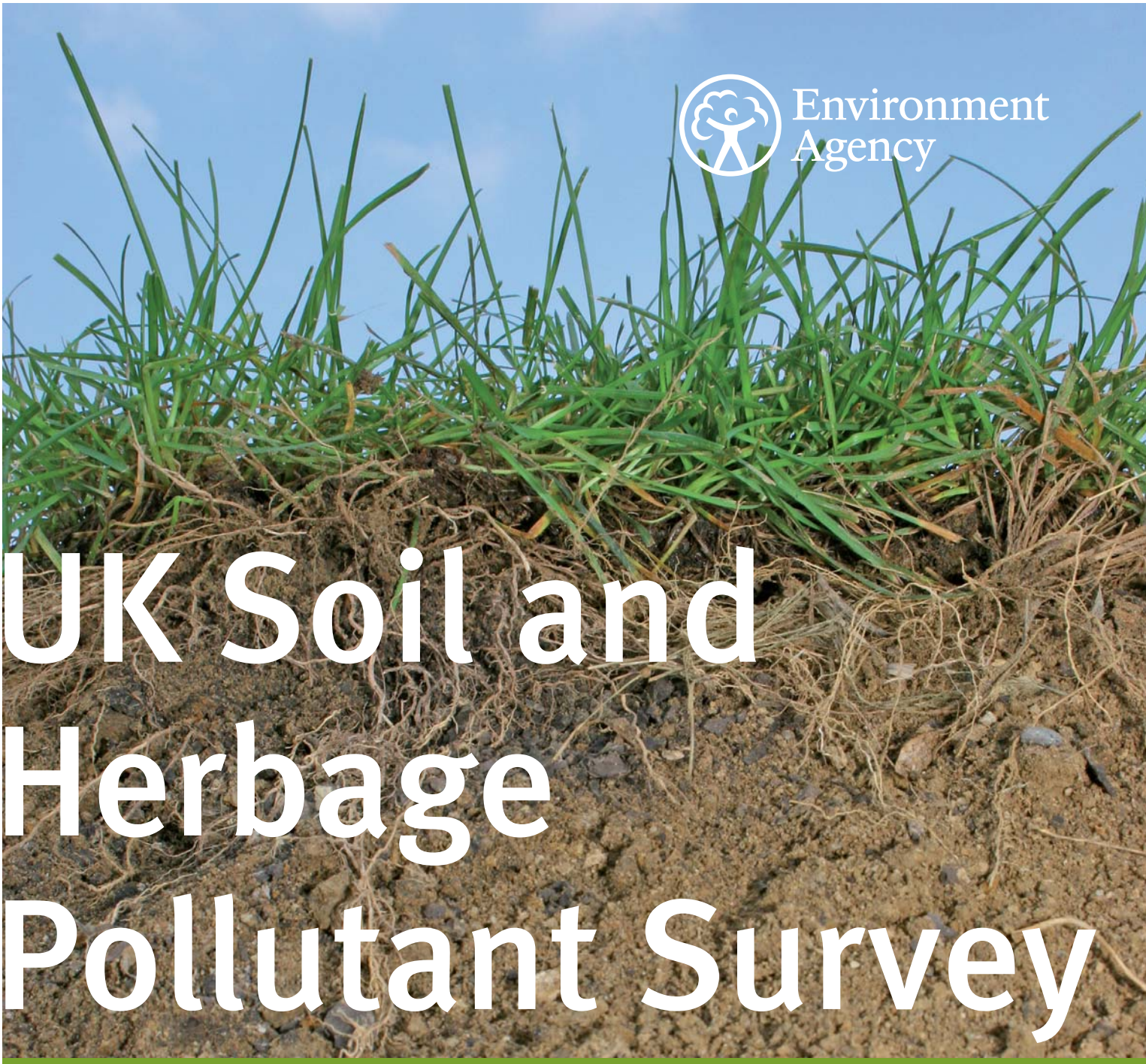

Environment Agency

UK Soil and Herbage Pollutant Survey

UKSHS Report No. 3

Chemical analytical methods

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Author(s):
Fox, I., Hunter C., Hudson, R.

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Research Contractor:
School of Biological Sciences, University of Liverpool
Liverpool, L69 3BX, UK
Tel: +44(0) 151 7945291

Environment Agency's Project Manager:
Dr Peter Crook, Block 1, Government Buildings,
Burghill Road, Westbury-on-Trym, Bristol, BS10 6BF

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Steve Killeen

Head of Science

Executive summary

This report describes the laboratory test methods used by the Environment Agency's National Laboratory Service (NLS) for the chemical analysis of samples utilised within the UK Soil and Herbage Survey (UKSHS). The report provides:

- method summaries for metals and organics;
- details of the method validation studies for these two categories;
- information about the peer reviews;
- a description of sample and data handling methods.

Supporting information and descriptions are provided on an accompanying CD-ROM.

The laboratory tests methods applied by the NLS to the UKSHS samples submitted for chemical analysis have the following important features:

- robust, peer-reviewed methods;
- modern analytical instrumentation;
- quality assurance procedures accredited by the United Kingdom Accreditation Service (UKAS);
- externally validated performance characteristics;
- experienced and qualified scientists.

As such, the chemical data produced for the survey are reliable and fit-for-purpose.

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Glossary of terms

Bias	Systematic error – a consistent difference between the mean of many results and the true value.
Control sample	A sample which is analysed for quality control purposes.
Determinand	General term for any numerical property of a sample chemical quality variable (e.g. cadmium).
Laboratory information management system (LIMS)	The electronic system by which the analytical information is managed within the laboratory (StarLIMS).
Limit of detection (LoD)	The concentration of a determinand at which there is a desirably small probability this it will not be detected.
Minimum reporting value (MRV)	A minimum concentration selected for reporting purposes (i.e. the less than value), which is usually higher than the statistically derived method limit of detection. It provides consistency of reporting as well as an allowance for sample variation.
Precision	The degree of agreement existing between repeated measurements on the same sample made under specified conditions. Precision of results (or rather lack of it) is usually expressed in terms of standard deviation.
Quantitation	The calculation process of converting instrument signal responses, from prepared test samples, into concentrations of compounds in the sample matrix concerned. Details of the quantitation process for each analytical technique are included in the test methods.
Sample registration	The process of registering samples into the laboratory's system of sample and data management which includes assigning identification and analysis.

1 Introduction

1.1 Background

The UK Soil and Herbage Pollutant Survey (UKSHS) is a research project sponsored jointly by:

- Environment Agency
- Department for Environment, Food and Rural Affairs (DEFRA)
- National Assembly for Wales
- Food Standards Agency
- Food Standards Agency Scotland
- Scottish Environment Protection Agency (SEPA)
- Environment and Heritage Service (Northern Ireland)
- Scotland and Northern Ireland Forum for Environmental Research (SNIFFER).

Dr Peter Crook from the Environment Agency provided overall project management on behalf of the sponsors. A consortium led by the University of Liverpool's School of Biological Sciences was commissioned to undertake the work. The consortium consisted of the Environment Agency's National Laboratory Service (NLS), Nottingham Trent University, the University of Stirling and the University of Liverpool (UoL), with additional assistance being provided by Parkman Ltd and the Environmental Advice Centre (EAC) Ltd.

