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Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This Technical Report has been prepared by Technical Committee CEN/TC 264, "Air quality", the secretariat of which is held by DIN.

This document is a working document.

This CEN Report has been prepared by an Ad-Hoc-Group of CEN/TC 264 "Air quality" in co-operation with the European Commission's Joint Research Centre, Ispra, Italy.

This CEN Report is an informative document.

1 Introduction

The European Framework Directive on Ambient Air-quality Assessment and Management, and its related Daughter Directives, require measurements to be made using specified Reference Methods (given in outline in Annex A of this Report), and the results to be reported, on specific air pollutants, with specified data quality objectives, by all Member States within the EU. The main purpose of this CEN Report is to provide guidance to those CEN/TC 264 working groups which are involved in the preparation of Reference Methods to measure ambient air quality as required by the EC Daughter Directives, on the uncertainty evaluation to be carried out in order to conform to these data quality objectives. One further purpose of this Report is to emphasize the essential requirements for appropriate quality assurance (QA) and quality control (QC) procedures in order to ensure that the ongoing field measurements are valid – so as to ensure the EC data quality objectives continue to be met.

It is important to understand that these Daughter Directives specify the principles of the Reference Method to be used, but they rely on the European standardisation body (CEN) to produce detailed Reference Method(s) for each pollutant as European Standard(s), and to arrange for their publication so that they can be made available to Member States. As noted above, the principles of these Reference Methods, which are to be used in the implementation of the Directives, are prescribed in the relevant EC Directives. In some cases, these Methods are specified as continuous or semi-continuous automated instruments, which comprise fully integrated systems, usually including a sampling line and the analytical equipment. In other cases, the Reference Methods are specified as manual or discontinuous methods, which comprise a field sampler and a separate laboratory analytical component. In addition, it should be noted that in certain scenarios the Directives allow indicative methods to be used, which are allowed to have poorer measurement uncertainties.

It is also a requirement of some of the Daughter Directives that Member States meet air quality limit values for a variety of different averaging periods (covering e. g. hourly, daily, and annual averages). For some of the pollutants specified, however, the requirement is only for annual averages.

The European Framework Directive on Ambient Air-quality, and the associated Daughter Directives, also state that the approaches given in the *Guide to the Expression of Uncertainty in Measurement* published by ISO, and given in the International Standards ISO 5725, are to be used for the estimation of the measurement uncertainties of these Reference Methods. Both approaches are mentioned in these Directives as alternatives for the estimation of measurement uncertainty. This Report describes, therefore, in broad terms, the methodology which should be employed by the relevant CEN working groups for the integration of the approaches described in these documents into European Standards, whilst still maintaining a valid overarching statistically-based methodology for determining these measurement uncertainties.

The issues outlined above are covered in this Report. The Report was produced following consultations with an Ad-hoc Group of experts which were convened to consider the issues. These experts are experienced in both ambient air monitoring and in measurement uncertainty statistics. The members of this group and its Chairman are listed in Annex G. It should be noted that all the existing working groups of CEN/TC 264, which are currently involved with the standardisation of ambient air quality measurements, were represented on this Ad-hoc Group.

It should also be noted that Member States may use other methods, as alternatives to these Reference Methods – provided they can demonstrate that these other methods produce results which have been shown to be equivalent to those obtained by the relevant Reference Method. It is not, however, the remit of this Report to discuss the issue of equivalence, or to define how it may be demonstrated.

2 Assumptions and interpretations in this Report of the EC Air Quality Framework and Daughter Directives

2.1 Annexes to the Daughter Directives

Each of the Daughter Directives contains an Annex which specifies the data quality objectives required for the air quality assessment for the particular pollutant species they address. These Annexes specify the 'accuracy' or 'uncertainty' that must be met by the Member States when reporting results, in order to satisfy the requirements of the Directives. For clarity, it is assumed in this Report that the two terms accuracy and uncertainty are synonymous, and therefore in the text of this Report reference will only be made to uncertainty, or to the uncertainty of measurement, as defined in the *Guide to the Expression of Uncertainty of Measurement* (1993) published by ISO – known hereafter in this Report as the GUM. An overview of the GUM is presented in clause 3.2 of this Report.

