PROPOSALS TO EXTEND THE ENVIRONMENT AGENCY'S MONITORING CERTIFICATION SCHEME (MCERTS) TO THE CHEMICAL TESTING OF SOILS

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A report prepared by the Environment Agency's National Compliance Assessment Service in collaboration with the Environment Agency's Land Quality function.

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Section 1 – Overview of Proposals

Introduction

1.1 The Environment Agency (the Agency) has specific responsibilities under environmental protection legislation to protect the environment and a duty to form an opinion and report on the state of the environment. The Agency's strategy focuses on the need to address the state of the environment at any time, to identify the pressures that are affecting it, to consider options and, where required, to ensure regulated industries implement appropriate controls.

1.2 An essential first step for implementing this strategy is to have measurements of contaminants in environmental media because of the potential harm they can cause to the environment and to human health. The Agency can then decide on the appropriate measures to be taken to prevent, minimise, or eliminate any harm from occurring. Clearly, if the Agency is to make informed quality decisions these need to be based on the assessment of reliable data that industry, regulators and the public can all have confidence in.

1.3 The Agency is pursuing several initiatives to help improve the quality and reliability of measurement data submitted to it for assessment. Central to these is the Agency's Monitoring Certification Scheme: MCERTS.

1.4 As part of the development of MCERTS, the Agency now proposes to extend the scheme to the chemical testing of contaminants in soil by establishing a register of qualifying laboratories. Qualification would be by third party accreditation to Agency performance requirements based on the European and international standard EN ISO/IEC 17025:2000. A MCERTS laboratory performance standard has been developed to provide an explanation and interpretation of the generally stated requirements of EN ISO/IEC 17025: 2000 for this particular application.

1.5 Comments are invited on these proposals in accordance with the Agency's arrangements for the technical review by stakeholders of new developments under MCERTS. Section 1 of this document provides an overview of the proposals. Section 2 describes the MCERTS performance standard.

Environmental legislation

1.6 New legislation is bringing the issue of reliance upon laboratory data from the chemical testing of soils into particular focus. Part IIA of the Environmental Protection Act 1990 (implemented in 2000) and the Pollution Prevention and Control (England and Wales) Regulations 2000 require testing to establish the concentration of particular contaminants in soil. The Agency and Local Authority regulators will rely upon the data produced by laboratories to make key regulatory decisions. It becomes increasingly important, therefore, that the data produced are reliable, and uncertainties associated with their production are explicitly stated.

Chemical testing of contaminants in soil

1.7 The chemical testing of contaminants in soil involves the taking of samples for laboratory analysis. However, the parameters normally considered for analysis depend upon a number of factors, including the nature of the site being investigated, its previous history of use, the intended new purpose of the site and any remedial action being considered to remove contamination. Hence, the range of parameters including associated concentrations to be determined is site-specific.

1.8 Although methods for the chemical testing of soils are developed and published by many organisations, there is, generally, no guidance on the levels of performance that these methods should achieve, and whether their performance is appropriate for use, especially for the assessment of potentially contaminated land.

1.9 Furthermore, it is recognised by the Agency, and other organisations, that the overall quality, reliability and consistency of the chemical testing of contaminants in soil, including the reporting of results, require improvement. Practices and procedures are often carried out to the detriment of quality in a predominantly price-driven, commercial market. The Agency, therefore, considers that its requirements for the chemical testing of contaminants in soil now need to be more clearly stated in order to improve confidence in, and reliability of, the measurement data it needs to be able to carry out proper assessments.

Performance requirements

1.10 The European and international standard EN ISO/IEC 17025:2000 sets the general requirements for the competence of testing and calibration laboratories. The standard is for use by laboratories in developing their quality, administrative and technical systems that govern their operations. Laboratory clients, regulatory authorities and accreditation bodies may also use it in confirming or recognising the competence of laboratories.

1.11 The standard recognises that it might be necessary to explain or interpret certain requirements to ensure that they are applied in a consistent manner. This is referred to as *elaboration* of the generally stated requirements. It provides guidance (Annex B to the standard) on elaborating the standard for application to specific fields. The MCERTS performance standard described in Section 2 of this document has been developed in line with this guidance for the application of EN ISO/IEC 17025:2000 to the chemical testing of soils.

