MCERTS standard for organisations undertaking sampling and chemical testing of water

July 2025

We are the Environment Agency. We protect and improve the environment.

We help people and wildlife adapt to climate change and reduce its impacts, including flooding, drought, sea level rise and coastal erosion.

We improve the quality of our water, land, and air by tackling pollution. We work with businesses to help them comply with environmental regulations. A healthy and diverse environment enhances people's lives and contributes to economic growth.

We cannot do this alone. We work as part of the Defra group (Department for Environment, Food & Rural Affairs), with the rest of government, local councils, businesses, civil society groups and local communities to create a better place for people and wildlife.

Published by:

Environment Agency
Horizon House, Deanery Road,
Bristol BS1 5AH

www.gov.uk/environment-agency

Further copies of this report are available from our publications catalogue: www.gov.uk/government/publications or our National Customer Contact Centre: 03708 506 506

Email: enquiries@environment-agency.gov.uk

Contents

Introduction

Where you submit data for the chemical testing of untreated sewage, treated sewage, and trade effluents to the Environment Agency for regulatory purposes, those data shall be generated using methods accredited to EN ISO/IEC 17025 and this MCERTS performance standard.

Accreditation is undertaken by an appropriate national organisation. In the United Kingdom UKAS (United Kingdom Accreditation Service) is the appropriate national organisation to undertake this.

This MCERTS performance standard provides criteria for applying ISO/IEC 17025 in the specific field of sampling and chemical testing of untreated sewage, treated sewage effluents and trade effluents.

There are also requirements for anyone who uses analytical services accredited to MCERTS and submits data to the Environment Agency for regulatory purposes.

We describe some of the requirements of the performance standard in general terms. This allows flexibility for an organisation to take advantage of technological developments. This also means you are not excluded because, for example, you lack specific equipment. Along with this flexibility is the need for the provision of appropriate information. For example, if you generate test data for a specific site over an extended period you must make consistent and meaningful comparisons. Where we assess data for regulatory purposes, you must record all relevant information and make it available, if requested.

The MCERTS performance standard does not restate all the provisions of EN ISO/IEC 17025 which must be fully complied with. It only states the additional requirements which organisations must comply with to become registered under MCERTS for the chemical testing of untreated sewage, treated sewage, and trade effluents.

The clause numbers in this document align with those of EN ISO/IEC 17025:2017 and will not be the same as those in other dated versions of EN ISO/IEC 17025.

If you have any questions about the accreditation process, or would like further information on how to apply, please contact:

UKAS
2 Pine Trees
Chertsey Lane
Staines-upon-Thames
TW18 3HR
Telephone: 01784 429000

Email: info@ukas.com

Find more information on MCERTS and copies of the performance standards and further guidance on our MCERTS page on GOV.UK.

Contact the Environment Agency

You can contact the Environment Agency if you need any help.

General enquiries
National Customer Contact Centre
PO Box 544
Rotherham
S60 1BY

Email enquiries@environment-agency.gov.uk

Telephone 03708 506 506

Monday to Friday, 8am to 6pm.

1 Scope

The chemical testing of untreated sewage, treated sewage effluents and trade effluents can be undertaken for a wide range of determinands using a wide range of methods, including on-site testing methods. The methods that a laboratory or other organisation uses to generate data for submission to the Environment Agency for regulatory purposes shall have accreditation to ISO/IEC 17025 for this MCERTS performance standard.

We use the term organisation to include laboratories, as organisations not normally referred to as laboratories may apply for accreditation to this performance standard for sampling only.

This performance standard is applicable to organisations that may wish to undertake sampling and chemical testing, or just sampling, or just chemical testing, of untreated sewage, treated sewage effluents and trade effluents.

Most of the requirements of this performance standard are laboratory activities. But users of analytical services must make sure that the requirements are satisfied and that the appropriate information is provided to us, or the laboratory, if requested.

If an organisation meets the appropriate requirements of this performance standard, it will have shown that it meets the Environment Agency's requirements for undertaking the sampling and chemical testing of untreated sewage, treated sewage effluents and trade effluents. The organisation shall publish its scope of accredited activities on the UKAS website.

2 Normative references

We refer to EN ISO/IEC 17025 - General requirements for the competence of testing and calibration laboratories in the text in such a way that some or all their content constitutes requirements of this document.

3 Terms and definitions

In the context of this performance standard, these terms and definitions apply. It is recognised that some terms used in this document may have slightly different meanings to those used in other publications.

Analytical Quality Control (AQC) – the overall process of ensuring that the application of an analytical method is controlled within specified tolerances.

Batch – a number of samples prepared for a discrete analytical run.

Bias – bias, which may be positive or negative is the difference (expressed as a percentage) between the mean of a number of determinations and the true or accepted concentration.

$$\%\text{Bias} = \frac{(\text{mean of determinations} - \text{true or accepted value}) \times 100}{\text{true or accepted value}}$$

Bias can be estimated where appropriate certified reference materials are available, and a stated (certified*)* concentration has been quoted. Recovery data can be used to estimate bias via spiking experiments (see spiking recovery).

Certified Reference Material (CRM) – reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure, which establishes its traceability to an accurate realisation of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence. [ISO/IEC-Guide 30]

Concentration – concentration, for chemical testing of waters, is usually expressed as mass per unit volume, for example mg l^{-1}. (In certain circumstances the term concentration is not appropriate, for example in the determination of pH values.)

Critical level of interest (CLOI) – this is the concentration value around which a decision is often required, for example is the concentration above or below a certain value. It may be a regulatory limit, or some other concentration of importance. A method is usually acceptable if, when used properly, it can establish within defined limits of bias and precision, whether a concentration is above or below the CLOI.

Determinand – within the sample, this is the measurand, analyte, substance, or group of substances, the concentration of which needs to be determined. It shall be clearly and unambiguously defined.

Laboratory – a laboratory, or sub-contracting laboratory, that undertakes the chemical testing of untreated sewage, treated sewage effluents and trade effluents. A laboratory may also undertake sampling activities.

Organisation – in the context of this performance standard the term organisation encompasses analytical laboratories.

Operator – “operator” is defined as: “in relation to an installation or mobile plant, the person who has control over its operation”.

Performance characteristics – those performance values, such as precision, bias (or recovery, as appropriate) and LOD (limit of detection) that need to be estimated before a method is used routinely.

Precision – this is the distribution of a number of repeated determinations, obtained under specific conditions, expressed in this document as the % relative standard deviation (RSD).

%RSD = SD/M x 100

Where:

- SD = total standard deviation
- M is the mean of results

You can obtain total standard deviation from estimates of both within batch and between batch standard deviations, using analysis of variance.

Reference material (RM) – material, sufficiently homogenous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in the measurement process. [ISO Guide 35:2006]

Sample – that (uniquely identified) material removed from a site and submitted to the laboratory for analysis or analysed on-site.

Spiking recovery – the addition of a known quantity of a determinand to a sub-sample, followed by analysis to establish that fraction or percentage recovered using a defined method. See details in Annex B.

Often the use of this technique is the only viable option for the analyst when appropriate certified reference materials are not available, and bias cannot be determined directly. When this is so, calculate bias from:

%Bias = %Recovery – 100

Statistical control – when the result or results of quality control samples are shown to be within defined limits of recognised acceptability, a method is in statistical control. When these limits are breached, the method is out of statistical control.

Sub-sample – a representative or homogenised portion of the sample. This portion is used in the analysis.

Traceability – property of a measurement result whereby the result can be related to a stated reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty.

4 General requirements

4.1 Impartiality

4.1.1 No additional requirements to EN ISO/IEC 17025.

4.1.2 Organisations conducting sampling shall have arrangements in place to ensure that its management and personnel conducting these activities are free from any undue

internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work.

4.1.3 Organisations shall have policies and procedures in place to ensure operational and sampling practices do not diminish confidence in competence, judgement, or integrity.

It is not acceptable for an organisation to manipulate the operation of their treatment plant or effluent inputs to a treatment plant to take account of sampling dates. The sampling programme shall represent the normal operation of that treatment plant.

4.1.4 – 4.1.5 No additional requirements to EN ISO/IEC 17025.

4.2 Confidentiality

No additional requirements to EN ISO/IEC 17025.

5 Structural requirements

5.1 to 5.3 No additional requirements to EN ISO/IEC 17025.

5.4 For data to be submitted to the Environment Agency for regulatory purposes, the organisation shall carry out its sampling, testing and calibration activities in such a way as to meet the requirements of this performance standard.

5.5 to 5.7 No additional requirements to EN ISO/IEC 17025.

6 Resource requirements

6.1 General

No additional requirements to EN ISO/IEC 17025.

6.2 Personnel

No additional requirements to EN ISO/IEC 17025.

6.3 Facilities and environmental conditions

6.3.1 Equipment, reagents and samples shall be protected from damage or degradation, during collection, transportation, and subsequent storage, as appropriate.

Note: There may be methods specifying the procedures necessary for protecting the integrity of samples and reagents during transportation and storage such

as collection into suitable containers and storage out of direct sunlight at specified temperatures.

The organisation shall have procedures in place and use appropriate practices to ensure that sample transport and storage conditions do not adversely affect the measurement result.

6.3.2 to 6.3.5 No additional requirements to EN ISO/IEC 17025.

6.4 Equipment

6.4.1 to 6.4.5 No additional requirements to EN ISO/IEC 17025.

6.4.6 The organisation shall calibrate equipment, and if appropriate with each batch of samples, using measurement standards that are traceable to national or international standards except where derived from natural physical constants, or where this degree of traceability is not possible.

