

SOIL TESTS

Determinand	Laboratory	MRL	Acreditation		Lab Test Method Ref	Summary
			UKAS	MCERTS		
Explosives	BAE	shown below	√	√	ESAL/QC/4k	Overnight 2:1 solvent extraction of prepared soil samples. Analysis of extracts by Isocratic and Gradient High Performance Liquid Chromatography incorporating Ultra Violet absorption detectors capable of dual wavelength detection
NC		5000mg/kg				
HMX		2mg/kg				
RDX		2mg/kg				
EGDN		0.1mg/kg				
Tetryl		1mg/kg				
NG		0.1mg/kg				
TNT		0.5mg/kg				
PETN		5mg/kg				
HNS		0.5mg/kg				
Picrite		0.25mg/kg				
Picric Acid		0.1mg/kg				
2, 6 DNT		1mg/kg				
2, 4 DNT		1mg/kg				
Metals	BAE	shown below	√	-	ESAL/QC/4c	Aqua Regia digestion of soil samples based on ISO11.466 using a semi sealed system to retain volatile elements of interest followed by analysis using ICP-OES.
As		1mg/kg				
Cd		0.3mg/kg				
Cu		0.3mg/kg				
Ni		5mg/kg				
Pb		3mg/kg				
Zn		3mg/kg				
Metals	BAE	shown below	-	-	ESAL/QC/4i	
Ba		5mg/kg				
Be		0.1mg/kg				
Bi		4mg/kg				
Mg		4mg/kg				
Mn		2mg/kg				

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Mo Sb Sn Sr Ti V		5mg/kg 5mg/kg 2mg/kg 3mg/kg 5mg/kg 5mg/kg				
Mercury	BAE	0.1mg/kg	√	-	ESAL/QC/4g	Aqua Regia digestion of soil samples as above are diluted to remove matrix effects. Using a semi-automated pumping system, these dilutions are mixed with a reductant in a gas/liquid separator where any ionic Mercury is reduced to the element. Argon gas carries the volatile Mercury vapour through a drying membrane into the optical path of a fluorescence detector and the resulting fluorescence is quantitatively measured.
Selenium	BAE	0.3mg/kg	√	-	ESAL/QC/4f	Aqua Regia digestion of soil samples as above, the digests are acidified and boiled to ensure all the Selenium present is in the correct oxidation state. The extracts are mixed with a reductant in a gas/liquid separator where any Selenium present in the extract is converted to its hydride form. Argon gas carries the hydride through a drying membrane into the optical path of a fluorescence detector and the resulting fluorescence is quantitatively measured.
Phosphorus	BAE	5mg/kg	-	-	ESAL/QC/4i	Aqua Regia digestion of soil samples based on ISO11.466 using a semi sealed system to retain volatile elements of interest followed by analysis using ICP-OES.
PAH	BAE	0.1mg/kg	√	√	ESAL/QC/4s	Overnight 2:1 Dichloromethane extraction of prepared soils followed by analysis using a fully automated GC-MS system.
TPH	BAE	shown below	√	√	ESAL/QC/4v	For the PRO range, an as-received sub-sample of soil is extracted with methanol. The extract is analysed by Headspace GC-MS. For the DRO range, an air-dried sub-sample of soil is extracted with Dichloromethane. The extract is analysed by GC-FID.
C5 – C6 C6 – C8 C8 – C10 C10 – C12 C12 – C16 C16 – C21 C21 – C40		1.5mg/kg 2mg/kg 1mg/kg 2mg/kg 20mg/kg 20mg/kg 20mg/kg				

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MTBE Benzene Toluene Ethyl Benzene m,p-Xylene o-Xylene		0.5mg/kg 0.5mg/kg 0.5mg/kg 0.5mg/kg 0.5mg/kg 0.5mg/kg				
PCB's	STL	2µg/kg	√	√	GE001	Samples are pre-extracted with propan-2-ol and then extracted into hexane. This extract is then concentrated prior to clean up with an alumina silver nitrate column. PCB's are determined by GC-ECD.
VOC's	BAE	0.025mg/kg	√	-	ESAL/QC/4t	An as-received sub-sample of soil is treated with a saturated sodium sulphate solution to which internal and surrogate standards are added. After equilibrating at an elevated temperature, an aliquot of the headspace is extracted and analysed by GC-MS.
Dioxins & Furans	Marchwood	0.0001ng/g	√	-	1613	The samples are dried, ground and passed through a 1mm sieve. A known weight is then extracted for 16hrs with toluene. The extract is concentrated to 1ml and subject to column chromatography to eliminate other interfering compounds. The sample is then concentrated and reconstituted in nonane and analysed by High Resolution Gas Chromatography/Mass Spectrometry using a Micromass Ultima NT mass spectrometer.
Solvents	Mountainheath	0.1mg/kg	-	-	MH0098	A known mass of sample is vialled and solvents are allowed to evaporate into the vial headspace. If required (e.g. for porous samples) a liquid in which the analyte is insoluble is added to the sample to purge any trapped headspace from the sample. The headspace is analysed by GCMS in broadscan mode.
Alcohols	Mountainheath	0.1mg/kg	-	-	MH0069	A known mass of sample is vialled and alcohols are allowed to evaporate into the vial headspace. If required (e.g. for porous samples) a liquid in which the analyte is insoluble is added to the sample to purge any trapped headspace from the sample. The headspace is analysed by GCMS in broadscan mode.
Sulphur	BAE	10mg/kg	√	√	ESAL/QC/4u	Overnight 5:1 Dichloromethane extraction of prepared soils followed by analysis of extracts by Isocratic High Performance Liquid Chromatography incorporating UV absorption detectors.
Sulphate	BAE	250mg/kg	-	-	ESAL/QC/ SULPHATE	10:1 Concentrated Hydrochloric Acid extraction of soil sample followed by analysis using ICP-OES.

