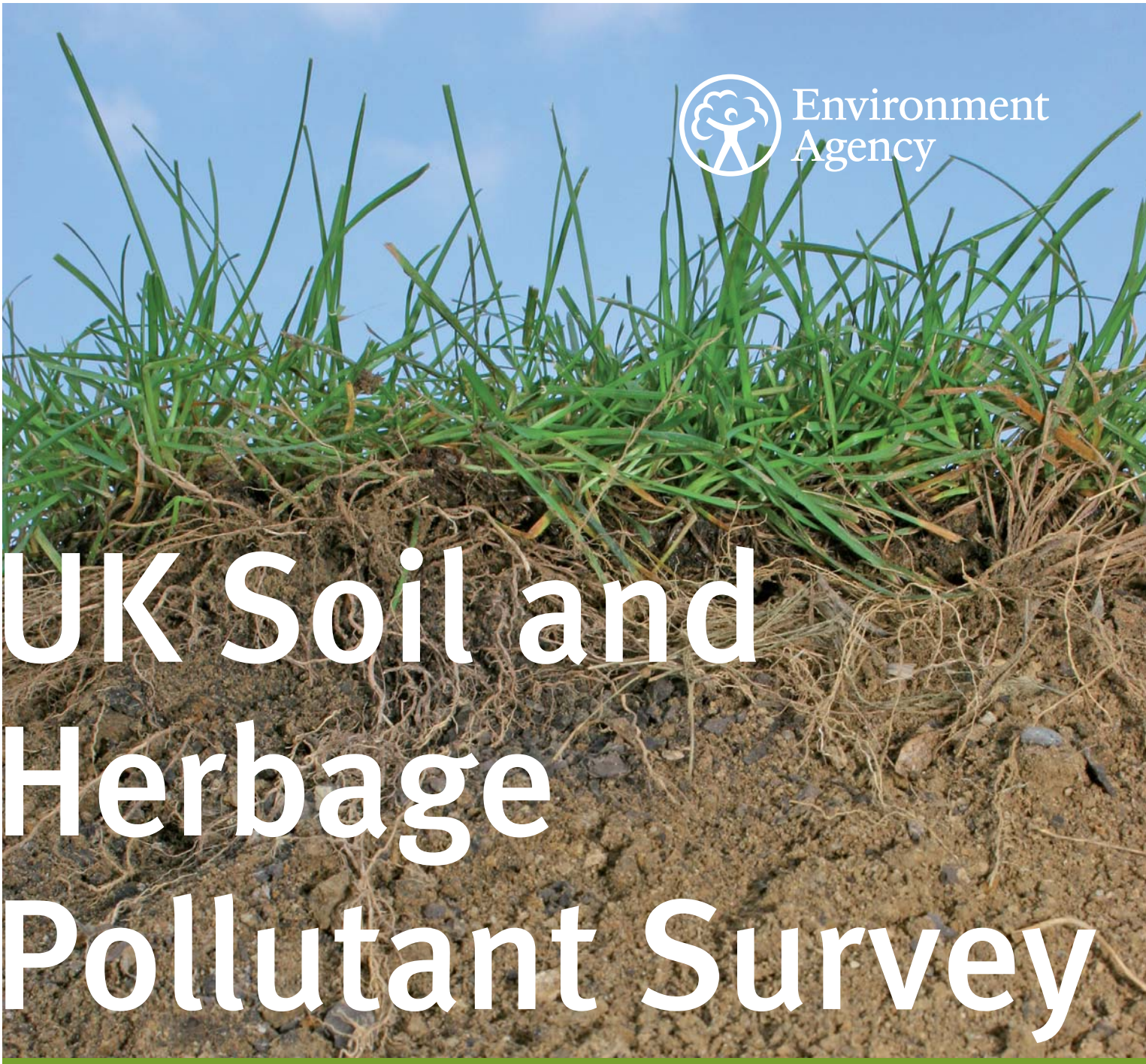

Environment Agency

UK Soil and Herbage Pollutant Survey

UKSHS Report No. 5

Intercomparison exercise

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Steve Killeen
Head of Science

Executive Summary

This report describes an intercomparison exercise conducted within the UK Soil and Herbage Pollutant Survey (UKSHS). Most laboratories now participate regularly in national and international round-robin and inter-laboratory comparison exercises. However, it was felt that this project would benefit from examining how the analytical laboratories involved in the UKSHS – (the Environment Agency's National Laboratory Service (NLS) and the University of Liverpool (UoL) – compared with other UK laboratories that might be involved in this type of exercise. This intercomparison exercise was therefore conducted to demonstrate whether the chemical analytical laboratories and the radio-analytical laboratory involved in this project was performing in a similar manner to other laboratories within the UK.

Each analytical method used within the UKSHS has been accredited by the United Kingdom Accreditation Service (UKAS) to ISO17025. Consequently, NLS and UoL participate regularly in intercomparison exercises run by national and international organisations. However, if the results of the UKSHS are to become a definitive dataset for others to use, it was felt that the project would benefit from demonstrating that the results obtained are similar to those that would be obtained from other UK laboratories.

There were two component parts to the intercomparison exercise:

- Chemical analysis for four contaminant groups – polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), dioxins and metals/metalloid – was conducted on nine samples, although each laboratory did not necessarily analyse all nine samples. The samples covered two sample types likely to be encountered in the UKSHS (soil and herbage). In addition, the UKSHS project was asked to evaluate laboratory performance in the analysis of incinerator ash. The soil samples used were representative of the main land use types covered by the UKSHS (rural, urban and industrial). The ash sample was supplied only to those laboratories conducting dioxin analysis.

- Radiometric measurement of both natural and anthropogenic gamma-emitting radionuclides was conducted on two soil samples and a herbage sample.

Six laboratories participated in the chemical exercise and five in the radiometric exercise.

Staff from the UoL prepared all the samples analysed by the participating laboratories. Samples of certified reference materials (CRMs) were weighed out from stock supplies purchased from approved suppliers while the unknowns were collected in the field, air-dried and homogenised before being sub-sampled.

To demonstrate the samples were homogeneous prior to their release to the participating laboratories, the sample preparation procedure was checked by:

- analysis of four different metals using in-house techniques for the chemical samples;
- counting all radiometric samples on the same high-purity germanium detector.

The data returned were analysed in a systematic manner to determine the percentage deviation (to give a measure of any bias in the measurement). The u-statistic was used to determine whether the results were significantly different.

Following analysis of the data reported by each of the laboratories participating in the laboratory intercomparison trial, the main conclusions were:

- Metal concentrations reported by the NLS were comparable with those reported by other laboratories for a given sample.

- PAH concentrations reported by the NLS were comparable with those reported by other laboratories for a given sample.

- PCB concentrations reported by the NLS were comparable with those reported by other laboratories for a given sample.

- Dioxin concentrations reported by the NLS were comparable with those reported by other laboratories for a given sample. There were significant difficulties, however, with the analysis of the ash samples with none of the participating laboratories performing the analysis of either the CRM or the prepared ash material well.

- Overall, the analytical performance of the NLS was shown to be comparable to, or better than, the analytical performance achieved by the other participating laboratories for both the analytical suite and samples in the intercomparison exercise.

- Radionuclide activity concentrations reported by the UoL were comparable with those reported by other laboratories for a given sample.

Both laboratories involved in the UKSHS were shown to produce results that were comparable with those obtained by other UK laboratories. This supports the findings of international and national intercomparison and round-robin exercise results for the NLS and UoL laboratories. The results of this intercomparison trial provide additional confidence in the data from the UKSHS project.

Contents

Glossary of terms

Industrial	A site dominated by some form of industry.
Rural	All other areas not categorised as industrial, urban, semi-urban or semi-rural. Predominantly agricultural land or undeveloped countryside.
Urban	An area which is ≥90 per cent urbanised/built up. A conurbation may be formed when a large town and city merge. Urban areas include large towns (20–50 km^2 in area) and cities (>50 km^2 in area).
u-statistic	A statistical test that uses the standard uncertainty to provide an estimate of the agreement between two values.

1 Introduction

The UK Soil and Herbage Pollutant Survey (UKSHS) was 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).

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.

The primary aim of the project was to establish a baseline for pollutant levels in soil and herbage in the UK. The study involved the collection of soil and herbage samples for chemical and radiometric analysis from industrial, rural and urban sites throughout the UK. Full details of the sites visited and the number of samples collected are given in UKSHS Report No. 1. The sampling techniques used are detailed in UKSHS Report No. 2.

The scale of the UKSHS has resulted in such a wealth of methodological information and analytical data that presenting the whole study in one report would be unwieldy. The information is therefore presented as a series of 11 standalone reports that users can read individually or as a complete set. This report describes the intercomparison exercise conducted for the UKSHS and is Report No. 5 in the series. Details of the other reports in the series can be found in UKSHS Report No. 1.

Each analytical method used within the UKSHS has been accredited by the United Kingdom Accreditation Service (UKAS) to ISO17025. The analytical laboratories involved in the UKSHS (NLS Leeds, NLS Nottingham and UoL) participate regularly in intercomparison exercises run by national and international organisations. But if the results of the UKSHS are to become a definitive dataset for others to use, it was felt that the project would benefit from a further demonstration of analytical laboratory performance to show that the results obtained were similar to those that would be obtained from other UK laboratories.

An intercomparison exercise was therefore conducted to demonstrate whether the chemical analytical laboratories and the radio-analytical laboratory involved in the UKSHS were performing in a similar manner to other laboratories within the UK. The participating laboratories are listed in Section 1.1.

The two component parts of the exercise involved:

determination of chemical contaminants in soil samples representing the three UKSHS land use types (rural, urban and industrial) and in a herbage sample. The contaminants of interest (see Section 2.2) were:

- polycyclic aromatic hydrocarbons (PAHs)
- polychlorinated biphenyls (PCBs)
- polychlorinated dibenzodioxins and dibenzofurans (dioxins);
- metals/metalloids.

measurement of both natural and anthropogenic gamma-emitting radionuclides in two soil samples and a herbage sample.

Following a specific request to the UKSHS project, the performance of laboratories in determining dioxin/furan concentrations in an incinerator ash sample was also evaluated.

For soil samples and the ash sample, certified reference materials (CRMs) were also supplied to the laboratories for the different determinand groups. A herbage CRM was not included in this exercise, but the NLS did use herbage CRMs throughout the UKSHS project for quality assurance of the results for chemicals from both soil and herbage. Details of the CRMs are given in Section 3.1.

The analysis approach adopted by the UKSHS project is described in UKSHS Report No. 3.

Details of the samples (including the CRMs) used in the intercomparison exercise are given in Section 2.1. Preparation of the CRMs and unknown samples for the intercomparison exercise is described in Section 3.

1.1 Participants in the intercomparison exercise

A selection of laboratories accredited to UKAS for conducting analysis of soil and herbage for the determinands present in the UKSHS project were contacted. They were asked if they would like to participate and, if so, to provide information on the determinands they could report on.

These laboratories were then evaluated and those that could cover the majority of the determinands were invited to participate in the intercomparison exercise. Other issues that were considered included the cost of analysis, the turnaround times and the similarities in the methods for sample analysis.

Table 2.1 lists the 11 laboratories invited to participate. Six laboratories were involved in the chemical intercomparison exercise – including the NLS, which conducted the chemical analysis of the UKSHS samples. Five laboratories were involved in the radioactivity exercise – including the Universities of Liverpool and Stirling, both of whom are involved in the UKSHS project. Only the NLS and the UoL are distinguished in the presentation of the results in Section 5; the other laboratories are anonymous.

Apart from the University of Stirling, all participating laboratories were UKAS-accredited for the majority, if not all, of the determinands.

Table 1.1 Contact names and details for the participating laboratories

Chemical intercomparison exercise	Radioactivity intercomparison exercise
Chris Hunter National Laboratory Service Environment Agency Olympia House Gelderd Lane Gerald Road Leeds LS12 6DD	David Copplestone School of Biological Sciences University of Liverpool Liverpool L69 3GS
Geraint Apps Harwell Scientifics 551 South Becquerel Avenue Harwell International Business Centre Didcot Oxfordshire OX11 0TD	Vicki Ritherdon Harwell Scientifics 551 South Becquerel Avenue Harwell International Business Centre Didcot Oxfordshire OX11 0TD
Karl Pettit Marchwood Scientific Services (MSS) Unit 4G Marchwood Industrial Park Marchwood Southampton Hampshire SO40 4PB	Greg Willets NNC Ltd Engineering Department Centre Birchwood Park Birchwood Warrington WA3 6BZ
David Wood Scientific Analysis Laboratories Ltd (SAL) Medlock House New Elm Road Manchester M3 4JW	Ian Maidment Geoffrey Schofield Laboratories, BNFL Westlakes Science and Technology Park Moor Row Cumbria CA24 3JZ
Alwyn Fernandes Central Science Laboratory Sand Hutton York YO41 1LZ	Andrew Tyler Department of Environmental Science University of Stirling Stirling FK9 4LA
Andrew Jervis Direct Laboratories Woodthorne Wergs Road Wolverhampton WV6 8TQ	

2 Sample types and determinands

2.1 Types

2.1.1 Chemical intercomparison exercise

Three different sample types (soil, herbage and ash) were provided to the laboratories participating in the chemical intercomparison exercise (Table 2.1). A number of soil samples were provided to cover the range of typical environmental concentrations anticipated within the UKSHS, making a total of nine samples available for the chemical intercomparison exercise:

Table 2.1 Sample types for the chemical intercomparison exercise

Number	Description
Soil	
C1	10 g of soil sample of known heavy metal composition (to be analysed by all participating laboratories carrying out metal analysis on C4 samples and/or C5).
C2	10 g of soil sample of known PCBs and PAHs composition (to be analysed by all participating laboratories carrying out PCB/PAHs analysis on C4 samples and/or C5)
C3	10 g of soil sample of known dioxin composition (to be analysed by all participating laboratories carrying out dioxin analysis on C4 samples and/or C5).
C4	30 g prepared soil sample. Three samples of unknown chemical composition representing typical levels of contamination observed in the environment (Unknown Soil 1, Unknown Soil 2, Unknown Soil 3).
Herbage	
C5	10 g of prepared herbage sample of unknown chemical composition (Herbage)
Ash	
C6	100 g of incinerator bottom ash of unknown chemical composition
C7	2 g of ash sample of known dioxin composition (to be analysed by all participating laboratories carrying out dioxin analysis on C6) (Ash)

2.1.2 Radioactivity intercomparison exercise

Two different sample types (soil and herbage) were provided to the laboratories participating in the radiometric intercomparison exercise. Table 2.2 lists the samples available for this part of the exercise.

Table 2.2 Sample types for the radio-analytical intercomparison exercise

Number	Description
Soil	
R1	250 g of soil sample of known radionuclide composition (to be analysed by all participants)
R2	500 g of prepared soil sample of unknown radionuclide composition representing a typical level of contamination observed in the environment (Unknown Soil)
Herbage	
R3	50 g of prepared herbage sample of unknown radionuclide composition representing a typical level of contamination observed in the environment (Herbage)

2.2 Determinands

The samples were a mix of CRMs and unknowns. Each laboratory was asked to:

- indicate whether it was able to report a value for the determinands listed in Tables 2.3–2.5;
- provide details of its UKAS accreditation.

All the determinands listed in Tables 2.3–2.5 are commonly found in environmental samples and are either naturally occurring or anthropogenic in origin. The laboratories were not told which CRMs were being used. They were given three months to return their analytical data.

Table 2.3 Organic determinands included in the intercomparison exercise

Dioxins and furans	PAHs	PCBs[1]
2,3,7,8-tetrachlorodibenzo-*p*-dioxin*	Acenaphthene	PCB 18*
1,2,3,7,8-pentachlorodibenzo-*p*-dioxin*	Acenaphthylene	PCB 28*
1,2,3,4,7,8-hexachlorodibenzo-*p*-dioxin*	Fluorene	PCB 31*
1,2,3,6,7,8-hexachlorodibenzo-*p*-dioxin*	Phenanthrene*	PCB 47
1,2,3,7,8,9-hexachlorodibenzo-*p*-dioxin	1-Methylphenanthrene	PCB 49*
1,2,3,4,6,7,8-heptachlorodibenzo-*p*-dioxin*	2-Methylphenanthrene	PCB 51
Octachlorodibenzo-*p*-dioxin*	Anthracene*	PCB 52*
2,3,7,8-tetrachlorodibenzofuran*	Fluoranthene*	PCB 77
1,2,3,7,8-pentachlorodibenzofuran*	Pyrene*	PCB 81
2,3,4,7,8-pentachlorodibenzofuran*	Benzo(a)anthracene*	PCB 99*
1,2,3,4,7,8-hexachlorodibenzofuran*	Chrysene*	PCB 101*
1,2,3,6,7,8-hexachlorodibenzofuran*	Benzo(a)pyrene*	PCB 105*
1,2,3,7,8,9-hexachlorodibenzofuran*	Benzo(b)fluoranthene*	PCB 114
2,3,4,6,7,8-hexachlorodibenzofuran*	Benzo(j)fluoranthene*	PCB 118*
1,2,3,4,6,7,8-heptachlorodibenzofuran*	Benzo(k)fluoranthene*	PCB 123
1,2,3,4,7,8,9-heptachlorodibenzofuran	Dibenzo(ac)anthracene*	PCB 126
Octachlorodibenzofuran*	Benzo(ghi)perylene*	PCB 128*
	Indeno(1,2,3-cd)pyrene*	PCB 138*
	Dibenzo(ah)anthracene*	PCB 153*
	Coronene	PCB 156*
		PCB 157
		PCB 167
		PCB 169
		PCB 170*
		PCB 180*
		PCB 189

* Determinands for which a certified value is available within the CRM.
[1] See UKSHS Report No. 8 for full chemical names.

Table 2.4 Inorganic determinands included in the intercomparison exercise

Arsenic (As)	Nickel (Ni)*
Cadmium (Cd)*	Platinum (Pt)
Chromium (Cr)*	Tin (Sn)
Copper (Cu)*	Titanium (Ti)
Lead (Pb)*	Vanadium (V)
Manganese (Mn)*	Zinc (Zn)*
Mercury (Hg)*	

* Determinands for which a certified value is available within the CRM.

Table 2.5 Radionuclide determinands included in the intercomparison exercise

Potassium-40 (^{40}K)*	Lead-214 (^{214}Pb)
Cobalt-60 (^{60}Co)	Radium-224 (^{224}Ra)
Caesium-134 (^{134}Cs)*	Radium-226 (^{226}Ra)*
Caesium-137 (^{137}Cs)*	Actinium-228 (^{228}Ac)
Thallium-208 (^{208}Tl)	Thorium-234 (^{234}Th)
Lead-210 (^{210}Pb)	Protoactinium-234m (^{234m}Pa)
Bismuth-212 (^{212}B)	Uranium-235 (^{235}U)
Lead-212 (^{212}Pb)	Americium-241 (^{241}Am)
Bismuth-214 (^{214}Bi)	

* Determinands for which a certified value is available within the CRM.

3 Sample preparation

3.1 CRMs

The individual CRMs used (Table 3.1) were obtained from a number of suppliers. The organic and inorganic CRMs were purchased on behalf of the exercise by the NLS and provided to the UoL. The radionuclide CRM was purchased by UoL from the International Atomic Energy Agency (IAEA).

Aliquots (10 g) of the chemical exercise CRM materials were weighed out and transferred to hexane-washed amber glass jars by UoL staff. The CRM material for the radiometric exercise was supplied in 250 g aliquots, which were transferred entirely into plastic containers for dispatch to the participating laboratories.

All sample containers were given a unique identifier known only to the co-ordinators of the intercomparison exercise.

Table 3.1 List of CRM materials used and their suppliers

CRM	Notes	Supplier
SETOC 738	Sediment with reference values for dioxins, PCBs and PAHs	Wageningen University, The Netherlands
CRM 490	Dioxins and furans in flyash	Community Bureau of Reference (BCR), purchased from LGC Promochem, UK
SRM 1944	Marine sediment containing certified concentration values for PCBs and PAHs	National Institute of Standards and Technology (NIST), purchased from LGC Promochem, UK
CRM 141R	Trace elements in calcareous loam soil	Community Bureau of Reference (BCR)
IAEA-375	Radionuclides and trace elements in soil	International Atomic Energy Agency

3.2 Unknown samples

3.2.1 Sample collection

The soil samples were collected from:

- an industrial location on Merseyside;
- an urban park in Liverpool;
- a rural site being visited by the field team as part of the main sampling programme.

Approximately 1 kg of soil was collected for the chemical analyses and 5 kg for the radiometric analyses.

The soil samples were collected by clearing an area of herbage and decaying plant litter, and then using a spade to excavate the soil to a depth of 5 cm (15 cm for the radiometric sample) until sufficient material was collected.

Herbage samples were collected using a pair of shears to cut an area of non-woody vegetation as described in UKSHS Report No. 2.

3.2.2 Chemical sample processing

The chemical samples were air-dried in aluminium foil trays (soil) or paper bags (herbage) within a fume hood at the UoL. The samples were dried to constant weight at <20^{o}C to avoid loss of highly volatile determinands. For the purposes of this project, constant weight is defined as the weight of the material ±0.1 per cent from the previous measurement.

The soil was then ground with a pestle and mortar, while the herbage was ground in small aliquots using a blender. The blender was operated in short bursts to avoid overheating of the sample, which may lead to loss of the determinands of interest. The material was ground until it passed through a 1 mm sieve.

Each processed sample was transferred into a clean hexane-washed demi-john size glass container and placed securely on an electric-powered mixer. The mixer was operated for a period of 24 hours.

Each 'rolled' sample was then passed through a riffle box to separate the material into 15–25 g (herbage) and 80–100 g (soil) aliquots, which were transferred into hexane-washed amber glass jars with lids lined with aluminium foil.

The samples were then labelled and made ready for transfer to the participating laboratories.

Between samples, the glass container was rinsed with distilled water before being washed with acetone and hexane. The riffle box was cleaned using compressed air and brushes to remove surface material.

To reduce the risk of cross-contamination, the herbage was processed first followed by rural, urban and industrial soils.

3.2.3 Radiometric sample processing

The radiometric samples were oven-dried in aluminium foil trays (soil) or paper bags (herbage) at <85°C. The samples were then ground with a rotary mill or blender (soil and herbage respectively) until the sample passed through a 1 mm sieve.

Each processed sample was then transferred to a large plastic drum, which was placed securely on an electric mixer. The mixer was operated for a period of 24 hours.

Each 'rolled' sample was then passed through a riffle box to separate the material into 80–110 g (herbage) and 450–550 g (soil) aliquots, which were transferred into 500 ml plastic containers.

The samples were then labelled and made ready for transfer to the participating laboratories.

The plastic drum was washed out with detergent before being rinsed with distilled water and the riffle box was cleaned using compressed air and brushes to remove surface material between samples.

3.2.4 Ash sample

A sample of bottom ash from an incinerator was supplied to UoL by staff from the Environment Agency. The nature of this type of sample makes sample processing and homogenisation difficult because it contains materials such as grit, metal, glass and rags (see Plate 3.1. After consultation, the following procedure was adopted.

Figure 3.1 Photograph of the bottom ash sample as received by University of Liverpool

The material was transferred to aluminium foil trays (Plate 3.2) and the wet weight recorded. The sample was then left to air-dry to constant dry weight. Drying the ash material took 12 days. The final dry weight was measured and recorded.

Figure 3.2 Photograph of the dried bottom ash sample

The samples were then examined visually and any obvious metal pieces were removed; the weight of the removed fraction was recorded. The material was then passed through a heavy-duty grinder operated by the NLS Waste Management section in Nottingham. The material was ground in this mill until it passed through a 1 mm sieve. The ground material was then returned to UoL for rolling as outlined in Section 3.2.2.

The rolled material was divided into eight sub-samples for release to the participating laboratories. Four laboratories participated in the analysis of the incinerator ash sample.

3.2.5 In-house testing of the sample preparation procedure

To assess the effectiveness of the preparation procedure in homogenising samples, a series of in-house tests were conducted by UoL staff.

Table 3.2 shows the concentrations for four metals (copper, lead, nickel and zinc) which were analysed in the herbage and three soil samples used in the chemical exercise. The results indicate that the coefficient of variation for the samples ranged from 2.3 to 8.17 (excluding the Pb results for the herbage) for the eight samples. Through careful selection of the five samples for release to the participating laboratories, it was possible to reduce the coefficient of variation to a range of 1.79 to 6.43. The five samples selected for sending out are marked in Table 3.2.

These results were determined using a different analytical technique (flame atomic absorption spectrometry) to that employed by the NLS (inductively coupled plasma mass spectrometry). Therefore, differences between the results listed in Table 3.2 and those for the NLS in the metals section of this report are likely to be related to differences in the analytical approach.

3.2.6 Sample analysis

Participating laboratories were asked to analyse the samples as received using their own analytical methods. The brief description of these methods provided by the laboratories as part of the reporting procedure are summarised in Section 5.1.

The only exceptions to this were that participants were advised to:

- report the aqua regia extractable metals for the inorganics analysis;
- treat the ash sample with dilute hydrochloric acid (HCl) (0.01M) for four hours followed by accelerated solvent extraction (ASE) with toluene and then treat as a 'normal' soil/sediment sample for dioxin analysis;
- seal the radiometric samples for three weeks prior to counting.

The participants in the radioactivity intercomparison exercise were asked to provide decay-corrected results to 1 January 2003.

The participants were asked to report any positive results or limit of detection (LOD) values for the determinands listed in Tables 2.3–2.5.

Table 3.2 Results of the in-house analysis for four metals (Cu, Pb, Ni and Zn) from the prepared soil and herbage samples (all results expressed in mg/kg dry weight)

Unknown Soil 1	Cu	Ni	Pb	Zn
Replicate 1	40.9	11.5	123.0	76.0
Replicate 2*	37.9	10.5	121.0	73.0
Replicate 3*	39.3	10.5	119.0	75.0
Replicate 4	33.4	8.5	101.0	66.0
Replicate 5**	37.6	10.5	114.0	72.0
Replicate 6*	38.2	10.5	116.0	75.0
Replicate 7*	37.7	10.0	115.0	75.0
Replicate 8*	40.3	10.5	117.0	75.0
Arithmetic mean	38.2	10.3	115.8	73.4
Standard deviation	2.3	0.8	6.7	3.2
Coefficient of variation	6.0	8.2	5.8	4.4
Unknown Soil 2				
Replicate 1*	1755.0	29.0	3900.0	4600.0
Replicate 2*	1740.0	30.5	3400.0	4500.0
Replicate 3	2090.0	29.5	3250.0	4700.0
Replicate 4	1660.0	27.0	3400.0	4100.0
Replicate 5*	1775.0	29.5	3450.0	4600.0
Replicate 6*	1745.0	28.0	3300.0	4400.0
Replicate 7**	1655.0	28.5	3300.0	4200.0
Replicate 8*	1705.0	27.5	3450.0	4300.0
Arithmetic mean	1765.6	28.7	3431.3	4425.0
Standard deviation	138.1	1.2	203.4	212.1
Coefficient of variation	7.8	4.1	5.9	4.8
Unknown Soil 3				
Replicate 1	12.5	14.0	23.0	61.0
Replicate 2*	11.5	12.5	21.0	57.0
Replicate 3*	11.4	12.5	22.0	55.0
Replicate 4	12.8	13.5	24.0	59.0
Replicate 5**	12.8	13.0	23.0	59.0
Replicate 6*	12.0	12.5	22.0	59.0
Replicate 7*	11.6	13.0	21.0	57.0
Replicate 8*	11.9	12.5	24.0	57.0
Arithmetic mean	12.1	12.9	22.5	58.0
Standard deviation	0.6	0.6	1.2	1.9
Coefficient of variation	4.7	4.4	5.3	3.2
Herbage				
Replicate 1	12.2	5.5	<1	56.0
Replicate 2	13.7	5.5	1.0	57.0
Replicate 3**	13.4	6.0	4.0	58.0
Replicate 4*	12.4	6.5	4.0	58.0
Replicate 5*	11.6	6.0	2.0	56.0
Replicate 6*	11.8	6.0	5.0	57.0
Replicate 7*	12.8	6.0	2.0	54.0
Replicate 8*	11.4	6.0	1.0	57.0
Arithmetic mean	12.4	5.9	2.7	56.6
Standard deviation	0.8	0.3	1.6	1.3
Coefficient of variation	6.7	5.4	59.1	2.3

* Replicate sample used in the intercomparison exercise.

** Held back as a spare.

4 Treatment of data

Data reporting forms were prepared and distributed with the samples. Laboratories were asked to report all chemical concentrations in μg/kg and all radionuclide activity concentrations in Bq/kg. The data returned were entered into a Microsoft ® Excel spreadsheet and checked for consistency. These data were then returned to the participating laboratories for checking (particularly of units reported). The data were then handled in one of two ways depending upon whether the sample was a CRM (Section 4.1) or an unknown (Section 4.2).

4.1 CRM data

The data returned were used in conjunction with the certified reference values to calculate two parameters.

First, the deviation from the reference value was calculated as a percentage:

$$\text{Deviation (\%)} = \frac{(\text{Analyst's result - Certified reference value}) \times 100}{\text{Reference value}} \quad (1)$$

Secondly, the value of the u-statistic (Brookes *et al*. 1979) was calculated:

$$\text{u - statistic} = \frac{(\text{Analyst's result - Certified reference value})}{\sqrt{(\text{Analyst's uncertainty}^2 + \text{Uncertainty on certified reference value}^2)}} \quad (2)$$

The u-statistic provides an indication of agreement between the two values. The uncertainty to be used is the standard uncertainty (where the coverage factor, k = 1). This statistic is compared with values in t-statistic tables (Murdoch and Barnes 1976) and can be interpreted as set out in Table 4.1.

Table 4.1 Criteria for u-tests

Value	**Conclusion**	Category
u < 1.64	The values do not differ significantly.	a
1.64 < u < 1.96	The values probably do not differ significantly, but more data are required to confirm this.	b
1.96 < u < 2.58	It is not possible to say whether there is a significant difference without further data.	c
2.58 < u < 3.29	The values probably differ significantly, but more data are required to confirm this.	d
3.29 < u	The values differ significantly.	e

Where data were reported for determinands present in the CRM samples that do not have a certified reference value, these were treated as unknowns (Section 4.2).

4.2 Data from unknowns

The data returned were used to calculate the mean, median and standard deviation of the results for each determinand in each sample.

Where a participating laboratory did not return a result for a particular determinand, this is listed as NR (not reported) in this report. Additionally, some laboratories reported LOD values for particular determinands.

In both these cases, the results were not included in the calculation of the mean, median and standard deviation. This sometimes led to a situation where less than three results were available for determining the mean, median and standard deviation. Consequently, the mean, median and standard deviation were not determined and no further evaluation of the data was undertaken for this particular determinand/sample.

Where sufficient data existed for the determination of the mean, median and standard deviation, an outlier test was performed using the method outlined in the other UKSHS Reports (No. 8, 9 and 10). The screening limits to identify outliers were set at the median ±2.5 standard deviations. If an outlier was identified, the data point was excluded and, if sufficient results remained, the mean, median and standard deviation were re-calculated. But in the end, no outliers were identified within the data returned using this approach.

The resulting mean and standard deviation for each determinand were used as the reference value. The percentage deviation and u-statistic were calculated for the dataset using equations (1) and (2).

5 Results

5.1 Summary of methods used by participating laboratories

All the participating laboratories analysed the samples as received and followed the requested procedure for both analysis (Section 3.2.6) and reporting. The analytical methods employed by the participating laboratories are outlined below.

5.1.1 Methods for PCBs, dioxins and furans

Samples were spiked with known amounts of ^{13}C-labelled isotopes of the compounds of interest and then extracted using organic solvents such as toluene. Laboratories used different extraction techniques (e.g. a Dionex accelerated solvent extraction (ASE™) system); others used a Soxhlet approach. The extract was cleaned up using adsorption chromatography to remove fats, sulphur and other interfering compounds. In most cases, the sample was then fractionated to separate the dioxins and furans from the PCBs. Each fraction was then concentrated to a volume suitable for injection into a capillary gas chromatograph (GC).

Dioxins and non-ortho PCBs were analysed by high-resolution gas chromatography mass spectrometry (HR GC-MS). Ortho-PCBs were determined using high-resolution gas chromatography coupled with low-resolution mass spectrometry (HRGC-LRMS).

5.1.2 Methods for PAHs

Samples were spiked with known amounts of ^{13}C-labelled isotopes of the compounds of interest (added to the sample before extraction) for use later in quantifying the sample. Samples were extracted using solvents such as acetone and dichloromethane. As with the PCBs and dioxins, laboratories used different extraction systems such as the Dionex ASE™ system or a sonic extraction technique. The extract was then cleaned up to remove fats, sulphur and other interfering compounds. This was often achieved using a Fluorisil solid phase extraction (SPE) clean-up column and gel permeation chromatography (GPC) system, where contaminants were removed on a size-exclusion basis.

Analysis was typically conducted using HRGC-LRMS.

5.1.3 Methods for metals/metalloid

As requested, all the participating laboratories used an aqua regia digestion of each sample. The extract was then made up to volume and analysed using a range of techniques including inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma optical emission spectrometry (ICP-OES). Some metals (e.g. Cd and Hg) were analysed using graphite furnace atomic absorption spectrometry (GFAAS) (for Cd) or a Leco-analyser (for Hg). One laboratory prepared the As digest by ashing with magnesium nitrate and then taking up the sample in HCl. The As concentration was then determined using atomic fluorescence.

5.1.4 Methods for radionuclides

Samples were transferred to the in-house counting geometry (frequently a Marinelli beaker or a 100–200 ml pot) and then counted on high-purity germanium detectors. Most laboratories used analytical software such as GammaVision-32, Canberra Genie or Fitzpeaks for photopeak identification and quantification, although a couple of laboratories supplemented these with in-house software routines.

All laboratories used calibration standards traceable to national or international standards for determining efficiency calibration curves for use in quantification. All laboratories also applied peak background corrections using GammaVision-32, Fitzpeaks or in-house software routines.

A number of laboratories commented on the problems associated with the determination of radionuclides from the U/Th natural series by gamma spectrometry, indicating that they would normally have instead employed radiochemical separation techniques followed by alpha spectrometry because of the inherent difficulties in measurement via gamma spectrometry.

5.2 Presentation of data

Section 5.3 contains the results for the metals/metalloid, PAHs, PCBs, dioxins and radionuclides respectively.

The results for each sample type are given first in terms of the performance of the laboratories for the CRM and then the unknowns. The figures present the percentage deviation of the result compared with:

- the CRM value;
- the mean calculated from the data returned for the unknowns.

