ANALYTICAL METHOD SUMMARIES

Analyte	Method	Reference Method
Organics		
1,4-Dioxane*	A water sample that has been dechlorinated and preserved with a microbial inhibitor is fortified with the isotopically labelled SUR, 1,4-dioxane-d8. The sample is extracted by one of two SPE options. In option 1, a 500-mL sample is passed through an SPE cartridge containing 2 g of coconut charcoal to extract the method analyte and SUR. In option 2, a 100-mL sample is extracted on a Waters AC-2 Sep-Pak or Supelco Supelclean ENVI-Carb Plus cartridge. In either option, the compounds are eluted from the solid phase with a small amount of dichloromethane (DCM), approximately 9 mL or 1.5 mL, respectively. The extract volume is adjusted, and the IS, tetrahydrofuran-d8 (THF-d8), is added. Finally, the extract is dried with anhydrous sodium sulfate. Analysis of the extract is performed by GC/MS. The data provided in this method were collected using splitless injection with a high-resolution fused silica capillary GC column that was interfaced to an MS operated in the SIM mode. The analyte, SUR and IS 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 GC/MS conditions. The concentration of the analyte is determined by comparison to its response in calibration standards relative to the IS.	**US EPA Method 522 DETERMINATION OF 1,4-DIOXANE IN DRINKING WATER BY SOLID PHASE EXTRACTION (SPE) AND GAS CHROMATOGRAPHY MASS SPECTROMETRY (GC/MS) WITH SELECTED ION MONITORING (SIM): EPA/600/R-08/101**
Methamphetamine in Swabs	**TARGET ANALYTES**: • Ephedrine • Pseudoephedrine • Amphetamine • Methamphetamine • MDA • MDMA SAMPLING: 1. Using a new pair of nitrile gloves, remove the wipe from its protective package that is contained in the supplied kit. NOTE: Do not use vinyl gloves due to the potential for leaching of phthalate	**NIOSH Method 9111 - METHAMPHETAMINE on Wipes by Liquid Chromatography/Mass Spectrometry**

Analyte	Method	Reference Method
	plasticisers and contamination of the samples. 2. Place the supplied 10 cm x 10 cm template over the area to be sampled (may tape in place along outside edge of template). Wipe the surface to be sampled with firm pressure, using vertical S-strokes. Fold the exposed side of the pad in and wipe the area with horizontal S-strokes. Fold the pad once more and wipe the area again with vertical S-strokes. 3. Fold the pad, exposed side in, and place in supplied shipping container and seal with cap. NOTE: Keep samples refrigerated (<6 ºC). While nicotine and related compounds are stable on the recommended wipe media for at least 7 days at room temperature, refrigeration is recommended as soon as possible. 4. Clean the template before use for the next sample or use a new disposable template. 5. Label each sample clearly with a unique sample identifier. 6. Prepare a minimum of two field blanks with one field blank for every ten samples. SAMPLE PREPARATION: 7. Desorption from media: a. Remove cap from shipping container. Sample media should fit loosely in the container. If not, rearrange media carefully with rinsed forceps or transfer to a larger container. If the sample media are transferred to a larger container, do not discard the original container. Samples may consist of more than one wipe. If this is the case, internal standard and desorption solution volumes may be adjusted accordingly. b. Spike exactly 50 µL of internal standard spiking solution onto each wipe sample. c. Add 30 mL desorption solution. If the samples were transferred to a larger container, the original shipping container must be rinsed with the desorption solution first, shaken, and the rinsate decanted into the larger container. d. Cap securely and mix contents by inverting the tubes end over end on a rotary mixer at 10-30 rpm for at least one hour. NOTE 1: The desorption solution must percolate freely through the gauze wipes.	

Analyte	Method	Reference Method
	e. Filter an aliquot of the sample through a 0.45 µm membrane. 8. Transfer the filtered sample into a vial and cap. 9. Analyse samples, standards, blanks, and Quality Control samples (QCs) by LC-MS/MS or LC-QToF-MS using in-house method LTM-ORG-2240.	
Methane in Water	The measurement of dissolved gases such as methane, ethane, and ethylene in ground water is important in determining whether intrinsic bioremediation is occurring in a fuel- or solvent contaminated aquifer. A helium headspace is generated above a water-filled bottle. Gases that are dissolved in the water partition between the gas and liquid phases and equilibrate rapidly. An aliquot of this headspace is analysed by gas chromatography to determine the gases' concentration in this phase. The concentration of the gas dissolved in the water can then be calculated based on its partitioning properties, as indicated by its Henry's Law constant using in-house LTM-ORG-2070	**Analysis of Dissolved Methane, Ethane, and Ethylene in Ground Water by a Standard Gas Chromatographic Technique., Don H. Kampbell* and Steve A. Vandegrift., U.S. Environmental Protection Agency, Journal of Chromatographic Science, Vol. 36, May 1998**
Per- and Polyfluoroalkyl Substances (PFAS) in Water	A water sample is fortified with isotopically labelled surrogates and passed through an IXR solid phase extraction (SPE) cartridge to extract the method analytes and surrogates. The compounds are eluted from the solid phase with a small amount of methanol with modifier. The extract is concentrated to near dryness under reduced pressure in an automated system and then adjusted to a final volume with mobile phase after adding the injection standards IS(s). An injection is made into an LC equipped with a C18 column that is interfaced to a tandem mass spectrometer (MS/MS) operating in scheduled MRM mode. 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 isotope dilution technique. Isotope dilution is used for calibration of each native compound for which an exact labelled analogue is available – see Table below. Labelled compounds are enriched with deuterium to produce ^{2}H-labeled analogues, stable isotopes of oxygen-18 to	**US EPA Method 533: Determination of Per- And Polyfluoroalkyl Substances in Drinking Water by Isotope Dilution Anion Exchange Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry., November 2019** **US EPA Method 537.1 Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction And Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)** **Version 1.0, November 2018**

Analyte	Method	Reference Method
	produce ^{18}O -labelled analogues or carbon-13 to produce ^{13}C-labeled analogues. The labelled analogues are spiked into each sample to allow identification and correction of the concentration of the native compounds in the extraction, clean-up and the analytical process. Correction of reported results along with a statement of the recovery for labelled analogues are included in the certificate of analysis. Typical recoveries are between 50-150% (± 50%) depending on media and the specific analyte. An initial calibration is prepared for each native compound. Internal standard calibration is applied to the determination of the native compounds that do not have exact labelled analogues and that are not being quantified by isotope dilution. The recoveries of the labelled analogues themselves are determined by internal standard quantitation (ISTD) and used as a quality control check on the overall analytical process. Branched and linear isomers are used for calibration standards when they are commercially available as a certified standard. Table 2 lists standards that are currently commercially available and used. The target analyte response for analytes containing branched and linear isomer result from the summation of peaks from all isomers. Limit of reporting is listed in Table 3: PFAS LORs - Water, Soil/Sediments & Biotic Matrices. The LOR obtainable is dependent on the matrix and method. The limit of reporting may be affected by the presence of other contaminants or components in individual samples that cause analytical interferences that raise the achievable LOR. This problem is more likely to occur in complex matrices such as soil, waste, biosolids and biota samples.	**US EPA Method 8327 - Per- and polyfluoroalkyl substances (PFAS) using external standard calibration and multiple reaction monitoring (MRM) liquid chromatography/tandem mass spectrometry (LC/MS/MS)** **United States of America's Department of Defense (DoD) Environmental Data Quality Workgroup (EDQW) and the Department of Energy (DOE) Consolidated Audit Program (DOECAP) Operations Team developed the Quality Systems Manual (QSM) for Environmental Laboratories. Version 5.3 May 2019** **UNITED STATES DEPARTMENT OF DEFENSE** **Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15** **Environmental Data Quality Workgroup** **1 May 2020**
Per- and Polyfluoroalkyl Substances (PFAS) in Soils	The sample is homogenised, spiked with isotopically labelled surrogates solution and digested with 1M NaOH by heating and ultrasonic agitation, followed by incubation. Samples are neutralised with	**USEPA Method EPA-821-R-11-007 Draft Procedure for Analysis of Perfluorinated Carboxylic Acids and Sulfonic Acids**

Analyte	Method	Reference Method
	HCl and extracted with solvent. Solvent extraction and clean-up is then performed using 50:50/ACN:MeOH (v/v) and then cleaned up using a carbon solid-phase extraction (SPE) cartridge. An injection is made into an LC equipped with a C18 column that is interfaced to a tandem mass spectrometer (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 isotope dilution technique. Isotope dilution is used for calibration of each native compound for which an exact labelled analogue is available – see Table below. Labelled compounds are enriched with deuterium to produce ^{2}H-labeled analogues, stable isotopes of oxygen-18 to produce ^{18}O - labelled analogues or carbon-13 to produce ^{13}C-labeled analogues. The labelled analogues are spiked into each sample to allow identification and correction of the concentration of the native compounds in the extraction, clean-up and the analytical process. Correction of reported results along with a statement of the recovery for labelled analogues are included in the certificate of analysis. Typical recoveries are between 50-150% (± 50%) depending on media and the specific analyte. An initial calibration is prepared for each native compound. Internal standard calibration is applied to the determination of the native compounds that do not have exact labelled analogues and that are not being quantified by isotope dilution. The recoveries of the labelled analogues themselves are determined by internal standard quantitation (ISTD) and used as a quality control check on the overall analytical process. Branched and linear isomers are used for calibration standards when they are commercially available as a certified standard. Table 2 lists standards that are currently commercially available and used. The target analyte response for analytes	**in Sewage Sludge and Biosolids by HPLC/MS/MS December 2011** **US EPA Method 8327 - Per- and polyfluoroalkyl substances (PFAS) using external standard calibration and multiple reaction monitoring (MRM) liquid chromatography/tandem mass spectrometry (LC/MS/MS)** **USEPA Method 533: Determination of Per- And Polyfluoroalkyl Substances in Drinking Water by Isotope Dilution Anion Exchange Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry., November 2019** **United States of America's Department of Defense (DoD) Environmental Data Quality Workgroup (EDQW) and the Department of Energy (DOE) Consolidated Audit Program (DOECAP) Operations Team developed the Quality Systems Manual (QSM) for Environmental Laboratories. Version 5.3 May 2019** **UNITED STATES DEPARTMENT OF DEFENSE**

