

SECTION 5: GUIDANCE ON ANALYSIS



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Section 5: Guidance on Analysis

Summary of Section 5

- ◆ Sets out the requirements of the Regulations on analysis and emphasises the importance of accreditation of laboratories.
- ◆ Advises Water Services Authorities (WSAs) and their laboratories on the competence, training and supervision of analysts and the monitoring and audit of analysts' performance.
- ◆ Provides guidance on the storage and preservation of samples in the laboratory.
- ◆ Describes the criteria for the suitability of laboratory equipment.
- ◆ Sets out the regulatory requirements for the performance of analytical methods.
- ◆ Provides advice on how to determine the performance of analytical methods for specified parameters, on those parameters for which analytical methods are specified and on those parameters for which performance is not specified.
- ◆ Sets out the requirements of the Regulations on analytical quality control.
- ◆ Provides advice on the internal and external analytical quality control procedures to satisfy the regulatory requirements.
- ◆ Advises WSAs and their laboratories on the calibration of analytical systems.
- ◆ Provides advice on how to correct analytical results for recovery losses when analysing for organic parameters.
- ◆ Provides advice on what information to retain in the laboratory's records of analysis.
- ◆ Describes the importance of the integrity of analytical results and advises on how to ensure integrity.
- ◆ Provides brief details of the annual reporting of analytical results to the Environment Protection Agency (the EPA).

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1. Introduction

1.1 | Part 3 of the schedule to the Regulations states that *“Each laboratory at which samples are analysed must have a system of analytical quality control that is subject from time to time to checking by a person who is not under the control of the laboratory and who is approved by the Agency [the Environment Protection Agency*

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(the EPA)] for that purpose". Part 3 also specifies the methods that must be used for the microbiological parameters and the performance that must be achieved for the non-microbiological parameters in terms of trueness, precision and limit of detection.

1.2 | Laboratories may satisfy these requirements and the guidance in this section for particular parameters if they have gained **accreditation for those parameters to the ISO/IEC Standard 17025 "General Requirements for the Competence of Calibration and Testing Laboratories"**. Assessment for compliance with the above standard is carried out by the **Irish National Accreditation Board (INAB)**. This International Standard contains all the requirements that laboratories have to meet to demonstrate that they are operating a quality management system and are able to produce valid analytical results. Laboratories that have not gained this accreditation from INAB will need to demonstrate to the EPA, or a person or organisation authorised by the EPA, that they have an appropriate quality management system in place and that they satisfy the requirements of the Regulations and the guidance in this section. The key requirements of a quality management system include document control of all procedures and analytical methods used in the laboratory, standards for sub-contracting analysis to another laboratory, procedures for dealing with complaints about the service, satisfactory laboratory accommodation, a self-assessment process including internal audit and management review, integrity and impartiality, valid test procedures, competence of personnel and traceability of measurements.

1.3 | **The EPA strongly recommends that laboratories carrying out analysis of drinking waters attain accreditation to ISO 17025 for all parameters** and as such the EPA considers that laboratories that have attained such accreditation will satisfy the requirements of Part 3 of the schedule to the Regulations. **EPA recommends that laboratories carrying out analysis for determining compliance with the water quality standards should aim to be accredited by the end of 2012 and that all analysis must be carried in accredited laboratories by the end of 2015.** The EPA will not accept monitoring results from unaccredited laboratories after the end of 2015. Meanwhile laboratories that have not attained accreditation to ISO 17025 should use the guidance in this section as the basis for the development of such a system in order to satisfy the requirements of Part 3. Such laboratories shall be subject to checking by the EPA or a person approved by the EPA to permit the EPA to ascertain whether the laboratory has a suitable system of analytical quality control in place and meets the specified performance requirements.

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2. Competency and training of analysts

2.1 | Water Services Authorities (WSAs) and their laboratories or their contract laboratories should ensure that samples are analysed by, or under the supervision of, a person who is competent to perform that task. As many laboratories will have some staff with only basic technical qualifications and limited experience in water analysis, the organisational and management structure of the laboratory is important. The following should be included in the laboratory structure:

- ◆ the laboratory manager is supported by an adequate number of qualified staff, trained in the principles and practice of relevant areas of analysis;
- ◆ there is a nominated deputy for the manager who is suitably qualified and experienced;
- ◆ an up-to-date record is kept of the structure and organisation of the laboratory;
- ◆ an up-to-date record is kept of the qualifications, experience and training of each member of staff;
- ◆ the proportion of senior to junior staff is such as to ensure a satisfactory level of supervision;
- ◆ unqualified temporary staff are adequately supervised and the proportion of unqualified staff to qualified staff does not impair the quality of analysis performed; and
- ◆ there is a suitably qualified quality control manager responsible for all quality control activities in the laboratory and who has direct access to senior management outside the laboratory.

2.2 | In order to carry out monitoring of drinking water quality correctly it is essential that all analysts are fully trained and competent before they are allowed to work unsupervised. WSAs and their laboratories or their contract laboratories should produce a comprehensive analyst training manual and programme to cover all aspects of analysis that as a minimum should include:

- ◆ the criteria for selection of persons suitable to train as analysts, if necessary sub-divided by type of analysis;

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- ◆ the relevant principles and practice of analysis, including calibration and internal and external analytical quality control;
- ◆ supervised training and experience of the relevant analytical systems;
- ◆ the criteria and method of assessment of competence to work supervised and unsupervised;
- ◆ the criteria and method of assessment of competence for senior analysts to train, audit and supervise others;
- ◆ the monitoring/audit of trained analysts to check that they continue to perform satisfactorily and the criteria for satisfactory performance;
- ◆ re-training when performance is not satisfactory; and
- ◆ an annual review of each analyst's training to assess whether further training is necessary.