All the figures are plotted on the same scale for ease of comparison though, as a consequence, the percentage deviation is occasionally greater than the scale selected. In these cases, the value is in excess of 250 per cent and the actual value is reported on, or by, the corresponding bar on the figure.

The table of data then presents the numeric value of the result from each participating laboratory and the calculated mean or CRM value. Limit of detection values are also reported, as are the code used to describe the u-statistic (see Table 4.1) and the u-statistic value.

Analytical difficulties were encountered with certain determinands. These are listed in Table 5.1, along with an indication of how the problem was solved.

Table 5.1 Problem determinands

Determinand	Problem	Solution
Dibenzo(ac)anthracene	Co-elutes with dibenzo(ah)anthracene	Data reported together
Dibenzo(ah)anthracene	Co-elutes with dibenzo(ac)anthracene	Data reported together
Benzo(b)fluoranthene	Co-elutes with benzo(j)fluoranthene	Data reported together
Benzo(j)fluoranthene	Co-elutes with benzo(b)fluoranthene	Data reported together

5.3 Metals/metalloid results

Results for metals/metalloid are presented in Figures 5.1–5.10 and Tables 5.2–5.6.

The four laboratories reporting data are indicated by NLS, A, B and C.

5.3.1 Analysis of CRM of known metal composition

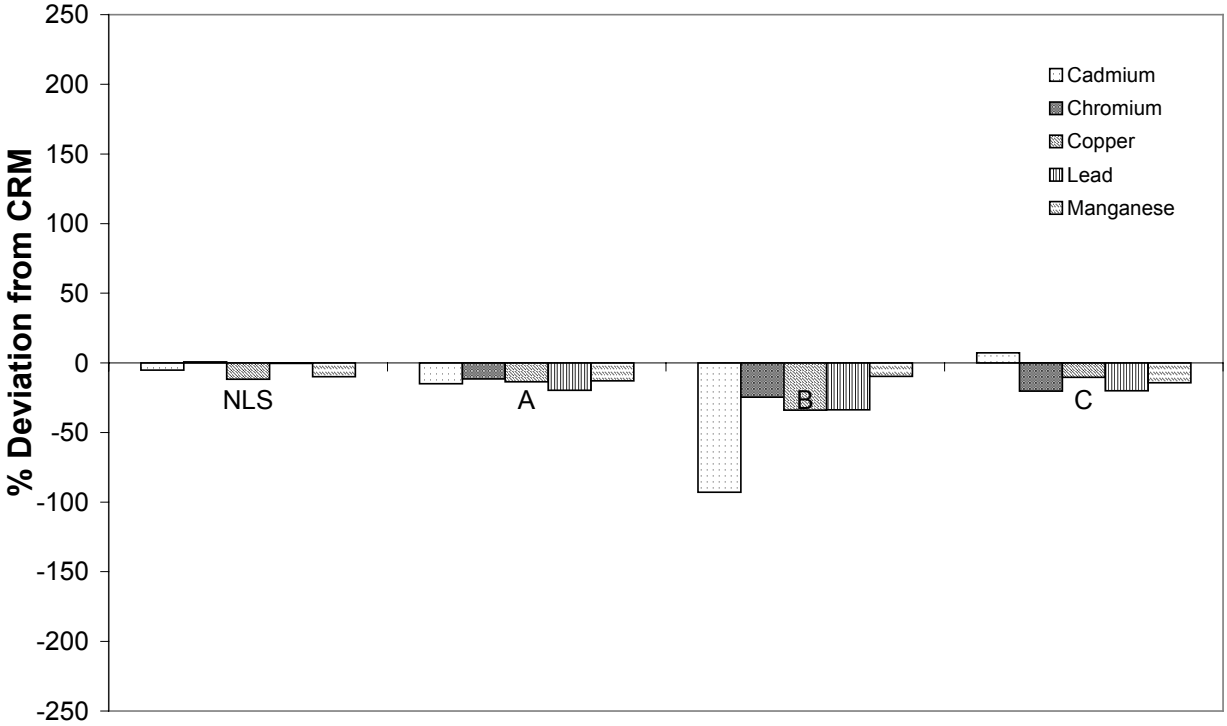

Figure 5.1 Comparison of laboratory performance for analysis of metals CRM (Cd, Cr, Cu, Mn and Pb)

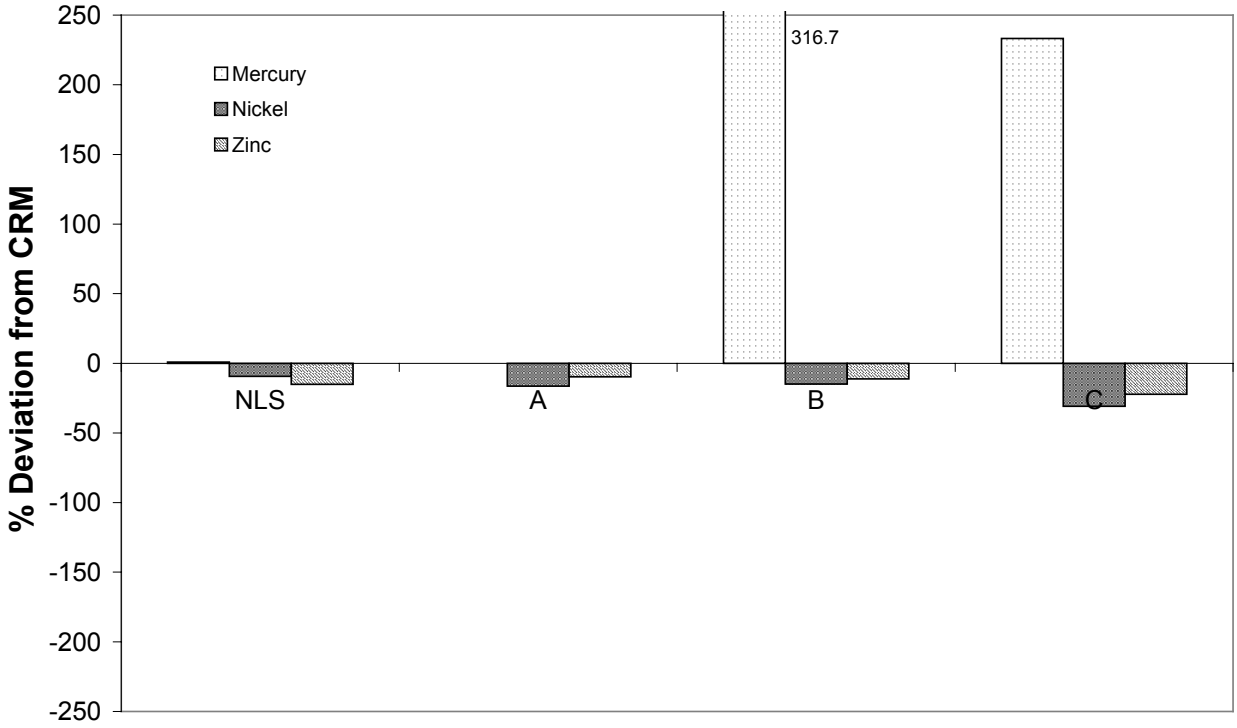

Figure 5.2 Comparison of laboratory performance for analysis of metals CRM (Hg, Ni and Zn)

Table 5.2 Analysis of metals/metalloid CRM and statistical assessment of laboratory performance

		NLS			A			B			C		
Determinand	CRM	Results $mgkg^{-1}$	u-statistic	u-statistic category	Results $mgkg^{-1}$	u-statistic	u-statistic category	Results $mgkg^{-1}$	u-statistic	u-statistic category	Results $mgkg^{-1}$	u-statistic	u-statistic category
Arsenic	*	6.15	*	*	5.83	*	*	< 1.00	*	*	< 0.500	*	*
Cadmium#	14.0	13.3	-1.83	b	11.9	-5.25	e	< 1.00	-32.5	e	15.0	2.50	c
Chromium#	138	139	0.200	a	122	-3.20	d	104	-6.80	e	110	-5.60	e
Copper#	46.9	41.4	-3.06	d	40.5	-3.56	e	31.0	-8.83	e	42.0	-2.72	d
Lead#	51.3	51.1	-0.1000	a	41.2	-5.05	e	34.0	-8.65	e	41.0	-5.15	e
Manganese#	653	588	-4.06	e	569	-5.25	e	590	-3.94	e	560	-5.81	e
Mercury#	0.240	0.242	0.0667	a	0.240		a	< 1.00	25.3	e	0.800	18.7	e
Nickel#	94.0	85.2	-1.76	b	78.7	-3.06	d	80.0	-2.80	d	65.0	-5.80	e
Platinum	*	< 0.0200	*	*	NR			NR			< 2.00	*	*
Tin	13.5	5.66	-0.424	a	4.40	-0.493	a	2.80	-0.580	a	41.0	1.50	a
Titanium	175	202	0.310	a	232	0.657	a	47.0	-1.48	a	220	0.518	a
Vanadium	41.2	48.2	0.615	a	41.6	0.0351	a	25.0	-1.42	a	50.0	0.773	a
Zinc#	270	229	-5.13	e	244	-3.25	d	240	-3.75	e	210	-7.50	e

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.
# A CRM value is available for this determinand.
Non # values are calculated using the approach described in Section 4.2.

5.3.2 Analysis of Unknown Soil 1 for metals/metalloid

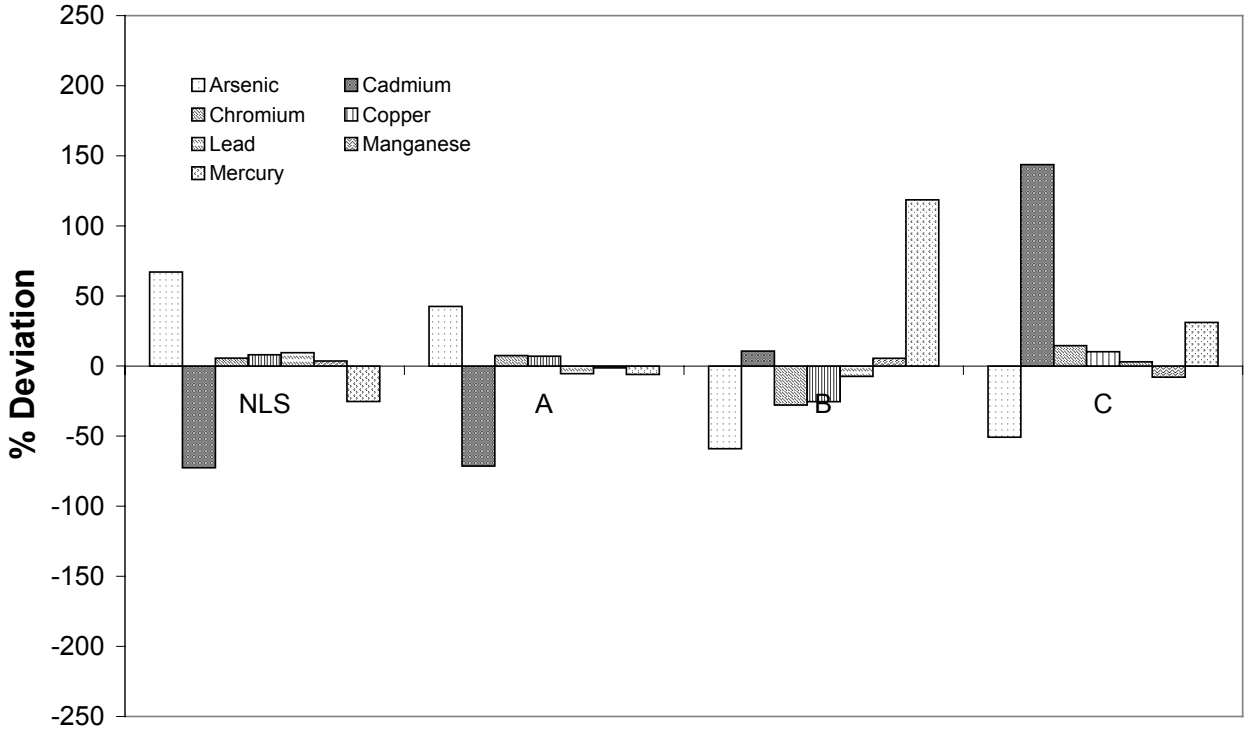

Figure 5.3 Comparison of laboratory performance for analysis of metals/metalloid in Unknown Soil 1 (As, Cd, Cr, Cu, Hg, Mn and Pb)

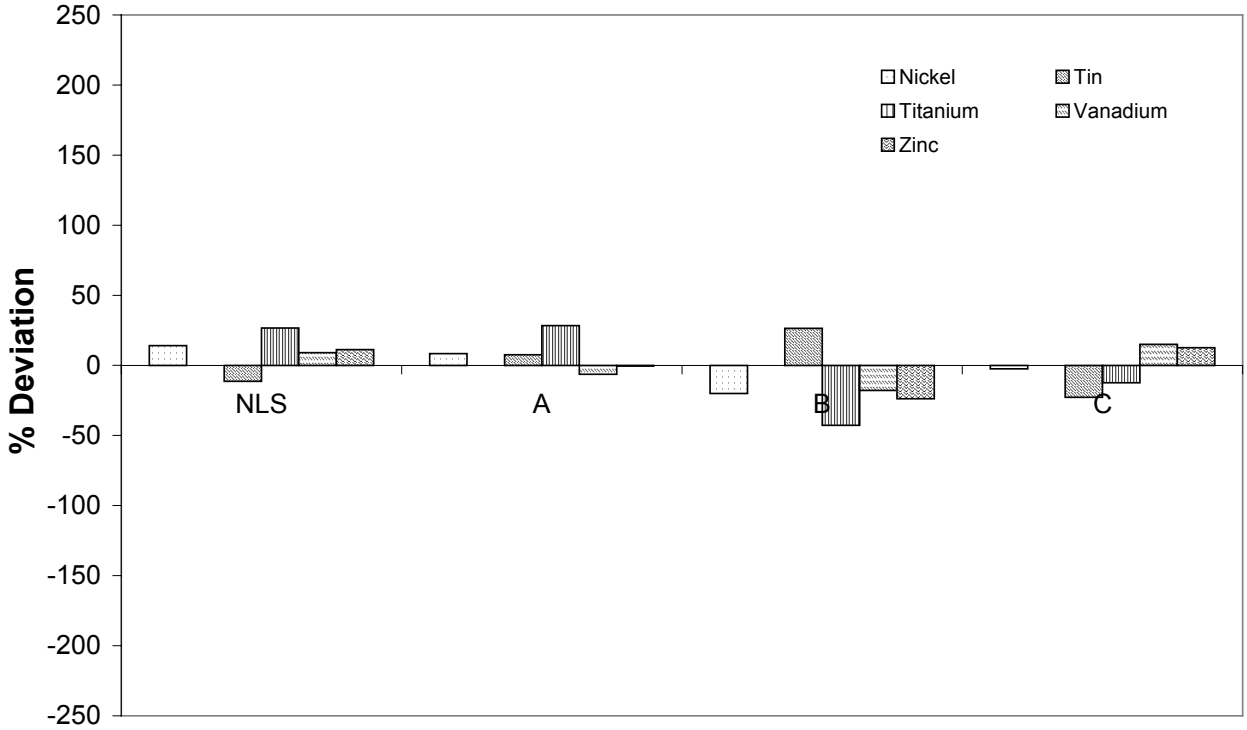

Figure 5.4 Comparison of laboratory performance for analysis of metals in Unknown Soil 1 (Ni, Sn, Ti, V and Zn)

Table 5.3 Analysis of metals/metalloid in Unknown Soil 1 and statistical assessment of laboratory performance

		NLS			A			B			C		
Determinand	Mean	Results $mgkg^{-1}$	u-statistic	u-statistic category	Results $mgkg^{-1}$	u-statistic	u-statistic category	Results $mgkg^{-1}$	u-statistic	u-statistic category	Results $mgkg^{-1}$	u-statistic	u-statistic category
Arsenic	6.08	10.2	1.05	a	8.67	0.663	a	2.50	-0.918	a	3.00	-0.790	a
Cadmium	0.903	0.248	-0.583	a	0.260	-0.572	a	< 1.00	*	*	2.20	1.15	a
Chromium	11.4	12.0	0.303	a	12.2	0.396	a	8.20	-1.47	a	13.0	0.770	a
Copper	30.8	33.3	0.473	a	33.0	0.416	a	23.0	-1.50	a	34.0	0.607	a
Lead	107	117	1.23	a	101	-0.689	a	99.0	-0.929	a	110	0.390	a
Manganese	104	108	0.601	a	103	-0.200	a	110	0.922	a	96.0	-1.32	a
Mercury	0.457	0.342	-0.879	a	0.430	-0.208	a	< 1.00	*	*	0.600	1.09	a
Nickel	10.3	11.7	0.943	a	11.1	0.553	a	8.20	-1.33	a	10.0	-0.163	a
Platinum	*	< 0.0200	*	*	NR			NR	*	*	< 2.00	*	*
Tin	5.30	4.70	-0.525	a	5.70	0.350	a	6.70	1.22	a	4.10	-1.05	a
Titanium	126	159	0.783	a	161	0.830	a	72.0	-1.25	a	110	-0.362	a
Vanadium	18.3	19.9	0.607	a	17.1	-0.423	a	15.0	-1.20	a	21.0	1.01	a
Zinc	57.7	64.3	0.675	a	57.6	-0.0128	a	44.0	-1.41	a	65.0	0.747	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.3.3 Analysis of Unknown Soil 2 for metals/metalloid

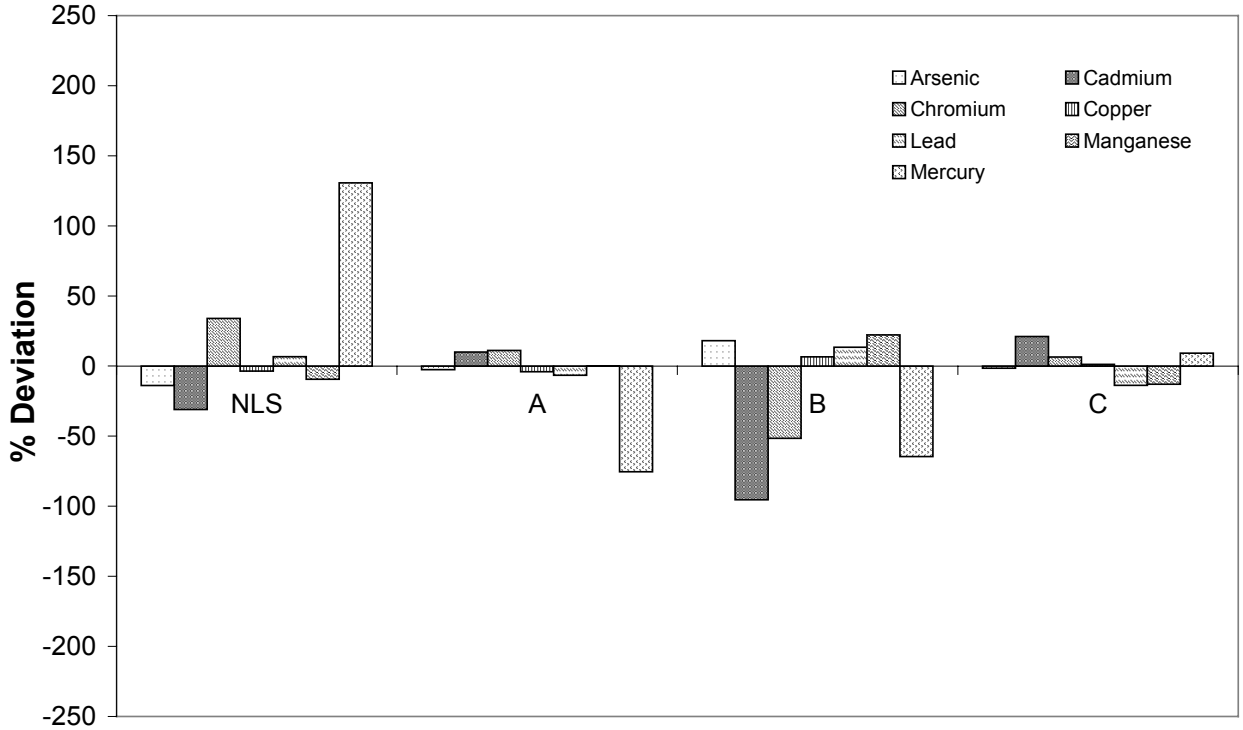

Figure 5.5 Comparison of laboratory performance for analysis of metals/metalloid in Unknown Soil 2 (As, Cd, Cr, Cu, Hg, Mn and Pb)

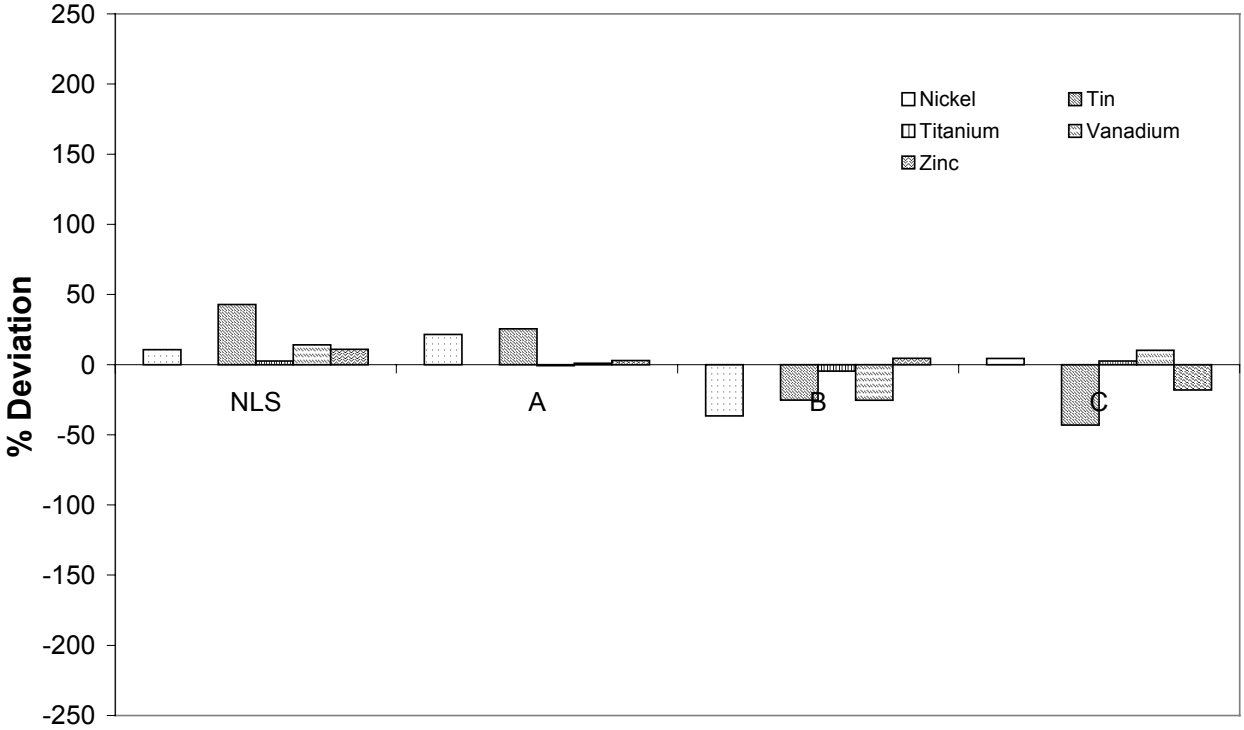

Figure 5.6 Comparison of laboratory performance for analysis of metals in Unknown Soil 2 (Ni, Sn, Ti, V and Zn)

Table 5.4 Analysis of metals/metalloid in Unknown Soil 2 and statistical assessment of laboratory performance

		NLS			A			B			C		
Determinand	Mean	Results $mgkg^{-1}$	u-statistic	u-statistic category	Results $mgkg^{-1}$	u-statistic	u-statistic category	Results $mgkg^{-1}$	u-statistic	u-statistic category	Results $mgkg^{-1}$	u-statistic	u-statistic category
Arsenic	2030	1750	-1.05	a	1980	-0.194	a	2400	1.36	a	2000	-0.120	a
Cadmium	21.5	14.8	-1.13	a	23.6	0.362	a	< 1.00	*	*	26.0	0.769	a
Chromium	20.7	27.7	0.932	a	23.0	0.308	a	10.0	-1.42	a	22.0	0.176	a
Copper	1880	1810	-0.724	a	1800	-0.831	a	2000	1.31	a	1900	0.241	a
Lead	4060	4330	0.550	a	3790	-0.530	a	4600	1.09	a	3500	-1.11	a
Manganese	4830	4370	-0.595	a	4830	0.00654	a	5900	1.41	a	4200	-0.818	a
Mercury	15.6	35.9	1.38	a	3.82	-0.794	a	5.50	-0.681	a	17.0	0.0978	a
Nickel	26.8	29.7	0.422	a	32.6	0.847	a	17.0	-1.44	a	28.0	0.172	a
Platinum	*	< 0.0200	*	*	NR			NR			5.00	*	*
Tin	33.4	47.7	1.05	a	41.9	0.625	a	25.0	-0.618	a	19.0	-1.06	a
Titanium	273	280	0.763	a	271	-0.184	a	260	-1.34	a	280	0.763	a
Vanadium	28.1	32.1	0.795	a	28.4	0.0550	a	21.0	-1.43	a	31.0	0.575	a
Zinc	4880	5410	0.862	a	5020	0.225	a	5100	0.355	a	4000	-1.44	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.3.4 Analysis of Unknown Soil 3 for metals/metalloid

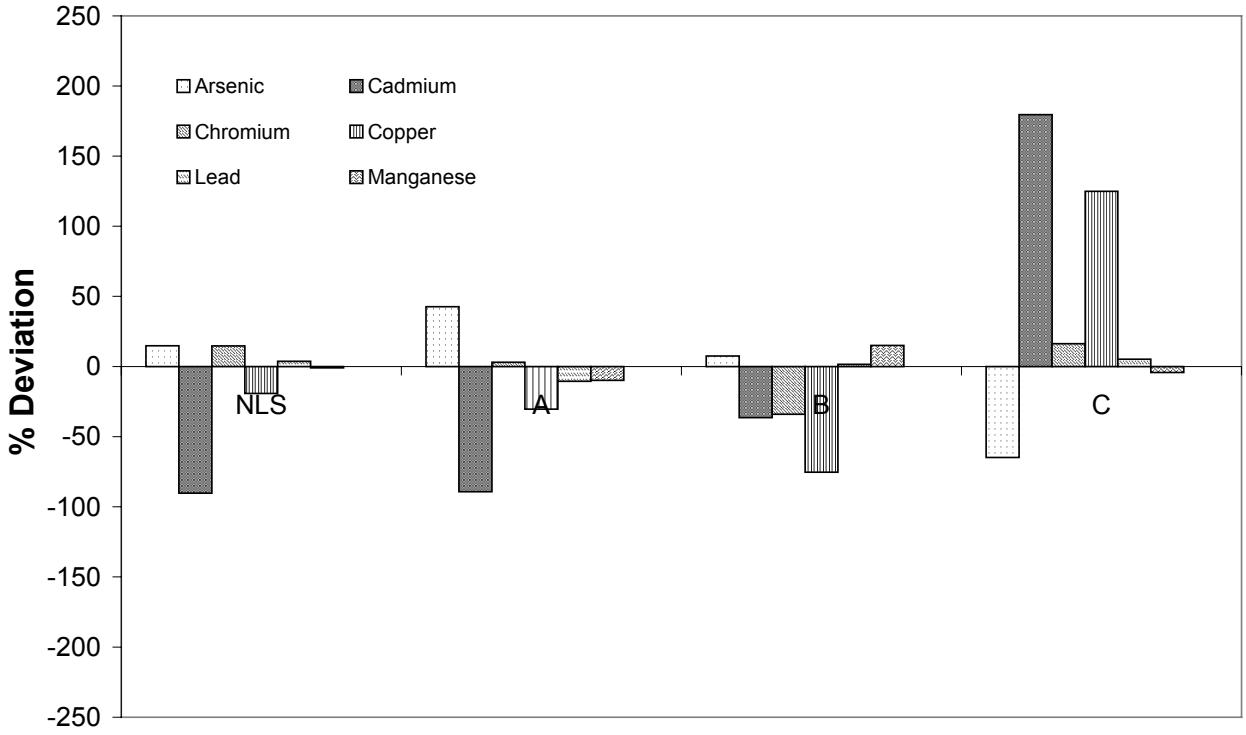

Figure 5.7 Comparison of laboratory performance for analysis of metals/metalloid in Unknown Soil 3 (As, Cd, Cr, Cu, Mn and Pb)

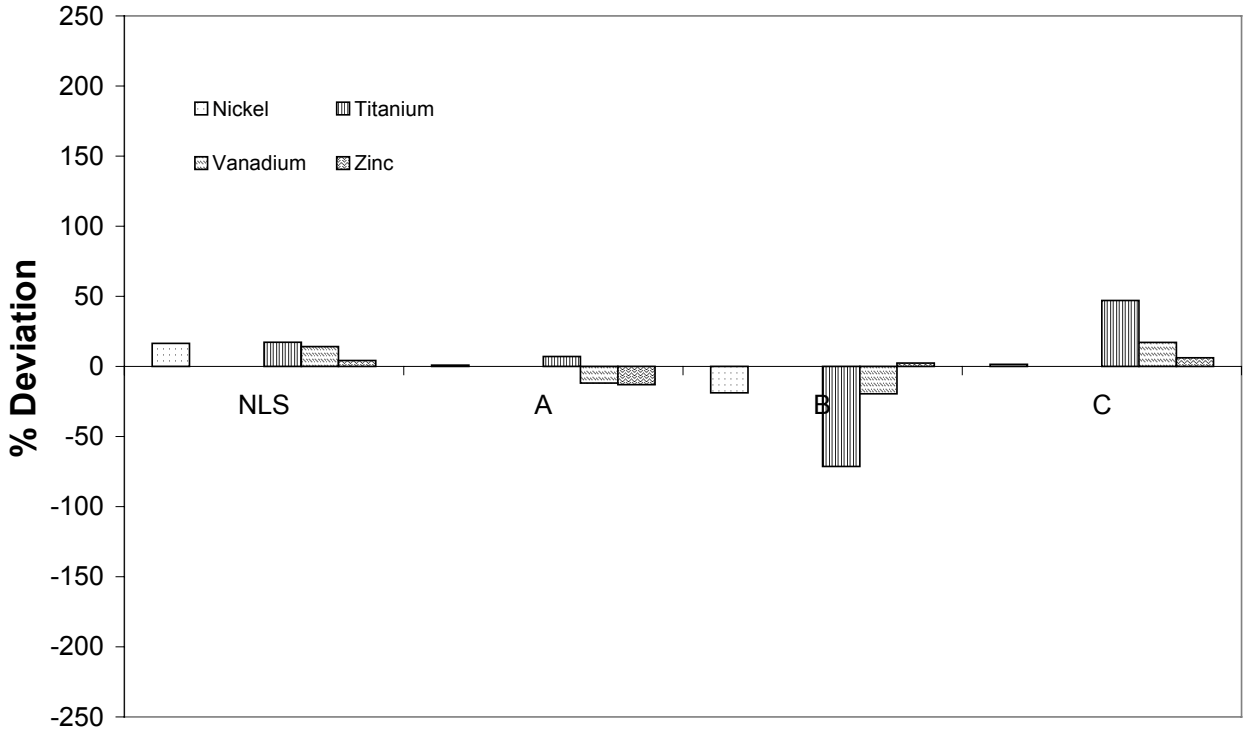

Figure 5.8 Comparison of laboratory performance for analysis of metals in Unknown Soil 3 (Ni, Ti, V and Zn)

Table 5.5 Analysis of metals/metalloid in Unknown Soil 3 and statistical assessment of laboratory performance

		NLS			A			B			C		
Determinand	Mean	Results $mgkg^{-1}$	u-statistic	u-statistic category	Results $mgkg^{-1}$	u-statistic	u-statistic category	Results $mgkg^{-1}$	u-statistic	u-statistic category	Results $mgkg^{-1}$	u-statistic	u-statistic category
Arsenic	5.68	6.52	0.323	a	8.10	0.931	a	6.10	0.162	a	2.00	-1.42	a
Cadmium	1.57	0.152	-0.581	a	0.170	-0.574	a	< 1.00	*	*	4.40	1.15	a
Chromium	25.8	29.6	0.627	a	26.6	0.132	a	17.0	-1.45	a	30.0	0.693	a
Copper	14.2	11.5	-0.221	a	9.90	-0.350	a	3.50	-0.869	a	32.0	1.44	a
Lead	26.6	27.6	0.523	a	23.8	-1.46	a	27.0	0.209	a	28.0	0.732	a
Manganese	157	155	-0.0898	a	141	-0.928	a	180	1.41	a	150	-0.389	a
Mercury	*	0.135	0.707	a	0.100	-0.707	a	< 1.00	*	*	< 0.400	*	*
Nickel	14.8	17.2	1.14	a	14.9	0.0586	a	12.0	-1.30	a	15.0	0.105	a
Platinum	*	< 0.0200	*	*	NR			NR			< 2.00	*	*
Tin	*	< 2.00	*	*	< 2.00	*	*	< 1.00	*	*	4.20	*	*
Titanium	265	311	0.342	a	284	0.140	a	76.0	-1.41	a	390	0.931	a
Vanadium	43.5	49.7	0.770	a	38.4	-0.639	a	35.0	-1.06	a	51.0	0.932	a
Zinc	51.8	54.0	0.492	a	45.0	-1.48	a	53.0	0.273	a	55.0	0.711	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.3.5 Analysis of herbage for metals

Figure 5.9 Comparison of laboratory performance for analysis of metals in herbage sample (Cd, Cr, Cu, Hg, Mn and Pb)

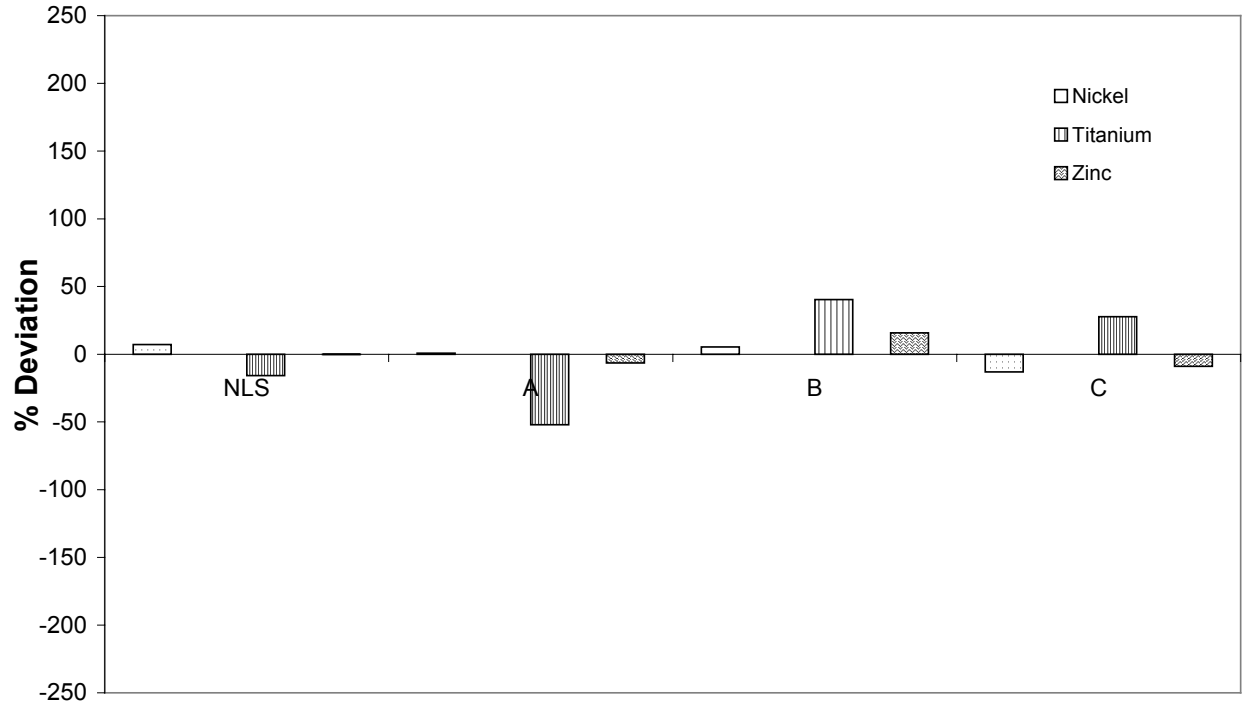

Figure 5.10 Comparison of laboratory performance for analysis of metals in herbage sample (Ni, Ti and Zn)

Table 5.6 Analysis of metals/metalloid in herbage sample and statistical assessment of laboratory performance

		NLS			A			B			C		
Determinand	Mean	Results $mgkg^{-1}$	u-statistic	u-statistic category	Results $mgkg^{-1}$	u-statistic	u-statistic category	Results $mgkg^{-1}$	u-statistic	u-statistic category	Results $mgkg^{-1}$	u-statistic	u-statistic category
Arsenic	*	< 0.500	*	*	0.520	*	*	< 1.00	*	*	< 0.700	*	*
Cadmium	0.170	0.129	-0.665	a	0.140	-0.485	a	< 1.00	*	*	0.240	1.15	a
Chromium	12.5	13.1	0.468	a	11.8	-0.506	a	14.0	1.14	a	11.0	-1.10	a
Copper	10.8	11.4	0.339	a	11.5	0.426	a	8.20	-1.48	a	12.0	0.715	a
Lead	9.22	8.15	-0.573	a	8.52	-0.375	a	12.0	1.49	a	8.20	-0.546	a
Manganese	135	121	-0.836	a	130	-0.308	a	160	1.45	a	130	-0.308	a
Mercury	0.724	0.101	-0.563	a	0.0700	-0.591	a	< 1.00	*	*	2.00	1.15	a
Nickel	6.56	7.02	0.779	a	6.60	0.0754	a	6.90	0.578	a	5.70	-1.43	a
Platinum	*	< 0.0200	*	*	NR			NR			< 3.00	*	*
Tin	*	< 2.00	*	*	< 2.00	*	*	< 1.00	*	*	3.00	*	*
Titanium	15.7	13.2	-0.373	a	7.49	-1.23	a	22.0	0.955	a	20.0	0.653	a
Vanadium	*	< 1.00	*	*	< 2.00	*	*	1.60	*	*	1.30	*	*
Zinc	52.7	52.6	-0.0214	a	49.3	-0.586	a	61.0	1.42	a	48.0	-0.808	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.4 PAH results

Results for PAHs are presented in Figures 5.11–5.25 and Tables 5.7–5.11.

