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Workplace air — Guidance for the measurement of respirable crystalline silica

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British Standards

National foreword

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

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Foreword

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ISO 24095 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

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.

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 place 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]

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]]

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.
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.

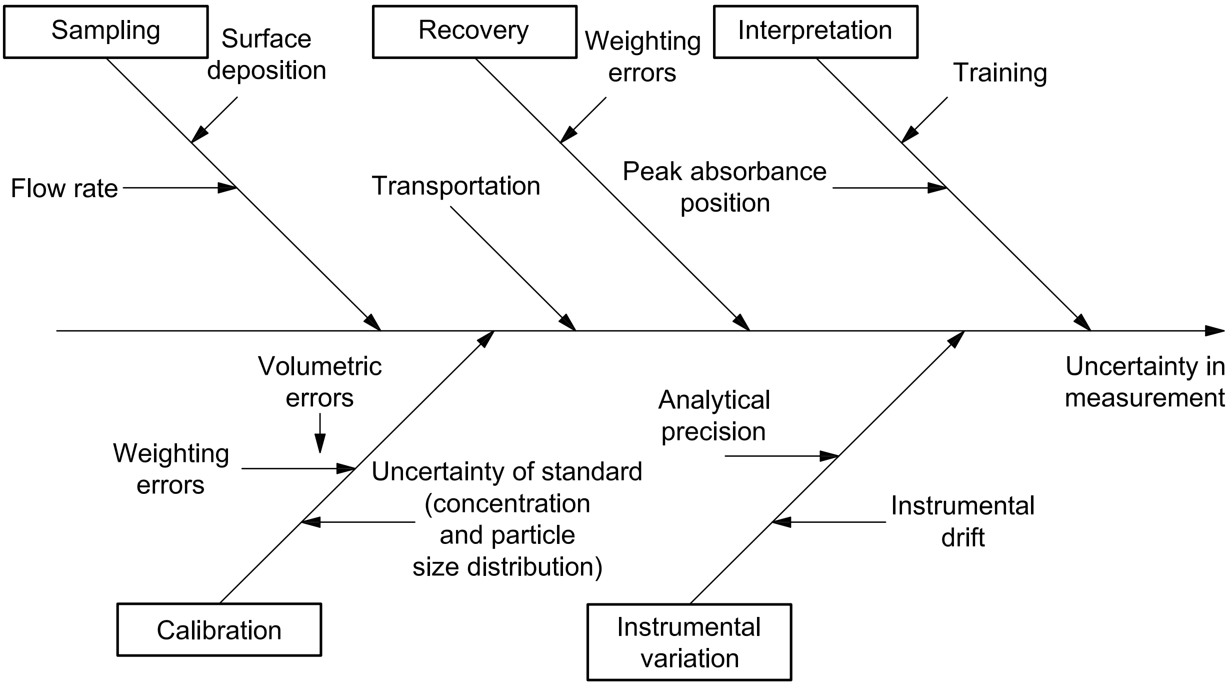


Figure 1 — Cause and effect diagram illustrating sources of error in the measurement of respirable crystalline silica

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 ± 30 % at the exposure limit value and ± 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 ± 25 % for its occupational hygiene methods (Reference [9]), based on laboratory tests. The NIOSH accuracy requirement can be expanded to ± 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 in mineralogy and/or advanced analytical techniques, such as peak profile fitting and spectral interferences.

e) An individual should also be identified to check that the quality assurance procedures are followed.

f) The quality system should be reviewed periodically.

The role of management in achieving analytical quality control is not considered further here, but laboratories are encouraged to obtain accreditation from a recognised accrediting body that demonstrates that the management and administrative procedures of the laboratory are sufficient to ensure that its work is conducted with integrity. The organisation of a laboratory may depend on it size, but it should have a clearly defined structure with the responsibilities of individuals also clearly defined.

7 Sampling

7.1 General

To avoid underestimation of exposure, special attention should be given to pumps and sampling equipment (see EN/TR 15230, EN 1232). It is important that the sampling equipment and the associated medium, such as filter or foam, are compatible with the intended method of analysis. In some recommended or official methods the sampling instrument is specified. Changes to official or recommended methods (Reference [14]) should be validated to ensure they are appropriate and comparable.

Sampling strategy should also be considered. EN 689 specifies one sampling strategy. Other examples include Reference [7] from NIOSH and Reference [16] from the American Industrial Hygiene Association (AIHA).

7.2 Sampler

A specific sampler should comply with ISO/CEN aerosol size-selective definitions, except where local regulation requires otherwise, and have a performance as required by EN 13205. The sampler should operate at a specific flow rate to collect the respirable size fraction. All connections included in the sampling trains should be checked prior to use to avoid leaks.

NOTE Health-related sampling conventions are a series of curves representing the probability of particle penetration to different regions of the human airways system for particles with an aerodynamic diameter up to 100 µm. For smaller particles, especially around the deposition minimum of 0,3 µm, a substantial quantity of inhaled particulate can be exhaled, but this is not accounted for in particle penetration conventions. The sampling convention to be followed for RCS sampling is the one termed “respirable”. Sampling convention probability varies from 0,1 % at an aerodynamic diameter of 10 µm to 100 % at 1 µm. The 50 % penetration efficiency, D_{50}, is, at 4 µm, a compromise between earlier recommendations from the British Medical Research Council ($D_{50} = 5$ µm) and the American Conference of Governmental Industrial Hygienists ($D_{50} = 3{,}5$ µm).

Because of the range of sampler types available, practical aspects of their use require consideration. One of the most important is the flow rate required, both with respect to the size, mass, and cost of pump needed, as well as with respect to the LOD (but conversely also the potential for overloading). Additional factors that should be considered are matrix effects, product information, and minimum sampling times for low levels. There may be issues specific to certain samplers such as particles adhering to walls, orientation to the wind, ability to clean and re-use or even complexity of calibration and checking the flow rates. Consult the product information before use.

