

Workplace atmospheres — General requirements for the performance of procedures for the measurement of chemical agents

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National foreword

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Foreword

This document (EN 482:2006) has been prepared by Technical Committee CEN/TC 137 "Assessment of workplace exposure to chemical and biological agents", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2007, and conflicting national standards shall be withdrawn at the latest by February 2007.

This document supersedes EN 482:1994.

The major technical change between this European Standard and the previous edition is the revision of the method of calculating the uncertainty of a measurement procedure to comply with ENV 13005.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

National laws and regulations based on European Directives require the assessment of the potential worker exposure to chemical agents in workplace atmospheres. One way of assessing such exposure is to measure the concentration of a chemical agent in the air in the worker's breathing zone. The procedures used for such measurements should give reliable and valid results so that when compared with set occupational exposure limit values a correct decision can be made, for instance, as to whether the exposure level is acceptable or control measures need to be applied.

Because of their importance in the process of exposure assessment, it is required that the measurement procedures should fulfil some general requirements, which are given in this document. Specific European Standards have been prepared for different types of measuring procedures and measuring devices. These include European Standards for dust samplers (EN 13205), diffusive samplers (EN 838), sorption tubes for active sampling (EN 1076), detector tubes (EN 1231), sampling pumps (EN 1232, EN 12919), metals and metalloids (EN 13890), mixtures of airborne particles and vapours (ENV 13936) and direct reading instruments (EN 45544 all parts). In these specific European Standards additional requirements have been included for the procedure or device in question, so that the general requirements of this document are not compromised. Where no specific European Standard exists, only the general requirements apply.

Performance requirements are given in this document for unambiguity, selectivity, expanded uncertainty for minimum specified measuring ranges, averaging time, etc. These requirements are intended to apply under environmental conditions present at the workplace. However, because a wide range of environmental conditions is encountered in practice, this document specifies requirements that have to be fulfilled by measuring procedures when tested under prescribed laboratory conditions.

It is the user's responsibility to choose appropriate procedures or devices that meet the requirements of this document. One way of doing this is to obtain information or confirmation from the provider of a procedure or the manufacturer of a device. Type-testing, or more generally, assessment of the performance of procedures or devices, can be undertaken by the manufacturer, user, test house or research and development laboratory, as is most appropriate. A number of existing procedures for workplace measurements have been tested over part of the required minimum specified measuring range but not over the entire range (see Table 1) or have not been tested for all environmental influences and potential interference's. If these partially validated procedures meet the performance requirements of this European Standard they can nevertheless be used at present. These procedures should be tested over the full ranges as soon as is reasonably practicable. If there is no measuring procedure for a chemical agent, which meet the requirements of this document, a procedure should be used, whose performance is nearest the specified requirements.

1 Scope

This document specifies general performance requirements for procedures for determination of the concentration of chemical agents in workplace atmospheres as required by the Chemical Agents Directive 98/24/EC (see [1]). These requirements apply to all measuring procedures, irrespective of the physical form of the chemical agent (gas, vapour, suspended matter) and of the sampling method and analytical method used.

This document is applicable to all steps of a measuring procedure.

This document is applicable to measuring procedures with separate sampling and analysis steps, and also to direct-reading devices.

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 applies.

EN 838:1995, *Workplace atmospheres – Diffusive samplers for the determination of gases and vapours – Requirements and test methods*

EN 1076:1997, *Workplace atmospheres – Pumped sorbent tubes for the determination of gases and vapours – Requirements and test methods*

EN 1231, *Workplace atmospheres – Short term detector tube measurement systems – Requirements and test methods*

EN 1232, *Workplace atmospheres – Pumps for personal sampling of chemical agents – Requirements and test methods*

EN 1540, *Workplace atmospheres – Terminology*

EN 12919, *Workplace atmospheres – Pumps for the sampling of chemical agents with a volume flow rate of over 5 l/min – Requirements and test methods*

EN 13205:2001, *Workplace atmospheres – Assessment of performance of instruments for measurement of airborne particle concentrations*

EN 13890, *Workplace atmospheres – Procedures for measuring metals and metalloids in airborne particles – Requirements and test methods*

EN 45544 (all parts), *Workplace atmospheres – Electrical apparatus used for the direct detection and direct concentration measurement of toxic gases and vapours*

ISO 78-2, *Chemistry – Layouts for standards – Part 2: Methods of chemical analysis*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 1540 and the following apply.

3.1

analytical uncertainty

u_a

combined uncertainty of the analytical procedure including, where relevant, contributions from analytical recovery, analytical variability, interferences, calibration, instrument response drift and blank correction

3.2

averaging time

period of time for which the measuring procedure yields a single value

[EN 1540:1998]

NOTE For direct reading instruments the averaging time is related to the internal electrical time constant. For other procedures it is normally equal to the sampling time.

3.3

bias

consistent deviation of the measured value from the value of the air quality characteristic itself or the accepted reference value

[ISO 6879:1995]

NOTE 1 In this European Standard the "air quality characteristic" means the concentration of a chemical agent in air.

NOTE 2 In this European Standard the "accepted reference value" can be, for example, the certified value of a reference material, the concentration of a standard test atmosphere or the target value of an interlaboratory comparison.