The project's primary objective was to establish a baseline for pollutant levels in soil and herbage in the UK. The field-based component of the UKSHS involved the collection of soil and herbage samples for chemical and radiometric analysis from industrial, rural and urban sites throughout the UK (see UKSHS Report No. 2). The samples were analysed by the NLS and the UoL's radiometric laboratory (see UKSHS Report Nos 3 and 4). All sample collection and laboratory-based methods used within the UKSHS were accredited to ISO17025 by the United Kingdom Accreditation Service (UKAS).

1.2 Aim of the report

This report provides a factual record of the laboratory test methods used for the chemical analysis of samples from the UKSHS. The report focuses on the analytical methods employed by the NLS and the efforts taken to ensure that the data produced for the survey are fit-for-purpose.

The report provides:

- method summaries for metals and organics (Sections 2 and 3);
- details of the method validation studies for these two categories (Sections 4 and 5);
- information about the peer reviews (Section 6);
- a description of sample and data handling methods (Section 7).

Supporting documents are provided in electronic format as on CD-ROM. These contain the full UKAS accredited test methods for both organics and metals analysis, along with all the method test data produced during validation of the methodology. Details are given of all interlaboratory calibration exercises, drying tests, recoveries, blanks and ^{13}C-labelling studies. Lists of files on the CD-ROM are given in Appendix 1.

2 Method summaries – organics

2.1 Dioxins/furans and polychlorinated biphenyls

Samples were air-dried at <20ºC (typically 18-20ºC) and then extracted into toluene using a Dionex Accelerated Solvent Extraction (ASE™) system. A three-stage clean-up/separation was performed to clean up the extract to remove fats, sulphur and other interfering compounds.

The sample was first treated with activated, acidic and basic silica gel to remove fats and organic contaminants. It was then passed through an automated gel permeation chromatography (GPC) system, where contaminants were removed on a size exclusion basis. The sample was then fractionated using an alumina liquid chromatography column to separate the dioxins and furans from the polychlorinated biphenyls (PCBs). Each fraction was then concentrated to a volume suitable for injection into a capillary gas chromatograph.

Analysis was by high-resolution gas chromatography mass spectrometry (HR GC-MS) using splitless injection onto a capillary gas chromatography (GC) column. The analysis was performed on a Finnigan-MAT 95 high-resolution mass spectrometer system, with a Hewlett Packard HP 6890 gas chromatograph inlet. In order to resolve the co-elution of PCB 28 and PCB 31, a GC column designed specifically for separation of these compounds was used (the parameters are given in Appendix K of Method HRMS 3 on the CD-ROM).

Quantitation was based upon the isotope dilution method. ^{13}C-labelled isotopes of the compounds of interest were added to the sample before extraction and these isotopes were used for quantitation. Internal standards were used to correct for losses of each individual isotope of interest in the extraction and clean-up stages.

Results were quoted on an individual native isomer basis, although total native dioxin and furan isomer results for each level of chlorination could also be quoted along with an international toxic equivalent (I-TEQ) value.

2.2 Polycyclic aromatic hydrocarbons (PAHs)

Solid samples were extracted into 50:50 acetone: dichloromethane (DCM), using a Dionex Accelerated Solvent Extraction (ASE™) system. A two-stage clean-up process was used to clean up the extract to remove fats, sulphur and other interfering compounds.

The extract was first passed through a Fluorisil solid phase extraction (SPE) clean-up column to remove non-organic contamination. The sample was then passed through an automated GPC system, where contaminants were removed on a size exclusion basis. Finally, the sample was concentrated to a volume suitable for injection into a gas chromatograph. Analysis was by high-resolution gas chromatography – low-resolution mass spectrometry (HRGC-LRMS) using programmed temperature vaporisation (PTV) injection onto a capillary GC column. The analysis was performed on a Hewlett Packard 5973n mass spectrometer system, with a Hewlett Packard HP 6890 gas chromatograph inlet.

The mass spectrometer was run in selected ion monitoring (SIM) mode and quantitation was based on the calibration curve method, with internal standard correction. Deuterated isotopes of the compounds of interest were added to the sample before extraction. These isotopes were

used as internal standards to correct for losses of each individual compound of interest in the extraction and clean-up stages. Results were quoted on an individual compound basis.

Full details of each method are included on the accompanying CD-ROM (Folder 2 Organics Data).

3 Method summaries – metals

3.1 Inductively coupled plasma mass spectrometry (ICPMS) – quantitative

A Perkin Elmer Elan ICPMS was used to analyse soil and herbage samples for:

- cadmium
- chromium
- copper
- lead
- nickel
- platinum
- tin
- titanium
- zinc.

The samples were refluxed with aqua regia to extract metals from the solid into solution. Digested samples were then introduced into the spectrometer via a nebuliser.

ICPMS generates singly charged ions from the elements present in a sample. These ions are directed into a quadrupole mass spectrometer, which separates the ions according to their mass to charge ratio. Ions of the selected mass/charge (specific for each element) are directed to a detector. The number of ions detected is proportional to the concentration of each element in the sample.

When platinum was added to the analytical schedule, it was decided to utilise the same sample digest and ICPMS technique to keep costs within the scope of the project. This approach does not yield as limits of detection (LoD) for platinum that are as low as other techniques, which employ a 'fire assay' and then determination by ICPMS or neutron activation analysis (NAA). The separation and pre-concentration of platinum group elements (PGE) by the fire assay technique is well established (Haffty *et al.* 1977). When coupled with ICPMS or NAA as the final step, detection limits at the low μg/kg level can be achieved; the main limitation is the purity of the reagents used in the fusion process. However, this approach requires a much larger sample (typically 50 g) than employed in the UKSHS and an extra analytical procedure.

3.2 Inductively coupled plasma mass spectrometry (ICPMS) – semi-quantitative scans

The ICPMS instrument can also be operated in semi-quantitative scan mode. Sample digests were analysed in a similar fashion to those described in Section 3.1, but with the instrument set to scan a range of masses. These scans provide qualitative and semi-quantitative information on the majority of the metals in the periodic table. For most metals, the scan gives an indication of the order of magnitude of the concentration of the element present (normally within ±50 per cent of the concentration in the digest).

This approach offers a cost-effective way of widening the scope of the analysis to include other toxic metals that were not included in the standard (quantitative analysis) suite. Other metals of potential interest, which would be detected by the scan, include:

- beryllium
- bismuth
- cobalt
- lithium
- molybdenum
- palladium
- silver
- thallium
- uranium.

Full quantitation of any interesting metals detected could then be achieved by following up with a quantitative method, 'homing in' on the specific areas of interest.

The additional information may enable patterns of contamination to be recognised more easily. It may also permit comparisons with regional geochemical data (e.g. British Geological Survey) to help distinguish between 'natural' levels and those introduced through anthropogenic contamination.

3.3 Inductively coupled plasma optical emission spectrometry (ICPOES)

This technique was used to analyse samples for manganese and vanadium in soil and herbage. The samples were refluxed with aqua regia to extract metals from the solid into solution. The sample extracts were introduced via a nebuliser into a Perkin Elmer Optima ICPOES instrument.