7.3 Filters and foams

Foams are generally associated with a specific sampler and only the type of foams supplied with the sampler should be used. Filters should be of a diameter suitable for use in the selected sampler. The chosen filter type should have a filtration efficiency of not less than 95 % over the range of particle sizes to be collected (1 µm to 10 µm) and be suitable for collection of samples of crystalline silica. Not all filters are suitable for all analytical methods. For example, silver filters are suitable for X-ray diffraction (XRD) analysis but not infrared

(IR) spectroscopy. For direct on-filter analysis, polyvinylchloride (PVC) filters or PVC-acrylonitrile co-polymer filters have suitable IR characteristics. Polycarbonate, PVC, and silver filters are frequently used for XRD analysis. The filter material may degrade if the sampling environment is particularly humid. It is possible to re-deposit the sample on to a different substrate from that used for sampling, although this step adds to the uncertainty budget of the procedure. The advantage or disadvantage of re-deposition should be balanced against other considerations (evenness and thickness of deposit, removal of interfering species, etc.).

7.4 Sampling pumps

Sampling pumps should comply with the provisions of EN 1232, or equivalent performance standard, with an adjustable flow rate, incorporating a flow-meter or a flow fault indicator, capable of maintaining the selected flow rate to within ± 5 % of the nominal value throughout the sampling period. If the flow rate is not within ± 5 % of the nominal value, the person recording the flow rate measurement should inform the person responsible for communicating the results to note the observation in the final report. This situation can occur when sampling very high concentrations. Efficient pulsation damping of the flow is important for correct size selection. The battery for the pump should be fully charged prior to the sampling exercise. The flow rate through the sampling train should be checked at the site and any necessary adjustments made prior to sampling. Extreme conditions of temperature and pressure have an influence on the volume of air sampled. In good conditions, the temperature may change by 1 °C for every 100 m change in altitude and this has very little effect. However, the temperature in mines can vary significantly because of the depth, machinery present and ventilation/air extraction rates. If the flow rate through the sampling train was originally checked with a float type flow meter (rotameter), then a correction to the flow rate may have to be made to take account of different conditions of temperature and pressure. Corrections are only necessary when the conditions at the site differ substantially from those where the flow rate check was originally made (e.g. using the universal gas law, a temperature change of 10 °C would change the volume sampled by about 3 %). A flow rate check of the sampling train should be made close to the site of the working operation to ensure the pump is functioning correctly at the beginning and again at the end of sampling period to ensure compliance with EN 1232. When taking personal samples, sampling pumps worn by personnel should not impede normal work activity.

7.5 Respirable size selectors

The pump should deliver a constant flow rate with low pulsations to be sure to respect the requirements of the respirable size selector and/or at least EN 1232 or EN 12919 (flow rate ± 5 %, pulsation ± 10 %). The reader should also refer to ISO 7708 or EN/TR 15230.

7.6 Transportation

At most normal loadings (< 4 mg), the sample (of respirable dust) is safely contained on the filter and is not lost if the sample is handled with care. Test samples containing relatively small amounts of material (< 1 mg) have been sent through postal systems worldwide without significant losses in material from the filter. However, samples from coalmines are particularly prone to surface abrasion and need particular care to avoid sample loss. Filters from coal mines should be retained within a specially designed sample cassette for transport. When samplers with foams are used, the foam is retained in place using a cap.

8 Procedures

8.1 Handling of filter cassettes

The user should refer to the manufacturer's handling instructions. For a cyclone selector, the respirable size selectors should be stored and opened with the grit pot for the non-respirable particles kept vertically below the cassette. Ensure that the filter cassettes are firmly closed. Care should be taken upon opening the filter cassette to avoid loss of dust. The condition of the cassette should be visually checked and any deposition of dust on the walls of the cassette should be noted, if it is not intended to be analysed by procedure. Static dissipative cassettes may be useful in minimising wall deposition of dust.

8.2 Method validation

It is recommended that laboratories use only official or validated methods. If other methods are used, then they should prove that they are validated methods that have comparable or better performance characteristics when compared with published methods. A Eurachem Guide[12] describes the method validation process. If deviations from a standard method are made, then the laboratory should prove that the changes give comparable results and note the modifications when the results are reported.

8.3 Calibration

Measurements should be traceable. When weighing calibration test samples, use a balance with a resolution of 1 µg. This resolution is important when weighing low microgram levels (< 200 µg). This also requires the use of E2 class certified check masses to verify the balance calibration. Humidity and electrostatic effects may affect the stability of the weighing (see ISO 15767). Weighing procedures for filters are given in ISO 15767.

Good practices use not less than six concentration levels of calibration test samples in order to reduce the standard error of the regression line to an appropriate level and use three replicates at each concentration level. To reduce the uncertainty of the calibration line, at low measurement levels, some direct on-filter analysis methods for RCS recommend 30 calibration test samples because of the difficulty of producing replicates. The calibration test samples need to be equally spread across the analytical range in order to avoid a regression or other calibration model from being unduly weighted by isolated responses that may lead to significant differences between results. Current evidence suggests that IR analysis produces data with a constant standard deviation, whereas XRD analysis produces data with standard deviation varying with mass of RCS. Therefore a weighted regression may be more appropriate for XRD analysis. The calibration should be tested against some known sample such as a proficiency testing or spiked quality control sample.

To minimise variability, the preparation of the calibration standards needs to be fit for its intended purpose. For direct on-filter methods, the procedure for the preparation of the standards should match the sample collection methods. For deposition methods, the procedure for the preparation of the calibration standards should match the sample preparation methods.