Accreditation

1.12 A laboratory would seek accreditation for the chemical testing of contaminants in soil directly from an appropriate national organisation, for example, in the United Kingdom, this would be the United Kingdom Accreditation Service (UKAS). In order for this to qualify for acceptance under MCERTS, the accreditation must be to EN ISO/IEC 17025:2000 as elaborated by the MCERTS performance standard for application to the chemical testing of soil.

MCERTS registration

1.13 The Agency's Monitoring Certification Scheme: MCERTS provides for the product certification of instruments, the competency certification of personnel and the accreditation of test-house laboratories[1]. These provisions reflect the fundamental quality aspects of the varied measurement techniques used in environmental monitoring.

1.14 Over time, a comprehensive MCERTS register will be established. This will include details of instruments, equipment, personnel and test-house laboratories that have been certified/accredited as conforming to MCERTS standards.

1.15 A laboratory that has achieved accreditation in accordance with paragraph 1.12 will be registered under MCERTS. The MCERTS register of accredited laboratories will be maintained by the Agency. This information will be made available on the Agency's web-site.

1.16 A registered laboratory would be eligible to market its services under MCERTS for its accredited scope. It is important to understand that accreditation would not be generic but would be for the analysis of the parameters specified by the laboratory. Consequently, whilst a laboratory could seek accreditation for one particular parameter, it would then not be able to analyse other parameters under MCERTS, nor market itself under MCERTS as offering a service outside of its scope of accreditation.

Benefits of including chemical testing of contaminants in soil within MCERTS

1.17 The development of performance standards and procedures for the accreditation of laboratories carrying out chemical testing of contaminants in soil under MCERTS will provide several benefits. These include:

- Establishing a level playing field based on the Agency's requirements, in the form of a MCERTS performance standard;
- Sending a clear message that the production of defensible data for the chemical testing of contaminants in soils is a crucial component of the Agency's regulatory requirements;
- Providing assurance to all stakeholders including contractors, regulators, laboratories and the public on the reliability of analytical data generated under MCERTS;
- Providing independent and impartial arrangements for the establishment of a MCERTS register based on formal accreditation of analytical laboratories undertaking chemical testing of contaminants in soil.

1.18 Overall, the extension of MCERTS to include the chemical testing of contaminants in soil will provide a powerful driver to improve the quality and reliability of the analysis of soils and improve confidence in the data generated.

Soil sampling

1.19 The Agency recognises that sampling and the competency of the individuals carrying out the sampling plays a very important part in the overall quality of the analytical results from the chemical testing of soil. These proposals do not cover performance standards for the competency of personnel in relation to sampling procedures and strategies. Also, these proposals do not cover on-site screening tests, where for example *in-situ* testing of soils is carried out. However, any analysis work carried out within a laboratory located on-site is included. MCERTS may be extended to cover sampling and on-site screening tests at a later date.

Implementation

1.20 In 1999, the Agency established a policy[2] in relation to the progressive introduction of these proposals over a period of 18 months. The original target dates defined full implementation by October 2001. However, the Agency recognises that the complexity and scope of these draft proposals may create some difficult challenges for some laboratories. In view of this, the staged introduction of the policy has been amended and the policy statement should now state:

"To allow a reasonable time for laboratories to complete the validation of their testing methods to the Agency's specification, the Agency proposes to implement this policy over a period of twelve months from April 2002. From this date, the Agency will require that all chemical testing data on contaminants in soils, which is presented to the Agency in support of regulatory compliance, must have an accompanying estimate of bias and precision and a description of the testing method that has been used. The Agency will also require the testing laboratory to be accredited to the international standard EN ISO/IEC 17025:2000 for the test method that has been used. The Agency will be flexible in its requirement for specification of bias and precision until April 2003. Thereafter, the proposals under MCERTS will be fully applied."