3.4

chemical agent

any chemical element or compound on its own or admixed as it occurs in the natural state or as produced, used, or released, including release as waste, by any work activity, whether or not produced intentionally and whether or not placed on the market

[Council Directive 98/24/EC Art. 2(a) [1]]

3.5

coverage factor

k

numerical factor used as a multiplier of the combined standard uncertainty in order to obtain an expanded uncertainty

NOTE A coverage factor, k , is typically in the range from 2 to 3.

[ENV 13005:1999]

3.6

combined standard uncertainty

u_c

standard uncertainty of the result of a measurement when that result is obtained from the values of a number of other quantities, equal to the positive square root of a sum of terms, the terms being the variances or covariances of these other quantities weighted according to how the measurement result varies with changes in these quantities

[ENV 13005:1999]

3.7

expanded uncertainty

U

quantity defining an interval about the result of a measurement, that may be expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurand

[ENV 13005:1999]

3.8

(occupational exposure) limit value

limit of the time-weighted average of a chemical agent in the air within the breathing zone of a worker in relation to a specified reference period

[Council Directive 98/24/EC Art. 2(d) [1]]

NOTE Limit values are mostly set for reference periods of 8 h, but can also be set for shorter periods or concentration excursions. Limit values for gases and vapours are stated in terms independent of temperature and air pressure variables in ml/m³ and in terms dependent on those variables in mg/m³ for a temperature of 20 °C and a pressure of 101,3 kPa. Limit values for suspended matter and mixtures of particles and vapours are given in mg/m³ or multiples of that for actual environmental conditions (temperature, pressure) at the workplace. Limit values of fibres are given in number of fibres/m³ or number of fibres/cm³ for actual environmental conditions (temperature, pressure) at the workplace.

3.9

measurand

particular quantity subject to measurement

[ENV 13005:1999]

3.10

non-random uncertainty

uncertainty associated with systematic errors

3.11

random uncertainty

uncertainty associated with random errors

3.12

repeatability conditions

conditions where independent test results are obtained by the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time

[ISO 3534-1:1993]

3.13

reproducibility conditions

conditions where test results are obtained by the same method on identical test items in different laboratories with different operators using different equipment

[ISO 3534-1:1993]

3.14

sampling uncertainty

u_s

combined uncertainty of the sampling procedure including, where relevant, contributions from sampled air volume, sampling efficiency, sample storage and, if applicable, transport

3.15**standard uncertainty**

uncertainty of the result of a measurement expressed as a standard deviation

[ENV 13005:1999]

3.16**uncertainty (of measurement)**

parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand

[ENV 13005:1999]

4 Classification**4.1 General**

In this document measuring procedures are classified according to measurement task. This classification is based upon the measurement strategy laid down in EN 689.

NOTE EN 689 describes a number of measurement categories that involve a combination of these measurement tasks (see Annex A).

4.2 Screening measurements of time weighted average concentration

Screening measurements of time weighted average concentration are performed to obtain relatively crude quantitative information on exposure levels in order to decide if an exposure problem exists, and if so, to appraise its possible seriousness. These measurements can also determine if the exposure is well below or well above the (occupational exposure) limit value.

4.3 Screening measurements of variation of concentration in time and/or space

Screening measurements of variation of concentration in time and/or space are used to:

- provide information on the likely pattern of the concentration of chemical agents in air;
- identify locations and periods of elevated exposure;
- provide information on the location and intensity of emission sources;
- estimate the effectiveness of ventilation or other technical measures.

4.4 Measurements for comparison with (occupational exposure) limit values and periodic measurements

Measurements for comparison with (occupational exposure) limit values are used to obtain results of known precision and accuracy for the average concentration of a chemical agent in the air in a worker's breathing zone.

Periodic measurements are used to determine whether exposure conditions have changed since the measurements for comparison with limit values were performed, or whether control measures remain effective (see EN 689).

NOTE Since the composition of the workplace atmosphere will have been investigated during the initial occupational exposure assessment, it might be appropriate for periodic measurements to use procedures with lower selectivity.

5 Performance requirements

NOTE Performance requirements for measuring procedures depend on the measurement task. The performance requirements for screening measurements are less stringent than for measurements for the comparison with limit values and periodic measurements. Therefore, the performance requirements for screening measurements in 5.1 and 5.2 are only given in general terms.

5.1 Screening measurements of time weighted average concentration

The measurement procedures shall have:

- adequate selectivity for the chemical agent (see 4.2);
- averaging time less than or equal to the limit value reference period;
- measuring range that includes the limit value;
- expanded uncertainty that is fit for purpose (see 4.2).

5.2 Screening measurements of variation of concentration in time and/or space

The measuring procedures shall have:

- adequate selectivity for the chemical agent (see 4.3);
- short averaging time (for variation of concentration in time ≤ 5 min; for variation of concentration in space ≤ 15 min);
- measuring range that is fit for purpose (see 4.3);
- expanded uncertainty that is fit for purpose (see 4.3).

5.3 Measurements for comparison with limit values and periodic measurements

5.3.1 Unambiguity

A measuring procedure shall produce an unambiguous result for the concentration of the chemical agent being measured in the specified measuring range, i.e. an analytically determined value shall correspond to one concentration only.

5.3.2 Selectivity

The measuring procedure shall contain appropriate information about the nature and magnitude of any interferences.

NOTE Selectivity requirements vary from case to case, depending on what is known in advance about the workplace air. If the identity of all contaminants present is not known in advance then the measuring procedure will need to have a high selectivity. If the identity of all contaminants is known prior to measurement, and there are no interferences present, then it might be possible to use a measurement method with a low selectivity.