The response from both XRD and IR analysis techniques is sensitive to the distribution of the size of the particles on the surface of the analysis filter and different standards may have a slightly different distribution of particle sizes, which may lead to differences in results between laboratories. Measurement of peak area shows less variability with particle size for XRD, because, as the median particle size decreases, the peak shape broadens, which decreases the height. Peak height is usually measured for IR analysis.

If the calibration dust used in a laboratory is not a certified reference material (CRM) or a secondary standard with a known amount of RCS, then the response of the calibration dust should be compared with these materials to determine any potential bias. If the absorbance or diffraction peak profile is outside the repeatability precision of the measurement and is considered significant at the 95 % confidence level, a correction factor should be applied to the final result or calibration. The result reported to the customer should be corrected for purity and crystallinity of the dust standard.

For example, if XRD is used and the difference between the area result for a sample of the bulk reference material is greater or less than 2,26 (Student t-distribution for nine degrees of freedom), standard deviations of 10 peak area measurements of the bulk dust used for calibration then the correction factor for crystallinity of the calibration dust is

$$\frac{\bar{A}_{\text{CM}}}{A_{\text{Ref}}} \; \frac{w}{100} \tag{1}$$

where

$\bar{A}_{\text{CM}}$ is the average area, in counts per second, of the principal peak(s) of the calibration material;

A_{Ref} is the area, in counts per second, of the same principal peak(s) of CRM or secondary standard with a known concentration of RCS;

w is the mass fraction, expressed as a percentage, of crystalline silica in the CRM or secondary standard.

Alternatively, the correction values for common calibration dusts for α-quartz can be obtained from published work (Reference [18]).

It is recommended to report the material used to prepare the calibration test samples. Treatment of the reference material during calibration may also change the particle size characteristics of the dust when they are sampled again and so lead to different absorbances for IR analysis and diffraction peak profiles.

The frequency of calibration depends on the stability of the instrument and the experience of the analysts. When using XRD, it is not necessary to recalibrate frequently, when a calibration is proved to achieve satisfactory results, provided the instrumental conditions and parameters are maintained for the method of analysis, an external monitor is used to correct the calibration for tube drift and quality control and/or external quality assurance programmes are in place to monitor the performance of the procedure.

8.4 Sample preparation

Some analysis methods require the dust to be recovered from the original air sample filter to allow for sample treatment to remove interferences and then the recovered dust is re-deposited on to another filter. The best practice is to prepare a number of standards (more than 1) from a calibration solution and involve these in the sample preparation process to test the recovery. If the difference between the target value and the results obtained from the recovery samples are significantly more than the expected precision of the procedure, then a correction factor for recovery should be applied. For example, if the target value is more than two standard deviations from the expected precision (determined from experience of proficiency testing, quality control, published or method validation data) of the recovered values.

For re-deposition methods, it is recommended that laboratories choose the most appropriate “clean-up” technique for the mineral matrix in the sample. Ashing in a furnace can remove carbonaceous materials and dilute acids can remove carbonates. There may be matrix interferences such as silicates that could be handled best by a sample clean-up step prior to analysis. This can be accomplished by an acid digestion according to Reference [19]. This procedure does not successfully remove some silicates such as feldspars (albite) and may cause the loss of very small particles of RCS and the formation of amorphous silica.

During the re-deposition process, care should be taken to insure that there is a complete seal for filtration of the reconstituted crystalline silica. Leakage leading to sample loss can occur if there is an insufficient seal. Ultrasound should be used before the filtration to overcome the agglomeration of particles in suspension and to ensure homogeneity. Placing about 2 ml of solvent in the filtration funnel before pouring the sample provides a cushion for the suspension and contributes to a homogenous surface.

XRD parameters related to sample preparation include sample homogeneity, size distribution of the particles, size of the sample surface exposed to the X-ray beam, thickness of the sample deposition, and choice of internal standards if any (including absorption correction factors if silver filters are used for XRD sample deposition).

8.5 Sample measurement

8.5.1 X-ray diffraction

XRD is a technique where X-rays are reflected from the surface of the sample and most methods assume the sample deposit is a thin layer and so does not need any correction for any absorption or depth effects. Some methods use the silver filter as an internal standard to help correct for any matrix effects. Measurement of the response of the silver from the filter sample helps correct the intensity of the signal when the relationship between response and intensity is no longer linear because of the depth of sample. Since crystalline silica often occurs in a matrix with other silicate minerals, some of which exhibit X-ray spectra that overlap with the primary quartz diffraction peak, the use of alternative peaks for quantification may be necessary. Annex A gives a list of potential mineral interferences. The use of secondary, tertiary, and even quaternary peaks results in decreased sensitivity. Good practice includes a qualitative XRD scan to characterise the environmental matrix for a set of samples from a given site. A determination revealing the correct peak ratios for the three largest peaks can be taken as an indicator of lack of interferences; otherwise, a clean-up procedure may be needed prior to analysis.

Sample spinning during XRD analysis allows for the alignment of each crystal in the X-ray beam and increases the precision by ensuring that all crystals are measured.

8.5.2 Infrared analysis

For IR, matrix effects from interferences with other polymorphs of crystalline silica and silicates, such as kaolinite, present analysis problems. There is a potential for bias when correcting for matrix absorption effects, with the bias increasing at low levels of quartz. In coal mining, the assumption is made that quartz is the only polymorph present due to the geological processes involved in coal formation. It is sometimes assumed that the only mineral interference found in coal is kaolinite. Spectral correction is possible to eliminate IR interference from kaolinite in coal.

For direct-on-filter IR analyses, good practice requires that samples be rotated and multiple measurements taken and averaged in order to minimise possible differences in deposition across the filter.

