
**Workplace air — Guidance for the
measurement of respirable crystalline
silica**

*Air des lieux de travail — Lignes directrices pour le mesurage de la
fraction alvéolaire de la silice cristalline*

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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.

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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.

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Introduction

Respirable crystalline silica (RCS) is a hazard to the health of workers in many industries through exposure by inhalation. Industrial hygienists and other public health professionals need to determine the effectiveness of measures taken to control workers' exposure. Taking samples of air during a work activity and then measuring the amount of RCS present is often done to assess the exposure of an individual, the effectiveness of their respiratory protection or effectiveness of other controls. Studies have found significant problems can be encountered if procedures to ensure the quality of RCS measurements are not followed. In addition, there is interest in accurately measuring RCS at lower levels where the variability of measurements is poorer. If proper controls to limit bias and measurement variability are not employed, a reasonable measurement uncertainty cannot be achieved and usefulness of RCS measurements to make informed decisions to protect worker health is reduced. This International Standard is intended to be of benefit to those involved in the determination of RCS in the workplace, e.g. agencies concerned with health and safety at work; industrial hygienists; safety and health professionals; analytical laboratories; industrial users and their workers. Readers should be aware that in some countries there are legal requirements for the quality assurance of these measurements.

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Workplace air — Guidance for the measurement of respirable crystalline silica

1 Scope

This International Standard gives guidelines for the quality assurance of measurements of respirable crystalline silica in air using direct on-filter or indirect X-ray diffraction and infrared analysis methods. The scope of this International Standard includes the following crystalline silica polymorphs: quartz, cristobalite, and tridymite.

These guidelines are also relevant to the analysis of filters obtained from dustiness measurements in accordance with EN 15051^[6].

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.

ISO 7708, *Air quality — Particle size fraction definitions for health-related sampling*

ISO 15767, *Workplace atmospheres — Controlling and characterizing uncertainty in weighing collected aerosols*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

EN 482:2006, *Workplace atmospheres — General requirements for the performance of procedures for the measurement of chemical agents*

EN 689, *Workplace atmospheres — Guidance for the assessment of exposure by inhalation to chemical agents for comparison with limit values and measurement strategy*

EN 1232:1997, *Workplace atmospheres — Pumps for personal sampling of chemical agents — Requirements and test methods*

EN 12919, *Workplace atmospheres — Pumps for the sampling of chemical agents with a volume flow rate 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/TR 15230, *Workplace atmospheres — Guidance for sampling of inhalable, thoracic and respirable aerosol fractions*

3 Terms and definitions

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

3.1 General definitions

3.1.1

respirable crystalline silica

RCS

inhaled particles of crystalline silica that penetrate into the unciliated airways

3.1.2 Breathing zone

NOTE Both definitions 3.1.2.1 and 3.1.2.2 are adapted from ISO 4225:1994^[2].

3.1.2.1

breathing zone

⟨general⟩ space around the face of a worker from where he or she takes his or her breath

3.1.2.2

breathing zone

⟨technical⟩ hemisphere (generally accepted to be 0,3 m in radius) extending in front of the human face, centred on the mid-point of a line joining the ears; the base of the hemisphere is a plane through this line, the top of the head and the larynx, when no respiratory protective equipment is in use

3.1.3

exposure by inhalation

situation in which a chemical agent is present in air that is inhaled by a person

[ISO 8518:2001^[3], 3.1.3]

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3.1.4

measuring procedure

procedure for sampling and analysing one or more chemical agents in the air, including storage and transportation of the sample

[ISO 8518:2001^[3], 3.1.4]

3.1.5

operating time

period during which a sampling pump can be operated at specified flow rate and back pressure without recharging or replacing the battery

[EN 1232:1997]

3.1.6

limit value

reference figure for concentration of a chemical agent in air

[EN 1540:1998^[5]]

3.1.7

workplace

defined area or areas in which the work activities are carried out

[EN 1540:1998^[5]]

3.2 Sampling definitions

3.2.1

air sampler

device for separating chemical and/or biological agents from air and collecting them on a collection substrate

3.2.2

static sampler

area sampler

device, not attached to a person, used in **static sampling** (3.2.3)

[ISO 8518:2001^[3], 3.3.5]