Procedures for measuring chemical agents present as airborne particles shall prescribe a method for sampling the particle size fraction, as defined in EN 481, for which the limit value for the chemical agent is set.

If different limit values are set for different species of a chemical agent, then the measuring procedure shall determine the individual species concerned.

5.3.3 Averaging time

The averaging time is equal to the sampling time, which shall be less than or equal to limit value reference period.

5.3.4 Measuring range

The measuring range of the procedure or instrument shall cover at least the concentrations from 0,1 to 2 times the limit value for long-term measurements and from 0,5 to 2 times the limit value for short-term measurements.

5.3.5 Expanded uncertainty

The requirements for expanded uncertainty are given in Table 1.

Table 1 — Expanded uncertainty requirements for measurements for comparison with limit values and periodic measurements

reference period	measuring range	relative expanded uncertainty
short-term (e.g 15 min)	0,5 to 2 times limit value	$\leq 50 \%$
long-term	0,1 to < 0,5 times limit value	$\leq 50 \%$
long-term	0,5 to 2 times limit value	$\leq 30 \%$

NOTE Variation of exposure to chemical agents in the workplace can be significantly greater than indicated by the uncertainty of a single measurement calculated according to this European Standard. This is due to the temporal and spatial variability of workplace exposure. See EN 689 for measurement strategy.

5.4 Composite procedures

The performance requirements detailed in 5.1, 5.2 and 5.3 shall be fulfilled for the whole measuring procedure even if the measuring procedure consists of several distinct steps. In this case each step of the measuring procedure may be tested individually as an alternative to testing the measuring procedure as a whole.

5.5 Transport and storage

Transport and storage of a sample, if appropriate, shall be carried out in such a way that the physical and chemical integrity is maintained between sampling and analysis.

5.6 Environmental conditions

The effect of environmental conditions (e.g. temperature, humidity, pressure) on the performance of the method shall be tested in the laboratory. Performance requirements concerning unambiguity, selectivity, expanded uncertainty, minimum measuring range and averaging time shall be fulfilled under the conditions likely to be encountered in the workplace.

NOTE It is not practicable to carry out a comprehensive evaluation of environmental influences in the field and therefore laboratory tests are specified in this document. However, field tests can provide valuable information on the performance of measurement methods.

The range of climatic conditions under which the performance requirements of 5.1 to 5.4 are fulfilled, shall be specified in the measuring procedure.

5.7 Description of measuring procedure

Measuring procedures shall be written in accordance with ISO 78-2. An example for the structure of a method description is given in Annex B.

The description shall contain all necessary information to carry out the measuring procedure, information about the attainable expanded uncertainty, measuring range, averaging time, interferences and environmental or other conditions that can influence the performance of the measuring procedure.

If correction factors are applied, e.g. for known and explainable bias due to environmental influences, these shall be justified in the measuring procedure and recorded in the validation report.

5.8 Dimension of result

The final result of a measuring procedure shall be expressed in the same units as those of the limit value. This can be achieved directly or by means of a suitable conversion.

This requirement is not obligatory for screening measurements of the variation of concentration.

5.9 Additional requirements

In addition to the requirements in 5.1 to 5.8, further requirements specified in EN 838, EN 1076, EN 1231, EN 1232, EN 12919, EN 13205, EN 13890 and EN 45544 (all parts) shall be met for particular types of measuring procedures and devices, as appropriate.

NOTE ENV 13936 should also be considered, if appropriate

6 Test method

6.1 Estimate the expanded uncertainty of results obtained using the measuring procedure by carrying out the tests prescribed in the relevant European Standard(s) specific to the type of procedure or device concerned, as listed in Clause 2. Carry out measurements at the lower and upper ends of the measuring range specified in Table 1 and at least one intermediate concentration. Prepare at least six replicate samples for each set of tests and analyse the samples under repeatability conditions.

Calculate the expanded uncertainty, expressed as a percentage, according to the following procedure (see e.g. [2], [4], [5] and ENV 13005).

- Specify the measurand;
- identify all possible sources of uncertainty;
- quantify the random sampling uncertainty (u_{s_r}) and non-random sampling uncertainty ($u_{s_{nr}}$);
- quantify the random analytical uncertainty (u_{a_r}) and non-random analytical uncertainty ($u_{a_{nr}}$);
- calculate the combined standard uncertainty (u_c) according to equation (3);
- calculate the expanded uncertainty (U), using a coverage factor $k = 2$, according to equation (4).

$$u_{c_r} = \sqrt{u_{s_r}^2 + u_{a_r}^2} \quad (1)$$

$$u_{c_{nr}} = \sqrt{u_{s_{nr}}^2 + u_{a_{nr}}^2} \quad (2)$$

$$u_c = \sqrt{u_{c_{nr}}^2 + u_{c_r}^2} \quad (3)$$

$$U = 2 \times u_c \quad (4)$$

NOTE 1 Annex C gives information about the different components of sampling uncertainty and analytical uncertainty.

NOTE 2 It is intended to give detailed calculation methods in EN 838, EN 1076, EN 1231, EN 1232, EN 12919, EN 13205, EN 13890, ENV 13936 and EN 45544 (all parts) when these are revised.