8.6 Instrumental variation

8.6.1 X-ray diffraction

8.6.1.1 Intensity

For optimum XRD instrument performance, the X-ray source should be aligned and monitored routinely for stability. An estimate of instrumental drift should be performed whenever measurements are made. For XRD, the intensity of the copper radiation deteriorates over time and a correction should be applied to correct for the instrumental drift. An aluminum plate or any other suitable stable robust material can be used as an external standard to correct a measurement for the gradual decline in X-ray tube emission from the calibration. Such a standard should be fine-grained, free from marked texture and have a strong XRD peak in the same range as the quartz or cristobalite peaks being used for analysis.

8.6.1.2 2θ position

In a diffractometer with Bragg-Brentano geometry, the diffracted intensity measured should be independent of 2θ. Displacement of the slit assemblies changes the semi-focusing geometry and may lead to intensity becoming slightly dependent on 2θ. Because the ratios of the intensities of peaks in different positions are used both for identification and quantitative analysis, such a displacement would lead to erroneous results, although a displacement large enough to have a serious effect is unlikely. Checking the intensity ratio of diffraction peaks from a reference sample is a method of detecting serious errors.

The peak width provides a further check on instrument operation. It is appropriate to use a standard reference material such as NIST 640c to check the instrument after service or to use other characterised material such as Arkansas stone that gives reproducible line positions.

8.6.2 Infrared analysis

A check sample, previously analysed proficiency testing sample or known standard should be used to check instrumental drift and resolution.

8.6.2.1 Absorbance

On modern IR instruments, the laser intensity (energy) should be checked periodically for deterioration.

8.6.2.2 Resolution

For IR, modern instruments employ their own checks for wavelength drift and the absorbance of silica is a relatively broad peak. It is recommended that the resolution for analysis should be set at no more than 8 cm^{-1}.

9 Internal quality control

The analytical precision is dependent on the particular conditions employed for the analysis. The aim of a quality control scheme is to check every stage of an analysis. One way to check that an analytical procedure is functioning correctly is to take a matrix-matched sample of known value through the entire analytical procedure in the same way as the field samples. Ideally, if an indirect analysis procedure is employed then the samples for quality control should be included with the samples during the preparation stage of the method, so that both accuracy and precision are evaluated. Any untoward losses or contamination experienced by the field samples during the analytical process is likely to be experienced by the quality control sample also and the analyst may be alerted to any deviation and take it into account.

It is recommended that for analyses, which are determined frequently, a batch quality control material should be prepared. A suitable quality control material might be a filter spiked with a known amount of crystalline silica added to it, or a past proficiency-testing sample.

A good practice is to plot the value obtained for the quality control sample on a chart. It is much easier to detect changes in this way than by looking at lists of figures recorded in a notebook. The simplest form is the Shewhart chart that is described in more detail in Annex C. A Shewhart chart with simple rules, i.e. warning at $\pm 2s$, action at $\pm 3s$, where s is standard deviation, is probably sufficient for a laboratory starting up a quality control scheme. A more complex set of rules can be employed once analysts are familiar with quality control procedures.

A good practice is to check the standard deviation of the method at intervals and revise the warning and action limits if necessary. The data used to calculate the initial standard deviation may not be representative and the application of a quality control scheme may result in an improvement in the precision of the results obtained, so that the old warning limits may no longer be appropriate.

10 External verification and assessment of uncertainty

A laboratory may be able to control its precision, but still report inaccurate results if the bias between the true value and the results it obtains is not assessed. An internal quality control scheme helps a laboratory control its precision but does not include potential bias from the calibration standards, preparation of calibration samples, unaccounted factors in recovery and interpretation of data. Participation in proficiency testing programmes helps a laboratory to compare the results it obtains on almost identical samples with the results from other laboratories and laboratory clients can use records of laboratory proficiency performance to determine the ability of the laboratory to produce measurements of consistent quality.

A laboratory should select a proficiency-testing program that is appropriate for its method of analysis. Proficiency testing is preferable but national methods may not be compatible with the particular requirements of the proficiency-testing scheme. In these cases, an internal assessment protocol is essential for monitoring the quality of the data.

For interpretation of analytical measurements, good practice should include an assessment of the uncertainty of the measurements. ISO/IEC Guide 98-3[4] describes an approach for estimating the uncertainty associated with measurement. An uncertainty range should be reported with the results to aid in the interpretation of data. An example of the ISO/IEC Guide 98-3[4] approach to estimate the uncertainty of results is shown in Annex D.

11 Test report

11.1 Minimum report requirements

The test report shall contain at least the following information:

a) details of the method used, with reference to this International Standard;

b) all information necessary for the complete identification of the sample;

c) the condition of the sampling medium such as damage, overloading and any residual dust in the container used for transport;

d) the dust standard used for the calibration;

e) the mass, in micrograms, of analyte;

f) the estimated expanded uncertainty;

g) the LOD and if appropriate the LOQ;

h) the name of the analyst;

i) the name and address of the laboratory;

j) the signature of the laboratory manager or a person delegated by the laboratory manager;

k) the date of the analysis;

l) the name and the model of the instrument used for the analysis;

m) identification of the report;

n) the interferences present during the analysis of the sample;

o) analytical range of the calibration standards;

p) the storage period of the samples;

q) all operating details not specified in this International Standard, or regarded as optional, together with details of any incident that may have influenced the result(s).

11.2 Data to be archived by the laboratory

The following is the minimum that should be archived by the laboratory:

a) a copy of the report sent to the customer;

b) a copy of the scans and parameters of the instrument;

c) the calibration curve;

d) the samples for a period of time to allow analyses to be verified;

e) a copy of the written procedures used to perform the analysis and the results from any tests used to demonstrate the comparability of any changes from the standard method.