Included in each of the Annexes of the Daughter Directives, noted above, is an explanatory paragraph on their accuracy (uncertainty) requirements, in which it is explained that “the Guide to the Expression of Uncertainty of Measurements 1993, or ISO 5725-1, Accuracy (Trueness and Precision) of Measurement Methods and Results, shall be used”. As an example of this, the relevant Annex of one of the (draft) Daughter Directives is reproduced in Annex B of this Report, where it can be seen that the uncertainty values required of the Reference Methods for benzene and carbon monoxide are listed.

It is also assumed in this Report that the GUM and ISO 5725 are not mutually exclusive, and that there is no intention in the Daughter Directives to restrict the use by CEN and others to only one or the other. Instead, it is proposed here that the principles of GUM is employed to identify and combine all uncertainties of the appropriate Reference Method, and that ISO 5725 (including some of its six parts – not just the one part mentioned in the Directives) may be used, where appropriate, as an aid to this evaluation (see clause 3 of this Report). It should also be noted in this context that the GUM is a (voluntary) European Standard (ENV 13005).

It has also been necessary for the Ambient-air working groups in CEN/TC 264 to assume that the data quality objectives of the Daughter Directives will be met, provided that the uncertainty of the measurement results obtained with the Reference Methods are within the uncertainties prescribed by these data quality objectives. This is also assumed in this Report, as it is judged that this is a valid assumption for the relevant European Standards.

There are also other statistical ISO standards that may be useful in this evaluation. Some of these are noted below in this Report. It is assumed that the statements in the Daughter Directives do not preclude the use of any of these, or of other relevant standards, where they are applicable, as long as the over-riding principles described in the GUM are followed.

2.2 Definitions and interpretation of measurement uncertainty, level of confidence, and confidence interval, within the Ambient Air Quality Daughter Directives

2.2.1 General

As discussed above, each of the EC Ambient Air Quality Daughter Directives specify data quality objectives (quantified in their Annexes) that EU Member States must conform to, when reporting the results obtained to the European Commission (EC). The definitions associated with measurement uncertainty that also relate to, or are referred to, in the Annexes of these Daughter Directives are discussed below for clarity, and also to serve as a background to the remainder of this Report.

2.2.2 Uncertainty of measurement

The Air Quality Directives require an evaluation of the ‘uncertainty of the assessment (or measurement) methods’ according to GUM and therefore it is clear that the definition of uncertainty used in GUM shall be used. The GUM definition of the term ‘uncertainty of measurement’ is therefore reproduced in this Report for completeness as:

Uncertainty of measurement: Parameter, associated with the result of a measurement, that characterizes the dispersion that would reasonably be attributed to the measurand.

It is also essential to express clearly the conditions under which this uncertainty of the measurement results is to be evaluated. This issue is also covered in this Report.

2.2.3 Level of confidence

The above term ‘uncertainty of measurement’ is used in the Annexes of the Daughter Directives. The term ‘confidence interval’ is, in addition, used in these Annexes of the Daughter Directives, and this is also defined in the GUM. The relationship between the terms ‘confidence interval’ and ‘uncertainty of measurement’ is discussed below in clause 2.2.4. However, it is first useful, for clarification purposes, to discuss the meaning of the term ‘level of confidence’ which is used throughout these discussions.

As described in the GUM, the usual method of determining the level of uncertainty of a given measurement parameter is to determine the combined standard uncertainty u_c of that measured parameter by combining all the individual standard uncertainties, which may be Type A or Type B. It is then necessary to provide a measure of this uncertainty that defines an interval about the measurement result that is expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurement. This is illustrated diagrammatically in Figure 1. This measure of uncertainty is termed the expanded uncertainty, denoted by U, which is obtained by multiplying the standard uncertainty by a coverage factor k:

$$U = k\,u_c$$

As is well known, the result of the measurement is then traditionally expressed as:

$$Y = y \pm U$$

This is interpreted to mean that the best estimate of the value attributed to Y is y and also that $y–U$ to $y+U$ is an interval that may be expected to encompass a large fraction of the distribution of the Y value.

More specifically, U may be interpreted as defining the interval about the measurement result that encompasses a large fraction p of the probability characterised by that result and its associated uncertainty. This fraction p is then the 'level of confidence' of that interval. Generally, and in the specific case of the requirements of the EC Air-quality Daughter Directives this level of confidence p is defined as 95 % – i.e. 95 % of all the individual measurement parameters lie within $\pm U$ of the best estimated value y and only 5 % lie outside of this interval.