6.2 Carry out further tests, as appropriate, to investigate the influence of interferences and environmental parameters, e.g. wind speed, wind direction or direction of sampling device.

6.3 For a measuring procedure consisting of several independent steps (preparation of equipment, sampling, transportation and storage of sample, analysis), each step of the measuring procedure may be tested individually as an alternative to testing the procedure as a whole. In this case, calculate the relative expanded uncertainty of the measuring procedure by an appropriate combination of the uncertainties of all the independent steps.

NOTE For some chemical agents the performance of one or more of the steps might have to be determined by means other than direct testing with the chemical agent. For details, see the appropriate specific European Standard.

7 Validation report

A validation report shall be prepared for each measuring procedure tested containing at least the test conditions, the results obtained and the extent to which the measuring procedure complies with the requirements of this document and other relevant European Standards or International Standards.

Annex A (informative)

Measurement categories described in EN 689 and involving a combination of measurement tasks

A.1 Worst case measurements

Screening measurements of variation of concentration in time and/or space (see 4.3) can clearly identify episodes where higher exposures occur, e.g. high emissions due to certain working activities. Sampling periods can be selected containing these episodes. This approach is called worst-case sampling. Subsequently, monitoring can be required either as a screening measurement of time weighted average concentration (see 4.2) or for comparison with limit values (see 4.4).

A.2 Fixed point measurements

Fixed point measurements can be used for comparison with limit values (see 4.4) if the results are representative of the exposure concentration. In other uses, the initial measurement task is the same as for emission sources (see 4.3) and any subsequent monitoring will be screening measurement of time weighted average concentration (see 4.2) or periodic measurements (see 4.4).

A.3 Short-term peak measurements

Screening measurements of variation of concentration in time (see 4.3) can find short-term peaks. These measurements can be followed by screening measurements of time weighted average concentration (see 4.2). Where the measurements are of exposure concentration, these can be compared with limit values (see 4.4). Short-term limit values are important in these cases.

Annex B (informative)

Structure of a method description

The following major clauses should be present, if applicable:

- Introduction;
- Title;
- Warning and safety precautions;
- Scope;
- Normative references;
- Definitions;
- Principle;
- Reactions;
- Reagents and materials;
- Apparatus and equipment;
- Sampling;
- Transport and storage;
- Analytical procedure;
- Calculation;
- Performance characteristics;
- Quality assurance and control;
- Special cases (e. g. interferences);
- Test report;
- Annexes.

Annex C (informative)

Calculation of uncertainty of measurement

C.1 General

The first step in estimating the uncertainty of a method according to ENV 13005, is to construct a cause and effect diagram to identify individual random and non-random uncertainty components (see [2], [4], [5] and ENV 13005). After simplifying to resolve any duplication, the resulting diagram can be used to identify components for which uncertainty estimates are required.

Workplace air measurement methods are normally composed of two major steps: sampling and analysis. Each of these has random and non-random uncertainty components:

a) sampling:

- 1) uncertainty associated with sampled air volume (see C.2);
- 2) uncertainty associated with the sampling efficiency (see C.3);
- 3) uncertainty associated with sample storage and transportation, if any (see C.4).

b) analysis:

- 1) uncertainty associated with method recovery (see C.5);
- 2) uncertainty associated with analytical variability (see C.6);
- 3) uncertainty associated with the calibration (see C.6.2.2 and C.6.2.3);
- 4) uncertainty associated with instrument response drift (see C.6.2.5 and C.6.3.4).

The uncertainty of each of these components is estimated or calculated and then combined to obtain an estimate of the uncertainty of the measurement method as a whole as described in Clause 6.

Assuming a rectangular probability distribution, a range $\pm A$, should be converted into a non-random uncertainty equal to $A/\sqrt{3}$.

C.2 Uncertainty associated with sampled air volume

C.2.1 Pumped sampling

C.2.1.1 Sources of uncertainty

For pumped sampling, the sampled air volume has the following sources of uncertainty: Flow rate measurement (see C.2.1.2), pump flow stability (see C.2.1.3) and sampling time (see C.2.1.4).

C.2.1.2 Flow rate measurement

Flow rate measurements can be carried out using a range of different devices, e.g. rotameters, mass flow meters, bubble flow meters or dry piston flowmeters. Flow rate measurement error arises from two sources: the calibration of the flow meter (non-random), the reading of the flow meter (random) and, where appropriate, correction of the flow rate reading to ambient pressure and temperature. The uncertainty of the flow rate calibration (u_{fc}) should be calculated from the data given on the flow meter test certificate. The uncertainty of the flow rate reading (u_{fr}) should be calculated from measurements carried out under repeatability conditions.

Examples of uncertainty of flow rate measurement for different types of flow meters are given in Table C.1.