Analyte	Method	Reference Method
	containing branched and linear isomer result from the summation of peaks from all isomers. Limit of reporting is listed in Table 3: PFAS LORs - Water, Soil/Sediments & Biotic Matrices. The LOR obtainable is dependent on the matrix and method. The limit of reporting may be affected by the presence of other contaminants or components in individual samples that cause analytical interferences that raise the achievable LOR. This problem is more likely to occur in complex matrices such as soil, waste, biosolids and biota samples.	**Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15** **Environmental Data Quality Workgroup** **1 May 2020**
Per- and Polyfluoroalkyl Substances (PFAS) in Biotic Matrices	The sample is cryogenically milled with dry ice, a homogenate taken and spiked with isotopically labelled surrogates solution. The sample is sonicated and vortexed and then neutralised with HCl before adding acetonitrile and again sonicated and vortexed before QuEChERS extraction is undertaken. Depending on the particular biotic matrix ENVI-carb SPE may be utilised prior to mixed-mode reversed phase WAX SPE was used before concentration to a known volume. An injection is made into an LC equipped with a C18 column that is interfaced to a tandem mass spectrometer (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 isotope dilution technique. Isotope dilution is used for calibration of each native compound for which an exact labelled analogue is available – see Table below. Labelled compounds are enriched with deuterium to produce 2H-labeled analogues, stable isotopes of oxygen-18 to produce 18O - labelled analogues or carbon-13 to produce 13C-labeled analogues. The labelled analogues are spiked into each sample to allow identification and correction of the concentration of the native compounds in the extraction, clean-up and the analytical process. Correction	**US EPA Method 8327 - Per- and polyfluoroalkyl substances (PFAS) using external standard calibration and multiple reaction monitoring (MRM) liquid chromatography/tandem mass spectrometry (LC/MS/MS)** **USEPA Method 533: Determination of Per- And Polyfluoroalkyl Substances in Drinking Water by Isotope Dilution Anion Exchange Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry., November 2019** **United States of America's Department of Defense (DoD) Environmental Data Quality Workgroup (EDQW) and the Department of Energy (DOE) Consolidated Audit Program (DOECAP) Operations Team**

Analyte	Method	Reference Method
	of report results along with a statement of the recovery for labelled analogues are included in the certificate of analysis. Typical recoveries are between 50-150% (± 50%) depending on media and the specific analyte. An initial calibration is prepared for each native compound. Internal standard calibration is applied to the determination of the native compounds that do not have exact labelled analogues and that are not being quantified by isotope dilution. The recoveries of the labelled analogues themselves are determined by internal standard quantitation (ISTD) and used as a quality control check on the overall analytical process. Branched and linear isomers are used for calibration standards when they are commercially available as a certified standard. Table 2 lists standards that are currently commercially available and used. The target analyte response for analytes containing branched and linear isomer result from the summation of peaks from all isomers. Limit of reporting is listed in Table 3: PFAS LORs - Water, Soil/Sediments & Biotic Matrices. The LOR obtainable is dependent on the matrix and method. The limit of reporting may be affected by the presence of other contaminants or components in individual samples that cause analytical interferences that raise the achievable LOR. This problem is more likely to occur in complex matrices such as soil, waste, biosolids and biota samples.	**developed the Quality Systems Manual (QSM) for Environmental Laboratories. Version 5.3 May 2019** **UNITED STATES DEPARTMENT OF DEFENSE** **Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15** **Environmental Data Quality Workgroup** **1 May 2020**
Total Oxidisable Precursor Analysis (TOPA) - Screen	Samples are treated via hydroxyl radical oxidation using an activated agent with overnight heating which converts the masked fluorinated precursors to their equivalent detectable perfluoroalkyl carboxylic acids (PFCAs) and perfluoroalkyl sulfonates (PFSAs). LC-MS/MS is done before and after TOP Analysis.	**Oxidative Conversion as a Means of Detecting Precursors to Perfluoroalkyl Acids in Urban Runoff, Erika F. Houtz and David L. Sedlak, Environ. Sci. Technol. 2012**

Analyte	Method	Reference Method
Total Oxidisable Precursor Analysis (TOPA) – Detailed	Detailed TOPA includes multiple mass-labelled analogues added prior to the "cooking" step and the recoveries provided along with dilutions required are reported in the certificate of analysis. Sum of all PFAS are reported pre- and post TOPA along with. Results for total PFAS concentration post-TOPA should be greater or equal to the total PFAS concentration pre-TOPA, (signifies no material losses observed in preparation steps, noting a decrease of up to 10% might be expected due to normal analytical variability). – the sum of PFCA post-TOPA should be equal to or greater than the sum of PFCA pre-TOPA, which signifies any precursors being converted to PFCA products. – the sum of PFSA post-TOPA should approximate the sum of PFSA pre-TOPA, signifying that precursors did not convert to PFSA products. – for a full oxidation, no PFAA precursors (e.g. 6:2 FTS, FOSA) are detectable post oxidation, signifying complete oxidation. – for situations where a near complete oxidation is acceptable, minimal PFAA precursors are detectable post oxidation signified by: • for aqueous samples, sum of [PFAA precursors] divided by sum of [Total PFAS] <5%. • for soil samples, sum of [PFAA precursors] divided by sum of [Total PFAS] <10%. • noting greater leniency may be applied for samples where PFAS were detected ≤ 10 times LOR.	**PFAS National Environmental Management Plan JANUARY 2020**

Analyte	Method	Reference Method

Table 1: Per- and Polyfluoroalkyl Substances (PFAS)