6.4.7 For instrumental methods, calibration solutions may go through the entire method or just the determination stage. In either case, solutions shall match the sample extract solutions, both in terms of acid strength and content or solvent composition. The calibration shall cover the range of interest for the samples, and should, ideally, be linear over that range. Use at least 3 calibration points (not including the calibration blank), but more shall be necessary for a non-linear calibration. Organisations should prepare calibration solutions, and standard solutions used for quality control purposes, where possible, using different analysts and from different lots or sources of materials.

When calibrating pH instruments, you may use the procedure in EN ISO 10523 Water quality. Determination of pH. This requires using 2 appropriate calibration standards, and a third to check linearity. Recalibrate if the third standard is outside limits. Full details are in the standard. This approach may also be used for electrical conductivity.

Organisations shall take at least one blank sample, containing negligible amounts of the determinands of interest, through the entire analytical system (including sample preparation if appropriate) with each batch of samples. Organisations shall demonstrate, according to written procedures, how they utilise blank samples. Organisations shall investigate blank sample results that show evidence of contamination and may have to repeat the analysis of the entire batch of samples. This may not be appropriate for some determinations, for example pH.

6.4.8 No additional requirements to EN ISO/IEC 17025.

6.4.9 The response of instruments may fall. For example, due to deterioration in a detector. This may not be immediately obvious from internal quality control sample results but might coincide with deterioration in both precision and LOD of the

analytical system. The initial calibration should, therefore, meet with appropriate predefined system suitability limits. Examples include the use of peak area or signal to noise ratio and for chromatographic methods criteria for acceptable peak shape and peak resolution for closely eluting peaks.

6.4.10 The organisation shall confirm the continuing validity of calibrations by regular analysis of calibration check standards throughout the analytical batch according to a defined procedure. Where calibration events only occur infrequently, thereby incorporating several batches across an extended period of time (that is greater than daily), then checks across the range of calibration shall be used. The instrument shall not be re-calibrated using the check standard. If a check standard fails to meet appropriate predefined limits the organisation shall recalibrate and reanalyse affected samples, unless they can demonstrate that the results are not affected. Where appropriate, procedures shall be in place to ensure calibration is valid through to the end of an analytical run.

6.4.11 to 6.4.13 No additional requirements to EN ISO/IEC 17025.

6.5 Metrological traceability

No additional requirements to EN ISO/IEC 17025.

6.6 Externally provided products and services

No additional requirements to EN ISO/IEC 17025.

7 Process requirements

7.1 Review of requests, tenders, and contracts

7.1.1 To submit data to the Environment Agency for regulatory purposes, the requirements of the methods used shall be clearly and unambiguously defined and documented. The organisation shall demonstrate that those who undertake the analysis understand the requirements of the methods used.

Note: The organisation may or may not be aware that the data it generates will be submitted to the Environment Agency for regulatory purposes. However, the organisation’s customer or user of the sampling and analytical service should be aware that if it wishes to submit the data to the Environment Agency for regulatory purposes, then the requirements of this performance standard need to be satisfied.

To submit data to the Environment Agency for regulatory purposes, the organisation shall select the appropriate sampling and test and calibration methods that satisfy the requirements of this performance standard.

An operator may sub-contract the sampling or chemical testing, or both, to another organisation. It is the responsibility of the operator to make sure that the sub-contracted organisation has registration under MCERTS for the scope of work sub-contracted. The terms of this clause do not apply to samples sent to an organisation by an external quality control or inter-laboratory proficiency-testing scheme organiser.

7.1.2 to 7.1.8 No additional requirements to EN ISO/IEC 17025.

7.2 Selection, verification, and validation of methods

7.2.1 Selection and verification of methods

7.2.1.1 The organisation shall demonstrate and provide justification that they use suitable methods (including sample pre-treatment and preparation) for the analysis of a particular matrix and determinand. They shall also show that it is appropriate for the concentration of the determinand in the sample. The organisation shall demonstrate and provide justification that method validation procedures have been undertaken in such a manner as is appropriate to the sample matrix undergoing analysis. The organisation shall make full details of the method and method validation procedures available to the Environment Agency, if requested.

7.2.1.2 to 7.2.1.3 No additional requirements to EN ISO/IEC 17025.

7.2.1.4 The Environment Agency will not prescribe those analytical methods that an organisation should use, but the method used shall be appropriate for the matrix and determinand at the level of concentration being analysed. Where results are submitted to the Environment Agency for regulatory purposes, the organisation shall provide a clear and unambiguous description of the method used to generate the results, if requested. This description does not need to be fully comprehensive. However, it should comprise more than the title of the method and shall clearly indicate the determinand, scope, principle and matrix or matrices for which the method is applicable.

You shall describe the method, determinand, and matrix in enough detail to allow direct comparisons with similar methods, determinands, and matrices that other analysts or laboratories may use. For example, if a laboratory uses an extraction technique to isolate or concentrate a particular determinand, they shall report:

- the name of the solvent or full details of the composition of the solvent mixture

- the amount of sample taken for analysis and the amount of solvent used in the extraction
- where the analytical determination of an extract involves the use of a specific wavelength or mass number, then details shall also be given

Organisations shall demonstrate that the methods they employ for each determinand are appropriate for the CLOI. Organisations may achieve this by submitting a list of the range of regulatory limits monitored by each method. Regulatory limits may change, so a mechanism shall be in place to ensure methods are still appropriate when changes take place. A fully documented method shall be made available to the Environment Agency, if requested.

7.2.1.5 to 7.2.1.7 No additional requirements to EN ISO/IEC 17025.

7.2.2 Validation of methods

7.2.2.1 Before using a method for a particular matrix and determinand for generating data for submission to the Environment Agency, that method shall be accredited to ISO/IEC 17025 for this performance standard. Only those results generated using these methods will be eligible for submission to the Environment Agency for regulatory purposes. The process of a full method validation provides confidence that the established performance characteristics of the method are robust experimental determinations and are statistically sound.

Validation procedures shall include assessment of:

- selectivity and interference effects
- range of applicability
- linearity
- calibration and traceability
- bias (recovery)
- precision
- limit of detection (LOD)
- uncertainty of measurement

Organisations shall estimate precision and bias (recovery) for each determinand and matrix covered by the method, and LOD for each determinand and method (see appendix C). Where available and appropriate, you shall analyse matrix certified reference materials relevant to the matrices, determinands and range of determinand concentrations under investigation. Sample pre-treatment and preparation is an important part in the validation process, but certified reference materials may not need any sample pretreatment. In these cases, the organisation shall undertake a separate exercise to determine the effects of sample pre-treatment and preparation.

Whilst it is not expected that every sample submitted should have its own validated method, we recognise that a single validated method established for one particular matrix but used for every sample, irrespective of its matrix, is unlikely to be appropriate. You cannot assume that one method is appropriate for all effluents. A number of appropriate matrices shall undergo full validation as described in clause 7.2.2.3, as appropriate to the requirements of the organisation. In addition to this, you may need further validations of a variety of complex trade effluent or untreated and treated sewage samples. This will in time represent the full range of sample matrices and concentrations received by the organisation. Organisations shall undertake this as described in clause 7.2.2.3.

You shall characterise each sample used in validation procedures in terms of basic analytical data. This shall include determinands appropriate to the matrix, for example chemical oxygen demand, pH, conductivity, suspended solids, hardness, and TOC.

In the absence of suitable certified reference materials, you shall determine recovery estimates relevant to the matrix and determinand under investigation, using spiking experiments. Where possible these experiments shall cover the entire method (including pre-treatment, extraction, and determination). The addition of a determinand to a sub-sample followed by immediate extraction may not be a satisfactory test for estimating spiking recovery, as insufficient time may elapse to allow possible matrix-determinand interactions to occur. A satisfactory period of time shall be allowed for such interactions to occur. The organisation shall show that its use of spiking experiments and the spiking procedures employed is appropriate.

It may be appropriate to use a mixture of spiked samples and CRMs to ensure a full coverage of all determinands and matrices, or to validate an additional CRM which may not exactly match spiked matrices but will give further confidence in the method validation.

For spiking experiments, the concentrations of the solutions used in the validation procedures shall be appropriate to the concentrations found in samples routinely analysed. Recovery estimates shall be obtained using 2 different but appropriate concentration levels, for example, 20% and 80% of the expected range, or at a CLOI. The organisation shall justify choice of sample and concentration level. If samples contain a significant amount of a determinand this approach may not be feasible, organisations must be able to find and justify an alternative approach. All solutions shall either be taken from bulk stock solutions that are known (and have been shown) to be stable over the entire period of testing or, if solutions are not stable over the entire period of testing, they may be prepared immediately before the analysis of each validation batch or stabilised by addition of appropriate reagents. The traceability of these solutions shall have been established.

You can find statistical procedures for dealing with sample instability during validation in:

A Manual on Analytical Quality Control for the Water Industry, R. V. Cheeseman and A. L. Wilson, revised by M. J. Gardner, NS 30, Water Research Centre, 1989. ISBN 0-902156-85-3

When Isotope Dilution Mass Spectrometry (IDMS) is employed (with appropriate labelled analogues of the determinands spiked into all samples, calibration standards and matrix AQC standards, and equilibrated before sample preparation is undertaken) then the results obtained will be recovery corrected. The recovery corrected values of spiked samples and CRMs obtained in this manner shall be used to estimate bias against the certified CRM and or added spike. Assign acceptable limits for surrogate recovery, such that reliability and confidence in results is maintained.