Table C.1 — Uncertainty of the flow rate measurement for different types of flow meters (example data)

Flow meter type	Scale	Uncertainty of flow rate calibration, u_{fc}^a %	Uncertainty of flow rate reading, u_{fr}^b %
rotameter 30 cm length	100 %	1,6	0,23
	50 %	2,0	0,45
	10 %	5,2	2,3
mass flow meter	Measuring range		
	0,1 to 15 l min ⁻¹ , flow 2 l min ⁻¹	0,61	2,0
bubble flow meter	Measuring cell		
	0 to 0,25 l min ⁻¹ , flow 0,12 l min ⁻¹	0,4	0,35
	0,2 to 6 l min ⁻¹ , flow 2,0 l min ⁻¹	0,12	0,1
	2 to 30 l min ⁻¹ , flow 3,0 l min ⁻¹	0,06	0,22
dry piston flow meter	0,5 to 5 l min ⁻¹ , flow 2,0 l min ⁻¹	0,59	0,26
	0,5 to 25 l min ⁻¹ , flow 3,0 l min ⁻¹	0,41	0,07
NOTE The uncertainty of the flow rate reading of an analogue flow meter depends upon the resolution of the scale of the instrument.			
a All uncertainty values are calculated with the assumption of a rectangular probability distribution $A/\sqrt{3}$, where A is the individual distribution value, e. g. from calibration certificates.			
b Based on ten measurements.			

If the flow rate is measured at several times, and not only at the beginning of the sampling, the uncertainty of the flow rate reading is reduced by a factor of $1/\sqrt{n}$, where n is the number of measurements of the flow rate.

C.2.1.3 Pump flow stability

Pumps for personal air sampling are usually self-regulating and maintain the set flow rate independent of variation in back pressure. EN 1232 and EN 12919 require that the flow rate is maintained within ± 5 % of the set value throughout the sampling period. Assuming a rectangular probability distribution, the maximum acceptable value of the non-random uncertainty component of the pump flow stability is $5/\sqrt{3}$ %.

C.2.1.4 Sampling time

Sampling time can be measured very exactly with a radio controlled clock, a quartz clock or stopwatch. The major source of uncertainty in measurement of sampling time is the accuracy with which the reading is taken, i.e. to the nearest minute or second. This non-random uncertainty component is very small in the case of long

term measurements (e.g. > 2 h) and can be disregarded, but for short term measurements it needs to be taken into account. For example, if the time is recorded to the nearest minute, the relative standard deviation is 3,8 % for a sampling time of 15 min (assuming a rectangular probability distribution).

C.2.2 Diffusive sampling

C.2.2.1 Sources of uncertainty

For diffusive sampling, the sampled air volume has the following sources of uncertainty: Uptake rate (see C.2.2.2) and sampling time (see C.2.2.3).

C.2.2.2 Uptake rate

The uptake rate is influenced by the pressure, humidity and temperature of the sampled air and by the mass of chemical sampled (which is a function of its concentration in the sampled air and sampling time). The random and non-random uncertainty components of the uptake rate are estimated from replicate samples obtained from a test atmosphere, as described in EN 838.

C.2.2.3 Sampling time

See C.2.1.4.

C.3 Uncertainty associated with sampling efficiency

C.3.1 Pumped sampling methods for gases and vapours

The adsorption of gases and vapours on pumped sorbent tubes is influenced by the pressure, humidity and temperature of the sampled air, the concentration of gases and vapours in the sampled air and by the flow rate. These factors affect the adsorption capacity and the performance of the adsorption process. In some circumstances (low flow rate) a simultaneous process of diffusion can occur.

For sorbent tubes, the sample volume is kept well below the experimentally established breakthrough volume, in which case the sampling efficiency is 100 % and the uncertainty of the sampling efficiency does not need to be taken into account. The determination of the breakthrough volume is specified in EN 1076.

Depending on the sorbent/vapour system, although 100 % of the vapour is retained on the sorbent, the differences in the adsorption process can give differences in the mass actually recovered. The uncertainty associated with these effects is estimated in conjunction with the uncertainty component of the method recovery (see C.5.2).

For bubblers/impingers, impregnated filters and impregnated sorbent tubes, random and non-random uncertainty components of sampling efficiency are estimated or experimentally determined using a procedure based on that given in EN 1076.

C.3.2 Diffusive sampling methods for vapours

The uncertainty components associated with the sampling efficiency of diffusive samplers are dealt with in C.2.2.

Back diffusion can occur if there is significant variation in the air concentration of the chemical agent during the sampling period. It is influenced by the characteristics of the sorbent and the chemical agent, by the pressure, humidity and temperature of the sampled air and by the mass of chemical agent sampled (which is a function of its concentration in the sampled air and sampling time).

The non-random uncertainty component due to back diffusion can be estimated from the difference of the means of the results in two sets of replicate samples obtained by exposure of the diffusive samplers for a short period to high concentration of the chemical agent, one of which is subsequently exposed to clean air for a long period, as described in EN 838.

C.3.3 Aerosol sampling methods

C.3.3.1 General

Aerosol samplers have to follow one or more of the sampling conventions defined in EN 481. Aerosol sampling methods have random and non-random uncertainty components that arise from how closely the samplers used match the required sampling convention(s).

EN 13205 describes two test methods to determine whether a sampler collects the required aerosol fraction(s) correctly. In the primary method (see EN 13205:2001, Annex A), this is done by determining the collection efficiency of the sampler as a function of particle size. In the secondary method (see EN 13205:2001, Annex B), this is done by comparing the concentration measured by the candidate sampler and that measured by a validated (reference) sampler for at least three test aerosols with widely different particle size distributions.

C.3.3.2 Uncertainty for aerosol samplers tested according to EN 13205:2001, Annex A

C.3.3.2.1 Principle

In this method the sampling efficiency is determined as a function of particle size.