Annex A
(informative)

Polymorphs of crystalline silica and their interferences

A.1 Interferences in the XRD and IR spectra of RCS

Industrial hygiene samples for RCS are often taken in environments that include mineral matrices such as graphite in the iron and steel industry and various silicates in agriculture and ceramics. The presence of mineral compounds that have characteristic peaks in close proximity to the peaks of the crystalline silica polymorphs can interfere with the measurement of the RCS. It is important to characterise the composition of the dust that was collected in order for the analyst to have a good understanding of any suspected interfering compounds. When the sampled dusts are submitted to the laboratory for analysis, it is advisable to provide all available information relating to the industrial process, including raw materials, extraction processes, transformation products and packaging of the final product.

A.1.1 X-ray diffraction

In XRD, interferences can be detected with the analytical technique. Most XRD reference methods call for a qualitative analysis of a representative bulk sample in order to investigate the presence of interfering minerals (NIOSH Method 7500[8], OSHA Method ID-142[10], and MDHS 101[15]). In this case, the interfering minerals can be identified in corroboration with any information provided by the hygienist who collected the bulk sample. If interferences are found, sample clean-up can be performed prior to analysis of the respirable samples or a secondary peak can be used with an associated loss in sensitivity. Other XRD reference methods require the analyst to measure all the principal peaks for each crystalline silica polymorph (OSHA Method ID-142[10]). The known relative intensities are concurrently compared during each sample run and deviation from the expected value is considered to be an indicator of the interferences in that sample. In XRD, the diffraction peaks of the main polymorphs may be subject to overlap, changes in intensity or distortion of peak shape. It is possible to obtain a reference profile from the calibration material and to use software to compare the relative intensity, peak width, and peak symmetry with the peak profile of the sample to estimate the amount of silica present. However, it may be simplest to reject results from peaks with interference and use a different silica peak form analysis. Alternatively, if the interfering phase is known, it may be possible to make a simple correction by measuring the intensity of another line of the interfering phase and then making an appropriate adjustment to the intensity. It is to the advantage of the analyst and the hygienist to have a working knowledge of the common interferences that are found in various industrial environments.

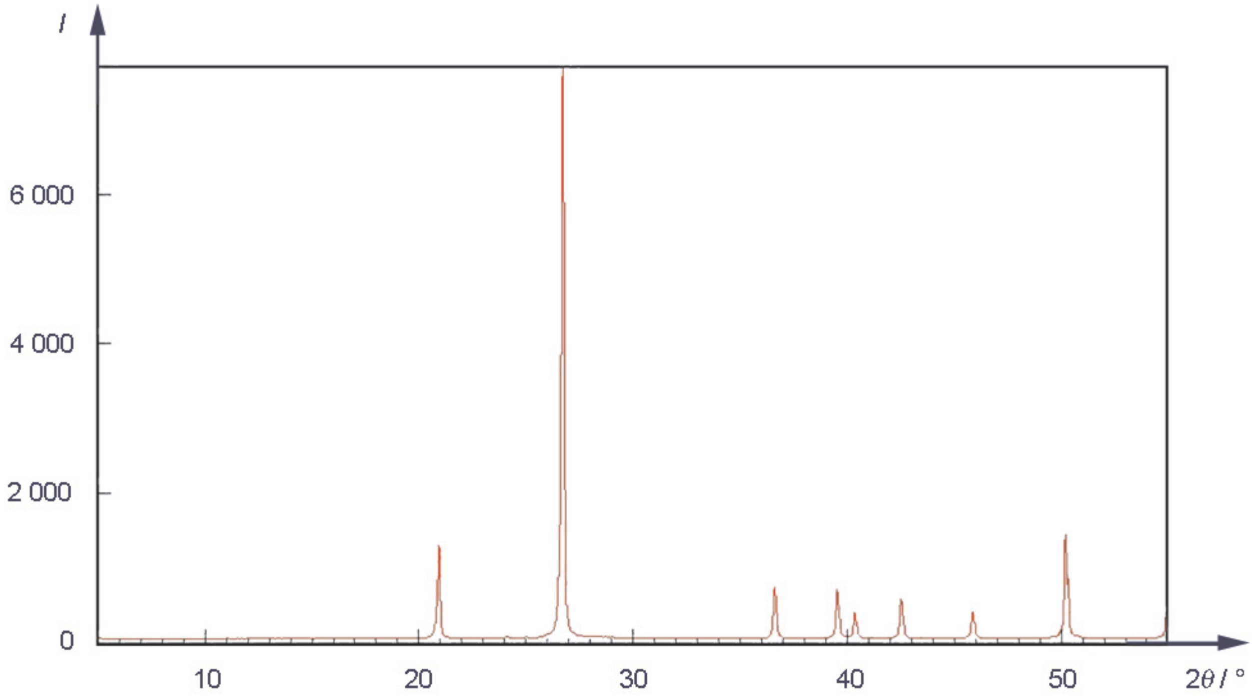
The four main peaks of α-quartz are given in Table A.1 along with several known mineral interferences.

Table A.1 — Peaks and intensities of α-quartz and known mineral interferences

Analysis peak	(100)	(101)	(112)	(211)
Relative intensity	22	100	14	9
d (Å)	4,26	3,34	1,82	—
Angle of diffraction (2θ) Cu K_α	20,85	26,65	50,17	59,96
Interferences				
Albite		X	X	X
Anorthite			X	X
Aragonite	X	XX	X	X
Barite		XX	XX	
Biotite		XX		X
Cristobalite				X
Graphite		XX		
Kaolinite	X	X	X	X
Maghemite	X	X	X	
Microcline	X	XX		
Mullite		XX		X
Muscovite		XX		
Orthoclase	XX	XX	X	
Sillimanite		XX	X	X
Tridymite	XX		X	X
Wollastonite		XX	X	X
Wüstite				X
Zircon		XX		X
XX indicates a serious interference				

A.1.2 Typical X-ray diffraction scans of α-quartz and potential interferences

For an XRD scan of crystalline a-quartz, see Figure A.1. For an XRD scan of a sample containing a feldspar and α-quartz, see Figure A.2. Figure A.3 shows an XRD scan of a sample containing gypsum and a trace amount of feldspar showing the potential for interference with α-quartz.



