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Guide to on-site test methods for the analysis of waters

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Foreword

Publishing information

This British Standard BS 1427 is published by BSI and came into effect on 1 January 2009. It was prepared by Subcommittee EH/3/2, *Physical, chemical and biochemical methods*, on behalf of Technical Committee EH/3, *Water quality*. A list of organizations represented on the committee can be obtained on request to its secretary.

Supersession

This standard supersedes BS 1427:1993, which is withdrawn.

Information about this document

This guide describes methods for the analysis of industrial and other waters which can be undertaken outside of the chemical laboratory, as on-site tests, when the purpose of the test is to characterize the water under test for either quality or process control purposes. With some determinands, which can be unstable after sampling and which cannot be stabilized, on-site tests provide a suitable means of obtaining fit-for-purpose test results. The test methods described in this Standard comprise simple procedures for use either by a trained analyst, or following appropriate training, by a suitable individual. Since 1993, many industrial laboratories have been shut down and the use of on-site methods increased significantly. A very significant proportion of these tests are carried out by non-analysts using proprietary test-kits.

This British Standard is not intended as a substitute for, or alternative to, other British Standards on the quantitative analysis of waters, namely BS 6068 (all parts) and BS 2690, which remain the reference methods for use in a laboratory. The methods described in this British Standard are mostly based on principles and/or procedures used in BS 6068 and BS 2690 and their associated updates.

This revision of BS 1427 contains updates to specific methods since the previous standard and provides guidance on the use of the much wider range of available commercial test-kits and test-kit equipment since the 1993 previous version of this standard. Since 1993, the range, type and quality of commercial test-kits/apparatus have improved considerably, producing significant benefits in terms of increased reliability and ease of use for on site testing. The use of pre-packaged chemicals and reagents has also reduced health and safety risks associated with handling chemicals and enhanced the simplicity of testing.

The layout of this British Standard has been significantly amended since the 1993 version to reflect the very much greater availability of commercial test-kits and the continuing need for effective quality control procedures, both of which feature in extended annexes.

Traditional colorimetric, titrimetric and instrumental methods for water analysis are still shown in this standard. The test procedures for the colorimetric and instrumental test methods are specific to the colour comparator or instrument described. The test procedures for each commercial test-kit method are specific to the parameter described. The detailed test procedure defined/described by the manufacturer are to be followed when the manufacturer's test-kit is used.

A standardized approach to describing tests undertaken with the colour comparator method is shown separately in Annex B.

Commercial test-kit methods available are shown in Annexes I, J and K. As a guide, this British Standard takes the form of guidance with a number of informative annexes. The full set is:

- Annex A, *Guidance on training, supervision and associated needs*
- Annex B, *Example of a documented test-kit method and associated sampling protocol for a colorimetric method*
- Annex C, *Typical expected result confidence intervals from a given QC solution from a range of test-kits from two manufacturers*
- Annex D, *An example of target test-kit requirements for a COD test-kit used in a designated room/area testing facility*
- Annex E, *An example of target test-kit requirements for a free and total chlorine test-kit used on-site at the sampling location*
- Annex F, *An example of routine QA/QC documentation for a COD test*
- Annex G, *An example of routine QA/QC documentation for a free and total chlorine test*
- Annex H, *Example of Shewart control charts*
- Annex I, *Test-kit method overview*
- Annex J, *Summary of test-kits*
- Annex K, *Availability of visual test sticks*

Use of this document

As a guide, this British Standard should not be quoted as if it were a specification, and particular care should be taken by manufacturers and users to ensure that claims of compliance are not misleading or taken out of context. Any user claiming compliance with this British Standard is expected to be able to justify any course of action that deviates from its recommendations. Any compliance claim should be supported with appropriate validation data.

Users cannot specify the on-site test performance required and how to monitor that this performance is met on an on-going basis. They can then either devise an on-site method or find a test-kit that can meet this specification for all relevant matrices. It is felt that the vast majority of users of this standard will adopt the latter option. This document aims to provide guidance on how to do this, but the user needs to decide the level of performance required to ensure that all results are fit for the intended purpose. Users in this context include the end user of the results and the organization carrying out the test.

It is not possible to cite or describe specific individual commercial water test-kits in a British Standard for commercial impartiality reasons. In Annex H, a general overview of the major commercial water test-kits currently available is given.

The water test-kit market is fast moving and extremely innovative. Users of test-kits are strongly advised to regularly check the availability of improved test-kits meeting their requirements. Most relevant information is available on the various test-kit manufacturers' websites and can be readily assessed via a search engine. It is important to appreciate that much of this performance data is best-case data, often related to aqueous standards. Users always have to assess any new proposed test-kit that they intend to use against all relevant sample matrices that are to be tested.

Notice has been taken of the proposed MCERTS *Performance Standards and Test Procedures for Water Test-kits* in the writing of this standard.

Hazard warnings

WARNING. This British Standard calls for the use of substances and/or procedures that can be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage.

Presentational conventions

The provisions in this standard are presented in roman (i.e. upright) type. Its recommendations are expressed in sentences in which the principal auxiliary verb is "should".

Commentary, explanation and general informative material is presented in smaller italic type, and does not constitute a normative element.

The word "should" is used to express recommendations of this standard. The word "may" is used in the text to express permissibility, e.g. as an alternative to the primary recommendation of the clause. The word "can" is used to express possibility, e.g. a consequence of an action or an event.

Notes and commentaries are provided throughout the text of this standard. Notes give references and additional information that are important but do not form part of the recommendations. Commentaries give background information.

Contractual and legal considerations

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard cannot confer immunity from legal obligations.

Section 1: General

1.1 Scope

COMMENTARY ON 1.1.

Emergency screening analysis for major pollution incidents is not covered. Information on this is available via the US EPA which has set up an Environmental Technology Verification (ETV) Programme [1] and published a series of verification reports developed for each verified technology. The reports contain the performance verification test results and meet the ETV programme's stringent quality assurance and peer review requirements for a wide range of advanced monitoring systems for water analysis mainly directed to emergency incidents. Further information on rapid analytical response issues in emergency situations is available (Thompson and O'Neill, 2006 [2] and Thompson, 2009 [3]).

This British Standard describes information and guidance on test methods for the analysis of industrial and other waters which are applicable outside of a conventional laboratory as either in situ on-site tests; or the use of a close suitable designated room/area testing facility. For some tests this could include dedicated space in a designated vehicle or caravan. These test methods can relate to compliance, water quality and process control purposes. No recommendation is given as to which test is applicable to a particular type of water, nor is it implied that in any given case all tests or any particular suite of tests are necessary. The selection of tests depends on local requirements and conditions. Samples requiring preservation for subsequent analysis are outside of the scope of this British Standard.

Certain tests such as adsorbable organic halides (AOX), chemical oxygen demand (COD), total organic carbon (TOC) and total nitrogen need a suitable designated test room/area facility and are not considered suitable for testing outside of these designated areas.

Sample stability is not considered to be a significant problem as most test-kit measurements are carried out with minimal delay.

Most of the methods described in this standard can be applicable for use on clean or lightly polluted waters which might have undergone treatment or been modified for industrial use. Typically, the types of waters for which the tests are intended include the following:

- a) boiler waters;
- b) cooling waters;
- c) waters from hot water systems;
- d) waters from air conditioning systems;
- e) waters from industrial air washing systems;
- f) potable waters;
- g) ground waters;
- h) surface waters;
- i) process waters; and
- j) swimming pool waters.

Certain more heavily polluted waters can be routinely tested for certain control purposes for specific determinands using the test methods cited in this British Standard. Such waters include:

- 1) sewage works influents and effluents;
- 2) selected industrial effluents.

Emergency screening analysis for major pollution incidents is not covered (see commentary).

The on-site methods outlined in this British Standard include titrimetric, colorimetric and instrumental analytical techniques and also includes consideration of commercial test-kits: their general principles and formats available. It is generally accepted that in the UK, the vast majority of on-site measurements are carried out using commercial test-kits rather than user-devised test-kit type methods.

following similar equivalent method procedures to those used in conventional laboratories. Also the designated room/area testing facility titrimetric methods described in Section 4 of this standard might require staff with a greater degree of training than commercial test-kit titrimetric methods which can be readily used outside of these areas. These methods are less likely to be used by non-analysts than the more robust test-kit titrimetric methods.

The technique used and principle of the designated room/area testing facility titrimetric method are described within each test method and each method is self-contained within a standard format.

This British Standard does not apply to radioactivity, ecotoxicity (See Persoone et al., 2000 [4]) or microbiological testing. Spot tests are not covered (Jungris, 1997 [5]).

Many of the relevant methods are suitable for analysis for the water framework directive (see Note and SWIFT 2003 [6]), water for human consumption (see Note) and for monitoring of discharges to water and sewer (EA, 2006 [7] and Dixon and Gardner, 1997 [8]).