The four laboratories reporting data are indicated by NLS, B, C and D.

5.4.1 Analysis of CRM of known PAH composition

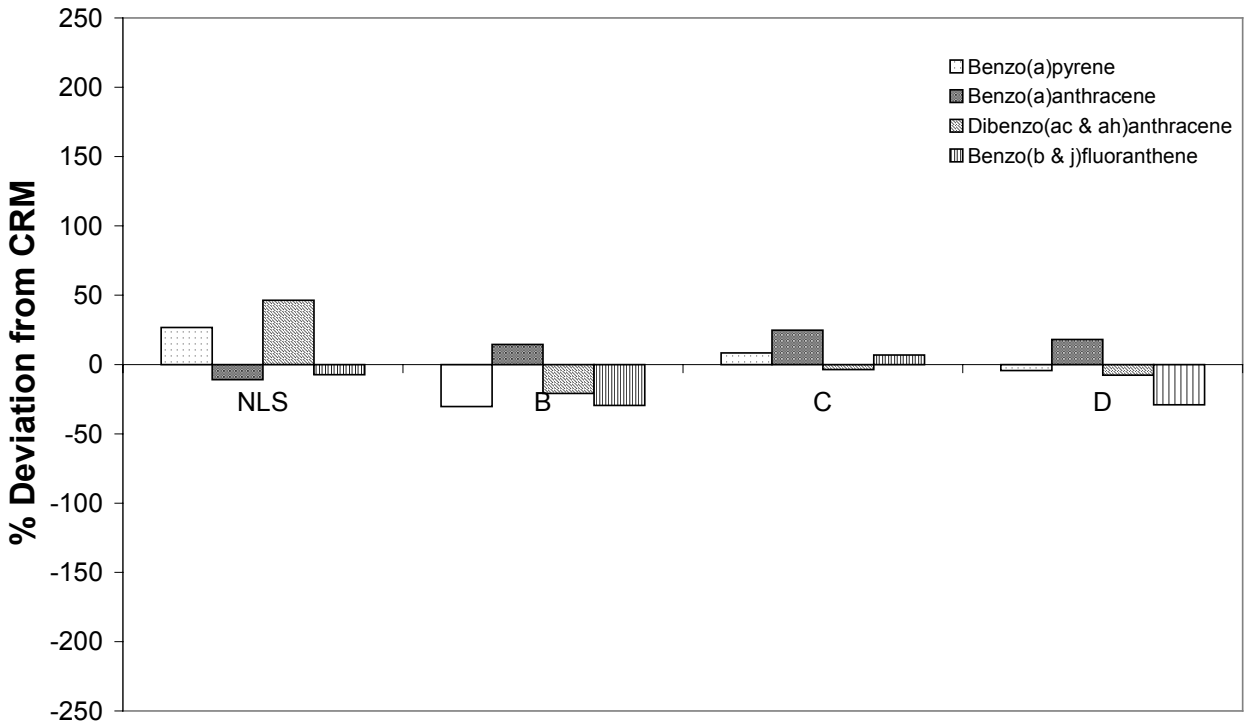

Figure 5.11 Comparison of laboratory performance for analysis of PAH CRM (1)

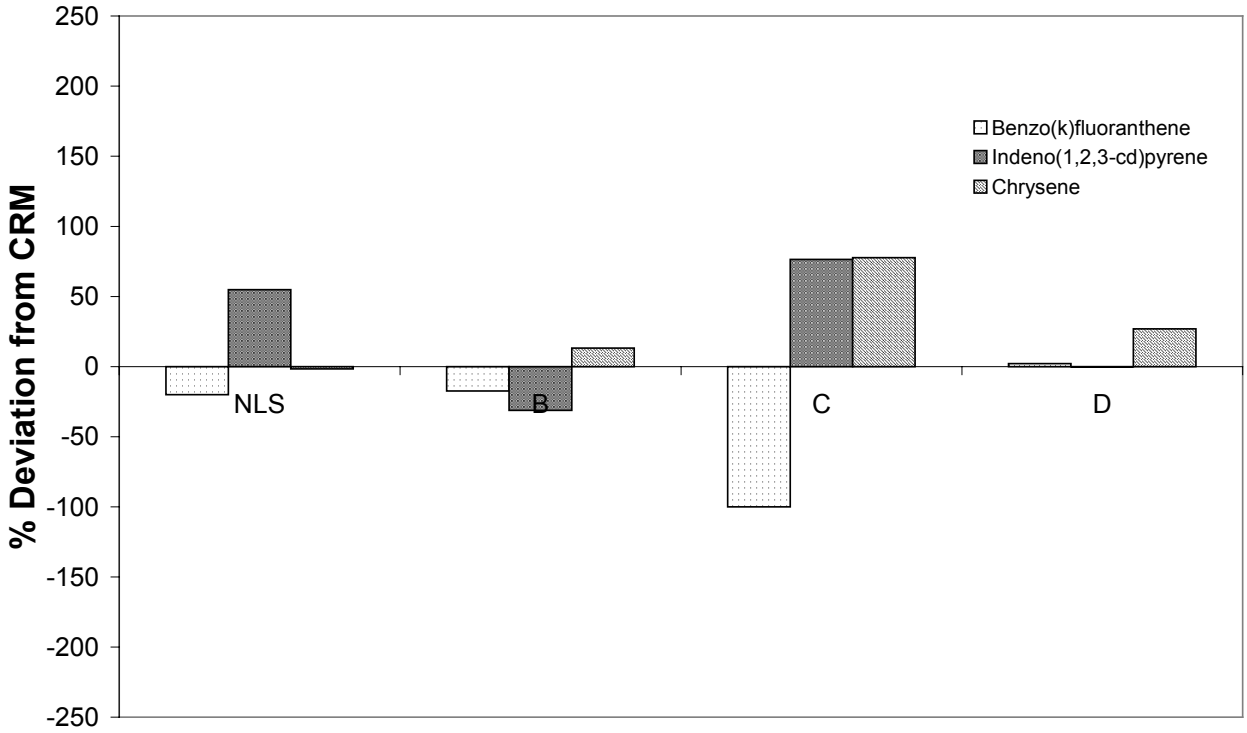

Figure 5.12 Comparison of laboratory performance for analysis of PAH CRM (2)

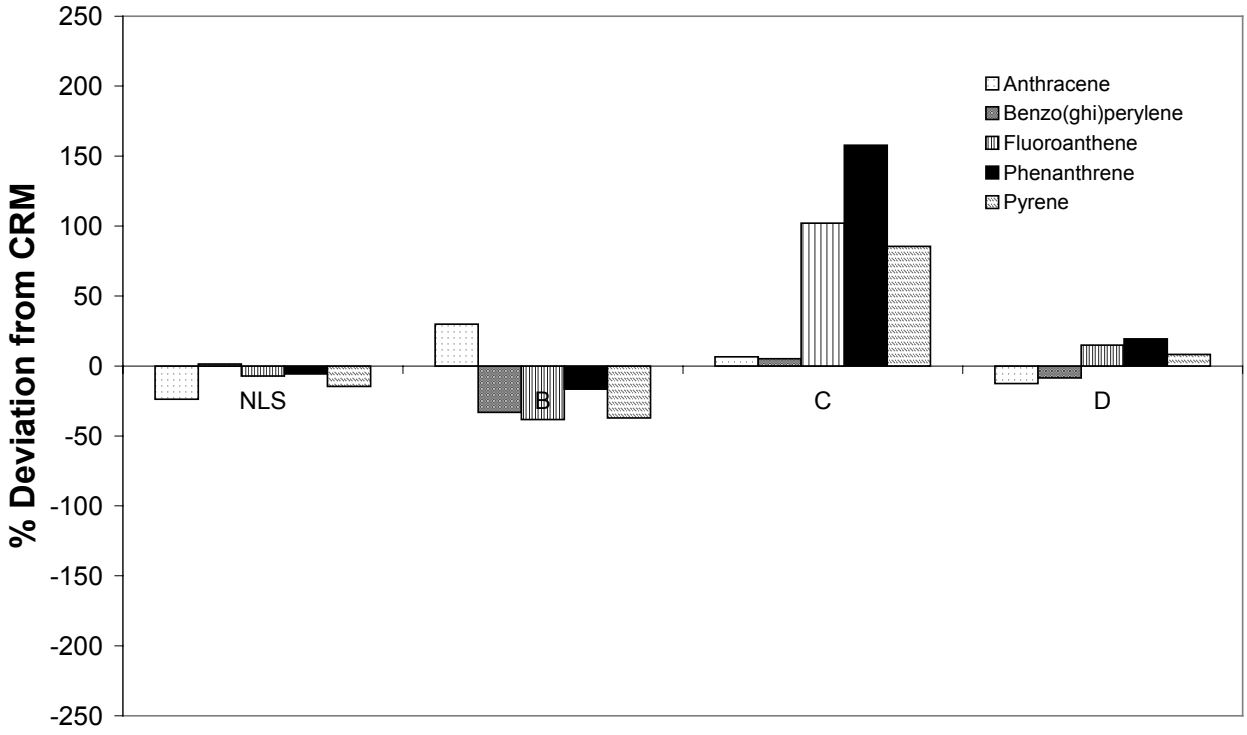

Figure 5.13 Comparison of laboratory performance for analysis of PAH CRM (3)

Table 5.7 Analysis of PAH CRM and statistical assessment of laboratory performance

		NLS			B			C			D		
Determinand	CRM	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category
Benzo(a)pyrene#	4300	5450	1.26	a	3000	-2.12	c	4660	0.742	a	4110	-1.47	a
Benzo(a)anthracene#	4720	4200	-1.21	a	5400	0.626	a	5890	1.95	b	5570	7.72	e
Dibenzo(ah & ac)anthracene#	759	1110	2.83	d	600	-1.09	a	731	-0.255	a	700	-0.720	a
Benzo(b & j)fluoranthene#	5960	5520	-0.433	a	4200	-1.46	a	6360	0.378	a	4220	-2.02	c
Benzo(k)fluoranthene#	2300	1840	-1.88	b	1900	-0.931	a	NR			2350	0.235	a
Indeno(1,2,3-cd)pyrene	2470	3830	0.762	a	1700	-0.435	a	4360	1.05	a	2470	-0.00287	a
Chrysene#	4860	4780	-0.198	a	5500	0.579	a	8640	4.34	e	6170	13.1	e
Acenaphthene	337	358	0.0988	a	300	-0.168	a	552	0.979	a	475	0.649	a
Acenaphthylene	1410	557	-0.502	a	1100	-0.179	a	4330	1.67	b	1050	-0.213	a
Anthracene#	1770	1350	-1.25	a	2300	0.936	a	1890	0.310	a	1550	-0.673	a
Benzo(ghi)perylene#	2840	2880	0.259	a	1900	-2.39	c	2990	0.467	a	2600	-2.40	c
Coronene	*	897	*	*	NR			NR			NR		
Fluoroanthene#	8920	8280	-0.300	a	5500	-2.99	d	18000	4.97	e	10300	4.16	e
Fluorene	412	458	0.176	a	400	-0.0460	a	705	1.10	a	499	0.336	a
1-Methylphenanthrene	*	1280	*	*	NR			5140	*	*	NR		
2-Methylphenanthrene	*	1610	*	*	NR			7740	*	*	NR		
Phenanthrene#	5270	4970	-0.592	a	4400	-0.959	a	13600	6.04	e	6290	4.64	e
Pyrene#	9700	8290	-0.857	a	6100	-2.79	d	18000	4.49	e	10500	1.90	b

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.
# A CRM value is available for this determinand.
Non # values are calculated using the approach described in Section 4.2.

5.4.2 Analysis of Unknown Soil 1 for PAHs

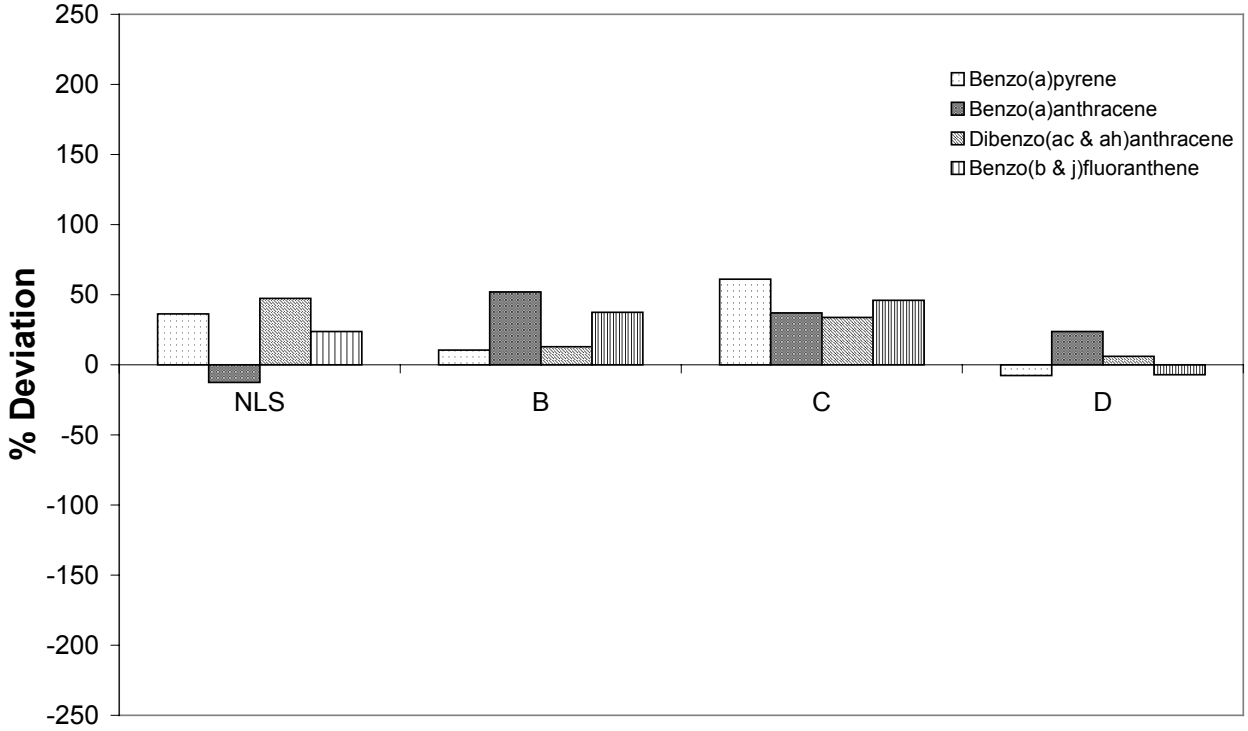

Figure 5.14 Comparison of laboratory performance for analysis of PAHs in Unknown Soil 1 (1)

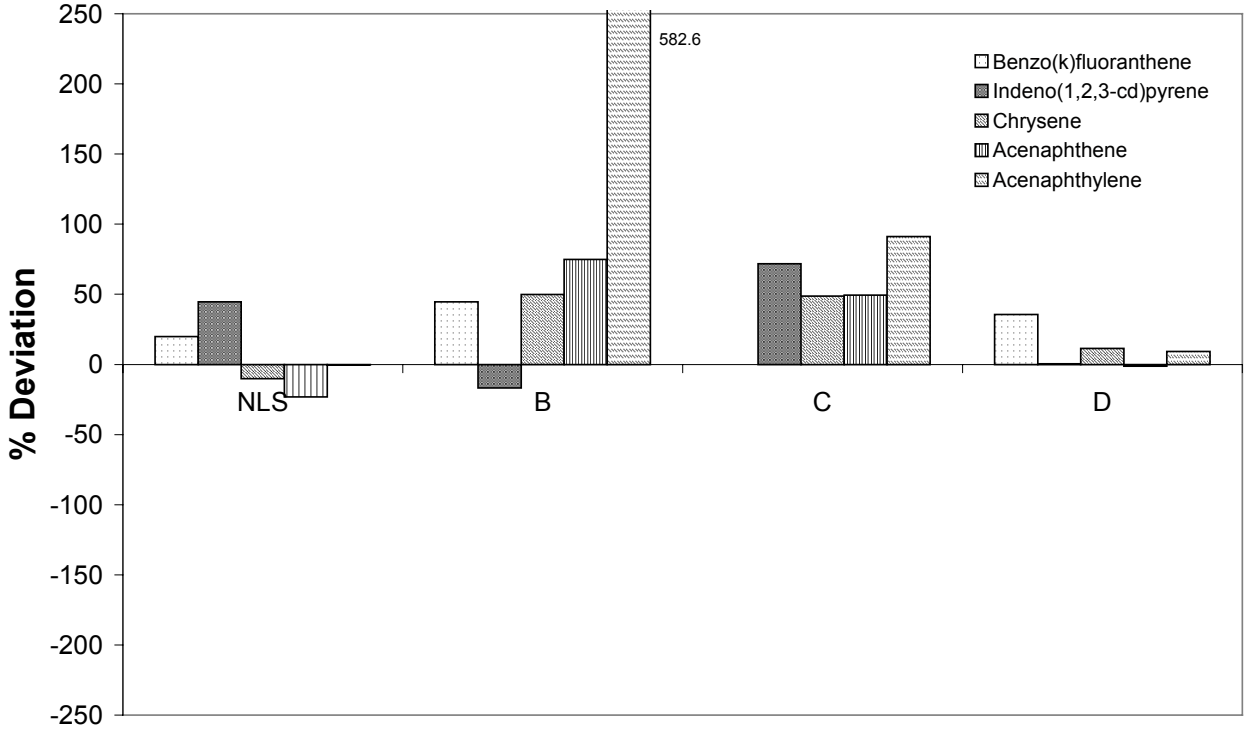

Figure 5.15 Comparison of laboratory performance for analysis of PAHs in Unknown Soil 1 (2)

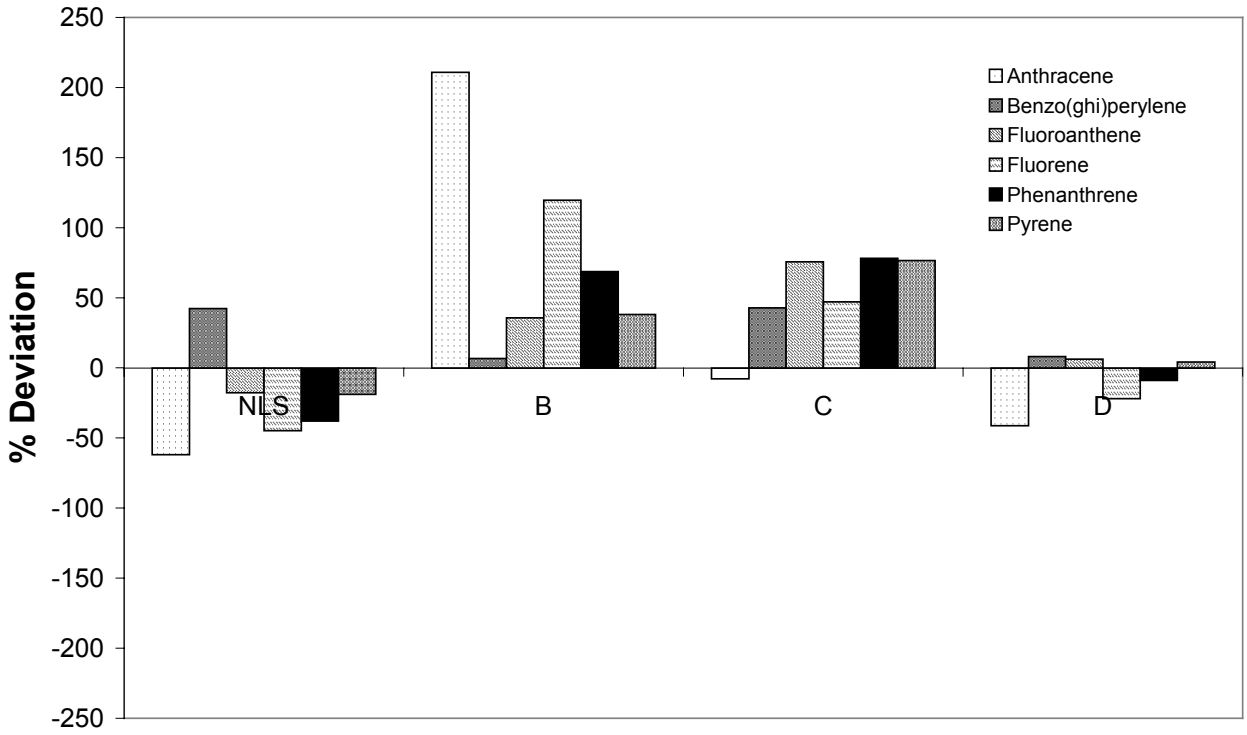

Figure 5.16 Comparison of laboratory performance for analysis of PAHs in Unknown Soil 1 (3)

Table 5.8 Analysis of PAHs in Unknown Soil 1 and statistical assessment of laboratory performance

		NLS			B			C			D		
Determinand	Mean	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category
Benzo(a)pyrene	1360	1850	0.551	a	1500	0.159	a	2190	0.958	a	1250	-0.124	a
Benzo(a)anthracene	1050	921	-0.204	a	1600	0.764	a	1440	0.593	a	1300	0.389	a
Dibenzo(ac & ah)anthracene	177	261	0.794	a	200	0.206	a	237	0.565	a	188	0.105	a
Benzo(b & j)fluoranthene	1380	1710	0.391	a	1900	0.572	a	2020	0.751	a	1280	-0.120	a
Benzo(k)fluoranthene	484	580	0.291	a	700	0.608	a	NR			656	0.527	a
Indeno(1,2,3-cd)pyrene	962	1390	0.660	a	800	-0.247	a	1650	1.05	a	966	0.00693	a
Chrysene	1270	1140	-0.163	a	1900	0.729	a	1890	0.771	a	1410	0.186	a
Acenaphthene	114	87.9	-0.340	a	200	0.977	a	171	0.709	a	113	-0.0177	a
Acenaphthylene	14.7	14.6	-0.00436	a	< 100	*	*	28.0	1.13	a	16.0	0.118	a
Anthracene	386	147	-0.505	a	1200	1.53	a	356	-0.0632	a	227	-0.336	a
Benzo(ghi)perylene	843	1200	0.719	a	900	0.108	a	1200	0.710	a	911	0.138	a
Coronene	*	433	*	*	NR			NR			NR		
Fluoroanthene	2360	1940	-0.257	a	3200	0.501	a	4150	1.11	a	2510	0.0961	a
Fluorene	91.1	50.3	-0.525	a	200	1.25	a	134	0.545	a	71.0	-0.259	a
1-Methylphenanthrene	*	126	*	*	NR			255	*	*	NR		
2-Methylphenanthrene	*	170	*	*	NR			313	*	*	NR		
Phenanthrene	1240	772	-0.506	a	2100	0.838	a	2220	1.02	a	1130	-0.121	a
Pyrene	2320	1880	-0.276	a	3200	0.529	a	4090	1.11	a	2410	0.0630	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.4.3 Analysis of Unknown Soil 2 for PAHs

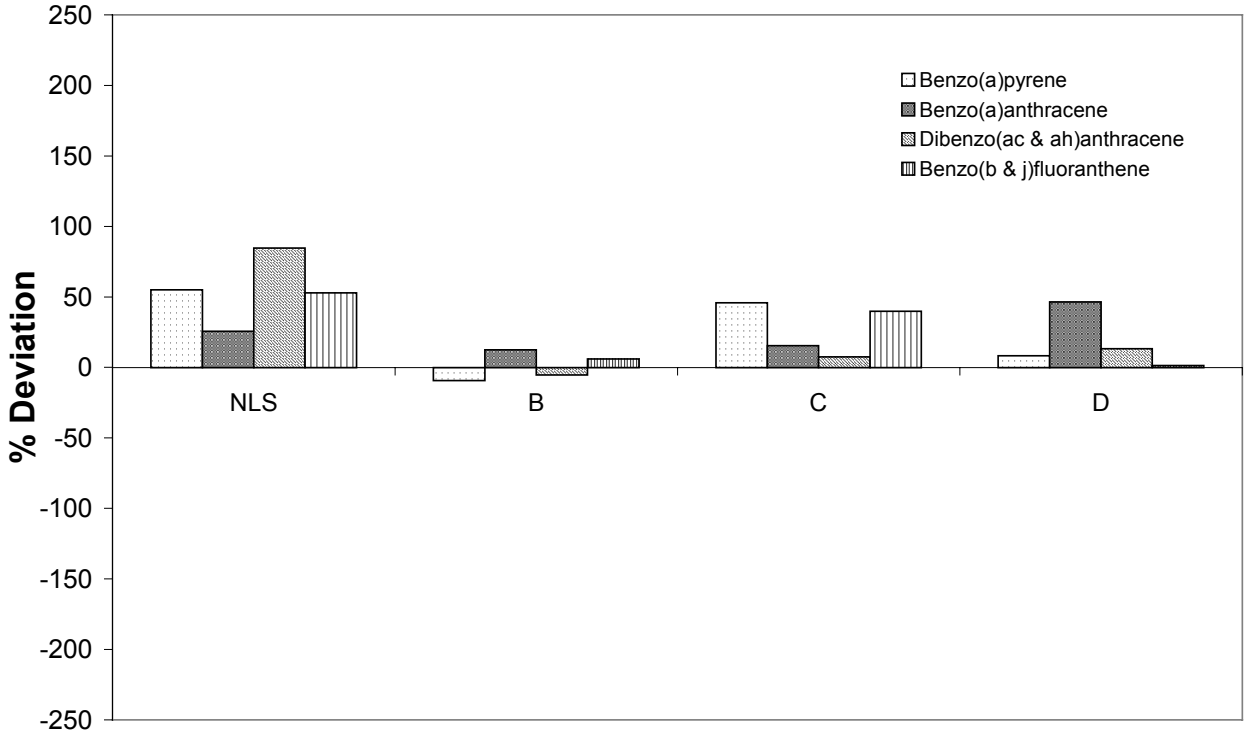

Figure 5.17 Comparison of laboratory performance for analysis of PAHs in Unknown Soil 2 (1)

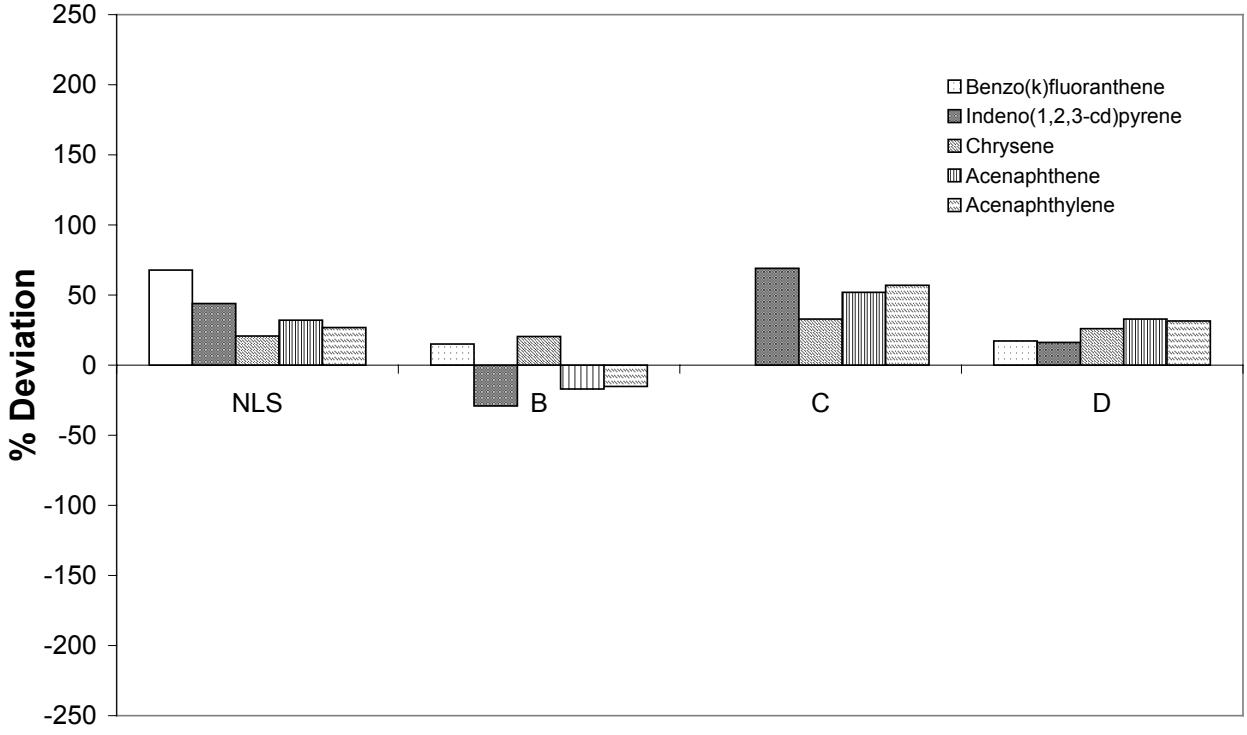

Figure 5.18 Comparison of laboratory performance for analysis of PAHs in Unknown Soil 2 (2)

