



Sample Preparation Instructions

Changes to Document from Previous Issue

- Inclusion of the following samples:
 - Sample 26 Organochlorine (OCs) Pesticides in Soil

General Instructions

Sample Storage

All standard solutions should be stored in a refrigerator at $4(\pm 2)^{\circ}\text{C}$, in the dark, from the time of arrival at your laboratory. Other samples should be stored in accordance with your normal procedures. If a preservative is routinely added to the type of sample provided, as part of your laboratory procedures, a suitable aliquot should be preserved as soon as possible in the normal way. Any dilutions that result from addition of preservatives should be corrected for before submission of results.

N.B. Group C Samples 15 and 19, and Samples 22 and 23 spiking solutions should be stored at -20°C or lower on receipt.

Sample Preparation

All samples should be equilibrated at room temperature $20(\pm 5)^{\circ}\text{C}$ before any dilutions or analyses are performed. Samples should be prepared in accordance with the specific instructions for the group. The dilutions specified should be conducted in such a way as to ensure that any errors introduced by this dilution are much smaller than the overall analytical error involved in your method. As a general rule it is suggested that the error from dilution should be less than 1%. Example dilutions are given for illustration to help clarify the meaning of the instructions. These procedures should be followed exactly to ensure comparability of results. **Any dilutions detailed as part of these procedures should not be used in the calculation of results. These dilutions simply provide a final sample for analysis within the expected concentration ranges.**

Sample Analysis

Samples should be analysed by the normal methods used for those determinands by your laboratory.

CONTEST samples should be treated like any other samples and all normal quality control procedures should be adopted.

Results should only be corrected for recovery and blank, if appropriate, **and** if this is the normal practice in the laboratory. **If the sample is diluted as part of the analytical process (this is apart from the dilutions in the sample preparation instructions), such dilutions should be corrected for.**

Soil Matrix

The classification for the soil matrix (soil type), for each round, will be provided on the sample label for Samples 3a, 3b, 3c and 18.

General Safety

Some of the CONTEST samples contain acids or alkalis and are corrosive. Others will contain low levels of carcinogens and toxic or flammable chemicals.



Sample Preparation Instructions

Group A – Metals

Sample 1a

Materials Supplied

- Approximately 8mL standard solution (in 0.2M nitric acid)

Preparation

Dilute an aliquot of the sample supplied by a factor of 10 (e.g. 1mL made up to 10mL) using **deionised** water or **acid** to achieve your normal final acid strength then analyse by your chosen instrumental technique for the following determinands. **Do not correct the results for these dilutions.**

- Barium
- Beryllium
- Cadmium
- Chromium
- Cobalt
- Copper
- Iron
- Lead
- Manganese
- Molybdenum
- Nickel
- Thallium
- Tin
- Vanadium
- Zinc

Sample 1b

Materials Supplied

- Approximately 8mL standard solution (in 0.2M nitric acid)

Preparation

Dilute the sample supplied by a factor of 10 (e.g. 1mL made up to 10mL) using **deionised** water or **acid** to achieve your normal final acid strength then analyse by your chosen instrumental technique for the following determinands. **Do not correct the results for these dilutions.**

- Antimony
- Arsenic
- Selenium



Sample Preparation Instructions

Sample 1c

Materials Supplied

- Approximately 8mL standard solution (in 0.2M nitric acid)

Preparation

Dilute the sample supplied by a factor of 10 (e.g. 1mL made up to 10mL) using **deionised** water or **acid** to achieve your normal final acid strength then analyse by your chosen instrumental technique for the following determinands. **Do not correct the results for these dilutions.**

- Mercury

N.B. Mercury may be present in both organic and inorganic forms.