NOTE EC 2000 [9] is known as 'the water framework directive'; EC 1998 [10], The Water Supply (Water Quality) Regulations 2000 [11] and DWI [12] relate to water for human consumption.

1.2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

BS 1704:1985/ISO 1770:1981, *Specification for solid-stem general purpose thermometers*

BS 2586, *Specification for glass and reference electrodes for the measurement of pH*

BS EN ISO 3696:1995, *Water for analytical laboratory use – Specification and test methods*

1.3 Terms and definitions

For the purposes of this British Standard, the following terms and definitions apply.

1.3.1 on-site tests

quantitative chemical and physical tests undertaken in situ on a site, or within a designated room/area testing facility, to enable water quality assessments to be undertaken

NOTE Some on-site tests do require a designated room/area testing facility and these tests are clearly indicated in this standard (See Annex I).

1.3.2 designated room/area testing facility

basic laboratory facilities in a designated room (or a designated secure area in a room)

NOTE 1 This could include provision of electricity, fixed bench facilities, water supply and suitable drainage; analytical balance, heating block, fume extraction, etc. Other uses of the room or area have to be controlled

so as not to interfere in any way with the analysis. This ought to include controlled access and restricted activities compatible with use as a test area. For some tests this could include dedicated space in a designated vehicle or caravan.

NOTE 2 It would appear that the majority of on-site (1.3.1) tests are carried out in a designated room/area testing facility.

1.3.3 designated test room/area test

test that requires a designated room/area testing facility (1.3.2)

NOTE This could include provision of electricity, fixed bench facilities, water supply and suitable drainage; analytical balance, heating block, fume extraction, etc. (See also 1.3.2).

1.3.4 ready to use test-kit

commercially packaged complete analytical system in a portable kit format designed for on-site testing

NOTE 1 Some test-kits can only be applied in designated test rooms/areas and laboratories.

NOTE 2 Test-kits are intended as simplified, easy-to-use tests for analysis of water quality, usually within specified concentration ranges and designated sample matrices.

1.3.5 precision

random error expressed as the standard deviation (within a batch and between batches) of the spread of results about the mean

1.3.6 trueness (the systematic error)

difference between the mean value of the large number of repeated measurements and the true value

1.3.7 bias

synonym of trueness (1.3.6)

1.3.8 uncertainty

parameter associated with the results of a measurement that characterises the dispersion of the values that could reasonably be attributed to the measurand

NOTE It therefore takes account of all the significant sources of variability in the measurement results and should include the effect of both random and systematic errors. No part of uncertainty can be corrected for.

1.3.9 confidence interval

CI

interval estimate of a population parameter; instead of estimating the parameter by a single value, an interval of likely estimates is given

NOTE Most confidence intervals are stated at the 95% level.

1.3.10 accuracy

closeness of agreement between a test result and the accepted reference value

NOTE Definition from BS ISO 5725-1:1994.

1.3.11 method validation

confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled

NOTE Definition from BS EN ISO IEC 17025.

1.3.12 system suitability checks**SSC**

simple tests to ensure that the associated test-kit instrumentation is functioning within specified limits before commencing analysis

1.3.13 quality assurance

part of quality management focussed on providing confidence that quality requirements will be fulfilled

1.3.14 quality control

part of quality management focussed on fulfilling quality requirements

1.3.15 repeatability

precision under repeatability conditions. i.e. conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time

1.3.16 reproducibility

precision under reproducible conditions, i.e. conditions where independent test results are obtained with the same method on identical test items in different laboratories by different operators using different equipment

1.3.17 limit of detection

lowest concentration of an analyte that can be detected with a specified degree of statistical certainty

1.3.18 proficiency testing

assessment of laboratory testing performance by means of interlaboratory comparison by distributing common samples to a number of different laboratories

1.3.19 control charts

routine plotting of analysis data obtained from the analysis of quality control materials to check if the results lie within pre-determined limits

1.3.20 target value chart

control chart with fixed rather than statistically derived limits

1.3.21 internal AQC

analysis of known samples or standards carried out with each batch of analysis

1.3.22 recovery

fraction of the amount of analyte added which is found using the method

NOTE If a known amount of an analyte is added to an aliquot of a test sample and the amended and unamended samples are analysed, then the recovery is the fraction of the amount of analyte added which is found using the method.

1.3.23 fitness for purpose

process of assessing that a method is suitable for a given application

1.3.24 instrumental method

method that employs non-photometric instrumentation that can be used for in situ on-site measurements

NOTE It can also be used in a designated room/area testing facility analysis.

1.3.25 standard addition

method of standard addition used in instrumental analysis to determine the concentration of a substance (analyte) in an unknown sample by comparison to a set of the test sample solutions with increasing additions of known concentration of the analyte

NOTE This is similar to using a calibration curve. The aim is to at least double the concentration of the analyte with the largest addition. Standard addition can be applied to most analytical techniques and can be used instead of a conventional calibration graph to minimize the matrix effects.

1.3.26 analyte

chemical or constituent being determined

1.3.27 carryover

contamination of a subsequent sample by a previous sample, typically due to incomplete cleaning of a reused test-kit component

1.3.28 interference

effect of a matrix component that might cause an analytical bias or that might prevent a successful analysis

1.3.29 matrix

sample contents other than the target analyte

1.3.30 in situ

within, or directly adjacent to, the water space

1.3.31 on-site

method that can be carried out at a location close to where the sample is taken

NOTE Generally, fixed facilities or services might not be available.

1.4 Information on MCERTS performance standards and test procedures for water test-kits

The Environment Agency (EA) established its Monitoring Certification Scheme (MCERTS) to deliver quality environmental measurements in 1997 and a number of performance standards have been developed for air, land and water (see www.mcerts.net).

The Environment Agency MCERTS scheme includes *Performance Standards and Test Procedures for Portable Water Monitoring Equipment* [13]. It provides a certification framework for instruments designed for use in the field and applies to any technology. It describes laboratory and field tests to assess the operation of sensors for the measurement of: dissolved oxygen; conductivity; pH; turbidity; temperature; ammonia; nitrate; nitrite; orthophosphate; and chlorophyll-a. Other parameters may be covered in future, if there is sufficient industry interest and support.

The *Performance Standards and Test Procedures for Portable Water Monitoring Equipment* [13] standard covers equipment used for monitoring of:

- waste water treatment;
- rivers, lakes and estuarine environments;
- water storage reservoirs;

- boreholes and groundwater;
- trade effluents.

A portable water monitor (PWM) comprises of all components required to make the measurement. This may include reagents, devices required for sample preparation and any sample delivery system. A PWM should provide a direct read-out of the determinand, in appropriate units of measurement and not require reference to a calibration chart or table. Relevant water test-kits can also be submitted for accreditation under this scheme.

The Environment Agency is in the process of considering the development of a separate MCERTS standard specifically for water test-kits.

Test-kit manufacturers can receive an MCERTS certificate for a given test-kit with a five year expiry date, if they demonstrate that they can meet the relevant MCERTS requirements.

Expected advantages of MCERTS for test-kits are:

- makes available a certification scheme that is formally recognized within the UK and is acceptable internationally;
- gives confidence to regulatory authorities that test-kits/instrumentation, once certified, are fit-for-purpose and capable of producing results of the required quality and reliability;
- gives confidence to users that the test-kits/instrumentation selected are robust and conform to performance standards that are accepted by the Environment Agency;
- supports the supply of accurate and reliable data to the public;
- provides test-kit manufacturers with an independent authoritative endorsement of their products, which will facilitate their access to international markets and increase the take-up of their products in the UK.

Product certification comprises three phases. These are as follows.

- Laboratory testing – used to determine performance characteristics, where such testing requires a highly controlled environment.
- On-site or designated test room/area testing, carried out on waters that are representative of the intended applications.
- Surveillance (initial and continuing), which comprises an audit of the manufacturing process to confirm that the manufacturer has provisions to ensure manufacturing reproducibility and to control any design changes to ensure that they do not degrade performance below the MCERTS standards.

Users of this British Standard are advised that, where there are any MCERTS services and equipment available relevant to the analysis being performed, users should consider the desirability of using these.

Section 2: Physical and chemical test methods: general information

2.1 Health and safety

2.1.1 General

The test methods described in this British Standard should be undertaken only after suitable training has been received (see 2.2). Where necessary, adequate supervision in undertaking the tests should be provided. Training and associated documentation in areas such as safe working practices should also be provided which should pay due regard to all potential hazards of on-site operations.

NOTE 1 Attention is drawn to the Health and Safety at Work etc. Act 1974 (HSE, 1974) [14] and the need for management and test operators to make adequate provisions for safe working.

The test operator should be fully trained with respect to and always following safe working procedures.

NOTE 2 Attention is drawn to the Control of Substances Hazardous to Health Regulations 2002 (COSHH, 2002) [15] which mention risk assessment being undertaken and appropriate safety measures to be implemented when using potentially hazardous chemicals.