3.2.3

static sampling

area sampling

process of air sampling carried out in a particular location

[ISO 8518:2001^[3], 3.3.4]

3.2.4

personal sampler

device attached to a person that samples air in the **breathing zone** (3.1.2)

[EN 1540:1998^[5]]

3.2.5

personal sampling

process of sampling carried out using a **personal sampler** (3.2.4)

[EN 1540:1998^[5]]

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3.2.6

sampling equipment

apparatus for collecting airborne respirable particles, including any size selection element and collection medium

3.2.7

sampling train

apparatus for collecting airborne particles including **sampling equipment** (3.2.6), pump and connecting tubing

3.3 Analytical definitions

3.3.1

sample preparation

operations carried out on a sample, after transportation and storage, to prepare it for analysis, including transformation of the sample into a measurable state

3.3.2

limit of detection

LOD

lowest amount of **respirable crystalline silica** (3.1.1) that is detectable with a given level of confidence

[EN 482:2006]

NOTE Many analysis procedures require laboratories to calculate an LOD by multiplying the standard deviation of measurements of a number of blank samples (~10) by three. Readers should note that there is some doubt about the relationship between signal and the mass when RCS is measured at very low masses and a specific formula to determine

the LOD using statistics based on a normal distribution is not given in this International Standard. The test samples used for calibration are not matrix matched and reporting an LOD based on three standard deviations of the background noise may give an optimistic impression of the capability of method when analysing “real” samples. Analysts should take this into consideration when analysing samples for RCS.

3.3.3
limit of quantification
LOQ

lowest reliable mass of **respirable crystalline silica** (3.1.1) that is quantifiable with a given level of confidence taking into consideration the matrix effects in the sample

[EN 482:2006]

3.4 Statistical terms

3.4.1
accuracy

closeness of agreement between a test result or measurement result and the **true value** (3.4.5)

NOTE In practice, the accepted reference value is substituted for the true value.

[ISO 3534-2:2006^[1], 3.3.1]

3.4.2
analytical recovery

ratio of the mass of analyte measured when a sample is analysed to the known mass of analyte in that sample

NOTE The analytical recovery is expressed as a percentage.

[ISO 8518:2001^[3], 3.5.1]

3.4.3
bias

difference between the expectation of a test result or measurement result and a **true value** (3.4.5)

NOTE Bias is the total systematic error as contrasted to random error. There may be one or more systematic error components contributing to the bias. A larger systematic difference from the true value is reflected by a larger bias value.

[ISO 3534-2:2006^[1], 3.3.2]

3.4.4
precision

closeness of agreement between independent test/measurement results obtained under stipulated conditions

[ISO 3534-2:2006^[1], 3.3.4]

3.4.5
true value

value which characterises a quantity or quantitative characteristic perfectly defined in the conditions which exist when that quantity or quantitative characteristic is considered

[ISO 3534-2:2006^[1], 3.2.5]

3.4.6
validation

process of evaluating the performance of a **measuring procedure** (3.1.4) and checking that the performance meets certain pre-set criteria

[EN 482:2006]

3.4.7**uncertainty (of measurement)**

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

[ISO/IEC Guide 98-3:2008^[4], 2.2.3]

NOTE 1 The parameter may be, for example, a standard deviation (or a given multiple of it), or the width of a confidence interval.

NOTE 2 Uncertainty of measurement comprises, in general, many components. Some of these components may be evaluated from the statistical distribution of the results of series of measurements and can be characterised by standard deviations. The other components, which also can be characterised by standard deviations, are evaluated from assumed probability distributions based on experience or other information.

4 Principle

The aim of this International Standard is to give information to minimise the errors in measurement. There are many factors that may influence the variability of a measurement from the true value. The uncertainty of an air measurement is the combination of the variation of measurements from the true value from both the sampling and chemical analysis. Within these two broad headings are other factors, some of which are illustrated below.

a) Organisational factors:

- 1) strategy;
- 2) method;
- 3) training and experience.

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b) Measurement factors:

- 1) sampling instruments;
- 2) calibration;
- 3) sample preparation;
- 4) instrumental variation;
- 5) interferences.

The variability of these individual factors combines to add to the uncertainty of the measurement. At stages within the sampling and analysis process, the factors that contribute to the variance of a measurement can be monitored and controlled to reduce the expanded uncertainty. With such an approach, the variation of the measurements is minimised.

