

ANALYTICAL METHOD SUMMARIES

Analyte	Method	Reference Method
Organics		
Per- and Polyfluorinated Alkyl Substances (PFASs) in Water	<p>A 250-mL water sample is fortified with surrogates and passed through a solid phase extraction (SPE) cartridge containing to extract the method analytes and surrogates. The compounds are eluted from the solid phase with a small amount of methanol. The extract is concentrated to dryness with nitrogen in a heated water bath, and then adjusted to a 1-mL volume with 96:4% (vol/vol) methanol:water after adding the IS(s). A 10-μL injection is made into an LC equipped with a C18 column that is interfaced to an MS/MS. The analytes are separated and identified by comparing the acquired mass spectra and retention times to reference spectra and retention times for calibration standards acquired under identical LC/MS/MS conditions. The concentration of each analyte is determined by using the internal standard technique. Surrogate analytes are added to all Field and QC Samples to monitor the extraction efficiency of the method analytes. Quantification of linear and branched isomers is conducted as a single total response using the relative response factor for the corresponding linear standard.</p>	<p>USEPA Method 537 Determination of Selected Perfluorinated Alkyl Acids in Drinking Water By Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS) Version 1.1 September 2009</p>
Per- and Polyfluorinated Alkyl Substances (PFASs) in Soils	<p>The sample is homogenised, spiked with the labeled compound spiking solution and digested with 1M NaOH by heating and ultrasonic agitation, followed by overnight incubation. Samples are neutralized with HCl and extracted with solvent. Solvent extraction and cleanup is then performed using 50:50/ACN:MeOH (v/v) and then cleaned up using a solid-phase extraction (SPE) cartridge.</p> <p>A 10-μL injection is made into an LC equipped with a C18 column that is interfaced to an MS/MS. The analytes are separated and identified by comparing the acquired mass spectra and retention times to reference spectra and retention times for calibration standards acquired under identical LC/MS/MS conditions. The concentration of each analyte is determined by using the internal standard technique. Surrogate analytes are added to all Field and QC Samples to monitor the</p>	<p>USEPA Method EPA-821-R-11-007 Draft Procedure for Analysis of Perfluorinated Carboxylic Acids and Sulfonic Acids in Sewage Sludge and Biosolids by HPLC/MS/MS December 2011</p>

Analyte	Method	Reference Method
	extraction efficiency of the method analytes. The recoveries of the labeled analogs themselves are determined by internal standard quantitation and used as a quality control check on the overall analytical process. Quantification of linear and branched isomers is conducted as a single total response using the relative response factor for the corresponding linear standard.	
TRH (Volatile)/BTEX C6-C10 – 2013 NEPM Fractions C6-C9 – 1999 NEPM Fractions	<p>10g soil extracted with 20mL methanol, tumbled for 1 hour, and analysed with solvent and instrument check surrogates. Clay samples must be completely disintegrated before an aliquot is taken for analysis. Water direct injection of supplied sample (unopened) and analysis with solvent and instrument check surrogates. Analysis by capillary column Purge and Trap GCMS (Eurofins mgt in-house method numbers: Total Recoverable Hydrocarbons (TPH), Method: LTM-ORG-2010, Method: LTM-GEN-7080 Moisture).</p> <p>Owing to the differential responses of mass spectrometric detectors towards aliphatic and aromatic compounds, it is essential that the standard contain representatives of both groups. This standard should therefore consist of about 40% aromatic and 60% aliphatic target analytes, to be representative of a typical Australian fuel. The aromatic compounds shall comprise the components of BTEX. The aliphatics shall comprise equal proportions of all n-alkanes in the C6 to C10 range.</p>	USEPA Method 8260B NEPM Appendix 1: Determination of total recoverable hydrocarbons (TRH) in soil
Total Recoverable Hydrocarbons C10- C36 – 1999 NEPM Fractions >C10-C40 – 2013 NEPM Fractions	<p>Soil - 10g soil and anhydrous sodium sulfate extracted with 20mL dichloromethane/acetone (1:1), and tumbled for a minimum of 1 hour. Clay samples must be completely disintegrated before an aliquot is taken for analysis.</p> <p>Water - One 250ml of water sequentially extracted in a separatory funnel three times with 20mL dichloromethane.</p> <p>Analysis by capillary column GC/FID (Eurofins mgt in-house method numbers: Total Recoverable Hydrocarbons (TRH), Method: LTM-ORG-2010, Method: LTM-GEN-7080 Moisture)</p>	USEPA Method 8015C NEPM Appendix 1: Determination of total recoverable hydrocarbons (TRH) in soil
TRH (Silica Gel)	Sample extracts obtained from the appropriate TRH method are exchanged to	USEPA Method 3630C