Native PFASs Perfluoroalkyl carboxylic acids (PFCAs)	Extracted Internal Standard Analytes (EIS) Isotope Dilution Quantification Standard
Perfluorobutanoic acid (PFBA)	Perfluoro-n-[1,2,3,4-$^{13}C_4$]butanoic acid
Perfluoropentanoic acid (PFPeA)	Perfluoro-n-[1,2,3,4,5-$^{13}C_5$]pentanoic acid
Perfluorohexanoic acid (PFHxA)	Perfluoro-n-[1,2,3,4,5-$^{13}C_5$]hexanoic acid
Perfluoroheptanoic acid (PFHpA)	Perfluoro-n-[1,2,3,4-$^{13}C_4$]heptanoic acid
Perfluorooctanoic acid (PFOA)	Perfluoro-n-[1,2,3,4,5,6,7,8-$^{13}C_8$]octanoic acid
Perfluorononanoic acid (PFNA)	Perfluoro-n-[1,2,3,4,5-$^{13}C_5$]nonanoic acid
Perfluorodecanoic acid (PFDA)	Perfluoro-n-[1,2,3,4,5,6-$^{13}C_6$]decanoic acid
Perfluoroundecanoic acid (PFUnA)	Perfluoro-n-[1,2-$^{13}C_2$]undecanoic acid
Perfluorododecanoic acid (PFDoA)	Perfluoro-n-[1,2-$^{13}C_2$]dodecanoic acid
Perfluorotridecanoic acid (PFTrDA)	Perfluoro-n-[1,2-$^{13}C_2$]tetradecanoic acid ISTD
Perfluorotetradecanoic acid (PFTeDA)	Perfluoro-n-[1,2-$^{13}C_2$]tetradecanoic acid
Perfluoroalkane sulfonic acids (PFSAs)	
Perfluoropropanesulfonic acid (PFPrS)	Sodium perfluoro-n-[2,3,4-$^{13}C_3$]butane sulfonate ISTD
Perfluorobutanesulfonic acid (PFBS)	Sodium perfluoro-n-[2,3,4-$^{13}C_3$]butane sulfonate
Perfluoropentane sulfonic acid (PFPeS)	Perfluoro-n-[1,2,3,4-$^{13}C_4$]octane sulfonic acid ISTD
Perfluorohexane sulfonate (PFHxS)	Sodium perfluoro-n-[$^{18}O_2$]hexanesulfonate
Potassium perfluorohexanesulfonate (linear and branched isomers) (br-PFHxSK)	Sodium perfluoro-n-[$^{18}O_2$]hexanesulfonate
Perfluoroheptane sulfonate (PFHpS)	Perfluoro-n-[1,2,3,4-$^{13}C_4$]octane sulfonic acid
Perfluorooctane sulfonate (PFOS)	Perfluoro-n-[1,2,3,4,5,6,7,8-$^{13}C_8$]octane sulfonate
Potassium perfluorooctanesulfonate (linear and branched isomers) (br-PFOSK)	Perfluoro-n-[1,2,3,4,5,6,7,8-$^{13}C_8$]octane sulfonate
Perfluorononanesulfonic acid (PFNS)	Perfluoro-n-[1,2,3,4-$^{13}C_4$]octane sulfonic acid ISTD
Perfluorodecanesulfonic acid (PFDS)	Perfluoro-n-[1,2,3,4-$^{13}C_4$]octane sulfonic acid ISTD
Perfluoroalkane sulfonamides (FASAs), Perfluoroalkane sulfonamido ethanols (FASEs) and N-alkyl perfluoroalkane sulfonamido ethanols (MeFASEs, EtFASEs) Perfluoroalkane sulfonamido acetic acids (FASAAs) and N-alkyl perfluoroalkane sulfonamido acetic acids (MeFASAAs, EtFASAAs)	
Perfluorooctane sulfonamide (FOSA)	Perfluoro-n-[1,2,3,4,5,6,7,8-$^{13}C_8$]octane sulfonamide
N-methylperfluoro-1-octane sulfonamide (N-MeFOSA)	N-methyl-d_3-perfluoro-n-octanesulfonamide
N-ethylperfluoro-1-octanesulfonamide (N-EtFOSA)	N-ethyl-d_5-perfluoro-n-octanesulfonamide
2-(N-methylperfluoro-1-octane sulfonamido)-ethanol (N-MeFOSE)	2-(N-methyl-d_3-perfluoro-1-octane sulfonamido)-ethanol-d_4
2-(N-ethylperfluoro-1-octane sulfonamido)-ethanol (N-EtFOSE)	2-(N-ethyl-d_5-perfluoro-1-octane sulfonamido)-ethanol-d_4
N-ethyl-perfluorooctanesulfonamidoacetic acid (N-EtFOSAA)	N-ethyl-d_5-perfluoro-n-octanesulfonamidoacetic acid
N-methyl-perfluorooctanesulfonamidoacetic acid (N-MEFOSAA)	N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid
Fluorotelomers **n:2 Fluorotelomer sulfonic acids (n:2 FTSAs)**	
1H,1H,2H,2H-Perfluorohexanesulfonic Acid (4:2 FTSA)	Sodium 1H,1H,2H,2H-perfluoro-1-[1,2-$^{13}C_2$]hexane sulfonate (4:2 FTSA)
1H,1H,2H,2H-Perfluorooctanesulfonic Acid (6:2 FTSA)	Sodium 1H,1H,2H,2H-perfluoro-1-[1,2-$^{13}C_2$]octane sulfonate (6:2 FTSA)
1H,1H,2H,2H-Perfluorodecanesulfonic Acid (8:2 FTSA)	Sodium 1H,1H,2H,2H-perfluoro-1-[1,2-$^{13}C_2$]decane sulfonate (8:2 FTSA)
1H, 1H, 2H, 2H-perfluorododecane sulfonic Acid (10:2 FTSA)	Sodium 1H,1H,2H,2H-perfluoro-1-[1,2-$^{13}C_2$, D4]dodecane sulfonate (10:2 FTSA)
Additional PFAS Compounds	
Hexafluoropropylene oxide dimer acid (HFPO-DA) [GenX)	$^{13}C_3$-Hexafluoropropylene oxide dimer acid ($^{13}C_3$-HFPO-DA)
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS) [11Cl-F53B]	Perfluoro-n-[1,2,3,4,5,6,7,8-$^{13}C_8$]octane sulfonate
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS) [9Cl-F53B]	Perfluoro-n-[1,2,3,4,5,6,7,8-$^{13}C_8$]octane sulfonate ISTD
4,8-dioxa-3H-perfluorononanoic acid (ADONA)	Perfluoro-n-[1,2,3,4,5,6,7,8-$^{13}C_8$]octanoic acid ISTD
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	Perfluoro-n-[1,2,3,4-$^{13}C_4$]heptanoic acid ISTD
Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA)	Sodium perfluoro-n-[2,3,4-$^{13}C_3$]butane sulfonate ISTD
Perfluoro-3-methoxypropanoic acid (PFMPA)	Perfluoro-n-[1,2,3,4-$^{13}C_4$]butanoic acid ISTD
Perfluoro-4-methoxybutanoic acid (PFMBA)	Perfluoro-n-[1,2,3,4,5-$^{13}C_5$]pentanoic acid ISTD
6:2 fluorotelomer sulfonamide alkylbetaine (6:2 FTAB)	N-ethyl-d_5-perfluoro-n-octanesulfonamide ISTD
3:3 Fluorotelomercarboxylic acid (3:3 FTCA)	Perfluoro-n-[1,2,3,4,5,6,7,8-$^{13}C_8$]octanoic acid ISTD
5:3 Fluorotelomer carboxylic acid (5:3 FTCA)	Perfluoro-n-[1,2,3,4,5,6,7,8-$^{13}C_8$]octanoic acid ISTD
Perfluoroethylcyclohexane sulfonate (PFECHS)	Perfluoro-n-[1,2,3,4-$^{13}C_4$]octane sulfonic acid ISTD

Analyte	Method	Reference Method
	Table 2: Currently Available Certified PFAS Standards Containing Branched and Linear Isomers Perfluorohexanesulfonic acid (PFHxS) Perfluorooctanesulfonic acid (PFOS) 2-(N-methylperfluorooctanesulfonamido) acetic acid (NMeFOSAA) 2-(N-ethylperfluorooctanesulfonamido) acetic acid (NEtFOSAA)	
Adsorbable Organofluorine (AOF)	For the trace level determination of adsorbable organic fluorine (AOF) in water, the sample must first be passed through a mixed-mode weak anion exchange solid-phase extraction (SPE) cartridge thereby adsorbing the PFAS compounds. AOF is then determined by eluting the contents of the SPE cartridge with NaOH in methanol, evaporating and reconstituting the extract, and finally determining the fluoride content of the extract by CIC. The LOR is dependent on the volume passed through the SPE, so the presence of suspended solids does impose limits on the procedure, but for clean waters the LOR is 0.001 mg F/L. Where significant levels of suspended solids are encountered the LOR may be limited to 0.01 mg F/L and the suspended solids may be determined separately by direct combustion.	**ASTM D7359-18 ASTM D7359 Standard Test Method for Total Fluorine, Chlorine and Sulfur in Aromatic Hydrocarbons and Their Mixtures by Oxidative Pyrohydrolytic Combustion followed by Ion Chromatography Detection (Combustion Ion Chromatography-CIC).**
Extractable Organofluorine (EOF)	For solid samples, where LORs lower than the direct combustion method of 0.05 mg F/kg are required, extraction can be performed using the same solvent systems used for conventional targeted LC-MS/MS methods. The resulting concentrate is then combusted giving an extractable organofluorine result. A LOR of 0.02 mg F/kg is achievable.	**ASTM D7359-18 Standard Test Method for Total Fluorine, Chlorine and Sulfur in Aromatic Hydrocarbons and Their Mixtures by Oxidative Pyrohydrolytic Combustion followed by Ion Chromatography Detection (Combustion Ion Chromatography-CIC).**
TRH (Volatile)/BTEX **C6-C10 – 2013 NEPM Fractions** **C6-C9 – 1999 NEPM Fractions**	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 in-house method numbers: Total Recoverable	**USEPA Method 8260D** **NEPM 2013 Schedule B3 Appendix 1: Determination of total recoverable hydrocarbons (TRH) in soil**

Analyte	Method	Reference Method
	Hydrocarbons (TPH), Method: LTM-ORG-2010, Method: LTM-GEN-7080 Moisture). 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.	
Total Recoverable Hydrocarbons C10-C36 – 1999 NEPM Fractions **>C10-C40 – 2013 NEPM Fractions**	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. Water - One 250ml of water sequentially extracted in a separatory funnel three times with 20mL dichloromethane. Analysis by capillary column GC/FID (Eurofins in-house method numbers: Total Recoverable Hydrocarbons (TRH), Method: LTM-ORG-2010, Method: LTM-GEN-7080 Moisture)	**USEPA Method 8015C** **NEPM 2013 Schedule B3 Appendix 1: Determination of total recoverable hydrocarbons (TRH) in soil**
TRH (Silica Gel)	Sample extracts obtained from the appropriate TRH method are exchanged to 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.	**USEPA Method 3630C** **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 in-house method numbers	**US EPA Method 8260D**

Analyte	Method	Reference Method
	Method: LTM-ORG-2150, LTM-ORG-2160, Method: LTM-GEN-7080 Moisture).	
Semi-volatile Organic Compounds (SVOCs)	The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation (refer to Method 3500) and, if necessary, sample clean-up procedures (refer to Method 3600). The semi-volatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph. Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve. NOTE: This method can be used in conjunction with the following sample preparation procedures: Water (including TCLP leachates) - Methods 3510, 3520, 3535 Soil/sediment - Methods 3540, 3541, 3545, 3546 3550, 3560, 3561	**USEPA Method 8270E**
Phenols/PAHs/PCBs/OPPs/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 in-house Methods LTM-ORG-	**USEPA Method 8270E**

Analyte	Method	Reference Method
	2130, LTM-ORG-2140 Method: LTM-GEN-7080 Moisture).	
Analysis of Phenoxy Acid Herbicides in Aqueous and Soil Samples by HPLC	A 100-mL water sample is adjusted to a basic pH with sodium hydroxide, shaken, and allowed to set for 1 hour to hydrolyse chlorinated esters. The sample is acidified with H_3PO_4, filtered, and the chlorinated acids are extracted from a 20-mL aliquot. The aliquot is pumped through a high performance liquid chromatography (HPLC) cartridge (containing C-18-silica), trapping the chlorinated acids. The concentrator cartridge is valved in-line with the C-18 analytical column following extraction. The acids are separated by HPLC and detected using an ultraviolet (UV) absorption spectrometer. LABORATORY TEST METHOD NUMBER: LTM-ORG-2180 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, adjusted to a basic pH with sodium hydroxide, shaken, and allowed to set for 1 hour to hydrolyse chlorinated esters. The sample is acidified with H_3PO_4, filtered, and the chlorinated acids are extracted from a 20-mL aliquot. The aliquot is pumped through a high performance liquid chromatography (HPLC) cartridge (containing C-18-silica), trapping the chlorinated acids. The concentrator cartridge is valved in-line with the C-18 analytical column following extraction. The acids are separated by HPLC and detected using an ultraviolet (UV) absorption spectrometer. LABORATORY TEST METHOD NUMBER: LTM-ORG-2180	**US EPA -NERL: Method 555: Chlorinated Acids in Water Using HPLC/UV**
EXPLOSIVES **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 surrogates are pre-concentrated using solid-phase extraction, as described in USEPA Method 3535 and then diluted with	**USEPA Method 8330B**