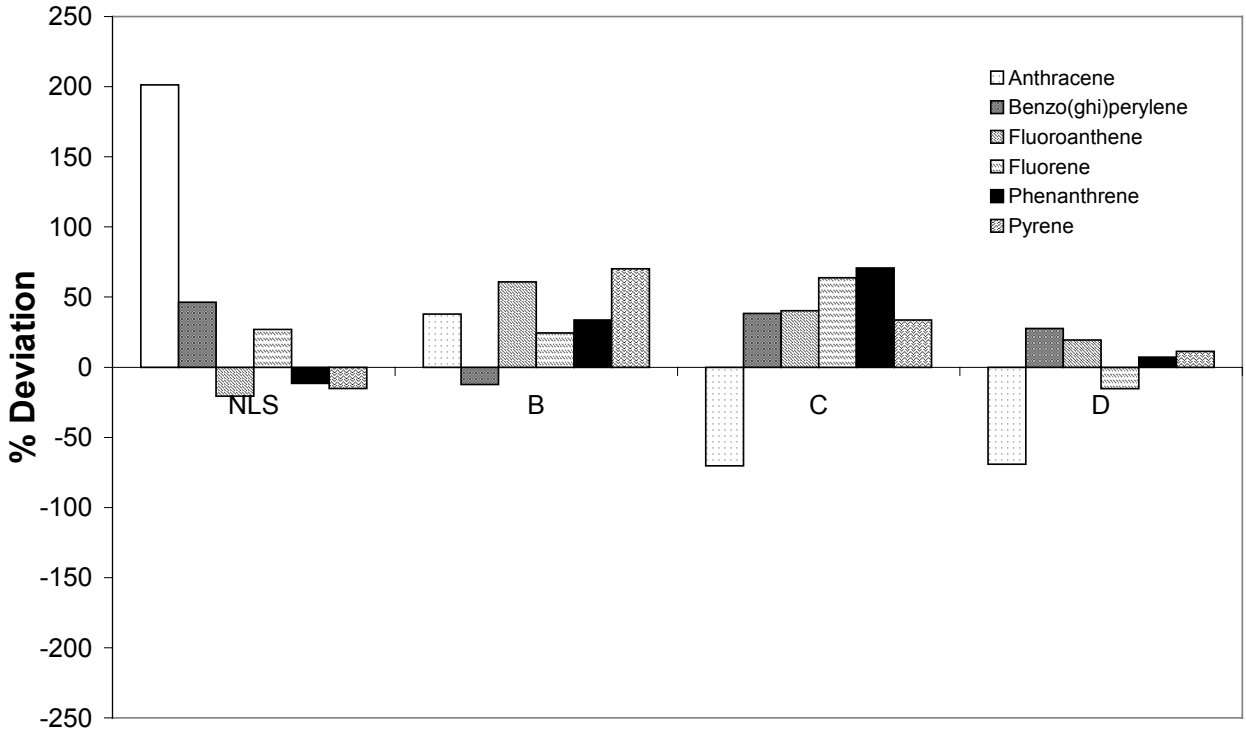

Figure 5.19 Comparison of laboratory performance for analysis of PAHs in Unknown Soil 2 (3)

Table 5.9 Analysis of PAHs in Unknown Soil 2 and statistical assessment of laboratory performance

		NLS			B			C			D		
Determinand	Mean	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category
Benzo(a)pyrene	2980	4620	0.823	a	2700	-0.145	a	4350	0.722	a	3230	0.135	a
Benzo(a)anthracene	3020	3800	0.438	a	3400	0.203	a	3490	0.263	a	4420	0.807	a
Dibenzo(ah & ac)anthracene	528	975	1.25	a	500	-0.0772	a	567	0.110	a	598	0.201	a
Benzo(b & j)fluoranthene	3960	6060	0.855	a	4200	0.0934	a	5540	0.645	a	4020	0.0242	a
Benzo(k)fluoranthene	1220	2040	0.939	a	1400	0.202	a	NR			1430	0.242	a
Indeno(1,2,3-cd)pyrene	2400	3450	0.643	a	1700	-0.427	a	4050	1.00	a	2780	0.242	a
Chrysene	3400	4110	0.364	a	4100	0.334	a	4520	0.570	a	4290	0.463	a
Acenaphthene	120	159	0.522	a	100	-0.266	a	183	0.821	a	160	0.535	a
Acenaphthylene	236	299	0.435	a	200	-0.238	a	370	0.895	a	310	0.511	a
Anthracene	2750	8300	1.61	a	3800	0.299	a	818	-0.566	a	852	-0.556	a
Benzo(ghi)perylene	1940	2840	0.765	a	1700	-0.198	a	2690	0.621	a	2480	0.458	a
Coronene	*	467	*	*	NR			NR			NR		
Fluoroanthene	7460	5920	-0.310	a	12000	0.855	a	10500	0.620	a	8910	0.306	a
Fluorene	161	204	0.430	a	200	0.365	a	263	0.987	a	136	-0.245	a
1-Methylphenanthrene	*	506	*	*	NR			770	*	*	NR		
2-Methylphenanthrene	*	790	*	*	NR			944	*	*	NR		
Phenanthrene	4190	3710	-0.179	a	5600	0.485	a	7160	1.07	a	4490	0.113	a
Pyrene	6460	5480	-0.230	a	11000	0.968	a	8630	0.514	a	7200	0.177	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.4.4 Analysis of Unknown Soil 3 for PAHs

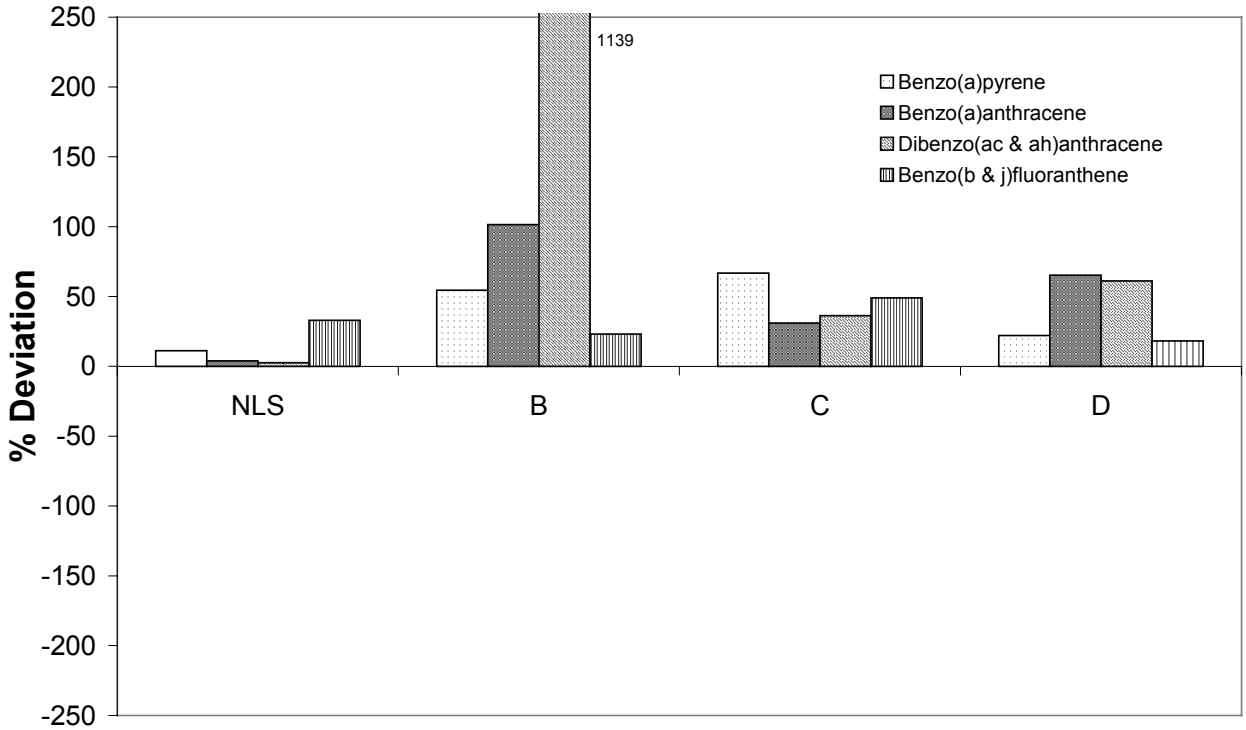

Figure 5.20 Comparison of laboratory performance for analysis of PAHs in Unknown Soil 3 (1)

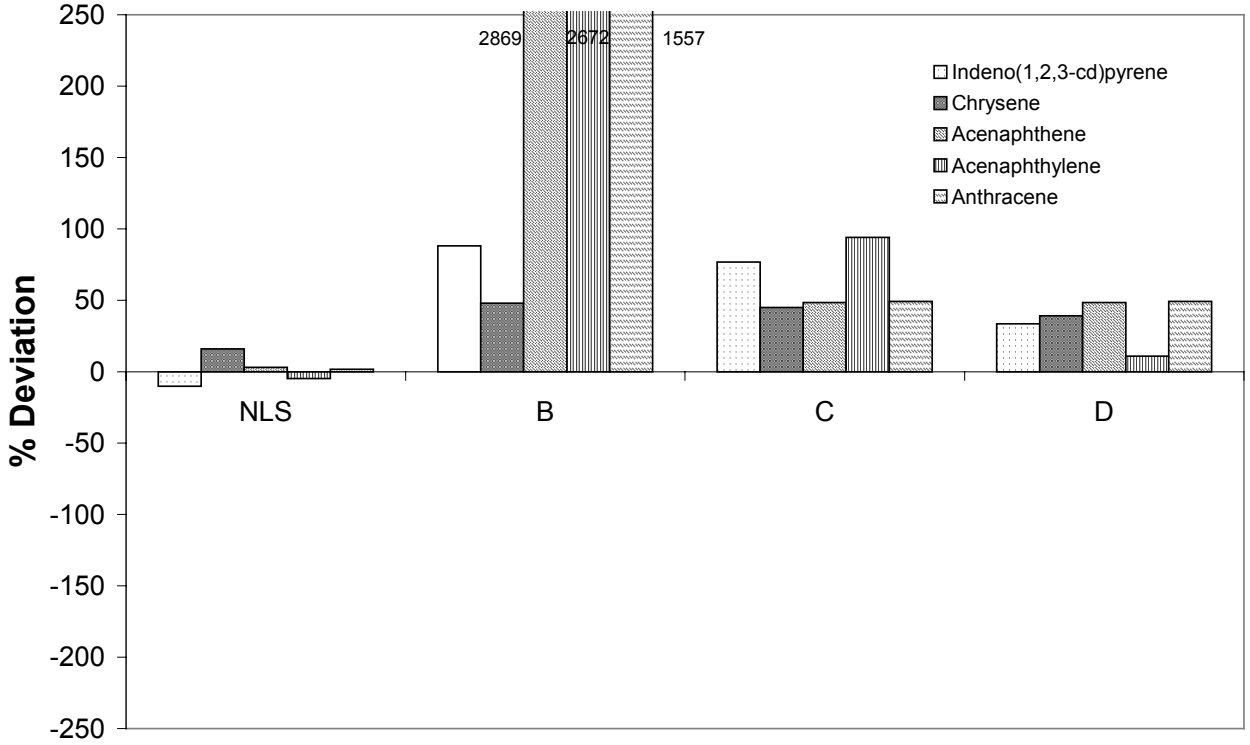

Figure 5.21 Comparison of laboratory performance for analysis of PAHs in Unknown Soil 3 (2)

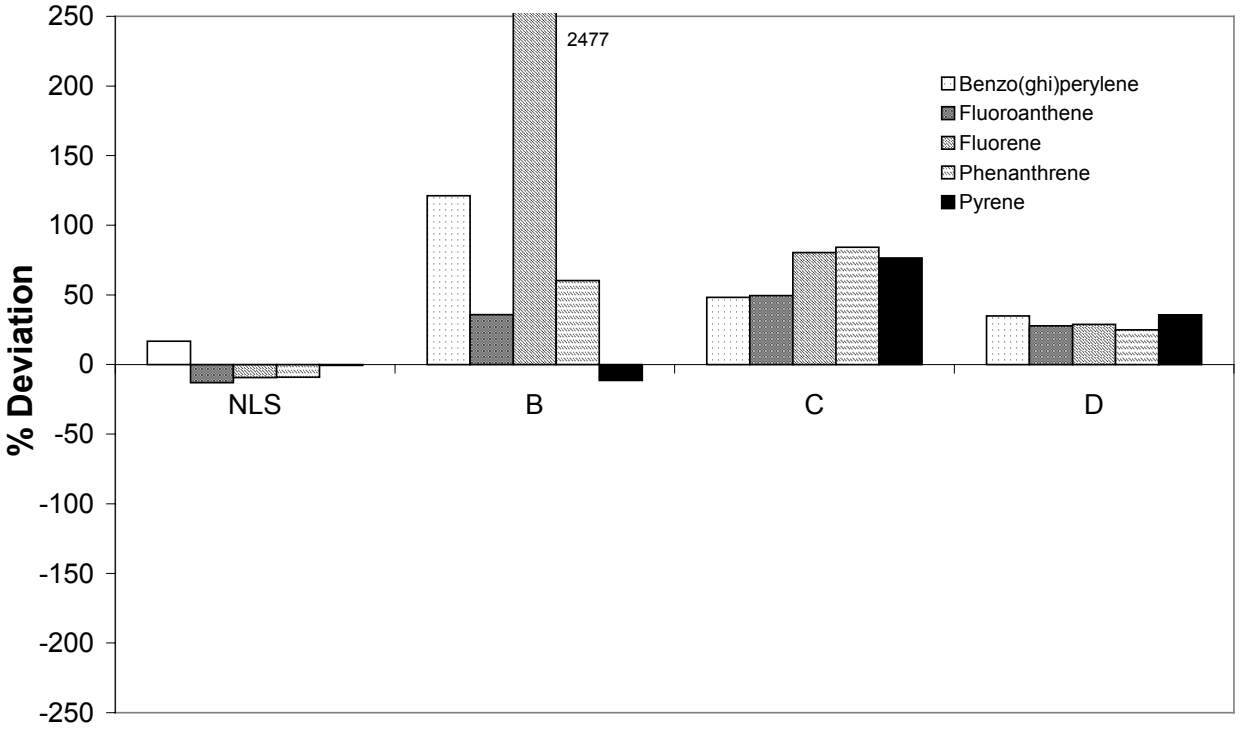

Figure 5.22 Comparison of laboratory performance for analysis of PAHs in Unknown Soil 3 (3)

Table 5.10 Analysis of PAHs in Unknown Soil 3 and statistical assessment of laboratory performance

		NLS			B			C			D		
Determinand	Mean	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category
Benzo(a)pyrene	64.8	72.0	0.153	a	< 100	*	*	108	0.917	a	79.0	0.310	a
Benzo(a)anthracene	49.6	51.5	0.0525	a	< 100	*	*	65.0	0.428	a	82.0	0.916	a
Dibenzo(ah & ac)anthracene	8.07	8.28	0.0365	a	< 100	*	*	11.0	0.503	a	13.0	0.862	a
Benzo(b & j)fluoranthene	81.3	108	0.477	a	< 100	*	*	121	0.704	a	96.0	0.268	a
Benzo(k)fluoranthene	*	35.5	*	*	< 100	*	*	NR			46.0	*	*
Indeno(1,2,3-cd)pyrene	53.2	47.7	-0.135	a	< 100	*	*	94.0	0.989	a	71.0	0.444	a
Chrysene	67.6	78.3	0.232	a	< 100	*	*	98.0	0.649	a	94.0	0.576	a
Acenaphthene	3.37	3.47	0.0435	a	< 100	*	*	5.00	0.677	a	5.00	0.692	a
Acenaphthylene	3.61	3.43	-0.0618	a	< 100	*	*	7.00	1.15	a	4.00	0.137	a
Anthracene	6.03	6.13	0.0229	a	< 100	*	*	9.00	0.684	a	9.00	0.699	a
Benzo(ghi)perylene	45.2	52.8	0.247	a	< 100	*	*	67.0	0.694	a	61.0	0.515	a
Coronene	*	16.1	*	*	NR			NR			NR		
Fluoroanthene	147	128	-0.202	a	200	0.540	a	220	0.793	a	188	0.458	a
Fluorene	3.88	3.52	-0.122	a	< 100	*	*	7.00	1.03	a	5.00	0.379	a
1-Methylphenanthrene	*	6.19	*	*	NR			10.0	*	*	NR		
2-Methylphenanthrene	*	9.06	*	*	NR			12.0	*	*	NR		
Phenanthrene	62.4	56.7	-0.118	a	< 100	*	*	115	1.06	a	78.0	0.324	a
Pyrene	113	112	-0.0104	a	100	-0.167	a	199	1.12	a	153	0.543	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.4.5 Analysis of herbage sample for PAHs

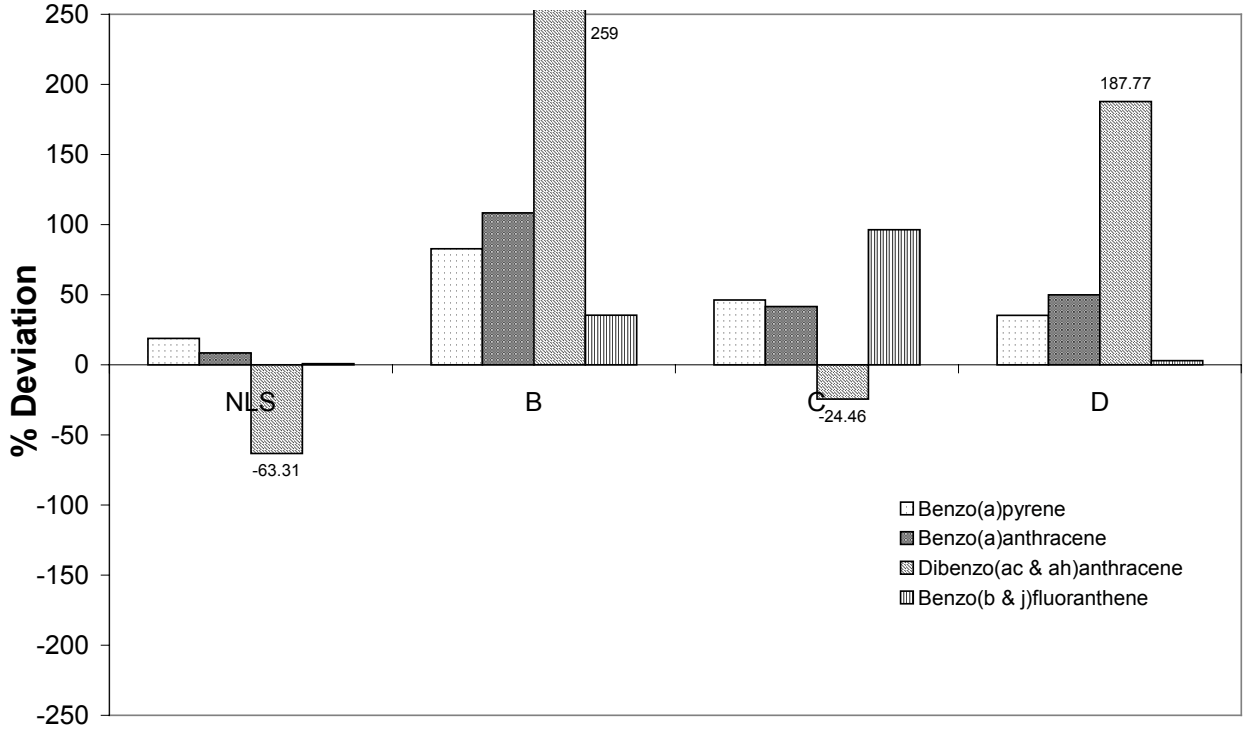

Figure 5.23 Comparison of laboratory performance for analysis of PAHs in herbage sample (1)

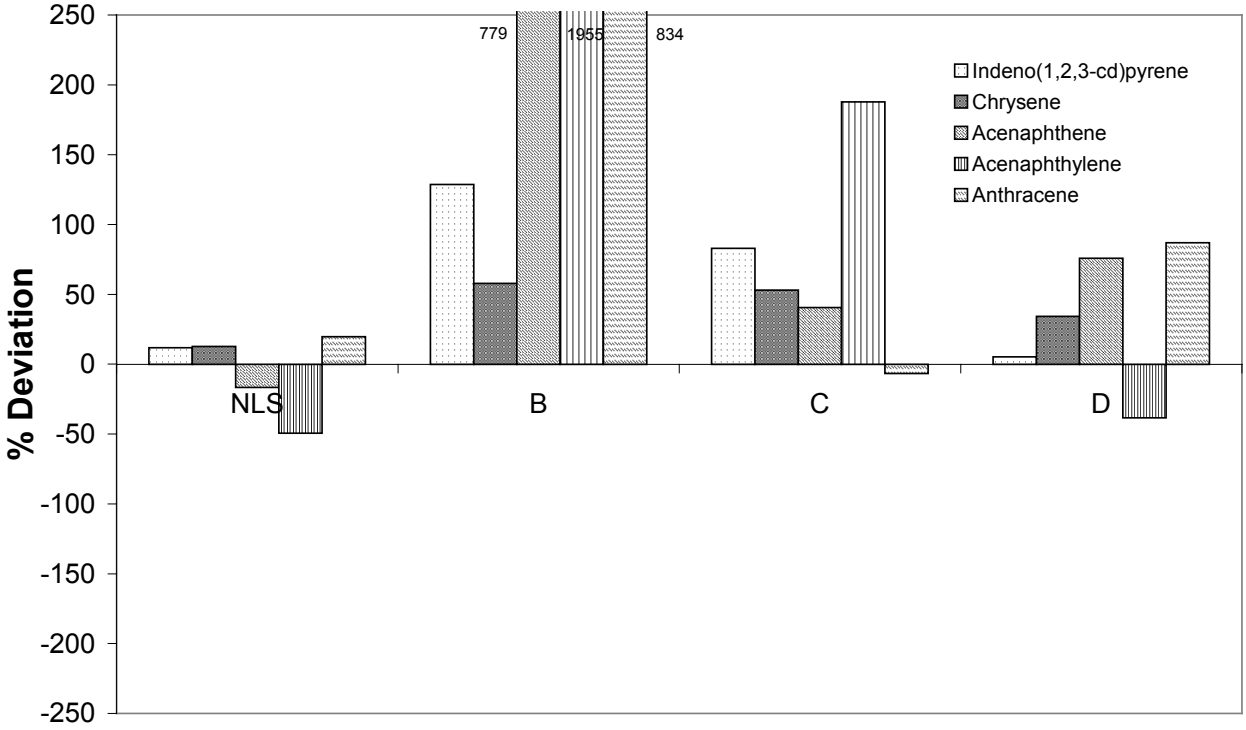

Figure 5.24 Comparison of laboratory performance for analysis of PAHs in herbage sample (2)

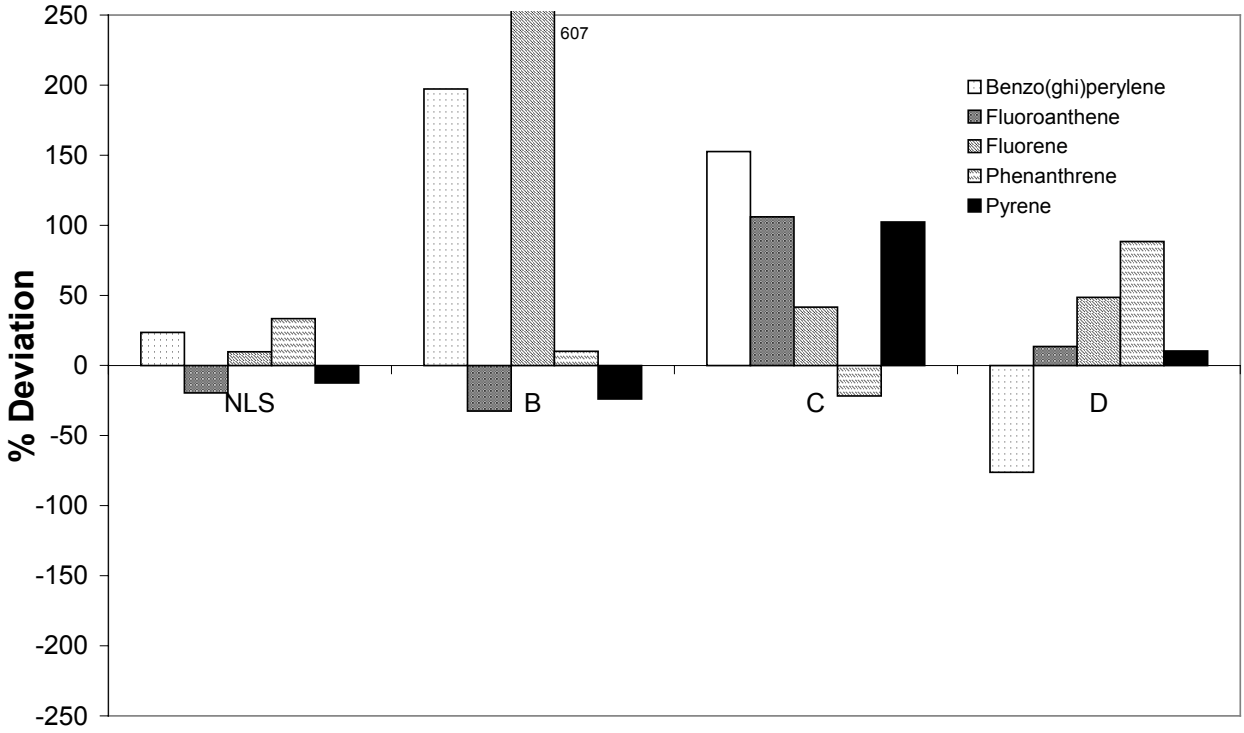

Figure 5.25 Comparison of laboratory performance for analysis of PAHs in herbage sample (3)

Table 5.11 Analysis of PAHs in herbage sample and statistical assessment of laboratory performance

		NLS			B			C			D		
Determinand	Mean	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category
Benzo(a)pyrene	54.8	65.0	0.266	a	< 100	*	*	80.0	0.667	a	74.0	0.520	a
Benzo(a)anthracene	48.0	52.1	0.121	a	< 100	*	*	68.0	0.590	a	72.0	0.723	a
Dibenzo(ah & ac)anthracene	27.8	10.2	-0.491	a	< 100	*	*	21.0	-0.189	a	80.0	1.46	a
Benzo(b & j)fluoranthene	73.9	74.4	0.00922	a	< 100	*	*	145	1.17	a	76.0	0.0363	a
Benzo(k)fluoranthene	*	24.5	*	*	< 100	*	*	NR			31.0	*	*
Indeno(1,2,3-cd)pyrene	43.7	48.9	0.155	a	< 100	*	*	80.0	1.07	a	46.0	0.0690	a
Chrysene	63.4	71.4	0.183	a	< 100	*	*	97.0	0.755	a	85.0	0.498	a
Acenaphthene	11.4	9.50	-0.215	a	< 100	*	*	16.0	0.521	a	20.0	0.988	a
Acenaphthylene	4.87	2.46	-0.386	a	< 100	*	*	14.0	1.43	a	3.00	-0.299	a
Anthracene	10.7	12.8	0.253	a	< 100	*	*	10.0	-0.0839	a	20.0	1.12	a
Benzo(ghi)perylene	33.7	41.6	0.205	a	< 100	*	*	85.0	1.30	a	8.00	-0.663	a
Coronene	*	12.9	*	*	NR			NR			NR		
Fluoroanthene	148	119	-0.223	a	< 100	*	*	305	1.21	a	168	0.158	a
Fluorene	14.1	15.5	0.141	a	< 100	*	*	20.0	0.592	a	21.0	0.708	a
1-Methylphenanthrene	*	13.5	*	*	NR			14.0	*	*	NR		
2-Methylphenanthrene	*	18.7	*	*	NR			28.0	*	*	NR		
Phenanthrene	90.8	121	0.410	a	< 100	*	*	71.0	-0.269	a	171	1.10	a
Pyrene	132	115	-0.148	a	< 100	*	*	266	1.20	a	145	0.124	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.5 PCB results

Results for PCBs are presented in Figures 5.26–5.37 and Tables 5.12–5.16.

The four laboratories reporting data are indicated by NLS, A, B and C.

5.5.1 Analysis of CRM of known PCBs

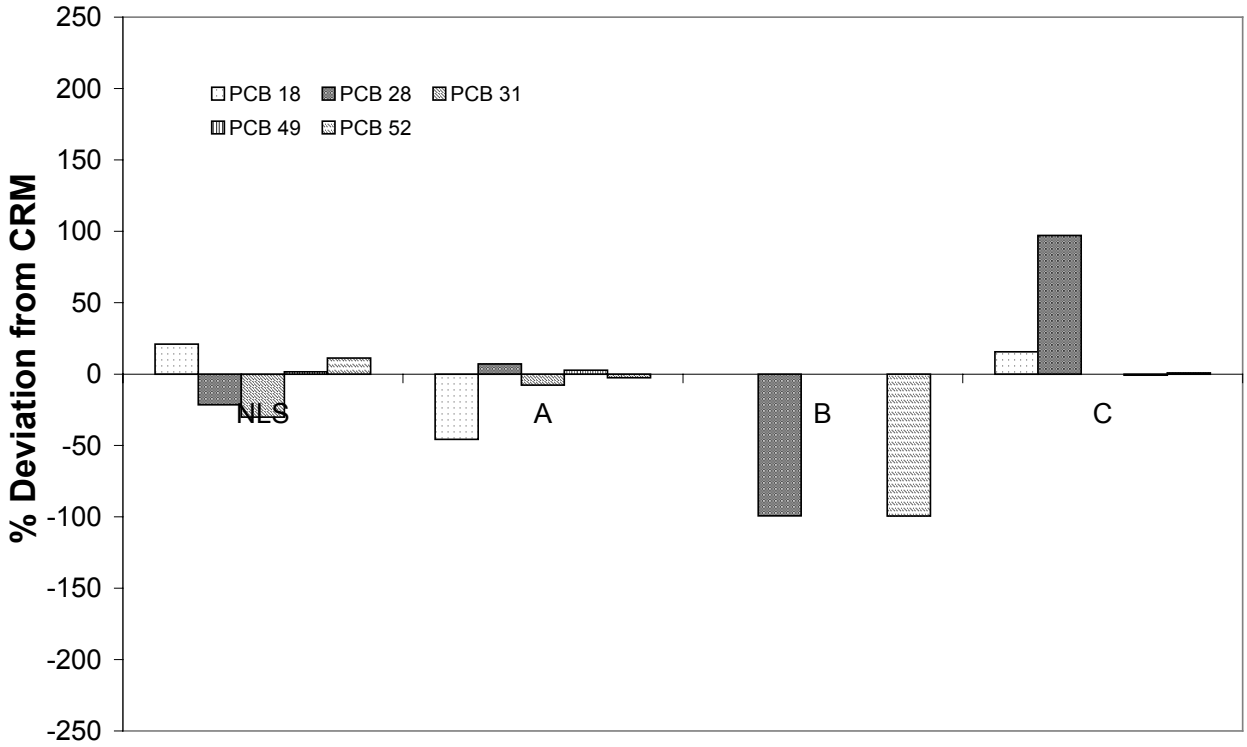

Figure 5.26 Comparison of laboratory performance for analysis of PCB CRM (PCBs 18, 28, 31, 49 and 52)

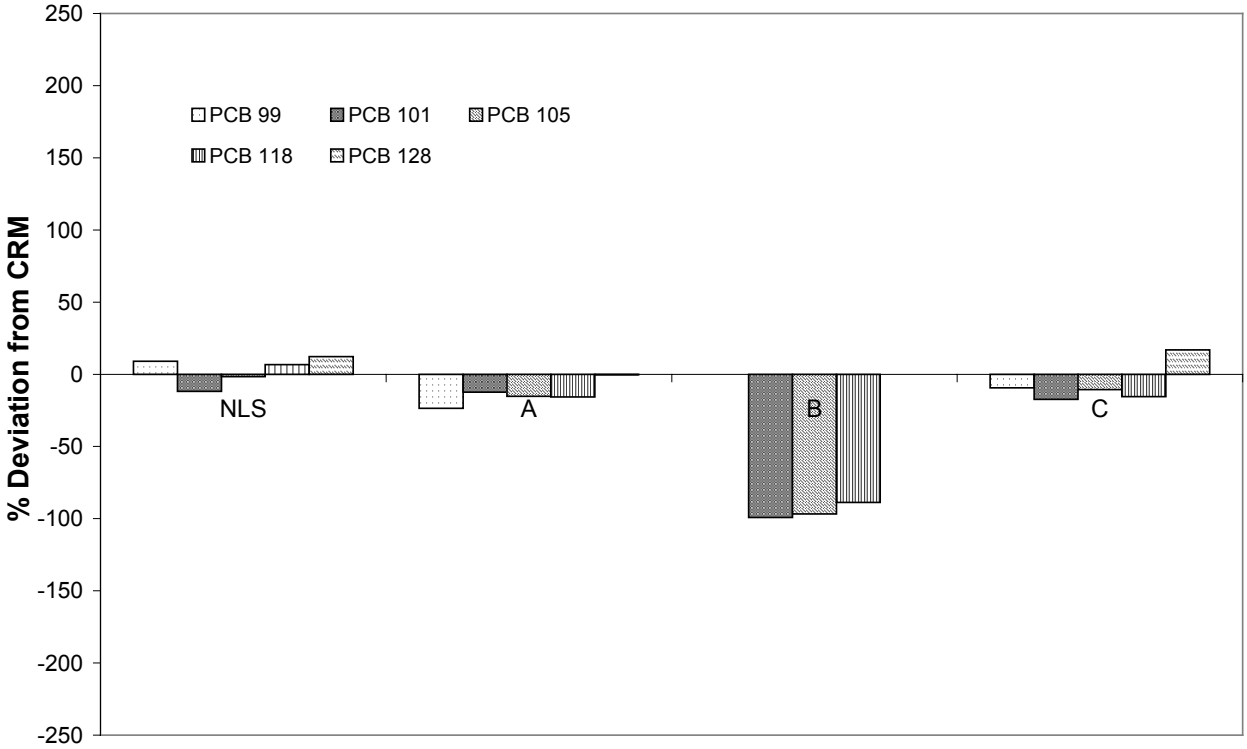

Figure 5.27 Comparison of laboratory performance for analysis of PCB CRM (PCBs 99, 101, 105, 118 and 128)

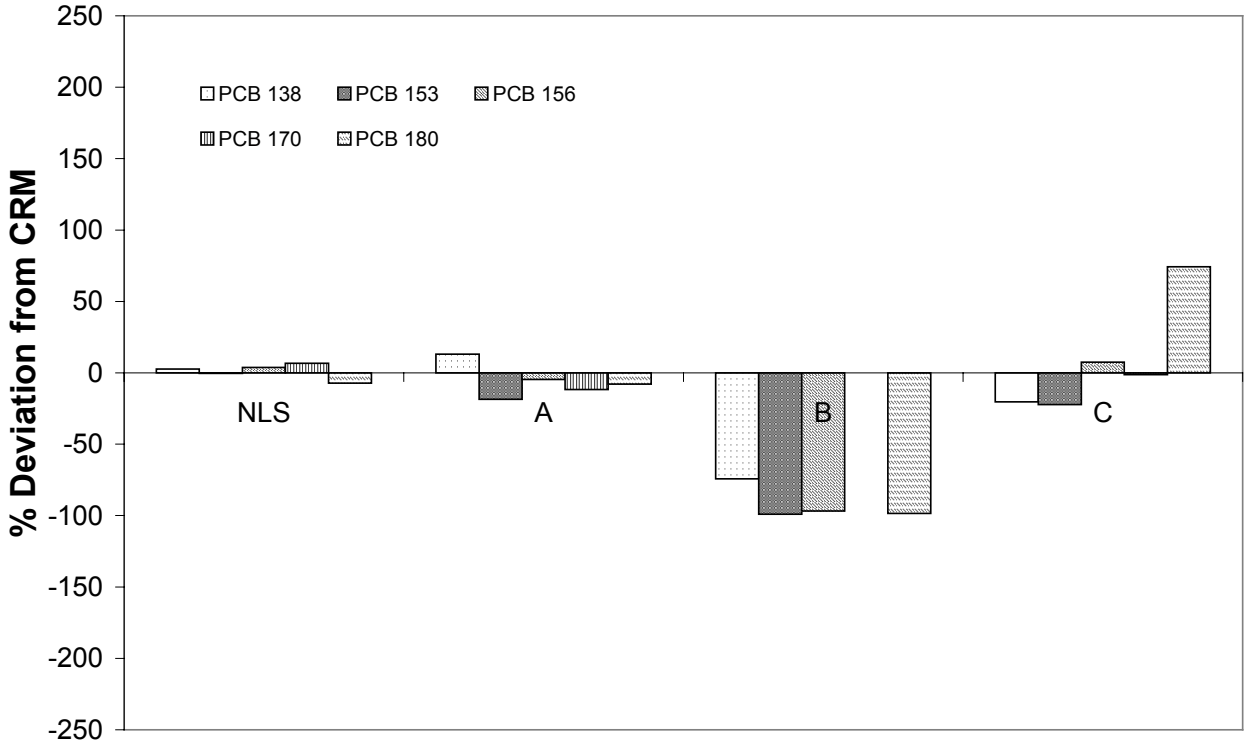

Figure 5.28 Comparison of laboratory performance for analysis of PCB CRM (PCBs 138, 153, 156, 170, 180)

Table 5.12 Analysis of he PCB CRM and statistical assessment of laboratory performance

		NLS			A			B			C		
Determinand	CRM	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category
PCB 18#	51.0	61.7	1.10	a	27.7	-8.97	e	NR			59.0	1.24	a
PCB 28#	80.8	63.4	-1.85	b	86.5	2.10	c	0.500	-29.7	e	159	4.86	e
PCB 31#	78.7	55.0	-3.19	d	72.5	-3.85	e	NR			NR		
PCB 47	26.8	20.6	-0.680	a	23.3	-0.413	a	NR			36.5	1.05	a
PCB 49#	53.0	53.8	0.0946	a	54.5	0.859	a	NR			52.6	-0.0724	a
PCB 51	3.26	2.47	-0.888	a	3.30	0.0566	a	NR			4.00	0.860	a
PCB 52#	79.4	88.3	0.620	a	77.3	-1.03	a	0.380	-39.5	e	80.0	0.0728	a
PCB 77	6.72	7.87	0.248	a	8.87	0.473	a	0.0500	-1.47	a	10.1	0.726	a
PCB 81	1.43	3.49	1.28	a	0.324	-0.705	a	0.100	-0.847	a	1.80	0.235	a
PCB 99#	37.5	40.9	0.606	a	28.7	-3.68	e	NR			34.0	-0.841	a
PCB 101#	73.4	64.8	-1.04	a	64.3	-3.63	e	0.630	-29.1	e	60.6	-1.95	b
PCB 105#	24.5	24.1	-0.143	a	20.8	-3.38	e	0.810	-21.3	e	21.9	-1.06	a
PCB 114	1.84	1.58	-0.574	a	1.39	-1.06	a	2.10	0.429	a	2.30	0.942	a
PCB 118#	58.0	61.9	0.475	a	48.9	-2.11	c	6.50	-11.5	e	49.0	-1.38	a
PCB 123	3.07	1.18	-0.878	a	2.62	-0.208	a	< 0.0500	*	*	5.40	1.05	a
PCB 126	0.896	0.291	-0.430	a	0.263	-0.450	a	0.0300	-0.615	a	3.00	1.46	a
PCB 128#	8.47	9.51	0.809	a	8.44	-0.107	a	NR			9.90	1.39	a
PCB 138#	62.1	63.7	0.161	a	70.2	2.70	d	16.0	-10.5	e	49.5	-2.18	c
PCB 153#	74.0	73.7	-0.0246	a	60.2	-4.76	e	0.640	-25.3	e	57.6	-2.54	c
PCB 156#	6.52	6.77	0.224	a	6.22	-0.455	a	0.210	-9.54	e	7.00	0.499	a
PCB 157	1.30	1.31	0.0168	a	1.82	0.597	a	0.0500	-1.41	a	2.00	0.781	a
PCB 167	2.05	2.64	0.425	a	2.62	0.423	a	0.0500	-1.49	a	2.90	0.618	a
PCB 169	0.686	0.0890	-0.468	a	0.0160	-0.525	a	0.0400	-0.506	a	2.60	1.47	a
PCB 170#	22.6	24.1	0.440	a	19.9	-1.90	b	NR			22.3	-0.114	a
PCB 180#	44.3	41.1	-0.584	a	40.8	-2.88	d	0.660	-36.1	e	77.2	4.21	e
PCB 189	0.575	0.774	0.932	a	< 0.990	*	*	0.550	-0.113	a	0.400	-0.908	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.
# A CRM value is available for this determinand.
Non # values are calculated using the approach described in Section 4.2.