Analyte	Method	Reference Method
	a non-polar solvent and are passed through a column containing 1 gram of 100% activated silica gel. Elution is achieved with a small volume of 1:1 DCM:pentane or 1:1 DCM:hexane. The eluted solvent is then concentrated and analysed by the appropriate TRH analysis procedure. A decanoic acid reverse surrogate is used to provide assurance of the effectiveness of the silica-gel clean-up.	NEPM Appendix 1: Determination of total recoverable hydrocarbons (TRH) in soil
VOCs	10g soil extracted with 20mL methanol, tumbled for 1 hour, and analysed with solvent and instrument check surrogates. Clay samples must be completely disintegrated before an aliquot is taken for analysis. Water direct injection of supplied sample (unopened) and analysis with solvent and instrument check surrogates. Analysis by capillary column Purge and Trap GC-MS (Eurofins mgt in-house method numbers Method: LTM-ORG-2150, LTM-ORG-2160, Method: LTM-GEN-7080 Moisture).	US EPA Method 8260B
Phenols/PAH/PCBs /OPP/OCs	Soil - 10g soil, surrogates, mixed with anhydrous sodium sulfate and extracted with 20mL dichloromethane/acetone (1:1), and tumbled for a minimum of 1 hour. Clay samples must be completely disintegrated before an aliquot is taken for analysis. Water - 250ml water sample plus surrogates triple extracted with dichloromethane (base and neutrals). Leachate - 250ml water sample plus surrogates triple extracted with dichloromethane (base and neutrals). Analysis by capillary column GC/MS (Eurofins mgt in-house Methods E008.1, E008.2, E015.1, E015.2, E017.1 and E017.2, E016.1, E016.2, E017.1 and E017.2, E007.1, E007.2, E015.1, E015.2, E017.1 and E017.2 Method: LTM-GEN-7080 Moisture).	USEPA Method 8270D
Nitroaromatics, nitramines, and nitrate esters by high performance liquid chromatography (HPLC)	Soil - 10g soil, surrogates, mixed with anhydrous sodium sulfate are extracted using acetonitrile in an ultrasonic bath, or shaker filtered, diluted with water as appropriate, and analysed by HPLC with UV/DAD detection. Clay samples must be completely disintegrated before an aliquot is taken for analysis. Water - 250ml water sample plus	USEPA Method 8330B

Analyte	Method	Reference Method
	<p>surrogates are pre-concentrated using solid-phase extraction, as described in USEPA Method 3535 and then diluted with water as appropriate for the selected separations.</p> <p>Leachate - 250ml water sample plus surrogates extracted with SPE.</p>	
Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS	<p>This method is for determination of tetra-through octa-chlorinated dibenzo-p-dioxins (CDDs) and dibenzofurans (CDFs) in water, soil, sediment, sludge, tissue, and other sample matrices by high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS). The seventeen 2,3,7,8-substituted CDDs/CDFs may be determined by this method. Specifications are also provided for separate determination of 2,3,7,8-tetrachloro-dibenzo-p-dioxin (2,3,7,8-TCDD) and 2,3,7,8-tetrachloro-dibenzofuran (2,3,7,8-TCDF).</p> <p>The detection limits and quantitation levels in this method are usually dependent on the level of interferences rather than instrumental limitations.</p> <p>Analysis subcontracted to Eurofins GfA Lab Service GmbH – Hamburg, Germany</p>	USEPA Method 1613B
Inorganics		
Total Metals (As, Cd, Cr, Cu, Ni, Pb, Zn)	A portion of soil undergoes acidic digestion using either microwave or automated hot block. Analysis by ICP/MS. (Eurofins mgt in-house method LTM-MET-3030, LTM-GEN-7080 Moisture).	USEPA Method 6010C
Total Mercury (Hg)	A portion of soil undergoes acidic digestion using either microwave or automated hot block. Analysis by ICP/MS.(Eurofins mgt in-house method LTM-MET-3030, LTM-GEN-7080 Moisture).	USEPA Method 6010C
Filtered Metals (As, Cd, Cr, Cu, Ni, Pb ,Zn)	Filtered (0.45µm) and acidified in the field prior to analysis. Analysis by ICP/MS. (Eurofins mgt in-house method LTM-MET-3040).	USEPA Method 6020A
Filtered Mercury (Hg)	Filtered, oxidation and final reduction. Analysis by FIMS. (Eurofins mgt in-house method LTM-MET-3040).	USEPA Method 7471B
Water Laboratory		
Phosphorus	<p>Phosphorus analyses embody two general procedural steps:</p> <p>(a) conversion of the phosphorus form of</p>	APHA 4500 P.

