorium isotopes is determined by the recovery of the Th-229 tracer (Th-234, a relatively short-lived beta-emitting daughter of U-238 is not detected).

ISOTOPIC THORIUM IN ORGANIC SOLIDS BY ALPHA SPECTROMETRY (PROCEDURE FM-RAD-0084)

Working Linear Range: Infinite with dilution
Reporting Limit: To be determined
Reporting Units: pCi/g
Matrix: Asphalt, wood, other organic solids or solids with high organic content.

1.0 Scope and Application

1.1 The method covers the measurement of thorium isotopes (Th-238, Th-230, and Th-232) in organic solids or solids with high organic content. The mass of the isotopes can be calculated from the measured activities using specific activity factors for each isotope. The analytical sensitivity is affected by the sample matrix; performance data will demonstrate the sensitivity. The beta emitting Th-234 isotope is not covered by the method.

1.2 This analytical method applies to Analytical Support Levels C and D.

2.0 Method Summary

2.1 The sample is granulated and Th-229 tracer is added. The sample is dry ashed and the residue decomposed by nitric acid-hydrofluoric acid digestion. Thorium is separated from uranium by organic extraction followed by back extraction into the aqueous phase. The extract is purified by adsorption on anion change resin followed by stripping with HCl. The eluted solution is electrodeposited onto a planchet.

2.2 The planchet is counted on a solid state detector (e.g., a silicon surface barrier) to determine the thorium isotopes by alpha spectrometry. The recovery of the thorium isotopes is determined by the recovery of the Th-229 tracer.

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ISOTOPIC THORIUM IN INORGANIC SOLIDS BY ALPHA SPECTROMETRY (PROCEDURE FM-RAD-0086)

Working Linear Range: Infinite with dilution
Reporting Limit: To be determined
Reporting Units: pCi/g
Matrix: Concrete, masonry, and earthen solids

1.0 Scope and Application

1.1 This method covers the measurement of thorium isotopes (Th-228, Th-230, and Th-232) in concrete, masonry and earthen solids with low organic content. The mass of the isotopes can be calculated from the measured activities using the specific activity factors for each isotope. Sensitivity is a function of recovery and counting time. Th-234, a short-lived beta-emitting daughter of U-238, is not addressed by the method.

1.2 This analytical method applies to Analytical Support Levels C and D.

2.0 Method Summary

2.1 If required, the sample is ground to enhance surface to volume ratio. A Th-229 tracer is added and residual organics are removed by heating. The sample is decomposed by nitric acid-hydrofluoric acid digestion. Thorium is separated from uranium by organic extraction followed by back extraction into the aqueous phase. The extract is purified by adsorption on anion change resin followed stripping with HCl. The eluted solution is electrodeposited onto a planchet.

2.2 The planchet is counted on a solid state detector (e.g., a silicon surface barrier) to determine the thorium isotopes by alpha spectrometry. The recovery of the thorium isotopes is determined by the recovery of the Th-229 tracer.

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ISOTOPIC URANIUM IN VEGETATION, MILK, WATER,AND AIR FILTERS BY ALPHA SPECTROMETRY (PROCEDURE FM-RAD-0100)

Working Linear Range: Greater than 1 pCi/L, infinite with dilution
Reporting Limit: ~1 pCi/L (water); TBD for other materials
Reporting Units: Milk, water, pCi/L; Solids, pCi/g; Air filters, pCi/air filter
Matricies: Milk, vegetation, water, air filters

1.0 Scope and Application

1.1 This procedure applies to determination of isotopic uranium in milk, water, vegetation, and air filters. Total uranium activity is determined by adding the results for the individual isotopes (U-233/234, U-235/236, U-238). Total uranium mass may be determined with the specific activity factors for each isotope. The nominal sensitivity may vary with each analysis but is approximately 0.06 pCi/L (U-235,238), 1 pCi/L (U-234) for water. The nominal sensitivity that may be obtained by this exact method for the other media requires additional performance data.

1.2 This analytical method applies to Analytical Support Levels C and D.

2.0 Method Summary

A uranium tracer (U-232) is added to the sample. A milk or vegetation sample is oven dried and ashed, a water sample is evaporated, and an air filter is dissolved. The sample residue is dissolved in strong HCl. Uranium is extracted into tri-iso-octylamine (TIOA) and then stripped from the TIOA with dilute nitric acid. Polonium is removed by absorption onto Ni foil. The uranium is electrodeposited onto a stainless steel disk, and the disk is counted on a solid state detector, e.g., silicon surface barrier, to determine the uranium isotopes by alpha spectrometry.

ISOTOPIC URANIUM IN SOIL/SEDIMENT BY ALPHA SPECTROMETRY (PROCEDURE FM-RAD-0110)

Working Linear Range: Infinite with dilution
Reporting Limit: ~0.04 pCi/g (U-235), ~0.8 pCi/g (U-238)
Reporting Units: pCi/g
Matricies: Soil/sediment

1.0 Scope

1.1 The method applies to the determination of uranium isotopes in soil/sediment samples. It has been applied to soils from various parts of the United States. Total uranium activity is determined by adding the results for the individual isotopes (U-233/234, U-235/236, U-238). Total uranium mass may be determined with the specific activity factors for each isotope. The nominal sensitivity may vary with each analysis but is approximately 0.8 pCi/g for U-238 and 0.04 pCi/g for U-235.

1.2 This analytical method applies to Analytical Support Levels C and D.

2.0 Method Summary

2.1 After adding U-232 tracer, organic matter is removed from the sample by heating. The sample is then decomposed by nitric acid-hydrofluoric acid digestion, and uranium is coprecipitated by making the solution basic with ammonium hydroxide (carbonate free). The hydroxide precipitate is dissolved in 8 M HCl, which is extracted with isopropyl ether to remove the bulk of the iron present. For samples with relatively low iron concentrations, this extraction can be omitted.

2.2 The 8 M HCl solution is passed through an anion exchange resin column. Uranium, polonium, and bismuth will be adsorbed on the resin, but thorium and radium will pass through the column. Plutonium and any unextracted iron are also retained by the resin but are eluted with 6 M HCl containing hydrogen iodide. The iodide ion reduces plutonium (IV) to plutonium (III) and reduces the iron (III) to iron (II); neither ion is retained by the ion exchange resin in 6 M HCl. The uranium is eluted from the column with 1.0 M HCl, whereas any zinc adsorbed on the column will remain. The uranium is electrodeposited onto a stainless steel planchet, and the planchet is counted on a silicon surface barrier detector to determine the uranium isotopes by alpha spectrometry.

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ALPHA-EMITTING NUCLIDES IN ORGANIC LIQUIDS (PROCEDURE FM-RAD-0112)

Working Linear Range: Nuclide and Matrix Specific
Reporting Limit: Nuclide and Matrix Specific
Reporting Units: pCi/L
Matrix: Organic liquids

1.0 Scope and Application

1.1 This method is applicable to the direct screening for alpha-emitting nuclides in organic liquids compatible with liquid scintillators. Sufficiently low count rates in specified beta energy regions demonstrate the absence of various alpha emitters above release levels, and indicate that further analyses are not required. Alpha energy resolution is approximately 300 keV FWHM

1.2 The compatibility of the matrix with the liquid scintillator is determined automatically by a quench indicating system.

2.0 Method Summary

2.1 An aliquot of the matrix is added directly to an "Environmentally Safe" commercial liquid scintillation solution in a 20 ml translucent scintillation vial. The vial is counted in a commercial liquid scintillation system.

2.2 For best results, a counting system with alpha/beta pulse shape discrimination and energy spectrum display should be used. Essentially 100% counting efficiency is possible with background count rates less than 1 cpm.

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ISOTOPIC URANIUM IN INORGANIC SOLIDS BY ALPHA SPECTROMETRY (PROCEDURE FM-RAD-0113)

Working Linear Range: Infinite with dilution
Reporting Limit: ~0.04 pCi/g (U-235), ~.8 pCi/g (U-238)
Reporting Units: pCi/g
Matrix: Inorganic Solids (Non-metallic)

1.0 Scope and Application

1.1 The method applies to the determination of uranium isotopes in granulated inorganic solids. Total uranium activity is determined by adding the results from the analyses of the individual isotopes (U-233/234, U-235/236, U-238). Total uranium mass may be determined with the specific activity factors for each isotope. The nominal sensitivity may vary with each analysis but is approximately 1.0 pCi/g for U-238 and 0.05 pCi/g for U-235.

1.2 This analytical method applies to Analytical Support Levels C and D.

2.0 Method Summary

2.1 Inorganic solids are ground to approximately 16 mesh. After U-232 tracer is added, organic matter is removed from the sample by heating. The sample is then decomposed by nitric acid-hydrofluoric acid digestion, and uranium is coprecipitated by adjusting the pH to make the solution basic by adding ammonium hydroxide (carbonate free). The hydroxide precipitate is dissolved in 8 M HCl. If iron is present in the sample, it is extracted from the 8 M HCl solution with isopropyl ether.

2.2 The 8 M HCl solution is passed through an anion exchange resin column. Uranium, polonium, and bismuth will be adsorbed on the resin, but thorium and radium will pass through the column. Plutonium and any unextracted iron are also retained by the resin. They are eluted with 6 M HCl containing hydrogen iodide. The iodide ion reduces plutonium (IV) to plutonium (III) and reduces the iron (III) to iron (II); neither ion is retained by the ion exchange resin in 6 M HCl. The uranium is eluted from the column with 1.0 M HCl (any zinc adsorbed on the column will remain). The uranium is then electrodeposited onto a stainless steel planchet, and the planchet is counted by using a silicon surface barrier detector to determine the uranium isotopes by alpha spectrometry.

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ALPHA-EMITTING NUCLIDES IN AQUEOUS LIQUIDS (PROCEDURE FM-RAD-0114)

Working Linear Range: Nuclide and Matrix Specific
Reporting Limit: Nuclide and Matrix Specific
Reporting Units: pCi/L
Matrix: Aqueous liquids

1.0 Scope and Application

1.1 This method is applicable to screening for alpha-emitting nuclides in aqueous liquids compatible with aqueous-accepting liquid scintillators. Sufficiently low count rates in specified alpha energy regions demonstrate the absence of various alpha emitters above release levels, and indicate that further analyses are not required. Alpha energy resolution is approximately 300 keV FWHM.