It is clear, however, from this discussion that whatever level of confidence is actually used, it shall be stated unambiguously, together with the result and its expanded level of uncertainty. It should also be noted that the coverage factor k which is used as a multiplicand with u_c to obtain an appropriate level of confidence will, of course, depend on the number of independent (uncorrelated) determinations of that measurand (i.e. the number of degrees of freedom), which make up the probability distribution (as described in the GUM). Therefore $k = 2$ shall be used only when there are a sufficiently large number of such degrees of freedom.

2.2.4 Relationship between confidence interval and measurement uncertainty

The relationship between uncertainty of measurement and the confidence interval I of a statistical distribution of measurement results is illustrated, in an exemplar manner, in Figure 1, where

$T_1 = \Theta - 1{,}96\sigma_0$ is the lower confidence limit for a large number of degrees of freedom - expressed in this example at a level of confidence of 95%

$T_2 = \Theta + 1{,}96\sigma_0$ is the upper confidence limit for a large number of degrees of freedom - expressed in this example at a level of confidence of 95%

σ_0 is the normally defined standard deviation (uncertainty) associated with the statistical population distribution

The length of the confidence interval I defined in the GUM, for a large number of degrees of freedom expressed at a level of confidence of 95 %, is then:

$$I = T_2 - T_1 = 2 \times 1{,}96 \times \sigma$$

Thus the standard deviation (uncertainty) associated with this confidence interval I is:

$$\sigma = \frac{I}{2 \times 1{,}96}$$

This then enables the expanded uncertainty U associated with the confidence interval I to be expressed (for a large number of degrees of freedom, expressed at a level of confidence of 95%) as:

$$U = 1{,}96 \times \sigma = 0{,}5 \times I$$

This interpretation is then fully consistent with the definition and use of the term 'expanded uncertainty' within the GUM, and it is also consistent with the intentions within the relevant Annexes of the Air Quality Daughter Directives.

As noted above, the key terms which are used to describe the statistical procedures associated with measurement uncertainty are defined in Annex E of this Report.

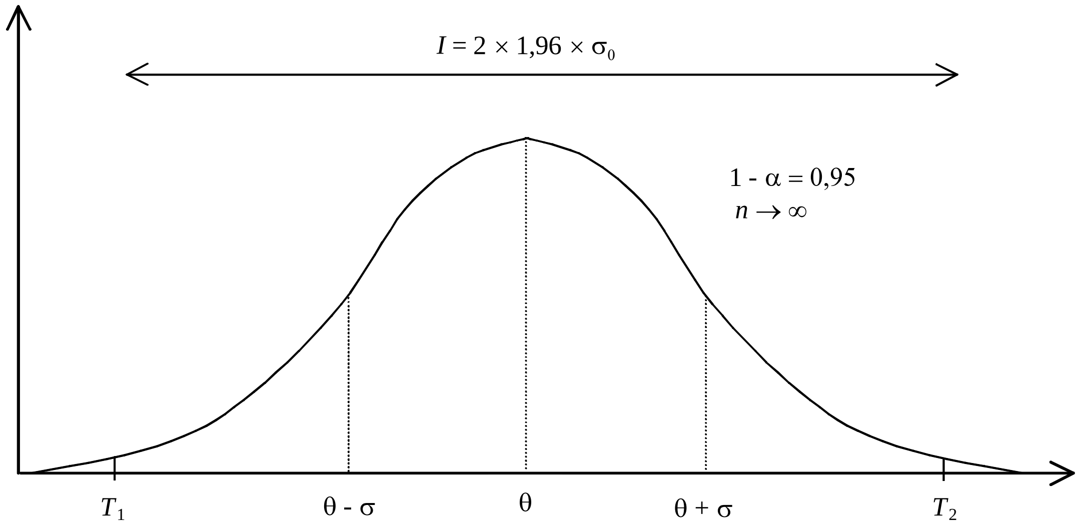

Figure 1 — Graphical illustration of the uncertainty distribution of a measurement quantity derived from repeated observations

2.3 EC Terms of Reference

The EC has produced certain Terms of Reference (ToR) which specify requirements for the contents of European Standards covering ambient air quality, and also to assist in their preparation. The latest draft of these is reproduced in Annex C of this Report. It should be noted that, although these ToR are still draft, and thus are liable to revision, they will retain the same under-lying principles given in the current draft. It should also be noted that because different types of instruments or equipment are available to measure air quality, the EC ToR allow for the type-approval of instruments, and therefore any instruments which operate on the same principle as those stated as meeting all the requirements of the Reference Method prescribed in the relevant European Standard are also acceptable – as long as the latter have been validated by demonstrating that they conform to all the relevant performance criteria applicable to the Reference Method, and also to the overall requirements of the data quality objectives in the appropriate EC Daughter Directive.