1.21 The following target dates for progressing the issue and implementation of the policy are proposed:

From	To	Activity
October 2001	December 2001	Distribution of proposals and issue of technical review and assessment of feedback from industry
January 2001	March 2002	Revise and finalise proposals
April 2002	April 2003	Period for allowing laboratories to achieve conformance
May 2003		Full application of policy

1.22 In summary, from May 2003, the Agency would only accept analytical data for the chemical testing of contaminants in soil from laboratories that have been accredited to EN ISO/IEC 17025:2000 as elaborated by the final version of the MCERTS performance standard proposed in section 2 of this document.

Review arrangements

1.23 The Agency invites comments on these proposals. Comments would be particularly welcomed on the following aspects of the proposals:

- *The proposals are not intended to be prescriptive with regard to sample preparation and pre-treatment, and some degree of flexibility is allowed; however, all relevant information must be recorded and reported with the results submitted for assessment.*

- *The proposals could include references to minimum levels of acceptability with respect to results submitted to a proficiency-testing scheme organiser but the Agency recognises that these levels may be adversely affected by a number of factors, for example, if very low concentrations of parameters are present and reported.*

- *Validation and routine analytical quality control procedures could be defined in a prescriptive manner; however, the Agency recognises that specific procedures would depend, for example on the availability of appropriate certified reference materials and the concentration levels found in specific matrices of interest.*

- *Some parameters can be described generically, for example mineral oil, phenols and polyaromatic hydrocarbons; what guidance should be provided to clarify issues of this nature?*

- *The recording and reporting of results could be specified within a standardised MCERTS reporting form.*

- *The proposed MCERTS performance standard represents best practice, as currently achievable, based upon information provided to the Agency. Is this performance standard sufficiently comprehensive when assessed against all the factors relating to the chemical testing of contaminants in soil?*

- *The proposed target dates for implementation.*

- *The possible later extension of MCERTS to the competency of personnel carrying out sampling, and on-site screening tests.*

1.24 Comments are sought on the proposals. In recognition of the comprehensive nature of the proposals, an eight week period has been set aside for technical review. Comments should be submitted in writing, or by e-mail, before 30 November 2001 to

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Section 2 - MCERTS performance standard for the chemical testing of soils

Introduction

2.1 This section sets out the Agency's proposals for establishing a MCERTS performance standard based on an elaboration of EN ISO/IEC 17025:2000 for application to the specific field of laboratories carrying out chemical testing of contaminants in soil. The proposed MCERTS standard states the Agency's requirements for ensuring that laboratory accredited procedures are fit for purpose and capable of delivering reliable quality data.

2.2 From May 2003, the Agency will only accept analytical data for the chemical testing of contaminants in soil from laboratories with third party accreditation (in the UK from UKAS) to EN ISO/IEC 17025:2000 as elaborated by the final version of this MCERTS standard. Conformance to these requirements will provide confidence to regulatory authorities, users of analytical data and the public that the data generated for management of contaminants in land are fit for purpose.

2.3 Soil is an important biological medium for supporting life and comprises complex mixtures of inorganic minerals and organic matter. The relative proportions of these mixtures vary enormously presenting a difficult range of sample matrices to the analyst. Furthermore, there is no single, standard soil material that can be used as a reference material either for contaminated or uncontaminated soil. It is, therefore, very difficult to prescribe specific analytical methods.

2.4 Consequently, the Agency's approach is to specify the performance characteristics that have to be met and leave the choice of method to the analytical laboratory. The performance characteristics are considered to represent best practice within the United Kingdom. Advice and guidance on the requirements for bias and precision (and how these characteristics are to be demonstrated by laboratories) are described, as are requirements for the production of validation data, proficiency testing and method descriptions. The proposed requirements for acceptable levels of bias, precision, limits of detection etc reflect the need to make meaningful and realistic judgements, taking into consideration other sources of variation.

The MCERTS performance standard

2.5 The proposed MCERTS performance standard elaborates the following main elements of EN ISO/IEC 17025:2000:

- description of methods;
- validation procedures;
- performance characteristics;
- pre-treatment of samples;
- routine analytical quality control;
- proficiency testing;
- reporting.

