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Workplace atmospheres — Protocol for evaluating the performance of diffusive samplers

Air des lieux de travail — Protocole pour l'évaluation de la performance des dispositifs de prélèvement par diffusion

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Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 16107 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

This second edition cancels and replaces the first edition (ISO 16107:1999), which has been technically revised.

Introduction

Gas or vapor sampling is often accomplished by actively pumping air through a collection medium such as activated charcoal. Problems associated with a pump, such as inconvenience, inaccuracy and expense, are inextricable from this type of sampling. The alternative covered by this International Standard is to use diffusion for moving the compound of interest onto the collection medium. This approach to sampling is attractive because of the convenience of use and low total monitoring cost.

However, previous studies have found significant problems with the accuracy of some samplers. Therefore, although diffusive samplers may provide a plethora of data, inaccuracies and misuse of diffusive samplers may yet affect research studies. Furthermore, worker protection may be based on faulty assumptions. The aim of this International Standard is to counter the uncertainties in diffusive sampling through achieving a broadly accepted set of performance tests and acceptance criteria for proving the efficacy of any given diffusive sampler intended for use.

This International Standard is intended specifically for the large-scale evaluation of many diffusive sampler/analyte pairs of practical application and is complementary to EN 838. An affordable, experimental evaluation determines a single performance value indicating how a sampler performs in a typical situation. A sampler can thereby be quickly judged as to acceptability. Additionally, sufficient data are obtained to predict performance in many atypical situations. For example, although sampling may normally be done at room temperature, a particular need may call for use in extreme cold. In such a case, the single performance value would be superseded by the particular needs.

Workplace atmospheres — Protocol for evaluating the performance of diffusive samplers

1 Scope

This International Standard specifies methods for evaluation of sampler performance in terms of workplace conditions: wind speed, humidity, temperature, atmospheric pressure, and analyte variation. The concise set of experiments specified aims to minimize cost to the user. The evaluation is limited to conditions commonly encountered in personal sampling in the indoor workplace setting, namely wind speeds of up to 0,5 m/s and for sampling periods typically from 2 h to 8 h.

Static or area sampling, unlike personal sampling where movement of the subject is significant, may sometimes be subject to sampling-rate reduction due to stagnation at very low wind speeds. This International Standard therefore does not apply to wind speeds of less than 0,1 m/s relative to static samplers. Samplers are also tested for compliance with the manufacturer's stated limits on capacity, possibly in the presence of interfering compounds. Given a suitable exposure chamber, the sampler evaluation protocol can be extended to cover sampler use for other sampling periods and conditions.

This International Standard indicates how to measure diffusive sampler uncertainty for characterizing concentration estimates obtained subsequent to the evaluation. It is impractical continually to re-evaluate diffusive sampler performance under various environmental conditions prevailing during application.

NOTE 1 In this International Standard, the confidence level for the initial method evaluation becomes an integral part of the measurement uncertainty. This approach slightly broadens the statistical protocols given in ISO Guide 98:1995. Furthermore, the possibility of sampler errors related to correctible sampler bias is addressed.

NOTE 2 This International Standard is an extension of previous research on diffusive samplers (References [1] to [17] inclusive and EN 838).

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.

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

ISO Guide 98:1995, *Guide to the expression of uncertainty in measurement*. BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 838 and ISO Guide 98:1995 and the following apply.

3.1
symmetric accuracy range

A

fractional range about the measurand concentration, c, within which 95 % of sampler measurements are found

NOTE See References [18] to [21] inclusive.

If the modulus of the bias is small, i.e. $|\varDelta| < R/1{,}645$, the symmetric accuracy range, A, can be shown to be closely approximated (Reference [21]) by Equation (1):

$$A = 1{,}960 \times \sqrt{\varDelta^2 + R^2} \tag{1}$$

where

$\varDelta$ is the bias, expressed relative to true concentrations;

R is the overall true relative standard deviation, expressed relative to true concentrations.

Otherwise

$$A = |\varDelta| + 1{,}645 \times R \tag{2}$$

If the bias is corrected, the expected value of $\varDelta^2$ in Equation (1) becomes equal to the variance of the bias correction, reflecting an uncorrectable residual bias due to uncertainty in the correction. Then if the bias uncertainty is under control, the low bias modulus model of Equation (1) indicates the proportionality of A to the root mean squared combined uncertainty components.