NOTE 3 The chemicals referred to in this British Standard are typical of chemicals used in a laboratory environment and hazard data sheets are obtainable from chemical suppliers. Commercial test-kits might also incorporate hazardous chemicals and manufacturers ought to provide warning notices and relevant hazard data sheet(s) with the kits.

This British Standard does not define safe working procedures for test methods or commercial test-kits; in addition to laboratory and site work safety training, the check-lists in 2.1.2 to 2.1.3 for working practices should be considered, as they include some general safeguards against potential hazards within routine control testing.

2.1.2 Personal safety precautions

Personal safety precautions that should be followed include the following.

- a) Where appropriate, protective safety glasses should be worn when following test methods or when handling hazardous chemicals. An emergency eyewash bottle or facility should be made available.
- b) Hand washing facilities should be provided whilst working on-site and/or in a designated test room/area.
- c) Where appropriate, suitable protective clothing when sampling, testing or when handling hazardous chemicals should be worn.
- d) Hand-to-mouth actions, such as smoking, drinking or eating should not be undertaken whilst testing or handling hazardous chemicals. Cosmetics or handcreams, etc. should not be applied.
- e) The use of self-adhesive labels should be encouraged.
- f) Solutions should never be pipetted by mouth.

- g) Because of potential microbiological contamination in water samples, samples should always be taken in bottles with secure caps and the hands washed after sampling or carrying out tests. The hands should also be washed before leaving a site location.
- h) Lone worker implications at unmanned sites should be considered.

2.1.3 Care in the work zone (i.e. on-site or in designated room/area testing facilities)

Work zone precautions that should be followed include the following.

- a) Designated room/area testing facilities should be kept clean and tidy.
- b) The test-kit user should ensure that chemicals and test-kits are stored safely using clear, unambiguous labelling. A regularly updated register of all chemicals and test-kits stored should be kept by a suitable nominated appropriate person.
- c) All spillages should be cleaned up immediately in a safe manner.
- d) Hazardous chemicals are usually sold in protective packaging clearly marked with appropriate hazard warnings; the chemicals should be stored securely and safely as directed on the label or material data safety sheet supplied with the chemical. Preparation of test reagents should be restricted to suitably trained staff unless the test operative is given suitable training and detailed instructions on preparation procedures.

2.1.4 General safeguards

General safeguards that should be implemented include the following.

- a) Safe working procedures for the designated room/area testing facility and when working on-site should always be followed.
- b) Damaged equipment or broken or chipped glassware should not be used.
- c) Test operatives and samplers should be trained and checked/audited in safe working procedures for testing.
- d) Where chemicals are transported for on-site testing, suitable protective containment should be identified and used.
- e) Documented instructions should be provided (and always followed by test operatives) for the safe disposal of any waste material.

2.2 Training, supervision and associated needs

Satisfactory training is essential to safeguard the test operator and to achieve acceptable test results. This British Standard does not define the training or criteria required for training, but Annex B gives some guidance on training, supervision and associated needs.

Training should be subject to objective assessment against preset criteria by a competent person: the final approval of the test operative lies with the trainer. It is essential that the test-kit user is fully trained to use the test-kit to give fit-for-purpose results and that this training is maintained.

2.3 Test facilities

Test results can have a significant random error associated with the physical and environmental conditions under which the test is carried out. There are only limited measures that can be implemented to improve the environment when carrying out open site in situ testing, but provision of a fit-for-purpose designated room/area testing facility within a fixed enclosed site can improve the quality of testing. This area should, ideally, be controlled-access, only subject to activities which are compatible with use as a testing area and ideally provide the following features:

- a) adequate environmental conditions, including provision of lighting and heating;
- b) services including power outlets, running water and drainage and a supply of RO/DI water to BS EN ISO 3696:1995, Grade 3;
- c) bench top working space and secure cupboard storage space for chemicals and apparatus;
- d) for health and safety reasons, access should be restricted and the designated testing room/area made secure when not in use; other uses of the room or area should be controlled so as not to interfere in any way with the analysis; and
- e) for any tests using significant amounts of hazardous reagents and/or involve complex procedures (e.g. AOX, COD, TOC and total Nitrogen) a designated room/area testing facility is essential.

The vast majority of test-kit measurements are carried out in designated room/area testing facilities.

2.4 Sampling, sampling preservation and sample pre-treatment considerations

NOTE 1 Some instrumental test methods can be applied in situ by direct immersion of measuring electrodes into the body of water, which obviates the need for sampling (e.g. DO; redox potential; pH and electrical conductivity). This assumes that it has been shown to be safe to carry out such measurements.

Where it is necessary to obtain a sample from a liquid stream, it is essential to obtain a representative sample, which should be tested as quickly as possible to retain sample integrity. Correct sampling procedures and, if relevant, the appropriate sample preservation and pre-treatment techniques are essential for fit-for-purpose analytical results. Sample preservation is seldom required for on-site measurements which are normally carried out within a few minutes of sampling. Guidance on sampling is available from a number of sources (see Note 2). Comprehensive details of sample preservation are given in BS EN ISO 5667-3.

NOTE 2 Further details on sampling and sample preservation are described in BS 6068-6.7, BS 3285, BS 6068-6 and BS EN ISO 5667-3.

NOTE 3 If the test-kit is used for field screening to indicate the need for samples requiring a high accuracy measurement, the user might need to provide a means of preserving a sample for later measurements at a laboratory.

For most tests carried out on-site, the water sample is tested without any sample pre-treatment other than, for example, 0.45 µm filtration

for toxic metals. This is then a filtered metal rather than a total analyte determination. However, some test-kits indicate sample pre-treatment procedures to obtain a “total analyte measurement”. Often it is not practically possible to implement these procedures for in situ on-site measurements. The pre-treatment procedures are normally carried out in a designated room/area testing facility.

Documented sampling and sub-sampling procedures are essential to ensure that the test operative takes a fit-for-purpose relevant samples and sub-samples. These can be included in the actual test method. It is paramount that an appropriately-taken sample is used to then obtain a fit-for-purpose analysis.

It is important that the sample temperature at the time of testing is within the limits of the testing method. For example, the speed of reaction of many colorimetric reactions is significantly reduced at low temperatures.

Sampling from enclosed vessels and sample lines might be hazardous and documented safe working procedures should be available and undertaken correctly. This is particularly important where the system is operated at elevated temperatures and/or pressures.

Open-channel sampling (including sampling from boats) or other in-situ, on-site or remote site sampling requires specific documented safe working procedures that should be followed.

Samples should be taken at or below points of turbulence to provide representative samples.

In this British Standard, information on spot-sampling procedures predominates. Where on-site time- or flow-weighted composite sampling is required, the collection bottle should be refrigerated during and after the sampling period until the analysis is carried out. The composite sampling period should be kept to a minimum to reduce potential sample deterioration. However, note that some regulatory tests mention 24-hour composite samples. Where sample deterioration is a problem and regulations permit, it might be better to take a number of shorter period samples and then average the results (e.g. instead of one 24-hour composite sample, it might be better to use six 4-hour composite samples), assuming each composite sample is analysed promptly.

Turbidity and colour could interfere with many of the test methods described in this British Standard, particularly the colorimetric test methods. It can be advantageous or necessary to clarify visibly-turbid samples either by quiescent settlement then sampling the supernatant liquor or by filtration through a membrane or glass filter of known pore size. Details of any pre-treatment used should be recorded; simple purpose-designed, easy-to-use, low-cost, disposable systems for filtration on-site (e.g. syringe filtration) are commercially available. Any determinand present in the removed particulate matter will not be included in the test result. This information should be recorded and reported. It is not common practice in on-site testing of waters to pre-treat samples to solubilize particulate matter or release inactivated (bound) determinand. Such pre-treatments are not identified in the test methods cited in many BS 6068 water quality methods which typically measure only the soluble and reactive (available) determinand. Any pre-treatment is a matter for local decision, but should be clearly recorded and reported. Pre-treatment should only be used where such treatment does not invalidate the use

of the test method. Pre-treatment for some parameters (e.g. metals) might require the use of a designated room/area testing facility; this should be determined by a risk or COSHH assessment.

NOTE 2 Further details on sampling and sample preservation are described in BS 6068-6.7, BS 3285, BS 6068-6 and BS EN ISO 5667-3.

2.5 Selection and application of test methods

2.5.1 General

The test methods outlined in Annex A for use for on-site testing purposes should be adequate for most purposes. Most of the test methods included are based upon laboratory reference test methods but are commonly modified or utilized in a form to make them usable under on-site conditions. The selected test methods fall into three categories; colorimetric methods, titrimetric methods and instrumental analysis methods.