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Hexamine	Mountainheath	0.5mg/kg	-	-	MH0028	Hexamine, dimethylamine and diphenylamine are extracted from solid samples into dichloromethane (1:1 solid/liquid extraction) by sonication and mechanical shaking. An aliquot of the solvent layer is vialled and analysed by GCMS against appropriate calibration standards.
Dimethylaniline	Mountainheath	0.5mg/kg				
Diphenylamine	Mountainheath	0.5mg/kg				
Carbamite	BAE		-	-	ESAL/QC/ CARBAMITE	Overnight 2:1 solvent extraction of prepared soil samples. Analysis of extracts by Isocratic and Gradient High Performance Liquid Chromatography incorporating Ultra Violet absorption detectors capable of dual wavelength detection.
Perchlorate	Mountainheath	0.1mg/kg	-	-	MH0029	Anions (including chlorate/perchlorate) are extracted from soil samples into water then analysed directly by ion chromatography, with conductivity detection.
Pesticides	STL	0.05mg/kg	-	-	OCPS1	Samples are extracted with hexane and cleaned up prior to analysis by GC-ECD
		0.1mg/kg	-	-	OPPS1	The OPP content of land samples is extracted with dichloromethane/acetone mix solvent using an accelerated solvent extractor (ASE). 1ml of extract is transferred to a vial and internal standard is added. The OPP content of this extract is then determined by GC-MS
Herbicides	STL	0.1mg/kg	-	-	THERBS1	The herbicide content of land samples is extracted with dichloromethane/acetone mix solvent using an accelerated solvent extractor (ASE). 1ml of extract is transferred to a vial and internal standard is added. The herbicide content of this extract is then determined by GC-MS.
Asbestos	Bodycote	-	√	-	ASB/01	A preliminary visual examination of the whole of the bulk sample is made to assess the sample type and the required sample treatment (if any): where possible a representative sub-sample may be taken at this stage. Sample treatment is undertaken (if required) to release or isolate fibres. A detailed and thorough search under the stereo microscope is made to classify the fibre types present. Representative fibres are mounted in appropriate RI liquids on microscope slides and the different fibrous components are identified using a polarised light microscope.
pH	BAE	0.1 Unit	-	-	ESAL/QC/pH	An aqueous soil suspension is prepared and the pH is measured using a calibrated pH meter.
Organic Matter	Fugro	0.10%	√	-	DETS 002	Organic matter in soil is oxidised with potassium dichromate in the

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						presence of concentrated sulphuric acid. The excess dichromate is titrated with ferrous sulphate using diphenylamine as an external indicator. The organic matter content is calculated from the amount of dichromate used during the oxidation process based on an empirical relationship.
Moisture Content	BAE	-	-	-	ESAL/QC/ DRY MATTER	Soil samples are dried to a constant mass at 105 ±3°C. The difference in mass of an amount of soil before and after the drying procedure shall be used to calculate the dry matter contents on a mass basis.
Calorific Value	BAE	<500 kJ/kg	-	-	Method 9	The calorific value of the sample is determined by combustion of the sample in a controlled environment under ~30 bars pressure of Oxygen.
Leachate (As, B, Be, Cd, Cr, Cu, Hg, Mg, Mn, Mo, Ni, Pb, Sb, Se, Sn, V, Zn)	STL	N/A	N/A	N/A	N/A	A mass of sample is weighed out and a volume of water 10 times its weight is added. The sample is then shaken for 24 hours and filtered through a 0.45µm membrane filter. The samples are then subjected to the test method as detailed in the Groundwater Test summary table.