1.2 The compatibility of the matrix with the liquid scintillator is determined automatically by a quench indicating system.

2.0 Method Summary

2.1 An aliquot of the matrix is added directly to an "Environmentally Safe" aqueous-accepting commercial liquid scintillation solution in a 20 ml translucent scintillation vial. The vial is counted in a commercial liquid scintillation system.

2.2 For best results, a counting system with alpha/beta pulse shape discrimination and energy spectrum display should be used. Essentially 100% counting efficiency is possible with background count rates less than 1 cpm.

ISOTOPIC URANIUM IN ORGANIC SOLIDS BY ALPHA SPECTROMETRY (PROCEDURE FM-RAD-0115)

Working Linear Range: Infinite with dilution
Reporting Limit: ~0.04 pCi/g (U-235), ~0.8 pCi/g (U-238)
Reporting Units: pCi/g
Matrix: Organic or high-organic solid materials

1.0 Scope and Application

1.1 The method applies to the determination of uranium isotopes in dry ashed organic solids or solids containing high concentrations of organic materials. Total uranium activity is determined by adding the results from the analyses of the individual isotopes (U-233/234, U-235/236, U-238). Total uranium mass may be determined with the specific activity factors for each isotope. The nominal sensitivity may vary with each analysis but is approximately 1.0 pCi/g for U-238 and 0.05 pCi/g for U-235.

1.2 This analytical method applies to Analytical Support Levels C and D.

2.0 Method Summary

2.1 The sample is granulated and U-232 tracer is added. The sample is dry ashed and the residue decomposed by nitric acid-hydrofluoric acid digestion. Uranium is coprecipitated by adjusting the pH to make the solution basic by adding ammonium hydroxide (carbonate free). The hydroxide precipitate is dissolved in 8 M HCl. If iron is present in the sample it is extracted from the 8 M HCl solution with isopropyl ether.

2.2 The 8 M HCl solution is passed through an anion exchange resin column. Uranium, polonium, and bismuth will be adsorbed on the resin, but thorium and radium will pass through the column. Plutonium and any unextracted iron are also retained by the resin. They are eluted with 6 M HCl containing hydrogen iodide. The iodide ion reduces plutonium (IV) to plutonium (III) and reduces the iron (III) to iron (II); neither ion is retained by the ion exchange resin in 6 M HCl. The uranium is eluted from the column with 1.0 M HCl (any zinc adsorbed on the column will remain). The uranium is then electrodeposited onto a stainless steel planchet, and the planchet is counted on a silicon surface barrier detector to determine the uranium isotopes by alpha spectrometry.

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URANIUM IN WATER, SOIL/SEDIMENT, AND AIR FILTERS BY PULSED-LASER PHOSPHORIMETRY (PROCEDURE FM-RAD-0120)

Working Linear Range: Greater than 50 ng/L, infinite with dilution
Reporting Limit: 50 ng/L Water; TBD for other matricies
Reporting Units: Water, mg/L; Solids, mg/g; Air filters, mg/air filter
Matricies: Water, soil/sediment, air filters

1.0 Scope and Application

1.1 The method covers the determination of uranium in water, soil/sediment, and air filters in the range of 50 ng/L or greater. Samples with uranium levels above the laser phosphorimeter dynamic range may be diluted to bring the concentration to a measurable level. The minimum detectable concentration is approximately 50 ng U/L of water. The nominal sensitivity that may be obtained by this exact method for the other media requires additional performance data.

1.2 The method applies to Analytical Support Levels C and D.

2.0 Method Summary

2.1 The method is based on the use of commercially available laser phosphorimeters to analyze uranium in the sample media.

2.2 The method is useful for the analysis of water either directly, following dilution, or following wet-ashing as required by the sample media.

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GROSS ALPHA AND GROSS BETA RADIOACTIVITY IN WATER AND GROSS BETA RADIOACTIVITY IN AIR FILTERS BY PROPORTIONAL COUNTING (PROCEDURE FM-RAD-0130)

Working Linear Range: Infinite with dilution
Reporting Limit: To be determined.
Reporting Units: Water, pCi/L; Air filter, pCi/air filter
Matricies: Water, air filters

1.0 Scope and Application

1.1 The method establishes a procedure for monitoring water for gross alpha and gross beta activity and air filters for gross beta activity. The method can measure alpha particles with energies above 3.9 million-electron-volts (MeV) and beta particles with maximum energies above 0.1 MeV.

1.2 The minimum detectable concentration for the method depends on sample size, counting system characteristics, background, and counting time. For water with extremely high solids content (>500 mg/L), EPA Method 900.1 is recommended.

1.3 This analytical method applies to Analytical Support Levels C and D.

2.0 Method Summary

2.1 An aliquot of a dissolved air filter or water sample is evaporated to a small volume and transferred quantitatively to a tared 2-inch stainless steel counting planchet. The sample residue is dried to constant weight and counted for alpha and/or beta radioactivity.

2.2 Counting efficiencies for both alpha and beta particle activities are selected from graphs (or best fit equations) of counting efficiency versus sample weight.

2.3 The radioactive constituents of the sample are not separated from the solids of the sample; therefore, the solids concentration is a limiting factor in the sensitivity of the method for any given (water) sample. For samples with very low radioactivity, it is essential to analyze as large a sample aliquot as possible to give reasonable counting times in meeting the required sensitivity (minimum detectable concentration).

2.4 The largest sample aliquot to be counted for gross alpha activity is the volume that gives a solids density thickness of 5 mg/cm^2 in the counting planchet. For a 2-inch-diameter counting planchet (20 cm^2), an aliquot containing 100 mg of solids is the maximum sample size to be evaporated and counted for gross alpha activity.

2.5 Sample density thickness on the planchet shall not be more than 5 mg/cm^2 for gross alpha and not more than 10 mg/cm^2 for gross beta.

2.6 The concentration of total dissolved solids is not as limiting for determining gross beta activity because beta particles are not stopped in solids as easily as alpha particles. Very often a single sample aliquot is evaporated and counted for both gross alpha and gross beta activity. In that case, the sample aliquot size would be dictated by the solids limitation for alpha particles. For beta counting only, the solids on the planchet shall be limited to 200 mg (for 2" planchet).

2.7 Radionuclides that are volatile under the sample preparation conditions of the method will not be measured. The sample is evaporated after nitric acid is added, and the nitrated solids that form will not remain at a constant weight after being dried and if exposed to high atmospheric humidity. Therefore, the samples need to be heated to a dull red glow for a few minutes to convert the nitrate salts to oxides. Sample weights are then usually stable enough to give consistent counting rates, and a correct counting efficiency can then be determined.

3298

GROSS ALPHA/BETA ON AIR FILTERS (PROCEDURE FM-RAD-0132)

Working Linear Range: 0.1 to 100,000 pCi/filter
Reporting Limit: Function of air volume and counting time
Reporting Units: pCi/m^3 (air); pCi/mg (particulate)
Matrix: Particulate material in air

1.0 Scope and Application

1.1 This method is applicable to the determination of total alpha and total beta activity in particulate material contained in air which is entrained on a filter. Minimal sample preparation is required.

1.2 Alpha and beta activities are determined independently. Spectrometric resolution of alpha and beta emitters is not provided.

2.0 Method Summary

2.1 If activity per volume of air drawn through the filter is to be reported, that information must be obtained per filter from the sampling protocol. If activity per mg of particulate is to be reported, the filter must be weighed after dehydration in a desiccator prior to deployment, and re-weighed after dehydration after deployment.

2.2 The filter is placed, upstream face upwards, on a metal planchet. The planchet is placed in the counting chamber of a thin-window commercial proportional counting system (models with automatic sample changers are available if the number of samples to be analyzed is large).

2.3 Alpha and beta events are distinguished by pulse shape discrimination circuitry and accumulated in separate registers in the counting device. Detection efficiencies are determined by alpha calibration sources configured in the same geometry as the filters. Correction factors for self-absorption may be required for high particulate mass loadings.

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RADIOANALYSIS OF SOIL/SEDIMENT, AIR FILTERS, MILK, AND WATER BY GAMMA SPECTROMETRY (PROCEDURE FM-RAD-0140)

Working Linear Range: Infinite with dilution
Reporting Limit: To be determined
Reporting Units: Milk, water, pCi/L; Solids, pCi/g; Air filters, pCi/air filter
Matricies: Water, soil/sediment, air filters, milk

1.0 Scope and Application

1.1 The method applies to determination of gamma ray emitting radionuclides in samples of soil/sediment, air filters, milk, water, and aqueous solutions (i.e., air filter solutions). Radionuclides covered by the method include Cs-137/Ba-137, Ru-106/Rh-106, K-40. The minimum detectable concentration for this method depends on sample size and characteristics, counting system characteristics, background, and counting time.

1.2 This analytical method applies to Analytical Support Levels C and D.

2.0 Method Summary

2.1 The entire sample or a homogeneous aliquot of the liquid or solid sample is put into a standard geometry for gamma counting. The counting efficiency for the geometry must have been determined with standards of known radionuclide activity. Sample aliquots are counted long enough to meet the required sensitivity. The gamma spectrum is printed out or stored in an appropriate computer-compatible device for data processing (calculation of sample radionuclide concentrations).

2.2 Gamma ray spectra are typically measured with a modular equipment system consisting of a detector, analyzer, memory, printer, and permanent data storage device. Germanium (Ge) detectors are used because of their excellent energy resolution.

RADIOANALYSIS OF BULK MATERIALS BY GAMMA SPECTROMETRY (PROCEDURE FM-RAD-0142)

Working Linear Range:	Infinite with dilution
Reporting Limit:	To be determined; isotope specific
Reporting Units:	Water, pCi/L; Solids, pCi/g
Matrix:	Concrete, masonry, asphalt, wood, sludge, sediment

1.0 Scope and Application

1.1 The method applies to determination of gamma ray emitting radionuclides in samples of granulated solids and aqueous solutions and slurries. Radionuclides covered by the method include Cs-137/Ba-137, Ru-106/Rh-106, and K-40. The minimum detectable concentration for this method depends on sample size and characteristics, counting system characteristics, background, and counting time.