<table>
<tr><th>Analyte</th><th>Method</th><th>Reference Method</th></tr>
<tr><td></td><td>water as appropriate for the selected separations.
Leachate - 250ml water sample plus surrogates extracted with SPE.</td><td></td></tr>
<tr><td>DIOXINS
Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS↟</td><td>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).
The detection limits and quantitation levels in this method are usually dependent on the level of interferences rather than instrumental limitations.
↟Analysis subcontracted to Eurofins GfA Lab Service GmbH – Hamburg, Germany</td><td>USEPA Method 1613B</td></tr>
<tr><td>PBDEs
Polybrominated diphenyl ethers by HRGC/HRMS↟</td><td>This method is for determination of polybrominated diphenyl ethers in water, soil, sediment, sludge, tissue, and other sample matrices by high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS).
<table>
<tr><th>TriBDE</th><th>TetraBDE</th><th>PentaBDE</th></tr>
<tr><td>BDE-17
BDE-28</td><td>BDE-47
BDE-49
BDE-66
BDE-71
BDE-77</td><td>BDE-85
BDE-99
BDE-100
BDE-119
BDE-126</td></tr>
<tr><th>HexaBDE</th><th>HeptaBDE</th><th>OctaBDE</th></tr>
<tr><td>BDE-138
BDE-153
BDE-154
BDE-156</td><td>BDE-183
BDE-184
BDE-191</td><td>BDE-196
BDE-197</td></tr>
<tr><th>NonaBDE</th><th>DecaBDE</th><th></th></tr>
<tr><td>BDE-206
BDE-207</td><td>BDE-209</td><td></td></tr>
</table>
The detection limits and quantitation levels in this method are usually dependent on the level of interferences rather than instrumental limitations.
↟Analysis subcontracted to Eurofins GfA Lab Service GmbH – Hamburg, Germany</td><td>Method 1614A
Brominated Diphenyl Ethers in Water, Soil, Sediment, and Tissue by HRGC/HRMS
May 2010</td></tr>
</table>

Analyte	Method	Reference Method
Inorganics		
Total Metals (As, Cd, Cr, Cu, Ni, Pb, Zn)	A portion of soil or water undergoes acidic digestion using either microwave or automated hot block. Analysis by ICP-AES or ICP-MS. (Eurofins in-house method ICP-AES LTM-MET-3030, ICP-MS LTM-MET-3040 LTM-GEN-7080 Moisture).	**USEPA Method 6010D** **USEPA Method 3050B** **USEPA Method 3051A** **USEPA Method 6020B** **USEPA Method 3010A** **USEPA Method 3015A**
Total Mercury (Hg)	A portion of soil or water undergoes acidic digestion using either microwave or automated hot block. Analysis by ICP/MS.(Eurofins in-house method LTM-MET-3030, LTM-GEN-7080 Moisture).	**USEPA Method 6010D** **USEPA Method 3050B** **USEPA Method 3051A**
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 in-house method LTM-MET-3040).	**USEPA Method 6020B** **USEPA Method 3010A** **USEPA Method 3015A**
Filtered Mercury (Hg)	Filtered, oxidation and final reduction. Analysis by FIMS. (Eurofins in-house method LTM-MET-3040).	**USEPA Method 7471B** **USEPA Method 3010A** **USEPA Method 3015A**
Water Laboratory		
Alkalinity	Alkalinity is a measure of the acid neutralising capacity of waters. It is a measure of how much acid (H+) is required to lower the pH to a specific level. In most waters, alkalinity is a function of the concentrations of carbonate [CO32-], bicarbonate [HCO3-] and hydroxyl [OH-] ions present. For this method it is assumed that other weak inorganic or organic acids, such as silicic, phosphoric and boric acids are absent. Measuring alkalinity is important in determining a stream's ability to neutralise acidic pollutants from rainfall or wastewater. Total alkalinity is affected by environmental factors; rain, acidic sanitisers, addition of fill water and other product applications can all change the alkalinity over time. Most alkalinity in surface water comes from calcium carbonate (CaCO3), being leached from rocks and soil. This process is enhanced if the rocks and soil have been broken up for any reason, such as mining or urban development. Alkalinity is significant in the treatment of wastewater and drinking water because it will influence treatment process such as anaerobic digestion. Water may be unsuitable for use in irrigation if the	**APHA 2320 B.**

Analyte	Method	Reference Method
	alkalinity level in the water is higher than the natural level of alkalinity in the soil. This method covers the determination of alkalinity of all types of water. Alkaline ions present in the sample are neutralised by titration with a standard acid solution. Titration to different pH endpoints allows ion speciation to be determined. This method determines alkalinity relative to pre-designated endpoints measured by a pH meter. The end-points designated are pH 8.3 (Phenolphthalein Alkalinity) and pH 4.5 (Total Alkalinity). Titration by colour can also be used to analyse alkalinity, refer to APHA Method 2320 B (2.1) for details. Alkalinity is expressed in terms of the amount of calcium carbonate that would need to be dissolved in fresh water to give the same alkalinity. Alkalinity is reported as mg CaCO3/L. The typical range of applicability is 20 – 4000 mg CaCO3/L. Range can be extended with smaller sample volume and/or alternate titrant concentration(s).	
Ammonia in Water	Alkaline phenol and hypochlorite react with ammonia to form indophenol blue that is proportional to the ammonia concentration. The blue colour is intensified with sodium nitroprusside. This method determines ammonia in drinking, surface, and saline waters; domestic and industrial wastes.	**APHA 4500-NH3 B, C, D, F, H**
Anions in Water	Bromide; bromate; chloride; chlorite; chlorate; fluoride; iodide; nitrate; nitrite; phosphate; sulfate by IC using in-house E045.1/ LM-LTM- INO-4300.	**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/filtered1:5 soil/water extracts. Bromide; bromate; chloride; chlorite; chlorate; fluoride; iodide; nitrate; nitrite; phosphate; sulfate by IC using in-house E045.1/ LM-LTM- INO-4300.	**APHA 4110 B**
Biochemical Oxygen Demand (5 days, 20°C)	The BOD test is an empirical bioassay-type test which measures the dissolved oxygen consumed by microbial life while assimilating and oxidising organic matter in a sample. A waste sample (or dilution) is incubated for five days 20°C in the dark. Dissolved oxygen is measured before and after incubation using a modified Winkler or oxygen probe method. The reduction in	**APHA 5210**.

Analyte	Method	Reference Method
	dissolved oxygen during the incubation period yields a measure of BOD.	
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**.
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 AgNO3 in conjunction with an Ag/AgNO3 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 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.	**APHA Method 4500-Cl Rayment & Higginson 1992, "Australian Laboratory Handbook of Soil and Water Chemical. Methods".** **NEPM 2013 - Schedule B3 - Guideline on Laboratory Analysis of Potentially Contaminated Soil**

Analyte	Method	Reference Method
Chromium - hexavalent	This procedure measures only hexavalent chromium, (Cr6+). The hexavalent chromium is determined colorimetrically by reaction with diphenylcarbazide in acid solution. A red-violet coloured complex of unknown composition is produced. The colorimetric method is useful for the determination of hexavalent chromium in a natural or treated water in the range from 0.005 to 1 mg/L. This range can be extended by appropriate sample dilution or concentration and/or use of longer cell paths. Normal level analyses in waters uses in-house LTM- INO-4100 Analysis of hexavalent chromium in water by discrete analyser.	**APHA Standard Methods for the Examination of Water & Wastewater. 23rd Edition 2017. 3500-Cr-B**
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**.
Cyanide	Free Cyanide (CN_F) Only hydrogen cyanide and the cyanide ion in solution can be classed as "free" cyanide. The proportions of HCN and CN- in solution are according to their equilibrium equation; this is influenced by the solution pH. Methods used to detect free cyanide should not alter the stability of weaker cyanide complexes, as they may otherwise be included in the free cyanide result. Methods used to detect free cyanide should be clear of interferences due to the presence of high concentrations of more stable cyanide complexes or other cyanide forms. If not, the interference must be quantified and allowed for in the result. Weak Acid Dissociable Cyanide (CN_{WAD}) Unlike the definition of "free cyanide" which identifies the specific cyanide species being measured, WAD cyanide refers to those cyanide species measured by specific analytical techniques. WAD cyanide includes those cyanide species	**APHA 4500-CN B, C, D, E, I, N, O and USEPASW 846 9010, 9013, 9014, 9213**.