7.2.2.2 Revalidation

After validation and accreditation of an analytical method, it is inevitable that in time some modification of procedures will take place. Any modifications to a method used may affect the resulting performance. Organisations shall notify UKAS of any changes made to a method already accredited against the MCERTS requirements. These changes could range from replacing a piece of equipment to a fundamental procedural modification, such as a different extraction procedure.

Minor changes to the analytical system may not need its revalidation, but organisations shall take care to make sure the cumulative effects of several changes do not affect system performance. For example, by closely monitoring internal and external AQC, and reanalysing CRMs used for validation.

If an instrument is being replaced by one of the same model, and performance is not expected to fundamentally change, organisations only need to demonstrate that the new instrument performs as well as the old instrument. Organisations can achieve this by analysing several replicates of a representative matrix such as a spiked sample, a CRM, or a matrix matched AQC sample.

If an organisation makes a fundamental change to the analytical procedure or the equipment used then they shall need to do a full validation on a minimum of 3 matrices in accordance with this performance standard. These changes may include, for example, replacing Inductively coupled plasma optical emission spectrometry (ICPOES) with inductively coupled plasma mass spectrometry (ICPMS) or using a new extraction technique.

Organisations should carry out an intermediate degree of validation if they make significant changes to a method that are not fundamental to performance or reinstate a method after a voluntary suspension. They shall perform a partial validation (for example analysis of 6 batches of duplicates), using only one spiked sample from the lower end of the calibration range, or preferably a CRM, for all appropriate matrices. They shall also make a new estimation of LOD. If an organisation judges that the method needs this level of validation, then it shall notify

and gain the approval of UKAS. Organisations shall make sure that they include amendments to the analytical system and any procedures that they may affect, in the revalidation.

7.2.2.3 Validation procedures

For the method, determinand and matrix, the organisation shall determine performance characteristics with a minimum of ten degrees of freedom. They shall carry this out by analysing each certified reference material or spiked sample in duplicate in different analytical batches. A certified reference material is the preferred option.

Ideally, organisations should analyse each analytical batch with a new calibration, to make sure you fully reflect between batch variations.

Eleven batches of duplicates will guarantee a minimum of 10 degrees of freedom, but you may achieve 10 degrees of freedom in less than 11 batches. You can check this after each batch of results, and find appropriate procedures in:

- A Manual on Analytical Quality Control for the Water Industry, R. V. Cheeseman and A. L. Wilson, revised by M. J. Gardner, NS 30, Water Research Centre, 1989. ISBN 0-902156-85-3
- ISO TR 13530:1997 Water Quality – A Guide to Analytical Quality Control for Water Analysis

The period of validation must be between 6 days and 3 months. If a method is routinely calibrated, for example monthly, organisations shall spread the analytical batches used for validation over the 3-month period.

Note 1: This procedure is often termed an 11 x 2 test, as you analyse 11 batches containing 2 replicates of each test material.

When you have collected the data, estimate precision using analysis of variance (ANOVA). From this you can estimate different sources of error (for example within batch and between batch random errors) and combined them to give a total error as a standard deviation. Details of the statistical procedures for ANOVA and recovery (bias) estimation are given in Annex B, and these references:

- A Manual on Analytical Quality Control for the Water Industry, R. V. Cheeseman and A. L. Wilson, revised by M. J. Gardner, NS 30, Water Research Centre, 1989. ISBN 0-902156-85-3
- ISO TR 13530:1997 Water Quality – A Guide to Analytical Quality Control for Water Analysis
- NORDTEST Handbook of Internal Quality Control NT TR 569

The following wastewater matrices shall be validated as required:

- Treated sewage effluent (mixed domestic and industrial)
- Untreated sewage

- Trade effluent discharges (from industry sector commonly encountered in the laboratory, for example food processing)

Note 2: The use of a validated method for one particular matrix may not be suitable for the analysis of a different matrix. This may also be the case when analysing samples of the same matrix containing significantly different concentrations of the same determinand.

If an organisation does not require accreditation for all 3 of these matrices, then initial validation shall be on a minimum of 3 matrices that best represent those received and analysed by the organisation. For example, if an organisation does not wish to become accredited for untreated sewage, then it may substitute that matrix with a second treated sewage effluent or trade effluent discharge. An organisations Schedule of accreditation shall clearly show the matrix types for which Accreditation has been granted.

The organisation shall demonstrate that the certified reference material for the matrix, methodology, determinand and concentration of determinand they are analysing is appropriate.

After validation of a method, its stated performance shall reflect the routine capability of the method. So, when the organisation uses the method routinely, its day-to-day performance shall be typical of and maintained at the level of the stated validation performance.

The LOD of a method used to analyse highly contaminated samples may be higher than the LOD of a method used to analyse slightly contaminated samples. The reported LOD shall be fit for the intended purpose and appropriate to the concentration level of interest required of the analysis. Organisations shall calculate the LOD as described in Annex C1.3. Never use the LOD in isolation of other method validation data to judge the appropriateness of a method.

The maximum value of the LOD usually regarded as being fit for purpose is 10% of the concentration regarded as the CLOI. For example, if the lowest effluent permit level is 1 mg l^{-1} for a particular determinand, then the LOD should be at least as low as 0.1 mg l^{-1}. It is possible that this 10% may not be achievable on all matrices. If this situation arises then before submitting results you shall seek agreement with the Environment Agency.

For on-site chlorine analysis, if the consent value or 10% of the consent value/CLOI is lower than the achievable LOD for the instrumentation used, this shall be highlighted and agreed with the Environment Agency.

Performance criteria

The Environment Agency has specified that the following performance characteristics are acceptable for the validation of methods for the chemical testing

of water. You should bear in mind the need to take meaningful decisions, current analytical capabilities, and other sources of variation.

The bias (or systematic error) of individual results determined for the entire method shall not be significantly greater than the figure indicated in Annex A (Tables 1 to 3) expressed as a percentage. Organisations shall use the certified reference value of the certified reference material as the true or accepted value when calculating bias for a known critical level of interest, you can use one-twentieth of the critical level of interest as the target bias, rather than the value in Annex A. You can use the greater of the 2 values. Organisations shall demonstrate that the bias satisfies the stated requirement at the critical level of interest.

The precision, as expressed as the percent RSD, of individual results determined for the entire method shall not be significantly greater than the figure indicated in Annex A (Tables 1 to 3). Organisations shall estimate precision using ANOVA to determine total standard deviation. For a known critical level of interest, you can use one-fortieth of the critical level of interest as the target precision, rather than the value in Annex A. You can use the greater of the 2 values. Organisations shall demonstrate that the precision satisfies the stated requirement at the critical level of interest.

If required, organisations shall carry out testing for significance as described in Annex C2. If, for a particular determinand, testing shows a significant difference exists, then the organisation may need to carry out further method development or refinement or use a different analytical method.

Note 3: Experience has shown that if a method has borderline performance with respect to the performance requirements of this standard, it may be difficult to maintain the analytical performance of the method when in routine use.

Annex A (Tables 1 to 3) specifies the performance characteristics for a selection of determinands (which is not exhaustive).

When an organisation requests accreditation of additional determinands not listed in Annex A of this standard, the performance requirements organisations shall use are:

- metals – 5% precision and 10% bias
- inorganics – 5% precision and 10% bias
- organics – 15% precision and 20% bias

Where there are precision and bias targets for treated sewage and trade effluent discharges to controlled waters and none for the other matrices, the precision and bias targets for treated sewage and trade effluent discharges to controlled waters shall apply.

If an organisation is unable to meet these requirements due to matrix effects or fitness for purpose issues it shall propose alternative performance characteristics and submit them to the Environment Agency through UKAS for assessment.

If the organisation is unable to meet requirements due to analysis of a one-off nature being required urgently then they shall report the performance characteristics they achieve using a partial validation (see clause 7.2.2.2). If they use this procedure, they shall inform UKAS. If the determinand is subsequently added to Annex A the performance characteristics for the determinand shall be determined in the manner and in accordance with the full validation requirements specified in this performance standard.

Organisations shall not report these results as accredited until UKAS has assessed the method, and the Environment Agency has prescribed target performance values.

Further method validation

Having completed validation to the MCERTS standard, an organisation may be required subsequently undertake a programme of further validation.

Note 4: Further validation will ensure that methods will be assessed against a wide range of matrices encountered, without the necessity of performing the full validation procedure on each matrix.

It is possible that the composition of an effluent may change, for example if manufacturing processes change. Organisations shall ensure that initial validation is still valid.

A further validation exercise shall comprise of a minimum of 7 replicates of a sample and 7 spiked replicates of the same sample undergoing analysis. Precision and recovery shall be estimated and compared with the MCERTS requirements to ensure compliance. A significance test shall be carried out if required (see Annex C2). If MCERTS requirements are not met, and the organisation undertaking the further validation consider this is due to insurmountable matrix effects, then the further validation data shall be sent to the Environment Agency through UKAS.

Consideration will be given to the performance criteria applied in this MCERTS standard.

The organisation shall demonstrate that it has or is progressing to a good coverage of the range of sample matrices it encounters. UKAS will assess the further validation at the time of the annual surveillance visit, and after a full accreditation cycle will assess if any further work is required on each method.

For some matrices, a high background concentration of the target determinand may make it difficult to assess spiking recovery. Organisations may need an alternative approach, such as pre-dilution of spiked samples before analysis.