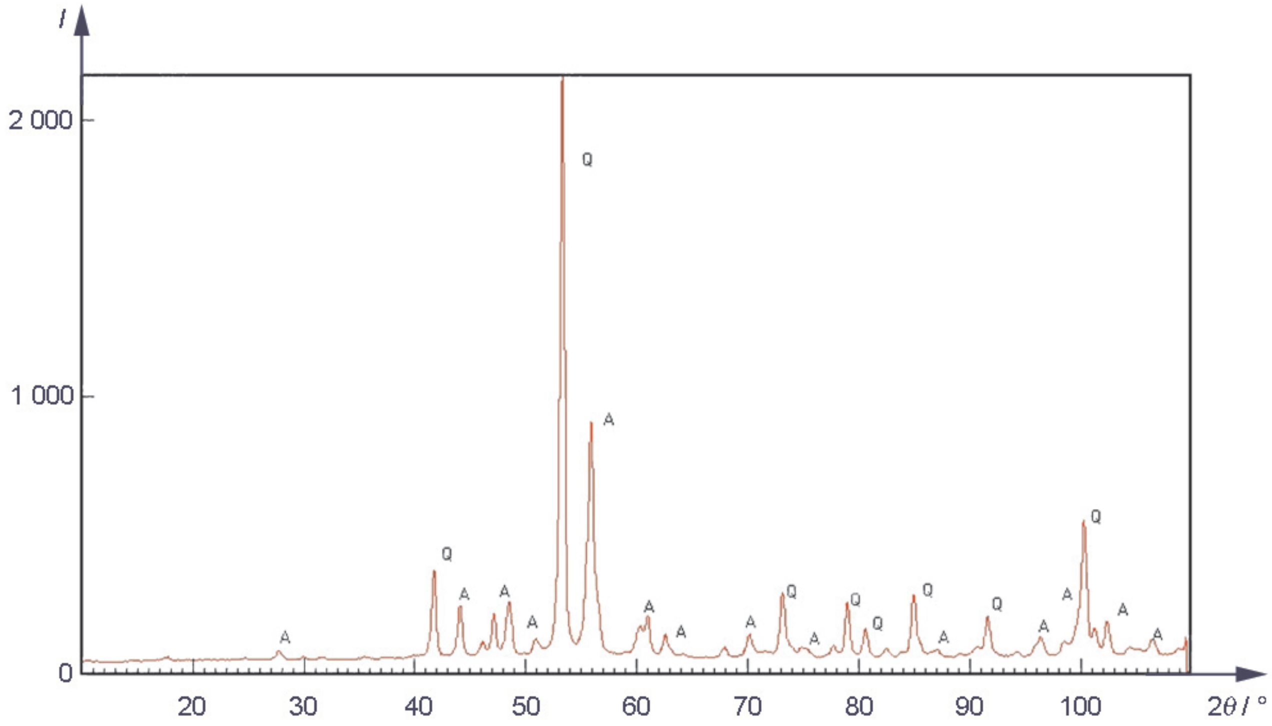
Key

2θ peak position

I intensity/counts

NOTE A scan of cristobalite is shown in Annex B.

Figure A.1 — An X-ray diffraction scan of crystalline α-quartz

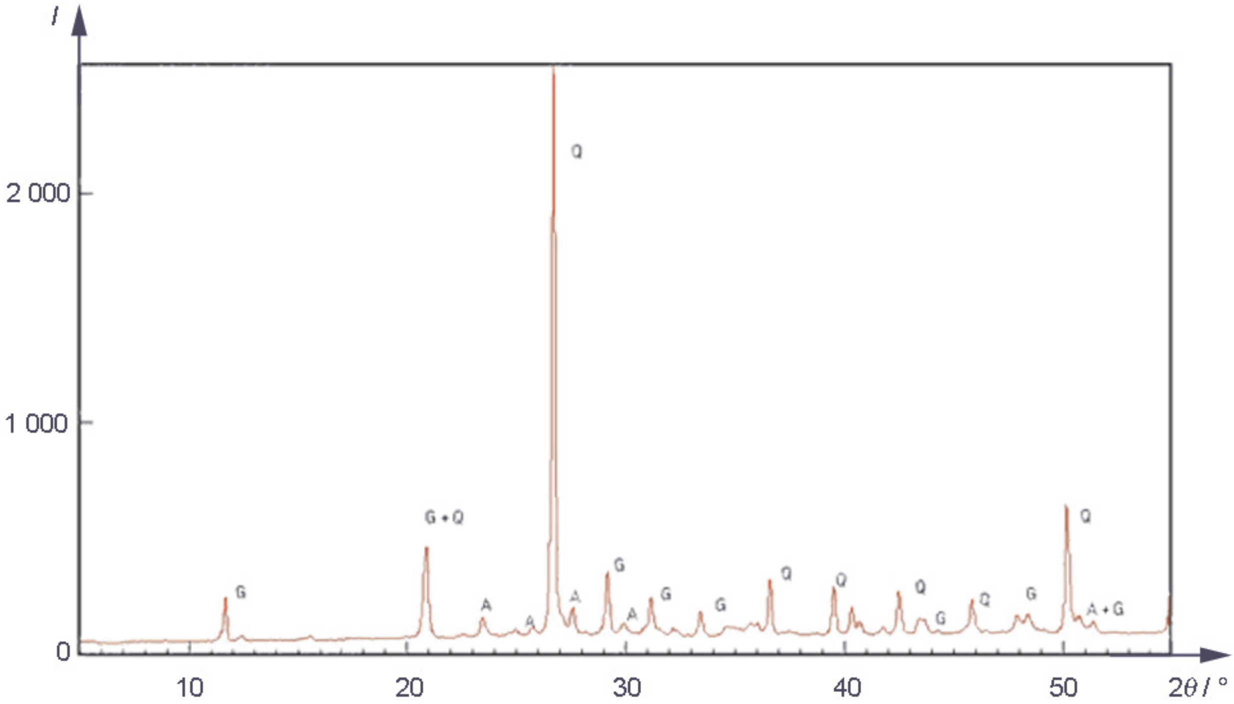


Key

2θ peak position
I intensity/counts
A feldspar mineral similar to albite low
Q α-quartz

NOTE The scan shows potential interference with the 26,65° and 50,1° 2θ reflections of α-quartz.