Commercial companies supply significant quantities of portable compact test-kits for water quality testing using a variety of techniques. The provision of test-kits is a competitive international market and variants of the same basic test method are commonplace between the different manufacturers. The primary factors when considering the use of commercial test-kits are accuracy, range, possible interferences, ease of use and possibly cost. Because of the scale of the international commercial test-kit market, there is no independent review of available systems. Manufacturers supply extensive information which is confined to their own products. This typically covers test method principle, test range, application areas in respect of test matrix, data on interferences, some typical best-case performance data and also health and safety information in respect of the reagents used. Detailed analytical performance data is not always provided. Defining standard testing conditions is difficult as there are currently no recommended test protocols for manufacturers to evaluate kit systems. It is important to appreciate that much manufacturer's data is best-case data, often related to aqueous standards. Users should always assess any new proposed test-kit that they intend to use against all relevant sample matrices that are to be tested. Where no validation data is available or the data is not relevant to the matrices of interest, the user should then carry out their own evaluation.

Because of their convenience and other defined advantages, the use of validated (see 3.4) commercial test-kits is recognized for test methods outlined in this British Standard. The observations in this section are intended as a brief review of some of the important factors concerning commercial test-kits which will assist any consideration of their use.

2.5.2 Colorimetric methods

2.5.2.1 General

Colorimetric is the most widely used chemical testing technique for water quality test-kits. The format and principle of the kits available vary widely, as do the accuracy and discrimination achievable. Modern

microelectronics have assisted in the development of miniature portable colorimeters with a variety of facilities to aid simple use on-site, including direct concentration read-out. In contrast, the widely-used visual colour comparison systems remain cheap and simple, but are more operator-dependent in terms of accuracy and colour discrimination.

The following systems are widely available in different commercial formats:

- a) visual comparison systems:
 - 1) paper test strips;
 - 2) printed colour comparator cards;
 - 3) colour comparison cubes;
 - 4) glass or plastics colour standard comparator discs; and
 - 5) sealed coloured liquid standards (ampoule/tube-based colour comparator);
- b) portable photometers:
 - 1) digital read-out of transmission; and
 - 2) optical absorbance or concentration.

The concentration output is often derived from a calibration graph supplied by the test-kit manufacturer.

Commercial colour disc visual comparison systems, because of their simplicity and wide application are still used by many organizations as the basic system for many of the colorimetric test methods described in this standard. Alternative visual colour comparison formats are commercially available for most of the determinands measured by colorimetry and described within this British Standard, e.g. ampoule-based colour comparators, consisting of a number of vacuum sealed glass ampoules containing a harmless dye, each corresponding to the calibrated increment for the relevant test range. Ampoule/tube-based colour comparators typically come in two types, either a round comparator (containing e.g. 8 ampoules, in a small cylinder; the test ampoule is placed inside the cylinder and the whole apparatus held up to the light and viewed from the bottom, viewing the long path length along the axis of the ampoules) or a flat comparator (containing e.g. 9 ampoules, held in place against a flat, white plastic backing. The test ampoule is laid between the comparator ampoules to find the two closest colour matches, viewing the short path length across the axis of the ampoules).

Some test-kits incorporate reagents in a sample/measuring tube which can then be inserted directly into a comparator or photometer after adding the sample.

Low-cost, rugged, portable photometer-based methods are now widely available from manufacturers and provide an instrumental alternative to visual comparison for the majority of the colour tests outlined in this British Standard (see Annex B).

2.5.2.2 Operation and reagents

Many colorimetric test-kits have a shelf life up to three years, sometimes without refrigeration. Some test-kit manufacturers also offer "eco-friendly test-kits" which attempt to avoid the use of hazardous chemicals and minimize the overall carbon footprint.

However, users should always ensure that any method selected is fit for the intended purpose.

Many test-kits now contain all or virtually all the reagents necessary for the analysis to be performed in the actual measuring cell. This is typically an optical grade glass tube 12 mm to 15 mm in diameter that is inserted directly into the test-kit manufacturer's photometer. Any reagents that need to be added for the test are typically added via dose metered bottles, single volume automatic pipettes with disposable tips or rapidly dissolving tablets. This can help minimize operator error. Other systems use vacuum-sealed glass ampoules that contain the pre-measured chemistry reagent, and which do not require measuring or mixing of the reagent or measuring of the sample volume. Such ampoule-based systems draw the correct sample volume automatically into the ampoule, and further help to reduce operator error and increase the likelihood of repeatable results. The vacuum-sealed ampoule helps to keep the reagent fresh and ready for use and leads to test-kits with long shelf lives.

Many test-kits have a barcode on each of these measuring cells, the photometer then automatically selects the correct method of analysis. Again, this can help reduce potential operator error.

2.5.3 Visual comparator versus photometric detection

In this British Standard, colorimetric methods refer to methods that rely upon the absorption of light. This can be detected visually using colour comparators or electronically using a photometer. The cost of reliable fit-for-purpose photometers is now not that much greater than colour comparators. It is generally agreed that photometric measurements are more reliable and robust than the subjective visual comparison measurements as they reduce operator error, especially that due to colour discrimination.

It is important to emphasize that with visual colour comparison methods, precision and accuracy are dependent upon the visual acuity of the test-kit user and are also influenced by the environmental test conditions (e.g. ambient light intensity). Therefore meaningful comparative performance data (data trends or comparisons) might be less accurate than instrumental methods – more reliable, accurate and consistent results might be obtained using the equivalent on-site photometric method. The key criterion for selecting a technique is that all data generated should be fit-for-purpose.

While photometric analysis does offer an "anticipated" advantage over the visual colorimetric finish, this will not always be the case. There are some cases where detection for a particular method can be improved by the use of a longer path length than typically offered on handheld or even bench top photometers and spectrophotometers; the visual approach then offers an advantage as it greatly increases the practical cell path length. While a standard photometer can read up to approximately 20 mm cell path length using standard cell holders, colour comparators that use equivalent vacuum sealed glass ampoule comparators (containing a dye equivalent to a specific colour concentration standard), allow the analyst to "read" the colour on path lengths of 100 mm and 250 mm, depending on the comparator being used. Assuming good visual discrimination by the operator, this additional sensitivity might not be achievable with a commercially-available photometer or spectrophotometer. In the

analysis of trace levels of chemicals in water, where detection limits are critical, this added benefit could be advantageous.

Most visual test-kits for a given format are usable over a concentration range of about one order of magnitude, whilst a photometric method might be usable over a range of up to two orders of magnitude. The user will need to confirm the actual calibration range.

Most test-kit manufacturers supply both visual and photometric options for a given parameter. On-site photometric equipment is relatively inexpensive, reliable and robust. Results can be made fully traceable using the memory storage facilities of most photometers and might help avoid transcription errors. In addition, some commercial diode-array test-kit photometers permit simultaneous measurement at several different wavelengths. As turbidity is generally independent of the wavelength over the wavelength range of a typical absorption band, any turbidity present in the sample solution can be recognized by the photometer and automatically corrected.

2.5.4 Titrimetric methods

The titrimetric test methods described in this British Standard are normally based upon laboratory reference test methods which are identified within the described test methods described in Section 4. To assist ease of use in on-site situations, only indicator colour end-point methods are described and instrumental end-points are not included. The test apparatus identified in the reference methods in Section 4 might not be suitable for use outside of a designated room/area testing facility.

Test-kit manufacturers now commonly supply alternative, more robust apparatus specifically designed for in situ on-site use. Commercial drop count or tablet count titrimetric test-kit methods are widely available.

Titrimetry is less widely used than colorimetry and typically it is less convenient to use and much less sensitive. There are a number of techniques used, depending on the accuracy and discrimination required, designed to increase the speed and convenience of in situ on-site titrimetry:

- a) *Tablet count procedure.* This uses solid reagents, bound into a tablet together with indicator, as is the titrant and is based on the number of tablets added required to reach the end point in a known test portion of test sample.
- b) *Drop count procedure.* This relies on counting the number of drops of titrant added from a special dropper used to reach the end point in a known test portion of test sample.
- c) *Digital titrators,* with the titrant supplied in disposable plastics containers to fit the titrator. These give titration accuracy equivalent to the use of standard burettes but can be used in situ, on site. There are a number of titrators available which are hand-held and can be operated one-handed. Associated meters are available which can be programmed to give a direct readout of concentration.
- d) *Reverse titration.* The indicator solution and reagent is contained inside a vacuum-sealed, self-filling ampoule. A flexible valve assembly is attached to the tip of the ampoule. The tip of the ampoule is snapped with the valve assembly in place. The tip

of the valve assembly is then immersed in the sample (using an optional titrettor), and small amounts of sample are drawn into the ampoule (the ampoule being gently rocked between each addition to ensure the reagent, indicator and sample are properly mixed) until the end point is reached. The ampoule is then inverted and the analyte concentration reading is read from the printed scale on the side of the ampoule.

2.5.5 Instrumental methods

NOTE An instrumental method is defined in this standard as a method that employs non-photometric instrumentation that can be used for in situ on-site measurements. It can also be used in a designated room/area testing facility analysis.