1.2 This analytical method applies to Analytical Support Levels C and D.

2.0 Method Summary

2.1 Bulk solids are granulated to facilitate reproducible placement in counting containers. The entire sample or a homogeneous aliquot is placed or configured into a standard geometry for gamma counting. The counting efficiency for the geometry must be determined with standards of known radionuclide activity. Sample aliquots are counted for a period of time which meets the required sensitivity. The gamma spectrum is printed or stored in an appropriate computer-compatible device for data processing (calculation of sample radionuclide concentrations).

2.2 Gamma ray spectra are typically measured with a modular equipment system consisting of a detector, analyzer, memory, printer, and permanent data storage device. Germanium detectors are used because of their excellent energy resolution.

3298

U-238, U-235, RA-226, AND RA-228 IN SOLIDS BY GAMMA SPECTROMETRY (PROCEDURE FM-RAD-0149)

Working Linear Range: Infinite with dilution
Reporting Limit: To be determined, nuclide dependent
Reporting Units: pCi/g
Matrix: Soil/sediment, granulated solids, slurries, liquids

1.0 Scope and Application

This method is applicable to the determination of U-238, U-235, Ra-226, and Ra-228 concentrations in bulk materials such as soils, concrete, masonry, asphalt, sludge, sediment, and most common building materials. Minimal sample preparation is required. U-234 concentrations may be inferred from the U-235 concentrations in depleated uranium samples (i.e., low U-235 content). Other gamma-emitting nuclides may be determined by this method, although the geometry is optimized for uranium determinations.

2.0 Method Summary

2.1 The entire sample or a homogenized aliquot of the liquid or solid sample is placed in a large (500 ml) airtight Marinelli beaker and the tared container is weighed. The sample is held for 30 days to achieve full equilibrium between Ra-226 and Ra-222 (alternatively, the equilibrium status can be confirmed by a second count one month after the beaker is sealed if a prompt preliminary count is required). Because Ra-226 interferes with the gamma analysis of U-235, its concentration is determined from the 0.609 MeV gamma of Bi-214 with which it is in equilibrium.

2.2 The counting efficiency for each isotope is a function of net sample weight and it is determined from standards of various net weights containing known concentrations. Sample aliquots are counted long enough to meet the required sensitivity. The gamma spectrum is printed or stored in an appropriate computer-compatible device for data processing (calculation of sample radionuclide concentrations).

2.3 Gamma ray spectra are typically measured with a modular equipment system consisting of a high resolution germanium detector, analyzer, memory, printer, and permanent data storage device. Germanium detectors are used because of their excellent energy resolution.

TECHNETIUM-99 IN AQUEOUS MEDIA (PROCEDURE FM-RAD-0150)

3298

Working Linear Range: 10 pCi/L to 10000 pCi/L
Reporting Limit: 30 pCi/L
Reporting Units: pCi/L
Matrix: Aqueous liquids, water, surface water, ground water

1.0 Scope and Application

This method is applicable to the determination of technetium-99 (Tc-99) activity in aqueous solutions.

2.0 Method Summary

2.1 This procedure provides for the determination of Tc-99 in aqueous liquids, water, surface water, and ground water. The aqueous sample is extracted to place the Tc-99 in a chloroform phase, which is taken to dryness and beta counted.

2.2 An appropriate volume of liquid is aliquoted for Tc-99 determination. If the sample is not clear, then the sample is filtered or centrifuged to remove any sediment or suspended material. Proceed on the soluble or liquid portion. Add to the sample ammonium citrate-ammonium hydroxide solution and hydrofluoric acid. Carefully adjust the pH of the sample to 5.0 to 5.1. Extract the Tc-99 into chloroform. Evaporate the chloroform in a planchet and obtain a constant weight of the residue. Beta count the planchet on a gas flow proportional counter to obtain the amount of Tc-99.

2.3 Each matrix type in the sample batch should have a yield tracer sample analyzed to determine the recovery for the matrix.

3298

TECHNETIUM-99 IN INORGANIC SOLIDS (PROCEDURE FM-RAD-0152)

Working Linear Range: To be determined, probably .1 g to 100 g
Reporting Limit: To be determined
Reporting Units: pCi/g
Matrix: Inorganic solids

1.0 Scope and Application

This method is applicable to the determination of technetium-99 (Tc-99) activity in inorganic solids.

2.0 Method Summary

2.1 This procedure provides for the determination of Tc-99 in inorganic solids. The solid sample is extracted to place the Tc-99 in an aqueous phase. The aqueous sample is then extracted to place the Tc-99 into a chloroform phase, which is taken to dryness and beta counted. A yield tracer sample should be run with each matrix type.

2.2 An appropriate weight (1-5 grams) of wet sample is used for the extraction. The percent of moisture content should be determined as a separate measurement if dry weight is required. For an unknown matrix proceed carefully. Add 5 mL of concentrated ammonium hydroxide, 5 mL of water, and 1 mL of 30% hydrogen peroxide. After any reaction stops, cap and shake the sample for 4 hours. Add water to bring the volume to 25 mL and warm in a hot water bath. Cool, centrifuge, and decant off the supernate. The supernate solution is used for Tc-99 determination. If the sample is not clear, then filter or centrifuge the sample to remove any sediment or suspended material. Proceed on the soluble or liquid portion. Add to the sample ammonium citrate-ammonium hydroxide solution and hydrofuoric acid. Carefully adjust the pH of the sample to 5.0 to 5.1. Extract the Tc-99 into chloroform. Evaporate the chloroform in a planchet and obtain a constant weight of the residue. Beta count the planchet on a gas flow proportional counter to obtain the amount of Tc-99.

3298

TECHNETIUM-99 IN ORGANIC SOLIDS (PROCEDURE FM-RAD-0154)

Working Linear Range: To be determined
Reporting Limit: To be determined, probably matrix dependent
Reporting Units: pCi/g
Matrix: Organic solids

1.0 Scope and Application

This method is applicable to the determination of technetium-99 activity in organic solids. Because of potential interferences from organic material extracting into the chloroform phase, each matrix will need testing.

2.0 Method Summary

2.1 This procedure provides for the determination of technetium-99 in organic solids. The solid sample is extracted to place the Tc-99 in an aqueous phase. The aqueous sample is then extracted to place the Tc-99 into a chloroform phase, which is taken to dryness and beta counted. A yield tracer sample should be run with each matrix type.

2.2 An appropriate weight (1-5) grams of wet sample is used for the extraction. The % moisture should be determined as a separate measurement if dry weight is required. For an unknown matrix proceed carefully. Add 5 mL of concentrated ammonium hydroxide, 5 mL of water and 1 mL of 30% hydrogen peroxide. After any reaction stops add additional hydrogen peroxide until indication of reaction with organic material stops. Cap and shake the sample for 4 hours. Add water to bring the volume to 25 mL and warm in a hot water bath. Cool, centrifuge, and decant off the supernate. The supernate solution is used for Tc-99 determination. If the sample is not clear, then filter or centrifuge to remove any sediment or suspended material. Proceed on the soluble or liquid portion. Add to the sample ammonium citrate-ammonium hydroxide solution and HF acid. Carefully adjust the pH of the sample to 5.0 to 5.1. Extract the Tc-99 into chloroform. Evaporate the chloroform in a planchet and obtain a constant weight of the residue. Beta count the planchet on a gas flow proportional counter to obtain the amount of Tc-99.

3298

STRONTIUM-90 IN AQUEOUS MEDIA (PROCEDURE FM-RAD-160)

Working Linear Range: With dilution, 0.5 pCi/L to 10000 pCi/L
Reporting Limit: To be determined
Reporting Units: pCi/L
Matrix: Aqueous liquid, groundwater, surface water

1.0 Scope and Application

This method is applicable to the determination of strontium-90 (Sr-90) in samples requiring Sr-90 analysis in an aqueous liquid. Sr-90 is determined as a total strontium, with no Sr-89 assumed present.

2.0 Method Summary

2.1 This procedure provides for the separation of the Sr-90 beta-emitting nuclide by a series of precipitation and separation steps.

2.2 An appropriate amount of the sample is aliquoted for analysis. Stable strontium and barium carriers are added to the sample. Strontium is precipitated from the water sample as insoluble carbonate. The precipitate is dissolved in nitric acid and strontium is precipitated as the nitrate from a fuming-acid solution. Barium and radium are removed as chromates and the yttrium is removed as a hydroxide.

2.3 The strontium is precipitated as the sulfate and filtered onto a preweighed filter. The sample is dried to constant weight to determine chemical recovery.

2.4 The filter is counted for its beta activity on a gas-proportional counter to determine the Sr-90 activity.

3298

STRONTIUM-90 IN INORGANIC SOLIDS (PROCEDURE FM-RAD-0162)

Working Linear Range: To be determined, probably .1 g to 100 g
Reporting Limit: To be determined
Reporting Units: pCi/g
Matrix: Inorganic solids

1.0 Scope and Application

This method is applicable to the determination of strontium-90 (Sr-90) in samples requiring Sr-90 analysis in an inorganic matrix. Sr-90 is determined as a total strontium, with no Sr-89 assumed present.

2.0 Method Summary

2.1 This procedure provides for the separation of the Sr-90 beta-emitting nuclide by a series of precipitation and separation steps.

2.2 An appropriate weight of sample is aliquoted for analysis. Stable strontium and barium carriers are added to the sample. As appropriate, the sample is dried and/or muffled to ash in a furnace. The exact muffling sequence and amount of sample must be determined on an individual matrix basis. The aliquot is dissolved with a series of additions of nitric acid (HNO_3) and hydrofluoric acid (HF) and evaporated to dryness. The sample is microwaved with HNO_3 and HF. The sample is converted to a nitrate form prior to further separation.

2.3 The strontium nitrate is precipitated and dissolved into water. Barium is separated out by precipitation.

2.4 The strontium is precipitated as the sulfate and filtered onto a preweighed filter. The sample is dried to constant weight to determine chemical recovery.