The Daughter Directives also contain air-quality concentration limit values set by the European Commission. These are expressed as limits to be met over stated temporal averaging periods, and some of these may be as long as a year. For discontinuous methods this poses potential difficulties for the development and validation of the relevant European Standards, not only with respect to the sampling method, but also in evaluation of the uncertainty of the results of the measurements. In these cases, it should be assumed that the uncertainty of the Reference Method, which is derived for the shorter averaging period used during the laboratory and field validation trials, applies to the longer averaging times specified in the Directives. This, although not completely rigorous, is a pragmatic solution. However, in these circumstances the longest averaging times which it is practical to employ during the laboratory and field trials should be used.

3 Approaches to uncertainty estimation

3.1 General

As noted above, the Air Quality Daughter Directives place specific data quality objectives on the uncertainty of the measurements carried out. This uncertainty of the measurement result is expressed in terms of a maximum allowable percentage of the air quality limit value(s), and it should be calculated with respect to the stated averaging period(s) referred to in the Directives. The ToR also state that these data quality requirements (‘objectives’ in Daughter Directives) are criteria against which the Reference Method is assessed using a combination of laboratory and field tests. The primary concern is therefore to ensure that the assessment of uncertainty of measurement that is used within the CEN/TC 264 standards to derive uncertainties from these assessment tests, allows valid comparisons to be made with the Directives’ objectives, both in terms of the concentration levels actually present, the prescribed limit values, and the associated averaging periods. This has implications for the subsequent use of the Reference Methods and the on-going quality assurance and quality control (QA/QC) regime which is adopted, as knowledge of these will be necessary to derive, for example, an uncertainty on a yearly average when the assessment tests are conducted over a matter of weeks or months.

The Daughter Directives allow the use of the GUM or the International Standards ISO 5725 (or equivalents) to assess the measurement uncertainty of the Reference Methods. It could be argued that these two approaches are not compatible. However, this is not necessarily correct, and the two procedures may be used in such a way that they are complimentary. This approach is explained further in clause 4 of this Report. However, it is useful first to review briefly the published documents covering uncertainty of measurements that are most relevant to this Report.

3.2 Guide to the Expression of Uncertainty in Measurement

The GUM provides the general concept for the harmonised estimation of measurement uncertainties, agreed on and adopted by important organisations in the field of general physical metrology and analytical chemistry (e.g. Bureau International des Poids et Measures (BIPM), International Union of Pure and Applied Chemistry (IUPAC), International Union of Pure and Applied Physics (IUPAP), International Electrotechnical Commission (IEC), and International Organisation of Legal Metrology (OIML)), and published by ISO. In overview, the GUM provides a procedure for assessing the combined uncertainty of a measurement method by assessing and combining all potential sources of uncertainty. In this procedure it is necessary to:

— Establish a model equation (or measurement equation) and a related uncertainty budget which should list fully all potential sources of uncertainty. It is also important at this stage to consider carefully the identified uncertainty contributions of the model equation, so as to ensure that there is no double counting and that any correlations between uncertainty sources are understood.

— Quantify the individual sources of uncertainty, as standard uncertainties (standard deviations), either by experiment and statistical analysis of repeated measurement (Type A uncertainties) or by other evaluation (generally an assumption about the likely distribution describing the uncertainty of the source).

NOTE The above methodology can, in principle, be implemented in all circumstances. However, in some cases, particularly those involving a complex measurement equation, it may not be practical to establish and evaluate all the uncertainty components of the measurement equation, and in these cases some components of the uncertainty budget might potentially be ignored. In these circumstances, the uncertainty evaluations described in clause 3.3 of this Report, which involve International Standards ISO 5725, may be used, where appropriate, to check and evaluate whether there are any neglected components of the uncertainty that are of significant magnitude. Any additional components identified by these means should be incorporated into the uncertainty of the measurement results, in an appropriate manner according to GUM. Where this is not practical, the field evaluations prescribed by the International Standard prEN ISO 14956 should, where possible, be used to establish appropriate (additional) components in the uncertainty budget (see clause 4.1 of this Report).