Figure A.2 — An X-ray diffraction scan of a sample containing a feldspar (similar to albite) and α-quartz



Key

2θ peak position

I intensity/counts

A feldspar mineral similar to albite low

G gypsum ($CaSO_4 \cdot 2H_2O$)

Q α-quartz

Figure A.3 — An X-ray diffraction scan of a sample containing gypsum and a trace amount of feldspar (albite low) showing the potential for interference with α-quartz

A.1.3 Infrared analysis

Different polymorphs of crystalline silica exhibit distinct absorption patterns. α-Quartz exhibits a characteristic doublet at 798 cm^{-1} to 790 cm^{-1} and 779 cm^{-1} to 780 cm^{-1} (Figure A.4) and secondary peaks at 694 cm^{-1}, 512 cm^{-1}, 460 cm^{-1}, 397 cm^{-1}, and 370 cm^{-1}. The 694 cm^{-1} peak can be used for quantification in cases where mineral interferences overlap with the primary doublet. α-Quartz may be quantified in the presence of amorphous silica by using the 694 cm^{-1} peak or, alternatively, a phosphoric acid digestion may be used prior to analysis. If the phosphoric acid digestion procedure is used, the analyst should take care not to prolong the contact between acid and particles because the acid may dissolve the smaller sized particles of crystalline silica. Cristobalite exhibits characteristic peaks at 798 cm^{-1}, 623 cm^{-1}, 490 cm^{-1}, 385 cm^{-1}, 297 cm^{-1}, and 274 cm^{-1}. The 623 cm^{-1} peak may be used for quantification in cases where mineral interferences overlap with the customarily used 798 cm^{-1} peak. Tridymite exhibits characteristic peaks at 793 cm^{-1}, 617 cm^{-1}, and 476 cm^{-1}. It may not be possible to quantify tridymite in the presence of α-quartz or cristobalite.

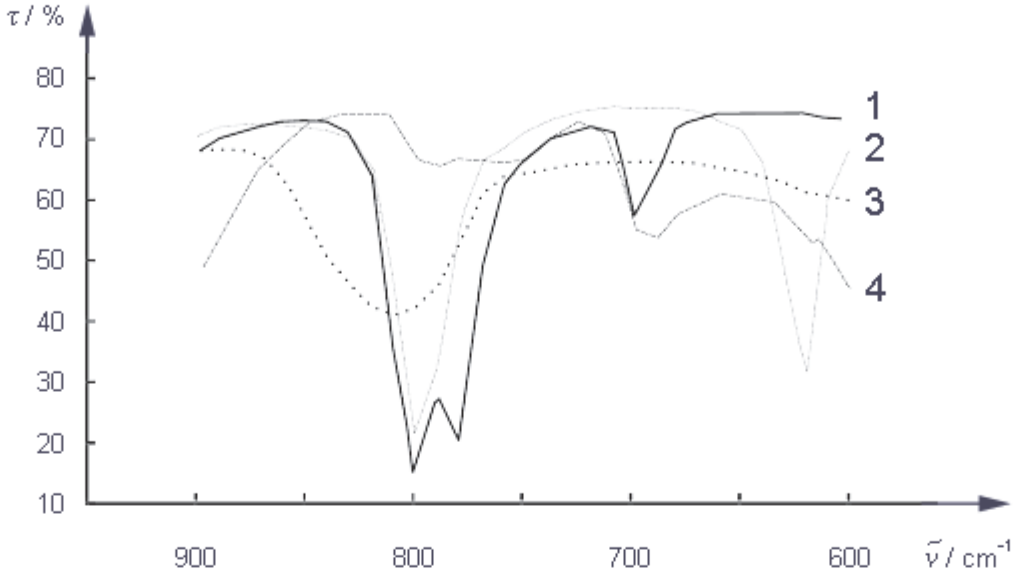
Matrix effects from interferences with other silicates present analysis problems. The following are examples of minerals groups that can cause interference:

a) clays e.g. kaolinite, dickite;

b) feldspars e.g. albite, anorthite, orthoclase;

c) micas e.g. muscovite.

The list is not exhaustive, since any mineral with a Si-O vibration stretch might contribute to absorbance in the same area as the principal absorbances of the silica polymorphs. There is a potential for bias when correcting for matrix absorption effects, with the bias increasing at low levels of quartz. In coal mining, the assumption is made that α-quartz is the only polymorph present due to the geological processes involved in coal formation. Generally, the only significant mineral interference found in coal is kaolinite. The analytical method MSHA Quartz Analytical Method P-7[11] gives a spectral correction procedure for eliminating IR interference from kaolinite. Although most analytical chemists are familiar with the IR technique as applied to organic analyses, mineralogical samples require additional knowledge of geology and mineralogy to correctly interpret data for the laboratory client. The example shown in Figure A.4 gives the transmission spectra of quartz, cristobalite, non-crystalline silica and kaolinite.