The instrumental test methods described in this British Standard are based upon laboratory reference test methods (the relevant BS is identified in the 'General' subclause of each method). Commercial instruments are not identified and only the general principles of the test instruments are described together with the test procedure. For all instrumental test methods described, instruments specially developed or adapted from laboratory systems are available for on-site use. Many parameters measured by instrumental test methods are temperature dependent. Wherever appropriate, on-site instruments should possess automatic temperature compensation and/or temperature measurement especially for "in-situ measurements".

Direct reading instruments for tests such as pH, turbidity, dissolved oxygen and electrical conductivity have portable equivalents to the fixed laboratory instruments specifically intended for testing external to the laboratory. Commercially available meters vary enormously in cost and degree of complexity. Most are battery operated, easily portable meters which are simple to use and with a read-out in direct concentration units. Fluoride analysis using an ion selective electrode (ISE) is only considered suitable for use in a designated room/area testing facility. (Haarhoff 2003 [16] and Noh and Coetzee 2006 [17])

2.5.6 Applicability of test methods

The test methods outlined in this British Standard have been chosen on the basis of being widely used to fulfil the circumstances and needs for on-site testing. Brief reference might also be made to alternative test methods or method formats within individual test methods which (depending on local circumstances) could be more applicable.

NOTE Some important determinands cannot be readily determined on-site because of method complexity and/or health and safety reasons. However, many of these can be determined in a designated room/area testing facility. Annex I identifies some of the tests that fall into this category.

2.5.7 Advantages and disadvantages of commercial test-kits versus laboratory testing

Assuming the available commercial test-kits for chemical testing of water quality are of acceptable performance as agreed with the user of the information, the advantages include:

- a) no preparation of reagents is required;
- b) greater safety results from pre-packaging of reagents;
- c) convenience, speed and ease of use are improved with simplified techniques;
- d) minimal sample deterioration from the use of fresh samples;

- e) no need to develop in-house portable systems;
- f) transport of kit and reagents is simpler and safer using the manufacturer's safety packaging;
- g) frequency of testing and/or immediacy of test results for control purposes might be increased; and
- h) the kit and reagents are readily stored and ready for use.

The disadvantages are chiefly involved with cost and the reliability and accuracy of the system, and include:

- 1) cost per test might be higher when using the more advanced commercial test systems; typically, commercial colour comparator or photometric test unit costs are relatively low when compared with small batches of laboratory tests;
- 2) the within batch and between batch reliability of test-kits for relevant matrices is sometimes not available from manufacturers; accuracy and reliability of the test-kits are probably inferior to reference test methods undertaken within a laboratory; however, they should be able to provide results of adequate accuracy and meet the principle of "fit-for-the-purpose-required"; it is the responsibility of the user to ensure that this is the case;
- 3) interferences might be incompletely documented and biased results might then be observed for some sample matrices; some test-kits do, however, document the most common interferences.

2.5.8 Performance characteristics

Performance data for the test methods in this British Standard might be compromised by the conditions encountered external to the laboratory environment. Direct comparison of commercial test-kits and reference test methods using known standards and typical test samples provides useful test data to establish the precision and accuracy of the test method and the merit of using an alternative test-kit. It is prudent to perform control determinations to act as a supervisory confirmatory test for both the operator and test method. This will also verify the continuing performance of commercial test-kits. Periodic comparative testing should be undertaken using the test-kit and an experienced person using both standards and real samples. If discrepancies are observed efforts should be made to identify the causes of the discrepancy (e.g. the test-kit operator or the test-kit method).

The basic performance characteristics of the test-kit are usually supplied by the kit manufacturer.

2.6 Apparatus and equipment

2.6.1 General

Portability and safety are essential requirements for all test-kits used for on-site measurements. Apparatus and equipment fabricated from suitable plastics material are widely used and available as alternatives to conventional laboratory glassware.

Commonly, commercial kits are housed in custom-made carrying cases designed to improve the safety, portability and ease of use of test-kits. The test equipment provided with commercial test-kits is dependent on the test format selected. Test equipment associated with the various formats available is described in 2.6.2 to 2.6.5.

2.6.2 Visual colour comparison test-kits

2.6.2.1 General

Test-kits are commercially available based on liquid, glass, plastics and printed card colour standards.

Competing systems differ in the number of colour standards provided, which affects the degree of discrimination afforded between individual concentration values. All the colour standards except for the printed standards are transparent standards. The accuracy achieved is very dependent on the ability of the manufacturer to achieve consistency, accuracy and long-term stability of representation of the colour and colour density of the standards. It also depends upon the discriminatory power in observing the colour by the test-kit user. Many test-kits employing visual measurements have low-cost portable photometer options which are not sensitive to ambient light levels or the visual acuity of the operator. It is generally accepted that these give more reliable results than equivalent visual methods.

2.6.2.2 Colour disc comparator system

The test equipment consists of a comparator unit which holds the colour disc and the test cuvettes for both blank and test samples. Both discs and cuvettes are interchangeable and are selected as specified by the test-kit manufacturer. The colour disc consists of a series of 8 to 10 discrete transparent glass or plastics standards, covering the test range, set around the circumference of the disc in ascending colour density. Alternatively, the entire disc is made of transparent glass or plastic which has a continuously increasing colour density gradient around its circumference. Both types are permanently marked in concentration units of the determinand being measured, equivalent to the colour density of the test solution. Background illumination aids for use where natural light levels are poor are available from most manufacturers of colour disc systems.

2.6.2.3 Liquid colour standards comparator system

The test equipment consists of a series of individual liquid colour standards, covering the test range, sealed into identical glass ampoules. Evacuated test ampoules containing the test reagents act as the test cuvettes and are identical to the standard ampoules. Up to 10 colour standards are provided ensuring colour discrimination equivalent to the colour disc system.

2.6.2.4 Colour cube comparator system

The test equipment is simple and consists of combined twin cuvettes. One cuvette is made up of a series of colour segments of ascending colour density. The sample is placed into the second empty cuvette and the appropriate reagents added, to allow direct visual

comparison. Where only a small number of colour segments are available, this limits the range and discrimination of the test method.

2.6.2.5 Printed colour card comparator system

The test equipment is usually minimal and consists of a card on which is printed a series of colour standards equivalent to the colours of the range of the test method. The colour standards are viewed through the blank solution until a colour match is achieved against the coloured-up test sample viewed against a white background. Colour matching by reflectance is typically not as exact as by transparency matching and additionally the colour printing needs to be exact. Colour standards might not be permanent – verification against known standards should be carried out on a regular basis.

NOTE Accurate results are very dependent on the ability of the manufacturer to represent correctly the colour and colour density of the colour standards and the person undertaking the test to discriminate differences between the colour of the standard and the colour of the test solution following addition of reagents. Also the colour (spectral distribution) of the incident light can also affect the accuracy of colour discrimination (due to the different response of the printed ink and the test solution).

2.6.3 Portable colorimeter systems

Test-kits incorporating portable colorimeters are very common. They provide consistent colour discrimination and eliminate the operator-dependence of comparator systems. Advances in microelectronics have allowed a significant amount of complexity to be incorporated within small hand-held instruments. Also, many systems allow storage of results that can subsequently be printed out or electronically downloaded. This helps to minimize transcription errors. Available systems vary significantly in cost and complexity but typically basic systems are not that much more expensive than the colour comparator test-kits. Selection is dependent on local requirements and needs.

Many test-kits now contain all or virtually all the reagents necessary for the analysis in the actual measuring cell. This is typically an optical grade glass tube 12 mm to 15 mm in diameter that is inserted directly into the test-kit manufacturer's photometer. Any reagents that need to be added for the test are typically added via dose-metered bottles, single-volume automatic pipettes with disposable tips or rapidly dissolving tablets. This should minimize operator error.

Many test-kits have a barcode on each of these cells, the photometer then automatically selects the correct method of analysis; this should reduce potential operator error.

Most test-kit photometers use narrow band interference filters with a photodiode detector or the more expensive versions can use a simple grating monochromator normally with photodiode or photodiode array (simultaneous multiwavelength detection).

2.6.4 Equipment for titrimetric on-site tests

Both complete test-kits and individual on-site-use burettes of innovative design are commercially available. The systems are designed for portability and ease of use and can be utilized for most standard

titrimetric tests. One simple option consists of a reverse titration test-kit employing a self-filling ampoule with valve assembly. Other titration test-kits options are available using a tablet count or drop count procedure, or a plastic burette with an integral titrant reservoir. Manual or electrically operated piston-based titrators with digital readout offer a greater degree of accuracy and ease of use. All manual or electrically operated piston based titrators should be regularly calibrated to ensure acceptable accuracy is maintained.

2.6.5 Equipment for instrumental tests

Advances in microelectronics have allowed the development of portable-use test equipment based on standard laboratory test equipment. There is a wide choice of commercially-available equipment. For example, turbidity; pH; dissolved oxygen, electrical conductivity; redox potential; anodic stripping voltammetry and fluorimetry. In the absence of formal testing protocols, selection of the most suitable equipment remains a local decision.