4 Symbols and abbreviated terms

A	symmetric accuracy range (3.1) in terms of bias and precision
A_{est}	estimated symmetric accuracy range, A
$A_{95\ \%}$	95 % confidence level on the symmetric accuracy range, A
c	true or reference analyte concentration, in milligrams per cubic metre, or parts per million as a ratio of analyte to air molecules, per instructions of the sampler manufacturer
c_{est}	mean of (four) concentration estimates (including pressure and temperature corrections), in milligrams per cubic metre or parts per million as a ratio of analyte to air molecules, per instructions of the sampler manufacturer
h	humidity, partial pressure of water vapor, in kilopascals
n	number of diffusive samplers tested for measuring sampler capacity
p	(atmospheric) pressure
R	overall true relative standard deviation of concentration estimates (dependent on assumed environmental variability), expressed as a percentage relative to a “true” concentration as estimated by reference sampling

R_{est}	estimated true relative standard deviation, expressed as a percentage
R_{run}	true relative standard deviation characterizing inter-run chamber variability, expressed as a percentage
R_{s}	intersampler component of the true relative standard deviation, expressed as a percentage
$R_{\text{s est}}$	estimated intersampler true relative standard deviation, R_{s}, expressed as a percentage
R_t	pulse-induced true relative standard deviation, expressed as a percentage
$R_{95\ \%}$	95 % confidence limit on the true relative standard deviation, expressed as a percentage
s	estimated standard deviation characterizing intersampler variation
$t_{0,95}(\nu)$	value which, at the 95 % probability level, exceeds random variables distributed according to the Student t-distribution with ν degrees of freedom
T	temperature, in degrees Celsius
u	ambient wind speed, in metres per second
α_x	concentration estimate dependence on environmental variable, x (T, h, u, or c)
Δ	bias relative to concentration, c
Δ_{est}	estimated bias, Δ
Δ_t	bias associated with concentration pulse
$\Delta_{95\ \%}$	95 % confidence limit on the bias, Δ
ν	degrees of freedom in determining R_{s}
ν_{eff}	effective number of degrees of freedom in determining R
σ_c	assumed concentration variability
σ_h	assumed humidity variability
σ_T	assumed temperature variability
σ_u	assumed ambient wind speed variability

5 Summary of test protocol

5.1 Factors affecting performance

5.1.1 Diffusive sampling may first of all suffer from error in the sampling rate as stated by the manufacturer of the sampler. As diffusive samplers are usually used without recalibration, this error implies a bias or systematic error in all concentration estimates made. As the bias may be in one direction, such error cannot be minimized by averaging several measurements. If the error is correctible it is not strictly part of the sampler uncertainty.

5.1.2 Aside from bias, several sources of random error exist. Some of these errors are associated with the sampler itself. For example, the sampler's dimensional tolerance may be low, leading to variation in the sampling rate, sampler to sampler. The sampler may become overloaded or may not follow concentration changes adequately.

5.1.3 Other errors relate to a non-constant environment which may affect the sampling rate. Potentially significant environmental *influence quantities* include temperature, humidity, wind speed, and atmospheric pressure. To predict the effect of any such parameter, an experiment on the sampler type may be carried out. Moreover, the variation expected of the parameter requires estimation. So as to result in a single performance measure, typical ranges of these environmental parameters are specified in this International Standard.

5.2 "Accuracy" as providing a single performance value linking to measurement uncertainty

5.2.1 A sampler presented by a manufacturer for evaluation may be found to exhibit both imprecision and bias. In the language of ISO Guide 98:1995, imprecision and bias together relate to the "accuracy" of a sampler, rather than "uncertainty". Accuracy refers to the closeness between measured values and measurand (in this case the concentration to be measured), whereas uncertainty refers to what is known about the measurand on the basis of the measurement. In other words, the manufacturer could recommend use of information about the bias for correction of the measured values. This International Standard provides means of judging a sampler as recommended for use by a manufacturer.

5.2.2 A useful statistical measure exists which summarizes the overall effect of both random variation and bias. This function has come to be known as the symmetric accuracy range, A (3.1).

5.2.3 The definition directly implies that A increases with both random error and bias magnitude. Therefore A is one particular quantification of accuracy as defined in ISO Guide 98:1995. The greater the disparity between measured values and measurand, the larger the value of A.

NOTE A is not the only way of quantifying accuracy. For example, the selection of 95 % as coverage level is arbitrary. Furthermore, other functions consistent with the qualitative idea of accuracy as described in ISO Guide 98:1995 are possible.

5.2.4 A indicates quantitatively how far off measurements would generally be if further correction of the sampler were not made. A further advantage of A is that a direct link to the uncertainty described in ISO Guide 98:1995 is obtained in the case that bias is minimized, e.g. through results of the evaluation.

5.2.5 Moreover, a measurement element not covered extensively within ISO Guide 98:1995 is dealt with explicitly. Namely, diffusive samplers are generally used as obtained from the manufacturer without re-evaluation at each use. Therefore, uncertainty in a measured value must account for confidence in the [single] evaluation itself as well as variation and possible residual bias at the point of use.

5.2.6 The result of approaching uncertainty from the point of view of accuracy results in an "expanded uncertainty" (in the language of ISO Guide 98:1995) proportional via a "coverage factor" to the root mean squared *combined uncertainty components* comprised of bias uncertainty, the effects of sampler variation, and influence quantities described above. The coverage factor is determined by the confidence required [e.g., 95 %] in the sampler evaluation and the coverage [also 95 %]. The final result is a value of expanded uncertainty with specific [tolerance or predictive] meaning, namely, in providing the 95 % coverage range about the [true] measurand at 95 % confidence in the evaluation.