An alternative to spiking samples for further validation of methods using mass spectrometry detection is the use of isotopically labelled surrogate compounds to establish the recovery of each determinand for each sample (see 7.2.2.1). In these cases, a known amount of the isotopically labelled surrogate compound shall be added to every sample prior to sample analysis. The recovery of the surrogate compound shall fall within acceptable limits and be reported with the associated sample results.

If the organisation is already using an analytical method based on the use of isotopically labelled surrogate standards for each of the determinands being analysed, then there is no need to take any additional measures for the analysis of samples with unvalidated matrices, provided that the recovery of each of the surrogate compound meets acceptable limits. An estimate of precision shall still be required, based on 7 replicate samples.

7.2.2.4 No additional requirements to EN ISO/IEC 17025.

7.3 Sampling

7.3.1 Each organisation that undertakes sampling activities relating to this performance standard shall operate a management system for relevant sampling activities. This may operate independently of a laboratory.

7.3.2 The sampling management system shall include, but not be limited to, the following procedures:

- sampling programme, including procedures for resampling
- methodologies for taking samples
- training and audit
- use of appropriate bottles and preservation techniques
- sample transport, receipt, handling, storage, retention, delivery, and chain of custody
- operation, maintenance, and calibration of equipment used in sampling, including autosamplers
- operation, maintenance, and calibration of on-site test equipment
- quality assurance procedures for assessing sampling activities

On site test equipment shall be validated in accordance with Annex D of this standard

Detailed guidance of the sampling procedures is not reproduced in this standard but organisations may wish to take account of the latest Environment Agency guidance on the MCERTS page on GOV.UK.

All samplers engaged in accredited sampling activities shall be audited by their own organisation at least once annually.

If organisations use automatic sampling devices, for example if composite samples are required, then the device shall have been tested and certified to the appropriate MCERTS performance standard:

MCERTS: performance standards and test procedures for continuous water monitoring equipment - part 1 automatic sampling equipment

7.3.3 No additional requirements to EN ISO/IEC 17025.

7.4 Handling of test or calibration items

7.4.1 When a sample undergoes stabilisation or preservation before analysis, then the organisation shall record this fact when they report the results and details of the stabilising or preserving agents used. Where a party independent of the analysing organisation performs this activity (such as the provider of the samples), the organisation should obtain this information and report it as above.

Organisations shall cooperate to ensure that sample preservation and handling procedures (including selection of sample containers) is appropriate for and compatible to the analytical method being employed in the organisation.

For some determinands on some samples it may be required that the dissolved portion of the determinand in the sample is analysed and reported on. The dissolved portion of the determinand in the sample shall be defined as that which will pass through a 0.45µm membrane filter. Filtration shall take place immediately at the point of sample collection. Any deviation from this prescribed procedure shall be justified and reported with results.

If preservation of samples by refrigeration is required, then during transportation and subsequent storage of samples, including retention time in an automatic sampling device, the sample storage environment shall maintain a temperature of 4.5 ± 3.5°C. An organisation carrying out sampling shall have appropriate procedures for demonstrating this. It is recognised that some time may be required to bring the sample temperature to within this range.

Note: The temperature range is to allow for the cycling of the refrigeration devices, their opening and closing during normal operation, and effects of adding a number of warm samples. For most analytical purposes best practice is to keep the samples at a constant temperature of not more than 5°C.

7.4.2 to 7.4.4 No additional requirements to EN ISO/IEC 17025.

7.5 Technical records

7.5.1 The organisation shall retain records for a defined period not less than 6 years. This period of time shall consider the need of the customer (user of the analytical

services) and the need to submit these records to the Environment Agency, if requested.

7.5.2 No additional requirements to EN ISO/IEC 17025.

7.6 Evaluation of measurement uncertainty

No additional requirements to EN ISO/IEC 17025.

Note: You can find information about the estimation of measurement uncertainty in these references:

- S L R Ellison and A Williams (Eds). Eurachem/CITAC guide: Quantifying Uncertainty in Analytical Measurement, ISBN 978-0-948926-30-3
- Handbook for Calculation of Measurement Uncertainty in Environmental Laboratories. Nordtest Report TR 537

7.7 Ensuring the validity of results

Having demonstrated that the method performance criteria prescribed in Annex A have been satisfied, on-going performance shall be monitored to:

- demonstrate that the method performance required by this performance standard is maintained in a statistically controlled manner
- identify at an early stage any changes (especially deterioration) in performance
- provide historical verification of this performance (keep records)
- enable aspects of measurement uncertainty to be estimated

Organisations shall achieve these objectives by carrying out the AQC procedures described in clauses 7.7.1 and 7.7.2.

7.7.1 Internal Quality Control

7.7.1.1 For internal quality control, the organisation shall verify the performance of each analytical method for each batch of samples analysed. Organisations shall analyse control samples within the analytical batch with which they prepare them.

In each analytical batch, a minimum of 5% of samples shall be analytical control samples If the batch size is less than twenty, one analytical control sample per batch is still required.

To monitor the variation of control samples, organisations shall record or plot control sample results on quality control charts. Organisations shall review the charts regularly and update the control limits as necessary (see Annex C). To demonstrate statistical control, plot a minimum of 30 points in a 12-month cycle, spread evenly over the period.

If an organisation carries out an analytical procedure infrequently, it shall be necessary to employ a greater degree of AQC to make sure you maintain statistical control of the method. The approach taken shall be fully justified.

Note 1: Examples of greater degree of quality control include increasing the number of control samples in a batch, use of the standard additions approach, and use of isotopically labelled surrogate compounds in organic analysis.

If organisation use their own reference materials or synthetic effluents, the actual values used must conform to the traceability criteria as described in EN ISO/IEC 17025. The following types of control material may be suitable:

1. CRM or RM (reference material) – a sample of the target matrix, the concentration of determinand being certified to a quoted uncertainty and preferably traceable to an international or national Standard.

Note 2: Where possible use reference materials from producers that meet ISO 17034. ISO Guide 33 provides guidance on the selection and use of reference materials.

2. In-house quality control material – a sample produced by the organisation that may be synthetic, containing known concentrations of determinands of interest.

It is vital that the sample is fully homogenised so that variations in repeat analyses reflect the analytical method performance and not inhomogeneity of the sample. The amount of material should be large enough to provide consistent and stable samples for as long a period as possible. An advantage of using in-house reference materials is the ability to match the determinand concentration and matrix of the material to samples normally encountered by the organisation.

The content of the synthetic matrix shall contain both inorganic and organic components to imitate a real effluent matrix. Reference method PD ISO/TS 21231 quoted below shall be considered the primary source for preparation of synthetic matrices.

Note 3: You can find guidance on the production of in-house reference materials and synthetic matrices in references:

- Water quality – Characterization of analytical methods – Guidelines for the selection of a representative matrix PD ISO/TS 21231
- Guidelines for the In-House Production of Reference Materials – version 2, B Brookman, R Walker 1998 LGC/VAM/1998/040
- Applications of Reference Materials in Analytical Chemistry – V. Barwick, S. Burke, R. Lawn, P. Roper and R. Walker Royal Society of Chemistry, Cambridge, 2001 ISBN 0-85404-448-5
- ISO guide 80 Guidance for the in-house preparation of quality control materials (QCMs)

Note 4: You may achieve traceability for this material by characterisation against a certified reference material, for example during method validation or by comparison with the analysis of the material by accredited third-party laboratories.

3. Spiked sample – a sample representative of the matrix being analysed, to which you add a known quantity of a determinand standard solution before analysis.

Standards used for spiking the sample shall be from a different source or lot number to that used for calibration, unless other independent checks of calibration stocks are undertaken. Suitable contact times between spiking and extraction shall be determined to provide adequate time for interaction between spike and sample while ensuring that there is no degradation of the determinand.

Note 5: Estimates of bias are often complicated with 'recovery' terms, especially if the method involves an extraction stage. An estimate of precision is easily obtainable, but the apparent precision of the spike is a combination of the precision of the sample and that of the spiked sample.

4. Other options – when you carry out a test infrequently consider duplicate analyses of individual samples as submitted to the organisation, and the use of duplicate control charts. Standard addition techniques may be appropriate. Other alternative procedures or a combination of approaches may be necessary to demonstrate control of infrequently performed tests.

7.7.1.2 For the individual determinands listed in Annex A (Tables 1 to 3) Organisations shall plot quality control results on appropriate control charts.

To monitor the variation of laboratory control samples, results shall be recorded or plotted on statistically based quality control charts.

Control charts should be set up using estimates of mean (M) and standard deviation (SD) obtained from results of at least 20 control samples obtained when the analytical system is under statistical control. Laboratories shall obtain this data during method validation procedures.

As further data are obtained, a new chart shall be produced based on the latest 60 to 100 results (depending on frequency of analysis), giving a new and more robust estimate of mean and standard deviation.

If any of the data points have breached the control rules and a cause is assigned (for example use of wrong standard, air in flow-cell), then it shall not be used in re-evaluation of chart limits. However, some results, which are part of the normal distribution, will breach the limits, and these shall be used where no specific reason for the breach can be assigned.

A senior member of staff shall review AQC performance on a regular basis. The timescale will depend on frequency of analysis. All significant changes shall be

investigated, even if precision and bias are still within the MCERTS targets. If a statistically significant change to precision or bias has occurred, then the new values shall be used in the control rules, and new control limits established and drawn on the control chart. Any decision made regarding updating of charts shall be justified and recorded.