5.5.2 Analysis of Unknown Soil 1 for PCBs

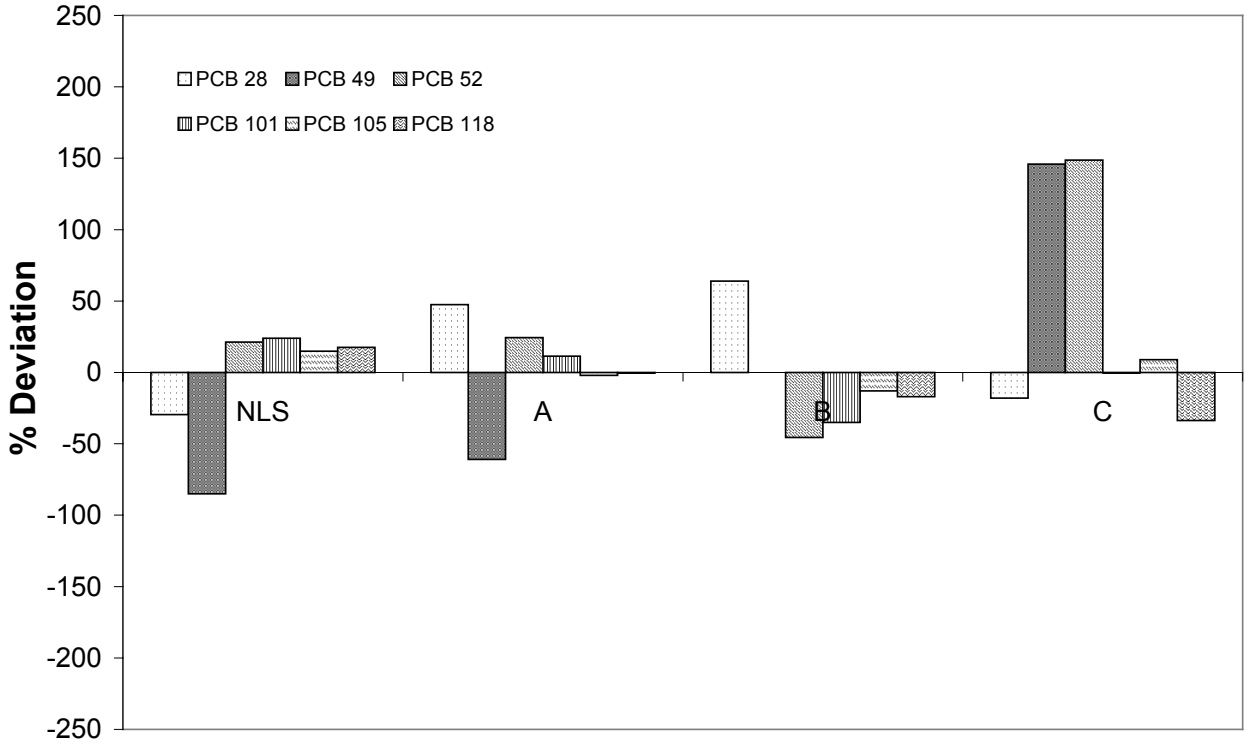

Figure 5.29 Comparison of laboratory performance for analysis of PCBs in Unknown Soil 1 (PCBs 28, 49, 52, 101, 105 and 118)

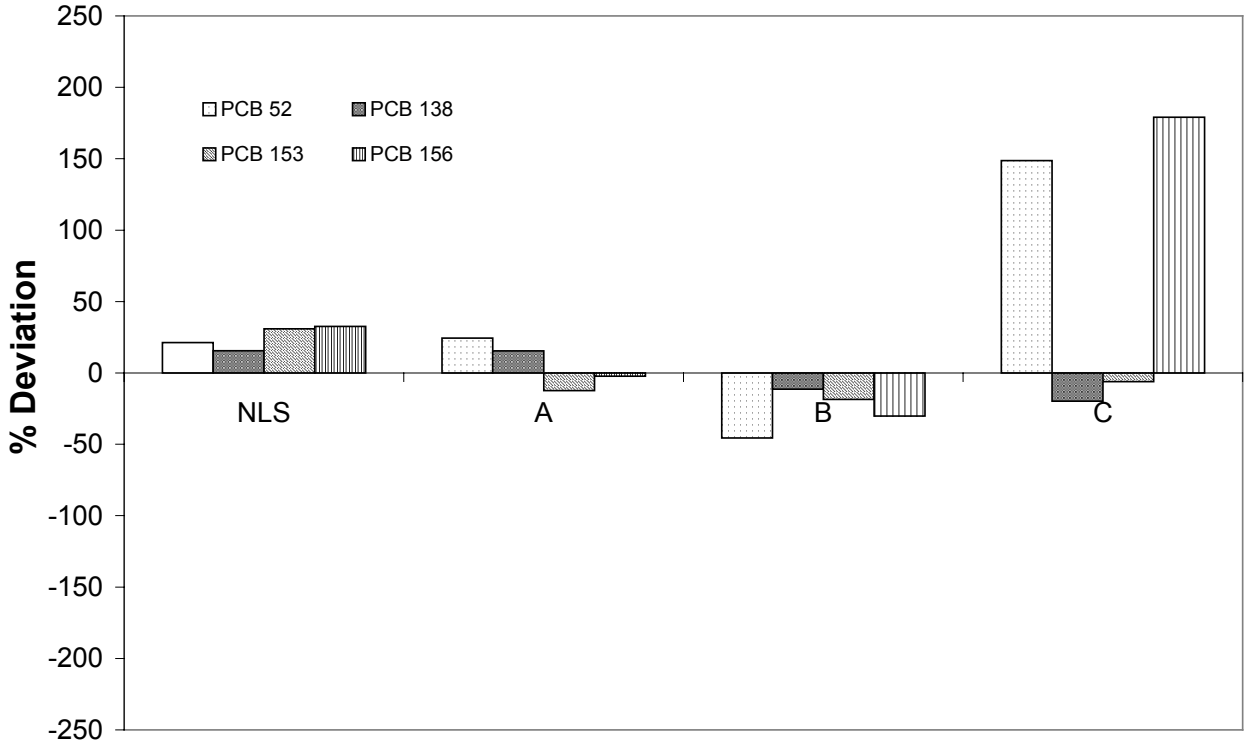

Figure 5.30 Comparison of laboratory performance for analysis of PCBs in Unknown Soil 1 (PCBs 52, 138, 153, 156)

Table 5.13 Analysis of PCBs in Unknown Soil 1 and statistical assessment of laboratory performance

		NLS			A			B			C		
Determinand	Mean	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category
PCB 18	*	0.0260	-0.707	a	< 0.0500	*	*	NR			1.09	0.700	a
PCB 28	0.0610	0.0430	-0.690	a	0.0900	1.14	a	< 0.100	*	*	0.0500	-0.426	a
PCB 31	*	0.0340	-0.696	a	0.0700	0.707	a	NR			NR		
PCB 47	*	0.0160	-0.705	a	0.0600	0.707	a	NR			< 0.200	*	*
PCB 49	0.179	0.0270	-0.669	a	0.0700	-0.480	a	NR			0.440	1.13	a
PCB 51	*	0.00500	*	*	< 0.0500	*	*	NR			< 0.460	*	*
PCB 52	0.129	0.156	0.482	a	0.160	0.616	a	0.0700	-1.11	a	< 0.320	*	*
PCB 77	*	0.0230	-0.674	a	0.0344	0.707	a	< 0.0500	*	*	< 0.300	*	*
PCB 81	*	< 0.00030	*	*	0.00240	*	*	< 0.0500	*	*	< 0.300	*	*
PCB 99	*	0.216	0.275	a	0.200	-0.707	a	NR			< 0.300	*	*
PCB 101	0.431	0.534	0.810	a	0.480	0.448	a	0.280	-1.23	a	0.430	-0.00852	a
PCB 105	0.184	0.211	0.799	a	0.180	-0.143	a	0.160	-0.577	a	< 0.200	*	*
PCB 114	*	0.0240	*	*	< 0.0500	*	*	< 0.0500	*	*	< 0.200	*	*
PCB 118	0.422	0.496	0.804	a	0.420	-0.0274	a	0.350	-0.712	a	< 0.280	*	*
PCB 123	*	0.0470	*	*	< 0.0500	*	*	< 0.0500	*	*	< 0.240	*	*
PCB 126	*	0.00700	-0.427	a	0.00790	0.707	a	< 0.0100	*	*	< 0.0400	*	*
PCB 128	*	0.159	0.563	a	0.120	-0.707	a	NR			< 0.400	*	*
PCB 138	0.710	0.821	0.621	a	0.820	0.847	a	0.630	-0.444	a	0.570	-0.990	a
PCB 153	0.639	0.837	0.904	a	0.560	-0.458	a	0.520	-0.590	a	< 0.600	*	*
PCB 156	0.0717	0.0950	0.903	a	0.0700	-0.0739	a	0.0500	-0.878	a	< 0.200	*	*
PCB 157	*	0.0200	*	*	< 0.0500	*	*	< 0.0500	*	*	< 0.140	*	*
PCB 167	*	0.0350	*	*	< 0.0500	*	*	< 0.0500	*	*	< 0.200	*	*
PCB 169	*	0.00400	0.672	a	0.00170	-0.707	a	< 0.0100	*	*	< 0.200	*	*
PCB 170	*	0.303	0.677	a	0.120	-0.707	a	NR			< 0.200	*	*
PCB 180	0.477	0.488	0.0286	a	0.220	-0.677	a	0.190	-0.752	a	1.01	1.36	a
PCB 189	*	0.00800	*	*	< 0.0500	*	*	< 0.0500	*	*	< 0.200	*	*

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.5.3 Analysis of Unknown Soil 2 for PCBs

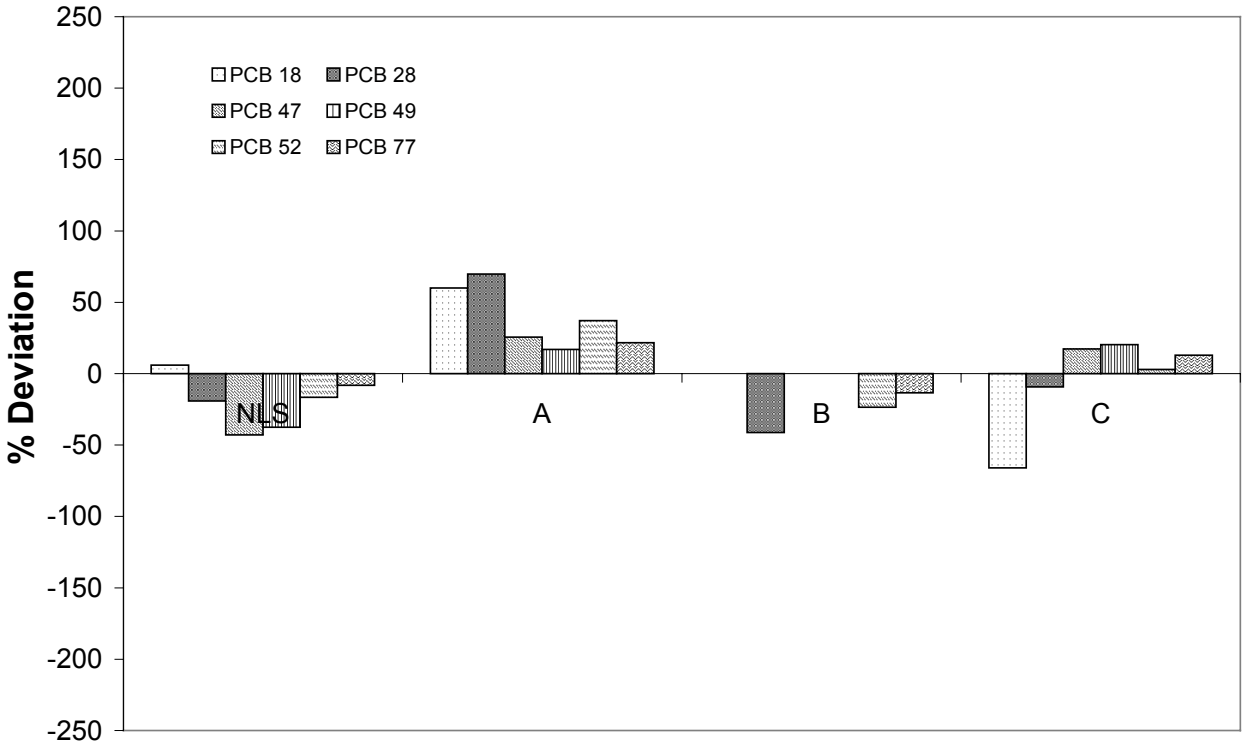

Figure 5.31 Comparison of laboratory performance for analysis of PCBs in Unknown Soil 2 (PCBs 18, 28, 47, 49, 52 and 77)

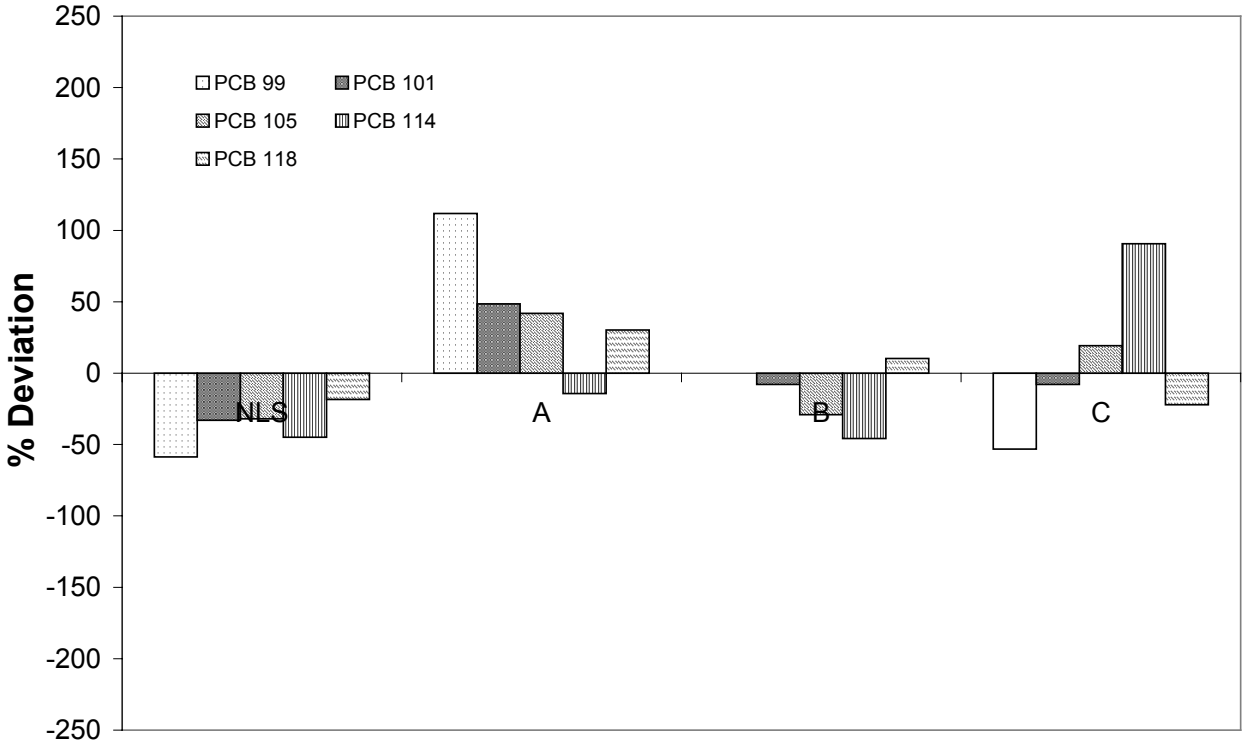

Figure 5.32 Comparison of laboratory performance for analysis of PCBs in Unknown Soil 2 (PCBs 99, 101, 105, 114 and 118)

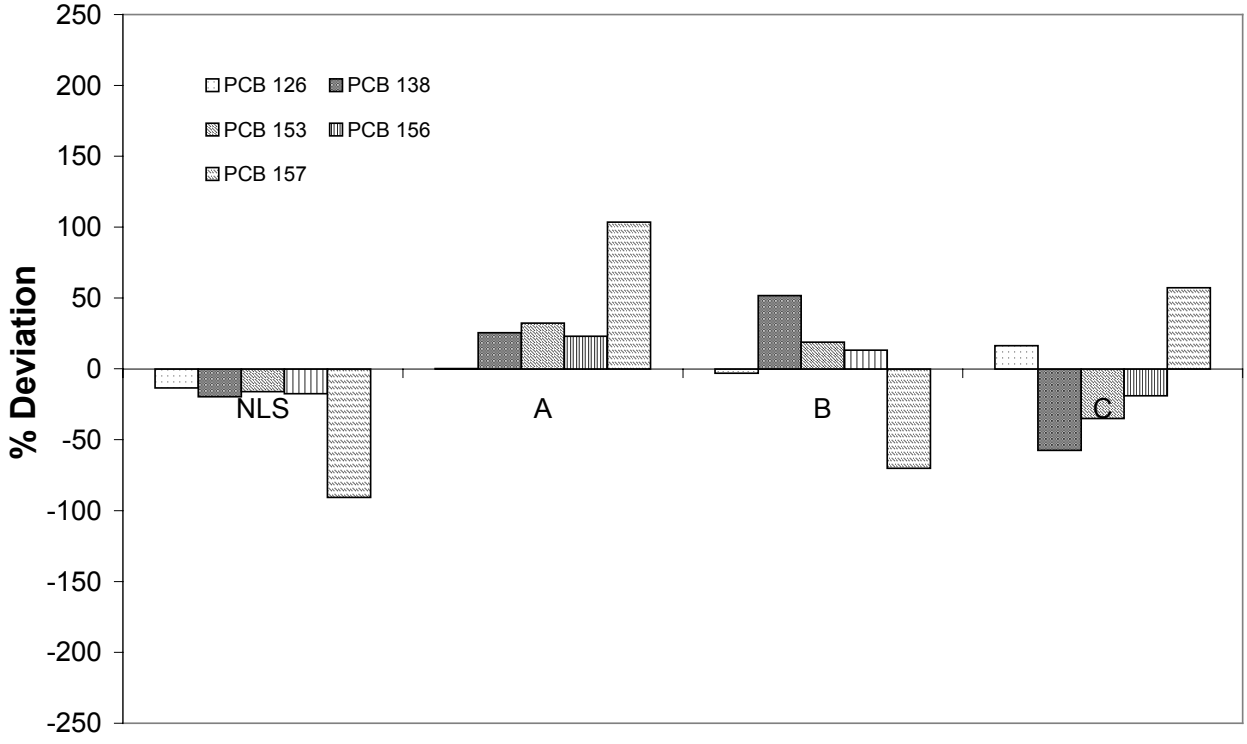

Figure 5.33 Comparison of laboratory performance for analysis of PCBs in Unknown Soil 2 (PCBs 126, 138, 153, 156 and 157)

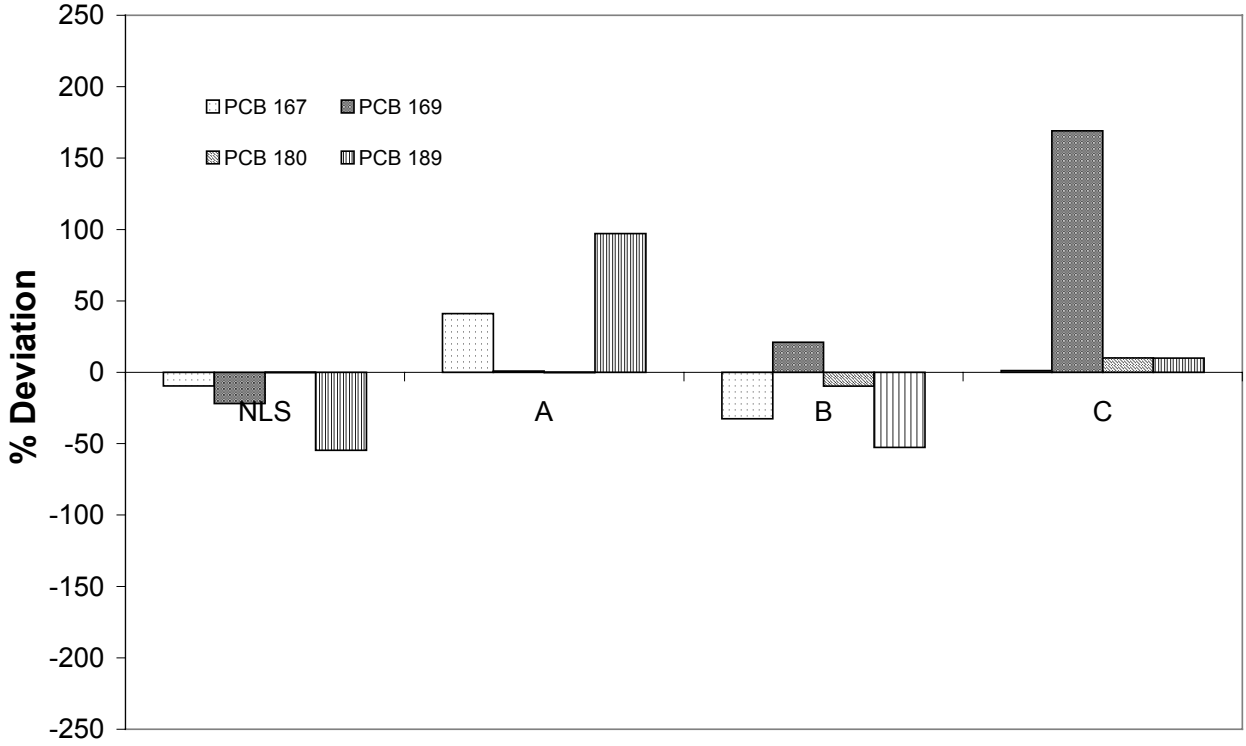

Figure 5.34 Comparison of laboratory performance for analysis of PCBs in Unknown Soil 2 (PCBs 167, 169, 180 and 189)

Table 5.14 Analysis of PCBs in Unknown Soil 2 and statistical assessment of laboratory performance

		NLS			A			B			C		
Determinand	Mean	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category
PCB 18	1.00	1.06	0.0920	a	1.60	0.949	a	NR			0.340	-1.04	a
PCB 28	2.72	2.20	-0.386	a	4.62	1.44	a	1.60	-0.829	a	2.47	-0.189	a
PCB 31	*	2.18	*	*	1.82	*	*	NR			NR		
PCB 47	0.605	0.346	-1.12	a	0.760	0.684	a	NR			0.710	0.442	a
PCB 49	0.598	0.374	-1.11	a	0.700	0.525	a	NR			0.720	0.589	a
PCB 51	*	0.0220	*	*	< 0.490	*	*	NR			< 0.460	*	*
PCB 52	1.28	1.07	-0.546	a	1.76	1.37	a	0.980	-0.755	a	1.32	0.100	a
PCB 77	0.266	0.244	-0.380	a	0.323	1.14	a	0.230	-0.524	a	< 0.300	*	*
PCB 81	*	0.106	-0.645	a	0.142	0.707	a	< 0.0500	*	*	< 0.300	*	*
PCB 99	1.39	0.574	-0.605	a	2.94	1.15	a	NR			0.650	-0.548	a
PCB 101	1.95	1.31	-0.929	a	2.90	1.41	a	1.80	-0.200	a	1.80	-0.219	a
PCB 105	1.28	0.873	-0.860	a	1.82	1.15	a	0.910	-0.744	a	1.53	0.501	a
PCB 114	0.572	0.315	-0.570	a	< 0.490	*	*	0.310	-0.577	a	1.09	1.12	a
PCB 118	2.81	2.29	-0.699	a	3.66	1.22	a	3.10	0.311	a	2.19	-0.848	a
PCB 123	*	0.0400	-0.707	a	< 0.490	*	*	< 0.0500	*	*	0.640	0.699	a
PCB 126	0.155	0.134	-0.832	a	0.155	0.0131	a	0.150	-0.134	a	0.180	0.963	a
PCB 128	*	0.458	-0.415	a	< 0.490	*	*	NR			0.520	0.456	a
PCB 138	2.97	2.38	-0.396	a	3.72	0.526	a	4.50	0.906	a	1.26	-1.18	a
PCB 153	2.53	2.12	-0.474	a	3.34	1.04	a	3.00	0.482	a	1.64	-1.11	a
PCB 156	0.715	0.590	-0.727	a	0.880	1.08	a	0.810	0.427	a	0.580	-0.826	a
PCB 157	0.737	0.0690	-0.953	a	1.50	1.09	a	0.220	-0.736	a	1.16	0.595	a
PCB 167	0.475	0.429	-0.293	a	0.670	1.33	a	0.320	-0.969	a	0.480	0.0341	a
PCB 169	0.0743	0.0580	-0.918	a	0.0750	0.0416	a	0.0900	0.650	a	< 0.200	*	*
PCB 170	*	1.19	-0.610	a	< 0.490	*	*	NR			1.56	0.607	a
PCB 180	1.66	1.66	-0.00983	a	1.66	-0.0186	a	1.50	-0.494	a	1.83	0.737	a
PCB 189	0.801	0.364	-0.761	a	1.58	1.36	a	0.380	-0.730	a	0.880	0.137	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.5.4 Analysis of Unknown Soil 3 for PCBs

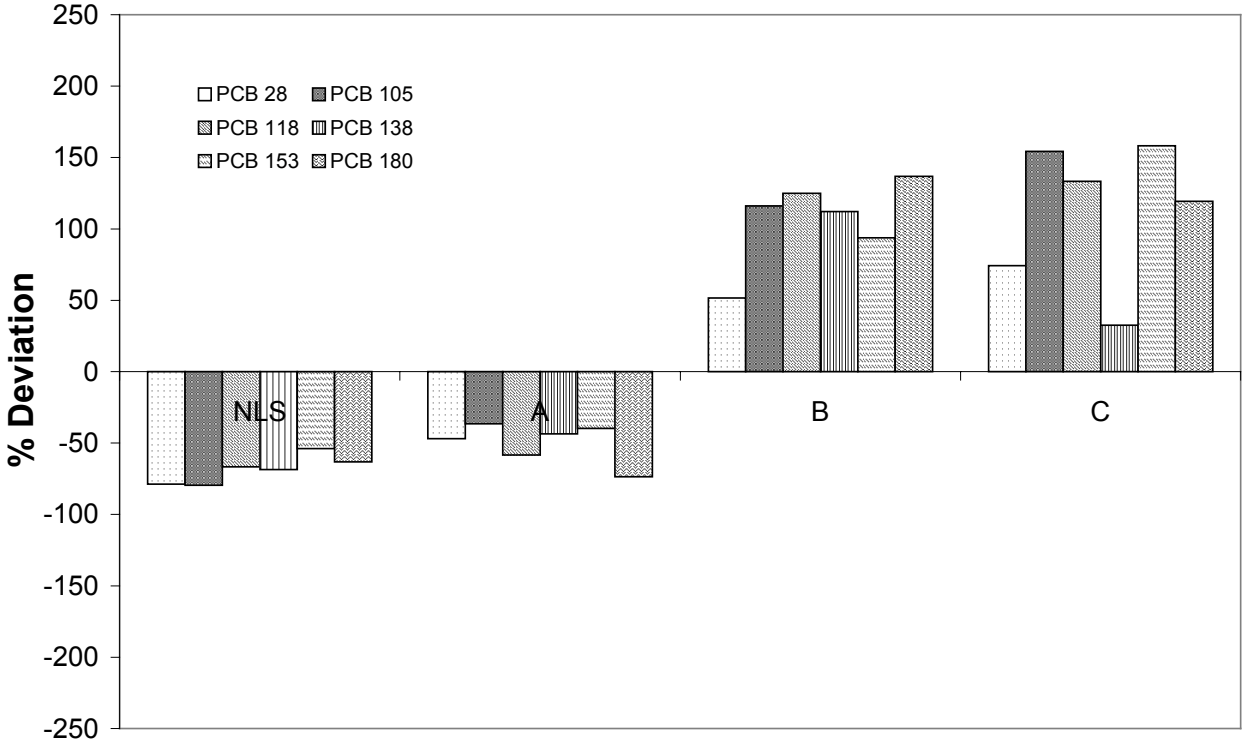

Figure 5.35 Comparison of laboratory performance for analysis of PCBs in Unknown Soil 3 (PCBs 28, 105, 118, 138, 153, 180)

Table 5.15 Analysis of PCBs in Unknown Soil 3 and statistical assessment of laboratory performance