The ICPOES instrument operates at a temperature of 6,000–8,000K. This high temperature breaks chemical bonds, liberates elements present and transforms them into a gaseous atomic state. A number of the atoms pass into the excited state and emit radiation when the atom or ion returns to a more stable configuration or the ground state. The wavelengths of radiation emitted are specific to the elements present in the sample. The intensity of the radiation is proportional to the amount of each element present within the solution and so can be used for quantitative purposes.

3.4 Cold vapour generation atomic absorption spectrometry (CV-AAS)

This technique was used to analyse for mercury in soil and herbage. The instrument used was a Perkin Elmer 4100 atomic absorption spectrometer, fitted with a FIAS-200 automated interface.

After refluxing with aqua regia to extract the mercury from the solid into solution, the samples were reacted with sodium borohydride to convert mercury (II) in the extracts to mercury (0). The mercury was removed from solution as a vapour and introduced into the atomic absorption spectrometer via the automated flow injection interface (FIAS).

Atomic absorption occurs when a ground state atom absorbs energy in the form of light of a specific wavelength and is elevated to an excited state. The amount of light energy absorbed

(absorbance) at this wavelength increases as the number of atoms of the selected element present increases.

3.5 Hydride generation atomic absorption spectrometry

The system described in Section 3.4 was also used to analyse the samples for arsenic. The solid sample was digested with nitric acid and ashed with magnesium nitrate to destroy organic matter under oxidising conditions. The inorganic arsenic resulting from this procedure was extracted into hydrochloric acid and reduced under carefully controlled conditions to arsenic (III).

The sample was mixed with sodium borohydride to produce a hydride. This volatile hydride was swept into a quartz cell on an atomic absorption spectrometer via the automated flow injection interface (FIAS). The hydride decomposes to give arsenic in its atomic state, which is measured as described in Section 3.4.

Appendix 2 demonstrates how the samples were processed through the laboratories. Full details of the various test methods are given on the CD-ROM (Folder 4 Metals Data).

4 Method validation studies – organics

4.1 Drying tests

Concerns were voiced about possible losses of the more volatile compounds (especially PAHs and PCBs) if vigorous drying protocols were used. A number of exercises were therefore conducted to ascertain the best method of drying the soil and herbage samples.

In the previous survey for the then Ministry of Agriculture, Fisheries and Food (MAFF), the soil samples were air-dried at 23–28°C. The associated herbage work involved freeze-drying the grass samples. The results of further work carried out for the current survey indicated that samples should be air-dried at <20ºC.

For this study, the following spiking protocol was adopted. The appropriate amount of labelled (either deuterated or ^{13}C) internal standard was diluted to approximately 1 ml with acetone. Using a Pasteur pipette, this solution was dripped over the wet soil or herbage matrix in an aluminium foil tray. The sample was then subjected to the relevant drying process at <20°C until dry.

A control matrix was prepared by spiking the appropriate internal standard after drying and extracting this along with the above dried samples using the laboratory's normal procedure.

The results indicated that air-drying was preferential to freeze-drying. As expected, some losses occurred, but adequate recoveries were achieved for all compounds of interest using the air-drying method. Details of the results achieved for all of the drying tests are provided as a separate file on the accompanying CD-ROM (Folder 3 Drying Tests).

4.2 Interlaboratory calibration exercises

In addition to the normal method performance testing procedures used to establish the precision, bias and limit of detection (LoD), a number of interlaboratory test exercises were conducted independently of the UKSHS and covering all of the compounds of interest.

NLS's performance in these exercises was very good and the results are given on the CD-ROM (Folder 2 Organics Data). The exercises conducted are listed in Table 4.1.

Table 4.1 – Summary of interlaboratory calibration exercises

Compounds	Exercise	Spreadsheets on CD-ROM
Dioxins and furans	International Sediment Exchange for Testing of Organic Contaminants (SETOC) – distributions 99.4, 00.4 and 01.4, organised by the Wageningen Agricultural University, the Netherlands	SETOC 99.4 SETOC 00.4 SETOC 01.4
	Consorzio Interuniversitano 'La Chimica per L'Ambiente' (INCA), Venice, Italy – Italian Sediment 2nd Exercise	Italian Sediment
	UMEÅ University, Sweden – Intercalibration Exercise 2000	Umea 2000
PCBs	Italian Sediment 2nd Exercise	Italian Sediment
	UMEÅ Intercalibration Exercise 2000	Umea 2000
PAHs	Laboratory of the Government Chemist (LGC) intercalibration exercise for river sediment and sewage sludge	LGC River Sediment LGC Sewage Sludge

4.3 Certified reference materials

The methods were further validated by analysing a number of certified reference materials (CRMs). The CRMs used are as shown in Table 4.2.

Table 4.2 – CRMs used in organics analysis

CRM	Description	Spreadsheet on CD-ROM
SRM 1941a New Jersey sediment	Covers PCBs and PAHs	CRM1941a

In addition to these exercises, an interlaboratory comparison exercise was designed and conducted specifically for the UKSHS to ensure that the analytical laboratories involved could analyses soil and herbage samples successfully. The design and results of this exercise are discussed in UKSHS Report No. 5.

4.4 Spiking recoveries

Where original reference values were not available (e.g. herbage), spiking recovery tests were conducted. These tests provided additional information on the suitability of the test methods (Table 4.3).

Herbage samples were spiked with known amounts of the compounds of interest. The results showed excellent recoveries, e.g. 75.9–97.5 per cent for dioxins/furans.

All the organics performance testing data are included on the CD-ROM (Folder 2 Organics Data).

Table 4.3 – Summary of additional information on spiking recoveries

Compounds	Spreadsheets on CD-ROM
Dioxins	Blanks Precision & Recovery AQC Dioxin Recoveries Grass Spiked Recoveries
PCBs	Blanks PCB ^{13}C Recovery Precision & Recovery Grass Spiked Recovery SETOC 738 spike SETOC 738
PAHs	Herbage Recoveries Soil (973) Spiked Recoveries Grass Spiking Recoveries SETOC 701 SETOC 738 SETOC 741

5 Method validation studies – metals

5.1 Certified reference materials

A number of CRMs were selected for the validation of the laboratory analysis (Table 5.1). These tests supplemented the normal method performance testing procedures employed by the NLS, which are used to establish the precision, bias and LoD.

The results obtained were in good agreement with the certified values, indicating that the methods were satisfactory for use in the UKSHS. These data were included in the peer review.

Table 5.1 – CRMs used in metals analysis

CRM	Description
CRM 281 Rye grass	Most appropriate herbage CRM available that covered the majority of metals of interest.
CMI 7003 Agricultural silty clay loam soil	Aqua regia extractable values are available for most metals of interest at low to medium values.
CRM 141R Calcareous loam soil	Aqua regia extractable values are available for most metals of interest at low to medium values.
LGC 6135 Hackney brickworks soil	Aqua regia extractable values are available for most metals of interest, with some elevated levels due to industrial contamination.

5.2 Spiking recoveries

Further spiking recovery tests were conducted when original metal reference values were low or not available. These tests provide additional information on the suitability of the test methods.

Both soil and herbage digests were spiked with known amounts of a range of heavy metals. For the herbage samples, CRM281 was used as the unspiked sample. For soil, CRM 7003 was similarly employed.

The results showed excellent recoveries (96.2–109.8 per cent).

Full results of the metals performance testing data are included on the CD-ROM (Folder 4 Metals Data).