Sample 2

Materials Supplied

- Approximately 60ml extract of soil (prepared following ISO 11466 for the extraction of metals, acid strength 21% HCl and 7% HNO₃)

Preparation

Analyse the sample by your chosen instrumental technique for the following determinands:

- Antimony
- Arsenic
- Barium
- Beryllium
- Cadmium
- Chromium
- Cobalt
- Copper
- Iron
- Lead
- Manganese
- Mercury
- Molybdenum
- Nickel
- Selenium
- Thallium
- Tin
- Vanadium
- Zinc



Sample Preparation Instructions

Sample 3a

Materials Supplied

- Approximately 30g prepared soil, ground to pass through a 200µm sieve

Preparation

Analyse the sample by your chosen instrumental technique for the following determinands:

- Antimony
- Arsenic
- Barium
- Beryllium
- Cadmium
- Chromium
- Cobalt
- Copper
- Iron
- Lead
- Manganese
- Mercury
- Molybdenum
- Nickel
- Selenium
- Thallium
- Tin
- Vanadium
- Zinc
- Chromium (VI)

Sample 13

Materials Supplied

- Approximately 60mL standard solution (containing potassium dichromate and another chromium salt)

Preparation

Analyse the sample by your chosen instrumental technique for the following determinands:

- Chromium (VI)



Sample Preparation Instructions

Group B – Inorganics

Sample 3b

Materials Supplied

- Approximately 150g prepared soil, ground to pass through a 200µm sieve

Preparation

Analyse the sample by your chosen instrumental technique for the following determinands:

- Ammonia
- Complex cyanide
- Free cyanide
- Total cyanide
- Dry matter
- Loss on ignition*
- pH
- Thiocyanate
- Total sulfate
- Easily liberated sulfide
- Water soluble boron*
- Water soluble chloride*
- Water soluble fluoride*
- Water soluble sulfate*

*Participants should use the temperature or water:soil ratio they use in their routine analysis.

Sample C6

Materials Supplied

- Approximately 60mL standard solution (preserved in 0.1M sodium hydroxide)

Preparation

Analyse the sample by your chosen instrumental technique for the following determinands:

- Total cyanide

Sample 8

Materials Supplied

- Approximately 60mL standard solution

Preparation

Analyse the sample by your chosen instrumental technique for the following determinands:

- Total sulfate



Sample Preparation Instructions

Sample 10

Materials Supplied

- Approximately 60mL standard solution

Preparation

Analyse the sample by your chosen instrumental technique for the following determinands:

- Water soluble boron

Sample 12

Materials Supplied

- Approximately 60mL standard solution (preserved with zinc acetate)

Preparation

Shake the sample well and take a 5mL sub-sample. Analyse the aliquot by your chosen instrumental technique for the following determinands:

- Easily liberated sulfide

N.B. Results should be reported as mg/L as the concentration of the solution.

Sample 16

Materials Supplied

- Approximately 60mL standard solution

Preparation

Analyse the sample by your chosen instrumental technique for the following determinands:

- Thiocyanate

N.B. Results should be reported as SCN.

Sample 17

Materials Supplied

- Approximately 60mL standard solution

Preparation

Analyse the sample by your chosen instrumental technique for the following determinands:

- Total fluoride



Sample Preparation Instructions

Group C – Organics

N.B. The weight of the sample plus the vial on preparation is given on all the vials. Please reweigh before analysis and if the weight difference is greater than 40mg please contact contest@lqcpt.com