2.7 Reagents

2.7.1 General

Commercial ready-to-use test-kits/ready-made appropriate test-kit reagents should always be used for the test methods cited in this British Standard as this avoids preparing test reagents under non-laboratory conditions.

Distilled or RO/de-ionized water conforming to grade 3 of BS EN ISO 3696:1995 should be used.

If any reagents are prepared, chemicals of recognized analytical quality should always be used.

2.7.2 Pre-packaged reagents for colorimetric test-kits

These are supplied by the manufacturer and are specific to each manufacturer's own test-kit methods. Dependent on the manufacturer, these can be in liquid, powder or tablet form or a combination of different forms for different test methods. Pre-packaged reagents can also consist of single or mixed reagents. The reagents are not usually interchangeable between different systems and replacement refills for pre-packaged reagents should normally be obtained from the relevant test-kit manufacturer. Refills save the expense of purchasing an entire kit by permitting the reuse of major kit components.

Some of these products can have a long shelf life (e.g. up to three years, sometimes without refrigeration).

Some test-kit manufacturers also offer "eco-friendly test-kits" an attempt to avoid the use of hazardous chemicals and minimize the overall carbon footprint. However, users should always ensure that any method selected is fit for the intended purpose.

2.7.3 Pre-packaged reagents for titrimetric test-kits

The concentration of test-kit titration solutions are frequently different from the concentrations described in the reference

titrimetric test methods. They are usually more concentrated, to reduce the titrant volume used, because of the small capacity of reagent cartridges or reagent containers used in portable titrimetric test-kits. It is important to identify the correct calculation factors for the reagent strength used. A calculation factor is not, however, applicable for a reverse-titration test-kit as a scale is provided on the side of the ampoule, providing concentration reading in the units specified for that particular analyte.

Tablet, drop test and reverse-titration methods are alternative commercially-available simplified titrations. These test methods have lower analytical accuracy expectations than conventional laboratory titration techniques. These tests are dependent on the accuracy and reproducibility of the tablets or test drops, which depend on manufacturing tolerances. The titration skills of the test-kit user also affects the accuracy. Endpoint overshoot by adding too many tablets or test drops is another potential source of error. This is affected by the number of drop or tablet incremental additions. The greater the number of additions (i.e. the smaller the discrimination of each tablet or drop), the smaller the potential error.

2.7.4 Calibration and working standards

Water quality test-kits are intended for testing waters which usually lie in the pH range 4 to 10 and standard solutions should be within the same range, otherwise erroneous results might occur. Acid-based metal and metalloid standards might be unsuitable and alternative standards prepared from neutral salts should be used. Advice should be obtained from the relevant test-kit manufacturer where any doubt exists regarding suitable standards. Most test-kit manufacturers will supply suitable standard solutions. These should be used within their shelf-life.

2.7.5 Further background information on reagents

Both liquid and solid reagents used in test-kits are pre-packaged in a variety of formats and formulations. Solid reagents are supplied in powder form as individual powder pillows for individual tests and as bottles of powder containing a micro measuring spoon. Alternatively, solid reagents can be supplied in tablet form. In either form, the solid reagents can combine test reagents, indicators and interference suppressants. Liquid reagents are also supplied with some test-kits, either in dropping bottles or as reagent bottles from which aliquots are taken. A significant development in test-kit systems has been the packaging of measured doses of reagents, both liquid and solid, into individual capped tubes which act as disposable cuvettes designed for a single individual test. This has enhanced the ease and convenience of use, requiring in many cases only the addition of a specified aliquot of test sample. A novel modification of this system seals the reagents in evacuated glass vials which act as the cuvette. When the seal at the tip of the glass vial is broken beneath the surface of the sample, it automatically draws in a fixed volume of the sample rather than requiring a measured addition of sample by the analyst. Sealing under vacuum additionally prolongs the reagent shelf life.

Typically these single pre-packaged cuvettes for colorimetric determinations are made by manufacturers for use only with the portable colorimeters or comparators sold by that manufacturer.

However, portable colorimeters are now commercially available which can be self-programmed to use the majority of pre-packaged single test cuvettes available from different manufacturers.

Section 3: Quality assurance and quality control (QA/QC)

3.1 Introduction

Not all necessary QA/QC requirements might be covered by the instructions of the manufacturer of the test-kit. More QA/QC requirements might also be needed, even if the test-kit manufacturer complies with BS EN ISO 9001 and/or MCERTS.

3.2 Causes of “unfit-for-purpose” results

3.2.1 General

The main causes of unfit-for-purpose test-kit results are considered to be:

- a) Incompetence of the operator (need for competence training).
- b) Matrix interferences (although the initial method validation should indicate if this is a potential problem, it is not always possible to cover all potential extreme sample matrices that might be encountered).
- c) Inappropriate method/test-kit employed (need a validated test-kit method that meets the client performance targets).
- d) Contamination (need for principles of good laboratory practice to be understood and employed). This also includes carryover (1.3.27) caused by contamination of a subsequent sample by a previous sample, typically due to incomplete cleaning of a reused test-kit component.
- e) Inappropriate sample pre-treatment (this needs to be agreed with the client).

3.2.2 Fitness-for-purpose-demonstration

In order to demonstrate fitness-for-purpose of the test-kit results, a hierarchical approach is recommended.

NOTE For example, the general approach outlined by the RSC Analytical Methods Technical Brief No. 28 (AMC 2007) [18].

- a) *Calibration.* Test-kit instrumentation should be calibrated or verified at specified intervals (or prior to use) against measurement standards traceable to international or national standards; where no such standards exist, the basis for calibration or verification should be recorded.
- b) *Qualification.* Qualification is the collection of documented evidence that an instrument performs suitably for its intended purpose and that it is properly maintained and calibrated. Use of qualified test-kit instruments in analysis contributes to confidence in the veracity of generated test-kit data.
- c) *Validation* (See also 3.4). Validation relates to the “fitness-for-purpose” of test-kit methods and associated analysis procedures. Ideally, this should include comparison data supplied by the test manufacturer that the test-kit method gives “equivalent” results to a fully characterized appropriate routine laboratory analysis

method for the parameter in question of a known and satisfactory performance for a range of relevant sample matrices. Any samples where there are significant discrepancies in the results should be checked using samples spiked with a known amount of the parameter by both methods to determine if one or both methods are in error.

A convenient illustration of this hierarchical approach is:

- 1) QC checks;
- 2) System suitability checks (1.3.12);
- 3) Analytical method validation;
- 4) Analytical instrument qualification [3.2.2b)].

3.2.3 Data integrity

A suitable framework for analytical data integrity is:

- a) The test-kit instrumentation used has been qualified and calibrated.
- b) The test-kit method selected is based upon sound scientific principles and has been shown to be robust, reliable and fit-for-purpose for the type of test materials being analysed under the conditions of use.
- c) The test sample is representative and sufficiently close to homogeneous.
- d) A person who is both competent and appropriately trained has carried out the analysis.
- e) The integrity of the calculation used to arrive at the result and its uncertainty is correct and statistically sound.
- f) Some form of internal quality control is carried out in every run of analysis. (A "run" is the period in which repeatability conditions prevail. This is typically a single batch of analysis where the user works without a break or without switching off any instrumentation. A quality control sample should be run at least every 20 samples.)
- g) Some form of proficiency testing is undertaken whenever practicable.
- h) Independent audits and assessments of the whole analytical system are carried out at intervals.

3.2.4 QA of on-site or test-kit methods

Similar to the routine analysis procedures employed in a laboratory, dependent upon the handling and nature of the method in question, the quality assurance of on-site or test-kit methods can be subject to very different requirements. In the case of a method which is used regularly (e.g. more than once per week), the same quality assurance measures can be similar to the reference method.

These include the following, and see DD ENV ISO 13530 and BS ISO 17381:

- a) multiple testing;
- b) measurements of standards and possible reference materials;

- c) internal AQC (1.3.21), analysis of known samples or standards carried out with each batch of analysis and all the results are used to statistically control the procedure using Shewhart or equivalent charts;
- d) plausibility tests by means of dilution and standard addition;
- e) comparative tests with reference methods;
- f) interlaboratory tests (proficiency testing);
- g) keeping a control chart.

In the case of methods giving discrete analytical results, Shewhart or Range type control charts (see DD ENV ISO 13530 and Annex H) can be employed, whereas a target value chart is suitable for methods stating result ranges. In the first case, the control values are subject to statistical evaluation. In the latter, the control values are not subjected to statistical evaluation (mean value, standard error, control limits, warning limits) but instead it is decided whether or not they lie within a pre-defined tolerance range (yes/no decision). In this way, target value charts for blank values, mean values, recovery rates and range can be maintained.