2.5 The filter is counted for its beta activity on a gas-proportional counter to determine the Sr-90 activity.

STRONTIUM-90 IN ORGANIC SOLIDS (PROCEDURE FM-RAD-0164) 3298

Working Linear Range: To be determined, probably .1 g to 100 g
Reporting Limit: To be determined
Reporting Units: pCi/g
Matrix: Organic solids

1.0 Scope and Application

This method is applicable to the determination of Strontium-90 (Sr-90) in samples requiring Sr-90 analysis in an organic matrix.

2.0 Method Summary

2.1 This procedure provides for the separation of the Sr-90 beta-emitting nuclide by a series of precipitation and separation steps.

2.2 An appropriate weight of the sample is aliquoted for analysis. Stable strontium and barium carriers are added to the sample. As appropriate, the sample is dried and/or muffled to ash in a furnace. The exact muffling sequence and amount of the sample must be determined on an individual matrix basis to prevent fires and/or explosions. The extent of removal of organic material will directly affect the recovery of strontium in the separation procedure. The aliquot is dissolved with a series of additions of nitric acid (HNO_3) and hydrofluoric acid (HF) and evaporated to dryness. The sample is microwaved with HNO_3 and HF. The sample is converted to a nitrate form prior to further separation.

2.3 The strontium nitrate is precipitated and dissolved into water. Barium is separated out by precipitation.

2.4 The strontium is precipitated as the sulfate and filtered onto a preweighted filter. The sample is dried to constant weight to determine chemical recovery.

2.5 The filter is counted for its beta activity on a gas proportional counter to determine the Sr-90 activity.

POLONIUM-210 IN AQUEOUS SAMPLES (PROCEDURE FM-RAD-0170) 3298

Working Linear Range: With dilution, 0.5 pCi/L to 100000 pCi/L
Reporting Limit: To be determined
Reporting Units: pCi/L
Matrix: Aqueous liquid, groundwater, surface water

1.0 Scope and Application

This method is applicable to the determination of Polonium-210 (Po-210) in aqueous samples.

2.0 Method Summary

2.1 Polonium-210 is determined by alpha spectrometry by counting a plated source. Polonium is plated from a hydrochloric acid (HCl) solution by a galvanic deposition technique on a nickel (Ni) disk. The yield and efficiency are determined by use of a polonium tracer.

2.2 Use an appropriate volume of sample, typically 1000 mL, and acidify with 50 mL of concentrated HCl. Add polonium tracer. Concentrate the solution to approximately 20 mL over low heat. Do not boil solution. Add 100 mg of ascorbic acid and plate polonium onto specially-cleaned and polished Ni disks. Plating time should be five hours. Determine polonium by alpha spectrometry.

POLONIUM-210 IN INORGANIC SOLIDS (PROCEDURE FM-RAD-0172) 3298

Working Linear Range: To be determined, probably .1 g to 10 g
Reporting Limit: To be determined
Reporting Units: pCi/g
Matrix: Inorganic solids

1.0 Scope and Application

This method is applicable to the determination of Polonium-210 (Po-210) in inorganic solids. The method is a controlled acid leach.

2.0 Method Summary

2.1 Polonium-210 is determined by alpha spectrometry by counting a plated source. Polonium is plated from a hydrochloric acid (HCl) solution by a galvanic deposition technique on nickel (Ni) disk. The yield and efficiency are determined by use of a polonium tracer. Matrix effects will be determined on a case-by-case basis.

2.2 Because some forms of polonium are volatile, total dissolution is not attempted. Low temperature acid leach is employed to remove to the extent possible any polonium.

2.3 Use an appropriate aliquot, typically two grams, of undried sample. Add lead carrier and polonium tracer. The sample is acid leached by use of a series of additions of nitric acid and hydrofluoric acid, with volume concentrations between each series. Do not evaporate to dryness and do not boil solution. Repeat with nitric acid. Dilute to 90 mL volume and filter. Insoluble sulfates are removed by heating with concentrated sulfuric acid. To the supernate add HCl and enough ascorbic acid to complex any iron present and plate polonium onto specially-cleaned and polished Ni disks. Plating time should be five hours. Determine polonium by alpha spectrometry.

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POLONIUM-210 IN ORGANIC SOLIDS (PROCEDURE FM-RAD-0174)

Working Linear Range: To be determined, probably .1 g to 10 g
Reporting Limit: To be determined
Reporting Units: pCi/g
Matrix: Organic solids

1.0 Scope and Application

This method is applicable to the determination of Po-210 in organic solids. The method is a controlled acid leach. Matrix effects must be determined on a case-by-case basis.

2.0 Method Summary

2.1 Polonium-210 is determined by alpha spectrometry by counting a plated source. Polonium is plated from a hydrochloric acid (HCl) solution by a galvanic deposition technique on Ni disk. The yield and efficiency are determined by use of a polonium tracer. Matrix effects will be determined on a case-by-case basis.

2.2 Because some forms of polonium are volatile, total dissolution is not attempted. Low temperature acid leach is employed to remove to the extent possible any polonium.

2.3 Use an appropriate aliquot, typically two grams, of undried sample. Add polonium tracer. The sample is acid leached by use of a series of additions of nitric acid and HF, with volume concentrations between each series. Add acid mixture carefully. Each new matrix must be treated with caution until the effect or magnitude of the sample/acid mixture reaction is known. Do not evaporate to dryness and do not boil solution. Repeat with nitric acid. Dilute to 90 mL volume and filter. To the supernate add HCl and enough ascorbic acid to complex any iron present and plate polonium onto specially-cleaned and polished Ni disks. Plating time should be five hours. Determine polonium by alpha spectrometry.

POLONIUM-210 IN AIR PARTICULATE FILTERS (PROCEDURE FM-RAD-0176) 3298

Working Linear Range: To be determined
Reporting Limit: Determined by number of filters and volume
Reporting Units: pCi/filter, pCi/mL, pCi/L, pCi/cc
Matrix: Filter(s)

1.0 Scope and Application

This method is applicable to the determination of polonium-210 (Po-210) on one or more air particulate filter(s). A controlled acid leach is used to remove polonium from the filter(s).

2.0 Method Summary

2.1 This procedure provides for the plating of Po-210 from a hydrochloric acid (HCl) solution onto nickel (Ni) disks. The polonium is counted by alpha spectrometry.

2.2 One or more air particulate filter(s) are selected for analysis. The sample is placed in a beaker with 150 mL of nitric and hydrofluoric (HF) acids and an appropriate amount of polonium tracer. Gently heat for six hours. Decant solution. Rinse sample twice with 50 mL portions of the acid mixture. Add concentrated HCl and gently concentrate to 20 mL solution. Add enough ascorbic acid to complex any iron and plate polonium onto specially-cleaned and polished Ni disks. Plating time should be 5 hours. Determine polonium by alpha spectrometry.

3298

RADON-222 IN AIR (GRAB) (PROCEDURE FM-RAD-0180)

Working Linear Range: ~0.5 pCi/L to >100,000 pCi/L
Reporting Limit: 4 pCi/L
Reporting Units: pCi/L
Matrix: Air

1.0 Scope and Application

This method is applicable to the direct determination of Rn-222 in air or other non-reactive gasses. The sample may be an instantaneous grab sample or an aliquot from an integrated sample collected over time in a Tedlar bag or other suitable container.

2.0 Method Summary

2.1 An alpha scintillation flask (Lucas Cell or George Cell) is evacuated and filled with the air sample. (Alternatively, the cell may be flushed with ten cell volumes or more of the air sample in flow-through mode.) After a minimum holding time of three hours in a dark chamber, the cell is counted directly on a suitable light-shielded PM tube connected to a scaler-ratemeter. The limit of detection is a function of the size and design of the scintillation flask. Volumes between 0.1 and 1 liters are commercially available.

3298

INTEGRATED RADON IN AIR BY ACTIVATED CARBON CANISTER (PROCEDURE FM-RAD-0182)

Working Linear Range: 0.5 pCi/L - Infinite
Reporting Limit: 4 pCi/L
Reporting Units: pCi/L
Matrix: Air

1.0 Scope and Application

This method is suitable for screening enclosed areas for Radon-222 levels in excess of EPA guidelines for dwellings.

2.0 Method Summary

2.1 A diffusion barrier activated carbon canister is placed in the environment to be screened for two to seven days, as recommended by the manufacturer. Ambient radon passing the diffusion barrier is absorbed onto a bed of activated carbon and is retained with high efficiency.

2.2 After exposure, the canister is sealed and held for at least three hours, then gamma counted within a week to ten days (some commercial systems employ vapor phase or liquid phase desorption and liquid scintillation counting as an alternative to gamma counting).

INTEGRATED RADON IN AIR BY PASSIVE ALPHA TRACK DETECTORS (PROCEDURE FM-RAD-0184)

Working Linear Range: 0.2 pCi/L - ~Infinite, (function of exposure time)
Reporting Limit: 4 pCi/L
Reporting Units: pCi/L
Matrix: Air

1.0 Scope and Application

This method is suitable for indoor and outdoor determinations of average Rn-222 concentrations over extended time periods.

2.0 Method Summary

2.1 Radon gas diffuses into a chamber through a filter designed to exclude alpha-emitting aerosols and Thoron gas. The chamber contains a sample of CR-39 film or related material that sustains alpha track damage proportional to the average radon concentration multiplied by the exposure time.

2.2 After exposure, the alpha track film is etched and the enlarged alpha tracks are examined microscopically and counted manually or automatically.

RADON PROGENY IN AIR BY MODIFIED KUSNETZ METHOD (PROCEDURE FM-RAD-0185)

Working Linear Range: Function of Air Volume
Reporting Limit: 0.1 WL
Reporting Units: Working Levels (WL)
Matrix: Air

1.0 Scope and Application

This method is suitable for indoor and outdoor determinations of Radon-222 progeny concentrations. Rn-220 progeny concentrations can also be determined with a modified counting protocol.

2.0 Method Summary

Breathing zone air is drawn through an open-faced membrane filter with a suitable air pump. The filter is counted on with an alpha scintillation counting system at a specified time after sampling. The count rate is converted to Working Levels of Radon progeny by correcting the rate using the instrument efficiency and by applying the time-dependent Kusnetz factor.