The quantifiable contributions to the uncertainty of crystalline silica in air measurement are illustrated in the classic cause and effect diagram often used as an aid to estimate uncertainty in measurement in Figure 1.

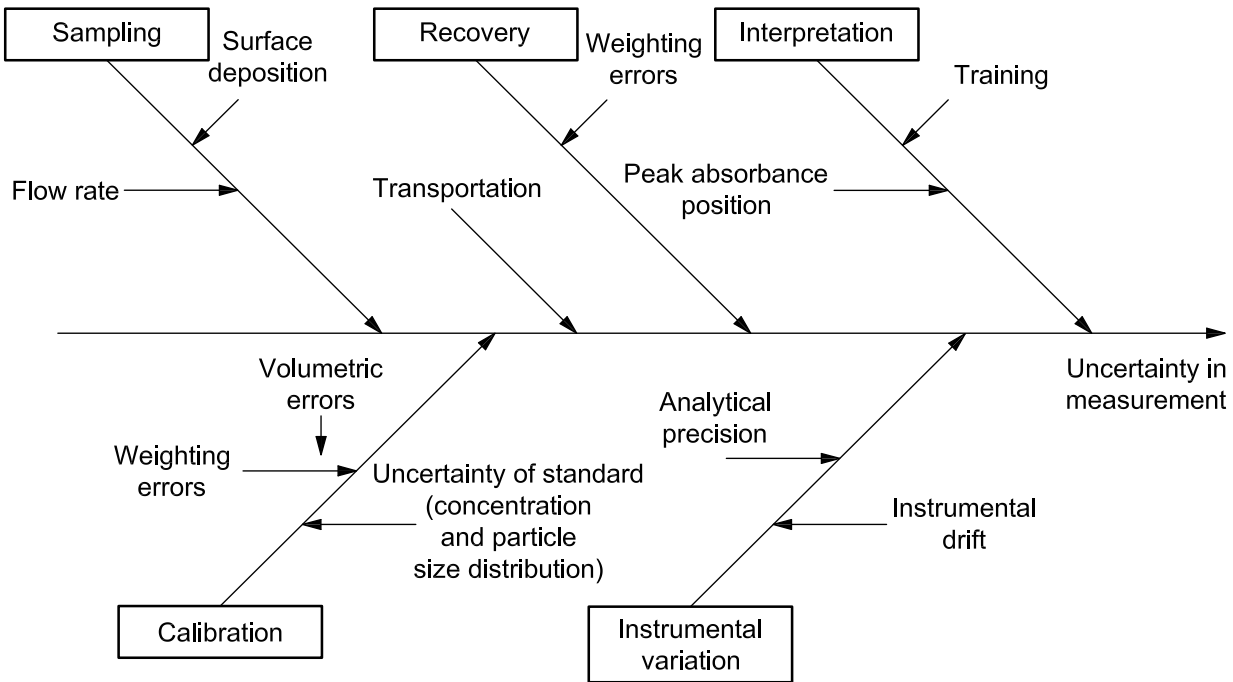


Figure 1 — Cause and effect diagram illustrating sources of error in the measurement of respirable crystalline silica

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5 Analytical quality requirements

The level of analytical quality required for effective occupational hygiene monitoring should be considered before procedures for establishing a quality assurance program are discussed. Statistical quality control procedures can determine what is currently achievable in terms of intra- and interlaboratory precision and bias and may throw some light on the relative accuracy of different methods, but they do not determine what are desirable levels of trueness and precision.

The existence of legislative requirements to take corrective action when exposure limits are exceeded has a bearing on analytical quality requirements. Laboratories with a negative analytical bias may be putting workers' health at risk; those with a positive bias may be placing an additional financial burden on an industrial company and so putting it at a competitive disadvantage.

NOTE The European Committee for Standardisation (CEN) Working Group on performance criteria has suggested limits to the expanded uncertainty in occupational hygiene analyses to reduce the potential for incorrect decisions due to the poor precision of results (EN 482). This uncertainty includes the imprecision in the sampling and analytical methods and is specified as a maximum of $\pm 30\%$ at the exposure limit value and $\pm 50\%$ at about half the limit value. The National Institute for Occupational Safety and Health (NIOSH) in the USA stipulates an expanded accuracy requirement of $\pm 25\%$ for its occupational hygiene methods (Reference [9]), based on laboratory tests. The NIOSH accuracy requirement can be expanded to $\pm 35\%$ for accuracy determined using field comparison of methods (Reference [17]).