6 Peer reviews

6.1 UKAS accreditation

All of the methods employed by the NLS for this survey were accredited to ISO17025 by the UKAS.

The organics analysis was carried out at NLS Leeds and the metals analysis at NLS Nottingham. The Schedules of Accreditation for these laboratories are shown in UKSHS Report No. 1.

6.2 Organics peer review – Central Science Laboratory

After obtaining UKAS accreditation, the test methods for dioxins, furans, PCBs and PAHs were peer reviewed by Dr Alwyn Fernandes of the Central Science Laboratory (CSL). He examined the laboratory test procedures with respect to the specific needs of the UKSHS (see Appendix 3).

6.3 Metals peer review – Cranfield University

The analytical test methods for the metals were subjected to a similar independent peer review by Professor Peter Loveland of Cranfield University. Details of Professor Loveland's review are shown in Appendix 4.

7 Sample and data handling

7.1 UKSHS sample identification protocol

The large number of similar samples arriving at the laboratory on a regular basis meant that automation of sample and data management was vital.

The risks of mixing up samples and their associated results were reduced to a minimum by adopting the procedures detailed below.

7.2 Pre-registration of samples

The agreement that all UKSHS samples would be submitted by UoL sampling teams to NLS Leeds for registration necessitated a significant amount of data allocation. A series of codes was created and made available on the NLS Laboratory Information Management System (StarLIMS) database at Leeds to facilitate effective registration and subsequent retrieval of all sample and result data.

First, a pair of method codes were obtained from the Environment Agency's National Data Code Set Team to allocate to the two types of matrix that were to be sampled (soil and herbage). The North West Region Data and Information Team manages the National Data Code Set (NDCS) for the whole of the Environment Agency. It allocated the code 38 for soil samples and 39 for vegetation samples to be taken as part of the survey. The analytes to be tested had to be allocated a determinand code (if they had not been previously analysed), which were also defined by the NDCS Team. Each determinand code was combined with the method code to dictate in which matrix the analyte was being measured, e.g. 737/38 is determinand code 737, method code 38 and indicates fluoranthene in soil.

The analytes were then grouped for laboratory use. This involved:

- compiling worksheets and worklists;
- adding quality control samples;
- designing the report layouts used in the laboratory.

Table 7.1 shows the total numbers of analytes.

From this point, all the soil analytes were built into one analytical suite (code XY) and all the vegetation analytes were built into another analytical suite (code XZ). This enabled any sampler to simply request the two-digit code to obtain the full suite of analysis required for the sample in the particular matrix.

The next stage involved creating a unique eight-digit code for each of the sampling points on StarLIMS. Each site and sample combination was assigned a different code. Full details of the codes used are included on the CD-ROM (Folder 5 Sample Identification Codes).

The final stage of the pre-registration process was the creation of unique sampler codes for UoL staff to allow them to register samples successfully onto StarLIMS at NLS Leeds. Two sampler codes were set up (4680 - David Copplestone and 4681 - Sally Bielby). If neither of these codes were used, then a default was used at the point of registration. The defaults were 0000 (Data Section User) or ZZZZ (Unassigned Sampler).

Table 7.1 – Total numbers of analytes

Type of analyte	Number of analytes	Type of sample
PAH	22	Soil
PAH	22	Vegetation
PCB	26	Soil
PCB	26	Vegetation
Dioxin	17	Soil
Dioxin	17	Vegetation
Total dioxin	8	Soil
Total dioxin	8	Vegetation
Metal	14	Soil
Metal	13	Vegetation

7.3 Sample submission

Once pre-registered, samples could be submitted and registered at NLS Leeds by completing a sample reception document. The sample reception document used was an optical character recognition (OCR) form, which interprets marks placed in predefined boxes on the form. An example of the type of form employed is shown in Appendix 5.

When completing a sample reception document, the sampler needed to provide the following information as a minimum for each sample:

- bar code (tag number) identifier
- sample date
- sample time
- sampling officer code
- sample point code
- analytical requirements.

In addition, the UKSHS samples required the addition of a specific sample point name to the comments box to pinpoint the site.

Samples were scheduled on the NLS bespoke scheduling system by creating pre-programmed bar codes that held:

- the sample point code
- the analytical requirements
- the date when the sample was due to be taken.

These bar codes were printed onto ‘document’ labels, which were applied to the sample reception documents at the time of issuing samples. A series of bottle labels were also created with a bar code (tag number) corresponding to the ones on the sample reception documents. This helped the samplers because they did not then need to fill out labels in the field; they simply applied the appropriate pre-printed label.

On occasions, samples were submitted on an ad hoc basis. This involved entering additional data in the form of the appropriate sample point code and analytical information on the back of the sample reception document.

7.4 Sample registration

On receipt of the UKSHS samples at NLS Leeds, the registration documents were passed through an optical scanner and the data interpreted into the StarLIMS database. At this point, each sample was allocated a unique laboratory reference number, thus allowing its progress to be monitored at any point in the laboratory.

Once a sample was registered in StarLIMS, the corresponding bottles were swiped past a bar-code scanner. This generated a label holding the unique laboratory reference number, plus other information such as sample point name and sample date.

The bottles were then distributed to the appropriate analytical sections of the laboratory and, in the case of the metals analysis, the bottles were crated safely and transported to NLS Nottingham via the daily interlaboratory courier. The analytical requests for metals analysis were transmitted across the Environment Agency's wide area network (WAN) from StarLIMS at Leeds to StarLIMS at Nottingham. The results for the metals analysis were transmitted back, on completion, across the WAN in a similar manner from StarLIMS at Nottingham to StarLIMS at Leeds.

7.5 NLS data quality control

Analytical results from laboratory instrumentation were entered onto the NLS StarLIMS laboratory database using a single input system.

The data for dioxins, PCBs and PAHs were cross-checked electronically against the original instrument datafiles using Microsoft® VBA macros in Microsoft® Excel. This automated comparison checked each individual result from the StarLIMS database against the raw instrument datafiles and highlighted any anomalies for investigation and amendment. In addition, a random audit of the data was performed by laboratory personnel to verify the correct operation of the system.

For the metals, the results were entered onto the StarLIMS database and then checked manually against the original raw analytical data. This process eliminated any possible errors arising from inaccurate calculation or transcription.

These validation routines were applied to every single analytical result before release of the data to UoL.

7.6 Data export

The NLS Leeds Data Team produced a script to extract sample and result data for all samples taken as part of the UKSHS. This script was written in structured query language (SQL) and was run against the StarLIMS database, which is an Oracle 8 database installation. The script produced the data in a format that would successfully import into a Microsoft® Access 97 database. A final validation was performed on this electronic export to verify that the correct number of samples and results were present. Minor reformatting was carried out in the Access database using macros. The Access database was then compressed using WinZip® software and transmitted to UoL. These procedures are discussed further in UKSHS Report No. 1.

7.7 Uncertainty of measurement in chemical analysis

Under the terms of its UKAS accreditation, the NLS is required to be able to quote an estimate of uncertainty of measurement (UoM) for any reported result (ISO17025 para. 5.4.6.2). UoM is determined by the NLS according to the principles set out in Section 7.7 of EURACHEM/CITAC Guide CG4 *Quantifying Uncertainty in Analytical Measurement* (Ellison *et al.* 2000).