2.3 | All analysts should have:

- ◆ a copy of the analytical methods that they are trained to use and access to a copy of the laboratory analysis manual;
- ◆ been trained in all the relevant analytical methods that they are, or could be, required to carry out;
- ◆ been trained in the principles and practices of calibration of equipment and methods and in analytical quality control; and
- ◆ a training record that sets out clearly those procedures and practices in which they have been trained, the dates and results (competency) of that training, the dates and results of audits of training and any re-training and the results of the annual review.

2.4 | Analysts should not carry out analytical procedures unless they have been successfully trained to an acceptable standard or they are being supervised by a competent and experienced analyst as part of their training.

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3. Sample storage and preservation

3.1 | Samples must be transported to the laboratory with the minimum of delay in an appropriate sampling vehicle under appropriate conditions (see paragraph 2.2 of section 4 of this handbook). The laboratory manual should contain written instructions for the storage and preservation of samples or sample portions that include:

- ◆ adequate refrigerated storage capacity and precautions to ensure that samples are not contaminated;
- ◆ monitoring and recording of refrigerator temperature;
- ◆ commencing and carrying out sample preservation within the maximum acceptable time, when it has not been carried before or at the time of sampling and it is necessary;
- ◆ procedures for dividing samples into portions and preserving such sample portions when necessary within the maximum acceptable time, when sample portions are required prior to analysis;
- ◆ clear labelling of preserved and unpreserved sample portions and preserved and unpreserved samples;
- ◆ commencing analysis within the maximum acceptable time when sample preservation has been carried out before or at the time of sampling;
- ◆ not analysing samples and sample portions that have not been preserved in sufficient time; and
- ◆ a requirement to carry out “blank checks” on reagents and/or apparatus used for sample preservation and for action to be taken in the event of an unsatisfactory blank.

3.2 | Further guidance on appropriate sample bottle types, sample preservation techniques and sample storage conditions is given paragraph 2.2 of section 4 of this handbook.

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4. Suitability of analytical equipment

4.1 | The analytical equipment (including the principal apparatus and all standard laboratory apparatus such as balances, glassware, thermometers, incubators etc) should be of the type specified in the analytical method and it should comply with each of the following criteria before it can be regarded as suitable for the purpose:

- ◆ located and used in appropriate conditions;
- ◆ maintained and serviced according to the manufacturer's or supplier's instructions or recommendations or equivalent procedures that are auditable;
- ◆ operated according to the manufacturer's or supplier's instructions or recommendations or equivalent procedures that are auditable;
- ◆ calibrated according to the manufacturer's or supplier's instructions or recommendations or equivalent procedures that are auditable;
- ◆ have a current calibration that is both valid and traceable to national or international standards; and
- ◆ all system suitability and analytical quality control criteria.

4.2 | Further guidance is given in ISO/IEC Standard 17025 "General Requirements for the Competence of Calibration and Testing Laboratories".

5. Performance of analytical methods

5.1 Introduction

5.1.1 | In order to ensure the accuracy of the results of monitoring drinking water quality, it is an essential requirement of the Regulations that laboratories use either the specified methods or alternative methods approved by the EPA (for microbiological parameters) or methods which meet the performance characteristics (trueness, precision and limit of detection) set out in part 3 of the schedule to the Regulations (for chemical and other parameters) and that they operate a system of analytical quality control that is checked by a person who is not under the control of the laboratory and who is approved by the EPA. For some indicator parameters there is no numerical indicator parameter value but there is a descriptive value either "no abnormal change"

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or “acceptable to consumers and no abnormal change”. For these parameters an analytical method or the performance to be achieved by an analytical method is not specified.

5.1.2 | Each laboratory should have tested the performance of the analytical methods used for each parameter or each determined constituent of a parameter (for chemical and other non-microbiological parameters), and to have demonstrated that the method is capable of meeting the performance requirements set out in Part 3 of the schedule to the Regulations before that method is used for routine analysis of compliance samples. Performance testing should cover the entire analytical method, including any sample preparation and concentration steps. Performance testing should be carried out in a manner emulating that used routinely, without taking special precautions that would not generally apply to achieve optimum performance. An analytical method is the specific combination of laboratory, analysts, instrumentation and analytical procedure used to analyse the sample, including any sample preparation or pre-treatment steps. Provided all analysts have been trained to the same standard and their competence has been assessed using the same criteria they can be regarded as equivalent for the purposes of initial performance testing of the analytical method.

5.1.3 | Laboratories may satisfy the performance requirements of the Regulations and the guidance in this section for particular parameters if they have gained accreditation for those parameters to the ISO/IEC Standard 17025 “General Requirements for the Competence of Calibration and Testing Laboratories” from the Irish National Accreditation Board (INAB). This is amplified in paragraph 1 of this section.