Analyte	Method	Reference Method
	liberated at moderate pH of 4.5 such as HCN(aq) and CN-, the majority of Cu, Cd, Ni, Zn, Ag complexes and others with similar low dissociation constants. Methods used to measure WAD should be free from interferences due to the presence of high concentrations of more stable cyanide complexes or other cyanide forms. If not, the interference must be quantified and allowed for in the result. Total Cyanide (CN_T) This measurement of cyanide includes all free cyanide, all dissociable cyanide complexes and all strong metal cyanide including ferro-cyanide $Fe(CN)_6^{4-}$, ferri-cyanide $Fe(CN)_6^{3-}$, and portions of hexacyano cobaltate $Co(CN)_6^{3-}$, and those of gold and platinum. Only the related or derived compounds cyanate (CNO-) and thiocyanate (SCN-) are excluded from the definition of total cyanide. Methods used to determine total cyanide must be shown to be capable of quantitatively determining all stable complexes of cyanide, including the cobalt cyanide complex. If methods determine other analytes as well (e.g. include SCN^-), those analytes need to be determined separately and allowed for in the total result. In-house method LTM-INO-4020 Total and Free plus Weak Acid Dissociable Cyanide by Continuous Flow Analysis	
Electrical Conductivity/Resistivity	This in-house method will determine the concentration of ions in a soil-water suspension, expressed in µS/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**
Ferrous (Fe^{2+})	Iron is brought into solution, reduced to the ferrous state by boiling with acid and hydroxylamine, and treated with 1,10-phenanthroline at pH 3.2 to 3.3. Three molecules of phenanthroline chelate each atom of ferrous iron to form an orange-red complex. The coloured solution obeys Beer's law; its intensity is independent of pH from 3 to 9. A pH between 2.9 and 3.5 insures rapid colour development in the presence of an excess of phenanthroline. Colour standards are stable for at least 6	**APHA 3500-Fe B Phenanthroline Method**

Analyte	Method	Reference Method
	months. Ferrous iron by DA using in-house LTM-INO-4190.	
Fluoride in Water	Fluoride is determined potentiometrically using a fluoride electrode in conjunction with a standard single junction sleeve-type reference electrode and a pH meter having an expanded millivolt scale or a selective ion meter having a direct concentration scale for fluoride using APHA 4500-F C. This method determines fluoride in drinking, surface, and saline waters; domestic and industrial wastes.	**APHA 4500-F^- C.**
Fluoride in Soils	Total fluoride by combustion ion chromatography (CIC) using in-house LTM-INO-4150 (Part A)	**ASTM D7359 Standard Test Method for Total Fluorine, Chlorine and Sulfur in Aromatic Hydrocarbons and Their Mixtures by Oxidative Pyrohydrolytic Combustion followed by Ion Chromatography Detection (Combustion Ion Chromatography-CIC).**
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 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 (CHCl3), followed by an aqueous backwash and measurement of the blue colour in the CHCl3 by spectrophotometry at 652 nm using in-house LTM-INO-4030 MBAS as MW: 288 (filtered).	**APHA 5540 C**
Nitrite, Total Oxidised Nitrogen	A discrete analysis is a system of quantitative spectrophotometric	**APHA 4500-NO_3^-**

Analyte	Method	Reference Method
(NOx) and Nitrate with photometric detection using Discrete Analyser	determinations utilising automated analytical techniques to perform chemical reactions with high precision and reliability. The sample and the reagents are pipetted by the instrument into a cell and mixed before incubation. After incubation, the absorbance of the solution is measured at the wavelength applicable to the determination. Nitrite (NO_2^-) is determined through formation of a reddish purple azo dye produced at pH 2.0 to 2.5 by coupling diazotised sulfanilamide with N-(1-naphthyl)-ethylenediamine dihydrochloride (NED dihydrochloride). The colour system is measured at 540 nm. The applicable detection range of spectrophotometric measurements, as dictated by Beer's Law, is 10 to 1000 µg NO_2^- N/L, with Method Detection Limits (MDL) and Limits of Reporting (LOR) determined within this range. See method validation for detail. Higher concentrations can be determined by diluting the sample. Total Oxidised Nitrogen (NO_X) Nitrate (NO_3^-) is reduced to nitrite (NO_2^-) by vanadium chloride. The total nitrite ions (Total Oxidised Nitrogen, TON, NO_X) are then reacted with sulphanilamide and NED dihydrochloride under acidic conditions to form a pink azo-dye. The absorbance is measured at 540 nm and is related to the NO_X concentration by means of a calibration curve. Higher concentrations can be determined by diluting the sample. Nitrate concentration is a result of the calculation of NOX concentration minus nitrite concentration noting the need for standardised units (i.e. mg N/L, or "as N"). Nitrogen-nitrate, nitrite, oxides of nitrogen, total by DA using in-house LTM-INO-4350	
Oil and Grease	This method is for determination of n-hexane extractable material (HEM; oil and grease) and n-hexane extractable material 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	**USEPA Method 1664, Revision A n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material)**

Analyte	Method	Reference Method
	related materials. The method is based on prior United States Environmental Protection Agency (US EPA) methods for determination of "oil and grease" and "total petroleum hydrocarbons". The term "n-hexane extractable material" reflects that this method can used to determine materials other than oils and greases. Similarly, the term "silica gel treated n-hexane extractable material" reflects that this method can be used to determine material that is not adsorbed by silica gel (non-polar material). This method is not applicable to measurement of materials that volatilise at temperatures below approximately 85°C. Petroleum fuels from gasoline through #2 fuel oil may be partially lost in the solvent removal operation. Some crude oils and heavy fuel oils contain a significant percentage of materials that are not soluble in n-hexane. Accordingly, recoveries of these materials may be low. This method is capable of measuring HEM and SGT-HEM in the range of 10 to 1000 mg/L, and may be extended to higher levels by analysis of a smaller sample volume collected separately.	**by Extraction and Gravimetry**
% Organic Matter	Gravimetric determination based on ashing at >600 °C	**NEPM Schedule B3**
pH in Soils (1:5 aqueous extract) **pH in Soils (1:5 $CaCl_2$ 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**
Phosphorus	Phosphorus analyses embody two general procedural steps: (a) conversion of the phosphorus form of 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. Filtration through a 0.45-µm-pore-diam membrane filter separates dissolved from suspended forms of phosphorus. No claim	**APHA 4500 P**.

Analyte	Method	Reference Method
	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. 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. The total phosphorus as well as the dissolved and suspended phosphorus fractions each may be divided analytically 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.	

Analyte	Method	Reference Method
Sulfate (as SO_4^{2-})	Sulfate ion (SO_4^{2-}) is precipitated in an acetic acid medium with barium chloride ($BaCl_2$) so as to form barium sulfate ($BaSO_4$) crystals of uniform size. Light absorbance of the $BaSO_4$ suspension is measured by a photometer and the SO_4^{2-} concentration is determined by comparison of the reading with a standard curve using in-house LTM-INO-4110 Sulfate by Discrete Analyser	**APHA 4500- SO_4^{2-} E. Turbidimetric Method***
Total Organic Carbon in Water	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. 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. TOC may be obtained as the difference of TC and IC.	**APHA 5310 B**
Total Dissolved Solids (TDS) Dried at 180°C	A well-mixed sample is filtered through a standard glass fibre filter. The filtrate is evaporated and dried to constant weight at 180°C. This method determines filterable residue in drinking, surface, and saline waters; domestic and industrial wastes. (A) Mineral Waters: Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing. (B) Bicarbonate: Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180oC to insure that all the bicarbonate is converted to carbonate. (C) High Residue Levels: Too much residue in the evaporating dish will crust over and entrap water that will not be	**APHA 2540 C.**

Analyte	Method	Reference Method
	driven off during drying. Total residue should be limited to about 200 mg. in-house method LTM-INO-4170.	
Total Suspended Solids (TSS) Dried at 103–105°C	Suspended solids are those that are retained on a glass-fibre filter. The unfiltered sample is mixed thoroughly and an appropriate volume is rapidly poured into a graduated cylinder. The suspended solids are collected on a glass fibre filter, and the insoluble residue is dried at 104 ± 1°C and weighed. This method may be used to determine the suspended-solids concentration of any natural or treated water or industrial waste. In-house method LTM-INO-4070	**APHA 2540 D.**
Fixed and Volatile Solids Ignited at 550°C	The residue from LTM-INO-4070 or LTM-INO-4170 is ignited to constant weight at 550°C. The remaining solids represent the fixed total, dissolved, or suspended solids while the weight lost on ignition is the volatile solids. The determination is useful in control of wastewater treatment plant operation because it offers a rough approximation of the amount of organic matter present in the solid fraction of wastewater, activated sludge, and industrial wastes.	**APHA 2540 E.**
Residue, Volatile (Gravimetric, Ignition at 550°C)	The residue obtained from the determination of total, filterable or non-filterable residue is ignited at 550°C in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile residue. This method determines the weight of solid material combustible at 550°C. The test is useful in obtaining a rough approximation of the amount of organic matter present in the solid fraction of sewage, activated sludge, industrial wastes, or bottom sediments.	**APHA 2540 E.**
General		
Cation exchange capacity (CEC)	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). The clay mineral and organic matter components of soil have negatively charged sites on their surfaces which	**NEPM** **Schedule B3**

Analyte	Method	Reference Method
	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 (Ca^{2+}), magnesium (Mg^{2+}), 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^{+}, Al^{3+} and Mn^{2+}, and common methods will produce CEC values much higher than what occurs in the field (McKenzie et al. 2004). NOTE: Only CEC & ESP are calculated by this method. Conducted by in-house Method LTM-MET-3060 – Cation Exchange Capacity (CEC) by bases & Exchangeable Sodium Percentage (ESP).	
Clay Content	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. The three categories of particles measured are defined as follows:- 1. Sand Ranges from 2000 to 50µm 2. Silt Ranges from 50-2µm 3. Clay Less than 2µm Settling rates of primary soil particles are measured using a hydrometer.	**AS1289.3.6.3**
Moisture	Gravimetric determination based on drying at 103-105 ^{o}C. MOISTURE CONTENT IN SOIL OR OTHER SOLID MATRICES BY GRAVIMETRY LTM-GEN-7080 Moisture.	**NEPM** **Schedule B3**