C.3.3.2.2 Sources of uncertainty for sampling efficiency

The sampling efficiency has the following sources of uncertainty:

- calibration of the sampler test system (see C.3.3.2.3);
- estimation of the sampled concentration (see C.3.3.2.4);
- deviation from the sampling convention (see C.3.3.2.5);
- excursions from the nominal flow rate [for samplers collecting the respirable and/or thoracic aerosol fraction] (see C.3.3.2.6);
- individual sampler variability [for samplers collecting the respirable and/or thoracic aerosol fraction] (see C.3.3.2.7).

C.3.3.2.3 Calibration of the sampler test system

In a properly designed and performed experiment, the random and non-random uncertainty components associated with calibration of the sampler test system should be very small. They can be calculated by propagation of errors from the uncertainty of the diameter of the calibration particles (and possibly by the use of calibration functions for particle sizers), to the uncertainty in sampled mass fraction.

C.3.3.2.4 Estimation of the sampled concentration

The random uncertainty component of the measured sampling efficiency of the sampler depends on the uncertainty of the concentrations measured by the reference sampler and the tested sampler, as described in EN 13205. It is calculated as propagation of errors from these uncertainties to uncertainty in sampled mass fraction.

C.3.3.2.5 Deviation from the sampling convention

The non-random uncertainty component due to deviation between the sampling efficiency of the sampler and the sampling convention is a function of the sampled aerosol size distribution (and in some cases even a steep function). It is calculated as specified in EN 13205.

C.3.3.2.6 Excursions from the nominal flow rate (for samplers collecting the respirable and/or thoracic aerosol fraction)

The separation efficiency for samplers collecting the respirable and/or thoracic aerosol fraction is highly dependent on the sampling flow rate. The non-random uncertainty component associated with excursions from the nominal flow rate is calculated by propagation of error (flow rate deviation) to variability in sampled mass fraction, as specified in EN 13205. This uncertainty component should be calculated in conjunction with the calculation of the uncertainty component of the sampled air volume (see C.2.1.3).

C.3.3.2.7 Individual sampler variability (for samplers collecting the respirable and/or thoracic aerosol fraction)

The random uncertainty component associated with individual sampler variability is calculated by propagation of error (individual sampler variability) to variability in sampled mass fraction, as specified in EN 13205.

C.3.3.3 Uncertainty for aerosol samplers tested according to EN 13205:2001, Annex B

C.3.3.3.1 Principle

In this method the sampling efficiency is evaluated by comparison with a validated (reference) sampler.

C.3.3.3.2 Sources of uncertainty for sampling efficiency

The sampling efficiency has the following sources of uncertainty:

- sampler variability (see C.3.3.3.3);
- correction factor (see C.3.3.3.4);
- reference sampler (see C.3.3.3.5);

C.3.3.3.3 Sampler variability

The random uncertainty component associated with sampler variability is determined as specified in EN 13205. This uncertainty component includes individual sampler variability, if several samplers of the same type are included in the test.

C.3.3.3.4 Correction factor

The correction factor gives the average systematic difference between the tested sampler and the reference sampler. This has non-random and random uncertainty components and these are calculated as specified in EN 13205.

C.3.3.3.5 Reference sampler

The uncertainty of the reference sampler has random and non-random uncertainty components. These should be given in the test report of the reference sampler.

C.3.3.4 Efficiency of collection substrate

C.3.3.4.1 Filter materials

Filter materials should be selected to have high collection efficiency for the particle size range of interest, in which case the uncertainty associated with collection efficiency is negligible.

C.3.3.4.2 Foams

When foams are used as a collection substrate, sampling efficiency and collection efficiency are interrelated and no uncertainty components need to be added.

C.4 Uncertainty associated with sample storage and transportation

C.4.1 Sample storage

The non-random uncertainty component associated with sample storage can be estimated by analysing samples collected from a test atmosphere or prepared by spiking sampling media with the chemical agent of interest. This can be calculated from the difference between the mean results of replicate samples analysed immediately after sampling/spiking and replicate samples analysed after the maximum storage period specified in the test method. Storage tests for gas and vapour samples are described in EN 838 and EN 1076.

C.4.2 Transportation

C.4.2.1 Gas samples and vapour samples

For gas samples and vapour samples, it is not necessary to take into consideration any component of uncertainty other than those associated with storage when samples are transported in an appropriate manner as specified in the measurement method.

C.4.2.2 Aerosol samples

The transport of aerosol samples normally has a component of uncertainty associated with material losses from the sample substrate or substrate contamination. The upper limit for the loading of the collection substrate can be determined as described in EN 13205 or ISO 15767. The non-random uncertainty component is determined from the acceptance criteria for the upper limit of sample loading.

C.5 Uncertainty associated with analytical recovery

C.5.1 Gases and vapours

C.5.1.1 General

Vapour method recovery is influenced by several factors (see C.3.1). The study of their influence is carried out following the tests described in EN 1076 and EN 838 by the use of standard test atmospheres. When the influence in recovery is significant, bias components are estimated and treated as uncertainty components from the results of these tests.

Results for vapour methods are normally corrected for desorption efficiency. It can be calculated using spiked sampling media following the specific tests described in EN 1076 and EN 838. When the influence of ambient conditions and other factors is not significant and the results are corrected for desorption efficiency, only the uncertainty component associated with this correction is taken into account.

The uncertainty of the method recovery bias can be estimated from:

- results obtained by analysing a suitable number of replicate samples of certified reference materials (see C.5.1.2);
- results obtained from replicate samples taken in the test gas atmospheres (see C.5.1.3);
- results from interlaboratory comparisons (see C.5.1.4); or
- results from recovery tests carried out on spiked sampling media (see C.5.1.5).