5.2 Parameters for which performance is not specified

5.2.1 | For the following parameters an analytical method or the performance to be achieved by an analytical method is not specified. The EPA advises the following:

Colour: qualitative assessments of the colour of water on different sampling occasions are unlikely to enable “no abnormal change” to be detected. WSAs and their laboratories should use an appropriate quantitative method for determining colour in mg/l Pt/Co that has a trueness, precision and limit of detection each equal to or better than 2 mg/l Pt/Co;

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Odour and taste: quantitative assessments of the odour and taste of water are time consuming and require a specialist panel of persons to smell and taste samples. Qualitative assessments by an experienced analyst are likely to be able to detect abnormal changes and therefore be able to determine whether the regulatory requirement of “no abnormal change” has been met. Analysts carrying out qualitative assessments of odour and taste must avoid in a period prior to the assessment activities that could affect the assessment, such as smoking, drinking and eating and wearing excessive cosmetics. **Taste assessments should not be carried out on any supply that is not disinfected or where disinfection is practised but may not be effective;**

Colony count at 22°C: WSAs and their laboratories should use the method in ISO 6222 for the enumeration of culturable micro-organisms or an alternative method approved by the EPA;

Total organic carbon (TOC): WSAs and their laboratories should use an appropriate quantitative method for determining TOC in mgC/l that has a trueness, precision and limit of detection each equal to or better than 0.5 mgC/l; and

Turbidity: qualitative assessments of the turbidity of water on different sampling occasions are unlikely to enable “no abnormal change” to be detected. WSAs and their laboratories should use an appropriate quantitative method for determining turbidity in nephelometric turbidity units (NTU) that has a trueness, precision and limit of detection each equal to or better than 0.25 NTU.

5.3 Parameters for which performance is specified

5.3.1 | For the most of the non-microbiological parameters, the methods of analysis are not specified in the Regulations. Instead the Regulations specify the performance to be achieved by the methods of analysis. **WSAs and their laboratories may use any analytical methods they wish provided they meet the performance specifications.** The table in appendix 1 reproduces the Regulations and sets out the performance characteristics (trueness, precision and limit of detection as a percentage of the standard or parametric value) that the methods of analysis used must, as a minimum, be capable of measuring.

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Initial performance testing

5.3.2 | The analytical method should be subjected to testing of its trueness, precision and limit of detection, including spiking recovery. **Laboratories should have a written procedure for the initial performance testing and validation of methods and the results should be kept for audit purposes.** The specifications for these performance characteristics are given in appendix 1 of this section. In addition any method that is not referenced to a fully validated authoritative method should be subjected to testing of its resilience against possible interferences. The minimum acceptable specifications for performance testing are given in the paragraphs below. The design of tests and calculation of performance characteristics should be in accordance with the guidance given in for example the UK publication 'A Manual of Analytical Quality Control for the Water Industry' (NS30) or any equivalent publication.

5.3.3 | A laboratory using an analytical method that is not referenced to a fully validated authoritative method should demonstrate that the method has been fully documented and tested to the standard currently expected of an authoritative reference method. It should demonstrate that the following have been established:

- ◆ the required tolerances of all measurements undertaken within the method (volumes, temperatures, masses etc);
- ◆ the forms of the determinand measured, including speciation;
- ◆ the effect of interferences has been widely investigated and quantified; and
- ◆ significant sources of error have been identified and adequate means of controlling them documented.

5.3.4 | For most parameters the minimum specification for the performance characteristics to be determined is as follows. Estimate the within-laboratory total standard deviation of individual analytical results for blanks, standard solutions, samples and spiked samples on at least 5 separate days (further advice on number of batches and period of testing is given in the paragraphs below). The number of replicate determinations of each solution in each batch should be the same and not less than two. All estimates of standard deviation used to estimate limit of detection or precision, or used in significance tests should have at least 10 degrees of freedom. The trueness for standard solutions, mean spiking recovery and standard deviation of spiking recovery should also be determined.

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Limit of detection is to be calculated as:

- ◆ three times the relative within batch standard deviation of a natural sample containing a low concentration of the parameter; **or**
- ◆ five times the relative within batch standard deviation of a blank sample.
- ◆ **Precision** (the random error) is to be calculated as twice the standard deviation (within batch and between batches) of the spread of the results about the mean.
- ◆ **Trueness** (the systematic error) is to be calculated as the difference between the mean value of the large number of repeated measurements and the true value.

5.3.5 | The range of the standard solutions tested should include the concentration or value of the parameter in tables A and B (the standards) and table C (the indicator parameter values) in the schedule to the Regulations wherever possible, but in all cases the whole calibrated range of the method should be covered subject to allowance for ensuring that all measurements fall within the calibrated range. This implies that a minimum of two different standard solutions should be included in the performance tests. All standard solutions should be prepared immediately prior to analysis for each batch, either from the pure substance or a stock solution that is known to be stable for the period of the tests.

5.3.6 | The sample(s) and spiked sample(s) selected for use should represent the type or types of drinking water normally analysed. The same bulk sample(s) should be used throughout the tests. Samples should be spiked immediately before analysis for each batch. The spiking standard should either be known to be stable for the period of the tests or be prepared in the same way as for standard solutions.