Analyte	Method	Reference Method
Leaching Procedures	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 in-house method LEACHING PROCEDURE FOR VOLATILE AND NON-VOLATILE ANALYTES FROM SOILS AND SOLID WASTES LTM-GEN-7010.	**Toxicity Characteristic Leaching Procedure (TCLP) USEPA Method 1311** **Australian Standard Leaching Procedure (ASLP) AS 4439.2: 2019; AS4439.3: 2019**
LEAF 1313	Liquid –Solid Partitioning as a Function of Extract pH for Constituents in Solid Materials using a Parallel Batch Extraction Procedure. Nine (9) Parallel extractions of a particle sized reduced solid material in dilute acid or base and reagent water. Series of eluates having pH values ranging from 2-13. Liquid solid ratio of 10:1. Eluate is centrifuged and filtered for COPCs. Designed to provide aqueous extracts representing the liquid-solid portioning [LSP] curve as a function of pH for inorganics and non-volatile organics in solid materials	**EPA SW-846 Method 1313**
LEAF 1314	Liquid –Solid Partitioning as a Function of Liquid-Solid Ration for Constituents in Solid Materials using an Up-Flow Percolation Column Procedure Eluent is introduced into a column with packed particle sized reduced solid material in an up-flow pumping mode. Flow rate is maintained between 0.5-1.0 LS/Day. Eluent is collected at predetermined times, filtered and analysed for COPCs. Total time of test is approximately 14 days. Designed to provide the liquid – solid portioning [LSP] of inorganic constituents and non-volatile organics in granular solid material as a function of liquid to solid [LS] ratio under percolation conditions.	**EPA SW-846 Method 1314**

Analyte	Method	Reference Method
LEAF 1315	Mass Transfer Rates of Constituents in Monolithic or Compacted Granular Materials using a Semi-dynamic Tank Leaching Procedure. Leaching of continuously water saturated monolithic or compacted granular material in an eluent-filled tank with periodic renewal of the leaching solution. LS ratio of 9 mL eluent per cm2 of surface area. Eluent is collected at predetermined times and analysed for COPCs. Eluate is centrifuged and filtered for COPCs. Total time of test is 63 days. Designed to provide the mass transfer [release rates] of inorganic analytes contained in a monolith or compacted granular material. Under diffusion controlled release conditions, as a function of leaching time.	**EPA SW-846 Method 1315**
LEAF 1315	Mass Transfer Rates of Constituents in Monolithic or Compacted Granular Materials using a Semi-dynamic Tank Leaching Procedure. Leaching of continuously water saturated monolithic or compacted granular material in an eluent-filled tank with periodic renewal of the leaching solution. LS ratio of 9 mL eluent per cm^2 of surface area. Eluent is collected at predetermined times and analysed for COPCs. Eluate is centrifuged and filtered for COPCs. Total time of test is 63 days. Designed to provide the mass transfer [release rates] of inorganic analytes contained in a monolith or compacted granular material. Under diffusion controlled release conditions, as a function of leaching time.	**EPA SW-846 Method 1315**
LEAF 1316	Liquid- Solid Partitioning as a Function of Liquid-Solid Ratio for Constituents in Solid Materials using a Parallel Batch Extraction Procedure. Five (5) Parallel extractions of a particle-size reduced solid material in reagent water over a range of L/S values from 0.5 to 10 mL eluant/g dry material. Depending on particle size, sample is tumbled between 24 and 72 hours. Eluate is centrifuged and filtered for COPCs. Designed to provide the liquid-solid portioning{LSP] of inorganic and non-	**EPA SW-846 Method 1316**

Analyte	Method	Reference Method
	volatile organics at the natural pH of the solid material as a function of liquid to solid ratio [L/S] under conditions that approach liquid-solid chemical equilibrium.	
Asbestos		
Asbestos in Soils	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-sampled for trace analysis in accordance with AS 4964-2004. 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	**AS 4964–2004**
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)**
Air		

Analyte	Method	Reference Method
Filters - Total Metals (As, Cd, Cr, Cu, Ni, Pb, Zn)	The filter is digested in a hot block set to 95°C for 2.5 hours using an extraction fluid containing hydrochloric acid (HCl) and nitric acid (HNO_3). Two aliquots of hydrogen peroxide (H_2O_2) are added after 1.5 hours and 2.0 hours of extraction and are allowed to effervesce. After extraction, the samples are filtered and diluted to a final volume of 50 mL. The extract is analysed by ICP-MS and the data are collected using the manufacturer's software.	**EQL-0512-201 - US Environmental Protection Agency**
Microbiology		
Method for Isolation, Enumeration and Confirmation of Sulfite Reducing Clostridia and *Clostridium perfringens* from Raw, Potable, Process and Recreational Waters by Membrane Filtration	Tests for sulfite-reducing clostridia play only a subsidiary role in water examination. The organisms form spores which are environmentally resistant and their presence may indicate soil contamination, although some species may grow in deposits, and be associated with corrosion of distribution pipes. *Clostridium perfringens* is a sulfite-reducing species and is associated with faecal contamination. The method involves a volume of sample that is filtered and the membrane filter placed on the surface of an agar medium containing sulfite, iron(III) and D-cycloserine (which inhibits other bacteria and reduces the size of colonies that develop). The agar medium is then incubated under anaerobic conditions at 37 °C. Sulfite-reducing clostridia usually produce black colonies as a result of the reduction of sulfite to sulfide, which then reacts with the iron(III) salt. If only a spore count is required then the sample is heat-treated at 60 °C prior to filtration in order to kill vegetative bacteria. In-house method Sulfite-Reducing Clostridia - Membrane Filtration Method: LTM-MIC-6617	**UK Environment Agency, The Microbiology of Drinking Water (2010), Part 6 – Methods for the isolation and enumeration of sulphite-reducing clostridia and *Clostridium perfringens* by membrane filtration.**
Detection of Male-specific & Somatic Coliphages in Water	Method uses the single agar layer (SAL) procedure. A 100-mL ground water sample is assayed by adding $MgCl_2$ (magnesium chloride), log-phase host bacteria (E. coli F_{amp} for F+ coliphage and E. coli CN-13 for somatic coliphage), and 100 mL of double-strength molten tryptic soy agar to the	**USEPA Method 1602: Male-specific (F+) and Somatic Coliphage in Water by Single Agar Layer (SAL) Procedure April 2001**

Analyte	Method	Reference Method
	sample. The sample is thoroughly mixed and the total volume is poured into 5 to 10 plates (dependent on plate size). After an overnight incubation, circular lysis zones (plaques) are counted and summed for all plates from a single sample. The quantity of coliphage in a sample is expressed as plaque forming units (PFU) / 100 mL. For quality control purposes, both a coliphage positive reagent water sample and a negative reagent water sample (method blank) are analysed for each type of coliphage with each sample batch.	
Air Toxics Laboratory		
TRH by Modified US EPA TO-15*	The laboratory performed analysis following modified EPA TO-15 for Total Recoverable Hydrocarbon (TRH) fractions using electron ionisation GC/MS in full scan mode. The method involves concentrating up to 0.2 litres of air. The concentrated aliquot is then flash vaporised and swept through a water management system to remove water vapour. Following dehumidification, the sample passes directly into the GC-MS for analysis. All sample-related peaks including BTEX and naphthalene eluting within their respective carbon range are included in the TRH result. The >C6-C10 TRH range is defined as the total ion area of peaks eluting after n-Hexane and including n-Decane referenced to the response factor of Toluene. The >C10-C12 TRH range is defined as the total area of peaks eluting after n-Decane and including n-Dodecane and reference to the response factor of n-Decane. Hydrocarbons heavier than C12 do not reliably recover from summa canisters due to their low vapour pressure. As a result, the reported range was limited to C12 rather than C16 as defined in Table C1[1]. If requested, the fraction >C6-C10 minus BTEX (F1) and >C10-C12 minus naphthalene (modified F2) were reported following the definition listed in the previous paragraph except BTEX and	**USEPA Compendium Method TO-15 Determination Of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS)**

[1] CRC CARE 2013, Petroleum hydrocarbon vapour intrusion assessment: Australian guidance, CRC CARE Technical Report no. 23, CRC for Contamination Assessment and Remediation of the Environment, Adelaide, Australia.