A pre-requisite for UKAS accreditation is that methods must be performance-tested using samples of a relevant matrix and shown to comply with whatever performance standards are in force.

The calculations for the UKSHS were based on replicate analyses of CRMs for both metals and organics – both during performance testing and alongside the UKSHS analysis. These were supplemented by replicate analysis of SETOC interlaboratory exercise samples for which reference values were established by analysis in parallel with the CRMs. For some determinands present at very low concentrations in the reference samples, a known spike was added so that recovery could be measured. Estimates of total standard deviation and bias/recovery were calculated from the data.

For the metals, UoM was quoted according to the equation given in the NLS Quality Manual, i.e.

$UoM = 2S_t + |\text{mean} - \text{nominal}|$

where:

S_t	=	total standard deviation for the specified period
$2S_t$	$\cong$	95 per cent confidence interval for the result (ref para 2.3.3. of Guide CG4)
mean	=	mean result for the specified period
nominal	=	certified concentration value of the CRM

The equivalent spike recovery term was substituted for the bias term for determinands for which recovery was calculated.

For the organics, the $2S_t$ component was quoted as ‘precision’ and bias/recovery term was quoted separately. This was because UKAS had posed the question of whether bias (being in essence a known quantity) should be included within uncertainty.

List of abbreviations and acronyms

AQC	analytical quality control
ASE	accelerated solvent extraction
CRM	certified reference material
CSL	Central Science Laboratory
CV-AAS	cold vapour atomic absorption spectrometry
DCM	dichloromethane
Defra	Department for Environment, Food and Rural Affairs
EAC	Environmental Advice Centre
FIAS	flow injection interface
GPC	gel permeation chromatography
GC	gas chromatography
HRGC-HRMS	high resolution gas chromatography – high resolution mass spectrometry
HRGC-LRMS	high resolution gas chromatography – low resolution mass spectrometry
ICPOES	inductively coupled plasma optical emission spectrometry
ICPMS	inductively coupled plasma mass spectrometry
INCA	La Chimica per L'Ambiente
I-TEQ	international toxic equivalent
LGC	Laboratory of the Government Chemist
LoD	limit of detection
MAFF	Ministry of Agriculture, Fisheries and Food
NDCS	National Data Code Set
NLS	National Laboratory Service
OCR	optical character recognition
PAH	polycyclic aromatic hydrocarbon
PDF	portable document format
PCB	polychlorinated biphenyl
PTV	programmed temperature vaporisation
SEPA	Scottish Environment Protection Agency
SETOC	International Sediment Exchange for Testing of Organic Contaminants
SIM	selected ion monitoring
SNIFFER	Scotland and Northern Ireland Forum for Environmental Research
SPE	solid phase extraction
SQL	Structured Query Language
StarLIMS	Laboratory Information Management System
UoL	University of Liverpool

UoM	uncertainty of measurement
UKAS	United Kingdom Accreditation Service
UKSHS	UK Soil and Herbage Pollutant Survey
VBA	Visual Basic for Applications
WAN	Wide Area Network

References

Ellison S L R, Rosslein M and Williams A, 2000 Editors *Quantifying Uncertainty in Analytical Measurement* (2nd edn.). EURACHEM/CITAC Guide CG4. Available from: http://www.eurachem.ul.pt/guides/QUAM2000-1.pdf [Accessed 20 September 2005]

Haffty J, Riley L B and Goss W D, 1977 *A manual on fire assaying and determination of the noble metals in geological materials*. US Geological Survey Bulletin 1445. Washington DC: US Geological Survey.

Appendix 1: Supporting files on CD-ROM

A number of files containing supporting information to this report are provided on an accompanying CD-ROM in six folders. The CD-ROM is available from the Environment Agency publications catalogue (publications.environment-agency.gov.uk) under the following product code: **SCHO0607BMTG-E-C**

These six folders contain information about:
1 Equipment Tests
2 Organics Data (includes method details and performance data)
3 Drying Tests
4 Metals Data (includes method details and performance data)
5 Sample Identification Codes
6 NLS Personnel involved in the UKSHS
For ease of reference, the contents of these files are outlined below

Drying tests

All the sample drying test data are held on the CD-ROM in the folder named '3 Drying Tests'. Tests were conducted on both soil and herbage for the more volatile PAHs and PCBs. Test data are held as a Microsoft® Excel workbook file. Table A1 lists details the contents of each spreadsheet within the workbook.

Table A1 – Contents of drying test spreadsheets (held in 3 Drying Tests)

Spreadsheet name	Data relating to:
PCB Drying 1	Grass and soil air-dried at 20–25ºC
PCB Drying 2	Grass and soil air-dried at <20ºC
PCB Drying 3	Grass and soil air-dried at <20ºC compared with freeze-drying
PCB Drying 4	Losses of PCB 18 from soil versus drying time at <20ºC
PAH Herbage Drying 1	Herbage air-dried at <20ºC
PAH Herbage Drying 2	Herbage air-dried at <20ºC (repeat tests)
PAH Soil Drying	Soil air-dried at <20ºC

Organics methods

The folder containing these methods is annotated as '2 Organics Data' on the CD-ROM. Within this folder are the full transcripts of each UKAS accredited method employed in the UKSHS in Microsoft® Word format. Method HRMS 2 relates to the analysis of PAHs, while method HRMS3 deals with dioxins and PCBs. The methods contain details of;

- principle
- hazards
- performance characteristics

- apparatus, reagents and equipment used for sample analysis.

Details of the quality control procedures are also given, along with the full analytical protocol.

Organics test method performance data

Three Microsoft® Excel workbook files are held on the CD-ROM in the folder named '2 Organics Data'. These files contain compilations of the test method performance data as listed in Tables A2–A4.

Table A2 – Compilation of dioxin data (held in 2 Organics Data)

Spreadsheet name	Data relating to:
Blanks	Measured concentrations in 10 batches
Precision & Recovery AQC	Precision and recovery data for 10 batches of analytical quality control (AQC) sample (includes mean, standard deviation and relative standard deviation, as well as mean recovery)
Dioxin Recoveries	Results of the recoveries of ^{13}C internal standards of one sample chosen at random from each of nine batches
Grass Spiked Recoveries	Recovery data for replicate (four) analyses over two separate batches (includes mean, standard deviation and relative standard deviation, as well as mean recovery)
CS3	Results (including mean and standard deviation) for 15 batches of analysis of the reference material CS3
Italian Sediment	NLS results for the Italian Sediment 2nd (INCA) interlaboratory exercise
SETOC 00 4	NLS results for the SETOC interlaboratory exercise, distribution 00.4
SETOC 01 4	NLS results for the SETOC interlaboratory exercise, distribution 01.4
SETOC 99 4	NLS results for the SETOC interlaboratory exercise, distribution 99.4
SETOC 738	NLS results for SETOC interlaboratory exercise 738
UMEA 2000	NLS results for the UMEA interlaboratory calibration exercise 2000

Table A3 – Compilation of PAH data (held in 2 Organics Data)