In the case of occasional use (e.g. in the event of a fault in a waste-water facility), the points mentioned above would necessitate an excessive, not goal-oriented, degree of time and expenditure. However, personnel should, as far as possible, be fully trained in the use of the method and the test method should be characterized and validated in terms of its performance characteristics before it is used for the first time and periodic (e.g. monthly) checks made of performance throughout the life of the kit, and especially after maintenance of any test equipment. In certain individual cases, multiple testing, the measurement of standards and plausibility checks through dilution of real samples or standard addition are useful as simple check measures. Sample dilution and standard addition are particularly useful when sample matrix effects are either unknown or it is suspected that they might significantly influence the result of the test.

Finally, the selection of suitable quality assurance measures depends upon the specific aim of the analysis. The decision concerning the extent of the measures to be implemented, the results of these measures and their assessment should be documented.

All test-kit methods should be fully documented by the test-kit users and include how to take a representative and relevant sample, QA/QC procedures, relevant simple system suitability checks (SSCs) and should also include the author, version number of the documentation and the method approval date. SSCs are simple tests to ensure that the associated test-kit instrumentation is functioning within specified limits before commencing analysis. Each step in the method should be described sequentially and in full, should be unambiguous and written as simply as possible.

Relevant QA/QC protocols need to be drawn up by the test-kit user for their particular application. The manufacturer's instruction leaflet supplied with the test-kit might not be sufficiently detailed for carrying out fit-for-purpose analysis. Users should not rely on basic test-kit instructions alone, they might need to be rewritten to cover the context of the particular application and need to include sampling, sample preservation if required and sub-sampling and sample pre-treatment; all QA/QC aspects; as well as health and safety considerations with

respect to the test-kit, and disposing of any waste after carrying out the test. The test method supplied with the kit should form the basis of the method. All method documentation should be fully traceable and signed off by a responsible experienced person.

Consideration should be given to laminating/encapsulating documented methods used in the "outside environment," so they can more readily withstand adverse environmental conditions. Also this will assist in preventing unapproved amendments being subsequently added.

It is the responsibility of the organisation/person carrying out the test method to ensure that it is capable of producing results meeting the performance requirements of the client. The use of the test methods described in this British Standard should not be taken as a guarantee that results will be of sufficient accuracy or indeed even fit-for-purpose. Additionally, the performance of the test method used can be affected by both non-standard test conditions, principally variable environmental site conditions (e.g. high or low temperatures and/or extreme ambient light intensity) and most importantly by the capability and experience of the person using the test method. As a consequence, no performance data are given for individual test methods in this standard. Most test-kit manufacturers will provide "best case" performance data upon request. The performance data generated with the equivalent laboratory methods, for example in the series BS 2690 and/or BS 6068, can be used as a guide for method performance or method selection.

The vast majority of users of this standard will not be trained analysts and are likely to use commercial test-kits rather than prepare their own test-kits or devise on-site methods.

It is not possible to be totally prescriptive with respect to the amount of QA/QC to be employed for all on-site methods. On a pragmatic basis, the amount of QA/QC that can reasonably be carried out in situ on-site is generally somewhat less than can be carried out in a designated room/area testing facility or laboratory. Some simple screening methods that are detecting presence or absence of a given parameter or demonstrating that the concentration of a given parameter is significantly below a maximum permitted concentration will require less QA/QC than a method trying to assess the actual concentration of a regulatory parameter.

EXAMPLE: Below maximum permitted concentration.

Assessment of the chloride concentration of a sample prior to carrying out a COD test to ensure that the chloride level is below a stated concentration (chloride is a significant interferent in the test and test-kits are designed to cope with differing levels of interference).

EXAMPLE: Actual concentration.

Measurement of the actual COD of the sample.

Measurements of a single well-characterized industrial effluent will normally require less QA/QC than measurements on a wide range of much less characterized effluent samples from a number of different sources. Some useful information on screening methods is available (SWIFT 2003 [6]).

The organisation responsible for the analysis should justify the adequacy of the QA/QC protocols adopted in demonstrating the fitness for purpose of results for a particular analysis.

Pritchard and Barwick (2007) [19] have produced a very concise overview of quality assurance in analytical chemistry; users are

strongly recommended to consult this open learning textbook that should be of considerable benefit to non-analysts in understanding this key subject area.

3.3 Confidence interval considerations

EXAMPLE

Direct electronic-readout test-kit instrumentation might indicate a result for a given parameter of, for example, 51.2 mg/L sulfate, the user of the result might erroneously assume that the result is exactly 51.2 mg/L rather than 51.1 mg/L or 51.3 mg/L that has then been "rounded" to 51.2 mg/L. The result uncertainty, however, can often be as much as $\pm 20\%$ of the concentration being measured for some test-kit parameters and can be higher. Assuming this to be the case in this instance, then this result would be better quoted as (51 ± 10) mg/L so the user can appreciate the uncertainty associated with the result.

Annex C gives an indication of the expected confidence intervals of top-of-the-range test-kits from two different manufacturers for the relevant manufacturer supplied quality control solutions. This Annex is only included in this standard for purely indicative purposes. Most major manufacturers of test-kits provide suitable QC standards with specified 95% confidence intervals for many parameters. These supplied QC test solutions can be directly traced back to national or international primary standards. If the user cannot meet their test-kit manufacturer's indicated confidence limits with the specified QC solutions, then a documented investigation should be carried out to determine the reasons for this and actions instituted to rectify the causes found.

Many commercial test-kits are also able to provide some indicative basic performance data. If this is not readily available, then the user should contact the manufacturer and if this information cannot be provided, then the user should seriously consider using an alternative test-kit where this information is available. This should be regarded as "best case" data as it is usually obtained from matrix-free standards. Performance in real sample matrices could be significantly worse.

3.4 Method validation (Assessing method performance characteristics)

3.4.1 Evaluation

A test-kit should be formally evaluated by the user in terms of:

- Concentration range.
- Accuracy/bias using typical sample matrices to be tested by appropriate spiking. A worst case matrix should be included.
NOTE Cheeseman et al. (1989) [20] give further advice on this issue.
- Within and between batch precision for typical relevant water sample matrices.
- Method detection limit.
- Potential sample matrix interference effects.
- Linearity of the calibration function.
- Operator bias (this would include visual acuity tests if relevant).

- h) Test-kit reproducibility (between different batches of test-kits). This information would normally be supplied by the test-kit manufacturer.
- i) Speed of analysis.
- j) If the test-kit is to be used on-site outside of a designated room/ area testing facility, then the likely extreme temperatures and the variation in ambient light considerations need to be taken into account. (e.g. some colorimetric reactions proceed much slower at 4 °C than at the normal laboratory or designated room/ area testing facility temperature of approximately 20 °C). Tests should be carried out on some standards at just below the lowest temperature likely to be encountered. Also the effect of bright sunlight should be assessed on colorimetric methods. and artificial light or low levels of natural light on visual comparison methods.
- k) QA/QC targets and system suitability (SSCs) checks of the analysis.
- l) Training and experience needed by test-kit users.

The results of this evaluation should then be formally documented.

3.4.2 Validation under typical operating conditions

If possible, a series of standards, a blank, a range of typical samples and some spiked samples should be run on-site under typical operating conditions with the test-kit. Then, all the tested samples should be brought back to a laboratory and tested again under laboratory environmental conditions, with minimal delay.

At the same time, the same set of samples should be analysed using an equivalent fully characterized appropriate routine laboratory analysis method procedure. This should demonstrate if the test-kit on-site results agree with test results obtained under defined laboratory environmental conditions. It will also show if the test-kit method gives equivalent results to a laboratory-equivalent routine, validated method. It should be appreciated that unstable determinands can result in an apparent positive bias of on-site measurement relative to the subsequent laboratory results. In this instance, it is the lab-based results that are biased rather than the on-site measurements.

If this is considered too onerous for the application, then a series of standards, a blank, a range of typical samples with the parameter of interest concentration at less than 30% of the calibration range and then the same samples spiked with 50 – 70% of the calibration range should be run.

Method performance targets should then be set up for all relevant measurements (confidence limits for blanks, standards and recoveries). Some test-kit manufacturers provide spiking solutions for their test-kits that can be used to detect measurement errors caused by the effect of the sample matrix on the sensitivity (i.e. sample matrix bias effects). These spiking solutions can also be prepared in a suitable designated room/area testing facility or a laboratory if they are not available from the test-kit manufacturer.

3.4.3 Critical concentration spiking and detection

Ideally, a typical sample with a low or negligible level of analyte should be spiked with the analyte using a spiking addition equivalent to the regulatory or critical concentration.

Other bias effects can be reduced by appropriately pre-treating the sample (e.g. filtering the sample to remove suspended solids for parameters such as chloride, sulfate, nitrite, ammonia or chloride) using photometric final detection. These parameters are unlikely to be associated with any suspended solids in the sample, so removal of the solids should not affect the result.