2.6 The MCERTS performance document does not re-state the provisions of EN ISO/IEC17025:2000 but maintains the international standard as the "governing document". For ease of reference back to EN ISO/IEC 17025:2000 the requirements elaborated for chemical testing of contaminants in soil are presented using the relevant clause numbers in the international standard.

Description of methods (clause 5.4.2)

2.7 In order for meaningful comparisons to be made and to establish whether methods can be considered equivalent, a clear and unambiguous description of the method used for the chemical test shall accompany any analytical data submitted under MCERTS. This description need not be fully comprehensive, but shall comprise more than the title of the method and shall clearly indicate the scope and matrix for which the method is applicable. The description of the method shall be in sufficient detail to allow direct comparisons with similar methods that might be used by other analysts or laboratories. For example, when an extraction technique is carried out to isolate or concentrate a particular parameter then the name of the solvent or full details of the composition of the solvent mixture shall be given. Also, the amount of soil taken for analysis and the amount of solvent used in the extraction shall be reported. In addition, where the analytical determination of an extract is undertaken and, for example, this involves the use of a specific wavelength or mass number, then details shall also be given.

2.8 Within the laboratory where chemical testing is undertaken, a fully documented method shall be made available, if required, to the Agency. The Agency will not prescribe analytical methods that laboratories use. Methods shall, however, be capable of satisfying the performance characteristics specified in this MCERTS standard. Laboratories shall demonstrate that they can achieve the overall level of performance specified, and continue to achieve this performance at every occasion analysis is carried out under MCERTS.

Validation procedures (clause 5.4.5)

2.9 Before any method for a particular matrix is brought into routine use within a laboratory that method should be fully validated and performance tested. Whilst there is little published information or guidance on validation and performance testing of analytical methods for contaminated soils, good practice for the water industry has been described elsewhere[3]. The process of full validation provides confidence that the performance characteristics that are established are based on robust experimental determinations and are statistically sound. When the performance characteristics have been established, the values shall be compared with those values specified by the Agency in this MCERTS standard as being suitable for the chemical testing of contaminants in soil (see paragraph 2.19). If the comparison is satisfactory, or regarded as satisfactory, then adoption of the method for routine use for chemical testing of contaminants in soil can proceed. If the comparison is not satisfactory, then further development of the method should be initiated and the validation procedures repeated, or an alternative method chosen.

2.10 Validation procedures should include a number of operations such as the analysis of blank, sample, spiked sample, and standard solutions using the entire method (for example pre-treatment, extraction and determination) as would be used in the analysis of a typical sample matrix for the particular parameter of interest. In the context of the chemical testing of contaminants in soil, whilst spiked samples are useful they do not often fully simulate natural samples. Also included in this process are the analyses of certified reference materials. However, it is recognised that these materials may not always be available for the types of sample matrix requiring analysis.

2.11 The method shall be tested to establish performance characteristics such as precision, bias, limit of detection and spiking recovery. The concentrations of the solutions used in the validation procedures should be reflective of and appropriate to the concentrations found in the samples being analysed and the calibration and working range of the method. All solutions should be prepared immediately before analysis for each batch, or be taken from bulk stock solutions that are known (and have been shown) to be stable over the entire period of testing. For methods where it is not possible to record values other than zero for blank solutions (for calculating limits of detection) then an appropriate standard containing a low concentration of parameter should be used, and the concentration reported. Complete details of all the validation procedures shall be made available.

2.12 Calibration solutions can be taken through the entire method or be prepared solely for the determination stage. In either case, solutions should be matrix-matched to the sample extract solution to be determined, and an estimate of the recovery established. In addition, the calibration should, ideally, be linear and cover the range of interest for the samples being analysed. Calibration solutions and standard solutions used for quality control purposes, should be prepared by different analysts and wherever possible, from different materials.

2.13 In order to provide greater confidence in the performance characteristics established for the method, the values should, ideally, be determined from a minimum number of degrees of freedom, typically not less than ten. This requirement can be fulfilled using a combination of batch numbers and replicate analyses. Whilst the analysis of 11 batches of samples in duplicate will result in a minimum number of ten degrees of freedom, any suitable combination may be used depending on the within-batch and between-batch variations. The replicate analysis of batches should be undertaken and completed within a period of time of not less than 6 days but not exceeding 3 months.