At least annually, mean and standard deviation values shall be estimated from new data and checked to see if any significant changes have occurred. If necessary, the significance of a change in precision (as standard deviation) can be tested using an F test at the 95% confidence level, and if the mean (bias) has changed significantly using a student's t test, again at the 95% confidence level (see Annex C).

The targets given in Annex A of the MCERTS standard for a given parameter shall not be statistically significantly exceeded, but all significant changes shall be investigated, even if precision and bias are still within the MCERTS requirements. If the MCERTS targets are significantly exceeded and cannot be corrected, then a statistically significant change in performance has occurred, and it will be necessary to re-validate the analytical method.

Note: When uncertainty of measurement is reported, it should reflect performance of the method at that time, including current precision as reflected in control charts.

7.7.1.3 For all determinands listed in Annex A (Tables 1-3) quality control results shall be plotted on appropriate control charts.

7.7.1.4 Organisations shall have documented procedures that define loss of statistical control and specify actions to take (control rules) when control samples breach control limits. They shall investigate all breaches, record the findings and actions, and make them available to the Environment Agency, if requested. Organisations shall reanalyse samples in an analytical batch where control samples breach the defined control rules. If it is not possible and results are reported a full justification shall be given.

Organisations shall include the following checks in their investigations, but may need to carry out other checks:

- changes in concentration of stock standard solutions and reagents, and that they do not exceed expiry date
- calibration of instruments used in the analytical process
- documented methods were strictly adhered to
- that system suitability check data meet requirements
- significant drift does not occur for automated determinations
- service and fault records
- recent proficiency testing scheme results

Records shall include:

- identification of control sample and all associated sample results
- control rules in force at time of breach and breach result
- investigation details, conclusions, and actions taken
- action taken with respect to affected sample results (such as analysis repeated, or results reported)

7.7.2 Participation in interlaboratory comparison or proficiency-testing programmes

7.7.2.1 The organisation shall participate in an appropriate external quality control or inter-laboratory proficiency-testing scheme. Where possible, samples from the scheme organiser should reflect typical matrices and determinand concentrations analysed within the laboratory, or if appropriate on-site.

Note: The Environment Agency will encourage scheme organisers to provide appropriate samples (in terms of matrices, determinands, and concentrations of determinands) for distribution that reflect real-life situations.

7.7.2.2 The methods, used by the organisation to generate analytical data for the chemical testing of untreated sewage, treated sewage effluents and trade effluents which are submitted under MCERTS, shall be the same as those methods used by the organisation 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 shall be treated by the organisation in the same manner as normal routine samples submitted for chemical testing of untreated sewage, treated sewage effluents and trade effluents. For example, procedures for registration, storage, analysis, and the recording and reporting of results should be similar.

7.7.2.3 Full details of the scheme, including the number of samples, determinands and analyses to be undertaken by the organisation and the types of matrices to be analysed, shall be made available for audit. The reports of the results of all analyses submitted by the organisation to the scheme organiser shall be made available.

7.7.2.4 The organisation shall have a documented system in operation to review, investigate and address unsatisfactory results that are submitted to the proficiency scheme organiser, and examine trends in performance. If the organisation detects a significant deterioration in method performance and cannot correct it within a reasonable period of time the method should be re-validated.

This review procedure shall take into consideration the relevance of the matrices and concentration provided by the scheme, the number of other organisations participating in the scheme and whether these organisations use the same or similar analytical methods.

7.7.3

No additional requirements to EN ISO/IEC 17025.

7.8 Reporting of results

7.8.1 General

No additional requirements to EN ISO/IEC 17025.

7.8.2 Common requirements for reports (test, calibration, or sampling)

No additional requirements to EN ISO/IEC 17025.

7.8.3 Specific requirements for test reports

7.8.3.1 For data submitted to the Environment Agency for regulatory purposes, the report shall include appropriate information that clearly identifies and locates the sample relating to the results. This information shall record all data necessary to allow a complete audit trail. Relevant information includes:

- location of sample
- unique sample code or reference
- date and time sample taken
- name of organisation (including sampling organisation if different)
- name of any sub-contracting laboratories, if used
- date sample analysis completed
- determinands analysed, including any sample preservation or stabilisation at sampling site
- result of analysis
- other relevant comments, for example, visual characteristics of sample

Note: Some of this information may only be available from, or be able to be provided by, whoever commissions the analytical service or takes the samples and not the laboratory.

Results submitted to the Environment Agency shall be accompanied with a statement indicating whether the results have been recovery corrected or not. If corrected you shall explain the criteria used, including the manner of calculation.

Whenever possible and where appropriate, individual compounds should be analysed, and individual results reported. Where a group of similar compounds is analysed and the combined concentrations of these compounds are expressed as the sum of individual concentrations, the organisation shall record the number and identity of each compound analysed. This information shall be reported with the

results. If this approach is not possible or appropriate, the organisation shall define the analysis undertaken and the calculated result. This information shall be reported with the result.

7.8.3.2 No additional requirements to EN ISO/IEC 17025.

7.8.4 to 7.8.8 No additional requirements to EN ISO/IEC 17025.

7.9 Complaints

No additional requirements to EN ISO/IEC 17025.

7.10 Non conforming work

No additional requirements to EN ISO/IEC 17025.

7.11 Control of data – information management

No additional requirements to EN ISO/IEC 17025.

8 Management system requirements

No additional requirements to EN ISO/IEC 17025.

Annex A (normative): Performance characteristics

Table 1: Metals (total and dissolved)

In the table:

- although no LOD is specified, it shall be fit for purpose, especially, when compared to critical levels of interest
- discharges to controlled waters are treated sewage and trade effluent discharges
- where there are precision and bias targets for discharges to controlled waters and none for the other matrices, the precision and bias targets for discharges to controlled waters apply (but see clause 7.2.2.3)

Determinand	Discharge to controlled waters Precision (%RSD)	Discharge to controlled waters Bias (%)	Trade effluent discharge to sewer Precision (%RSD)	Trade effluent discharge to sewer Bias (%)	Untreated sewage Precision (%RSD)	Untreated sewage Bias (%)
Aluminium	5	10	7.5	10	-	-
Antimony	7.5	10	7.5	10	-	-
Arsenic	7.5	10	10	10	10	10
Barium	5	10	7.5	10	-	-
Beryllium	5	10	7.5	10	-	-
Boron	5	10	10	10	10	10
Cadmium	5	10	7.5	10	-	-
Calcium	5	10	7.5	10	-	-
Chromium	5	10	7.5	10	-	-
Hexavalent chromium	5	10	7.5	10	-	-
Cobalt	5	10	7.5	10	-	-

Determinand	Discharge to controlled waters Precision (%RSD)	Discharge to controlled waters Bias (%)	Trade effluent discharge to sewer Precision (%RSD)	Trade effluent discharge to sewer Bias (%)	Untreated sewage Precision (%RSD)	Untreated sewage Bias (%)
Copper	5	10	7.5	10	-	-
Iron	5	10	7.5	10	-	-
Lead	5	10	7.5	10	-	-
Magnesium	5	10	7.5	10		
Manganese	5	10	7.5	10	-	-
Mercury	7.5	10	7.5	10	10	15
Molybdenum	5	10	7.5	10		
Nickel	5	10	7.5	10	-	-
Potassium	5	10	7.5	10	-	-
Selenium	7.5	10	10	10	10	10
Silver	7.5	10	7.5	10		
Sodium	10	10	10	10	10	10
Strontium	5	10	7.5	10		
Thallium	7.5	10	7.5	10		
Tin	5	10	10	10	10	10
Titanium	5	10	7.5	10		
Uranium	5	10	7.5	10	-	-
Vanadium	5	10	7.5	10	-	-
Zinc	5	10	10	10		

Table 2: General determinands

In the table:

- although no LOD has been specified, it shall be fit for purpose, especially, when compared to critical levels of interest
- discharges to controlled waters are treated sewage and trade effluent discharges
- where there are precision and bias targets for discharges to controlled waters and none for the other matrices, the precision and bias targets for discharges to controlled waters apply (but see clause 7.2.2.3)
- for pH, precision and bias are in terms of pH units, not percentage

Determinand	Discharge to controlled waters Precision (%RSD)	Discharge to controlled waters Bias (%)	Trade effluent discharge to sewer Precision (%RSD)	Trade effluent discharge to sewer Bias (%)	Untreated sewage Precision (%RSD)	Untreated sewage Bias (%)
Alkalinity (to pH 4.5)	5	10	-	-	-	-
Ammonia	5	10	5	10	5	10
BOD	10	10	10	10	10	10
COD	5	10	5	10	5	10
Chloride	5	10	-	-	-	-
Chlorine (all forms)	10	10	-	-	-	-
Cyanide (all forms)	5	10	-	-	-	-
Detergents (anionic, MBAS)	7.5	10	-	-	-	-
Dissolved oxygen	2	2	-	-	-	-
Fluoride	5	10	-	-	-	-
Formaldehyde	5	10	-	-	-	-

Determinand	Discharge to controlled waters Precision (%RSD)	Discharge to controlled waters Bias (%)	Trade effluent discharge to sewer Precision (%RSD)	Trade effluent discharge to sewer Bias (%)	Untreated sewage Precision (%RSD)	Untreated sewage Bias (%)
Nitrite nitrogen	5	10	-	-	-	-
Nitrogen total oxidised	5	10	5	10	5	10
Nitrogen kjeldahl	5	10	5	10	5	10
Nitrogen total	5	10	5	10	5	10
Optical density	5	10	-	-	-	-
pH	0.2	0.2	0.2	0.2	0.2	0.2
Phosphorus total	5	10	5	10	5	10
Phosphorus soluble reactive	5	10	-	-	-	-
Specific conductivity	2	2	2	2	2	2
Sulfide	7.5	10	7.5	10	7.5	10
Sulfate	5	10	5	10	5	10
Suspended solids (105°C)	7.5	10	7.5	10	7.5	10
Turbidity	5	10	-	-	-	-