C.5.1.2 Certified reference materials (CRMs)

The non-random uncertainty component associated with the analytical method bias can be estimated by the replicate analysis of CRMs.

C.5.1.3 Test gas atmospheres

C.5.1.3.1 Sources of uncertainty

The random and non-random uncertainty components associated with the method recovery can be estimated from the replicate samples taken in test gas atmospheres at different concentrations, ambient conditions, flow rates, etc. covering the ranges of the method application as specified in EN 1076 and EN 838.

The method recovery has the following sources of uncertainty:

- principal sources (see C.5.1.3.2);
- reference concentration (see C.5.1.3.3);
- desorption efficiency (see C.5.1.3.4);

C.5.1.3.2 Principal sources (of uncertainty)

The experimental data collected during the tests specified in EN 838 and EN 1076 give representative information about the effects causing variation and bias (relative to a reference value) that occur in routine application of the specified method of measurement, e.g. concentration, sampling time, temperature and humidity. These data can be used to estimate the method uncertainty components.

The random component of uncertainty associated with the difference between samples is calculated as the relative standard deviation of the replicate samples at all exposure conditions.

The non-random components of uncertainty are calculated as the square root of the corresponding between group variances obtained according to EN 1076:1997, Annex B and EN 838:1995, Annex A.

C.5.1.3.3 Reference concentration

In a properly designed and performed experiment, the random and non-random uncertainty components associated with the test gas atmosphere concentrations are expected to be small. They depend on the system used for generation and can be calculated by a propagation of errors from the uncertainty of the parameters of the atmosphere generation. For example, for a dynamic system the random uncertainty associated with the reference concentration is usually < 3 %.

C.5.1.3.4 Desorption efficiency

Desorption efficiency can be calculated from the results of replicate analyses of certified reference material or of sampling media spiked at several levels covering the range of the application of the method, dividing the mass of analyte recovered by the mass applied, as specified in EN 838 and EN 1076.

When desorption efficiency does not vary significantly with the concentration (general case) and the results are corrected for desorption efficiency, the random uncertainty component associated with this correction is estimated and treated as an uncertainty component. The random uncertainty component associated with incomplete desorption is estimated as the relative standard deviation of the mean of the desorption efficiencies for all levels.

If desorption efficiency correction is not applied to the results, the bias component is estimated and treated as an uncertainty component. The non-random uncertainty component associated with incomplete desorption is estimated as the difference of the mean of the desorption efficiency at all concentrations from unity and converted to a standard uncertainty.

In some cases desorption efficiency varies with concentration. In this case the random and non-random uncertainty components will be estimated from the function relating the desorption efficiency to concentration.

C.5.1.4 Interlaboratory comparisons

The non-random uncertainty component associated with the analytical method bias can be estimated from the results of interlaboratory comparisons, as described in [2].

C.5.1.5 Spiked sampling media

The random and non-random uncertainty components associated with the analytical method (bias) recovery can be estimated by the analysis of replicate test samples prepared by spiking blank sampling media with a known volume of standard solution containing the analyte or analytes of interest, as described in [2].

When spiked sampling media are used, some of the components of the uncertainty mentioned in C.5.1.3.2 cannot be estimated. Therefore the analysis of spiked sampling media should be used only in those circumstances in which the effects of concentrations, environmental conditions, flow rates etc. on sampled mass are either known to be negligible or can be estimated from previous information or other tests.

C.5.2 Measurement of chemical agents in airborne particles

C.5.2.1 General

Bias is normally eliminated during the development of a method, but this is not always possible. According to ENV 13005 measurement results should be corrected for bias if significant. However, this is often not practicable for methods for measuring chemical agents in airborne particles, since bias, when it exists, can vary depending on the sample matrix. The range of biases therefore has to be estimated and treated as an uncertainty component.

The uncertainty of the analytical method bias can be estimated from:

- results obtained by analysing a suitable number of replicate samples of certified reference materials and/or pure compounds, representative of the range of substances that could be present in workplace air (see C.5.2.2);
- results from interlaboratory comparisons (see C.5.2.3); or
- results from recovery tests carried out on spiked sampling media (see C.5.2.4).

Alternatively, some methods that cover a wide range of sample matrices specify an acceptable range for the bias, within which it has to be demonstrated that the method performs, when used in a particular laboratory for a particular application, e.g. ISO 15202. In such instances the uncertainty of the analytical method bias can be estimated from the acceptable range, assuming a rectangular probability distribution.

C.5.2.2 Certified reference materials (CRMs) or pure compounds

The non-random uncertainty component associated with the analytical method bias can be estimated by the replicate analysis of CRMs and/or pure compounds as described in EN 13890.

Analysis of CRMs and/or pure compounds is a good means of estimating the uncertainty of the bias for methods that involve a sample dissolution step. However, it is not generally useful for methods that do not involve sample dissolution, since CRMs are generally bulk materials rather than airborne particles deposited on sampling media. For such methods, it could be possible to produce test samples by depositing CRMs and/or pure compounds on sampling media in a controlled manner.

C.5.2.3 Interlaboratory comparisons

The non-random uncertainty component associated with the analytical method bias can be estimated from the results of interlaboratory comparisons, as described in [2].