Analyte	Method	Reference Method
	naphthalene peaks were removed from the total ion peak area. Naphthalene elutes outside the >C10-C12 range on the system used for sample analysis. As a result, >C10-C12 TRH value is equivalent to the modified F2 value.	
Modified US EPA TO-15 & VPH Fractions*	The laboratory performed analysis via EPA Method TO-15 and Eurofins Air Toxics VPH (Volatile Petroleum Hydrocarbon) methods for the Determination of VPH Fractions using GC/MS in the full scan mode. The method involves concentrating up to 0.5 litres of air. The concentrated aliquot is then flash vaporised and swept through a water management system to remove water vapour. Following dehumidification, the sample passes directly into the GC/MS for analysis. This method is designed to measure gaseous phase aliphatic and aromatic compounds in ambient air and soil gas collected in stainless steel Summa canisters. Eurofins Air Toxics VPH method is a hybrid of EPA TO-15 method viz chromatographic peaks were identified via mass spectrum as either aliphatic or aromatic petroleum hydrocarbons and included in the appropriate range as defined by the method. The volatile Aliphatic hydrocarbons are collectively quantified within the C5 to C6 range, C6 to C8 range, C8 to C10 range and the C10 to C12 range. Additionally, the volatile Aromatic hydrocarbons are collectively quantified within the C8 to C10 range and the C10 to C12 range. The Aromatic ranges refer to the equivalent carbon (EC) ranges. (Please note that benzene constitutes the >C5-C7 aromatic range and toluene constitutes the >C7-C8 aromatic range. Benzene and toluene concentrations are reported on the TO-15 workorder fraction.) Aliphatic data is calculated from the Total Ion Chromatogram (TIC) which has been reprocessed in a duplicate file differentiated from the original by the addition of an alphanumeric extension. The Aromatic calculation also uses the information contained in the associated extracted ion file.	**USEPA Compendium Method TO-15 Determination Of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS)**
Modified Natural Gas Analysis by ASTM D-1946*	The laboratory performed analysis via Modified ASTM Method D-1946 for Methane and fixed gases in air using	**ASTM D1946-77**

<table>
<tr><th>Analyte</th><th>Method</th><th>Reference Method</th></tr>
<tr><td></td><td>GC/FID or GC/TCD. The method involves direct injection of 1.0 mL of sample. On the analytical column employed for this analysis, Oxygen co-elutes with Argon. The corresponding peak is quantitated as Oxygen.</td><td>Standard Method for Analysis of Reformed Gas by Gas Chromatography</td></tr>
<tr><td>Analysis of volatile and semivolatile organic compounds in vapor by thermal desorption GC/MS full scan using modified EPA method TO-17,
SOP#109</td><td>The laboratory performed analysis via The laboratory performed the analysis via modified EPA Method TO-17 using GC/MS in the full scan mode. TO-17 'VI' sorbent tubes are thermally desorbed onto a secondary trap. The trap is thermally desorbed to elute the components into the GC/MS system for compound separation and detection.
A modification that may be applied to EPA Method TO-17 at the client's discretion is the requirement to transport sorbent tubes at 4 deg C. Laboratory studies demonstrate a high level of stability for VOCs on the TO-17 'VI' tube at room temperature for periods of up to 14 days. Tubes can be shipped to and from the field site at ambient conditions as long as the 14-day sample hold time is upheld. Trip blanks and field surrogate spikes are used as additional control measures to monitor recovery and background contribution during tube transport. Since the TO-17 VI application significantly extends the scope of target compounds addressed in EPA Method TO-15 and TO-17, the laboratory has implemented several method modifications outlined in the table below.
Specific project requirements may override the Eurofins Air Toxics modifications.
<table>
<tr><td>Requirement</td><td>TO-17</td><td>Eurofins Air Toxics Modifications</td></tr>
<tr><td>Initial Calibration</td><td>%RSD
≤ 30% with 2 allowed out up to 40%</td><td>VOC list:
%RSD ≤ 30% with 2 allowed out up to 40%</td></tr>
<tr><td>SVOC list: %RSD</=30 % with 2 allowed out up to 40%</td><td></td><td></td></tr>
<tr><td>Daily Calibration</td><td>%D for each target compound within ± 30%.</td><td>Fluorene, Phenanthrene, Anthracene, Fluoranthene,</td></tr>
</table></td><td>Modified EPA Method TO-17 (VI Tubes)*</td></tr>
</table>

<table>
<tr><th>Analyte</th><th>Method</th><th>Reference Method</th></tr>
<tr><td></td><td>
<table>
<tr><td></td><td></td><td>and Pyrene within ± 40%D</td></tr>
<tr><td>Audit Accuracy</td><td>70-130%</td><td>Second source recovery limits for Fluorene, Phenanthrene, Anthracene, Fluoranthene, and Pyrene = 60-140%.</td></tr>
<tr><td>Distributed Volume Pairs</td><td>Collection of distributed volume pairs required for monitoring ambient air to insure high quality.</td><td>If site is well-characterised or performance previously verified, single tube sampling may be appropriate. Distributed pairs may be impractical for soil gas collection due to configuration and volume constraints.</td></tr>
<tr><td>Analytical Precision</td><td>≤ 20% RPD</td><td>≤ 30% RPD for Fluorene, Phenanthrene, Anthracene, Fluoranthene, and Pyrene.</td></tr>
</table>
</td><td></td></tr>
<tr><td>Volatile organic compounds (VOCs) - chemically desorbed with CS_2</td><td>Code RAD130 cartridge is a stainless steel net cylinder, with 100 mesh grid opening and 5.8 mm diameter, packed with 530 ± 30 mg of activated charcoal, particle size is 35-50 mesh. Volatile organic compounds are trapped by adsorption and recovered by carbon disulfide desorption, analysis is performed by GC-MS.
white diffusive body code RAD120 
supporting plate code RAD121 
vertical adapter code RAD122 (optional) 
Chemi-adsorbing cartridge code RAD130  </td><td>Passive Sampler – radiello® User Manual 2019</td></tr>
</table>

Analyte	Method	Reference Method
	Extraction: A volume of 2.0 ml of CS_2 and 100 µL of internal standard solution is added directly in the radiello glass tube. The tube is shaken gently for 30 minutes. Sampling rates varies from the value at 298 K on the effect of temperature (in Kelvin) as expressed by the following equation: $$Q_K = Q_{298}(\frac{K}{298})$$ where Q_K is the sampling rate at the temperature K and Q_{298} is the reference value at 298 K. This produces a variation of ±5% for 10 °C variation (upwards or downwards) from 25 °C. Sampling rate is invariant with humidity in the range 15 - 90% and with wind speed between 0.1 and 10 $m.s^{-1}$. NOTE: where uptake rates (Q_K) are unpublished then they have been estimated from like compounds. Results for these compounds are semi-quantitative. Average concentration (in $\mu g.m^{-3}$) over the whole exposure time is calculated according to the following expression: $$C\ (\mu g.m^{-3}) = \frac{m\ (\mu g)}{Q_K\ (mL.min^{-1}).t(min)}.10^6$$ m = mass of analyte in µg determined by GC-MS t = exposure time in minutes	
Volatile organic compounds (VOCs) - thermally desorbed	Code RAD145 is a stainless steel net cylinder, with 3 x 8 µm mesh opening and 4.8 mm diameter, packed with 350 ± 10 mg of graphitised charcoal (Carbograph 4), particle size is 35-50 mesh. Volatile organic compounds are trapped by adsorption and recovered by thermal desorption, analysis is performed by GC-MS. yellow diffusive body code RAD1202 	**Passive Sampler – radiello® User Manual 2019**

Analyte	Method	Reference Method
	supporting plate code RAD121  vertical adapter code RAD122 (optional)  Chemi-adsorbing cartridge code RAD145  Code RAD145 cartridge has been dimensioned to fit the diameter of the Markes Unity thermal desorption system that is used in conjunction with an Agilent GC-MS. Sampling rates varies from the value at 298 K on the effect of temperature (in Kelvin) as expressed by the following equation: $$Q_K = Q_{298}(\frac{K}{298})$$ where Q_K is the sampling rate at the temperature K and Q_{298} is the reference value at 298 K. This produces a variation of ±5% for 10 °C variation (upwards or downwards) from 25 °C. Sampling rate is invariant with humidity in the range 15 - 90% and with wind speed between 0.1 and 10 $m.s^{-1}$. Do not expose directly radiello to rain: even if small amounts of water are adsorbed by Carbograph 4, they can nevertheless interfere with analysis. NOTE: where uptake rates (Q_K) are unpublished then they have been estimated from like compounds. Results for these compounds are semi-quantitative. Average concentration (in $\mu g.m^{-3}$) over the whole exposure time is calculated according to the following expression: $$C\ (\mu g.m^{-3}) = \frac{m\ (\mu g)}{Q_K\ (mL.min^{-1}).t(min)}.10^6$$ m = mass of analyte in µg determined by GC-MS t = exposure time in minutes	

Analyte	Method	Reference Method
Volatile organic compounds (VOCs) – passive samplers	Companion EPA Methods 325A (Sampler Deployment and VOC Sample Collection) and 325B (Sampler Preparation and Laboratory Analysis) select benzene as the representative compound to evaluate the overall emissions from refineries. Passive sampling onto sorbent tubes followed by Thermal Desorption-Gas-Chromatography/Mass Spectrometry (TD-GC/MS) analysis has been established as the standard air monitoring technology for the EPA's new rule. Passive sampling tube shelter assemblies will be hung at various locations along the fence line/property boundary surrounding refineries. After two weeks (14 days) passive sampling tubes can be detached from their shelters, re-sealed and sent to a laboratory equipped with TD-GC/MS for analysis. Per EPA Method 325, all tubes must be replaced with freshly conditioned and qualified sampling tubes every 14 days to ensure continuous monitoring. The methods provide a low cost alternative to screen fugitive or area emissions as compared to active sampling methods that involve pumped sorbent tubes or time weighted average canister sampling. While the rule is currently limited to the monitoring of benzene, Method 325 can also be extended to include other compounds of concern at ambient monitoring sites. Additional target VOCs include 1,3-Butadiene, Toluene, Ethyl Benzene, and Xylenes as well as other chemicals for which diffusive sampling rates have been determined. Reporting limits less than 1 µg/m^3 can be easily achieved over a 7-day period. Extending the sampling period to 14 days translates to reporting limits less than 0.5 µg/m^3.	**US EPAMethod 325B— Volatile Organic Compounds from Fugitive and Area Sources:** **Sampler Preparation and Analysis**