Table 3: Organics

In the table:

- although no LOD is specified, it shall be fit for purpose, especially when compared to critical levels of interest
- discharges to controlled waters are treated sewage and trade effluent discharges
- the data for 'explosive substances' covers explosive compounds listed as the "11 most common" in the R&D Technical Report P5-042/TR/03
- performance targets are for individual compounds within these groups. If a total (for example total PAH) result is requested, then each individual component should be determined and reported with the total
- where there are precision and bias targets for discharges to controlled waters and none for the other matrices, the precision and bias targets for discharges to controlled waters shall apply (see clause 7.2.2.3)

Determinand	Discharge to controlled waters Precision (%RSD)	Discharge to controlled waters Bias (%)	Trade effluent discharge to sewer Precision (%RSD)	Trade effluent discharge to sewer Bias (%)	Untreated sewage Precision (%RSD)	Untreated sewage Bias (%)
Acid herbicides	15	20	15	20	-	-
Alcohols/Ketones	10	15	-	-	-	-
Explosive substances	15	20	15	20	-	-
Hexachloro-1,3-butadiene	15	20	15	20	-	-
Hydrocarbon oils (Infra red)	10	12.5	10	12.5	10	12.5
Mothproofers	15	20	-	-	-	-
Nitroaromatics	15	20	-	-	-	-
Nonyl phenols	15	20	-	-	-	-
Organochlorine compounds	15	20	15	20	-	-

Determinand	Discharge to controlled waters Precision (%RSD)	Discharge to controlled waters Bias (%)	Trade effluent discharge to sewer Precision (%RSD)	Trade effluent discharge to sewer Bias (%)	Untreated sewage Precision (%RSD)	Untreated sewage Bias (%)
Organophosphorus compounds	15	25	15	25	-	-
Organotin compounds	15	20	-	-	--	-
Phenols	15	20	-	-	-	-
Phenols Monohydric colorimetric	6	10	-	-	--	-
Polyaromatic hydrocarbons	15	20	-	-	-	-
Polychlorinated biphenyls	15	20	-	-	-	-
Volatile organic compounds	15	20	-	-	-	-
Pyrethroids	15	20	-	-	-	-
Triazines	15	20	-	-	-	-
Urons/carbamates	15	20	-	-	-	-

Annex B (informative): Validation protocol

A typical validation protocol is described

Organisations shall only carry out performance tests to estimate precision, bias (recovery) and LOD on a stable analytical system. The following samples shall be put through the entire analytical procedure in a random order:

- matrix blank or sample with determinand concentration close to the expected LOD
- samples of appropriate matrices
- AQC material
- CRMs and samples of appropriate matrices plus spike if CRM not available

Each sample shall be analysed in duplicate, on 11 separate occasions (analytical batches) to guarantee at least 10 degrees of freedom but you may achieve 10 degrees of freedom in less than 11 batches. You should treat them as normal including the calculation of results.

You should estimate precision (within batch, between batch and total standard deviation) using ANOVA (analysis of variance) procedures for each solution. You should also estimate of the number of degrees of freedom associated with each total standard deviation. The following references give the appropriate procedures:

- ISO TR 13530: Water Quality – A Guide to Analytical Quality Control for Water Analysis
- A Manual on Analytical Quality Control for the Water Industry, R. V. Cheeseman and A. L. Wilson, revised by M. J. Gardner, NS 30, Water Research Centre, 1989. ISBN 0-902156-85-3

The total standard deviation should be compared with the appropriate precision targets listed in Annex A. If the value determined is greater than the target value, then it may be appropriate to ascertain if the difference is statistically significant using an F test at the $\alpha = 0.05$ level. The target standard deviation will be the denominator with infinite degrees of freedom. The procedure in Annex C2 shall be followed. If the difference is significant, then you may need further method development or to use of a different analytical method.

Recovery should be assessed as follows:

Calculate recovery for each pair of results, using the equation:

$$\text{Recovery (spiked samples)} = \frac{(C_m (V+W) - UV)}{C_s W} \times 100\ \%$$

Where:

- U = measured conc. in unspiked sample

- C_m = measured conc. in spiked sample
- C_s = conc. of spiking solution
- W = volume of spiking solution added
- V = volume of sample to which spike is added

Then calculate the mean recovery of each analytical batch. Calculate the mean recovery of all analytical batches and its standard deviation (s), the standard deviation of the 11 batch means.

The standard error (S) of this estimate of the mean recovery can now be calculated from:

$S = \frac{s}{\sqrt{m}}$where m is number of analytical batches, 11.

The true recovery should therefore lie in the range mean recovery ± $t_{(\alpha = 0.05)}$ S where $t_{(\alpha = 0.05)}$ = students t statistic at 95% probability with m-1 degrees of freedom.

LOD shall be calculated using the procedure outlined in Annex C.

Results of these validation tests can then be presented with method documentation in a tabular format.

Annex C (normative): Statistical analysis

C1 Limits of detection and reporting

C1.1 Introduction

We do not specify the LOD in this performance standard. But a common approach to the estimation of LOD is, to allow an evaluation of an organisation's performance in a consistent and comparable way. If data reported to the Environment Agency are to include results reported as less than values, the LOD shall be estimated using the following protocol.

C1.2 Choice of sample and sample pre-treatment

The sample used for estimating LOD should be as similar as possible to the matrix being analysed. Using a single sample for the determination of LOD for a given method will not take into account different matrix effects.

As a minimum LOD shall be estimated for one appropriate effluent matrix. It may be necessary to estimate LOD using different effluents for different methods due to background concentration of target determinands. When reporting results, it shall be made clear that the reported LOD may not be appropriate for samples with a 'complex' matrix.

If a more complex sample matrix is analysed, for example a crude sewage, and an estimation of matrix LOD is considered crucial, then the procedure for an on-going check in C1.3 shall be used.

Ideally analysis of the blank sample will produce normally distributed results scattered around zero, that is, both negative and positive results will be seen. It is usually possible for the blank sample to have a sufficiently small background concentration of the determinand to fulfil this requirement. However, this may not always be possible because in some analytical systems negative or low results cannot be obtained. In these cases, the blank sample should be spiked with a small amount of the determinand, sufficient to produce a small but significant response from the analytical system that is close to the expected LOD.

The blank or spiked sample shall be put through the entire analytical process (including, as necessary, extraction, clean-up, and measurement). The extraction and measurement of blank solutions based only on solvent or reagent blanks is not sufficient for estimating LODs for the purpose of satisfying MCERTS requirements. The blank samples or spikes shall be processed in the same way and using the same equipment and reagents as other samples in a batch.

Note 1: For commonly occurring substances such as iron, zinc, chloride, and sulfate, where waters may contain a significant amount of these substances, the method

used to determine an LOD for that substance using blank can give an optimistic (lower concentration) LOD. Alternatively, if an 'uncontaminated natural' sample is used to determine the LOD and it contains a significant amount of these substances then a pessimistic (higher concentration) LOD will be obtained.

Note 2: It is important that users of results should appreciate that the LOD for these common substances obtained by all MCERTS accredited organisations should be adequate for all these commonly (naturally) occurring substances. However, it is unlikely that LOD will be an issue with these substances, as adequate precision, and bias at the level of interest is more pertinent.

Note 3: For commonly occurring substances the variation in blank values should be consistent and within acceptable limits. Ideally all blank values for these substances should be less than 10% of the critical level of interest.

C1.3 Calculation

In this standard, LOD is defined by the equation:

$$LOD = 2\sqrt{2}.t_{(df,\ \alpha= 0.05)}.\ S_w$$

Where:

- df = number of degrees of freedom (minimum 10)
- t = one-sided Student's t-test statistic (95% confidence level)
- S_w = within-batch standard deviation of results from samples ideally containing negligible concentration of the determinand of interest

An estimate of the LOD can be made when initial validation studies are undertaken. Pairs of sample blanks shall be analysed in at least 10 different analytical runs or batches. Ideally these blanks should contain a negligible amount of the determinand being measured and should be consistent with and similar to the matrices of the samples being analysed. These sample blanks shall not be used as a calibration blank, and if the analytical procedure requires samples to be blank corrected, then the sample blanks used to estimate LOD should also be blank corrected.

Results shall not be rounded before being used for the estimation of LOD.

In the most general case, where m batches of different numbers of replicates n_i give a series of within-batch standard deviations, S_i:

The pooled value of S_w is given by:

$$S_w\,(pooled) = \sqrt{\frac{\sum {S_i}^2 \times (n_i - 1)}{\sum (n_i - 1)}}$$

Where:

- S_i= individual batch standard deviation
- n_i = number of results in the batch

Where the batches all contain the same number of results, this equation simplifies to:

$$S_w\,(pooled) = \sqrt{\frac{\sum {S_i}^2}{m}}$$ with m(n-1) degrees of freedom

for example, if at validation 11 batches of 2 blanks are analysed:

$$S_w\,(pooled) = \sqrt{\frac{\sum {S_i}^2}{11}}$$ with 11 degrees of freedom

Since $t_{(\alpha = 0.05)}$ for a one-sided t-test with 11 degrees of freedom is 1.796

Then LOD = $2\sqrt{2}.t.$ S_w = $5.08S_w$

Note: at infinite degrees of freedom the value of t (α = 0.05) becomes 1.645 and LOD = $4.65S_w$

If you use a different number of batches and replicates a minimum of 10 degrees of freedom shall be obtained. Where more than 10 batches of replicates are determined, all valid results shall be used in calculating the LOD. Further data shall be collected during routine analysis and pooled with the data obtained during validation to give a more robust estimation of LOD.