5.3.7 | Where there is a choice of key instruments, including electrodes and chromatographic columns, each combination used should be regarded as a separate analytical method. For instruments that are not identical full testing should be carried out for each analytical method. For identical instruments full validation should be carried out for each method except where the results of limited testing of the instruments under the conditions used in the analytical method have demonstrated that there is no statistically significant (at the 95% confidence level) difference in performance between the instruments, in which case only one method requires full validation. The tests should be performed on a minimum of five separate days and include the analysis of typical real samples and spiked samples. Limited testing should be appropriate for

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electrodes or chromatography columns from the same manufacturer or supplier. If the internal AQC record subsequently shows a significant difference in performance between methods each method should then be fully tested. Alternatively, independent data may be available, for example from the manufacturers or suppliers, to demonstrate the equivalence of items such as electrodes and chromatographic columns.

5.3.8 | WSAs and their laboratories should note that 5 batches of duplicate analyses cannot give 10 degrees of freedom. While many combinations of number and size of batch may give 10 degrees of freedom, a minimum of 11 batches is required to guarantee that number of degrees of freedom, irrespective of the number of replicates included in the batch. Laboratories are therefore strongly recommended to adopt 11 batches of duplicates as their minimum specification. The formula for calculating the number of degrees of freedom is given on page 57 of NS30 (or equivalent publication). A laboratory may however check whether at least 10 of degrees of freedom have been achieved by performing the calculation any time after at least 6 batches of duplicate analysis have been carried out provided they have been done on at least 5 separate days.

5.3.9 | For methods where the discrimination of the method is insufficient to record values other than zero for most blank determinations, the within-batch standard deviation of either the low standard solution or the within batch standard deviation of the sample may be used to calculate the limit of detection. Alternatively, a very low standard solution, at a concentration approximately two to three times the expected limit of detection when using the best currently available method, may be used as a surrogate blank. Some methods, particularly those involving simple titrations or the use of colour comparators, may be incapable of measuring any within-batch differences. In such cases the limit of detection should be quoted as the lowest measurable concentration or value.

5.3.10 | The bulk sample may not always be stable over the entire period of testing, resulting in an artificially high estimate of between-batch standard deviation. This instability may be recognised by a distinct trend in results for the sample over the period of testing and a between-batch standard deviation that, statistically, is significantly greater (at the 95% confidence level) than would be expected from the estimates obtained for the standard solutions. In such cases a surrogate between-batch standard deviation should be calculated using procedure (a) on page 53 of NS30 (or equivalent publication). Where the instability is so great that the estimate of within-batch standard deviation is significantly affected it may be possible to improve stability

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by ageing of the sample. Where ageing is either impractical or ineffective in reducing sample instability sufficiently to avoid a statistically significant effect on the estimate of within-batch standard deviation, procedure (b) on pages 53 and 54 of NS30 should be used (or equivalent publication).

5.3.11 | The period of testing should be continuous and not unduly long. Not more than 2 batches may be analysed on any day. When 2 batches are analysed on the same day all instruments used should be shut down to overnight conditions, daily reagents freshly prepared and all test solutions freshly prepared between the first and second batches.

5.3.12 | For physical parameters for which values are not truly additive spiking recovery tests may yield little useful information and need not be done. It is not possible to either analyse a blank or do spiking recovery tests for hydrogen ion concentration (pH value). For these parameters the calibrated range (or ranges) must include the full range of values encountered and the value in table B (the standards) and table C (the indicator parameter values) in the schedule to the Regulations.

5.3.13 | Methods may be used for compliance monitoring against the standards and indicator parameter values in the Regulations once it has been established that the performance characteristics determined by the procedures set out above meet the specifications for trueness, precision and limit of detection in part 3 of the schedule to the Regulations and set out in appendix 1 of this section.

Re-determination of performance characteristics

5.3.14 | Once a method is in routine use it will be necessary from time to time to re-determine its performance for a variety of reasons to make sure it still meets the performance characteristics in part 3 of the schedule to the Regulations. The performance characteristics of an analytical method should be re-determined whenever a significant change has occurred such as a change in:

- ◆ the analytical procedure used (a);
- ◆ the key equipment used (b);
- ◆ the laboratory environment I; or

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- ◆ a change of staff carrying out the procedure (this does not include routine changes that normally occur within the laboratory that are supported by appropriate training and properly trained supervisors) (d).
- ◆ The significance of any change should be assessed by a competent analyst, and any decision that a change is not significant supported by the results of limited but adequate testing.

5.3.15 | When a change of premises occurs it is not always possible to revalidate all analytical methods before they are used. In such cases it is essential that methods which on transfer also undergo a change of one of the types (a), (b) and (d) in paragraph 5.3.14 above are revalidated before they are used, as should those which are known to be susceptible to changes in laboratory environment e.g. ammonium and trihalomethanes. Other analytical methods should normally be revalidated within three months of relocation.

5.3.16 | The performance characteristics of analytical methods should also be re-determined whenever the results of routine analytical quality control (AQC) (internal or external) indicate that a statistically significant deterioration in performance has occurred which cannot be corrected, or that there is a significant discontinuity in the routine AQC record, whether due to a failure to perform routine AQC or disuse of the analytical method. Laboratories may also wish to re-determine the performance characteristics whenever routine AQC indicates that a statistically significant improvement in performance has occurred. Statistical significance should normally be assessed at the 95% confidence level.

5.3.17 | When an analytical method has been in continuous use for several years, typically between three and five years, without re-determination of performance characteristics, the method should be re-evaluated and the need for re-determination of the performance characteristics considered.