2.14 Validated performance characteristics should be obtained for each parameter and sample matrix containing typical concentrations of interest analysed routinely within the laboratory. It is acknowledged that the use of a validated method for one matrix may not be suitable for use in the analysis of a sample from a different matrix. This may also be the case in the analysis of the same matrix but containing significantly different concentrations of the same parameter.

2.15 When a method has been validated, its stated performance should reflect the routine capability of the method. When a method is being developed and optimised, analysts may require special skills. After the development process has been completed and the method is then to be validated, validation procedures should be undertaken by staff,

such that, when the method is used routinely, its day to day performance is typical of and maintained at the level of the stated performance. It is essential that all analysts are able to demonstrate their own capabilities in the use of the method. In this respect, a laboratory shall operate appropriate internal quality control schemes to demonstrate that the performance of the method does not significantly deteriorate and to ensure the analysis remains in statistical control. Statistical techniques are available to establish whether or not different analysts are, or can be regarded as being, equivalent[4]. Similar techniques can be used to establish the equivalency of different methods.

2.16 Whenever a method that is routinely used within a laboratory is modified in any way, the changes made to it may affect the resulting performance. Hence, any changes made to a method already accredited to the MCERTS requirements shall be notified to the national accreditation organisation for further scrutiny. In addition, it shall be reported whether or not the changes incorporated into the method have made statistically significant differences to the performance characteristics. Details of the statistical analyses shall also be provided. Wherever statistical analyses or techniques are used, the stated level of confidence shall be 95%.

Performance characteristics (clause 5.4.6)

2.17 Results shall provide information that is relevant for their intended purpose[5]. For example, if a concentration of 20 $mgkg^{-1}$ is not to be exceeded and most of the results generated lie in a region much below this value, then a variation of ± 20 % might not be a cause for concern. If, however, the results generated were to lie close to the value of 20 $mgkg^{-1}$, then the same level of variation might not be acceptable.

2.18 For the purpose of these proposals, the following performance characteristics have been defined as those that the Agency deems acceptable for chemical testing of contaminants in soil, bearing in mind the need for meaningful decisions to be taken, current analytical capabilities and other likely sources of variation. The performance characteristics include bias, precision, limit of detection and spiking recovery. It is recognised that some of the performance characteristics are not applicable for certain parameters, for example limit of detection and spiking recovery for the determination of the pH value, and this will be taken into consideration when data are submitted.

2.19 The performance characteristics for selected parameters are specified in Tables 1 – 3 (where a parameter is not listed in these tables, see paragraph 2.21). Whether results are determined on a "wet-weight", "as submitted" or "air-dried" basis, all values shall be calculated and reported on a dry-weight basis.

- The bias (or systematic error) of individual results determined for the entire method should be no greater than the figure indicated in Tables 1 - 3 as a percentage of the result.

- The precision, as expressed as the total standard deviation, of individual results determined for the entire method should be no greater than the figure indicated in Tables 1 - 3 as a percentage of the result.

- The spiking recovery determined for the entire method expressed on a percentage basis should lie between 80 - 120 %.

- The limit of detection (LOD) determined for the entire method, for a particular parameter, should not be statistically higher than the figure indicated in Tables 1 - 3, expressed in units of $mgkg^{-1}$ on a dry-weight basis, except for those parameters marked * where units are expressed in $ngkg^{-1}$ on a dry-weight basis.

2.20 Where spiking recoveries are significantly outside the range 80 - 120 %, consideration should be given to correcting the result recorded or changing the method to another more suitable for the concentration of interest or particular matrix. Full details of any procedure used shall be recorded when the results are reported.

2.21 When the analysis of a parameter not listed in Tables 1 - 3 of this document is carried out, the laboratory shall report the performance characteristics actually achieved. The performance characteristics for the parameter shall be determined in the same manner and in accordance with the same requirements as specified in this section. In addition, where a group of similar compounds are analysed, for example phenols, and the combined concentrations of these compounds is expressed as the concentration of a single compound, for example phenol, then the number and identity of each compound analysed shall be recorded when the results are reported.