GROUNDWATER TESTS

Determinand	Laboratory	MRL	UKAS	Lab Test Method Ref	Summary
Explosives	BAE	50µg/l	√	ESAL/QC/4r	Analysis of preconcentrated and filtered water samples by Isocratic and Gradient High Performance Liquid Chromatography incorporating Ultra Violet absorption detectors capable of dual wavelength detection.
Metals (Al, B, Ba, Be, Ca, Cd, Cr, Cu, K, Mg, Mn, Mo, Na, Ni, Pb, Sn, Zn)	STL	Various	√	WASO12	For dissolved metals samples are filtered through a 0.45µm membrane filter. Metals are determined in the filtrate following acidification with nitric acid pH 2. This prevents the metals from adsorbing onto the surface of the container. Metal concentrations are determined by ICP-OES.
Metals As Se Sb	STL	0.1µg/l	√	WAS014 WAS015 WAS016	Arsenic, selenium and antimony form volatile covalent hydrides when reduced with sodium borohydride. Samples are digested with hydrochloric acid – a hot digest is used for selenium analysis and for those samples where solids are present. Potassium iodide and ascorbic acid are added for arsenic and antimony analysis to convert all species to the appropriate hydride generation. The hydride produced is readily atomised and the resulting atoms are detected by atomic fluorescence.
Mercury	STL	0.1µg/l	√	WAS013	A mixture of acidic bromate and bromide is used to digest and preserve the sample. Tin(ii) chloride is used to reduce mercury to mercury vapour. The mercury vapour produced is detected by atomic fluorescence.
PAH (speciated)	STL	0.05µg/l	√	GE019	The sample is solvent extracted into dichloromethane prior to analysis by GCMSD in selective ion mode.
TPH	STL	50µg/l	√	GE035	This analysis is referred to as TPH by GC-FID and is a measure of hydrocarbons associated with both gasoline range (C6-C9) and diesel range (C10-C40). Samples are analysed by solvent extraction followed by GC-FID.
VOC	STL	5µg/l	√	GE032	P&T Extraction followed by GCMSD detection of a list of 59 compounds, with an option for qualitative identification of tentatively identified compounds
SVOC	STL	1-5µg/l	√	GE031	A target suite of organic compounds is analysed using solvent extraction followed by GC-MS

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Sulphide	STL	0.01mg/l	√	WAS033	Sulphide reacts with DPD and potassium dichromate to produce ethylene blue (or DPD blue). The concentration of sulphide is determined by measuring this blue complex using a spectrophotometer at 670nm.
Sulphate	STL	5mg/l	√	WAS036	Each determinand reacts with a specific reagent to produce a coloured complex that can be measured colorimetrically. In the case of sulphate an insoluble precipitate is formed which is determined turbidimetrically.
Chloride	STL	2mg/l	√		
Ammoniacal Nitrogen	STL	0.3mg/l	√		
Nitrite	STL	0.1mg/l	√		
Nitrate	STL	0.3mg/l	√	WAS036	This is a calculated result and equals Total oxidised nitrogen minus nitrite.
Alkalinity as CaCO ₃	STL	5mg/l	√	WAS025	The total alkalinity is measured by autotitration of the sample with sulphuric acid standard solution with instrumental detection at endpoint at pH 4.5.
Hardness as CaCO ₃	STL	1MG/L	√	WAS012	These are calculated results from the Calcium and Magnesium results. Total Hardness as CaCO ₃ = (Ca * 2.5) + (Mg * 4.12).
Suspended Solids	STL	2mg/l	√	WAS006	Suspended matter is removed from a measured volume of sample by filtration under reduced pressure through a pre-treated, pre-weighed glass fibre filter paper and determined gravimetrically after washing and drying at 105°C. In addition samples containing oil or other organic liquids undergo a solvent wash before drying, whilst samples containing a high level of dissolved solids undergo a hot wash prior to drying.
pH	STL	N/A	√	WAS023	The pH is measured directly by pH probe, calibrated using buffer solutions of known pH
Electrical Conductivity	STL	10µS/cm	√	WAS024	The conductivity is measured using a conductivity meter, calibrated using a standard solution of known conductivity. The meter automatically compensates to give values at 20°C or 25°C depending on which is specified.
COD	STL	20mg/l	√	WAS040	Samples are oxidised by heating in vials with sulphuric acid and potassium dichromate. Mercuric sulphate and nitrate are used to suppress chloride interference. The chromate produced is determined colorimetrically using a spectrophotometer, which gives results directly in mg/l as Oxygen.

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BOD	STL	1mg/l	√	WAS001	Samples are mixed with dilution water saturated with oxygen and containing nutrients, bacterial seed and ATU to suppress nitrification. The initial dissolved oxygen value is determined prior to leaving the samples to incubate in the dark for 5 days or 20 days. Robots with dissolved oxygen probes are used to take the dissolved oxygen readings at the beginning and end of the test.