5.4 Microbiological parameters

5.4.1 | The Regulations do not specify the performance to be achieved by the methods to be used for determining the microbiological parameters because performance cannot be specified in the same way as for non-microbiological parameters. Instead the Regulations require that WSAs and their laboratories or contract laboratories must use the methods for microbiological parameters specified in section 1 of part 3 of the

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schedule to the Regulations unless an alternative method has been approved by the EPA, in which case the authorised alternative may be used subject to any conditions given in the approval. Appendix 2 reproduces the specified methods for the microbiological parameters in the Regulations.

5.4.2 | Any WSA or laboratory wishing to use an alternative method that has not been approved must first make an application in writing to the EPA and must include a full description of the method to be used along with results of tests demonstrating both the reliability of the method and its equivalence to the specified method. Further information on the testing requirements and criteria to demonstrate equivalence are given in ISO/TR 17944:2004 – Water Quality – Criteria for establishing the equivalence between microbiological methods. An alternative method will only be approved if it is adequately documented and the results of tests demonstrate to the satisfaction of the EPA that results obtained using the method are at least as reliable as those produced by the use of the prescribed method. The EPA may make any approval subject to such conditions as it considers appropriate, e.g. limitation of the types of sample matrix it may be used to analyse or specific extra quality control requirements.

5.4.3 | The EPA is satisfied that the results obtained by the Idexx (Colilert 18) Quanti-Tray™ method for coliform bacteria and *E. coli* are at least as reliable as the results obtained by the method specified in the Regulations (ISO 9308-1). Therefore laboratories may use the Idexx (Colilert 18) Quanti-Tray™ method instead of the ISO 9308-1 method specified in the Regulations.

6. Analytical quality control (AQC)

6.1 Introduction

6.1.1 | Part 3 of the schedule to the Regulations states that *“Each laboratory at which samples are analysed must have a system of analytical quality control that is subject from time to time to checking by a person who is not under the control of the laboratory and who is approved by the Agency [the Environment Protection Agency (the EPA)] for that purpose”*. It follows that each laboratory must operate a system of routine internal AQC when analysing batches of samples for each parameter. Each laboratory should participate in external AQC schemes (proficiency testing schemes) if such schemes are available. The EPA operates a suitable scheme for some parameters.

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6.2 Non-microbiological parameters

Routine internal AQC

6.2.1 | As a minimum, the laboratory should run with each batch of samples an analytical quality control solution that contains a known concentration at, or close to, the standard or indicator value for each parameter or determined constituent of a parameter for each analytical method, except as provided for below. The term “close to the standard or indicator value” should be interpreted as meaning the standard or indicator value $\pm 25\%$. The frequency of use of AQC solutions will vary according to the particular analytical technique used but normally between five and twenty percent of all samples analysed should be AQC solutions, subject to a minimum of one per batch of analyses for batches of less than 20 samples. All AQC solutions should be subject to the full analytical procedure that is used for analysing samples and analysed with each batch of analyses.

6.2.2 | For permanent laboratory tests a “batch of analyses” should be regarded as a group of measurements or observations of standards, samples and/or AQC solutions that have been performed together in respect of all procedures, either simultaneously or sequentially, by the same analysts using the same reagents, equipment and calibration. For field tests (such as pH and conductivity tests) a “batch of analyses” should be regarded as a group of measurements or observations of standards, samples and/or control solutions which have been performed on the same day by the same analysts using the same reagents, equipment and calibration.

6.2.3 | In the following cases the guidance on selection of AQC solutions given above is not appropriate:

- ◆ the standard or indicator parameter value represents a concentration or value outside the normal analytical range of a particular method;
- ◆ there is no standard or indicator parameter value;
- ◆ the indicator parameter value is descriptive; or
- ◆ the indicator parameter value is a range.

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In these cases, as a minimum, an AQC solution with a known concentration or value within both the calibrated range of the method and the range of interest should be used.

6.2.4 | When a wide range of concentrations or values is calibrated that includes the standard or indicator value for a parameter but the overwhelming majority of drinking water samples have concentrations or values that are within a narrow band of the calibration range for which control at the standard is inappropriate, as a minimum two AQC solutions should be used, one with a known concentration or value at or close to the standard or indicator value and the other with a known concentration or value within the range of interest.

6.2.5 | As a minimum, all the results obtained from all AQC solutions should be used to plot, for each solution or calculated quality control characteristic, a Shewhart chart that is used to decide whether a method is in statistical control. When other types of chart are used, including those using statistics calculated from individual values, the laboratory should demonstrate that its arrangements effect adequate statistical control over the systematic error, and both the within-batch and between-batch components of random error, though not necessarily as separate items. Further guidance on the construction and use of control charts is given in NS 30 (or equivalent publication) and the Drinking Water Inspectorate's 'Guidance on the Interpretation of Aspects of Analytical Control' (or equivalent publication).

6.2.6 | The WSA and its laboratory or its contract laboratory should have properly documented policy and procedures for routine AQC that stipulate what action or actions should be followed when an out of statistical control condition is shown to exist, include a definition of an out of control condition and detail the records to be made when such a condition exists. The results of analyses obtained using a method not in statistical control should not be released except in exceptional circumstances, when each result so released should carry an appropriate commentary in all records and reports. The circumstances in which such results can be released should be fully documented and state that the cause of the out of control condition should first be identified and shown not to affect the results of analysis of samples intended for release.