Pre-treatment of samples (clause 5.7.1)

2.22 The analysis of a sample shall be performed on a representative or homogenised sub-sample. The preparation of this is a very important part of generating the correct result. However, depending on the parameter to be determined, the type of sub-sample will not necessarily be the same for all analyses. Normally, certain analyses can be carried out on air-dried samples. Indeed, for comparative purposes where a variety of samples are analysed, it is essential that all results should be reported on a dry-weight basis. When so reported, results can be compared directly between one sample and another, or between different laboratories. Conversely, it is known, for instance, that certain parameters are unstable or begin to degrade once the sample has been taken. In these situations, the analysis shall be carried out, without undue delay, on a representative sub-sample of the sample as removed from the site or preserved or stabilised on-site prior to analysis. The results of this analysis shall then be converted to, and reported on, a dry-weight basis. Thus, when an analysis of the sample is required for a parameter before any degradation occurs, the sample shall be analysed on a "wet-weight" or "as submitted" basis, but results reported on a dry-weight basis, and this fact recorded. Furthermore, when samples are stabilised, or preserved on-site, and subsequently analysed, then this fact shall be recorded when the results are reported. In addition, the procedures used to prepare an air-dried sample shall be reported, as shall the procedures used to establish the dry-weight basis.

2.23 When samples that have been air-dried and are subsequently analysed, then sufficient information shall accompany the reported results to establish the stability of the parameters analysed in this manner. Such information shall provide justification for analysing samples after air-drying has been carried out, rather than analysing samples

on a "wet-weight" or "as submitted" basis.

2.24 When samples are removed from the site, they often contain a variety of substances and constituent parts other than the soil under investigation. Hence, before analysis begins, the sample is often divided into constituent parts. If, after air-drying, the sample is to be sieved, or certain constituent parts removed, then appropriate details of any material remaining on the sieve, or any constituent parts removed, shall be recorded. These details shall include for example, the amount, type and nature of such materials. In addition, it shall be noted whether or not the remaining material on the sieve, or constituent parts removed, undergo the same analysis as that carried out on the material passing through the sieve. All relevant information shall be provided when results are reported to establish whether the analysis of sub-samples relates to all, or constituent parts, of the sample as submitted.

Proficiency testing (clause 5.9)

2.25 Repeated analysis of a sample can provide information on random variability, and give an indication of the precision of the method. However, repeated determinations of this type do not reveal, or minimise the effects of, bias or systematic error. Hence, a laboratory that undertakes chemical testing of contaminants in soil and submits results under MCERTS for assessment shall participate in an appropriate external quality control or inter-laboratory proficiency-testing scheme.

2.26 The methods, used by the laboratory to generate analytical data for the chemical testing of contaminants in soil which are submitted under MCERTS, shall be the same as those methods used by the laboratory for the analysis of samples distributed by the proficiency-testing scheme organiser. In addition, as far as is possible, samples distributed by the proficiency-testing scheme organiser should be treated by the laboratory in the same manner, for example in respect of registration, storage, analysis, recording and reporting of results etc, as normal routine samples submitted for chemical testing of soils.

2.27 Full details of the scheme, including the number of samples, parameters and analyses to be undertaken by the laboratory and the types of matrices to be analysed shall be made available. The results, of all analyses submitted by the laboratory to the scheme organiser, shall also be available. The laboratory shall have a documented system in operation to investigate and address those results submitted to the proficiency scheme organiser that are considered unsatisfactory.

Routine analytical quality control (clause 5.9)

2.28 The laboratory where chemical testing of contaminants in soil is undertaken shall operate appropriate routine analytical quality control schemes as a means of continually checking analytical performance. These schemes should ensure that, following full validation of the method, the routine application of the method is shown to be in statistical control and that the laboratory is able to maintain the stated level of performance for the entire method.

2.29 The demonstration of continued performance can be achieved in several ways. With

each batch of samples, for example, blank solutions, and spiked sample solutions and standard solutions etc of known concentrations relevant to the samples being analysed, can be analysed and results plotted on appropriate quality control charts. In addition, certified (or in-house) reference materials can be similarly treated. Depending upon the number of samples within a batch, each batch of samples shall be analysed with quality control solutions amounting to between 5-20 per cent of the total number of solutions.