3298

RADON-222 IN WATER (PROCEDURE FM-RAD-0190)

Working Linear Range: 50 pCi/L to >10,000,000 pCi/L
Reporting Limit: 300 pCi/L
Reporting Units: pCi/L
Matrix: Water

1.0 Scope and Application

This method is applicable to determining the concentration of Rn-222 in water samples.

2.0 Method Summary

2.1 A 10 ml sample of water is drawn without aeration into a syringe, then promptly injected beneath a 10 ml layer of water-immiscible liquid scintillation solution in a glass liquid scintillation vial ("Environmentally Safe" scintillators based on DIPN or PXE are recommended). The phases are mixed by vigorous agitation, then allowed to separate. Radon originally in the aqueous phase is held in the scintillator phase.

2.2 After three hours, the vial is placed in a liquid scintillation counting system for analysis (for optimum results, a system equipped with alpha/beta discrimination circuitry should be used).

GROSS ALPHA IN SMEAR SAMPLES (PROCEDURE FM-RAD-0200)

Working Linear Range: One (1) smear
Reporting Limit: 1 pCi/smear
Reporting Units: pCi/smear, pCi/cm2
Matrix: Smear

1.0 Scope and Application

This method is applicable to the determination of gross alpha on one smear. The method is limited to direct counts with no correction for self absorption.

2.0 Method Summary

This procedure provides for the determination of gross alpha by counting the smear on a gas flow proportional counter or alpha scintillation counter to determine the number of alpha counts.

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GROSS BETA IN SMEAR SAMPLES (PROCEDURE FM-RAD-0210)

Working Linear Range: One (1) smear
Reporting Limit: 1 pCi/smear
Reporting Units: pCi/smear, pCi/cm2
Matrix: Smear

1.0 Scope and Application

This method is applicable to the determination of gross beta on one smear. The method is limited to direct counts with no correction for self absorption.

2.0 Method Summary

This procedure provides for the determination of gross beta by counting the smear on a gas flow proportional counter to determine the number of beta counts.

SOIL CLASSIFICATION (LAB) (PROCEDURE FM-GTT-0011)

3298

Applicable Standard: ASTM D 2487, Standard Test Method for Classification of Soils for Engineering Purposes

Exceptions

1.0 ASTM D 2487 Test Options

1.1 Separate samples into two fractions for preparation. The dry method of preparation of soil samples shall be used to determine grain size of one fraction and the wet method of preparation of soil samples to determine Atterberg limits of the other fraction.

1.2 Determine grain size analysis, including hydrometer analysis in accordance with Method No. FM-GTT-0031 (ASTM D 422) using dry method of preparation in accordance with Method No. FM-GTT-0015 (ASTM D 421).

1.3 Determine Atterberg limits in accordance with Method No. FM-GTT-0032 (ASTM D 4318) using wet method of preparation in accordance with Method No. FM-GTT-0014 (ASTM D 2217).

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) A, B, and C: Report all information specified in ASTM D 2487.

2.2 ASL D: Report all information specified in ASTM D 2487, and provide:

- Copies of all moisture calculations, including tare weights, wet weights, and dry weights of all containers
- Copies of all grain size calculations, including tare weights of sieves and total weight of material retained on each sieve
- Plot of percentage passing each sieve versus grain size, in millimeters, showing D_{10}, D_{30}, and D_{60} grain sizes
- Copies of all liquid limit, plastic limit, and plasticity index calculations
- Plot of moisture content versus number of blows of the liquid limit device

- Copies of calculations for coefficient of curvature (C_c) and coefficient of uniformity (C_u), where applicable

- Certification of calibration of Atterberg limit apparatus (Section 9 of ASTM D 4318) and apparatus used for grain size analyses (ASTM D 422)

- Demonstration of laboratory precision, as specified in Section 21.1 of ASTM D 4318

SOIL CLASSIFICATION (VISUAL) (PROCEDURE FM-GTT-0012)

3298

Applicable Standard: ASTM D 2488, Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)

Exceptions

1.0 Data Package Deliverables

1.1 Analytical Support Levels (ASLs) A and B: Report all information specified in ASTM D 2488.

1.2 ASLs C and D: The method provides qualitative information only and is not considered applicable for ASLs C and D.

2.0 Quality Control Requirements

2.1 ASL A: Provide visual field classification at the time of sample collection before placing sample in container or before sealing ends of Shelby tube samples.

2.2 ASL B: Verify visual field classification in the laboratory by a second visual classification by a technically qualified person.

TRANSPORTING SAMPLES (PROCEDURE FM-GTT-0013)

3298

Applicable Standard: ASTM D 4220, Standard Practices for Preserving and Transporting Soil Samples

Exceptions

1.0 ASTM D 4220 Method Options

1.1 Section 4.1.2: Group B samples are not to include thin-walled tubes. Thin-walled Shelby tube samples shall be Group C samples.

1.2 Section 8.3: Group B samples for moisture content and classification tests must be stored in glass jars with rubber-ringed lids or lids lined with a wax-coated paper seal.

1.3 Section 8.3: Group B samples for proctor tests and other bulk samples that will be remolded or compacted into sample molds are to be stored and transported in polyethylene inner bags with woven polypropylene outer bags.

1.4 Section 8.3.6: Thin-walled tubes are to be Group C samples and are to be sealed with plastic expandable packers and plastic end caps. Tubes stored for longer than seven days shall have the end caps sealed in wax.

2.0 Data Package Deliverables

2.1 Analytical Support Level (ASL) A: Report as a minimum the project number, sampling date, sample number, boring/sample location number, and sampling depth.

2.2 ASLs B, C, and D: Report all information specified in ASTM D 2487, plus the following:

- Description of packaging containers, shipping containers, and type of insulation, as well as a dimensioned sketch of the shipping container
- Sample identification/traceability record (controlled document) as in Figure 1 of ASTM D 4220 and chain-of-custody form

WET PREPARATION OF SAMPLES (PROCEDURE FM-GTT-0014)

3298

Applicable Standard: ASTM D 2217, Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants

Exceptions

1.0 ASTM D 2217 Method Options

Section 1.2: Use Procedure B only.

2.0 Data Package Deliverables

2.1 Analytical Support Level (ASL) A: No data package deliverable.

2.2 ASLs B, C, and D: Record sample preparation procedure on accompanying traceability record (Method No. FM-GTT-0013; ASTM D 4220). The laboratory report of subsequent analyses must include a brief description of sample preparation procedure and a copy of the traceability record.

DRY PREPARATION OF SAMPLES (PROCEDURE FM-GTT-0015)

3298

Applicable Standard: ASTM D 421, Standard Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants

Exceptions

1.0 Data Package Deliverables

1.1 Analytical Support Level (ASL) A: No data package deliverable.

1.2 ASLs B, C, and D: Record sample preparation procedure on accompanying traceability record (Method No. FM-GTT-0013; ASTM D 4220). The laboratory report of subsequent analyses must include a brief description of sample preparation procedure and a copy of the traceability record.

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MOISTURE CONTENT (PROCEDURE FM-GTT-0021)

Applicable Standard: ASTM D 2216, Standard Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures

Exceptions

1.0 ASTM D 2216 Test Options

1.1 Section 6.3, Note 2: Remove any large coarse-grained particle from test specimen when working with small, fine-grained soil sample.

1.2 Section 7.3, Note 4: Keep test specimens in drying oven for at least 16 hours.

1.3 Section 7.3, Note 5: Maintain oven-drying at 110° ± 5°C.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) A and B: Report sample identifying information and moisture content of specimen to nearest 0.1%.

2.2 ASL C: Report all information specified in ASTM D 2216, including moisture content of specimen to nearest 0.1%.

2.3 Analytical Support Level D: Report all information specified in ASTM D 2216, including moisture content of specimen to nearest 0.1%. Provide copies of all moisture calculations, including tare weights, wet weights, and dry weights of all containers.

MOISTURE CONTENT (MICROWAVE) (PROCEDURE FM-GTT-0022)

Applicable Standard: ASTM D 4643, Standard Test Method for Determination of Water (Moisture) Content of Soil by the Microwave Oven Method

Exceptions

1.0 ASTM D 4643 Test Options

1.1 Section 5.4: The method must not be used when the specimen contains more than 10% material coarser than the No. 4 sieve.

1.2 Section 5.6: Specimens used in the test must not be used for other tests subsequent to drying.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) A and B: Report sample identifying information and moisture content of specimen to nearest 0.1%.

2.2 ASL C: Report all information specified in ASTM D 4643.

2.3 ASL D: The method is not considered appropriate for Level D analytical support.

MOISTURE CORRECTION (OVERSIZE) (PROCEDURE FM-GTT-0023) 3298

Applicable Standard: ASTM D 4718, Standard Practice for Correction of Unit Weight and Water Content for Soils Containing Oversized Particles

Exceptions

1.0 ASTM D 4718 Method Options

Section 1.5: This practice shall be applied to samples containing a minimum of 5% of oversized particles.

2.0 Data Package Deliverables

2.1 Analytical Support Level (ASL) B and C: Report all information specified in ASTM D 4718.

2.2 ASLs C and D: Report all information specified in ASTM D 4718, and provide copies of all calculations.

SPECIFIC GRAVITY (PROCEDURE FM-GTT-OO24)

3298

Applicable Standard: ASTM D 854, Standard Test Method for Specific Gravity of Soils

Exceptions

1.0 ASTM D 854 Test Options

1.1 Section 5.1: The pycnometer should consist of a volumetric flask having a capacity of at least 100 mL.

1.2 Section 6.1, Note 3: Use distilled water as the wetting agent.

1.3 Section 7.1: The sample should consist of at least 25 grams of material on the basis of oven-dried weight.

1.4 Section 8.2: Remove trapped air from sample in pycnometer by boiling for ten minutes.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) B and C: Report all information specified in ASTM D 854.