Sample 3c

Materials Supplied

- Approximately 70g prepared soil, ground to pass through a 200µm sieve

Preparation

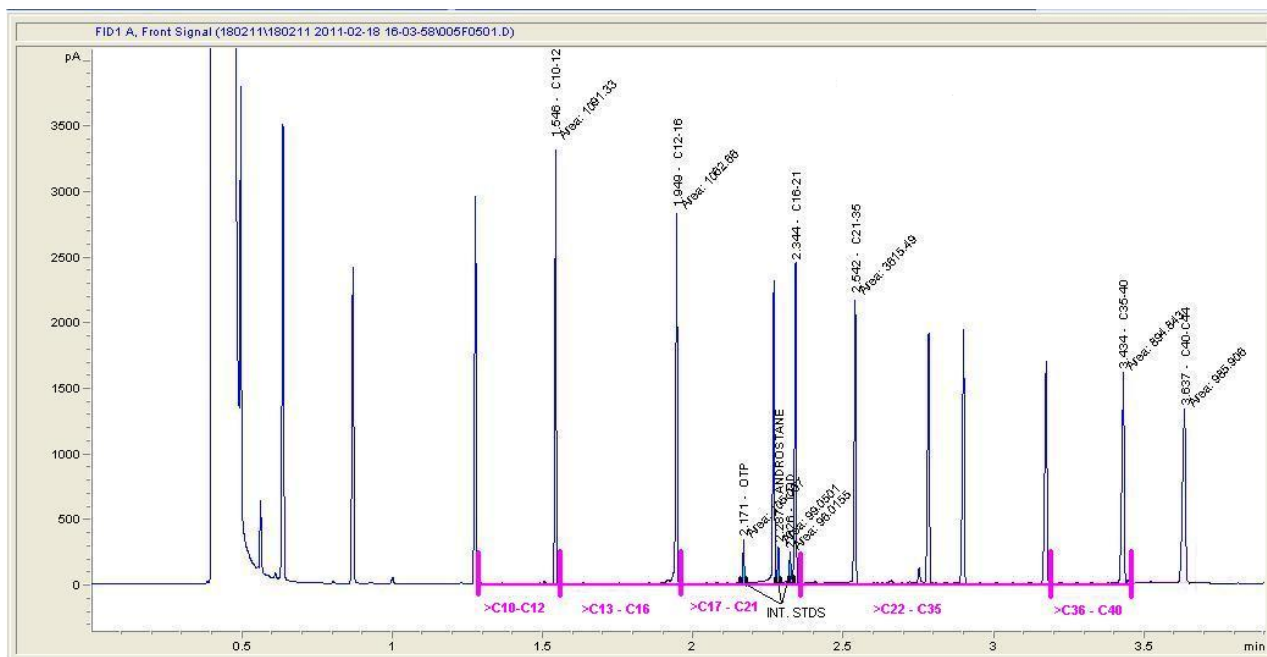
Analyse the sample by your chosen instrumental technique for the following determinands:

- Acenaphthene
- Acenaphthylene
- Anthracene
- Benz(a)anthracene
- Benzo(b)fluoranthene
- Benzo(k)fluoranthene
- Benzo(b/k)fluoranthene (sum of the 2 isomers)
- Benzo(ghi)perylene
- Benz(a)pyrene
- Chrysene
- Dibenz(ah)anthracene
- Fluoranthene
- Fluorene
- Indeno(123-cd)pyrene
- Naphthalene
- Phenanthrene
- Pyrene
- Total PAH
- Phenols
- Cresols
- Xylenols
- Distillable phenolic substances
- PCB 28
- PCB 52
- PCB 101
- PCB 118
- PCB 138
- PCB 153
- PCB 180
- Elemental sulphur
- Total organic carbon
- Total petroleum hydrocarbons (C₁₀-C₄₀ inclusive)
- TPH aliphatic >C₁₀-C₁₂
- TPH aliphatic >C₁₂-C₁₆
- TPH aliphatic >C₁₆-C₂₁
- TPH aliphatic >C₂₁-C₃₅
- TPH aliphatic >C₃₅-C₄₀
- TPH aromatic >EC₁₀-EC₁₂
- TPH aromatic >EC₁₂-EC₁₆
- TPH aromatic >EC₁₆-EC₂₁
- TPH aromatic >EC₂₁-EC₃₅
- TPH aromatic >EC₃₅-EC₄₀

Sample Preparation Instructions

Please find below a visual indication of how to correctly integrate the TPH bands for reporting in the CONTEST scheme:

Note: The bands shown in the chromatogram may not be those required for reporting in the CONTEST scheme.