As an ongoing check, an estimate of LOD can be obtained by analysing 11 blank samples in the same batch, here SD (total standard deviation) equates to $S_{w,}$ (the within batch standard deviation), with 10 degrees of freedom. This procedure should be used when a matrix is analysed by a method that has not been fully validated for that matrix.

C1.4 Form of expression

For a multi-determinand method such as PAH, each individual PAH will need to have its own LOD estimated.

For TPH and similar determinands, it would not be appropriate to estimate the LOD using just one of the hydrocarbons within the analytical range. Blank sample data shall be generated in the same way as normal sample data to obtain the results used in estimating LOD.

LOD values shall always be reported in the same units as the determinands they represent. The calculated value may be rounded up for convenience and ease of use.

C1.5 Reporting limit

For the purposes of this MCERTS performance standard the reporting limit will be the LOD calculated as above. However, an organisation may use higher reporting limits than

calculated LODs. For example, an organisation calculated LOD for a method as <0.2 mg l^{-1} but prefer to report <1 mgl^{-1} due to issues with reporting software and customer requirements. This is acceptable to the Environment Agency, if LOD is calculated in the correct way and is substantially below any reporting limit. The consent value and critical level of interest shall be considered when making these judgements.

If you dilute samples before analysis, then the LOD shall be scaled up, that is, if a sample is diluted 1:5, and the analytical result is <5, then <25 should be reported.

C2 The use of statistical significance tests in the interpretation of method performance

C2.1 Introduction

Method validation aims to produce data on the precision of analysis and to provide an indication of any susceptibility to systematic error or bias.

Assuming that validation has been carried out as described in section 7.2.2 and that Analysis of Variance (ANOVA) has been applied to the results, there should be sufficient data to assess whether method performance complies with Annex A criteria (see section 7.2.2.3).

C2.2 Assessment of precision

The convention in analysis has been to consider precision to be satisfactory if the measured standard deviation is found not to be statistically significantly larger than the target standard deviation.

This implies there is uncertainty about the measured standard deviation value, although this uncertainty is minimised by specifying its calculation with at least 10 degrees of freedom.

Assessment of precision is in 3 stages:

1. Determine the target standard deviation at the concentration of interest, in accordance with section 7.2.2.3.
2. If the measured standard deviation is less than the target standard deviation, the target is achieved.
3. If the measured standard deviation is greater than the target it is still possible to comply with the requirements of this standard if it is not significantly greater. To assess this significance a statistical test is required.

C2.3 F-Test of standard deviation

The F-test or variance ratio test is a way of determining whether differences between 2 standard deviations are statistically significant (at a chosen probability level). The procedure is to calculate the F ratio as shown below:

$F = S_t^2 / Z^2$

where S_t is the measured total standard deviation, estimated using between batch and within batch mean squares in ANOVA, and Z is the target standard deviation.

Compare the calculated value of F with a reference value obtained from statistical tables. The reference value of F is obtained using the correct probability (5% for this performance standard) and using the relevant degrees of freedom for S_t and Z.

Z is a target standard deviation and therefore has infinite degrees of freedom. In the case of S_t, the number of degrees of freedom is calculated during the analysis of variance. For a complete 11x2 validation the equation can be simplified to:

$$df = \frac{110[M_1 + M_0]^2}{11{M_1}^2 + 10{M_0}^2}$$

where M_1 and M_0 are the within batch and between batch mean squares respectively, each obtained from ANOVA.

If the F ratio is less than the tabulated reference F value then the measured standard deviation is not significantly greater than the target value, that is, performance is satisfactory.

If the F ratio is greater than the tabulated reference F value, then the measured standard deviation is significantly greater than the target value, meaning performance is not satisfactory.

C2.4 Assessment of systematic error or bias

This assessment is only relevant and should only be carried out if the assessment of precision is acceptable.

The assessment of bias depends on independent knowledge of a ‘true’ value with which to compare the average of measured data. This is accomplished using reference materials or by spiking recovery experiments.

To assess bias and its associated uncertainty first calculate the mean recovery for each batch. Then use the batch mean recoveries to estimate the overall recovery and its standard deviation (strictly its standard error).

Significance is assessed by means of calculating the confidence interval about the mean and checking to see if this overlaps the limits of tolerable bias.

Overall Mean Recovery = $M = \Sigma R_i / m$

Standard error of recovery = $S_e = S_R / \sqrt{m}$

90% Confidence interval of recovery = $M \pm S_e \times t_{(0.05,\ m-1)}$

Where:

- m = number of batches
- R_i = %Recovery of the ***i***th batch
- S_R = standard deviation of batch recoveries
- $t_{(0.05,\ m-1)}$ = single-sided Student's t value at 5% probability level and (m-1) degrees of freedom

If the calculated recovery range overlaps with the required target bias range the recovery is not significantly different to the MCERTS requirement and is regarded acceptable.

Note: When you estimate a bias, it is either positive or negative, therefore a one-sided t-test at the 95% confidence level is used to assess if observed bias is greater than permitted bias. However, by definition, a confidence interval is two sided, therefore the significance test is at the 95% confidence level, but the resulting confidence interval is 90%.

C2.5 Example validation exercise

This example illustrates the application of the statistical tests in this Annex. It considers a spiking exercise for ammonia, a low-level spike of a sewage effluent, and a higher-level spike of an industrial discharge. Spiking solution concentration is 5000 mg l^{-1} N; for the sewage effluent sample 1 ml of this solution was made to 1 litre with sample, for the trade effluent sample, 3 ml of the spiking solution was made to 1 litre with sample.

	Sewage effluent	Spiked sewage effluent	Sewage effluent recovery	Trade effluent	Spiked trade effluent	Trade effluent recovery
Batch 1: Replicate 1	0.327	5.073	4.746	9.133	22.899	13.766
Batch 1: Replicate 2	0.45	5.311	4.861	9.55	22.33	12.78
Batch 1: Mean	0.3885	5.192	4.804	9.3415	22.6145	13.273
Batch 1: SD	0.08697	0.16829	0.08132	0.29486	0.40234	0.69721
Batch 2: Replicate 1	0.614	5.431	4.817	9.688	24.227	14.539
Batch 2: Replicate 2	0.519	5.138	4.619	9.376	23.38	14.004
Batch 2: Mean	0.5665	5.285	4.718	9.532	23.8035	14.2715
Batch 2: SD	0.06718	0.20718	0.14001	0.22062	0.59892	0.3783
Batch 3: Replicate 1	0.281	5.427	5.146	9.56	23.637	14.077
Batch 3: Replicate 2	0.412	5.394	4.982	9.417	24.336	14.919
Batch 3: Mean	0.3465	5.411	5.064	9.4885	23.9865	14.498
Batch 3: SD	0.09263	0.02333	0.11597	0.10112	0.49427	0.59538
Batch 4: Replicate 1	0.43	5.87	5.44	9.77	21.871	12.101
Batch 4: Replicate 2	0.557	6.086	5.529	9.564	21.039	11.475
Batch 4: Mean	0.4935	5.978	5.485	9.667	21.455	11.788
Batch 4: SD	0.0898	0.15274	0.06293	0.14566	0.58831	0.44265
Batch 5: Replicate 1	0.698	5.289	4.591	10.189	23.114	12.925
Batch 5: Replicate 2	0.744	5.899	5.155	10.882	23.565	12.683
Batch 5: Mean	0.721	5.594	4.873	10.5355	23.3395	12.804
Batch 5: SD	0.03253	0.43134	0.39881	0.49002	0.31891	0.17112
Batch 6: Replicate 1	0.495	5.395	4.9	10.055	23.389	13.334
Batch 6: Replicate 2	0.415	5.845	5.43	10.72	22.773	12.053
Batch 6: Mean	0.455	5.62	5.165	10.3875	23.081	12.6935
Batch 6: SD	0.05657	0.3182	0.37477	0.47023	0.43558	0.9058
Batch 7: Replicate 1	0.787	5.414	4.627	9.239	22.304	13.065
Batch 7: Replicate 2	0.57	5.735	5.165	9.678	23.836	14.158
Batch 7: Mean	0.6785	5.575	4.896	9.4585	23.07	13.6115
Batch 7: SD	0.15344	0.22698	0.38042	0.31042	1.08329	0.77287
Batch 8: Replicate 1	0.94	5.391	4.451	10.271	23.437	13.166
Batch 8: Replicate 2	0.647	5.201	4.554	10.31	23.736	13.426
Batch 8: Mean	0.7935	5.296	4.503	10.2905	23.5865	13.296
Batch 8: SD	0.20718	0.13435	0.07283	0.02758	0.21142	0.18385
Batch 9: Replicate 1	0.364	5.574	5.21	9.501	22.513	13.012
Batch 9: Replicate 2	0.49	4.934	4.444	10.149	23.835	13.686
Batch 9: Mean	0.427	5.254	4.827	9.825	23.174	13.349
Batch 9: SD	0.0891	0.45255	0.54164	0.45821	0.9348	0.47659
Batch 10: Replicate 1	0.434	5.102	4.668	9.802	22.552	12.75
Batch 10: Replicate 2	0.588	5.219	4.631	9.92	23.382	13.462
Batch 10: Mean	0.511	5.1605	4.65	9.861	22.967	13.106
Batch 10: SD	0.10889	0.08273	0.02616	0.08344	0.5869	0.50346
Batch 11: Replicate 1	0.516	5.249	4.733	10.172	22.952	12.78
Batch 11: Replicate 2	0.468	5.047	4.579	10.277	22.642	12.365
Batch 11: Mean	0.492	5.148	4.656	10.2245	22.797	12.5725