Spreadsheet name	Data relating to:
Herbage Recoveries	Two duplicate batches of spiked recoveries on herbage
Soil (973) Spiked Recoveries	Duplicate analysis of spiking recoveries, using SETOC 973 soil as the base sample
LGC River Sediment	NLS results for the LGC interlaboratory calibration exercise
Grass Spiking Recoveries	Data on recoveries for five separate batches of grass
LGC Sewage Sludge	NLS results for the LGC interlaboratory calibration exercise
CRM1941a	NLS data on the analysis of CRM 1941a
SETOC 701	NLS data from five batches of analysis using the sample supplied as part of the SETOC interlaboratory exercise
SETOC 738	NLS data from five batches of analysis using the sample supplied as part of the SETOC interlaboratory exercise
SETOC 741	NLS data from five batches of analysis using the sample supplied as part of the SETOC interlaboratory exercise

Table A4 – Compilation of PCB data (held in 2 Organics Data)

Spreadsheet name	Data relating to:
Blanks	Measured concentrations in 19 batches
CS4	Data for 18 analyses of system suitability standard (includes mean, standard deviation and relative standard deviation)
PCB 13C Recovery	Results of the recoveries of ^{13}C internal standards of one sample chosen at random from each of 12 batches
Precision & Recovery	Precision and recovery data for 11 batches of AQC samples (includes mean, standard deviation and relative standard deviation, as well as mean recovery)
Grass Spike Recovery	Recovery data for replicate (four) analyses over two separate batches (includes mean recovery)
SETOC 738 Spike	Spiking recovery results for four batches using the sediment supplied as part of the SETOC 738 interlaboratory exercise
SETOC 738	NLS data from seven batches of analysis using the sample supplied as part of the SETOC interlaboratory exercise
CRM1941a	NLS data on the analysis of CRM 1941a
Italian Sediment	NLS results for the Italian Sediment 2nd (INCA) interlaboratory exercise
UMEA 2000	NLS results for the UMEA interlaboratory calibration exercise 2000

Metals methods

These methods are contained in the folder named ‘4 Metals Data’ on the CD-ROM.

Within this folder are the full transcripts of each UKAS accredited method employed in the UKSHS. The methods (see Table A5) contain details of:

- principle
- hazards
- performance characteristics
- apparatus, reagents and equipment used for sample analysis.

Details of the quality control procedures are also given, along with the full analytical protocol.

Table A5 – Methods used for metals analysis (held in 4 Metals Data)

File Name	Content
NM_07304 Aqua Regia Digest	Aqua regia digestion for ICPMS, ICPOES and cold vapour AAS
NM_07503 Hydride Analysis Sample Prep	Pretreatment and analysis by hydride generation
NM_04202 Elan 5000 ICPMS Operating Instructions	Set-up and maintenance for the Elan 5000 instructions
NM_07704 Elan ICPMS Calibration	Calibration standards, reagents and instrumental procedure for the Elan 5000
NM_07403 Optima ICPOES Operating Instructions & Calibration	Set-up and calibration for the Optima instructions and calibration 3000/3300 ICPOES
NM_02302 Mercury Calibration	Preparation of mercury calibration standards and reagents for aqua regia digests using the Perkin Elmer 4100 and FIAS 200
NM_07002 Hydride_FIAS Operating Instructions	Operating Instructions for Perkin Elmer flow instructions injection mercury/hydride system (FIAS) using ‘AA Winlab’ software
NM_08001 Elan 6000 ICPMS Scan	Procedure for performing ‘Total Quant Analysis’ (semi-quantitative element scan) using the Elan 6000 ICPMS

Metals test method performance data

Six Microsoft® Word files are held on the CD-ROM under the folder named ‘4 Metals Data’ . These files contain details of the test method performance data (see Table A6).

Table A6 – Test method performance data files (held in 4 Metals Data)

File Name	Content
CRM 141R	CRM 141R trace elements in calcareous loam soil
CRM281	CRM 281 trace elements in rye grass
CMI 7003	CMI 7003 trace elements in silty clay loam
LGC 6135	LGC 6135 trace elements in Hackney brickworks soil
SPIKING RECOVERY HERBAGE	Recovery data using CRM281 as unspiked sample
SPIKING RECOVERY SOIL	Recovery data using CRM7003 as unspiked sample

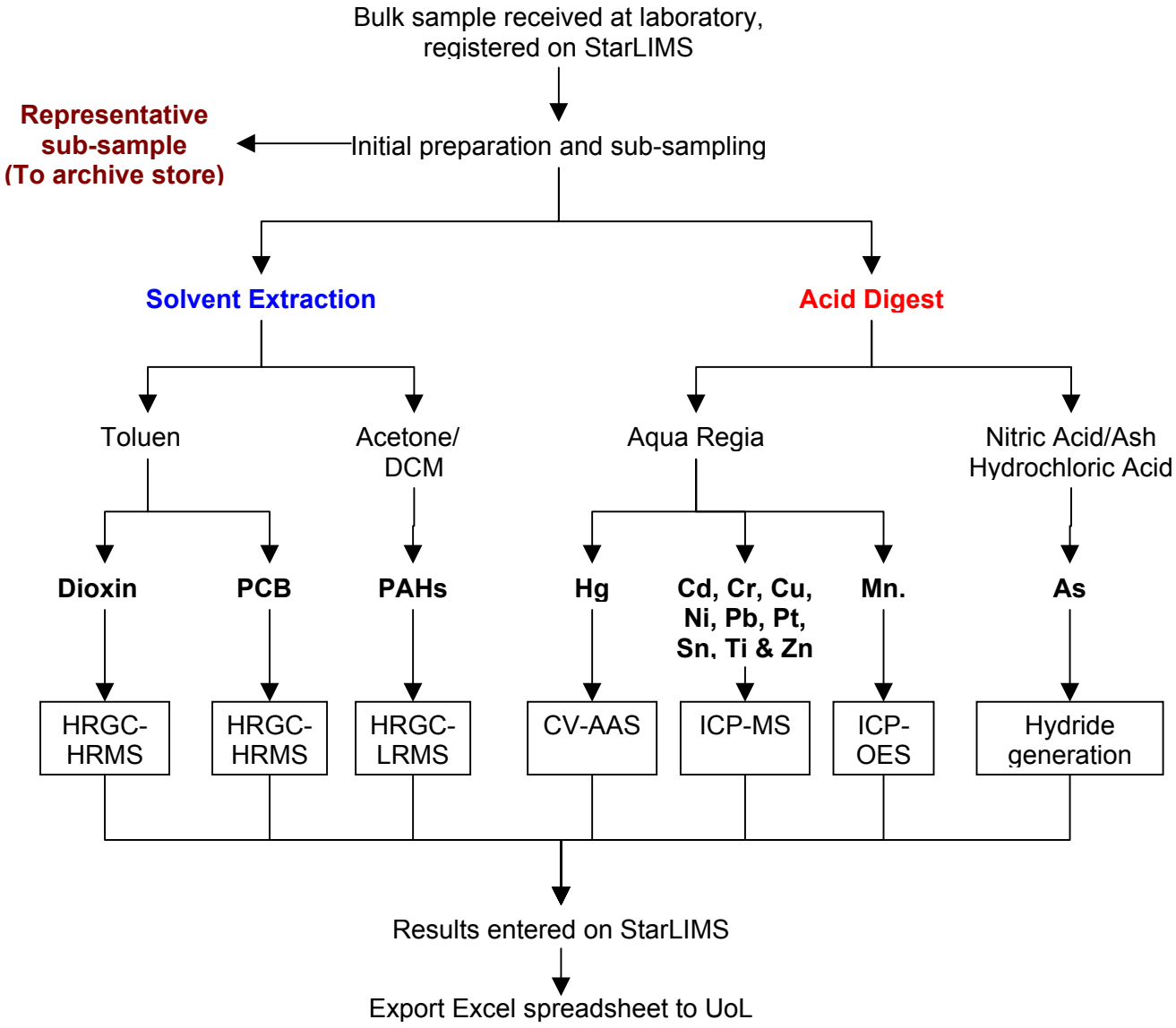
Sample identification codes

The sample identification codes for the UKSHS are held, in full, in the folder named ‘5 Sample Identification Codes’.
An archive of dried samples is held by the NLS at the Leeds Laboratory and can be accessed by application to the Environment Agency's Head of Land Quality.