Sample 5

Materials Supplied

- Approximately 2mL standard solution in hexane (for GC analysis)

N.B. These solutions must be stored in a refrigerator.

Preparation

Analyse the sample by your chosen instrumental technique for the following determinands:

- | | |
|---|------------------------|
| • Acenaphthene | • Chrysene |
| • Acenaphthylene | • Dibenz(ah)anthracene |
| • Anthracene | • Fluoranthene |
| • Benz(a)anthracene | • Fluorene |
| • Benzo(b)fluoranthene | • Indeno(123-cd)pyrene |
| • Benzo(k)fluoranthene | • Naphthalene |
| • Benzo(b/k)fluoranthene (sum of isomers) | • Phenanthrene |
| • Benzo(ghi)perylene | • Pyrene |
| • Benz(a)pyrene | |



Sample Preparation Instructions

Sample 7a

Materials Supplied

- Approximately 60mL standard solution in sodium hydroxide (for analysis by distillation and subsequent colorimetric analysis)

Preparation

Analyse the sample by your chosen instrumental technique for the following determinands:

- Monohydric phenols (by distillation)

Sample 7b

Materials Supplied

- 2 x approximately 2mL standard solutions in methanol/water (for instrumental analysis)

Preparation

Analyse the sample by your chosen instrumental technique for the following determinands:

- Total phenols (sum of phenol, cresols and xylenols)
- Total cresols
- Total xylenols
- Phenol

Sample 11

Materials Supplied

- Approximately 2mL standard solution in isooctane

Preparation

Analyse the sample by your chosen instrumental technique for the following determinands:

- PCB 28
- PCB 52
- PCB 101
- PCB 118
- PCB 138
- PCB 153
- PCB 180



Sample Preparation Instructions

Sample 14a

Materials Supplied

- Approximately 2mL standard solution in hexane

N.B. This solution must be stored at 4(±2)°C.

Preparation

Analyse the sample by your chosen instrumental technique for the following determinands:

- Total petroleum hydrocarbons (C₁₀-C₄₀ inclusive)

Sample 14b

Materials Supplied

- Approximately 2mL standard solution in methanol

N.B. This solution must be stored at 4(±2)°C.

Preparation

Dilute the sample supplied by a factor of 100 (e.g. 0.1mL made up to 10mL) using **deionised** water then analyse the resulting solution plus a blank by your chosen instrumental technique for the following determinands. **Do not correct the results for these dilutions.**

- TPH Aliphatic ≤C₆
- TPH Aliphatic >C₆-C₈
- TPH Aliphatic >C₈-C₁₀
- TPH Aromatic C₆
- TPH Aromatic >C₆-C₈
- TPH Aromatic >C₈-C₁₀

Sample 15

Materials Supplied

- Approximately 2mL standard solution in methanol

N.B. This solution must be stored at -20°C or below

Preparation

Dilute the sample supplied by a factor of 10,000 (e.g. 0.1mL made up to 1L) using **deionised** water then analyse the resulting solution plus a blank by your chosen instrumental technique for the following determinands. **Do not correct the results for these dilutions.**

- Benzene
- Toluene
- Ethylbenzene
- o-Xylene
- m- and p-xylene



Sample Preparation Instructions

Sample 19

Materials Supplied

- Approximately 2mL standard solution in methanol

N.B. This solution must be stored at -20°C or below

Preparation

Dilute the sample supplied by a factor of 10,000 (e.g. 0.1mL made up to 1L) using **deionised** water then analyse the resulting solution plus a blank by your chosen instrumental technique for the following determinands. **Do not correct the results for these dilutions.**

- Chlorobenzenes (Sum of 1,2,4-trichlorobenzene and 1,2,3-trichlorobenzene)
- 2-Chlorotoluene
- 1,2-Dichloroethane
- 1,2-Dichloroethene
- Dichloromethane
- Hexachloro-1,3-butadiene
- 1,1,1,2-Tetrachloroethane
- 1,1,1-Trichloroethane
- Tetrachloromethane
- Trichloroethene
- Trichloromethane
- Vinyl chloride

N.B. Vinyl chloride is extremely volatile. This solution must be analysed promptly.