Interlaboratory comparison results can be used to estimate the uncertainty of the bias for methods that do not involve sample dissolution, e.g. X-ray fluorescence methods. However, using results from interlaboratory comparisons is not generally a good approach for estimating the uncertainty of the bias for methods that involve a sample dissolution step. This is because test samples used in most interlaboratory comparisons are sampling media spiked with analyte solution and the analysis of such samples does not effectively test the performance of the sample dissolution method. However, where this is not the case (e.g. for interlaboratory comparisons in which the test samples are prepared by collection of airborne particles on sampling media), or where the effectiveness of sample dissolution is not an issue (e.g. measurement of sulphuric acid on a filter), using results from interlaboratory comparisons is a good approach for estimating the uncertainty of the analytical method bias.

C.5.2.4 Spiked sampling media

The non-random uncertainty component associated with the analytical method bias can be estimated by analysing replicate test samples prepared by spiking blank sampling media with a known volume of standard solution containing the analyte or analytes of interest, as described in [2].

Once again, this approach is applicable to methods that do not involve sample dissolution and methods for which the effectiveness of sample dissolution is not an issue, see C.5.2.3.

C.6 Uncertainty associated with analytical variability

C.6.1 General

The uncertainty associated with analytical variability may be estimated either from data obtained under repeatability conditions or from data obtained under reproducibility conditions. When the analytical precision is determined from within-laboratory reproducibility data, i.e. using quality control data, most random and randomized uncertainty components are included (see C.6.3.1). See ISO/TS 21748 for further guidance.

C.6.2 Estimation using repeatability data

C.6.2.1 Analytical precision

The uncertainty associated with the analytical precision is determined by analysing calibration standards of the same composition under repeatability conditions. In vapour methods this contribution is already incorporated in contributions from the determination of the desorption efficiency and it does not need to be taken into account.

C.6.2.2 Concentration of calibration standards

The non-random uncertainty components associated with the concentration of the calibration standards can be estimated from one or more of the following:

- certificate provided by the manufacturer of a stock standard solution;
- certificate provided by the manufacturer of a pressurised test gas;
- purity of the starting material (e.g. pure compound);
- uncertainties in weighing compounds and solutions, i.e. the uncertainty of a balance;
- uncertainties in preparing a test gas; and
- random uncertainty components associated with a dilution procedure.

C.6.2.3 Calibration function

The random uncertainty component associated with the calibration function can be calculated from parameters obtained by the least-squares linear regression.

C.6.2.4 Dilution of the sample solutions (if applicable)

If sample solutions are diluted before analysis it is necessary to take into consideration the random and non-random uncertainty components associated with the dilution process.

C.6.2.5 Instrument response drift

Methods and/or laboratory operating procedures generally specify a maximum instrument response drift that is permitted before recalibration (often monitored by repeat analysis of a calibration solution). It is necessary to take this non-random uncertainty component into consideration.

C.6.3 Estimation using within-laboratory reproducibility data

C.6.3.1 Analytical variability

The random uncertainty component associated with analytical variability can be estimated from within-laboratory reproducibility data obtained from the analysis of stable quality control samples, normally at both low and high concentrations. It is important to cover long-term random variations, and separate estimates need to be made for any sources of systematic error, e.g. non-random uncertainty associated with the concentration of the standard solution, dilution of the sample solutions, if applicable, and instrument response drift (see C.6.3.2 to C.6.3.4).

C.6.3.2 Concentration of the standard solution

The non-random uncertainty component associated with the concentration of the standard solution used to prepare the calibration solutions can be calculated from the concentration range given on the certificate provided by the manufacturer of a stock standard solution or the purity of a starting material (e.g. pure compound).

C.6.3.3 Dilution of the sample solutions (if applicable)

See C.6.2.4.

C.6.3.4 Instrument response drift

See C.6.2.5.

C.6.4 Blank subtraction

If applicable, the random uncertainty component associated with blank subtraction, or non-random uncertainty component when no blank subtraction is performed, need to be included.

C.7 General equation for combination of uncertainty components

To calculate the random and non-random components of sampling uncertainty and analytical uncertainty, the relevant individual uncertainty components are combined according to equations (C.1 to C.4):

$$u_{s_r} = \sqrt{\sum_{i=1}^{j_{s_r}} u_{s_{r_i}}^2} \quad (C.1)$$

$$u_{s_{nr}} = \sqrt{\sum_{i=1}^{j_{s_{nr}}} u_{s_{nr_i}}^2} \quad (C.2)$$

$$u_{a_r} = \sqrt{\sum_{i=1}^{j_{a_r}} u_{a_{r_i}}^2} \quad (C.3)$$

$$u_{a_{nr}} = \sqrt{\sum_{i=1}^{j_{a_{nr}}} u_{a_{nr_i}}^2} \quad (C.4)$$

where

u_{s_r} , $u_{s_{nr}}$, u_{a_r} and $u_{a_{nr}}$ are defined in 6.1;

$u_{s_{r_i}}$, $u_{s_{nr_i}}$, $u_{a_{r_i}}$ and $u_{a_{nr_i}}$ are the corresponding relevant individual uncertainty components;

j_{s_r} , $j_{s_{nr}}$, j_{a_r} and $j_{a_{nr}}$ are the corresponding numbers of relevant individual uncertainty components.

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¹ Recently withdrawn; replacement by new EN intended

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