Laboratory staff

Full details of all NLS personnel involved in handling samples and the analysis for the UKSHS are given on the CD-ROM in the folder named ‘6 NLS Personnel’.

Appendix 2: Chemical analysis flow chart



Appendix 3: Peer review – Central Science Laboratory

CENTRAL SCIENCE
LABORATORY

UK Soil and Herbage Pollutant Survey: Review of Analytical Method Documents for Dioxins, PCBs and PAHs

At the request of the Environment Agency project manager for the above survey a review of the analytical method documents that will be used to effect measurements of soil and herbage samples taken for the survey, was undertaken.

The following documents produced by the Environment Agency's laboratory at Leeds have been reviewed:

1. Method Manual HRMS 3 "Analysis of tetra through octa chlorinated dioxins and furans and polychlorinatedbiphenyls in sediment, soil and herbage samples by HR GC-MS"
   (Latest issue: July 2002),

2. Method Manual HRMS 2 "Analysis of polycyclic aromatic hydrocarbons (PAHs) and organochlorine pesticides (OCPs) in sediment, soil and herbage samples by GC-MS"
   (Latest issue: July 2002),

   and providing supporting data for the above,

3. HR GC-MS section, quality data summary (Issue as per 1),

4. PCB/PAH drying tests.

The Method Manuals described above are currently UKAS accredited documents.

Discussion meetings of the reviewer's comments on previous issues of the above documents were held at the Central Science Laboratory on 31 July and 6 November 2001 where detailed clarification of comments and recommendations were provided. Present were the Manager of the Survey – Dr Peter Crook, the senior analyst from the Leeds laboratory – Mic Daniel, the project consultant – Prof. Colin Creaser (not present at the 6 Nov meeting) and the peer reviewer – Dr Alwyn Fernandes. Further reviews of later issues of the method manuals were also carried out and specific comments and recommendations for method optimisation were made in consultation with the parties listed above.

In general, the method documents were found to be well focused and provided a good level of detail on the methodology used, as well as supporting data on the quality control tests that had been carried out. A number of technical issues arising from the descriptions given in the documents were clarified. Textual and procedural amendments have been made to the original documents and these have been validated where appropriate.

The latest issues of the method manuals listed above are therefore the result of a number of reviews and revisions following consultation and discussions between

Sand Hutton • York
YO41 1LZ • U.K.
Tel: +44 (0)1904 462000
Fax: +44 (0)1904 462111
Website: www.csl.gov.uk
E-mail: science@csl.gov.uk

DEFRA
Department for Environment, Food & Rural Affairs

8

the parties listed above. Whilst accredited data on dioxin/PCB and PAH levels in soil and herbage may be produced using the methodology that has resulted from this review the following points should be taken into consideration:

- Limits of acceptable measurement tolerance for all analytes are currently derived from the replicate analysis of reference materials. These limits should be continuously reviewed with reference to certificated data and contracted in line with technology and methodology improvements. The choice of reference material should also be reviewed as newer and more appropriate products become available.

- Estimates of measurement uncertainty on an ongoing basis should be made in accordance with the Eurachem guide and used in the interpretation of survey results.

- It is recommended that the positive identification and quantification criteria that are used for dioxins/PCBs and PAHs, conform to or exceed those given in the following documents:
  1. P Ambidge, E Cox, C Creaser, M Greenberg, M Gem, J Gilbert, P Jones, M Kibblewhite, L Levy, S Lisseter, T Meredith, L Smith, P Smith, J Startin*, I Stenhouse, and M Whitworth. Acceptance criteria for analytical data on polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzofurans. Chemosphere (1990) **21** (8) 999-1006.
  2. Determination of Tetra- through Octa-chlorinated dioxins and furans in waters and waste waters – Isotope dilution HRGC/HRMS method. Draft CEN method: ISO TC 147/SC2 [2001-08-06] (ISO/DIS/18073).

- It is recommended that the laboratory adopt a policy of continuous optimisation of the methods, to incorporate improvements in technology and improvements in analytical measurement methodology as they occur, in order to produce the most robust data for the survey.

- It is strongly recommended that participation in proficiency testing schemes and inter-laboratory trials for dioxins, PCBs and PAHs continue for the duration of the survey.

Due consideration of the above points as well as those discussed during the review period should result in data that is consistent with the requirements of the survey.

Dr Alwyn Fernandes
Food Safety and Quality
Central Science Laboratory

1st August 2002

T: ../OEC.../Public.../Environment Agency

Appendix 4: Peer review – Cranfield University

National Soil Resources Institute

Cranfield UNIVERSITY
Silsoe

Cranfield University
Silsoe
Bedfordshire MK45 4DT
England
Direct Tel. (with voicemail) +44 (0) 1525 863246
Switchboard: +44 (0) 1525 863000
Fax +44 (0) 1525 863253
Email: p.loveland@cranfield.ac.uk

Dr P. Crook,
The Environment Agency,
Block 1, Government Buildings,
Burghill Road,
Westbury-on-Trym,
Bristol BS10 6BF

November 1st 2001

Dear Dr Crook,

UK Soil and herbage pollutant survey (your ref.: letter of 30 October 2001; LQ/04/61/007)

Thank you for your letter and the enclosed documents relating to the inorganic chemical analytical protocols. I have examined these documents. The additional comments seem perfectly satisfactory explanations of the points raised earlier. I can confirm, therefore, that these protocols have been peer reviewed and approved.

I am returning the protocols to you with this letter.