— Combine the individual uncertainties, taking into account the sensitivity coefficients of each component determined from the partial differential of the model equation, or by experiment. If correlations are present they should also be taken into account at this stage (according to methods described within the GUM).

— Expand the combined uncertainty using a *coverage factor* (usually by a factor derived from the student-*t* distribution using the appropriate number of degrees of freedom) to express the expanded uncertainty at a stated level of confidence, which in the case of the Daughter Directives is 95 %. Note that in this case, as the model equation will, by definition, match the averaging time and limit values required in the Daughter Directive, the concept of bias and standard deviation used in certain versions of the text in the Daughter Directives is not required.

3.3 International Standards ISO 5725 Parts 1 to 6

International Standards ISO 5725 Parts 1 to 6 provide tools and methods for planning, conducting, and analysing, the results obtained from inter-laboratory studies, which aim to characterise the accuracy of measurement methods and measurement results. Part 5 of ISO 5725 is the main standard relevant to this Report, but Part 1 (definitions) and Parts 3 and 4, are also of some relevance. These standards distinguish between:

— repeatability: variations of results within a laboratory using identical methods, equipment, operators etc;

— reproducibility: variations between different laboratories with differing equipment and/or different operators etc.

NOTE: Reproducibility does not define specifically which variables shall be changed and, therefore, this term is not completely specific. In any specific context therefore the term reproducibility should specify which variables are changed.

These International Standards do not use the term 'uncertainty', using instead 'accuracy (trueness and precision)'. However, the results obtained from the application of these standards may be used to provide information, which can then be applied in an over-arching GUM evaluation. ISO 5725 is particularly suited to the assessment of information on the uncertainty of analytical methods, where complex matrices or large numbers of specific laboratory-related uncertainty sources mean that it is impractical (or possibly too expensive) to assess, individually, the contributions of all these sources of uncertainty to the overall uncertainty estimate. Instead, these may be evaluated by means of a series of well-designed inter-laboratory evaluations as given in ISO 5725-5. The relevant ISO 5725 investigation should be carried out as a part of the preparation of any European Standard where it is deemed appropriate to adopt this approach. The information obtained through its application may then be combined with the information on other potential uncertainty contributions (Type A and Type B) using the GUM approach. Care is needed, however, in using the results obtained from an ISO 5725-5 study in a GUM evaluation, so as to ensure that all measures of precision and trueness are expressed correctly in terms of variances and covariances, and also to avoid incorrect duplicate counting of sources of uncertainty. It should also be noted that under ISO 5725 a large number of laboratories and test samples may be required to give an acceptable level of confidence for the derived uncertainties. It is important therefore that any study is large enough to handle correctly the expected number of influence variables.

This methodology should also enable subsequent ('equivalent') measurement methods, which meet the requirements of the standard, to be adopted. However, these equivalent methods may also require an assessment of their uncertainties using ISO 5725-5 (or one using the GUM approach, if this is more appropriate) to be carried out by the user - in order to demonstrate compliance.

3.4 International Vocabulary of Basic and General Terms in Metrology

The International Vocabulary of Basic and General Terms of Metrology (known as the VIM) was first published by ISO in 1984 under the aegis of BIPM, IEC, ISO and OIML, and was re-issued by ISO in 1993. It is a useful addition to the GUM, in that it contains the definitions of the key terms used in this Report (some of which are listed in Annex E).

3.5 Other International Standards

A number of other International Standards provide tools that may be used within a GUM approach to assessing the uncertainty of a measurement result. These include:

— **ISO 9169 Performance characteristics of air quality measuring methods**, with its associated standard ISO 6879 *Performance characteristics and related concepts for air quality measuring methods*, which are both being revised. This defines laboratory (and field) tests that may be used to determine certain performance characteristics of air quality measurement methods.

— **prEN ISO 14956 Evaluation of the suitability of a measurement method by comparison with a stated measurement uncertainty**, which details the procedure used to calculate the combined uncertainty of the measurement method from its performance characteristics, specifically related to the air quality measuring methods given in the (draft) standards ISO 9169 and ISO 6879 (including, for example, the treatment of the component of measurement uncertainty of the results which arises from interference by other species that may be present during field monitoring). It also includes a requirement for field tests which are used to establish the validity of the measurement uncertainty derived from the laboratory tests. This is being progressed as a dual CEN/ISO standard. The DIS is under revision, to allow for the fact that the variance of the result of a measurement is, in general, a weighted sum of the variances and the covariance of all the influence variables. This is necessary in order to make prEN ISO 14956 fully consistent with the GUM.