		NLS			A			B			C		
Determinand	Mean	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category
PCB 18	*	0.0210	*	*	< 0.0500	*	*	NR			< 0.300	*	*
PCB 28	0.132	0.0280	-1.06	a	0.0700	-0.632	a	0.200	0.642	a	0.230	0.972	a
PCB 31	*	0.0190	*	*	< 0.0500	*	*	NR			NR		
PCB 47	*	0.0160	*	*	< 0.0500	*	*	NR			< 0.200	*	*
PCB 49	*	0.00600	*	*	< 0.0500	*	*	NR			< 0.200	*	*
PCB 51	*	0.00700	*	*	< 0.0500	*	*	NR			< 0.460	*	*
PCB 52	*	0.0100	*	*	< 0.0500	*	*	0.110	*	*	< 0.320	*	*
PCB 77	*	0.00400	*	*	0.00610	*	*	< 0.0500	*	*	< 0.300	*	*
PCB 81	*	< 0.00100	*	*	0.00040	*	*	< 0.0500	*	*	< 0.300	*	*
PCB 99	*	0.0200	*	*	< 0.0500	*	*	NR			< 0.300	*	*
PCB 101	*	0.0420	*	*	< 0.0500	*	*	0.110	*	*	< 0.300	*	*
PCB 105	0.0787	0.0160	-0.774	a	0.0500	-0.354	a	0.170	1.04	a	< 0.200	*	*
PCB 114	*	< 0.00200	*	*	< 0.0500	*	*	< 0.0500	*	*	< 0.200	*	*
PCB 118	0.120	0.0400	-0.615	a	0.0500	-0.538	a	0.270	1.07	a	< 0.280	*	*
PCB 123	*	0.00400	*	*	< 0.0500	*	*	< 0.0500	*	*	< 0.240	*	*
PCB 126	*	< 0.00400	*	*	0.00210	*	*	< 0.0100	*	*	< 0.0400	*	*
PCB 128	*	0.0200	*	*	< 0.0500	*	*	NR			< 0.400	*	*
PCB 138	0.302	0.0950	-0.699	a	0.170	-0.446	a	0.640	1.05	a	< 0.400	*	*
PCB 153	0.232	0.107	-0.660	a	0.140	-0.488	a	0.450	1.04	a	< 0.600	*	*
PCB 156	*	0.0160	-0.707	a	< 0.0500	*	*	0.110	0.671	a	< 0.200	*	*
PCB 157	*	< 0.00100	*	*	< 0.0500	*	*	< 0.0500	*	*	< 0.140	*	*
PCB 167	*	0.00900	*	*	< 0.0500	*	*	< 0.0500	*	*	< 0.200	*	*
PCB 169	*	< 0.00400	*	*	0.00070	*	*	< 0.0100	*	*	< 0.200	*	*
PCB 170	*	0.0730	*	*	< 0.0500	*	*	NR			< 0.200	*	*
PCB 180	0.228	0.0840	-0.532	a	0.0600	-0.621	a	0.540	1.07	a	< 0.500	*	*
PCB 189	*	< 0.00200	*	*	< 0.0500	*	*	< 0.0500	*	*	< 0.200	*	*

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.5.5 Analysis of herbage sample for PCBs

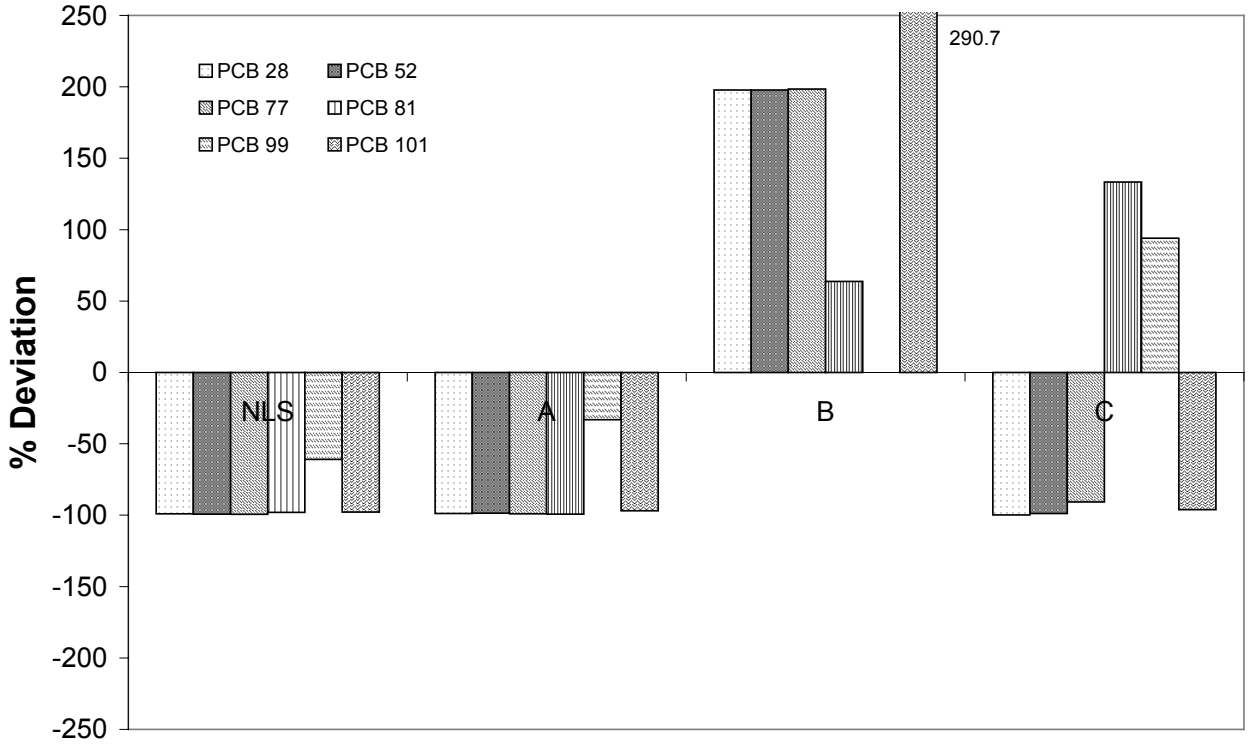

Figure 5.36 Comparison of laboratory performance for analysis of PCBs in herbage sample (PCBs 28, 52, 77, 81, 99 and 101)

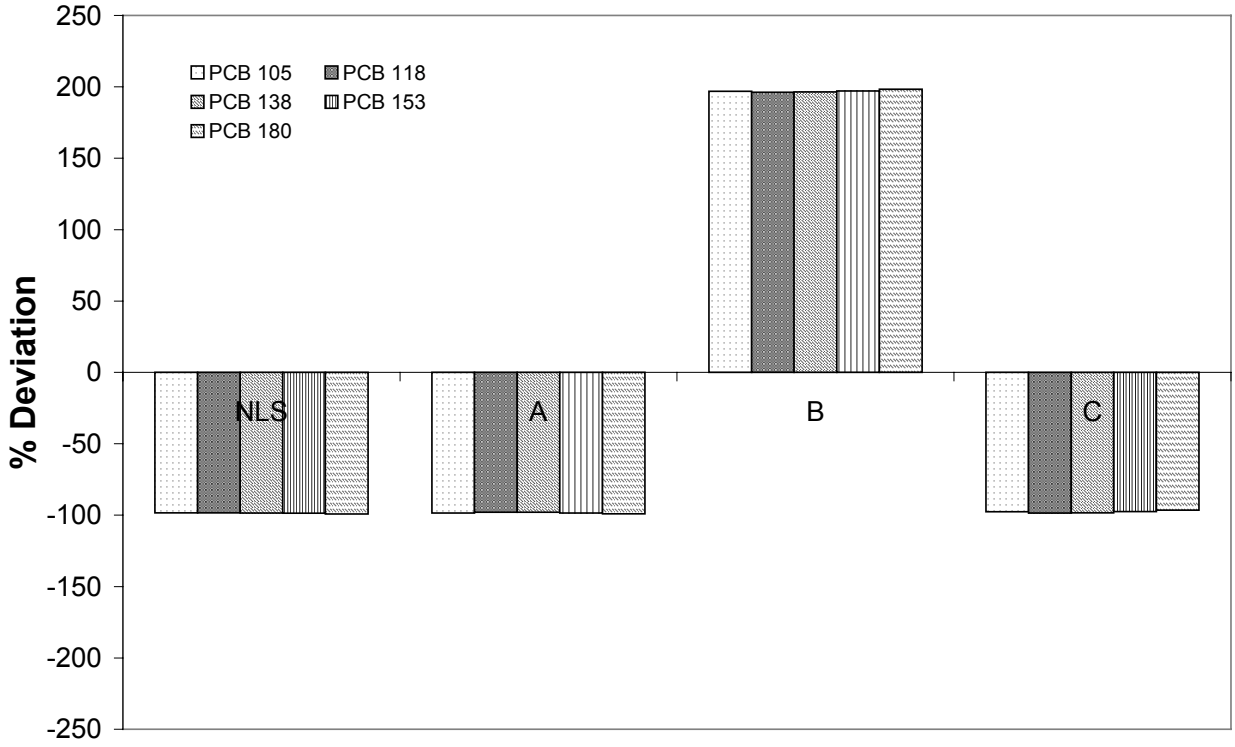

Figure 5.37 Comparison of laboratory performance for analysis of PCBs in herbage sample (PCBs 105, 118, 138, 153 and 180)

Table 5.16 Analysis of PCBs in herbage sample and statistical assessment of laboratory performance

		NLS			A			B			C		
Determinand	Mean	Results µgkg^{-1}	u-statistic	u-statistic category	Results µgkg^{-1}	u-statistic	u-statistic category	Results µgkg^{-1}	u-statistic	u-statistic category	Results µgkg^{-1}	u-statistic	u-statistic category
PCB 18	*	0.118	-0.686	a	0.220	0.707	a	NR			< 0.300	*	*
PCB 28	28.5	0.262	-0.578	a	0.360	-0.576	a	85.0	1.09	a	< 0.0400	*	*
PCB 31	*	0.244	-0.671	a	0.380	0.707	a	NR			NR		
PCB 47	*	0.0890	-0.681	a	0.160	0.707	a	NR			< 0.200	*	*
PCB 49	*	0.0800	-0.668	a	0.130	0.707	a	NR			< 0.200	*	*
PCB 51	*	0.0190	*	*	< 0.100	*	*	NR			< 0.460	*	*
PCB 52	25.5	0.207	-0.579	a	0.330	-0.576	a	76.0	1.09	a	< 0.320	*	*
PCB 77	3.25	0.0190	-0.578	a	0.0303	-0.576	a	9.70	1.09	a	< 0.300	*	*
PCB 81	0.201	0.00400	-0.835	a	0.00170	-0.845	a	0.330	0.524	a	0.470	1.11	a
PCB 99	0.299	0.117	-0.736	a	0.200	-0.401	a	NR			0.580	1.11	a
PCB 101	16.1	0.355	-0.505	a	0.510	-0.500	a	63.0	1.39	a	0.630	-0.496	a
PCB 105	8.42	0.137	-0.577	a	0.130	-0.578	a	25.0	1.09	a	< 0.200	*	*
PCB 114	*	0.0160	-0.707	a	< 0.100	*	*	2.00	0.680	a	< 0.200	*	*
PCB 118	17.9	0.289	-0.579	a	0.370	-0.576	a	53.0	1.09	a	< 0.280	*	*
PCB 123	*	0.0290	*	*	< 0.100	*	*	< 0.0500	*	*	< 0.240	*	*
PCB 126	*	< 0.0160	-0.658	a	0.00210	-0.707	a	0.400	0.680	a	< 0.0400	*	*
PCB 128	*	0.0620	*	*	< 0.100	*	*	NR			< 0.400	*	*
PCB 138	23.3	0.357	-0.579	a	0.490	-0.576	a	69.0	1.09	a	< 0.400	*	*
PCB 153	24.2	0.321	-0.578	a	0.380	-0.577	a	72.0	1.09	a	< 0.600	*	*
PCB 156	*	0.0470	-0.707	a	< 0.100	*	*	6.20	0.680	a	< 0.200	*	*
PCB 157	*	< 0.00500	*	*	< 0.100	*	*	1.40	*	*	< 0.140	*	*
PCB 167	*	0.0140	*	*	< 0.100	*	*	2.70	0.680	a	< 0.200	*	*
PCB 169	*	< 0.00200	-0.643	a	0.00020	-0.707	a	0.0400	0.680	a	< 0.200	*	*
PCB 170	*	0.0810	*	*	< 0.100	*	*	NR			< 0.200	*	*
PCB 180	14.1	0.107	-0.578	a	0.130	-0.577	a	42.0	1.09	a	< 0.500	*	*
PCB 189	*	< 0.00200	*	*	< 0.100	*	*	0.710	*	*	< 0.200	*	*

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.6 Dioxin results

Results for dioxins/furans are presented in Figures 5.38–5.51 and Tables 5.17–5.23.

The five laboratories reporting data are indicated by NLS, A, B, C and D.

5.6.1 Analysis of CRM of known dioxin and furan composition

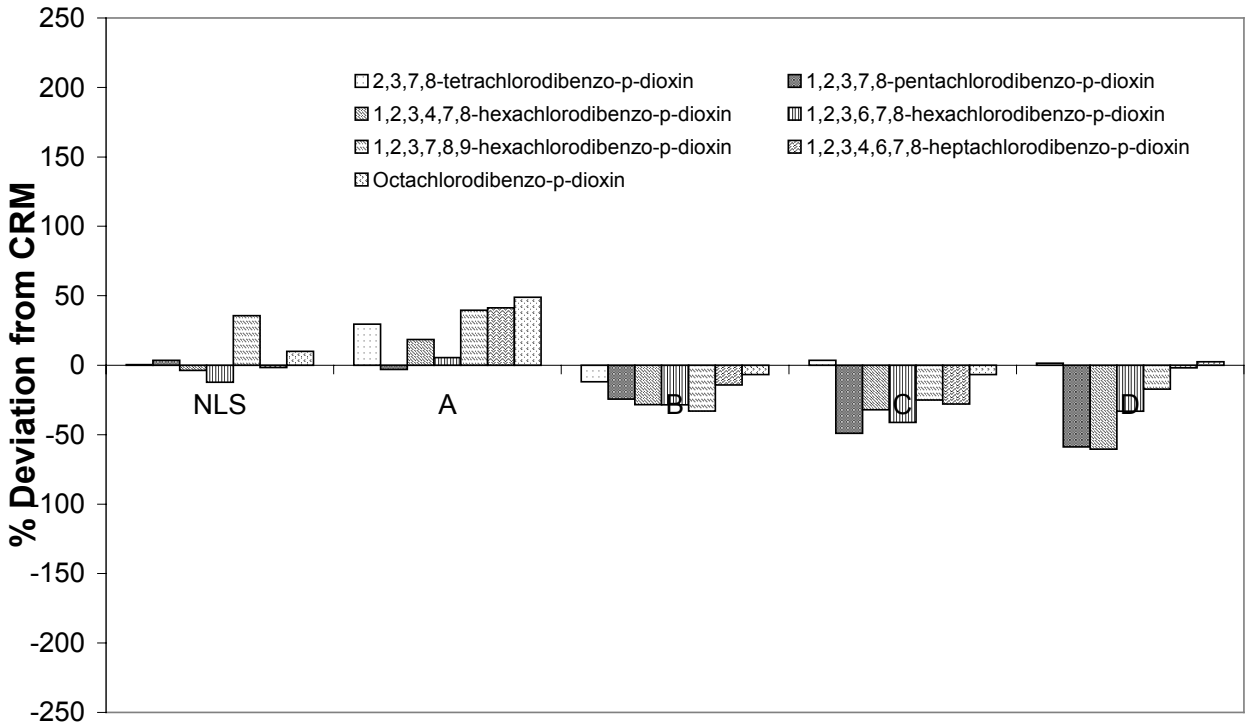

Figure 5.38 Comparison of laboratory performance for analysis of CRM (dioxins)

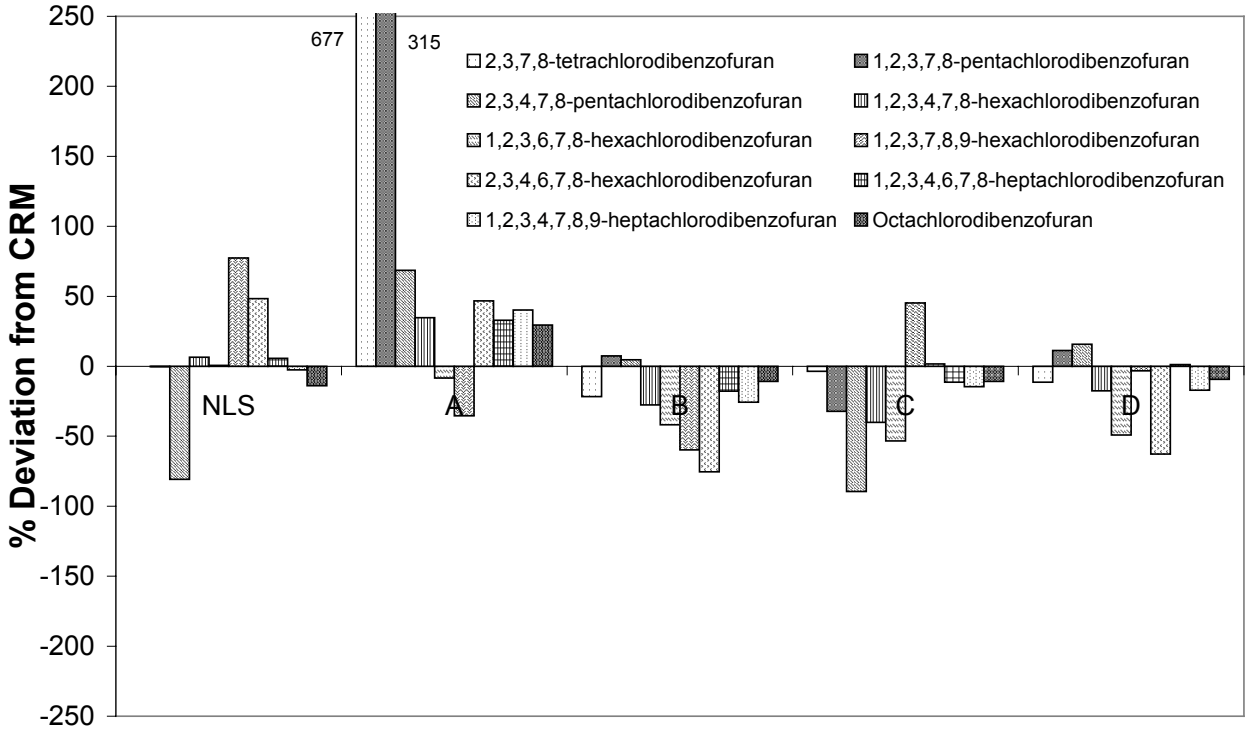

Figure 5.39 Comparison of laboratory performance for analysis of CRM (furans)

Table 5.17 Analysis of dioxin/furan CRM and statistical assessment of laboratory performance

		NLS			A			B			C			D		
Determinand	CRM	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category
2,3,7,8-tetrachlorodibenzo-p-dioxin#	0.0193	0.0194	0.0175	a	0.0250	1.06	a	0.0170	-0.310	a	0.0200	0.113	a	0.0196	0.0557	a
1,2,3,7,8-pentachlorodibenzo-p-dioxin	0.00608	0.00630	0.0886	a	0.00590	-0.0732	a	0.00460	-0.525	a	0.00310	-1.19	a	0.00250	-1.46	a
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin#	0.00810	0.00780	-0.270	a	0.00960	1.60	a	0.00580	-1.16	a	0.00550	-2.08	c	0.00320	-5.21	e
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin#	0.0238	0.0209	-0.281	a	0.0251	0.128	a	0.0170	-0.598	a	0.0140	-0.944	a	0.0159	-0.777	a
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin	0.0127	0.0172	0.841	a	0.0177	1.14	a	0.00850	-0.820	a	0.00950	-0.685	a	0.0105	-0.494	a
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin#	0.361	0.355	-0.0759	a	0.510	2.02	c	0.310	-0.429	a	0.260	-1.21	a	0.354	-0.0973	a
Octachlorodibenzo-p-dioxin#	2.57	2.83	0.528	a	3.83	3.70	e	2.40	-0.218	a	2.40	-0.351	a	2.64	0.191	a
2,3,7,8-tetrachlorodibenzofuran#	0.0166	0.0166	0	a	0.129	65.3	e	0.0130	-0.845	a	0.0160	-0.203	a	0.0147	-1.10	a
1,2,3,7,8-pentachlorodibenzofuran#	0.00782	0.00780	-0.00925	a	0.0325	11.6	e	0.00840	0.176	a	0.00530	-1.11	a	0.00870	0.415	a
2,3,4,7,8-pentachlorodibenzofuran#	0.0344	0.00660	-3.10	d	0.0580	2.64	d	0.0360	0.114	a	0.00360	-3.44	e	0.0398	0.603	a
1,2,3,4,7,8-hexachlorodibenzofuran#	0.0400	0.0426	0.491	a	0.0539	3.14	d	0.0290	-1.13	a	0.0240	-2.80	d	0.0330	-1.58	a
1,2,3,6,7,8-hexachlorodibenzofuran#	0.0165	0.0166	0.0129	a	0.0151	-0.183	a	0.00960	-0.846	a	0.00770	-1.14	a	0.00840	-1.06	a
1,2,3,7,8,9-hexachlorodibenzofuran#	0.00248	0.00440	0.954	a	0.00160	-0.442	a	0.00100	-0.735	a	0.00360	0.543	a	0.00240	-0.0402	a
2,3,4,6,7,8-hexachlorodibenzofuran#	0.0118	0.0175	2.62	d	0.0173	2.75	d	0.00290	-4.08	e	0.0120	0.0743	a	0.00440	-3.70	e
1,2,3,4,6,7,8-heptachlorodibenzofuran#	0.158	0.167	0.321	a	0.210	2.00	c	0.130	-0.597	a	0.140	-0.539	a	0.160	0.0692	a
1,2,3,4,7,8,9-heptachlorodibenzofuran#	0.0117	0.0114	-0.113	a	0.0164	1.87	b	0.00870	-0.828	a	0.0100	-0.581	a	0.00970	-0.797	a
Octachlorodibenzofuran#	0.258	0.222	-1.03	a	0.334	2.45	c	0.230	-0.370	a	0.230	-0.604	a	0.234	-0.781	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.
# A CRM value is available for this determinand.
Non # values are calculated using the approach described in Section 4.2.

5.6.2 Analysis of Unknown Soil 1 for dioxins/furans

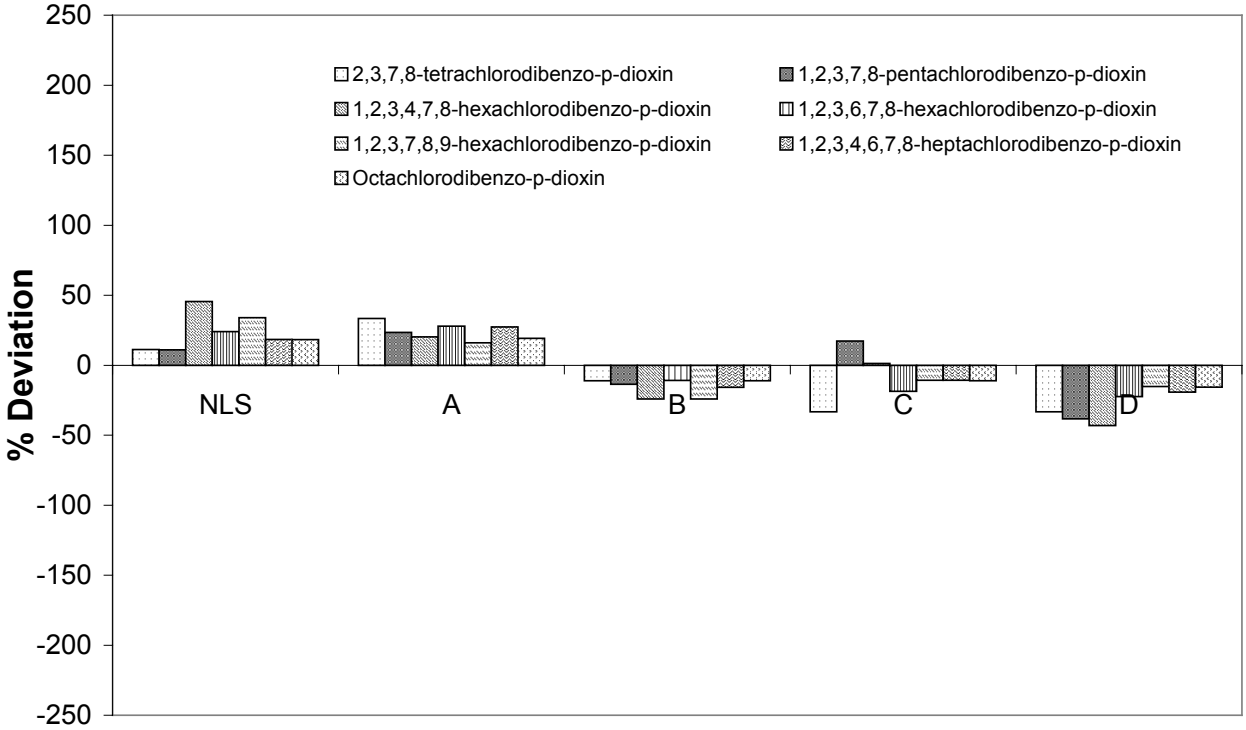

Figure 5.40 Comparison of laboratory performance for analysis of dioxins in Unknown Soil 1

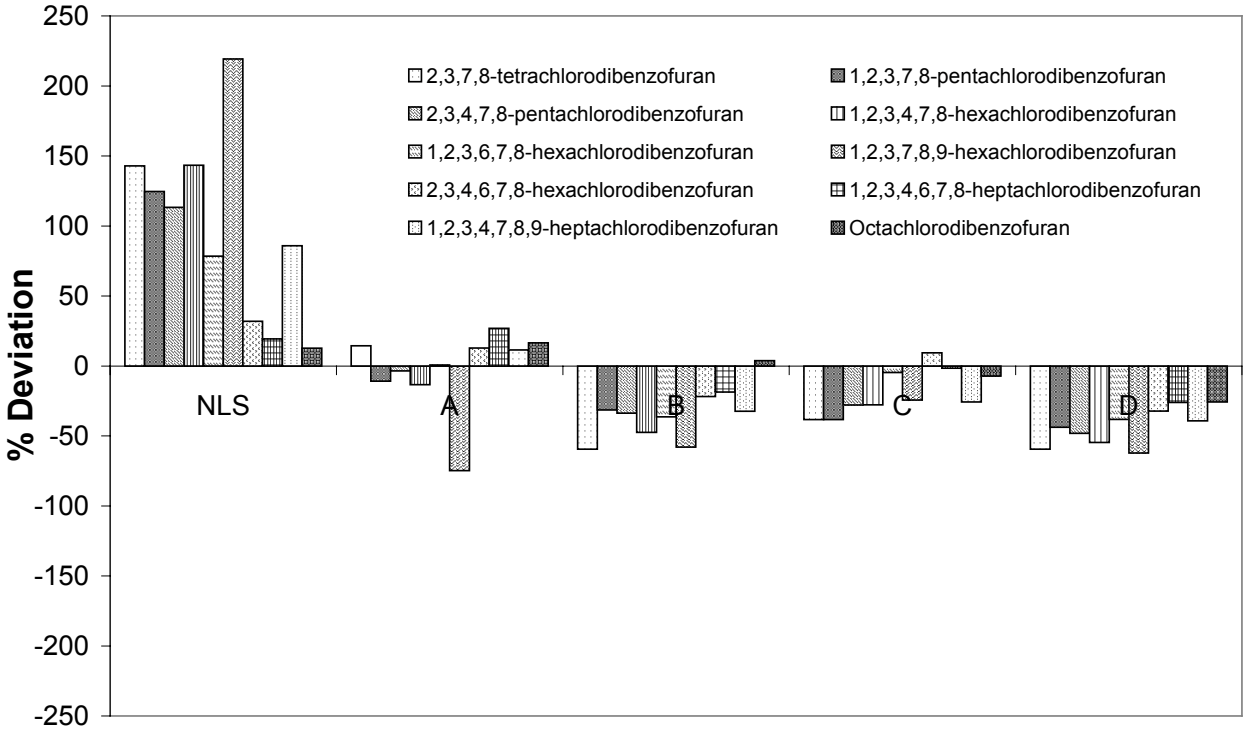

Figure 5.41 Comparison of laboratory performance for analysis of furans in Unknown Soil 1

Table 5.18 Analysis of dioxins and furans in Unknown Soil 1 and statistical assessment of laboratory performance

		NLS			A			B			C			D		
Determinand	Mean	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category
2,3,7,8-tetrachlorodibenzo-p-dioxin	0.000450	0.000500	0.363	a	0.000600	1.16	a	0.000400	-0.284	a	< 0.000300	*	*	0.000300	-1.16	a
1,2,3,7,8-pentachlorodibenzo-p-dioxin	0.00162	0.00180	0.423	a	0.00200	0.916	a	0.00140	-0.373	a	0.00190	0.556	a	0.00100	-1.49	a
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin	0.00158	0.00230	1.24	a	0.00190	0.578	a	0.00120	-0.575	a	0.00160	0.0331	a	0.000900	-1.23	a
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin	0.00258	0.00320	0.922	a	0.00330	1.16	a	0.00230	-0.301	a	0.00210	-0.688	a	0.00200	-0.932	a
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin	0.00224	0.00300	1.000	a	0.00260	0.665	a	0.00170	-0.726	a	0.00200	-0.388	a	0.00190	-0.628	a
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin	0.0202	0.0239	0.793	a	0.0257	1.28	a	0.0170	-0.476	a	0.0180	-0.429	a	0.0163	-0.901	a
Octachlorodibenzo-p-dioxin	0.0719	0.0851	0.816	a	0.0857	1.11	a	0.0640	-0.346	a	0.0640	-0.504	a	0.0607	-0.903	a
2,3,7,8-tetrachlorodibenzofuran	0.00988	0.0240	1.62	a	0.0113	0.168	a	0.00400	-0.690	a	0.00610	-0.445	a	0.00400	-0.697	a
1,2,3,7,8-pentachlorodibenzofuran	0.00730	0.0164	1.73	b	0.00650	-0.155	a	0.00500	-0.427	a	0.00450	-0.537	a	0.00410	-0.619	a
2,3,4,7,8-pentachlorodibenzofuran	0.00694	0.0148	1.71	b	0.00670	-0.0529	a	0.00460	-0.494	a	0.00500	-0.422	a	0.00360	-0.737	a
1,2,3,4,7,8-hexachlorodibenzofuran	0.00970	0.0236	1.72	b	0.00840	-0.164	a	0.00510	-0.570	a	0.00700	-0.338	a	0.00440	-0.668	a
1,2,3,6,7,8-hexachlorodibenzofuran	0.00566	0.0101	1.60	a	0.00570	0.0149	a	0.00360	-0.713	a	0.00540	-0.0929	a	0.00350	-0.806	a
1,2,3,7,8,9-hexachlorodibenzofuran	0.00238	0.00760	1.74	b	0.000600	-0.603	a	0.00100	-0.465	a	0.00180	-0.196	a	0.000900	-0.501	a
2,3,4,6,7,8-hexachlorodibenzofuran	0.00576	0.00760	1.17	a	0.00650	0.486	a	0.00450	-0.619	a	0.00630	0.301	a	0.00390	-1.22	a
1,2,3,4,6,7,8-heptachlorodibenzofuran	0.0295	0.0352	0.800	a	0.0374	1.17	a	0.0240	-0.554	a	0.0290	-0.0595	a	0.0218	-1.13	a
1,2,3,4,7,8,9-heptachlorodibenzofuran	0.00296	0.00550	1.60	a	0.00330	0.222	a	0.00200	-0.583	a	0.00220	-0.484	a	0.00180	-0.756	a
Octachlorodibenzofuran	0.0366	0.0413	0.671	a	0.0427	0.969	a	0.0380	0.105	a	0.0340	-0.327	a	0.0272	-1.51	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.6.3 Analysis of Unknown Soil 2 for dioxins/furans

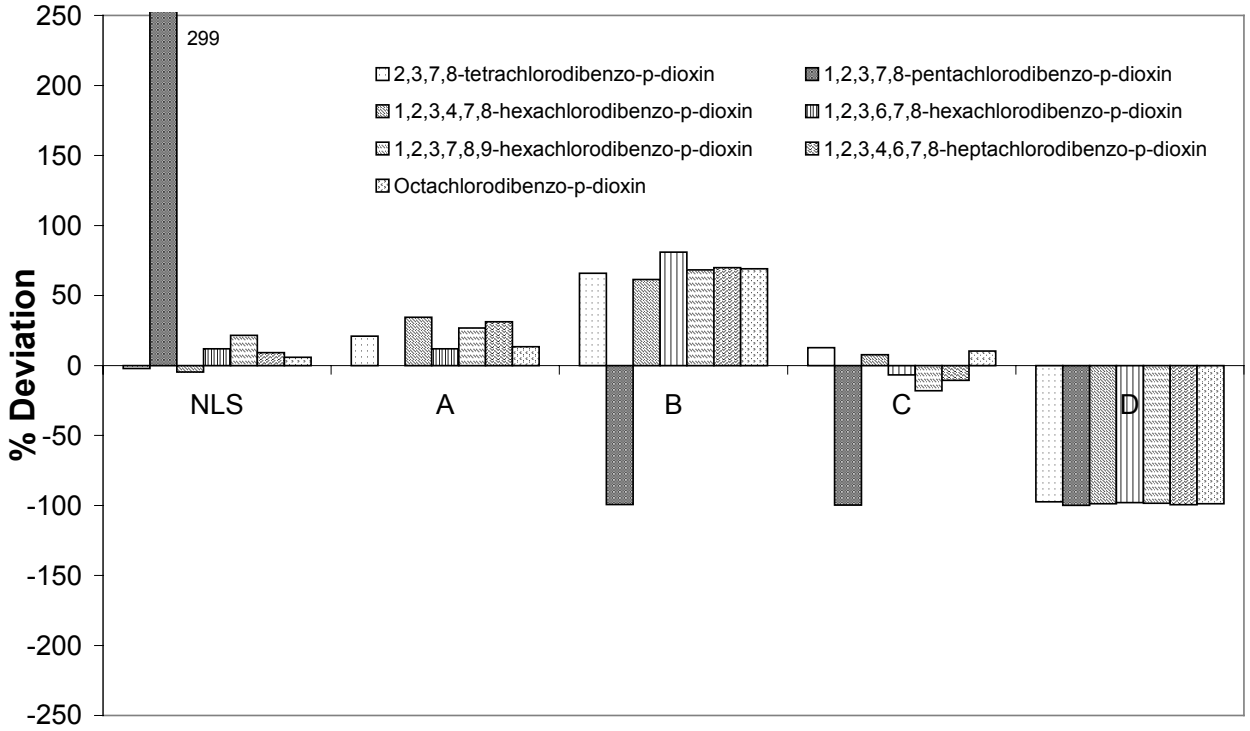

Figure 5.42 Comparison of laboratory performance for analysis of dioxins in Unknown Soil 2