3.4.4 Blanks

A sample-specific blank value for a photometric method can be obtained by carrying out the cuvette test, replacing the colour reaction reagent with RO/DI water, on a sample and then measuring the sample. The result is attributable purely to the characteristics of the sample (turbidity/colour), and is subsequently deducted from the result of the analysis using the actual sample.

3.4.5 Adsorbed metals

Many toxic metals in natural waters and effluents tend to be adsorbed on to suspended particulate matter at neutral sample pH values and (in order to determine the total amount present in the sample) might require an acid digestion step followed by filtration and neutralization if a colorimetric method is to be employed. Careful consideration of the key aspect of sample pre-treatment is required before any method is adopted. For river and raw water metal analysis, the filtrate from a 0.45 µm filtered sample is usually analysed – this determines the dissolved (available) metals. The filtration should be carried out at the time of sampling.

3.5 Routine quality control

There is a need to ensure fit-for-purpose analysis on a long-term on-going basis: basic system suitability checks (SSCs) should be run for each batch of analysis; an appropriate blank and QC should also be run. Then, on a regular basis (e.g. weekly or monthly) check spike recovery on a typical sample (as described above). All data should be recorded on suitable control sheets or charts. Many test-kit manufacturers will supply suitable standards and spiking solutions.

The action limit values on the QC chart for test-kits will often be fixed limits agreed with the result end user rather than statistically derived from the method validation data. This is considered adequate for test-kit usage for many simple applications, but might not be sufficient when the value of the result is critical. Arbitrary warning limits can also be set up for simple applications if required.

For applications where the value of the result is critical (e.g. regulatory monitoring or trend monitoring), a full Shewhart-type control chart or charts should be used with statistically derived limits.

All QC failures should be fully investigated as to the reason for the failure and the investigation findings and any remedial action instituted should be recorded.

With very few exceptions (e.g. redox potential and pH), a result of “zero” should never be quoted. The appropriate validated less than (detection limit) value should always be cited. Where a result of zero is appropriate it should be stated as 0 or 0.0 or 0.00 etc. depending on the precision of the result.

All users should seriously consider participating in a relevant proficiency testing scheme: some test-kit manufacturers run such schemes for certain of their test-kits, third party testing comparison results are useful for confirming on-going fitness-of-purpose results. All failures should be fully investigated as to the reason for failure and the investigation, findings and any remedial action instituted should be recorded.

The primary purpose of routine quality control checks is to give early indication of a problem and instigate an investigation and corrective action. If carried out correctly and proper records of investigations and actions kept, it also provides demonstration of continuing fitness for purpose of results.

3.6 Target test-kit requirements

Before selecting a test-kit for a given parameter, users should carefully consider with the users of the data, the target requirements (including sampling considerations and performance characteristics) that are needed to obtain fit-for-purpose results in all sample matrices likely to be encountered. These requirements should then be carefully documented and formally agreed with the final end user(s) of the data. Then the test-kit performance requirements should be discussed with the test-kit supplier with respect to the range of sample matrices likely to be encountered to help ensure that an appropriate fit-for-purpose test-kit is employed.

Annexes D and E set out examples of how this might be set out in a simple pro-forma, both for a parameter analysed in a designated room/area testing facility and for a parameter analysed on-site at the actual sampling location. These pro formas then need to be assessed and signed off when considered satisfactory. Obtaining the data to complete the pro forma can be carried out either by the user or (if they have the necessary facilities) then some of the data can be obtained by a competent laboratory, after discussions with the test-kit user. If the targets set cannot be met with the chosen test-kit, then either a more appropriate test-kit should be selected and tried or the target specification might be relaxed with the agreement of the end user. The competent laboratory might also be able to cite performance data generated using the equivalent laboratory methods, for example those in the series BS 2690 or/and BS 6068, can be used as a guide for method performance. The test-kit user will also have to demonstrate competence in using the test-kit as part of their training (see Annex A).

The most important factor is that both the user of the test-kit and the primary user of the results from the test-kit are aware of the basic performance characteristics of the test-kit and any limitations.

Major manufacturers of test-kits provide advice on QA/QC and as stated previously many also provide suitable QC standards with specified confidence intervals. In addition, some provide spiking solutions for their test-kits that can be used to detect measurement errors caused by the effect of the sample matrix (i.e. bias effects) (see 1.3.7). Some manufacturers supply secondary standards for checking the wavelength accuracy and linearity of the absorbance of the photometer as well as simple portable electronic temperature sensor for temperature control and calibration of heating blocks.

Also photometric standards are available for checking the calibration of pipettes on-site by pipetting the supplied standard into a specified volume volumetric flask and diluting to volume with water and measuring the absorbance at a specified wavelength on the test-kit photometer.

The routine QA/QC that should be employed needs careful consideration and should never be overlooked and should always form part of the test-kit user's training.

Annexes F and G set out examples of how this might be set out in a simple pro forma for test room/area and general on-site parameters. Annex H gives an example of a control chart. Some parameters might require a modified form. These pro formas then need to be assessed and signed off when considered satisfactory. The data required to complete the pro forma should be obtained by the trained user of the test-kit with each batch of analysis and immediately documented.

3.7 Concentration ranges of methods

The application range of a test method should be stated by the test-kit manufacturer in their literature or test-kit instructions. The stated range in many cases can be extended by pre-dilution of the test sample or (where the result is independent of the sample volume) a smaller test portion can be used. Depending on local requirements, results can be quoted as "less than" for concentrations below a stated level, or "greater than" for concentrations above a stated level. These upper and lower "levels of interest" should be clearly defined wherever they are applied. Where more than one application range is necessary for any determinand, alternative test methods for the different ranges might be required. For visual colour comparator methods, the user often needs to estimate readings falling between two adjacent colour discs - this is not necessary when photometric measurements are made as a discrete figure is displayed.

3.8 Interferences

Because of the wide range of water types covered in this British Standard the information given on interferences is not comprehensive. There is always the possibility that the water under test might contain substances which will interfere with the test procedure. Test method parameter summaries listed in Annex H identify typical interfering substances, but (in order to keep test methods as simple and as easy to use as possible) information on procedures for overcoming interferences is not always included. The most common interference, particularly for colorimetric test methods, is turbidity, which can easily be identified. The removal of turbidity and the manner of its removal is dependent upon local conditions (see also 2.5.3). Where the test method is similar or traceable to an existing ISO/CEN/British Standard, reference might be made to the standards for information on overcoming interferences. Pre-packaged reagents might include reagents designed to minimize or eliminate known interferences. Commercial test-kit systems might also indicate sample pre-treatment requirements designed to overcome interferences. Realistically, most of these need to be carried out in a designated room/area testing facility (see 2.3).

Section 4: Titrimetric methods for use in a designated room/area testing facility

COMMENTARY ON SECTION 4

It is important to emphasize that in titrimetric methods the precision and accuracy are considerably influenced by the environmental test conditions. However, with the use of a designated test room/area equivalent results to that of a conventional laboratory might be obtained with appropriately trained personnel. The use of titrimetric commercial test-kits suitable for on-site testing are covered in Section 4.

The test procedure and apparatus required for reverse titration based test-kits where the sample is titrated against a fixed volume of appropriate reagent(s) are not described in Section 4. This is because these test-kits require no pre-measurement of a sample aliquot and follow a simplified test methodology. Small volumes of sample are taken up using a simple ball-valve assembly on the tip of an evacuated ampoule containing the appropriate reagents until the colour changes, indicating the equivalence point of the titration. Quantitative results are obtained by reading the scale off of the graduated ampoule. [See also 2.5.4 d)].

Reverse titration test-kits are described in 2.5.4, 2.6.4 and 2.7.3. Reverse titration test-kits are currently available for all the analyte tests specified in Section 4, with the exception of 4.6 Sulfate and 4.8 Zinc.

4.1 Determination of alkalinity

4.1.1 General

This method is based on BS 2690-109. The quantitative capacity of aqueous media to react with hydrogen ions is known as alkalinity.

The alkalinity of a water is primarily a function of the bicarbonate, carbonate, and hydroxide concentrations. This method measures the alkalinity due to the presence of each of the three anions.

Commercial titrimetric test-kits based on the same principle are available. They typically employ novel titration systems in place of conventional burettes and also use modified test reagents (see 2.7.3).

4.1.2 Scope of this method

This test method is recommended as a designated room/area testing facility test for the determination of alkalinity. It is applicable to potable, raw and lightly polluted waters.

The analytical range for alkalinity is from 2.0 mg/L to 500 mg/L expressed as calcium carbonate (CaCO_3) using a test portion of 100 mL. The range might be extended using smaller test portions. Where the total volume of titrant exceeds 50 mL the test is repeated using a smaller test portion.

NOTE *Strongly coloured or turbid solutions interfere with the colorimetric end-point.*

Chemically treated waters can include other ions such as phosphate, silicate and borate which affect the pH of the sample and the measurement of alkalinity.

Oxidizing agents present in the sample might bleach indicators.