Sample 20

Materials Supplied

- 2 x approximately 10mL standard solutions in dichloromethane (sample and blank)

Preparation

Analyse the sample as received, or dilute if necessary, to bring into the working range of your instrument. Identify the six unknowns present in the test sample and select the appropriate compound from the drop down list on the result entry page of the reporting system.

A blank is supplied to eliminate adventitious contaminants. The unknowns should be present in the solution only and not present (or present at trace levels) in the blank.

Possible chemicals found in this sample can be found on the following page:



Sample Preparation Instructions

Sample 20 possible chemicals

Chemical	CAS Number
1,2,4,5-Tetrachlorobenzene	95-94-3
1,2,4-Trichlorobenzene	120-82-1
1,3-Dinitrobenzene	99-65-0
2,3,4,6-Tetrachlorophenol	58-90-2
2,4,5-Trichlorophenol	95-95-4
2,4,6-Trichlorophenol	88-06-2
2,4-Dichlorophenol	120-83-2
2,4-Dinitrotoluene	131-14-2
2,6-Dinitrotoluene	606-20-2
2-Acetylaminofluorene	53-96-3
2-Chloronaphthalene	91-58-7
2-Chlorophenol	95-57-8
2-Methylnaphthalene	91-57-6
2-Nitroaniline	88-74-4
3-Methylcholanthrene	56-49-5
4-Bromophenyl phenyl ether	101-55-3
4-Chloro-3-methylphenol	59-50-7
4-Chlorophenyl phenyl ether	7005-72-3
4-Nitrophenol	100-02-7
5-Nitro-o-toluidine	99-55-8
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Acetophenone	98-86-2
Anthracene	120-12-7
Benz(a)anthracene	56-55-3
Benzo(a)pyrene	50-32-8
Benzo(b)fluoranthene	205-99-2
Benzo(g,h,i)perylene	191-24-2
Benzo(k)fluoranthene	207-08-9
Benzyl alcohol	100-51-6
Bis(2-chloroethoxy)methane	111-91-1
Bis(2-chloroethyl)ether	111-44-4
Bis(2-chloroisopropyl)ether	108-60-1
Bis(2-ethylhexyl)phthalate	117-81-7
Butyl benzyl phthalate	85-68-7
Carbazole	86-74-8

Chemical	CAS Number
Chrysene	218-01-9
Dibenz(a,h)anthracene	53-70-3
Dibenzofuran	132-64-9
Diethyl phthalate	84-66-2
Dimethyl phthalate	131-11-3
Dimethylaminoazobenzene	60-11-7
Di-n-butyl phthalate	84-74-2
Ethyl methanesulfonate	62-50-0
Fluoranthene	206-44-0
Fluorene	86-73-7
Hexachlorobenzene	118-74-1
Hexachlorobutadiene	87-68-3
Hexachloroethane	67-72-1
Hexachloropropene	1888-71-7
Indeno(1,2,3-cd)pyrene	193-39-5
Isophorone	78-59-1
Isosafrole	120-58-1
m-Cresol	108-39-4
Naphthalene	91-20-3
Nitrobenzene	98-95-3
N-Nitrosodiethylamine	55-18-5
N-Nitrosodi-n-butylamine	924-16-3
N-Nitrosodi-n-propylamine	621-64-7
N-Nitrosomethylethylamine	10595-95-6
N-Nitrosomorpholine	59-89-2
N-Nitrosopiperidine	100-75-4
N-Nitrosopyrrolidine	930-55-2
o-Cresol	95-48-7
p-Cresol	106-44-5
Pentachlorobenzene	608-93-5
Pentachloronitrobenzene	82-68-8
Phenacetin	62-44-2
Phenanthrene	85-01-8
Phenol	108-95-2
Pyrene	129-00-0
Safrole	94-59-7