6 Administrative controls

ISO/IEC 17025 specifically addresses the establishment of a management system to ensure the traceability of measurements. This section outlines some essential good practice for measurements of crystalline silica in air.

The term “ensuring quality” is used in the sense of the monitoring and control of the precision and accuracy of laboratory measurement, and this International Standard is concerned mainly with this use of the term. However, the statistical control of analytical quality is only one aspect of laboratory quality control. A broader

definition would include the monitoring and control of all the influencing factors on the test result within the laboratory from the receipt of the sample to the dispatch of the report.

In some respects, even this definition is too narrow if quality is to be seen in the wider terms of the role of the laboratory in ensuring the usefulness of a result. Thus, for example, the quality of communication between the laboratory and the occupational hygienist is a very important aspect of quality assurance that should not be overlooked. Good communication on the part of the industrial hygienist should include additional qualitative information about the sample, such as sample matrix, sampling time, and sampling instrument, so that appropriate calibration standards and limits of detection can be applied and interferences anticipated (see Annex A). Good communication practices help to ensure: the production of timely results; a clearly presented report; or even that an appropriate analysis was undertaken. All of these are essential if results are to be understood and appropriate action taken. A report that arrived too late or one that was presented badly and was thus misinterpreted negates the effort put into the analysis.

Analytical quality does not depend solely on the skill of the analyst and on statistical techniques for monitoring the accuracy and precision of analytical methods. An important decision in analytical quality assurance is the initial selection of the analytical method to be used, which should be robust, and whose performance characteristics (bias, precision, LOD, etc.) are determined and documented. The method selected should be appropriate for the level of qualification and skill of the analyst who is to use it. Any modifications to a validated analytical method should be recorded and evidence of data equivalency should be available for audit.

The laboratory manager has a central role to play in choosing appropriate analytical methods and is also responsible for several equally important influences on analytical quality. These include the establishment and auditing of administrative procedures for sample identification, for the reporting of results and for the maintenance of records; the management of the purchase and maintenance of equipment and the purchase of reagents and other consumable supplies; and the training and deployment of staff.

A person should be responsible for establishing and monitoring the quality assurance program of the laboratory. Responsibility for day-to-day quality control matters may be devolved to the analyst, but more thorough analyses of the quality control data should be conducted to look for trends and to associate changes in results with specific events.

A quality assurance scheme should not simply assess the performance of individual members of staff. It is the job of the laboratory manager to ensure that analysts are properly trained and that they are not required to perform analyses for which their level of knowledge, training or skill is inadequate. A high level of expertise is required to optimise instrument parameters and correct for matrix interferences either during the sample preparation phase or the data analysis and interpretation phase. For many methods, unknown factors may influence results. A quality assurance programme helps to identify those parts of the method that are operator dependent so that steps can be taken to eliminate this dependency. If a method gives different results with different analysts, the fault may lie with the method and not the analysts.

In small laboratories, it is possible that more than one task is performed by one individual, but it is important that an individual is responsible for specific tasks, such as those listed in paragraphs a) to f).

- a) The individual responsible for sampling should clearly identify each of the samples taken and record essential information. Essential information required by the laboratory for the analysis of the samples consists of identification of the sample, type of size-selective device and its flow rate, the volume sampled, and information about the industrial process; such as the composition of materials and the temperatures involved. If the person taking the samples provides this information to the laboratory it avoids the need for the analyst to contact the person responsible for sampling.
- b) An individual should be responsible for recording the receipt of the sample(s) and allocating each sample a unique identification that is correlated with any original field identification provided with the sample(s).
- c) An individual should be responsible for analysis and checking the performance of the method.
- d) An individual should be responsible for checking any answer recorded including its compliance with any instrumental checks and quality control tests. The interpretation of measurements for crystalline silica in air requires experience, so the individual responsible for checking should be familiar with the interpretation of spectra or scans. Managers should consider whether analysts would benefit from training