6.2.7 | The procedures should also include regular and frequent examination and review of all charts and include guidance for checking and investigating significant trends or changes in either random or systematic error, and for correct operation

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of the chart. The minimum examination and review periods for each chart should depend on the frequency with which datum points are produced but should not be less frequent than monthly for examination and annually for review. The examination and review should be carried out by a suitably qualified and competent person who is not directly involved in the analysis, such as the laboratory quality manager. There should be appropriate rules for assessing revised control limits.

6.2.8 | An analytical method that is not in statistical control must be investigated and the cause determined and rectified. The performance characteristics of the method may need re-determining in accordance with paragraphs 5.3.14 – 17.

External AQC

6.2.9 | The laboratory should participate in an appropriate external AQC scheme for each parameter or determined constituent of a parameter for which an appropriate scheme is available. The laboratory should also have a properly documented procedure for investigating and recording all failures notified by the organiser of a scheme. Guidance on the suitability of a scheme is given in “The International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories” M Thompson, R Wood, Journal of AOAC International, Volume 76, No 4, 1993.

6.2.10 | In line with the recommendations of this document, laboratories are recommended to participate in schemes distributing drinking water samples of appropriate matrix and which conform to the relevant parts of the protocol. Samples should contain, or be spiked with, concentrations of interest (approximate range one tenth of the standard to twice the standard) and with appropriate speciation where this is of interest. When, in respect of any parameter, a laboratory participates only in schemes that do not meet all the recommended criteria it will be expected to demonstrate that it is participating in the most appropriate scheme currently available. The EPA operates a suitable scheme for some parameters.

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6.3 Microbiological parameters

Routine internal AQC

6.3.1 | Although WSAs must use the methods for microbiological methods specified in part 3 of the Schedule to the Regulations or alternative methods approved by the EPA, it is still necessary to carry out AQC to demonstrate that the methods are detecting the micro-organism of interest and that any organisms detected have been present in the original sample and have not been introduced inadvertently during sampling or in the laboratory.

6.3.2 | As a minimum the following internal AQC should be practised:

- ◆ equipment used for sterilisation should be regularly checked to ensure sterilisation is achieved. It is not sufficient to rely on autoclave tape as an indicator of sterility;
- ◆ all culture media and reagents should be sterile and every batch of completed culture medium should be checked for sterility before use;
- ◆ media should also be checked to ensure that each batch will support the growth of the organism to be detected and it will not support, or will minimise the support, of unwanted organisms;
- ◆ all media and reagents should be stored under conditions that ensure that deterioration does not occur and be marked with their shelf life. Media and reagents that have exceeded their shelf life should not be used;
- ◆ incubators should be fan assisted and incubation temperatures should be checked each day of use both when the incubator is loaded and unloaded;
- ◆ all cultures and sub-cultures should be labelled in such a way that they are clearly identifiable with the original sample;
- ◆ appropriate records should be kept to demonstrate that all necessary procedures have been followed during the examination of a particular sample or batch of samples; and
- ◆ AQC samples containing a known organism should be examined regularly to provide a check on method performance. For example a positive control containing *E coli*, such as natural water known to contain the organism, should be analysed

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with each batch of samples for *E coli*. Alternatively water to which reference organisms have been added should be examined with each batch of samples for that organism.

External AQC

6.3.3 | WSAs and their laboratories or contract laboratories should also participate in external quality control schemes involving the distribution of samples containing specific organisms when such schemes are available. Any evidence from participation in such schemes that shows that there are deficiencies in procedures should trigger immediate investigation of the cause and appropriate remedial action.

7. Calibration of analytical systems

7.1 | It is essential that the calibration procedure for each analytical system or method is fully documented and is sufficient to establish fully or check fully the calibration each time the system or method is used. The procedure will vary with the system or method used and the parameter being analysed, but in all cases the calibration should be established or checked over the entire range of the method and all results of analysis falling outside the applicable calibration range of the method should be rejected.

7.2 | Instrumental systems of analysis (such as chromatography, absorption and emission spectroscopy and automated colorimetric analysis) often require full calibration each time they are used. At least three calibration points are required to demonstrate a straight line. Generally the more complicated the calibration the greater the number of calibration points required. With long instrument runs it is essential that the validity of the calibration throughout the run is demonstrated and therefore as a minimum a repeat measurement of one of the calibration standards should be made at the end of the run.

7.3 | It is also essential that all other apparatus (apart from the analytical systems covered in the above paragraphs) used in the analytical procedure are also calibrated at appropriate intervals. Such apparatus includes, but is not limited to, balances and weights, volumetric equipment including micro-syringes and micropipettes and thermometers.

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8. Correction for analytical recovery losses for organic parameters

8.1 | Some methods used for the analysis of very low concentrations of organic chemical parameters do not fully recover the particular organic chemical sought by the method or the method may be prone to contamination from the environment. Recovery is the extent to which a known added quantity of a parameter can be measured by the analytical system. It is calculated from the difference between results obtained from a spiked and unspiked aliquot of the sample and is usually expressed as a percentage of the added parameter recovered as follows:

$$\% \text{ Recovery} = 100 \times (S(V+W) - UV)/CW$$

Where

- C = concentration of parameter in spiking solution
- V = volume of sample aliquot
- W = volume of the spiking solution added
- S = measured concentration in the spiked sample aliquot
- U = measured concentration in the unspiked sample aliquot

8.2 | Recoveries between 90% and 110% are acceptable and no correction to analytical results is required. Recoveries of less than 90% and more than 110% should be investigated and any cause of loss, contamination or interference within the laboratory's control eliminated. When the recovery at a concentration close to the standard or indicator parameter value is significantly greater than 110% (at the 95% confidence level), an alternative analytical method should be sought.