Sample Preparation Instructions

Group D – Leachate

Sample 18

Materials Supplied

- Approximately 200g prepared soil, ground to pass through a 200µm sieve

Preparation

Using a 'dry' sample (as received), extract the sample using a 10:1 leachate:soil ratio. Analyse the sample by your chosen instrumental technique for the following determinands:

- Antimony
- Arsenic
- Barium
- Cadmium
- Calcium
- Chromium
- Copper
- Iron
- Lead
- Magnesium
- Mercury
- Molybdenum
- Nickel
- Potassium
- Selenium
- Sodium
- Tin
- Zinc
- Sulfate (as SO₄)
- Chloride (as Cl)
- Fluoride (as F)
- Nitrate (as NO₃)
- Boron
- Chromium (VI)
- Phosphate (as P)
- Ammonia (as N)
- Free cyanide (as CN)
- Total cyanide (as CN)
- Thiocyanate (as SCN)
- pH
- Conductivity (20°C)
- COD (as O₂)
- TOC/ DOC*
- Phenol Index

N.B. Results should be reported as mg/L

Because of the very low levels of some analytes found in the leachate, many are not at a sufficient concentration for z scores to be calculated. The soil for leaching has therefore been spiked with a number of different parameters to raise the concentration of these to measureable levels.

***N.B. Total organic carbon and dissolved organic carbon are regarded as the same determinand in the leachate sample due to the filtration carried out as part of the analytical procedure.**



Sample Preparation Instructions

Group E - Soils for Waste Acceptance Criteria (in accordance with EN 12457-2:2002 (10:1 single stage leaching test))

Sample 21

Materials Supplied

- Approximately 200g prepared soil, ground to pass through a 4mm sieve

Preparation

- Determine the moisture content (at 105°C) of the material provided PRIOR to determining the concentration of any other analyte.
- Calculate the Dry Matter Content Ratio using the following calculation:

$$DR = 100 \times M_D / M_W$$

Where

DR is the dry matter content ratio

M_D is the mass of the dried test portion (kg)

M_W is the mass of undried test portion (kg).

- Report this value in PORTAL.
- Calculate the required test portion using the following calculations. The amount of sample should be equivalent to 0.09kg of dry mass.

$$\text{Required weight (kg)} = 100 \times 0.09 / DR$$

- Calculate the Moisture Content Ratio using the following calculation:

$$\text{Moisture Content} = 100 \times (M_W - M_D) / M_D$$

Where

M_D is the mass of the dried test portion (kg)

M_W is the mass of undried test portion (kg).

- Calculate the required amount of leachant using the following calculations. This should establish a 10:1 liquid to solid ratio.

$$L = (10 - MC/100) \times 0.09$$

Where

L is the volume of leachant used (in l)

MC is the moisture content ratio (in %) (calculated above)



Sample Preparation Instructions

- Leach the sample according to EN 12457-2
- Analyse the sample by your chosen instrumental technique for the following determinands:

• Antimony
• Arsenic
• Barium
• Cadmium
• Chromium
• Copper
• Lead
• Mercury
• Molybdenum
• Nickel
• Selenium
• Zinc
• Sulfate (as SO ₄)
• Chloride (as Cl)
• Fluoride (as F)
• Phenol Index
• DOC
• Total dissolved solids

N.B. Results obtained by the leaching test will be expressed in mg/L, however the results obtained from the eluate should be expressed as the amount of constituent leached relative to the total mass of the sample, in mg/kg of dry matter. Participants must use the following calculation to convert their results:

$$A = C \times ((L/0.09) + (MC/100))$$

Where

A is the concentration of the analyte in mg/kg dry matter (this must be reported as the result);

C is the concentration of the analyte in the eluate (in mg/l);

L is the volume of leachant used (in l);

MC is the moisture content ratio.