2.2 ASL D: Report all information specified in ASTM D 854, and provide copies of all calculations.

VOLUME-WEIGHT RELATIONSHIPS (PROCEDURE FM-GTT-0025)

3298

Applicable Standard: COE Manual EM 1110-2-1906, Appendix II, Unit Weights, Void Ratio, Porosity, and Degree of Saturation

Exceptions

1.0 EM 1110-2-1906, Appendix II, Test Options

1.1 Section 3: The volumetric method shall be used. The following methods for obtaining a regularly-shaped mass may be used:

- Calibrated ring-shaped specimen cutter (cylinder)
- Cylindrical carving or trimming device (vertical lathe)
- Thin-walled tube specimens, with saw-cut square ends
- Cylindrical compaction mold (compacted specimen)

Prepare samples that contain gravel, shells, or foreign materials by carving or trimming, remove the foreign matter, and be careful filling voids on the surface of the specimen with remolded soil from the trimmings. The diameter of the trimmed specimen must be at least four times the largest dimension of the pebble or shell.

1.2 Section 3.c.2: Make at least three height measurements and nine diameter measurements to determine the average height and diameter of the cylinder for each sample.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) A and B: Report all identifying information and unit weight, void ratio, porosity, and degree of saturation for the sample.

2.2 ASL C: Report all information specified in paragraph 2.1 above and all sample identifying information, and provide copies of original data logs (ENG Form No. 3836, Plate II-1, or equivalent).

2.3 ASL D: Submit all information specified in Paragraph 2.1 above and all sample identifying information, and provide copies of original data logs (ENG Form No. 3836, Plate II-1, or equivalent), all calculations, all tare weights, wet weights, and dry weights of all containers, and certification of calibration for all scales and calipers.

GRAIN SIZE ANALYSIS (PROCEDURE FM-GTT-0031)

3298

Applicable Standard: ASTM D 422, Standard Method for Particle Size Analysis of Soils

Exceptions

1.0 ASTM D 422 Test Options

1.1 Section 3.2: Stirring Apparatus A must be used.

1.2 Section 3.6: A full set of sieves must be used. A No. 100 sieve may be substituted for the No. 140 sieve.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASL) A and B: Report sample identifying information and results of grain-size distribution in tabular form (grain size and corresponding percent passing) and graphic form (grain size versus percent passing).

2.2 Analytical Support Level C and D: Report all information specified in ASTM D 422, with results presented in both tabular and graphic form.

2.3 ASL D: Report all information specified in ASTM D 422, and provide:

- Results in both tabular and graphic form
- Tabular summary of moisture data, sieve analysis data (wash sieve analysis, tare weights, soil weights, etc.), and hydrometer analysis data
- Copies of laboratory data sheets and other calculations not included in the tabular summary, including hydroscopic moisture correction, percentage passing each fraction, percentages in suspension, diameters of soil particles, and other calculations
- Copies of all scale, hydrometer, and sieve calibration

ATTERBERG LIMITS (PROCEDURE FM-GTT-0032)

3298

Applicable Standard: ASTM D 4318, Standard Test Method for Liquid Limit, Plastic Limit, and Plasticity Index of Soils

Exceptions

1.0 ASTM D 4318 Test Options

Section 1.1.1: The test procedure for performing the liquid limit must be in accordance with Procedure A, multipoint test using a wet preparation procedure, as described in Sections 10.1, 11, and 12 of ASTM D 4318.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) A and B: Report sample identifying information, and liquid limit, plastic limit, and plasticity index rounded to the nearest whole number.

2.2 ASL C: Report all information specified in ASTM D 4318.

2.3 ASL D: Report all information specified in ASTM D 4318, and provide:

- Copies of all moisture calculations, including tare weights, wet weights, and dry weights of all containers
- Plot of moisture content versus number of blows of liquid limit device
- Certification of calibration of apparatus as specified in Section 9 of ASTM D 4318
- Demonstration of laboratory precision as specified in Section 21.2 of ASTM D 4318

SHRINKAGE LIMIT (PROCEDURE FM-GTT-0033)

3298

Applicable Standard: ASTM D 427, Standard Test Method for Shrinkage Factors of Soils

Exceptions

1.0 Data Package Deliverables

1.1 Analytical Support Levels (ASLs) A, B, and C: Report sample identifying information and calculated shrinkage factors. Report moisture content, shrinkage limit, volumetric shrinkage, and linear shrinkage rounded to the nearest whole number. Report shrinkage ratio and specific gravity rounded to the nearest hundredth of a unit.

1.2 ASL D: Report the information required in paragraph 1.1, and provide:

- Copies of all moisture calculations, including tare weights, wet weights, and dry weights of all containers
- Copies of all other calculations
- Certification of calibration of applicable apparatus (balance, graduates, etc.)

2.0 Quality Control Requirements

2.1 ASLs A and B: Conduct one test per sample (single soil pat).

2.2 ASLs C and D: Conduct two tests per sample (two soil pats).

STANDARD PROCTOR (PROCEDURE FM-GTT-0041)

3298

Applicable Standard: ASTM D 698, Standard Test Methods for Moisture Density Relations of Soils and Soil-Aggregate Mixtures Using 5.5-lb (2.49-kg) Rammer and 12-in. (305-mm) Drop

Exceptions

1.0 ASTM D 698 Test Options

1.1 Section 1.1: Method D shall be used.

1.2 Section 3.2: The mechanical rammer must be used and must be calibrated before each use.

1.3 Section 4.1: The dry preparation procedure must be used unless otherwise specified.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) A and B: Report the sample identifying information and all information specified in ASTM D 698, Section 8.

2.2 ASL C: Report the information specified in paragraph 2.1, and provide a graph of the moisture-density relationship per ASTM D 698, Section 7.

2.3 ASL D: Report all information specified in paragraph 2.1, and provide:

- Graph of the moisture-density relationship per ASTM D 698, Section 7, showing the location of the optimum moisture content and maximum density and including the zero air voids curve. Report specific gravity as determined in accordance with Method No. FM-GTT-0024 (ASTM D 854)

- Calibration of mold volume in accordance with Section 3.1.3 of ASTM D 698

- Calibration of rammer weight, diameter, and fall distance in accordance with Section 3.2.3 of ASTM D 698

- Copies of all moisture calculations (including tare weights, wet weights, and dry weights of all materials), density calculations (including weights and volumes), and correction factors

- Demonstration of laboratory precision in accordance with Section 9 of ASTM D 698

MODIFIED PROCTOR (PROCEDURE FM-GTT-0042)

3298

Applicable Standard: ASTM D 1557, Standard Test Methods for Moisture Density Relations of Soils and Soil-Aggregate Mixtures Using 10-lb (4.54-kg) Rammer and 18-in. (457-mm) Drop

Exceptions

1.0 ASTM D 1557 Test Options

1.1 Section 1.1: Method D shall be used.

1.2 Section 3.2: The mechanical rammer shall be used, and shall be calibrated prior to each use.

1.3 Section 4.1: The dry preparation procedure shall be used unless otherwise specified.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) A and B: Report the sample identifying information and all information specified in ASTM D 1557, Section 8.

2.2 ASL C: Report the information specified in paragraph 2.1, and provide a graph of the moisture-density relationship per ASTM D 1557, Section 7.

2.3 ASL D: Report all information specified in paragraph 2.1, and provide:

- Graph of the moisture-density relationship per ASTM D 1557, Section 7, showing the location of the optimum moisture content and maximum density, and including the zero air voids curve. Report also the specific gravity, as determined in accordance with Method No. FM-GTT-0024 (ASTM D 854)
- Calibration of mold volume, in accordance with Section 3.1.3 of ASTM D 1557
- Calibration of rammer weight, diameter, and fall distance, in accordance with Section 3.2.3 of ASTM D 1557
- Copies of all moisture calculations (including tare weights, wet weights, and dry weights of all materials), density calculations (including weights and volumes), and correction factors
- Demonstration of laboratory precision, in accordance with Section 9 of ASTM D 1557

MAXIMUM (RELATIVE) DENSITY (PROCEDURE FM-GTT-0043)

3298

Applicable Standard: ASTM D 4253, Standard Test Methods for Maximum Index Density of Soils Using a Vibratory Table

Exceptions

1.0 ASTM D 4253 Test Options

1.1 Section 1.3.2: Use the dry preparation (Method A) procedure unless otherwise specified.

1.2 Section 4.4: Double amplitudes of vibration other than 0.013 ± 0.002 inch @ 60 Hz or 0.019 ± 0.003 inch @ 50 Hz must not be used unless otherwise specified.

1.3 Section 6: Standard molds must be used.

1.4 Section 8.4: The moisture content of the field sample must be determined in accordance with Method No. FM-GTT-0021 (ASTM D 2216).

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) A, B, and C: Report sample identifying information, all information specified in ASTM D 4253 Section 13, and the minimum void ratio and moisture content of the sample.

2.2 ASL D: Report the items described in paragraph 2.1, and provide:

- Copies of laboratory data sheets showing calculations per ASTM D 4253, Section 12
- Copies of laboratory data sheets showing moisture content calculations
- Certification of calibration of apparatus used to perform the test per ASTM D 4253, Section 10
- Certification of calibration of other test equipment used to perform the test (sieves, scales, etc.)
- Demonstration of laboratory precision, as specified in Section 14 of ASTM D 4253

MINIMUM (RELATIVE) DENSITY (PROCEDURE FM-GTT-0044)

3298

Applicable Standard: ASTM D 4254, Standard Test Methods for Minimum Index Density of Soils and Calculation of Relative Density

Exceptions

1.0 ASTM D 4254 Test Options

1.1 Section 1.3: Method A, using a funnel pouring device or hand scoop to place material in a mold, must be used.

1.2 Section 6.3: Standard molds must be used.

1.3 Section 7.5: Determine moisture content of the field sample in accordance with Method No. FM-GTT-0021 (ASTM D 2216).

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) A, B, and C: Report the sample identifying information, all information specified in ASTM D 4254 Section 11, and the maximum void ratio and moisture content of the sample.

2.2 ASL D: Report the items described in paragraph 2.1, and provide:

- Copies of laboratory data sheets showing calculations per ASTM D 4254, Section 10
- Copies of laboratory data sheets showing moisture content calculations
- Certification of calibration of apparatus used to perform the test per ASTM D 4254 Section 8
- Certification of calibration of other test equipment used to perform the test (sieves, scales, etc.)
- Demonstration of laboratory precision, as specified in Section 12 of ASTM D 4254

CONSOLIDATION (PROCEDURE FM-GTT-0051)

3298

Applicable Standard: ASTM D 2435, Standard Test Methods for One-dimensional Consolidation Properties of Soils

Exceptions

1.0 ASTM D 4235 Test Options

1.1 Section 1: Method B shall be used. Successive load increments must not be applied in time increments less than 24 hours.