Batch 11: SD	0.03394	0.14284	0.10889	0.07425	0.2192	0.29345
Overall	0.53391	5.41	4.876	9.874	23.08	13.206

From the table:

- Sewage effluent sample overall mean (mean of batch mean values) is 0.53391 mgl^{-1}
- Spiked sewage effluent sample overall mean (mean of batch mean values) is 5.41 mgl^{-1}
- Spiked sewage effluent sample overall mean recovery (mean of batch mean recovery values) is 4.876 mgl^{-1}
- Trade effluent sample overall mean (mean of batch mean values) is 9.874 mgl^{-1}
- Spiked trade effluent sample overall mean (mean of batch mean values is 23.080 mgl^{-1}
- Spikes trade effluent sample overall mean recovery (mean of batch mean recovery values) is 13.206 mgl^{-1}

Precision test (from ANOVA)

In the table:

- the tabulated $F_{0.05}$ value is obtained from statistical tables for the estimated degrees of freedom at the 5% probability level (p=0.05)
- the F-value is calculated as (total SD / target SD)^2

	Sewage effluent	**Spiked sewage effluent**	**Trade effluent**	**Spiked trade effluent**
Mean	0.53391	5.410	9.874	23.080
Within-Batch SD	0.104619	0.249369	0.293543	0.594442
Between-Batch SD	0.121437	0.186605	0.365231	0.534918
Total SD	0.160288	0.311459	0.468574	0.799687
Relative SD %	30.02%	5.76%	4.75%	3.46%
Target SD (5% of mean)	0.125	0.2705	0.4937	1.154
$F_{0.05}$ from tables	1.67	1.60	1.69	1.64
F-Value calculated	1.64	1.33	0.90	0.48
Estimate degrees freedom	15.14	18.02	14.68	16.86

	Sewage effluent	Spiked sewage effluent	Trade effluent	Spiked trade effluent
Assessment	PASS	PASS	PASS	PASS

In this example the precision in terms of the observed relative standard deviation of the sewage effluent is much higher than the target value of 5%, so we need to do an F test. For this sewage effluent, the CLOI is 5 mgl^{-1} so we can increase the target standard deviation to one-fortieth of the CLOI (that is 0.125 mgl^{-1}). The 95% calculated F value (1.64) for the sewage sample is less than the tabulated reference F value of 1.67. So, the standard deviation of the sewage sample is not significantly different from the target value, and thus meets the MCERTS requirement.

With the spiked sewage effluent, the observed relative standard deviation (5.76%) is higher than the 5% target value of the mean (that is 0.2705). Following the F test calculation, the data for the spiked sewage sample passes and so meets MCERTS requirements. Again, the sample passes the F test. The trade effluent sample and spiked trade effluent are within the target values, so no need for the F test.

Recovery for high sample

In the table, the:

- mean measured value is the average of the mean recovery for each batch
- standard error of mean recovery is the relative SD of overall mean recovery divided by the square root of the number of batches
- 90% confidence interval of recovery is the standard error of mean recovery multiplied by the student's t value (p=0.05 single sided) for degrees of freedom equal to number of batches minus 1, (t=1.812 for 11 batches)

	Sewage effluent	Trade effluent
Expected recovery concentration	4.9995	14.9704
Mean measured recovery	4.8763	13.2057
Overall mean recovery	97.5%	88.2%
SD of mean recovery	5.5192	5.11
Standard error of mean recovery	1.664	1.5402
90 % Confidence interval of recovery	3.015	2.7909

Recovery range	94.52% to 100.55%	85.42% to 91.0%
Assessment	PASS	PASS

The bias target value for ammonia is 10%, so the tolerable range of recovery in this example is 90 to 110%. At 97.5% the sewage sample is well within this range. In the case of the trade effluent sample, the overall mean recovery is lower than the tolerable range. However, the overlap of the confidence interval with the tolerable range means that although recovery is nominally outside this range it is not significantly so and is therefore statistically acceptable**.**

The precision must be acceptable before you can apply this test.

Annex D (normative): Validation and use of portable instruments and test kits

D1. Introduction

If an organisation uses portable instrumentation or test kits in the field for regulatory compliance monitoring of effluents, then procedures and practices shall comply with MCERTS and ISO 17025.

Where available, organisations should use instruments certified to the MCERTS 'Performance Standards and Test Procedures for Portable Water Monitoring Equipment' they shall follow manufacturer's instructions for calibration and operation as appropriate.

All test kits and instruments with or without MCERTS certification to the MCERTS 'Performance Standards and Test Procedures for Portable Water Monitoring Equipment,' shall undergo a validation procedure, as well as routine calibration and AQC, to make sure they can achieve the required performance.

One of the main criticisms of the use of portable instruments and test kits in the field is the lack of training given to staff that use them. Hence inconsistent and erroneous results from their misuse often occur, which is not acceptable for regulatory compliance monitoring. Organisations shall use manufacturer's training resources if available and appropriate. At least one member of staff shall be fully trained in the use of the instrument and/or test kit, have a good understanding of its basis of operation, fault finding and quality control, and be able to train others in its use. All who operate portable instruments and test kits shall have a training record including objective evidence of competency.

Operating procedures shall be fully documented and available in the field for users.

As the analytical systems are used outside of a controlled laboratory environment, organisations should give particular care to their cleaning, storage, and maintenance.

D2. Validation

pH, specific conductivity, and dissolved oxygen field instruments

Organisations shall carry out the full validation procedure detailed in clause 7.2.2 for each model and probe or electrode combination in use. For each determinand one validation exercise could encompass all instruments used in the field if all the model and probe or electrode combinations are identical. Validation may be performed under laboratory conditions. If it is not appropriate to use spiking experiments, you may use matrix samples and standards.

Carry out further limited verification tests on any additional instrumentation (if it has the same model and probe/electrode combination) using the further validation procedures in clause 7.2.2.3. You can do this after instrument calibration before first use in the field. You must use an appropriate standard and one appropriate matrix sample. Seven replicates of each are acceptable.

For dissolved oxygen it is acceptable to test matrix solutions at 0% and 100% oxygen saturation. Precision and bias targets only need to be met at 100% saturation.

Chlorine test kits

Organisations shall carry out the full validation procedure detailed in clause 7.2.2 for each model in use. Organisations shall carry out further limited verification tests on any additional instrumentation (same model) using the further validation procedures in clause 7.2.2. You can do this after instrument calibration before first use in the field. You shall use an appropriate standard and one appropriate matrix sample. Seven replicates of each is acceptable.

D3. Performance requirements

Use the performance requirements in Annex A.

For pH and conductivity bias can be determined from standard solutions used in validation. Precision shall be determined from samples used in validation.

For dissolved oxygen bias can be estimated by comparison with Winkler titrations, for which you shall demonstrate traceability.

D4. Calibration

Organisations shall:

- uniquely identify and record each piece of equipment
- draw up a calibration timetable, and clearly label each instrument as to when recalibration is required. It may not be necessary to calibrate pH and Electrical Conductivity [EC] meters daily (see AQC checks)
- keep a record of calibration events
- for pH, conductivity, and dissolved oxygen measurements, also calibrate thermocouples and thermometers
- apply Clause 6.4

D5. AQC requirements

Quality assurance checks using AQC samples shall be carried out during sampling runs when the instrument or test kit is in use. Results shall be recorded and plotted on

appropriate control charts after analysis of the AQC and checked against current control chart limits before the associated sample results are reported.

Clauses 7.7.1.2 and 7.7.1.4 shall apply where appropriate.

Note 1: An example of good practice is measuring an AQC sample at the beginning of the day before the first sample reading is taken and at the end of the day after the last sample has been analysed. Other approaches can be used if adequate control can be demonstrated.

Sufficient AQC samples should be measured to ensure that AQC samples comprise at least 5% of the samples measured.

If making pH measurements in low conductivity samples then a low conductivity pH check solution shall be used.

Note 2: For conductivities of <100μS.cm^{-1} pH 4 dilute acid buffers are recommended. Other buffers are available for samples with conductivity around 500μS.cm^{-1}. Some electrodes may not be appropriate for measurement of pH in low conductivity waters.

If measuring specific conductivity in low conductivity or saline water, then an additional more appropriate conductivity AQC sample shall be used.

Note 3: It is possible to use the same check and AQC solutions for conductivity and pH.

In addition, for all instruments, manufacturer recommended system suitability checks shall be carried out and the results recorded.

Proficiency testing shall be undertaken for all determinands for which appropriate schemes are available and shall be undertaken on-site.

D6. Instrument care

The following procedures shall be documented, and where appropriate records of implementation shall be kept:

- storage conditions for instruments and probes, when in use and when not, short term and long term
- replacement of consumables, such as reagents, O-rings, and membranes
- cable and connector inspections and replacement
- cleaning of instruments and probes
- updating firmware and software

D7. Temperature

Temperature measurement is not in Annex A but accreditation for the MCERTS (waters) standard can be granted for this determinand provided the relevant requirements of ISO 17025 are met.