Sample Preparation Instructions

Sample 24

Materials Supplied

- Approximately 1L leachate solution

Preparation

Analyse the sample by your chosen instrumental technique for the following determinands:

- Antimony
- Arsenic
- Barium
- Cadmium
- Chromium
- Copper
- Lead
- Mercury
- Molybdenum
- Nickel
- Selenium
- Zinc
- Sulfate (as SO₄)
- Chloride (as Cl)
- Fluoride (as F)
- Phenol Index
- DOC
- Total dissolved solids

Results should be reported in mg/L



Sample Preparation Instructions

BTEX in Soil

Sample 22

Materials Supplied

- Approximately 2mL standard solution in methanol
- Approximately 30g soil material

N.B. This solution must be stored at -20°C or below

Preparation

Accurately transfer 0.5mL of the standard solution to 10g of the soil supplied. Perform an extraction of the entire sub-sample and analyse by your chosen instrumental technique for the following determinands. **Do not correct the results for these dilutions.**

- Benzene
- Toluene
- Ethylbenzene
- o-Xylene
- m- and p-xylene

Report each analyte result in mg/kg of the prepared soil sample.

TPH in Soil

Sample 23

Materials Supplied

- Approximately 2mL standard solution in methanol
- Approximately 30g soil material

N.B. This solution must be stored at -20°C or below

Preparation

Accurately transfer 0.5mL of the standard solution to 10g of the soil supplied. Perform an extraction of the entire sub sample and analyse by your chosen instrumental technique for the following determinands. **Do not correct the results for these dilutions**

- TPH, Aliphatic $\leq C_6$
- TPH, Aliphatic $>C_6-C_8$
- TPH, Aliphatic $>C_8-C_{10}$
- TPH, Aromatic C_6
- TPH, Aromatic $>C_6-C_8$
- TPH, Aromatic $>C_8-C_{10}$

Report each analyte result in mg/kg of the prepared soil sample.



Sample Preparation Instructions

VOCs in Soil

Sample 25

Materials Supplied

- Approximately 2mL standard solution in methanol
- Approximately 30g soil material

N.B. This solution must be stored at -20°C or below

Preparation

Accurately transfer 0.5mL of the standard solution to 10g of the soil supplied. Perform an extraction of the entire sub sample and analyse by your chosen instrumental technique for the following determinands. **Do not correct results for these dilutions.**

- Chlorobenzenes
- 2-Chlorotoluene
- 1,2-Dichloroethane
- 1,2-Dichloroethene
- Dichloromethane
- Hexachloro-1,3-butadiene
- 1,1,1,2-Tetrachloroethane
- 1,1,1-Trichloroethane
- Tetrachloromethane
- Trichloroethene
- Trichloromethane

Report each analyte result in mg/kg of the prepared soil sample.

Organochlorine pesticides (OCs) in Soil

Sample 26

Materials Supplied

- Approximately 2mL standard solution in methanol
- Approximately 30g soil material

N.B. This solution must be stored at -20°C or below

Preparation

Accurately transfer 0.5mL of the standard solution to 10g of the soil supplied. Perform an extraction of the entire sub sample and analyse by your chosen instrumental technique for the following determinands. **Do not correct results for these dilutions.**

- Endrin
- Dieldrin



CONTEST

Contaminated Land Proficiency Testing Scheme

Version 17

Issue Date 05/09/2014

Sample Preparation Instructions

- Aldrin
- p,p'-DDT
- o,p'-DDT
- p,p'-DDE
- p,p'-DDD
- Alpha Hexachlorocyclohexane
- Beta Hexachlorocyclohexane
- Delta Hexachlorocyclohexane
- Lindane (Gamma HCH)
- Trifluralin
- Alpha Endosulphan
- Beta Endosulphan
- Hexachlorobenzene
- Heptachlor
- Heptachlor epoxide
- Pentachlorobenzene

Report each analyte result in mg/kg of the prepared soil sample.