2.30 The laboratory shall have documented systems in operation to investigate and address those quality control results that are regarded as being "out of control". These systems shall establish definitions for "out of control" situations, and document procedures for reporting results of samples, that are analysed with quality control solutions which are regarded as "out of control". Full details of the documented systems shall be available.

2.31 In order to monitor the variation of quality control samples, results are often recorded or plotted on quality control charts or Shewhart charts. Where these control charts are used, they shall be reviewed regularly, and the "out of control" limits updated periodically as necessary. Full details of the results that are "out of control" and the remedial actions undertaken to ascertain the causes shall be recorded and be available.

Reporting (clause 5.10)

2.32 In order to make checking easier and to facilitate meaningful comparisons, appropriate information should be included in the report to clearly identify and locate the sample relating to the results. This information shall require the recording of all data necessary to allow a complete audit trail to be made. The following information should, for example, be included:

- location of sample, including depth where necessary;
- unique sample code or reference;
- date/time sample taken;
- name of sampling officer(s);
- name of laboratory, including sub-contracting laboratory where necessary;
- date sample analysed;
- parameter analysed, including whether sample preserved or stabilised on-site and whether analysis carried out on air-dried or "as submitted" basis;
- result of analysis on dry-weight basis;
- name of analyst(s) undertaking analysis;
- other relevant comments.

This information could be specified in a standardised reporting form under MCERTS.

Table 1 - Performance characteristics (metals and organometallics)

Parameter	LOD[1]	Precision[2]	Bias[2]
antimony	1	7.5	15
arsenic	1	7.5	15
barium	10	5	10
beryllium	0.1	5	10
boron (water soluble)	10	5	10
cadmium	0.5	5	10
cobalt	5	5	10
copper	5	5	10
chromium	5	5	10
lead	5	5	10
mercury	0.1	5	10
molybdenum	5	5	10
nickel	2	5	10
organolead compounds (as tetraethyl lead)	0.5	15	30
organotin compounds (as tetraethyl tin)	0.5	15	30
selenium	1	7.5	15
vanadium	5	5	10
zinc	5	5	10

Notes
1. LOD expressed as mg kg^{-1} on a dry weight basis.
2. Precision and bias expressed as a percentage of the result.

Table 2 - Performance characteristics (inorganics)

Parameter	LOD[1]	Precision[2]	Bias[2]
easily liberated cyanide	2	15	30
complex cyanide	2	15	30
sulphide	20	15	30
sulphate	20	10	20
sulphur	20	10	20
thiocyanate	20	15	30

Notes
1. LOD expressed as mg kg^{-1} on a dry weight basis.
2. Precision and bias expressed as a percentage of the result.

Table 3 - Performance characteristics (organics)

Parameter	LOD[1]	Precision[2]	Bias[2]
aldrin	0.05	15	30
atrazine	0.1	15	30
azinphos-methyl	0.1	15	30
benzene	0.1	15	30
benzo[*a*]pyrene	0.1	15	30
chlorobenzenes (as chlorobenzene)	1	15	30
chloromethane	0.1	15	30
chlorophenols (as 1,2-chlorophenol)	1	15	30
chlorotoluenes (as 1,2-chlorotoluene)	1	15	30
cresols (as 1,2-methylphenol)	1	15	30
dichloroethanes (as 1,2-dichloroethane)	0.1	15	30
1,2-dichloroethene	0.1	15	30
dichloromethane	0.1	15	30
dichlorvos	0.1	15	30
dieldrin	0.05	15	30
diesel range organics (as n-decane)	5	15	30
dioxins (as 2,3,7,8-tetrachlorodibenzo-p-dioxin)	1*	15	30
ethylbenzene	0.2	15	30
fenitrithion	0.1	15	30
furans (as 2,3,7,8-tetrachlorodibenzo-p-furan)	1*	15	30
hexachloro-1,3-butadiene	0.1	15	30
hexachlorocyclohexanes (as lindane)	0.1	15	30
malathion	0.1	15	30
nitroaromatics (as 2,4,6-trinitrotoluene)	0.1	15	30
pentachlorophenol (PCP)	0.1	15	30
petroleum range organics (as n-decane)	5	15	30
phenols (as phenol)	0.1	15	30
phthalate esters (as dimethylphthalate)	1	15	30
polyaromatic hydrocarbons (as naphthalene)	1	15	30
polychlorinated biphenyls (as biphenyl)	0.1	15	30
tetrachloroethanes (as 1,1,2,2-tetrachloroethane)	0.1	15	30
tetrachloroethene	0.1	15	30
tetrachloromethane (carbon tetrachloride)	0.1	15	30
toluene	0.2	15	30
trichloroethanes (as 1,1,1-trichloroethane)	0.1	15	30
trichloroethene	0.1	15	30
trichloromethane (chloroform)	0.1	15	30
trifluoralin	0.1	15	30
vinyl chloride	0.05	15	30
xylenes (as 1,2-xylene)	0.2	15	30