8.3 | Recoveries for some organic analyses are generally less than 90%. In such cases recoveries and standard deviations should not be significantly different (at the 95% confidence level) from those obtained using the best currently available methods. If they are significantly different an alternative method should be sought. If they are not significantly different the guidance in the paragraphs below should be followed.

8.4 | One approach to the calibration of methods for organic parameters is to submit the calibration standard solutions to the whole procedure applied to samples, including any extraction and concentration steps. When this approach is adopted, a check standard at the same concentration as one of the calibration standards and preferably close to the standard or indicator parameter value that has been subjected

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to only the final measurement procedure should be analysed in order to monitor the actual recovery for that batch of analyses. The actual recovery should be recorded as a performance check and appropriate action taken when abnormal recovery is recorded.

8.5 | When the within batch standard deviation of the method is such that the approach in the above paragraph is not appropriate (for example calibration is not possible because of variability due to random errors), correction for recovery should be considered when recoveries are less than 90%. In such cases the recommended approach is to calculate a long-term mean correction factor using data from analyses of spiked samples. Results for AQC solutions must not be used. The actual recovery for the batch should be recorded as a performance check and appropriate action taken when abnormal recovery is recorded.

8.6 | The use of recovery correction factors should be regarded as a last resort and should only be applied after exhaustive attempts to eliminate the source of bias have been documented and proved unsuccessful. This information will often form part of an authoritative reference method. Good analytical methods that require neither compensation nor correction should be used in preference to those with built in compensation for poor recovery or those requiring correction.

8.7 | When an approach does not reduce the uncertainty associated with an individual result (as represented by the total error, calculated as bias plus twice the total standard deviation, after any relevant correction) that approach should not be adopted. In the absence of any acceptable procedure results should not be corrected. Results obtained with a method having a poor recovery that have not been corrected for recovery should carry an appropriate commentary on the analytical report.

9. Records of laboratory analysis and integrity of results

9.1 Records of laboratory analyses

9.1.1 | WSAs and their laboratories or contract laboratories should keep adequate records of key aspects of analytical procedures and the results. It is suggested that these records be kept for at least three years. As a minimum these records should include:

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- ◆ all key instrument installation, commissioning, maintenance and repair records, including any instrument log or diary;
- ◆ all basic calibration records (including proof of traceability), method suitability checks and any other record necessary to demonstrate the suitability of any equipment used at the time of the analysis;
- ◆ the analytical procedure used;
- ◆ all initial method performance testing data, including raw data, and similarly for any re-determination of performance;
- ◆ routine internal and external AQC data, including charts, investigations of out of control conditions and corrective action; and
- ◆ raw data for the whole analytical run and all calculations to obtain the final result of the analysis.

9.2 Integrity of results

9.2.1 | It is vitally important for public confidence in the results of compliance monitoring that WSAs and their laboratories or contract laboratories have arrangements and procedures in place to prevent unauthorised alteration of results at all stages of the production of the results in the laboratory and during the transfer of those results to the WSA's database

9.2.2 | The initial result in the laboratory may be a print out from the analytical equipment or the record of an analytical measurement in the analyst's workbook. The analyst may be required to manipulate the initial result and to make calculations to obtain the final compliance monitoring result. If the analyst makes a mistake during this process the result should be corrected in a way that shows exactly what the analyst has done – for example by putting a line through the mistake, entering the correct result alongside and initialling and dating the entry, but not by using correcting fluid to substitute the correct result for the incorrect result.

9.2.3 | A designated (experienced) person in the laboratory should be responsible for validating the result and authorising its transfer to the WSA's database. This person should check the analyst's result, that the analytical method is in statistical control from the AQC results and that the result relates to the appropriate compliance sample.

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If this person is satisfied the result can be validated and released to the database. Once a result is on the database it must not be deleted or altered. If it is subsequently discovered that a result on the database is incorrect the result may be qualified by a suitable explanation that gives the correct result. If the result is so wrong that it affects the statistical summary of compliance then the incorrect result may be replaced by the correct result, but the incorrect result must continue to be displayed with an appropriate explanation.

9.2.4 | Some laboratory methods may involve computers and laboratory results may be recorded on a computerised laboratory database. Computer access should be controlled by passwords that are set with sufficient level of access (analysts may not need the same level of access as the person validating the results) and passwords should be changed regularly. Any corrections to computerised data should follow the principles described in the previous paragraph.

10. Annual reporting to the EPA of results of monitoring public water supplies

10.1 | The requirement for WSAs to report annually to the EPA the results of all monitoring of public water supplies is covered fully in Section 9 of this handbook. The section sets out the monitoring results to be included, the format for the submission of the information (excel spreadsheet) including the source of supply, the type of treatment, supply zone code, *Cryptosporidium* risk score, remedial action list, sample information, analysis information and the timing for submission of this information.