1.2 Section 11.4: The test must include one unload-reload cycle; the unload-reload cycle must not be initiated until at least two load increments after the preconsolidation pressure have been applied. The final load following the unload-reload cycle must be greater than eight times the preconsolidation pressure. Specific loading and unloading increments must be at the direction of the project geotechnical engineer.

1.3 Section 11.4.2: The unloading load increments must be equal to those used in loading but in reverse order.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) B and C: Report the sample identifying information and all information specified in ASTM D 2435 Section 13. Provide both a graph of deformation versus log time and a graph of deformation versus square root of time. Provide both a graph of void ratio versus log of pressure and a graph of percent compression versus log of pressure.

2.2 Analytical Support Level D: Report the items described in paragraph 2.1, and provide:

- Copies of all moisture calculations, including tare weights, wet weights, and dry weights of all containers
- Copies of calculations for coefficient of consolidation, void ratio, and vertical stress for each load increment
- Graphical derivation of t_{100} from the plot of deformation versus log of time and graphical derivation of t_{90} from the plot of deformation versus square root of time

- Graphical derivation of preconsolidation pressure, consolidation index, C_c, and reconsolidation index, C_r, from the plot of void ratio versus log of pressure

- Certification of calibration of apparatus used to perform the test per ASTM D 4235 Section 7

- Certification of calibration of other test equipment used to perform the test (sieves, scales, etc.)

UNCONFINED COMPRESSION (PROCEDURE FM-GTT-0061)

Applicable Standard: ASTM D 2166, Standard Test Method for Unconfined Compressive Strength of Cohesive Soil

Exceptions

1.0 ASTM D 2166 Test Options

1.1 Section 1.3 and Section 6.4, Note 5: This test method is applicable only to cohesive soils with a degree of saturation less than 90%.

1.2 Section 4.3, Note 2: Soils that cannot retain their shape shall not be tested.

1.3 Section 6.1: Take three height measurements 120° apart, and take three width measurements at the quarter points of the height.

1.4 Section 6.2: Trim and square the ends of all tube samples.

1.5 Section 7.1: All specimens must be tested to failure. If failure occurs in less than 5 minutes or after 15 minutes, the unconfined compression test results shall be rejected and the sample retested.

2.0 Data Package Deliverables

2.1 Analytical Support Level (ASL) A: Report all sample identifying information and unconfined compressive strength and shear strength.

2.2 ASL B: Report all sample identifying information, unconfined compressive strength, shear strength, and a plot of stress versus strain.

2.3 ASL C: Report all sample identifying information and all information specified in ASTM D 2166, including a plot of stress versus strain.

2.4 Analytical Support Level D: Report all sample identifying information and all information specified in ASTM D 2166, and provide:

- Copies of all moisture calculations, including tare weights, wet weights, and dry weights of all containers
- Copies of all measurements made, including laboratory logs and curves

- Copies of all axial strain calculations, including length measurements and cross-sectional area measurements
- Copies of all unconfined compressive strength calculations
- Copies of all degree of saturation calculations, including all information specified in (ASTM D 854) for specific gravity
- Copies of all sensitivity calculations, including data on remolded strength determination
- Certification of calibration of compression device

DIRECT SHEAR (CONTROLLED-DISPLACEMENT METHOD) (PROCEDURE FM-GTT-0062)

3298

Applicable Standard: ASTM D 3080, Direct Shear Test of Soils under Consolidated Drained Conditions (Controlled-Displacement Method)

Exceptions

1.0 ASTM D 3080 Test Options

1.1 This procedure applies to the controlled-displacement method only, where both ultimate stress and maximum stress can be determined.

1.2 Section 1.1: The direct shear test must be conducted in single shear only.

1.3 Section 4.1: The shear device must be round. Square devices may be used only when the soil material type is too large to be compatible with the standard shear device and only when special approval has been obtained from the project geotechnical engineer.

1.4 Section 4.3.2: The load-indicating device must be a proving ring only.

1.5 Section 5.2: Prepare remolded samples as follows:

- Compact cohesive samples in at least three equivalent layers in an oversized mold and trim to inside dimension of direct shear device
- Compact noncohesive samples in at least three equivalent layers directly in the direct shear device
- The dry density of all compacted specimens must be within 1% of the target dry density specified in the sampling and analysis plan

1.6 Section 5.3: The sample must be at least two inches wide and at least one inch thick, and the ratio of width to thickness must be at least 2:1 and no more than 3:1.

1.7 Section 7.1, Note 4: The porous stone shall be dampened only for saturated, undisturbed samples taken from below the water table.

1.8 Section 7.2: The final normal force for each test specimen must be as specified in the sampling and analysis plan. Normal force must be applied in four increments so that the respectively applied loads are equal to $^1/_8$ the final load, $^1/_4$ the final load, $^1/_2$ the final load, and the final load.

1.9 Section 7.3: The rate of shear must be held constant throughout the test. The proposed rate must be determined approximately by dividing the estimated shear deformation at maximum shear stress by the computed time to failure. The estimated shear deformation at maximum shear stress must be estimated by the project geotechnical engineer based on consideration of grain size, plasticity, moisture content, stiffness, and other factors. The actual time to failure must be no less than $25t_{50}$ and no greater than $100t_{50}$. If actual time to failure exceeds these limits, the test shall be repeated for that normal stress specimen.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) A and B: Report the sample identifying information, and provide a plot of shear stress versus displacement for each normal stress increment (test specimen) and a plot of shear stress versus normal stress showing both maximum and ultimate shear stress.

2.2 ASL C: Report all information specified in ASTM D 3080, and provide plot of ultimate shear stress versus normal stress and plots of consolidation (normal displacement over time under each normal stress increment).

2.3 ASL D: Report all information specified in ASTM D 3080, and provide:

- Copies of all moisture calculations, including tare weights, wet weights, and dry weights of all containers, and including void ratio before and after consolidation and after shear for each test specimen, showing initial and final degrees of saturation for each test specimen

- Copies of all stress data calculations, including normal stress and shear stress, and calculation of t_{50}, time to failure, and constant rate of shear

- Plot of shear stress versus normal stress, showing both maximum and ultimate shear stress and estimated slope angle

- Plot of consolidation under each normal stress increment (normal displacement versus time) and showing the applicable t_{50} times

- Certification of calibration of the single shear direct-shear device as specified in ASTM D 3080 Section 6, and certification of proving ring calibration

- Description of the type of shear device, including the porous stone size, permeability, and dampening process
- Demonstration of technician training and experience for method validation

DIRECT SHEAR (CONTROLLED-STRESS METHOD) (PROCEDURE FM-GTT-0063)

Applicable Standard: ASTM D 3080, Direct Shear Test of Soils under Consolidated Drained Conditions (Controlled-Stress Method)

Exceptions

1.0 ASTM D 3080 Test Options

1.1 The procedure applies to the controlled-stress method, where the maximum shear stress can be approximately determined.

1.2 The analyst must comply with all test options specified for Method No. FM-GTT-0062, except that the shear-force load-indicating device and constant rate of shear are not required.

1.3 Section 7.3: The initial shearing force increments must be approximately 10% of the estimated maximum shear force and reduced as specified in ASTM D 3080. The project geotechnical engineer must estimate maximum shear force (failure force) based on consideration of applied normal load, grain size, plasticity, moisture content, stiffness, and other factors. The shearing force increment applied just prior to the failure load must be less than 2.5% of the maximum shear force. If the actual increment exceeds these limits, the test must be repeated for that normal stress specimen.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) A and B: Report the sample identifying information, and provide a plot of shear stress versus displacement for each normal stress increment (test specimen) and a plot of maximum shear stress versus normal stress.

2.2 ASL C: Report all information specified in ASTM D 3080, including plots of consolidation (normal displacement over time under each normal stress increment) and plots of consolidation under each shear stress increment (shear displacement over time).

2.3 ASL D: The procedure is not considered appropriate for Level D analytical support.

TRIAXIAL COMPRESSION (UU) (PROCEDURE FM-GTT-0064)

Applicable Standard: ASTM D 2850, Standard Test Method for Unconsolidated, Undrained Compressive Strength of Cohesive Soils in Triaxial Compression

Exceptions

1.0 ASTM D 2850 Test Options

1.1 Section 6.3: The dry density of any remolded specimen must be within 1% of the dry density determined for the original, undisturbed specimen.

1.2 Section 6.4: The dry density of all compacted specimens must be within 1% of the target dry density as specified in the Sampling and Analysis Plan.

1.3 Section 8.5: Latex membranes shall be used. The effect of the membrane on the lateral stress may be assumed to be negligible.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) A and B: Report the sample identifying information, the initial dry unit weight and moisture content of the sample, the value of the deviator stress, and the value of the minor and major principal total stresses at failure.

2.2 ASL C: Report all information specified in ASTM D 2850.

2.3 ASL D: Report all information specified in ASTM D 2850 and provide:

- Copies of all moisture calculations, stress calculations, strain calculations, and other calculations, including measurements of all dimensions, weights, volumes, or other units
- Copies of all logs and notes taken during testing
- Plots of the stress-strain curve (deviator stress versus axial strain) for each specimen showing maximum deviator stress and axial strain at failure and tangential and secant Young's moduli
- A single plot of the Mohr stress circles for all specimens of the one sample based on total stresses, showing minor and major principal stresses and maximum deviator stress

TRIAXIAL COMPRESSION (CU) (PROCEDURE FM-GTT-0065)

Applicable Standard: ASTM D 4767, Standard Test Method for Consolidated-Undrained Triaxial Compression Test on Cohesive Soils

Exceptions

1.0 ASTM D 4767 Test Options

1.1 Section 6.3: The dry density of all compacted specimens must be within 1% of the target dry density as specified in the Sampling and Analysis Plan.