Analyte	Method	Reference Method
	<p>interest to dissolved orthophosphate, and (b) colorimetric determination of dissolved orthophosphate. The separation of phosphorus into its various forms is defined analytically but the analytical differentiations have been selected so that they may be used for interpretive purposes.</p> <p>Filtration through a 0.45-μm-pore-diam membrane filter separates dissolved from suspended forms of phosphorus. No claim is made that filtration through 0.45-μm filters is a true separation of suspended and dissolved forms of phosphorus; it is merely a convenient and replicable analytical technique designed to make a gross separation. Pre-filtration through a glass fibre filter may be used to increase the filtration rate.</p> <p>Phosphates that respond to colorimetric tests without preliminary hydrolysis or oxidative digestion of the sample are termed "reactive phosphorus." While reactive phosphorus is largely a measure of orthophosphate, a small fraction of any condensed phosphate present usually is hydrolysed unavoidably in the procedure. Reactive phosphorus occurs in both dissolved and suspended forms. Acid hydrolysis at boiling-water temperature converts dissolved and particulate condensed phosphates to dissolved orthophosphate. The hydrolysis unavoidably releases some phosphate from organic compounds, but this may be reduced to a minimum by judicious selection of acid strength and hydrolysis time and temperature. The term "acid-hydrolysable phosphorus" is preferred over "condensed phosphate" for this fraction. The phosphate fractions that are converted to orthophosphate only by oxidation destruction of the organic matter present are considered "organic" or "organically bound" phosphorus. The severity of the oxidation required for this conversion depends on the form—and to some extent on the amount—of the organic phosphorus present. Like reactive phosphorus and acid-hydrolysable phosphorus, organic phosphorus occurs both in the dissolved and suspended fractions.</p> <p>The total phosphorus as well as the dissolved and suspended phosphorus fractions each may be divided analytically</p>	

Analyte	Method	Reference Method
	into the three chemical types that have been described: reactive, acid hydrolysable, and organic phosphorus. As indicated, determinations usually are conducted only on the unfiltered and filtered samples. Suspended fractions generally are determined by difference; however, they may be determined directly by digestion of the material retained on a glass-fibre filter.	
Chemical Oxygen Demand (COD)	Most types of organic matter are oxidized by a boiling mixture of chromic and sulfuric acids. A sample is refluxed in strongly acid solution with a known excess of potassium dichromate ($K_2Cr_2O_7$). After digestion, the remaining unreduced $K_2Cr_2O_7$ is titrated with ferrous ammonium sulfate to determine the amount of $K_2Cr_2O_7$ consumed and the oxidisable matter is calculated in terms of oxygen equivalent. Keep ratios of reagent weights, volumes, and strengths constant when sample volumes other than 50 mL are used. The standard 2-h reflux time may be reduced if it has been shown that a shorter period yields the same results. Some samples with very low COD or with highly heterogeneous solids content may need to be analysed in replicate to yield the most reliable data. Results are further enhanced by reacting a maximum quantity of dichromate, provided that some residual dichromate remains.	APHA 5220 C.
Colour - Visual Comparison Method	Colour is determined by visual comparison of the sample with known concentrations of coloured solutions. Comparison also may be made with special, properly calibrated glass colour disks. The platinum-cobalt method of measuring colour is the standard method, the unit of colour being that produced by 1 mg platinum/L in the form of the chloroplatinate ion. The ratio of cobalt to platinum given (2120B.4) matches the colour of natural waters.	APHA 2120 B.
Methylene blue active substances (MBAS)	Methylene blue active substances (MBAS) bring about the transfer of methylene blue, a cationic dye, from an aqueous solution into an immiscible organic liquid upon equilibration. This occurs through ion pair formation by the MBAS anion and the methylene blue cation. The intensity of the resulting blue colour in the organic phase is a measure of MBAS. Anionic surfactants are among the most prominent of many	APHA 5540 C