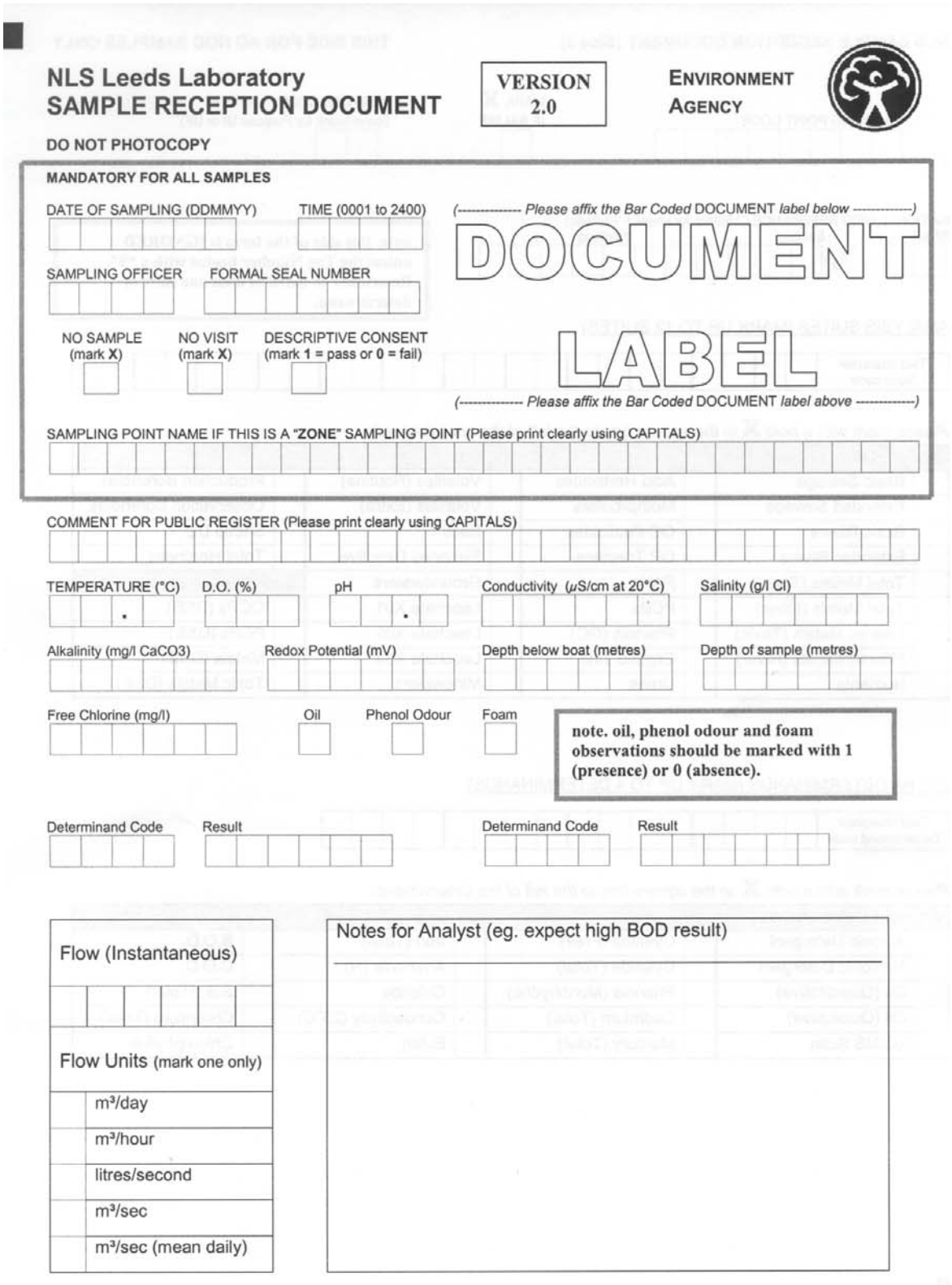
I can confirm that no originals have been retained, nor copies made of these or any other documents sent to us in the course of this review.

Yours sincerely

P. Loveland.

Peter J. Loveland, Ph.D.
Professor of Applied Soil Chemistry

Appendix 5: Example OCR form



NLS Leeds Laboratory
SAMPLE RECEPTION DOCUMENT

VERSION 2.0

ENVIRONMENT AGENCY

DO NOT PHOTOCOPY

MANDATORY FOR ALL SAMPLES

DATE OF SAMPLING (DDMMYY) | TIME (0001 to 2400)

(---------- Please affix the Bar Coded DOCUMENT *label below ----------)*

DOCUMENT

SAMPLING OFFICER | FORMAL SEAL NUMBER

NO SAMPLE (mark **X**) | NO VISIT (mark **X**) | DESCRIPTIVE CONSENT (mark **1** = pass or **0** = fail)

LABEL

(---------- Please affix the Bar Coded DOCUMENT *label above ----------)*

SAMPLING POINT NAME IF THIS IS A "**ZONE**" SAMPLING POINT (Please print clearly using CAPITALS)

COMMENT FOR PUBLIC REGISTER (Please print clearly using CAPITALS)

TEMPERATURE (°C) | D.O. (%) | pH | Conductivity (µS/cm at 20°C) | Salinity (g/l Cl)

Alkalinity (mg/l CaCO3) | Redox Potential (mV) | Depth below boat (metres) | Depth of sample (metres)

Free Chlorine (mg/l) | Oil | Phenol Odour | Foam

note. oil, phenol odour and foam observations should be marked with 1 (presence) or 0 (absence).

Determinand Code | Result | Determinand Code | Result

Flow (Instantaneous)

Flow Units (mark one only)

- m³/day
- m³/hour
- litres/second
- m³/sec
- m³/sec (mean daily)

Notes for Analyst (eg. expect high BOD result)

PLEASE COMPLETE THE REVERSE SIDE FOR ALL AD HOC SAMPLES.

NLS SAMPLE RECEPTION DOCUMENT (Side 2) **THIS SIDE FOR AD HOC SAMPLES ONLY**

SAMPLING POINT CODE

MARK **X** IF **SALINE**

PURPOSE CODE (leave blank for Purpose **UI** or **UF**)

NATIONAL GRID REFERENCE (2 letters for sheet + 10 digits)
Sheet Easting Northing

note. this side of the form is IGNORED unless the Tag Number begins with a "9". Remember to mark at least one suite or determinand.

ANALYSIS SUITES (MARK UP TO 12 SUITES)

Two character Suite code

Please mark with a bold **X** *in the square box to the left of the analytical suite.*

Aqueous Suites			
Basic Sewage	Acid Herbicides	Volatiles (Routine)	Production Boreholes
Extended Sewage	Mothproofers	Volatiles (Extra)	Observation Boreholes
Basic Rivers	OC Pesticides	Bacti	Sheep Dip
Extended Rivers	OP Triazines	Fisheries Directive	Total Hardness
Total Metals (Toxic)	PAHs	Groundwaters	Sediment Suites
Total Metals (River)	PCBs	Leachate X01	OCPs (DSB)
Filtered Metals (Toxic)	Phenols (GC)	Leachate X02	PCBs (DSB)
Filtered Metals (River)	Organo Tins	Leachate X03	Metals (DSB)
Nutrients	Urons	Minewaters	Toxic Metals (DSB)

EXTRA DETERMINANDS (MARK UP TO 4 DETERMINANDS)

Four character Determinand code

Please mark with a bold **X** *in the square box to the left of the determinand.*

Aqueous Determinands			
Anionic Detergent	Cyanide (Free)	Iron (Total)	B.O.D.
Nonionic Detergent	Cyanide (Total)	Ammonia (N)	C.O.D.
Oil (Quantitative)	Phenols (Monohydric)	Chloride	S.S. (Total)
Oil (Qualitative)	Cadmium (Total)	Conductivity (25°C)	Chromium (Total)
GCMS Scan	Mercury (Total)	Eulan	Chlorophyll-a

We are The Environment Agency. It's our job to look after your environment and make it **a better place** – for you, and for future generations.

Your environment is the air you breathe, the water you drink and the ground you walk on. Working with business, Government and society as a whole, we are making your environment cleaner and healthier.

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