1.2 Section 7.2, Note 12: The wet mounting method must be used for all test specimens. Record latex membrane thickness and combined height of cap, base, porous discs, and filter discs, and determine specimen dimensions after the specimen has been mounted.

1.3 Section 8.2.3: Back pressure increments shall be at the judgment of the analyst in consultation with the project geotechnical engineer.

1.4 Section 10.2.2: Calculate the cross-sectional area of the specimen after consolidation using both Method A and Method B. The average of both methods shall be used in subsequent calculations. If the value calculated by both methods differs by more than 5%, the error must be noted in the data package.

1.5 Section 5.3: Filter paper discs may be used, but filter paper strips must not be used.

1.6 Section 10.7.1: Latex membranes must be used. The effect of the membrane on the lateral stress may be assumed to be negligible.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) A and B: The procedure is not considered appropriate for Levels A and B analytical support.

2.2 ASL C: Report all information specified in ASTM D 4767.

2.3 ASL D: Report all information specified in ASTM D 4767 and provide:

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- Copies of all moisture calculations, stress calculations, strain calculations, back pressure calculations, and other calculations including measurements of all dimensions, weights, volumes, or other units

- Copies of all logs and notes taken during testing

- Plots of consolidation (volume change versus time) for each specimen

CALIFORNIA BEARING RATIO (PROCEDURE FM-GTT-0066)

Applicable Standard: ASTM D 1883, Standard Test Method for CBR (California Bearing Ratio) of Laboratory-Compacted Soils

Exceptions

1.0 ASTM D 1883 Test Options

1.1 Section 1.6: All specimens shall be soaked before penetration.

1.2 Section 7.2.4: Allow all specimens to soak for 96 hours.

1.3 Section 8.2, Note 4: Do not attach strain gauge to testing machine support bars.

2.0 Data Package Deliverables

2.1 Analytical Support Level (ASL) A: Report the sample identifying information, calculated California Bearing Ratio, and the density at which the test was performed.

2.2 ASL B: Report the sample identifying information plus:

- Dry density of sample before and after soaking
- Moisture content of sample in percent before and after compaction
- Bearing ratio of sample

2.3 ASL C: Report the sample identifying information and all information specified in ASTM D 1883, Section 10.

2.4 ASL D: Report the information specified in paragraph 2.3, and provide:

- Percent shrink/swell change during soaking
- Copies of all calculations
- Copies of all tare weights, wet weights, and dry weights
- Copies of all curve plots for each specimen

- Certification of calibration of apparatus used to perform the test per ASTM D 1883 Sections 7 and 8

- Certification of calibration of other test equipment used to perform the test (i.e., sieves, scales, etc.)

PERMEABILITY (CONSTANT HEAD) (PROCEDURE FM-GTT-0071)

Applicable Standard: ASTM D 2434, Standard Test Method for Permeability of Granular Soils (Constant Head)

Exceptions

1.0 ASTM D 2434 Test Options

1.1 Section 4.2, Note 1: Do not use tap water as permeant. Use de-ionized, de-mineralized, and de-aired water.

1.2 Section 6.5: "Minimum" and "maximum" densities are to be reported as "loose" and "compact" densities and are not to be confused with Method Nos. FM-GTT-0043 and FM-GTT-0044 (ASTM D 4253 and D 4254) to determine relative density. The dry density of compacted specimens for each run must be within 10% of the targeted dry density.

1.3 Section 7.2: Begin the initial test run at a head difference of 0.5 cm, and increase by 0.5 cm for subsequent runs in the laminar flow range and 1.0 cm for subsequent runs in the turbulent flow range. The test shall be stopped after making 10 successive runs in the turbulent flow range.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) A and B: Report the sample identifying information, the dry unit weight and moisture content of each run specimen, and the value of the coefficient of permeability in the laminar flow range of each run.

2.2 ASL C: Report all information specified in ASTM D 2434. Include plots of velocity versus hydraulic gradient for each run and a plot of the coefficient of permeability versus dry density.

2.3 ASL D: Report all information specified in ASTM D 2434, and provide:

- Copies of all moisture calculations and other calculations used to determine temperature correction, velocity, gradient, and permeability (include measurements of all dimensions, weights, volumes, temperature, or other units)

- Copies of all logs and notes taken during testing 1
- Plots of velocity versus hydraulic gradient for each run, and 2
  coefficient of permeability versus hydraulic gradient for each run 3
- A plot of the coefficient of permeability versus dry unit weight and 4
  a plot of permeability versus void ratio, indicating "loose" and 5
  "compact" relative compaction 6

PERMEABILITY (TRIAXIAL) (PROCEDURE FM-GTT-0072)

Applicable Standard: ASTM D 5084, Standard Test Method for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible-Wall Permeameter

Exceptions

1.0 ASTM D 5084 Test Options

1.1 Section 5.1: Constant head test (Method A) shall be used.

1.2 Section 6.1.2, Note 4: Permeant solution shall be 0.005 N $CaSO_4$, prepared by dissolving $CaSO_4$ in de-aired, distilled water.

1.3 Section 8.3.3: Verify back-pressure saturation by measuring the B coefficient in accordance with Section 8.3.3.1.

1.4 Section 8.4: Consolidate the specimen to the effective stress specified by the project geotechnical engineer.

1.5 Section 8.5.1: The hydraulic gradient shall not exceed ten.

1.6 Section 8.5.3: Continue permeation until at least two pore volumes of permeant have flowed through the sample.

2.0 Data Package Deliverables

2.1 Analytical Support Level (ASL) B: Report the sample identifying information, the dry unit weight and moisture content of each run specimen, and the average hydraulic conductivity of the last four determinations.

2.2 ASL C: Report all information specified in ASTM D 5084, including plots of hydraulic conductivity versus time and hydraulic conductivity versus pore volume, and measurement of inflow and outflow rates throughout the test.

2.3 ASL D: Report all information specified in ASTM D 5084 and provide:

- Copies of all moisture calculations, stress calculations, back-pressure calculations, and other calculations used to determine gradient and hydraulic conductivity, including measurements of all dimensions, weights, volumes, or other units

- Copies of all logs and notes taken during testing
- Plots of hydraulic conductivity versus time and hydraulic conductivity versus pore volume
- Certification of calibration of all equipment, gauges, scales, etc.

BTU CONTENT (SOLIDS) (PROCEDURE FM-GTT-0082)

Applicable Standard: ASTM D 2015, Standard Test Method for Gross Calorific Value of Coal and Coke by the Adiabatic Bomb Calorimeter

Exceptions

1.0 ASTM D 2015 Test Options

1.1 Section 1.1: The method may also be applied to determine the gross calorific value of organic soils and organically-contaminated soils.

1.2 Section 8.1: The sample must not be pulverized nor screened to pass the No. 40 sieve. Keep sample sealed to avoid loss or volatilization of organics until inserted into the calorimeter.

1.3 Section 8.2: Moisture content shall be determined in accordance with Method No. FM-GTT-0021 (ASTM D 2216).

1.4 Section 8.3: Sulfur analysis shall not be performed.

1.5 Section 11.1: It will be necessary to recalibrate the calorimeter so that the water equivalent will be based on the same temperature rise as that obtained with the sample.

1.6 Section 12.1: Do not mix the sample. Take special precaution to avoid loss or volatilization of organics within the soil sample.

1.7 Section 12.2, Note 8: Do not use asbestos lining method. Increase the mass of the sample or add benzoic acid as specified.

1.8 Section 13.2: Do not correct for difference between heat of formation of sulfuric acid and nitric acid, but include correction for the heat of combustion of benzoic acid if used to obtain a complete burn.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASLs) A, B, and C: Report the sample identifying information and gross calorific value per dry weight basis (Btu/lb).

2.2 ASL D: Provide:

- Sample identifying information

- Gross calorific value per dry weight basis (Btu/lb)
- Demonstration of equipment calibration, including calorimeter heat rise during mixing, thermometer accuracy, and pressure-gauge accuracy
- Demonstration of reagent purity, including water and oxygen purity
- Demonstration of restandardization procedure, including dates of restandardization, latest oxygen supply change, and latest change to calorimeter
- Oxygen pressure measured for all tests
- Copies of all calculations, including correction of water volumes for density changes due to temperature, correction for combustion of benzoic acid if used to obtain complete combustion, other thermochemical or thermometric corrections, and calculation of gross calorific value

ASH CONTENT (PROCEDURE FM-GTT-0083)

Applicable Standard: ASTM D 3174, Standard Test Method for Ash in the Analysis Sample of Coal and Coke from Coal

Exceptions

1.0 ASTM D 3174 Test Options

1.1 Section 7.2: The alternative method of drying shall not be used.

1.2 Section 7.3, Note 3: All samples will remain in the incinerator for at least three hours and until a constant weight (± 0.001 gram) has been maintained.

2.0 Data Package Deliverables

2.1 Analytical Support Levels (ASL) A, B, and C: Report all sample identifying information and the percentage of ash content, rounded to the nearest 1%.

2.2 ASL D: Report all sample identifying information and provide:

- Copies of all calculations, including measurements of tare weights, wet weights and dry weights
- Copies of all laboratory logs and notes, including temperature readings over time
- Certification of calibration of furnace temperature, scales, sieve, etc.

ORGANIC CONTENT (PROCEDURE FM-GTT-0084)

Applicable Standard: ASTM D 2974, Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils

Exceptions

1.0 ASTM D 2974 Test Options

1.1 Section 2.3: Method C, igniting the oven-dried sample from the moisture content determination in a muffle furnace, shall be used.

1.2 Section 3.2: The muffle furnace must be capable of producing a constant temperature of 440°C.

1.3 Section 9.3: Hold the furnace temperature for at least four hours, or until there is no change of mass after further heating.

2.0 Data Package Deliverables

2.1 ASL A: Report the sample-identifying information and moisture content, ash content, and organic matter content, rounded to the nearest 1%.

2.2 ASLs B and C: Report the sample identifying information and all information specified in ASTM D 2974.

2.3 ASL D: Report all sample-identifying information and all information specified in ASTM D 2974 and provide:

- Copies of all calculations, including measurements of tare weights, dry weights, and wet weights
- Copies of all laboratory logs and notes, including temperature readings over time
- Certification of calibration of furnace temperature and scales