Analyte	Method	Reference Method
	substances, natural and synthetic, showing methylene blue activity. The MBAS method is useful for estimating the anionic surfactant content of waters and wastewaters, but the possible presence of other types of MBAS always must be kept in mind. This method is relatively simple and precise. It comprises three successive extractions from acid aqueous medium containing excess methylene blue into chloroform (CHCl ₃), followed by an aqueous backwash and measurement of the blue colour in the CHCl ₃ by spectrophotometry at 652 nm using in-house LTM-INO-4030 MBAS as MW: 288 (filtered).	
Ferrous	Ferrous iron by DA using in-house E058.1	APHA 3500-Fe B
Nitrate	Nitrogen-nitrate, nitrite, oxides of nitrogen, total by FIA using in-house E037.1	APHA 4500-NO₃- F
Anions in Water	Bromide; bromate; chloride; chlorite; chlorate; fluoride; iodide; nitrate; nitrite; phosphate; sulfate by IC using in-house E045.1	APHA 4110 B
Anions in Soils	Tests for water-soluble anions on milled air-dry sample are suitable for use on all soils in clarified/filtered 1:5 soil/water extracts. Bromide; bromate; chloride; chlorite; chlorate; fluoride; iodide; nitrate; nitrite; phosphate; sulfate by IC using in-house E045.1	APHA 4110 B
Total Organic Carbon in Water	<p>Total Carbon (TC) is measured by injecting a portion of the water sample into a heated combustion tube packed with an oxidation catalyst. The water is vaporised and TC, the organic carbon and the inorganic carbon, is converted to carbon dioxide (CO₂). The carbon dioxide is carried with the carrier gas stream from the combustion tube to a NDIR (non-dispersive infrared gas analyser) and concentration of carbon dioxide is measured. The TC concentration of the sample is obtained by using the calibration curve prepared with standard solutions.</p> <p>Inorganic Carbon (IC) is measured by injecting a portion of the sample into an IC reaction chamber filled with phosphoric acid solution. All IC is converted to carbon dioxide and concentration of carbon dioxide is measured with a NDIR.</p> <p>TOC may be obtained as the difference of</p>	APHA 5310 B

Analyte	Method	Reference Method
	TC and IC.	
Electrical Conductivity/ Resistivity	This in-house method will determine the concentration of ions in a soil-water or soil-calcium chloride suspension, expressed in $\mu\text{S}/\text{cm}$ units. The conductivity is measured electrometrically at constant temperature (e.g. 25°C). E032.2 in soil type matrices by conductivity meter	NEPM Schedule B3
% Organic Matter	Gravimetric determination based on ashing at $>600^{\circ}\text{C}$	NEPM Schedule B3
Alkalinity	Alkalinity by FIA and classical using in-house E035.1	APHA 2320
Chloride - 1:5 soil/water extract	Tests for water-soluble chloride (Cl) on milled air-dry sample are suitable for use on all soils. For method 5A1, Cl ⁻ in clarified 1:5 soil/water extracts is determined by potentiometric titration with AgNO ₃ in conjunction with an Ag/AgNO ₃ electrode array. For method 5A2a, Cl ⁻ in clarified 1:5 soil/water extracts is determined by an automated, continuous flow colorimetric procedure based on the formation — in the presence of ferric ions and free thiocyanate ions — of highly coloured ferric thiocyanate in proportion to the Cl ⁻ concentration. Method 5A2b is similar, except it pertains to the use of flow injection analysis (FIA). For 5A1 and 5A2 methods, it is assumed there are no chemical interferences of significance. Moreover, Method 5A2a has proven more precise than method 5A1, particularly at soil concentrations $<50\text{ mg Cl/kg}$. Other analytical finish options involve chemically-suppressed ion chromatography (5A3a), single-column electronically suppressed ion chromatography (5A3b), and direct measurement by ICPAES (Method 5A4). The methodology specifies reporting results on an air-dry basis.	
pH in Soils (1:5 aqueous extract)	This in-house method will determine the concentration of hydrogen ions (H ⁺) in a soil-water or soil-calcium chloride suspension, expressed in pH units. The pH is measured electrometrically at constant temperature (e.g. 25°C). LTM-GEN-7090_R0 pH electrometric measurement in water & soil-type matrices by ISE.	NEPM Schedule B3
Oil and Grease	This method is for determination of n-hexane extractable material (HEM; oil and grease) and n-hexane extractable material	USEPA Method 1664, Revision A n-Hexane Extractable