— **ISO/DIS 11222: Determination of the uncertainty of the time average of air quality measurements**. In general, this Draft International Standard is applicable to quantifying the uncertainty of, for example, daily, monthly, or yearly average (concentration) values of air quality monitoring data where this has sampling times that are shorter than the required averaging time period. The standard may be used to determine the uncertainties of long-term average values, from data obtained from validation and calibration procedures performed over short times. The input data required to enable this standard to be applied is the uncertainty information attached to all of the QA/QC procedures carried out – i.e. the uncertainties in validation, calibration and drift control procedures as well as inter-laboratory comparisons. The uncertainties in the average values obtained by using ISO/DIS 11222 are quantified either as a (combined) standard uncertainty or as an expanded uncertainty at a stated level of confidence.

4 Recommendations for the assessment of uncertainty of ambient air measurement methods

4.1 Introduction

4.1.1 General

The assessment of measurement uncertainty shall be based on the approach described in the GUM. The specific approach to the employed will in detail, however, depend on the type of the measurement method to be employed to cover the selected ambient air pollutant(s). A summary of the principles of the Reference Methods to be used is given in Annex A. These methods can be generally divided into automated and non-automated methods, and the uncertainty estimation procedures will differ to some degree between these, as outlined below.

4.1.2 Automated measurement methods

On-line, automated measurement procedures may be employed where it is practical for the complete measurement system to be located in the field – i. e. where the results are obtained using a 'black box' with its own sample inlet, which is calibrated and checked at regular intervals. For these systems, possible variations are restricted to the make of the instrument used, and to the artefacts used for calibration (although for example, in the case of automated benzene measurements which use gas chromatography, the sampling procedure and the column of the gas chromatograph may also be varied). In these cases, the uncertainty of the results obtained using automated methods should be considered to be covered mainly by:

— Performing a set of (mainly) laboratory-based type-approval tests on all the various types (manufacturers and models) of instrumentation which utilise the principles of the reference measurement technique(s) prescribed in the appropriate European Standard(s) - in order to assess the intrinsic uncertainty contribution associated with the results of the measurements obtained with this type of instrumentation. These tests should encompass all performance criteria that contribute significantly to the uncertainty of the results, as specified in the relevant European Standard.

— Ensuring that the type-approval tests are designed so that they are as comprehensive and rigorous as practical. It should be noted, however, that since these tests are surrogates for actual field conditions, it may not always be possible to achieve this fully. For example, it may sometimes be difficult to mimic, in full, the effects on the measurement results of transient fluctuations of the influence variables. Therefore, to overcome this potential limitation the test procedures should be specified so as to be as comprehensive as possible within the relevant European Standard, and then in addition, well designed field trials should be carried out as noted below (and prescribed by prEN ISO 14956) to establish whether this uncertainty is robust under field conditions;

— Ensuring that all the tests are carried out with sufficient rigour (accuracy, precision, linearity etc) so that uncertainties arising from the test procedures themselves do not contribute significantly to the uncertainty of all the results obtained during the type-approval procedure. (For example, if a continuous monitor is required by the relevant European Standard to have a linearity of ±2 % relative value, then the tests which are carried out to establish this should be such that if they themselves have any non-linearities, these should be demonstrably smaller than this - by a significant factor).

— Carrying out a set of well-designed field trials, with each type of instrumentation, so as to confirm that the measurement uncertainty derived from the type-approval testing is valid when the automated method is used in the field (as prescribed in prEN ISO 14956).

In the above circumstances, it is essential that, when these automated measurement methods are deployed subsequently in the field, very rigorous and comprehensive QA/QC procedures are specified to ensure, as far as achievable, that the uncertainty of measurement derived during these type-approval test procedures continues to be valid for all results obtained from these measurement systems operating in field conditions. These QA/QC tests in the field should cover, inter alia, those components of the complete Method which are the most difficult to control within the type-approval tests, and also should target on any components of the method which are excluded by automated methods for type-approval (e. g. the sample lines and manifolds of the monitoring stations with automated measurement methods – which are excluded by the EC ToR). These requirements are discussed further in clause 4.1.4 and 5.