Notes

1. LOD expressed in mg kg^{-1} on a dry weight basis except where marked * which are expressed in ng kg^{-1}.
2. Precision and bias expressed as a percentage of the result.

Terms and definitions

The following terms and definitions, with associated meanings as used within this document, are given below.

As submitted basis. - The sample as it is removed from the site from where it is taken and submitted to the laboratory for analysis of a representative sub-sample.

Concentration. - Concentration is usually expressed as mass per unit mass, for example mg kg^{-1}. It is recognised that, in certain circumstances, the term concentration is not appropriate, for example in the determination of pH values.

Bias. - This is the difference (expressed as a percentage) between the mean of a number of determinations obtained under repeatability conditions and the true or accepted concentration. Bias can be estimated where appropriate certified reference materials are available and a stated concentration has been quoted.

Laboratory. - A laboratory, or sub-contracting laboratory, where chemical testing of contaminants in soil is carried out.

Limit of detection. - This is usually the minimum concentration that is capable of being determined with a particular confidence. The calculation used to record the limit of detection, LOD, is

$$LOD = 2t\sqrt{2}.\, s_w$$

Where t is the 1 - sided Student t-value at the 95% confidence level and s_w is the within-batch standard deviation of the blank solutions, or the corresponding value for a series of solutions containing a low concentration of standard. For an infinite number of degrees of freedom, this equates to approximately, LOD = 4.645 s_w. Where blank solutions are not used for the calculation of LOD, then the concentration of the low standard solution shall be reported.

On-site analysis. - Analysis carried out on-site in proximity to the location where the sample is taken.

Out of control. - An out of control situation arises when the result of a quality control solution is shown to be outside the defined limits of recognised acceptability.

Parameter. – Within the sample, this is the determinand, analyte, substance, or group of substances that is analysed. It should be clearly and unambiguously defined.

Performance characteristics. - Those performance values, such as bias, precision, limit of detection etc, that need to be estimated before a method is used routinely. Performance characteristics can be expressed in terms of standard deviations, random and systematic differences, etc.

Precision. - This is the distribution of a number of repeated determinations and in this document is expressed as the total standard deviation of results.

Repeatability conditions. – Normally, those conditions where the analyses are carried out in one laboratory by one or more analysts using the same equipment and reagents, within a short period of time.

Spiking recovery. - The addition of a known quantity of parameter to a sample or matrix followed by complete analysis to establish that fraction or percentage recovered. The concentration of added parameter should be close to the level expected in the sample. The addition of parameter to a sample or matrix followed by immediate extraction is not a satisfactory test for estimating spiking recovery, as sufficient time should elapse to allow possible matrix-parameter interactions to occur. This technique is often used as the only viable option accessible to the analyst when reference materials are not available.

Wet-weight basis. - The sample as it is removed from the site from where it is taken and submitted to the laboratory for analysis of a representative sub-sample.

References

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(4) Valid Analytical Methods and Procedures, C Burgess, Royal Society of Chemistry, 2000. ISBN 0-85404-482-5.

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