Analyte	Method	Reference Method
	that is not adsorbed by silica gel (SGT-HEM; non-polar material) in surface and saline waters and industrial and domestic aqueous wastes. Extractable materials that may be determined are relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases, and related materials. The	Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry
General		
Moisture	Gravimetric determination based on drying at 103-105 °C. MOISTURE CONTENT IN SOIL OR OTHER SOLID MATRICES BY GRAVIMETRY LTM-GEN-7080 Moisture.	NEPM Schedule B3
Clay Content	<p>Cation exchange capacity (CEC) is a measure of the soil's ability to hold positively charged ions. It is a very important soil property influencing soil structure stability, nutrient availability, soil pH and the soil's reaction to fertilisers and other ameliorants (Hazleton and Murphy 2007).</p> <p>The clay mineral and organic matter components of soil have negatively charged sites on their surfaces which adsorb and hold positively charged ions (cations) by electrostatic force. This electrical charge is critical to the supply of nutrients to plants because many nutrients exist as cations (e.g. magnesium, potassium and calcium). In general terms, soils with large quantities of negative charge are more fertile because they retain more cations (McKenzie et al. 2004) however, productive crops and pastures can be grown on low CEC soils. The main ions associated with CEC in soils are the exchangeable cations calcium (Ca²⁺), magnesium (Mg²⁺), sodium (Na⁺) and potassium (K⁺) (Rayment and Higginson 1992), and are generally referred to as the base cations. In most cases, summing the analysed base cations gives an adequate measure of CEC ("CEC by bases"). However, as soils become more acidic these cations are replaced by H⁺, Al³⁺ and Mn²⁺, and common methods will produce CEC values much higher than what occurs in the field (McKenzie et al. 2004). This "exchange acidity" needs to be included when summing the base cations and this measurement is referred to as effective CEC (ECEC). NOTE: Only CEC & ESP are calculated by this method.</p>	NEPM Schedule B3

Analyte	Method	Reference Method
Clay Content	<p>This method is based on the Soil Classification assessment by Hydrometer outlined in the Australian Standard 1289.3.6.3 (Determination of the particle size distribution of a soil – Standard method of fine analysis using a hydrometer). This method quantitatively determines the physical proportions of three sizes of primary soil particles, by determining their settling rates in an aqueous solution using a hydrometer.</p> <p>The three categories of particles measured are defined as follows:-</p> <ol style="list-style-type: none"> 1. Sand Ranges from 2000 to 50µm 2. Silt Ranges from 50-2µm 3. Clay Less than 2µm <p>Settling rates of primary soil particles are measured using a hydrometer.</p>	AS1289.3.6.3
Toxicity Characteristic Leaching Procedure (TCLP) Australian Standard Leaching Procedure (ASLP)	<p>This in-house method is for the preparation of leachates collected from soil, sediments, sludges, and other solid matrices using a rotary vessel extraction procedure. The method allows for the substitution of laboratory grade de-ionised water, EP or SPLP fluids, or site water supplied by the client as the extraction fluid. The solid portion of the sample is reduced in particle size, if necessary, and leached by rotary vessel agitation with a selected leaching fluid. The sample leachate is then extracted/ analysed by an additional test method, as per client request. (Eurofins mgt in-house method LEACHING PROCEDURE FOR VOLATILE AND NON-VOLATILE ANALYTES FROM SOILS AND SOLID WASTES LTM-GEN-7010.</p>	USEPA Method 1311 AS 4439.2; AS4439.3
Asbestos		
Asbestos in Soils	<p>The whole sample submitted is first dried and then sieved through a 10mm sieve followed by a 2mm sieve. All fibrous matter viz greater than 10mm, greater than 2mm as well as the material passing through the 2mm sieve are retained and analysed for the presence of asbestos. If the sub 2mm fraction is greater than approximately 30 to 60g then a sub-sampling routine based on ISO 3082:2009(E) Iron ores - Sampling and Sample preparation procedures is employed. Depending on the nature and size of the soil sample, the sub-2 mm residue material may need to be sub-</p>	AS 4964–2004

Analyte	Method	Reference Method
	<p>sampled for trace analysis in accordance with AS 4964-2004.</p> <p>Conducted in accordance with the Australian Standard AS 4964 – 2004: Method for the Qualitative Identification of Asbestos in Bulk Samples and in-house Method LTM-ASB-8020 by polarised light microscopy (PLM) and dispersion staining (DS) techniques. Bulk samples include building materials, soils and ores</p>	
Bonded asbestos-containing material (ACM)	The material is first examined and any fibres isolated and where required interfering organic fibres or matter may be removed by treating the sample for several hours at a temperature not exceeding 400 ± 30°C. The resultant material is then ground and examined in accordance with AS 4964-2004.and ores	AS 4964–2004
Asbestos fibres in Air	Conducted in accordance with the National Occupational Health & Safety Commission - Guidance Note on The Membrane Filter Method For Estimating Airborne Asbestos Fibres 2nd Edition [NOHSC:3003(2005)] and in-house Method LTM-ASB-8010.	NOHSC:3003(2005)