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Appendix 1: parameters for which performance characteristics are specified

For the following parameters, the specified performance characteristics are that the method of analysis used must, as a minimum, be capable of measuring concentrations equal to the parametric value with a trueness, precision and limit of detection specified. Whatever the sensitivity of the method of analysis used, the result must be expressed using at least the same number of decimals as for the parametric value considered in tables B and C in part I of the schedule.

Parameter number	Parameter	Trueness % of parametric value (note 1)	Precision % of parametric value (note 2)	Limit of detection % of parametric value (note 3)	Notes
3	Acrylamide				*
29	Aluminium	10	10	10	
30	Ammonium	10	10	10	
4	Antimony	25	25	25	
5	Arsenic	10	10	10	
6	Benzo(a)pyrene	25	25	25	
7	Benzene	25	25	25	
8	Boron	10	10	10	
9	Bromate	25	25	25	
10	Cadmium	10	10	10	
31	Chloride	10	10	10	
11	Chromium	10	10	10	
34	Conductivity	10	10	10	
12	Copper	10	10	10	
13	Cyanide	10	10	10	Note 4
14	1,2 Dichloroethane	25	25	10	
15	Epichlorohydrin				*
16	Fluoride	10	10	10	
36	Iron	10	10	10	
17	Lead	10	10	10	

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Parameter number	Parameter	Trueness % of parametric value (note 1)	Precision % of parametric value (note 2)	Limit of detection % of parametric value (note 3)	Notes
37	Manganese	10	10	10	
18	Mercury	20	10	20	
19	Nickel	10	10	10	
20	Nitrate	10	10	10	
21	Nitrite	10	10	10	
39	Oxidisability	25	25	10	Note 5
22	Pesticides	25	25	25	Note 6
24	Polycyclic aromatic hydrocarbons	25	25	25	Note 7
25	Selenium	10	10	10	
41	Sodium	10	10	10	
40	Sulphate	10	10	10	
26	Tetrachloroethene	25	25	10	Note 8
26	Trichloroethene	25	25	10	Note 8
27	Trihalomethanes – Total	25	25	10	Note 7
46	Turbidity	25	25	25	^
28	Vinyl chloride				*

* To be controlled by product specification

^ As specified in the note in section 3 of part 3 of the schedule to the Regulations

For hydrogen ion concentration the specified performance characteristics are that the method of analysis used must be capable of measuring concentrations equal to the parametric value with a trueness of 0.2 pH unit and a precision of 0.2 pH unit.

Note 1: Trueness is the systematic error and is the difference between the mean value of a large number of repeated measurements and the true value (this term is further defined in ISO 5725).

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Note 2: Precision is the random error and is usually expressed as the standard deviation (within and between batches) of the spread of results about the mean. Acceptable precision is twice the relative standard deviation (this term is further defined in ISO 5725).

Note 3: Limit of detection is either:

- three times the relative within batch standard deviation of a natural sample containing a low concentration of the parameter, or
- five times the relative within batch standard deviation of a blank sample.

Note 4: **The method should determine total cyanide in all forms.**

Note 5: **Oxidation should be carried out for 10 minutes at 100°C under acid conditions using permanganate.**

Note 6: The performance characteristics apply to each individual pesticide and will depend on the pesticide concerned. The limit of detection may not be achievable for all pesticides at present, but sanitary authorities should strive to achieve this standard.

Note 7: The performance characteristics apply to the individual substances specified at 25% of the parametric value in part 1 of the schedule.

Note 8: The performance characteristics apply to the individual substances specified at 50% of the parametric value in part 1 of the schedule.

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Appendix 2: parameters for which methods of analysis are specified

The following principles for methods of microbiological parameters are given for reference whenever a CEN/ISO method is given for guidance, pending the possible future adoption, in accordance with the Committee procedure laid down in Article 12 of Council Directive 98/83/EC of further CEN/ISO international methods for these parameters. Sanitary authorities [WSAs] may use alternative methods, providing the provisions of sub-articles 7 (4) (a) and (b) are adhered to.

Coliform bacteria and *Escherichia coli* (*E. coli*) (ISO 9308-1)

Enterococci (ISO 7899-2)

Clostridium perfringens (including spores):

Membrane filtration followed by anaerobic incubation of the membrane on m-CP agar (Note 1) at $44 \pm 1^\circ\text{C}$ for 21 ± 3 hours. Count opaque yellow colonies that turn pink or red after exposure to ammonium hydroxide vapours for 20 to 30 seconds. The composition of the m-CP agar is:-

Basal medium	
Tryptose	30 g
Yeast extract	20 g
Sucrose	5 g
L-cysteine hydrochloride	1 g
MgSO ₄ 7H ₂ O	0.1 g
Bromocresol purple	40 mg
Agar	15 g
Water	1000 ml
Dissolve the ingredients of the basal medium, adjust pH to 7.6 and autoclave at 121° for 15 minutes. Allow the medium to cool and add:	
D-cycloserine	400 mg
Polymyxine-B sulphate	25 mg
Indoxyl – β-D-glucoside to be dissolved in 8ml sterile water before addition	60 mg

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Filter-sterilised 0.5% phenolphthalein diphosphate solution	20 ml
Filter-sterilised 4.5% FeCl ₃ .6H ₂ O	2 ml

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