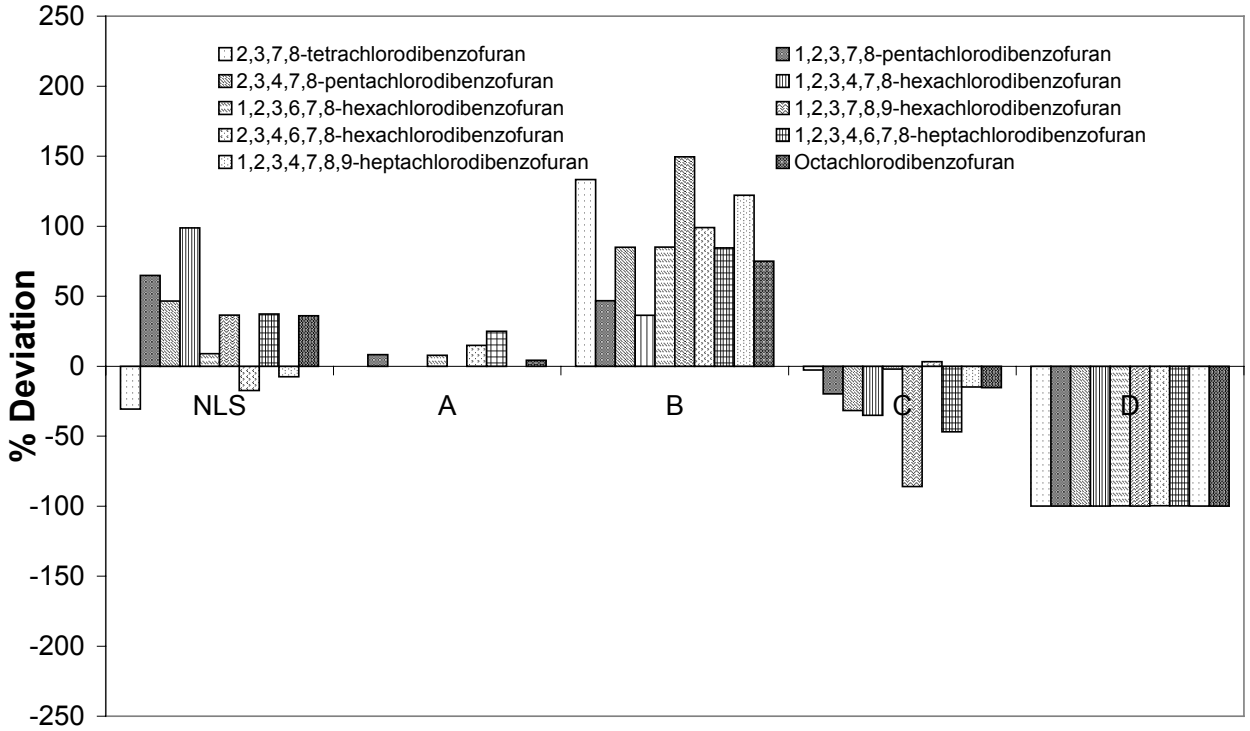

Figure 5.43 Comparison of laboratory performance for analysis of furans in Unknown Soil 2

Table 5.19 Analysis of dioxins and furans in Unknown Soil 2 and statistical assessment of laboratory performance

		NLS			A			B			C			D		
Determinand	Mean	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category
2,3,7,8-tetrachlorodibenzo-p-dioxin	0.0603	0.0590	-0.0360	a	0.0730	0.350	a	0.100	0.844	a	0.0680	0.204	a	0.00160	-1.62	a
1,2,3,7,8-pentachlorodibenzo-p-dioxin	61.1	244	1.49	a	NR			0.380	-0.498	a	0.200	-0.500	a	0.00420	-0.502	a
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin	0.205	0.195	-0.0760	a	0.275	0.567	a	0.330	0.790	a	0.220	0.120	a	0.00260	-1.62	a
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin	0.365	0.408	0.183	a	0.408	0.185	a	0.660	0.963	a	0.340	-0.103	a	0.00710	-1.53	a
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin	0.232	0.282	0.324	a	0.294	0.425	a	0.390	0.845	a	0.190	-0.282	a	0.00350	-1.56	a
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin	2.24	2.44	0.142	a	2.94	0.494	a	3.80	0.861	a	2.00	-0.164	a	0.0115	-1.58	a
Octachlorodibenzo-p-dioxin	4.08	4.32	0.0948	a	4.62	0.219	a	6.90	0.872	a	4.50	0.163	a	0.0525	-1.62	a
2,3,7,8-tetrachlorodibenzofuran	15.4	10.7	-0.313	a	NR			36.0	1.11	a	15.0	-0.0282	a	0.0195	-1.02	a
1,2,3,7,8-pentachlorodibenzofuran	11.6	19.1	0.990	a	12.5	0.126	a	17.0	0.596	a	9.30	-0.299	a	0.00660	-1.54	a
2,3,4,7,8-pentachlorodibenzofuran	7.03	10.3	0.563	a	NR			13.0	0.855	a	4.80	-0.382	a	0.00750	-1.21	a
1,2,3,4,7,8-hexachlorodibenzofuran	15.4	30.6	1.13	a	NR			21.0	0.381	a	10.0	-0.404	a	0.00690	-1.16	a
1,2,3,6,7,8-hexachlorodibenzofuran	3.67	4.00	0.134	a	3.96	0.119	a	6.80	0.988	a	3.60	-0.0296	a	0.00620	-1.52	a
1,2,3,7,8,9-hexachlorodibenzofuran	2.73	3.72	0.311	a	NR			6.80	1.08	a	0.380	-0.735	a	0.000300	-0.854	a
2,3,4,6,7,8-hexachlorodibenzofuran	1.36	1.12	-0.245	a	1.56	0.209	a	2.70	1.07	a	1.40	0.0437	a	0.00490	-1.40	a
1,2,3,4,6,7,8-heptachlorodibenzofuran	8.67	11.9	0.506	a	10.8	0.343	a	16.0	0.922	a	4.60	-0.639	a	0.0147	-1.37	a
1,2,3,4,7,8,9-heptachlorodibenzofuran	2.70	2.50	-0.0807	a	NR			6.00	1.08	a	2.30	-0.160	a	0.00100	-1.09	a
Octachlorodibenzofuran	17.7	24.1	0.544	a	18.5	0.0635	a	31.0	0.894	a	15.0	-0.230	a	0.0132	-1.53	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.6.4 Analysis of Unknown Soil 3 for dioxins/furans

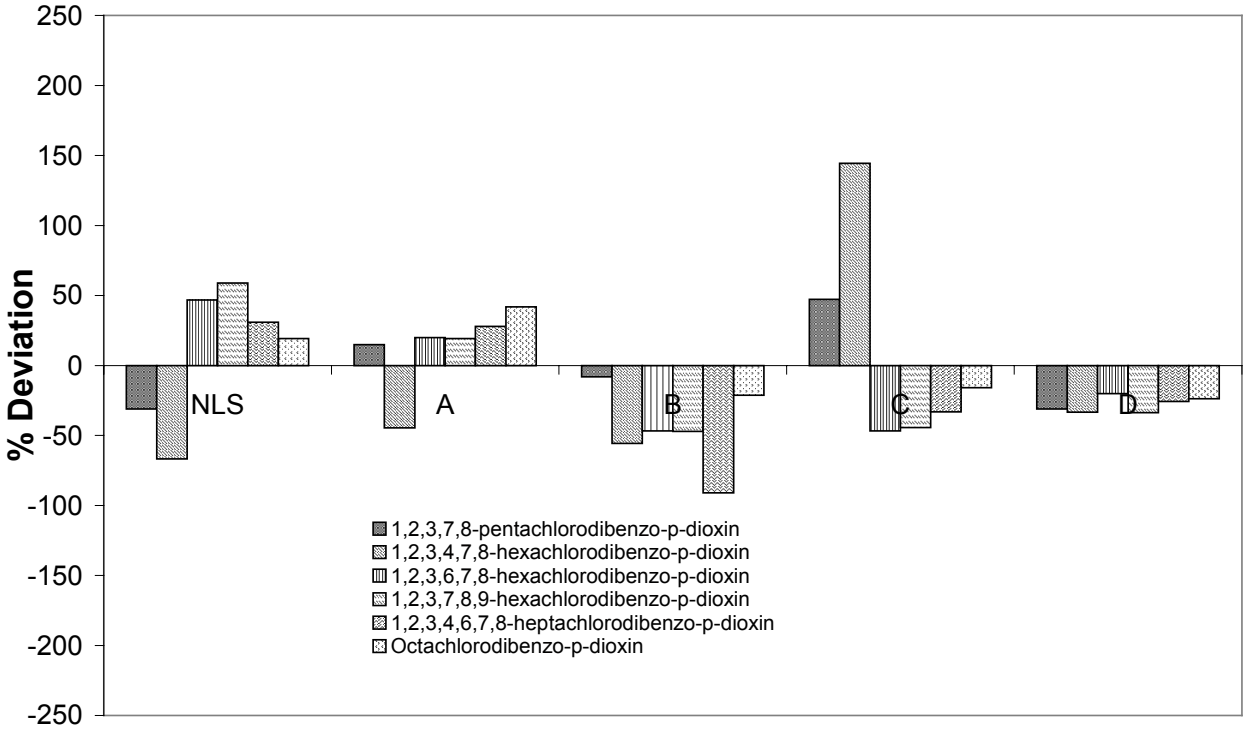

Figure 5.44 Comparison of laboratory performance for analysis of dioxins in Unknown Soil 3

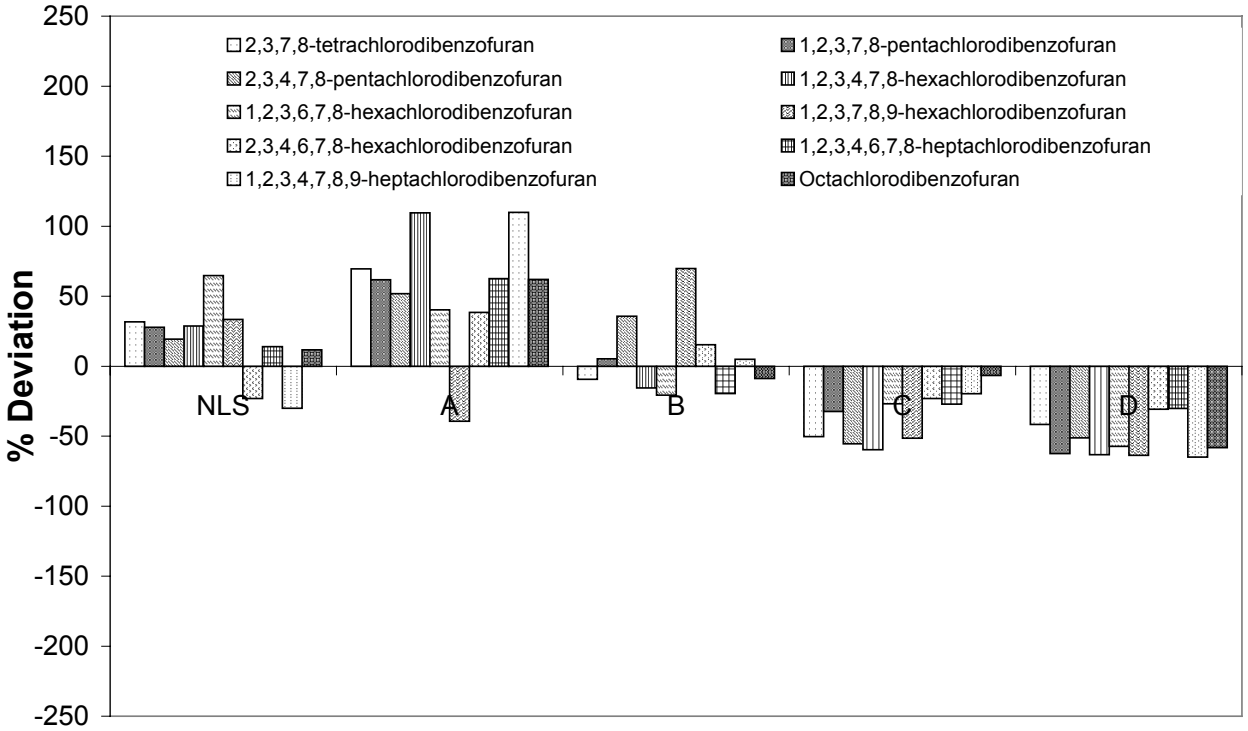

Figure 5.45 Comparison of laboratory performance for analysis of furans in Unknown Soil 3

Table 5.20 Analysis of dioxins and furans in Unknown Soil 3 and statistical assessment of laboratory performance

		NLS			A			B			C			D		
Determinand	Mean	Results µgkg^{-1}	u-statistic	u-statistic category	Results µgkg^{-1}	u-statistic	u-statistic category	Results µgkg^{-1}	u-statistic	u-statistic category	Results µgkg^{-1}	u-statistic	u-statistic category	Results µgkg^{-1}	u-statistic	u-statistic category
2,3,7,8-tetrachlorodibenzo-p-dioxin	*	0.000200	*	*	0.000200	*	*	< 0.000200	*	*	< 0.000300	*	*	NR		
1,2,3,7,8-pentachlorodibenzo-p-dioxin	0.000435	0.000300	-0.810	a	0.000500	0.391	a	< 0.000400	*	*	0.000640	1.07	a	0.000300	-0.813	a
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin	0.000900	0.000300	-0.685	a	0.000500	-0.457	a	< 0.000400	*	*	0.00220	1.39	a	0.000600	-0.343	a
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin	0.000750	0.00110	1.08	a	0.000900	0.482	a	< 0.000400	*	*	0.000400	-1.11	a	0.000600	-0.482	a
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin	0.000755	0.00120	1.06	a	0.000900	0.399	a	< 0.000400	*	*	0.000420	-0.908	a	0.000500	-0.702	a
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin	0.00673	0.00880	0.868	a	0.00860	0.818	a	< 0.000600	*	*	0.00450	-0.932	a	0.00500	-0.753	a
Octachlorodibenzo-p-dioxin	0.0190	0.0227	0.591	a	0.0270	1.44	a	0.0150	-0.566	a	0.0160	-0.503	a	0.0145	-0.819	a
2,3,7,8-tetrachlorodibenzofuran	0.00342	0.00450	0.610	a	0.00580	1.38	a	0.00310	-0.163	a	0.00170	-0.986	a	0.00200	-0.823	a
1,2,3,7,8-pentachlorodibenzofuran	0.00266	0.00340	0.564	a	0.00430	1.26	a	0.00280	0.0905	a	0.00180	-0.648	a	0.00100	-1.28	a
2,3,4,7,8-pentachlorodibenzofuran	0.00184	0.00220	0.383	a	0.00280	1.04	a	0.00250	0.551	a	0.000820	-1.10	a	0.000900	-1.02	a
1,2,3,4,7,8-hexachlorodibenzofuran	0.00272	0.00350	0.397	a	0.00570	1.53	a	0.00230	-0.203	a	0.00110	-0.827	a	0.00100	-0.881	a
1,2,3,6,7,8-hexachlorodibenzofuran	0.00164	0.00270	1.24	a	0.00230	0.796	a	0.00130	-0.371	a	0.00120	-0.518	a	0.000700	-1.13	a
1,2,3,7,8,9-hexachlorodibenzofuran	0.000825	0.00110	0.531	a	0.000500	-0.634	a	0.00140	0.868	a	< 0.000400	*	*	0.000300	-1.02	a
2,3,4,6,7,8-hexachlorodibenzofuran	0.00130	0.00100	-0.702	a	0.00180	1.18	a	0.00150	0.323	a	< 0.00100	*	*	0.000900	-0.943	a
1,2,3,4,6,7,8-heptachlorodibenzofuran	0.00658	0.00750	0.351	a	0.0107	1.60	a	0.00530	-0.423	a	0.00480	-0.665	a	0.00460	-0.769	a
1,2,3,4,7,8,9-heptachlorodibenzofuran	0.000858	0.000600	-0.452	a	0.00180	1.66	b	0.000900	0.0667	a	0.000690	-0.290	a	0.000300	-0.981	a
Octachlorodibenzofuran	0.00932	0.0104	0.263	a	0.0151	1.43	a	0.00850	-0.172	a	0.00870	-0.146	a	0.00390	-1.34	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.6.5 Analysis of the herbage sample for dioxins/furans

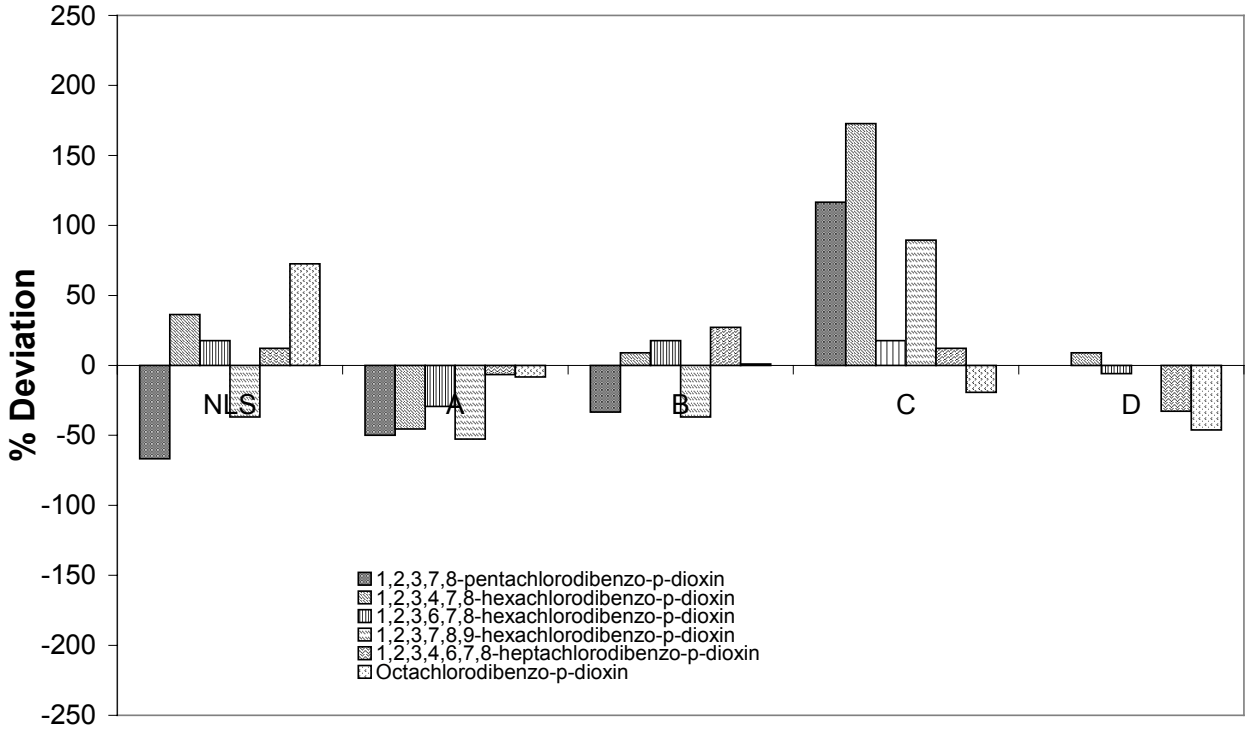

Figure 5.46 Comparison of laboratory performance for analysis of dioxins in herbage sample

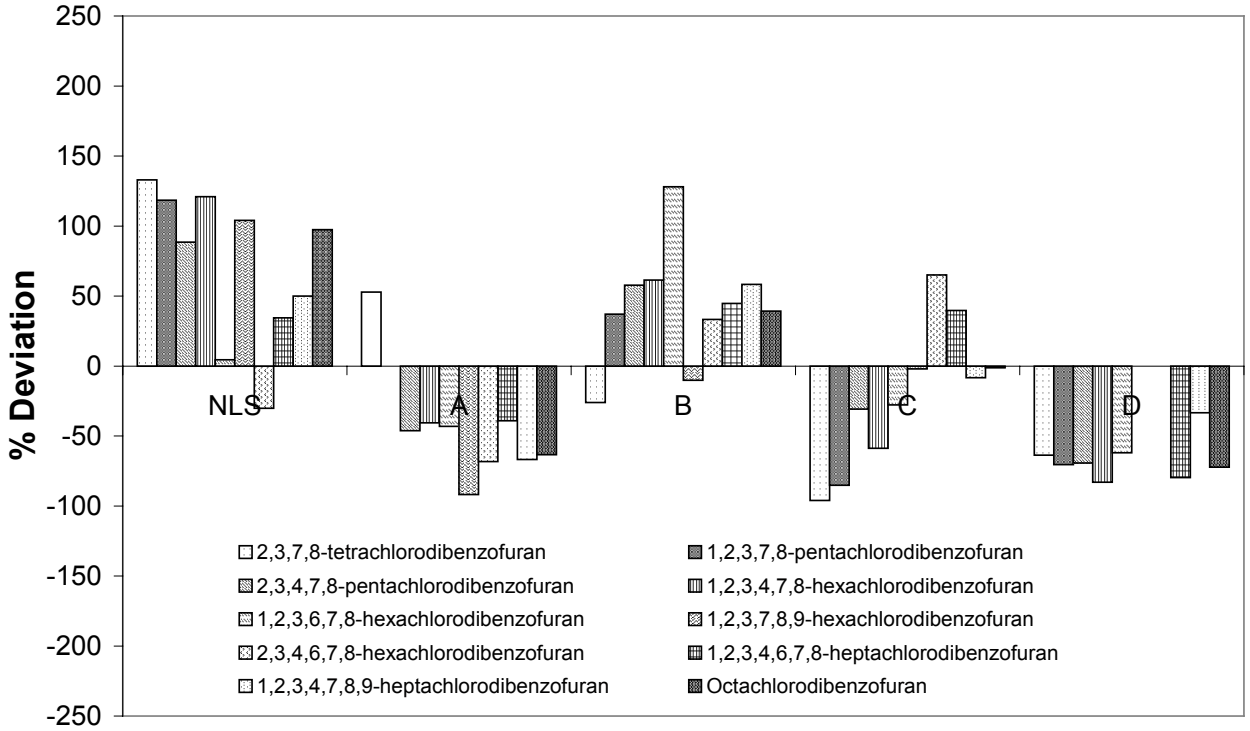

Figure 5.47 Comparison of laboratory performance for analysis of furans in herbage sample

Table 5.21 Analysis of dioxins and furans in herbage sample and statistical assessment of laboratory performance

		NLS			A			B			C			D		
Determinand	Mean	Results µgkg^{-1}	u-statistic	u-statistic category	Results µgkg^{-1}	u-statistic	u-statistic category	Results µgkg^{-1}	u-statistic	u-statistic category	Results µgkg^{-1}	u-statistic	u-statistic category	Results µgkg^{-1}	u-statistic	u-statistic category
2,3,7,8-tetrachlorodibenzo-p-dioxin	*	0.000400	0.696	a	0.000100	-0.707	a	< 0.000200	*	*	< 0.000800	*	*	NR		
1,2,3,7,8-pentachlorodibenzo-p-dioxin	0.000600	0.000200	-0.657	a	0.000300	-0.493	a	< 0.000400	*	*	0.00130	1.10	a	NR		
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin	0.000367	0.000500	0.847	a	0.000200	-1.09	a	< 0.000400	*	*	< 0.00100	*	*	0.000400	0.218	a
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin	0.000425	0.000500	0.723	a	0.000300	-1.31	a	0.000500	0.421	a	< 0.000500	*	*	0.000400	-0.261	a
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin	0.000633	0.000400	-0.468	a	0.000300	-0.676	a	< 0.000400	*	*	0.00120	1.08	a	NR		
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin	0.00268	0.00300	0.446	a	0.00250	-0.254	a	0.00340	0.589	a	< 0.00300	*	*	0.00180	-1.27	a
Octachlorodibenzo-p-dioxin	0.0119	0.0205	1.48	a	0.0109	-0.186	a	0.0120	0.0188	a	0.00960	-0.418	a	0.00640	-1.04	a
2,3,7,8-tetrachlorodibenzofuran	0.00824	0.0192	1.40	a	0.0126	0.570	a	0.00610	-0.273	a	0.000320	-1.04	a	0.00300	-0.686	a
1,2,3,7,8-pentachlorodibenzofuran	0.00270	0.00590	1.41	a	0.00270	*	*	0.00370	0.399	a	0.000400	-1.02	a	0.000800	-0.847	a
2,3,4,7,8-pentachlorodibenzofuran	0.00260	0.00490	1.27	a	0.00140	-0.669	a	0.00410	0.690	a	0.00180	-0.441	a	0.000800	-1.00	a
1,2,3,4,7,8-hexachlorodibenzofuran	0.00235	0.00520	1.37	a	0.00140	-0.465	a	0.00380	0.616	a	0.000970	-0.673	a	0.000400	-0.952	a
1,2,3,6,7,8-hexachlorodibenzofuran	0.00105	0.00110	0.0600	a	0.000600	-0.568	a	0.00240	1.26	a	0.000760	-0.363	a	0.000400	-0.819	a
1,2,3,7,8,9-hexachlorodibenzofuran	0.00123	0.00250	1.28	a	0.000100	-1.14	a	0.00110	-0.120	a	0.00120	-0.0250	a	NR		
2,3,4,6,7,8-hexachlorodibenzofuran	0.00158	0.00110	-0.499	a	0.000500	-1.13	a	0.00210	0.461	a	0.00260	0.998	a	NR		
1,2,3,4,6,7,8-heptachlorodibenzofuran	0.00394	0.00530	0.607	a	0.00240	-0.695	a	0.00570	0.629	a	0.00550	0.660	a	0.000800	-1.42	a
1,2,3,4,7,8,9-heptachlorodibenzofuran	0.00120	0.00180	0.911	a	0.000400	-1.24	a	0.00190	0.814	a	0.00110	-0.150	a	0.000800	-0.621	a
Octachlorodibenzofuran	0.00790	0.0156	1.34	a	0.00290	-0.889	a	0.0110	0.476	a	0.00780	-0.0174	a	0.00220	-1.01	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.6.6 Analysis of flyash CRM for dioxins/furans

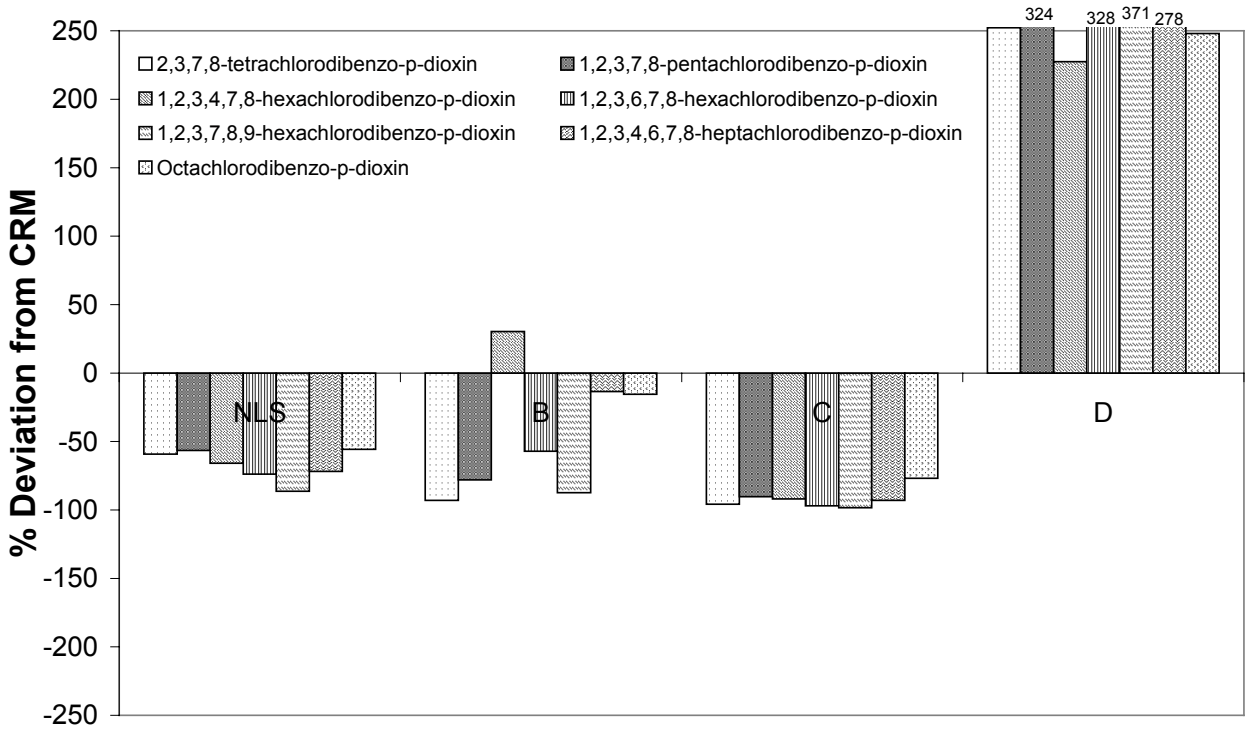

Figure 5.48 Comparison of laboratory performance for analysis of dioxins in flyash CRM

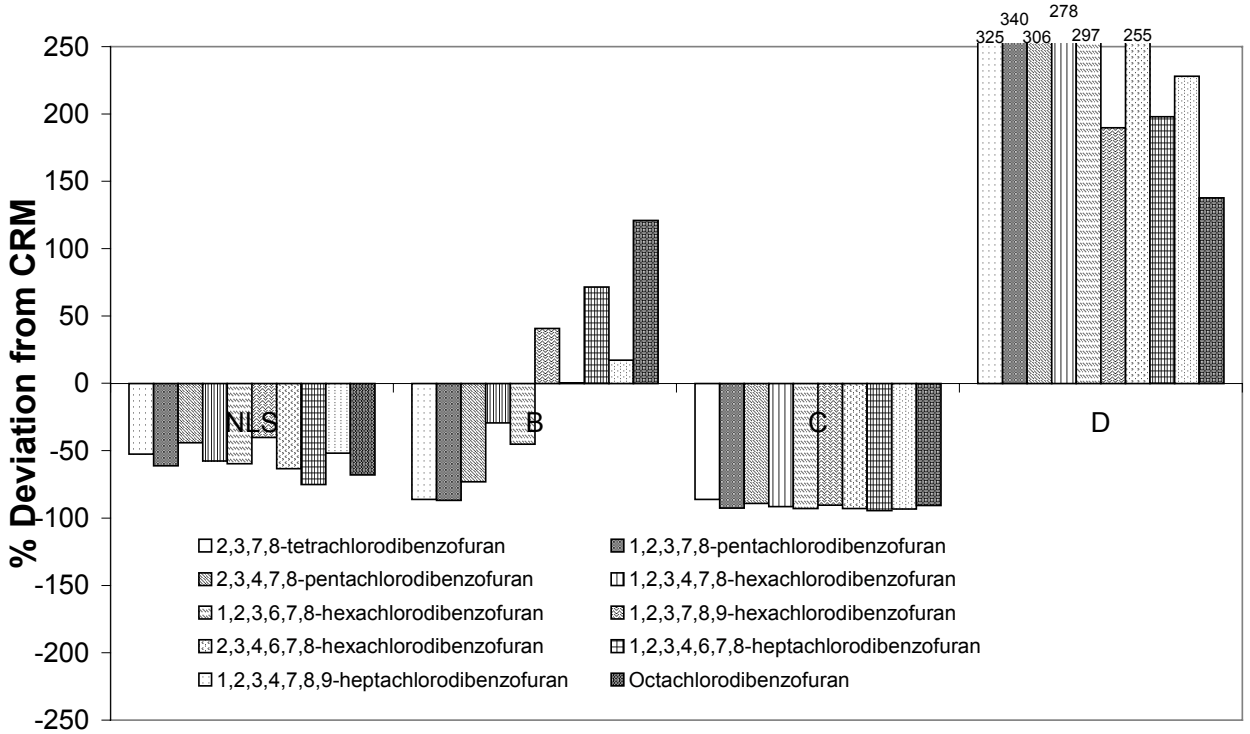

Figure 5.49 Comparison of laboratory performance for analysis of furans in flyash CRM

Table 5.22 Analysis of dioxins and furans in flyash CRM and statistical assessment of laboratory performance

		NLS			B			C			D		
Determinand	CRM	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category
2,3,7,8-tetrachlorodibenzo-p-dioxin#	0.169	0.136	-1.85	b	0.0340	-8.57	e	0.0910	-4.29	e	0.000800	-14.0	e
1,2,3,7,8-pentachlorodibenzo-p-dioxin#	0.670	0.476	-4.12	e	0.110	-10.8	e	0.310	-5.87	e	0.00100	-16.7	e
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin#	0.950	0.621	-2.75	d	0.130	-7.03	e	0.310	-5.36	e	0.00320	-8.61	e
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin#	4.80	3.30	-3.13	d	0.850	-8.33	e	1.40	-7.53	e	0.00720	-12.0	e
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin#	2.84	1.79	-2.91	d	0.570	-9.41	e	0.980	-8.28	e	0.00880	-16.7	e
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin	7.96	22.5	1.51	a	5.20	-0.287	a	12.0	0.418	a	0.0863	-0.829	a
Octachlorodibenzo-p-dioxin	12.2	36.4	1.53	a	7.30	-0.320	a	17.0	0.311	a	0.353	-0.781	a
2,3,7,8-tetrachlorodibenzofuran#	0.900	0.556	-4.92	e	0.180	-9.78	e	0.330	-8.10	e	0.00600	-17.9	e
1,2,3,7,8-pentachlorodibenzofuran#	1.71	1.31	-2.86	d	0.320	-9.05	e	0.660	-6.75	e	0.00760	-14.2	e
2,3,4,7,8-pentachlorodibenzofuran#	1.85	1.79	-0.433	a	0.420	-8.55	e	0.760	-6.88	e	0.00860	-16.7	e
1,2,3,4,7,8-hexachlorodibenzofuran#	2.37	1.73	-3.81	e	0.390	-11.8	e	0.990	-7.23	e	0.00720	-19.7	e
1,2,3,6,7,8-hexachlorodibenzofuran#	2.64	1.81	-4.28	e	0.420	-11.8	e	1.10	-7.12	e	0.00860	-18.8	e
1,2,3,7,8,9-hexachlorodibenzofuran#	0.340	0.917	7.22	e	0.160	-2.60	d	0.370	0.402	a	0.00220	-6.76	e
2,3,4,6,7,8-hexachlorodibenzofuran#	2.47	2.34	-0.634	a	0.530	-8.33	e	1.10	-5.78	e	0.0108	-14.5	e
1,2,3,4,6,7,8-heptachlorodibenzofuran	2.36	7.18	1.60	a	1.50	-0.287	a	3.10	0.244	a	0.0359	-0.781	a
1,2,3,4,7,8,9-heptachlorodibenzofuran	0.413	1.31	1.62	a	0.240	-0.316	a	0.510	0.177	a	0.00480	-0.751	a
Octachlorodibenzofuran	1.18	3.18	1.45	a	0.790	-0.282	a	1.90	0.519	a	0.0275	-0.847	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.
# A CRM value is available for this determinand.
Non # values are calculated using the approach described in Section 4.2.