Table 3: PFAS LORs - Water, Soil/Sediments & Biotic Matrices

Per- and Polyfluoroalkyl Substances (PFASs)	CAS No.[a]	MW	WATER (Potable, surface, groundwater, saline		SOLIDS (Soil, sediment, biosolids)		BIOTA*			
			LOR (µg/L)	LOR Trace (µg/L)	LOR (µg/kg)	LOR Trace (µg/kg)	Type 1 LOR (ng/mL)	Type 2 LOR (µg/kg)	Type 3 LOR (µg/kg)	Type 2 Trace LOR (µg/kg)
Perfluoroalkyl carboxylic acids (PFCAs)										
Perfluorobutanoic acid (PFBA)	375-22-4	214.04	0.05	0.005	5	0.1	0.5	0.5	1	0.1
Perfluoropentanoic acid (PFPeA)	2706-90-3	264.05	0.01	0.001	5	0.1	0.5	0.5	1	0.1
Perfluorohexanoic acid (PFHxA)	307-24-4	314.05	0.01	0.001	5	0.1	0.5	0.5	1	0.1
Perfluoroheptanoic acid (PFHpA)	375-85-9	364.06	0.01	0.001	5	0.1	0.5	0.5	1	0.1
Perfluorooctanoic acid (PFOA)	335-67-1	414.07	0.01	0.001	5	0.1	0.5	0.5	1	0.1
Perfluorononanoic acid (PFNA)	375-95-1	464.08	0.01	0.001	5	0.1	0.5	0.5	1	0.1
Perfluorodecanoic acid (PFDA)	335-76-2	514.08	0.01	0.001	5	0.1	0.5	0.5	1	0.5
Perfluoroundecanoic acid (PFUnA)	2058-94-8	564.09	0.01	0.001	5	0.1	0.5	0.5	1	0.5
Perfluorododecanoic acid (PFDoA)	307-55-1	614.10	0.01	0.001	5	0.1	0.5	0.5	1	0.5
Perfluorotridecanoic acid (PFTrDA)	72629-94-8	664.11	0.01	0.001	5	0.1	0.5	0.5	1	0.5
Perfluorotetradecanoic acid (PFTeDA)	376-06-7	714.11	0.01	0.001	5	0.1	0.5	0.5	1	0.5
Perfluoroalkyl sulfonic acids (PFSAs)										
Perfluoropropanesulfonic acid (PFPrS)	423-41-6	250.09	0.01	0.001	5	0.1	0.5	0.5	1	0.1
Perfluorobutanesulfonic acid (PFBS)	375-73-5	300.10	0.01	0.001	5	0.1	0.5	0.5	1	0.1
Perfluoropentane sulfonic acid (PFPeS)	2706-91-4	350.11	0.01	0.001	5	0.1	0.5	0.5	1	0.1
Perfluorohexane sulfonate (PFHxS)	355-46-4	400.11	0.01	0.001	5	0.1	0.5	0.5	1	0.1
Potassium perfluorohexanesulfonate (linear and branched isomers) (br-PFHxS)			0.01	0.001	5	0.1	0.5	0.5	1	0.1
Perfluoroheptane sulfonate (PFHpS)	375-92-8	450.12	0.01	0.001	5	0.1	0.5	0.5	1	0.1
Perfluorooctane sulfonic acid (PFOS)[g ,h]	1763-23-1	500.13	0.01	0.001	5	0.1	0.5	0.5	1	0.1
Potassium perfluorooctanesulfonate (linear and branched isomers) (br-PFOS)			0.01	0.001	5	0.1	0.5	0.5	1	0.1

Per- and Polyfluoroalkyl Substances (PFASs)	CAS No.[a]	MW	WATER (Potable, surface, groundwater, saline		SOLIDS (Soil, sediment, biosolids)		BIOTA*			
			LOR (µg/L)	LOR Trace (µg/L)	LOR (µg/kg)	LOR Trace (µg/kg)	Type 1 LOR (ng/mL)	Type 2 LOR (µg/kg)	Type 3 LOR (µg/kg)	Type 2 Trace LOR (µg/kg)
Perfluorononanesulfonic acid (PFNS)	68259-12-1	550.13	0.01	0.001	5	0.1	0.5	0.5	1	0.1
Perfluorodecanesulfonic acid (PFDS)	67906-42-7	617.18	0.01	0.001	5	0.1	0.5	0.5	1	0.1
Perfluoroalkane sulfonamides (FASAs), Perfluoroalkane sulfonamido ethanols (FASEs) and N-alkyl perfluoroalkane sulfonamido ethanols (MeFASEs, EtFASEs) Perfluoroalkane sulfonamido acetic acids (FASAAs) and N-alkyl perfluoroalkane sulfonamido acetic acids (MeFASAAs, EtFASAAs)										
Perfluorooctane sulfonamide (FOSA)	754-91-6	499.14	0.05	0.005	10	1	5	0.5	5	0.5
N-Methylperfluorooctane sulfonamide (MeFOSA)	31506-32-8	513.17	0.05	0.005	10	1	5	0.5	5	0.5
N-Ethylperfluorooctane sulfonamide (EtFOSA)	4151-50-2	527.19	0.05	0.005	10	1	5	2	5	0.5
N-Methylperfluorooctane sulfonamidoethanol (MeFOSE)	24448-09-7	557.22	0.05	0.005	10	1	5	1	5	0.5
N-Ethylperfluorooctane sulfonamidoethanol (EtFOSE)	1691-99-2	571.25	0.05	0.005	10	1	5	1	5	0.5
N-Ethylperfluorooctanesulfonamido acetic acid (EtFOSAA)	2991-50-6	585.23	0.05	0.005	10	1	5	0.5	5	0.5
N-Methylperfluorooctanesulfonamido acetic acid (N-MeFOSAA)	2355-31-9	571.21	0.05	0.005	10	1	5	0.5	5	0.5
n:2 Fluorotelomer sulfonic acids (n:2 FTSAs)										
1H,1H,2H,2H-Perfluorohexanesulfonic Acid (4:2 FTSA)	757124-72-4	328.15	0.01	0.001	5	0.5	5	0.5	5	0.1
1H,1H,2H,2H-Perfluorooctanesulfonic Acid (6:2 FTSA)	27619-97-2	428.16	0.01	0.001	5	0.5	5	0.5	5	0.1
1H,1H,2H,2H-Perfluorodecanesulfonic Acid (8:2 FTSA)	39108-34-4	528.18	0.01	0.001	5	0.5	5	1	5	0.1
1H, 1H, 2H, 2H-perfluorododecane sulfonate (10:2 FTSA)	120226-60-0	628.20	0.01	0.001	5	0.5	5	1	5	0.5
Additional PFAS Compounds										
Hexafluoropropylene oxide dimer acid (HFPO-DA) [GenX)	13252-13-6[b]	285[f]	0.01		5					
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF_3OUdS) [11Cl-F53B]	763051-92-9[c]	631	0.01		5					
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF_3ONS) [9Cl-F53B]	756426-58-1[d]	531	0.01		5					
4,8-dioxa-3H-perfluorononanoic acid (ADONA)	919005-14-4[e]	377	0.01		5					

Per- and Polyfluoroalkyl Substances (PFASs)	CAS No.[a]	MW	WATER (Potable, surface, groundwater, saline		SOLIDS (Soil, sediment, biosolids)		BIOTA*			
			LOR (µg/L)	LOR Trace (µg/L)	LOR (µg/kg)	LOR Trace (µg/kg)	Type 1 LOR (ng/mL)	Type 2 LOR (µg/kg)	Type 3 LOR (µg/kg)	Type 2 Trace LOR (µg/kg)
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	151772-58-6	296.045	0.01		5					
Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA)	113507-82-7	316.09	0.01		5					
Perfluoro-3-methoxypropanoic acid (PFMPA)	377-73-1	230.038	0.01		5					
Perfluoro-4-methoxybutanoic acid (PFMBA)	863090-89-5	280.046	0.01		5					
6:2 fluorotelomer sulfonamide alkylbetaine (6:2 FTAB)[i]	34455-29-3	570.37	0.01		5					
3:3 Fluorotelomercarboxylic acid (3:3 FTCA)	356-02-5	242.093	0.01		5					
5:3 Fluorotelomer carboxylic acid (5:3 FTCA)	914637-49-3	342.108	0.01		5					
Perfluoropropane sulfonic acid (PFPrS)	423-41-6	248.90	0.01		5					
Perfluoroethylcyclohexane sulfonate (PFECHS)	67584-42-3	500.22	0.01		5					

[a] Some PFAS are commercially available as ammonium, sodium and potassium salts. This method measures all forms of the analytes as anions while the counterion is inconsequential. Analytes may be purchased as acids or as any of the corresponding salts (see Section 7.2.3 regarding correcting the analyte concentration for the salt content).
[b] HFPO-DA is one component of the GenX processing aid technology.
[c] 11Cl-PF_3OUdS is available in salt form (e.g. CASRN of potassium salt is 83329-89-9).
[d] 9Cl-PF_3ONS analyte is available in salt form (e.g. CASRN of potassium salt is 73606-19-6)
[e] ADONA is available as the sodium salt (no CASRN) and the ammonium salt (CASRN is 958445-448).
[f] HFPO-DA is not stable in the ESI source and the [M-H]- is not observed under typical ESI conditions. The precursor ion used during method development was [M-CO2]-.
[g] Analyte has multiple resolved chromatographic peaks due to linear and branched isomers. All peaks summed for quantitation purposes.
[h] To reduce bias regarding detection of branched and linear isomers, the m/z 80 product ion must be used for this analyte.
[i] NICNAS 6:2 Fluorotelomer sulfonamide surfactants: Environment tier II assessment 12 December 2019

BIOTA Key
Type 1 - Human and Animal Blood (whole blood & plasma)
Type 2 - Citrus, tomato, zucchini, grasses, squash; muscle tissue of fish, crustaceans, cheese, cow, sheep; kidney tissue of sheep and cow; milk and chicken egg
Type 3 - Sheep and cow liver; olives and avocado

This page left intentionally blank