5.3 Bias, intersampler variability and the effects of environmental uncertainty

5.3.1 This International Standard gives a procedure for assessing the effects of variability in the following workplace variables: temperature, T; humidity, h (expressed in terms of the water vapor partial pressure to minimize interaction with the temperature); ambient wind speed, u, across the sampler face [see (5.7) regarding wind direction]; and concentration, c. An experiment is carried out which provides information about the dependencies of the concentration estimates on these variables near conditions of intended sampler use (T_0, h_0, u_0, and c_0). Testing is required at the concentration, c_0, of intended use, as well as at concentrations reduced at least to $c_0/2$. Furthermore, the sampler bias and the intersampler standard deviation are measured.

Finally, the effect of diffusion of material out of the sampler is measured. Pressure effects result in correctable bias and are not evaluated in this International Standard (5.8).

5.3.2 Using four samplers for each of five experimental runs (the minimum possible), the dependencies α_T, α_h, α_u, and α_c (relative to the chamber reference concentration and target environmental parameters) on changes in T, h, u, and c are measured, following the sampler manufacturer's instructions regarding pressure and temperature corrections (if any). These experiments also give a value for the estimated sampler bias, Δ, relative to the chamber reference concentration (defined for the target conditions). Two further runs describing time-dependent effects (5.4.5) from diffusive loss of analyte are also carried out. The chamber reference concentration must be traceable to primary standards of mass and volume.

5.3.3 Error in the estimates of the dependencies α_T, α_h, α_u, and α_c will exist on account of the intersampler component of the true relative standard deviation R_s and an inter-run chamber true relative standard deviation, R_run. The latter results in part from uncertainty in the reference concentration. R_s is obtained by pooling the variance estimates from each run and therefore is estimated with $7 \times 3 = 21$ degrees of freedom (or 15 degrees of freedom if the reverse diffusion experiment is omitted). So as to avoid re-measurement at each sampler/analyte evaluation, R_run is obtained by a separate characterization of the chamber with several runs at (for example) fixed environmental conditions. An example in which the dependencies α and R_s are estimated is presented in Annex A.

NOTE It is up to the user as to how traceability is established. In Reference [12], the concentration estimate, as calculated from the chamber's analyte generation parameters, is regarded as the "benchmark", although an independent estimate is required and must be within 5 % of the calculated estimate. If these estimates differ, then a third independent estimate is required to establish the reference concentration through agreement with one of the other independent estimates. One possibility for such an independent estimate is the mean of at least five independent, active sampler estimates per run within the chamber. Experiment (Reference [12]) on the accuracy of such reference measurements using sorbent tubes indicates that a true relative standard deviation of the order of 2 % can be achieved for the individual measurements. Alternatively, Reference [3] requires averaging of at least two independent methods (possibly including calculated estimates) with at least four samples per method. EN 838 has adopted the looser requirement that calculated and independent measurements shall agree within 10 %.

5.3.4 A further consolidation of tests may be made by observing that the dependence of concentration estimates on the wind speed, v, is only sampler specific, i.e. does not depend on the specific analyte. Therefore, after a single measurement for a given sampler type, the set of tests can be narrowed to five runs with $5 \times 3 = 15$ degrees of freedom in the estimate of R_s.

5.4 Reverse diffusion

5.4.1 A potential problem with diffusive samplers is presented by the possibility of reverse diffusion (sometimes denoted as "back-diffusion" or "off-gassing") of the analyte. Reverse diffusion is generally only significant in the case that an analyte is only weakly bound to the sorbent (Reference [6]). Therefore, inaccuracy associated with these effects may usually be minimized through proper sorbent selection.

5.4.2 Because of reverse diffusion, estimates of a varying concentration may in some cases be biased. The worst-case situation occurs with the concentration in the form of an isolated pulse at either the beginning or end of the sampling period. A pulse at the beginning of the period allows the entire sampling period (4 h to 12 h) for sample loss, possibly resulting in a low estimate relative to a pulse at the end.

5.4.3 In some cases, the time dependence of a specific workplace concentration correlates strongly with the sampling period. For example, a cleanup operation at the end of a workday could introduce analyte only then. This could imply a positive bias in the concentration estimates obtained from a day's sampling. For simplicity, however, this International Standard is designed to assess performance of samplers for use in a concentration fluctuating in a stationary manner, so that time-dependent effects are treated simply as components of sampler variance. Specifically, the effect of an isolated 0,5 h pulse occurring at random within the sampling period is estimated.

5.4.4 Challenging samplers to 0,5 h pulses is similar to the tests of NIOSH (Reference [3]) and EN 838.