5.6.7 Analysis of the incinerator ash sample for dioxins/furans

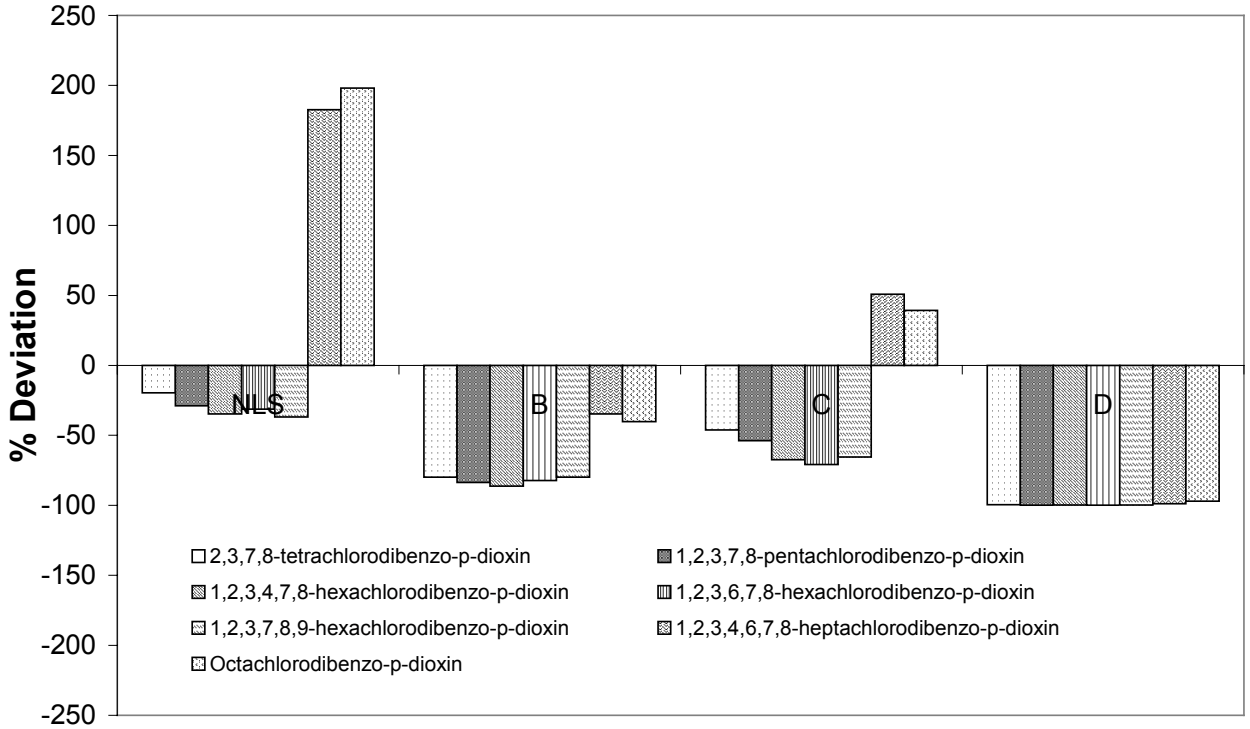

Figure 5.50 Comparison of laboratory performance for analysis of dioxins in ash sample

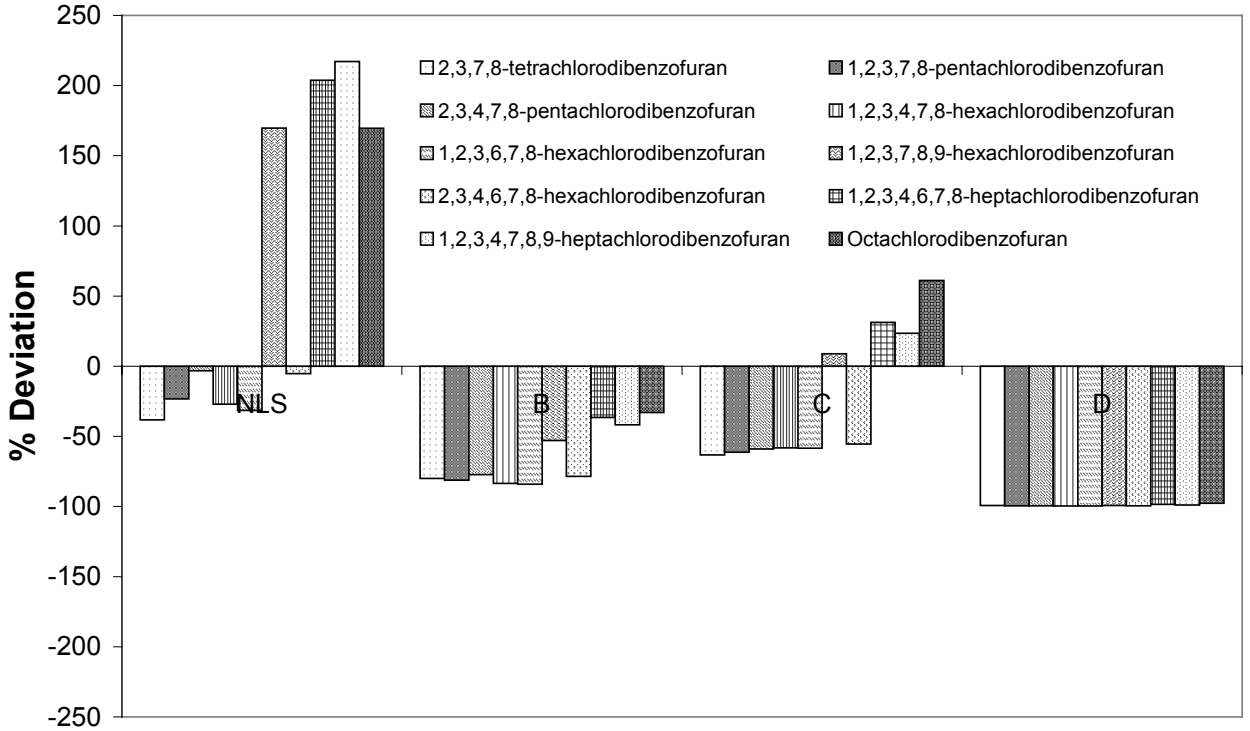

Figure 5.51 Comparison of laboratory performance for analysis of furans in ash sample

Table 5.23 Analysis of dioxins and furans in ash sample and statistical assessment of laboratory performance

		NLS			B			C			D		
Determinand	Mean	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category	Results μgkg^{-1}	u-statistic	u-statistic category
2,3,7,8-tetrachlorodibenzo-p-dioxin	0.00713	0.00290	-0.351	a	0.000500	-0.550	a	< 0.000300	*	*	0.0251	1.49	a
1,2,3,7,8-pentachlorodibenzo-p-dioxin	0.0205	0.00890	-0.310	a	0.00450	-0.428	a	0.00200	-0.495	a	0.0871	1.78	b
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin	0.0261	0.00890	-0.480	a	0.0340	0.213	a	0.00210	-0.669	a	0.0854	1.66	b
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin	0.152	0.0397	-0.401	a	0.0650	-0.310	a	0.00460	-0.526	a	0.650	1.78	b
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin	0.190	0.0259	-0.415	a	0.0240	-0.420	a	0.00330	-0.472	a	0.896	1.79	b
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin	1.05	0.297	-0.451	a	0.910	-0.0840	a	0.0730	-0.584	a	3.98	1.75	b
Octachlorodibenzo-p-dioxin	1.42	0.627	-0.393	a	1.20	-0.107	a	0.330	-0.540	a	4.94	1.75	b
2,3,7,8-tetrachlorodibenzofuran	0.0259	0.0123	-0.288	a	0.00360	-0.472	a	0.00360	-0.472	a	0.110	1.78	b
1,2,3,7,8-pentachlorodibenzofuran	0.0553	0.0215	-0.320	a	0.00720	-0.455	a	0.00410	-0.485	a	0.244	1.78	b
2,3,4,7,8-pentachlorodibenzofuran	0.0558	0.0312	-0.256	a	0.0150	-0.423	a	0.00610	-0.516	a	0.227	1.78	b
1,2,3,4,7,8-hexachlorodibenzofuran	0.0666	0.0282	-0.364	a	0.0470	-0.184	a	0.00560	-0.579	a	0.252	1.76	b
1,2,3,6,7,8-hexachlorodibenzofuran	0.0766	0.0309	-0.355	a	0.0420	-0.268	a	0.00540	-0.553	a	0.305	1.77	b
1,2,3,7,8,9-hexachlorodibenzofuran	0.0220	0.0132	-0.334	a	0.0310	0.321	a	0.00210	-0.755	a	0.0638	1.58	a
2,3,4,6,7,8-hexachlorodibenzofuran	0.0927	0.0340	-0.427	a	0.0930	0.00242	a	0.00660	-0.626	a	0.330	1.72	b
1,2,3,4,6,7,8-heptachlorodibenzofuran	0.362	0.0903	-0.572	a	0.620	0.507	a	0.0200	-0.720	a	1.08	1.51	a
1,2,3,4,7,8,9-heptachlorodibenzofuran	0.0504	0.0243	-0.381	a	0.0590	0.122	a	0.00340	-0.687	a	0.165	1.68	b
Octachlorodibenzofuran	0.172	0.0550	-0.572	a	0.380	0.890	a	0.0160	-0.763	a	0.409	1.16	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.7 Radionuclide results

The radionuclide results are presented in Figures 5.52–5.54 and Tables 5.24–5.26.

The four laboratories reporting data are indicated by UoL, A, B, C and D.

5.7.1 Analysis of CRM of known radionuclide composition

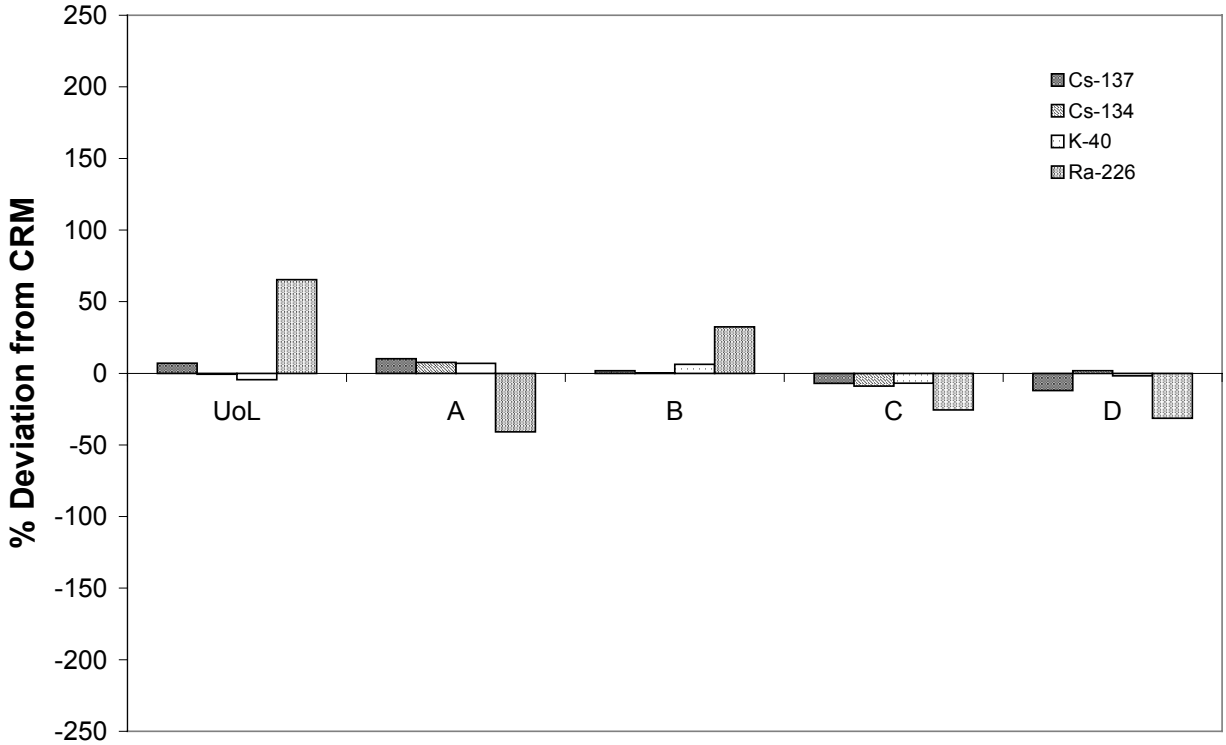

Figure 5.52 Comparison of laboratory performance for analysis of radionuclide CRM

Table 5.24 Analysis of radionuclide CRM and statistical assessment of laboratory performance

Determinand	CRM	UoL			A			B			C			D		
		Result $Bqkg^{-1}$	u-statistic	u-statistic category	Result $Bqkg^{-1}$	u-statistic	u-statistic category	Result $Bqkg^{-1}$	u-statistic	u-statistic category	Result $Bqkg^{-1}$	u-statistic	u-statistic category	Result $Bqkg^{-1}$	u-statistic	u-statistic category
Cs-137	5098	5455	0.69	a	5621	1.07	a	5192	0.17	a	4741	-0.66	a	4484	-1.29	a
Cs-134	488	484	-0.06	a	525	1.04	a	488	0.01	a	444	-0.77	a	496	0.10	a
K-40	408	390	-0.61	a	436	1.02	a	434	0.71	a	380	-0.68	a	401	-0.18	a
Ra-226	24.18	<40.0	*	*	14.3	-0.37	a	32.0	0.35	a	<18.0	*	*	16.6	-0.57	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.
# A CRM value is available for this determinand.
Non # values are calculated using the approach described in Section 4.2.

5.7.2 Analysis of Unknown Soil sample for radionuclides

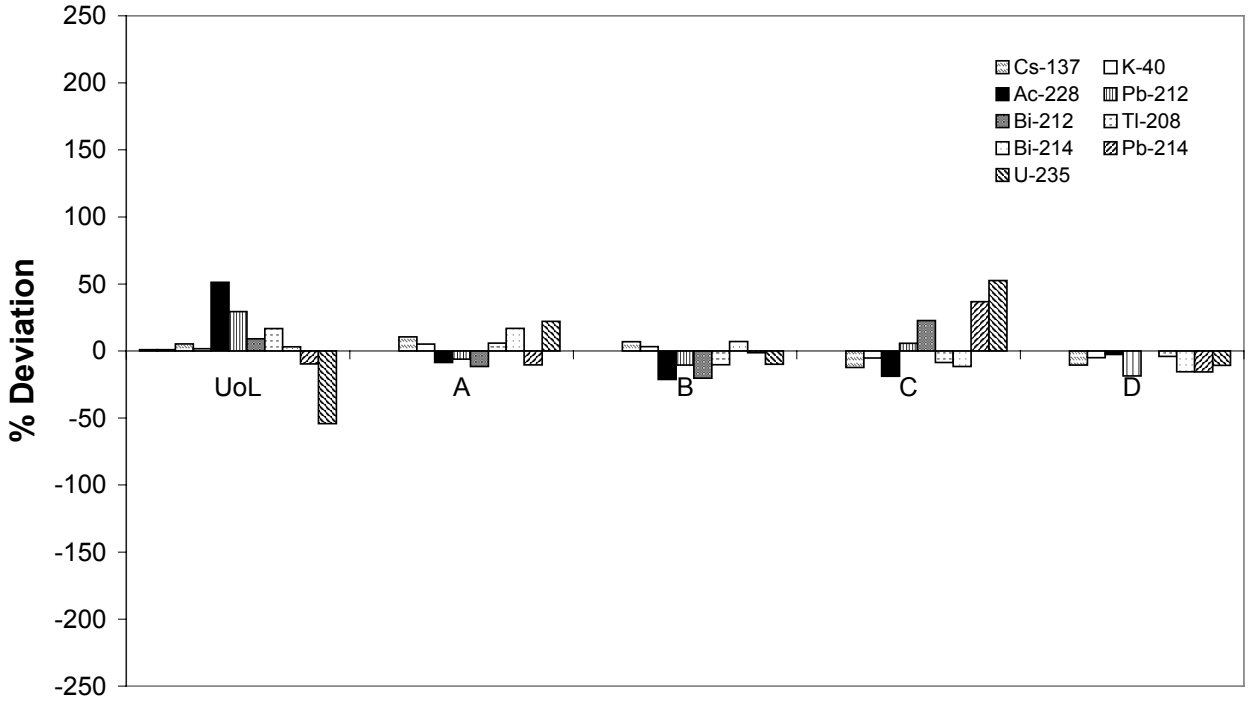

Figure 5.53 Comparison of laboratory performance for analysis of radionuclides in Unknown Soil sample

Table 5.25 Analysis of radionuclides in Unknown Soil sample and statistical assessment of laboratory performance

Determinand	Mean	UoL			A			B			C			D		
		Result $Bqkg^{-1}$	u-statistic	u-statistic category	Result $Bqkg^{-1}$	u-statistic	u-statistic category	Result $Bqkg^{-1}$	u-statistic	u-statistic category	Result $Bqkg^{-1}$	u-statistic	u-statistic category	Result $Bqkg^{-1}$	u-statistic	u-statistic category
Co-60	*	<0.60	*	*	<0.60	*	*	<1.20	*	*	<0.70	*	*	<1.72	*	*
Cs-137	11.4	12.0	0.47	a	12.6	0.93	a	12.2	0.40	a	10.0	-0.82	a	10.2	-0.79	a
Cs-134	*	<0.40	*	*	<0.48	*	*	<0.41	*	*	<0.70	*	*	<1.98	*	*
Am-241	*	<0.6	*	*	<0.70	*	*	<1.70	*	*	<0.70	*	*	NR		
K-40	327	333	0.33	a	344	0.85	a	338	0.40	a	310	-0.52	a	311	-0.72	a
Ac-228	17.3	26.1	1.51	a	15.8	-0.27	a	13.6	-0.62	a	14.0	-0.50	a	16.8	-0.08	a
Ra-224	*	<10.6	*	*	16.8	*	*	NR			NR			35.9	*	*
Pb-212	16.1	20.8	1.50	a	15.1	-0.32	a	14.4	-0.52	a	17.0	0.27	a	13.1	-0.93	a
Bi-212	15.1	17.8	0.41	a	14.4	-0.40	a	13.0	-0.31	a	<20.0	*	*	NR		
Tl-208	5.57	6.5	1.14	a	5.90	0.41	a	5.00	-0.45	a	5.10	-0.43	a	5.35	-0.18	a
Th-234	*	33.5	*	*	<17.8	*	*	<35.0	*	*	<20.0	*	*	25.4	*	*
Pa-234m	*	5.40	*	*	<1.00	*	*	<150	*	*	<2.00	*	*	NR		
Ra-226	*	<10.0	*	*	<10.5	*	*	24.0	*	*	<30.0	*	*	20.4	*	*
Bi-214	10.5	10.5	0.21	a	11.9	1.14	a	10.9	0.27	a	<9.00	*	*	8.60	-0.72	a
Pb-214	13.2	11.9	-0.43	a	11.8	-0.47	a	13.0	-0.04	a	18.0	1.43	a	11.1	-0.60	a
Pb-210	*	19.4	*	*	<0.04	*	*	16.0	*	*	<20.0	*	*	NR		
U-235	1.32	<0.60	*	*	1.60	0.49	a	1.18	-0.17	a	<2.00	*	*	1.17	-0.14	a

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

5.7.3 Analysis of herbage sample for radionuclides

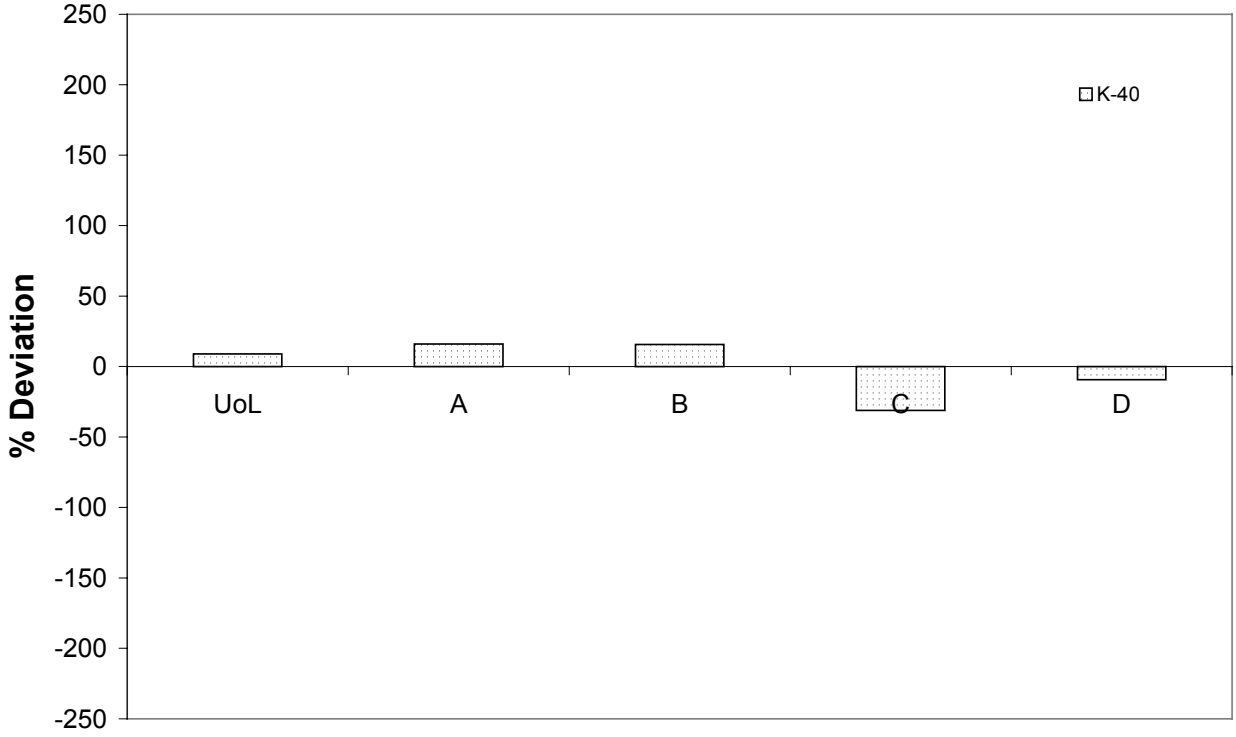

Figure 5.54 Comparison of laboratory performance for analysis of radionuclides in herbage sample

Table 5.26 Analysis of radionuclides in herbage sample and statistical assessment of laboratory performance

Determinand	Mean	UoL			A			B			C			D		
		Result Bqkg^{-1}	u-statistic	u-statistic category	Result Bqkg^{-1}	u-statistic	u-statistic category	Result Bqkg^{-1}	u-statistic	u-statistic category	Result Bqkg^{-1}	u-statistic	u-statistic category	Result Bqkg^{-1}	u-statistic	u-statistic category
Co-60	*	<4.00	*	*	<1.66	*	*	<2.90	*	*	<4.00	*	*	<6.11	*	*
Cs-137	*	1.70	*	*	2.44	*	*	<4.70	*	*	<6.00	*	*	<5.66	*	*
Cs-134	*	<2.00	*	*	<1.74	*	*	<1.70	*	*	<2.00	*	*	<7.28	*	*
Am-241	*	<0.40	*	*	<1.79	*	*	<2.50	*	*	<0.60	*	*	NR		
K-40	712	775	0.43	a	826	0.78	a	824	0.66	a	490	-1.39	a	645	-0.38	a
Ac-228	*	21.1	*	*	<8.23	*	*	<16.0	*	*	<7.00	*	*	NR		
Ra-224	*	<7.60	*	*	<31.0	*	*	NR			NR			NR		
Pb-212	*	3.10	*	*	5.21	*	*	<3.80	*	*	<10.0	*	*	NR		
Bi-212	*	<5.80	*	*	<28.5	*	*	<38.0	*	*	<20.0	*	*	NR		
Tl-208	*	1.30	*	*	2.41	*	*	<3.50	*	*	<5.00	*	*	NR		
Th-234	*	<5.80	*	*	NR			<50.0	*	*	<7.00	*	*	NR		
Pa-234m	*	<1.10	*	*	<2.19	*	*	<480	*	*	<3.00	*	*	NR		
Ra-226	*	<8.20	*	*	<30.2	*	*	<200	*	*	<20.0	*	*	NR		
Bi-214	*	3.30	*	*	<4.44	*	*	<6.50	*	*	<9.00	*	*	NR		
Pb-214	*	3.20	*	*	<3.39	*	*	<5.20	*	*	<6.00	*	*	27.1	*	*
Pb-210	*	29.2	*	*	113	*	*	<63.0	*	*	<20.0	*	*	NR		
U-235	*	<1.00	*	*	<2.12	*	*	<5.20	*	*	<0.90	*	*	NR		

NR = Laboratory did not report this determinand.
* There are insufficient data to enable the mean and u-statistic to be calculated using the approach detailed in Section 4.

6 Discussion and conclusions

6.1 Chemical analysis

In general, all the laboratories that participated in the chemical analysis part of the intercomparison trial performed well. There were no major inconsistencies in performance by any individual laboratory for all the determinand groups across the range of sample types. Of particular importance to the UKSHS, the NLS were consistently successful in producing analytical results comparable with the other laboratories.

6.1.1 Metals/metalloid

In terms of the ability of the participating laboratories to analyse the CRM and the unknowns for metals/metalloid, there were few instances where a laboratory failed to meet the u-statistic criteria for 'no significant difference', i.e. category a (Tables 5.2-5.6). For all unknown samples, every laboratory achieved a 'no significant difference' classification for all determinands for which a u-statistic could be calculated.

The main problem arose from the measurement of the CRM where, for a number of metals (e.g. Mn, Pb and Zn), the reported concentrations were lower than the values given for the CRM. However, this was a consistent problem across all participating laboratories. One suggestion is that these differences relate to problems associated with the aqua regia extraction of these metals.

Of the elements included in the UKSHS analytical suite, platinum appears to have caused all laboratories some measurement difficulties. However, this is due to the extremely low concentration of platinum in both the CRM and the unknowns. Laboratories A and B were unable to report any results for platinum, while the NLS and Laboratory C reported values as being below the limit of detection (LOD).

6.1.2 PAHs

All laboratories performed well, with the majority of the results being categorised as 'not significantly different' (Tables 5.7-5.11). The highest u-statistic values were again associated with the CRM, although laboratories C and D were the only ones to report data that were 'significantly different'.

The NLS performed in a similar manner to the other laboratories and was the only one able to report concentrations for the full analytical suite. None of the other laboratories reported for coronene, and only the NLS and one other laboratory reported for 1-methylphenanthrene and 2-methylphenanthrene. Laboratories C and D reported high values for chrysene compared with the CRM and Laboratory C reported a value 2.5 times that of the CRM for phenanthrene. All participating laboratories, with the exception of the NLS, also appeared to have had difficulty with the measurement of fluoranthene and pyrene in the CRM.

Laboratory B was only able to report two determinand concentrations above the LOD for Unknown Soil 3 and no concentrations above the LOD for the herbage sample. This was because the LODs for Laboratory B for the compounds in the PAH analytical suite were generally 50–100 times higher than those of the other laboratories.

6.1.3 PCBs

All participating laboratories were consistent in their PCB analytical performance (Tables 5.12–5.16). Their reported concentrations for PCBs in each of the samples analysed did not differ significantly for any of the unknowns where a u-statistic was determined. But for some samples, particularly for Unknown Soil 3, a large number of LOD values were reported which prevented calculation of the u-statistic. Laboratory C generally reported higher LOD values than the others.

Laboratory B reported fewer PCB congeners than the other laboratories (congeners 18, 31, 47, 49, 51, 128 and 170 were not reported) for all samples. Of the results it did report for the CRM, Laboratory B had the greatest proportion of discrepant values (u-statistic >3.29) (Table 5.12). Laboratories A and C had a smaller number of discrepant values from the CRM (6 and 2 respectively). Although the NLS did not have any discrepant values, it did report data for PCB 28 and PCB 31 which, while closer to the CRM value than those obtained by the other laboratories, were in categories b ('probably do not differ significantly but more data are required to confirm this') and d ('the values probably differ significantly but more data are required to confirm this'). However, all the laboratories appear to have had difficulty with these congeners.

The results reported by Laboratory B were queried with it and verified. Looking at the data, however, it would appear that some of the results are out by a factor of 100 and there may still be some form of reporting error.

With regard to the data in general, there were some cases where one laboratory's results, while not an outlier, were consistently higher than those of the other laboratories. A good example of this is given in Table 5.16 for PCB 180 where the results for the NLS and Laboratories A and C are 0.107, 0.130 and <0.500 µg/kg respectively whereas Laboratory B reported a value of 42 µg/kg. In this case, the results from Laboratories A and C and the NLS are likely to correct but, because the standard deviation was 24, the Laboratory B value was not excluded by the outlier test (Section 4.2).

6.1.4 Dioxins

Dioxin analysis performance was comparable across all participating laboratories (Tables 5.17–5.23). The results for the dioxin analyses were generally consistent though a couple of determinands (2,3,4,7,8-pentachlorodibenzofuran and 2,3,4,6,7,8-hexachlorodibenzofuran) appeared to be difficult to analyse due to the low concentrations present in the CRM. As with the other determinand groups, it can be concluded the analytical performance of all laboratories was comparable and that the NLS successfully demonstrated its analytical capabilities.

The majority of the NLS's dioxin measurements were at the higher end of the data range produced by the participating laboratories. Therefore, it is possible that conclusions based on the NLS data may be more conservative because the results are slightly higher than might otherwise be reported by other laboratories. This is demonstrated by the NLS performance for Unknown Soil 1, where the NLS data for four determinands returned gave rise to a u-statistic in category b which, while not significant, indicates that the NLS results are a little different to those from the other laboratories.

The ash samples proved difficult to analyse.

According to the u-statistic test, the vast majority of the results for the CRM are significantly different from the CRM values quoted. This indicates the level of difficulty in analysing the ash material. Looking at the data (in particular the value of the u-statistic in Table 5.22), the NLS results were slightly better than those from the other participating laboratories (e.g. it reported data that gave rise to five discrepant values compared with 11, 11 and 12 for Laboratories B, C

and D respectively). Essentially none of the laboratories measured the concentrations accurately. There are some results (e.g. Laboratory D) that appear to be a factor of 10 or 100 out from the other data. All the ash data were queried with the participants and the data given are the confirmed results. However, some form of reporting error is still suspected.

For the prepared ash sample, the range of reported concentrations is much larger than that for the CRM. Consequently, each determinand has a high standard deviation (in the order of 1.5–3 times the value of the mean). In this case, use of the u-statistic – which is based on the mean and standard deviation determined from the data returned – was not successful in discriminating between the participating laboratories. Although an outlier test was performed, the data were not sufficiently different from each other to be excluded. These difficulties reflect the analytical problems associated with incinerator bottom ash, which is heterogeneous and known to cause analytical difficulties.

6.2 Radiometric analysis

All five laboratories participating in the radiometric part of the UKSHS intercomparison trial performed consistently, with comparable results being reported by each laboratory. All radiometric results reported are in category ‘a’ (‘no significant difference’) following the calculation of the u-statistic. This applies to the CRM, soil samples and the herbage K-40 result. However, the majority of results reported for the herbage sample are below the LOD reflecting the very low radionuclide activity concentrations likely to be present in herbage from a rural site away from possible sources of anthropogenic radionuclides. These data are consistent with other available datasets.

Of particular importance to the UKSHS, the UoL radiometric results were comparable with those of the other laboratories.

6.3 Conclusions

Most laboratories now participate regularly in national and international round-robin and inter-laboratory comparison exercises. However, it was felt that this project would benefit from examining how the performance of the NLS and UoL laboratories compared with other UK laboratories that might be involved in this type of exercise.

Following analysis of the data reported by each of the participants in the laboratory intercomparison trial, the main conclusions are as follows:

- Metal concentrations reported by the NLS are comparable with those reported by other laboratories for a given sample, although there is an indication that there are certain metals where the aqua regia extraction gave rise to a lower concentration than that reported for the CRM.

- PAH concentrations reported by the NLS are comparable with those reported by other laboratories for a given sample.

- PCB concentrations reported by the NLS are comparable with those reported by other laboratories for a given sample.

- Dioxin concentrations reported by the NLS are comparable with those reported by other laboratories for a given sample. However, there were significant analytical difficulties with the analysis of ash samples with none of the participating laboratories performing the analysis of either the CRM or the prepared ash material well.

- Overall, the NLS's analytical performance was shown to be comparable to, or better than, the analytical performance achieved by the other participating laboratories for the analytical suite and samples included within this intercomparison exercise.

- Radionuclide activity concentrations reported by the UoL are comparable with those reported by other laboratories for a given sample.

Both laboratories that have conducted analysis for the UKSHS were shown to produce results comparable with those obtained by other UK laboratories. This supports the findings of international and national intercomparison and round-robin exercise results for the NLS and UoL laboratories. The exercise provides additional confidence in the data being reported within the UKSHS project.

List of abbreviations and acronyms

ASE	accelerated solvent extraction
CRM	Certified Reference Material
Defra	Department for Environment, Food and Rural Affairs
Dioxins	polychlorinated dibenzodioxins and dibenzofurans
GC	gas chromatogram
GFAAS	graphite furnace atomic absorption spectrometry
GPC	gel permeation chromatography
HR GC-MS	high-resolution gas chromatography mass spectrometry
HRGC-LRMS	high-resolution gas chromatography, low-resolution mass spectrometry
IAEA	International Atomic Energy Agency
ICP-MS	inductively coupled plasma mass spectrometry
ICP-OES	inductively coupled plasma optical emission spectrometry
LOD	Limit of detection
NLS	Environment Agency's National Laboratory Service
PAHs	polycyclic aromatic hydrocarbons
PCBs	polychlorinated biphenyls
ppb	parts per billion
ppm	parts per million
SEPA	Scottish Environment Protection Agency
SNIFFER	Scotland and Northern Ireland Forum for Environmental Research
SPE	solid phase extraction
UKAS	United Kingdom Accreditation Service
UKSHS	UK Soil and Herbage Pollutant Survey
UoL	University of Liverpool

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