Key

ṽ wavenumber

τ transmission

1 quartz

2 cristobalite

3 amorphous

4 kaolin

Figure A.4 — FT-IR transmission spectra of quartz, cristobalite, amorphous silica and kaolinite

Annex B
(informative)

The quantification of cristobalite using X-ray diffraction

B.1 Quantification of cristobalite

The quantification of cristobalite is difficult because of the variations of position and intensity of the diffraction maxima of both α- and β-cristobalite. The intensity of the diffraction lines has been shown to vary by a factor of three in some samples. It is possible that some of this variation is due to the two distinctly different forms of cristobalite.

The high temperature β-cristobalite is stable at temperatures above 1 470 °C but undergoes rapid conversion to the α-form when reaching about 200 °C on cooling. Therefore, it is assumed that workplace samples contain the α-form. See Figure B.1. However, studies of devitrified aluminosilicate ceramic insulation material have shown that the β-form can exist. If the β-cristobalite is analysed using the α-cristobalite calibration standard, the result has significant bias.

B.2 Quantification of cristobalite in the presence of opal and amorphous silica

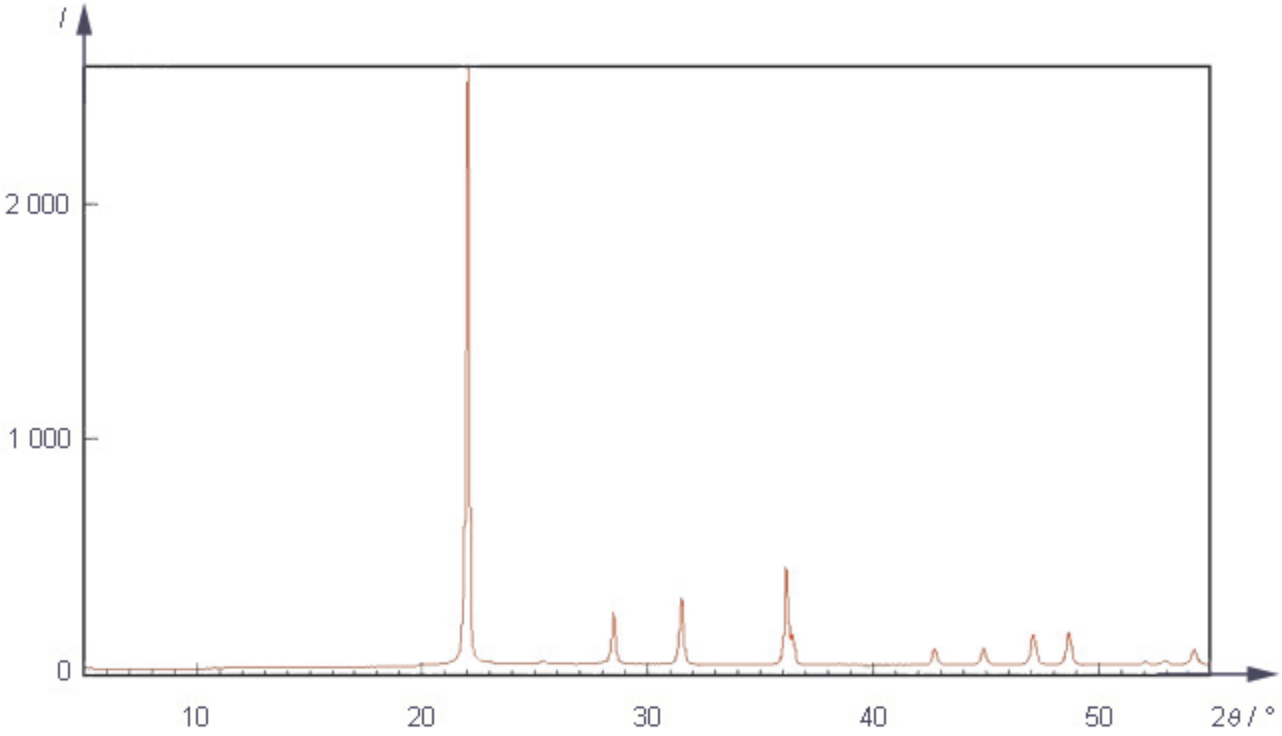
Some cristobalite samples show a diffuse scattering under the cristobalite maxima, either from the diffuse scattering of amorphous material with localised areas of arrangement such as silica glass or from some disordered structure within the crystalline silica. The question for the analyst is where to set the baseline to quantify the crystalline material. This is a subjective judgement by the analyst since, in some samples, the inclusion of some of the broading of the base of the cristobalite peak may increase the area counted by 30 % to 50 %. The essential requirement for occupational hygiene is the measurement of the crystalline component, since this is thought to cause the most serious health problems. Therefore it is reasonable to quantify the sharp crystalline maximum since this represents the most crystalline material. Profile fitting or Rietvelt analysis may be a useful software tool to separate the crystalline from the amorphous or poorly crystalline material.

Samples of opal show a full range of crystalline behaviour from highly ordered cristobalite (opal-C and some samples of opal CT) through to a broad amorphous peak in the region of the α-cristobalite maxima (opal-A).

B.3 Quantification of cristobalite in the presence of tridymite

Some samples of cristobalite show a broadening of the diffraction maxima and occassionally tridymite stacking is also observed with the cristobalite peak. In a sample with a mixture of cristobalite and tridymite, the tridymite should also be quantified or reported as present, since it is a crystalline silica. Several of the tridymite peaks coincide with the principal cristobalite peak and the reported result needs careful interpretation. In the absence of a calibration for tridymite, best practice is to report the value obtained for cristobalite and note it as a minimum value due to the presence of trydmite.

Figure B.2 shows an XRD scan of crystalline tridymite, while Figure B.3 depicts the combined XRD scans of cristobalite, quartz, and tridymite.

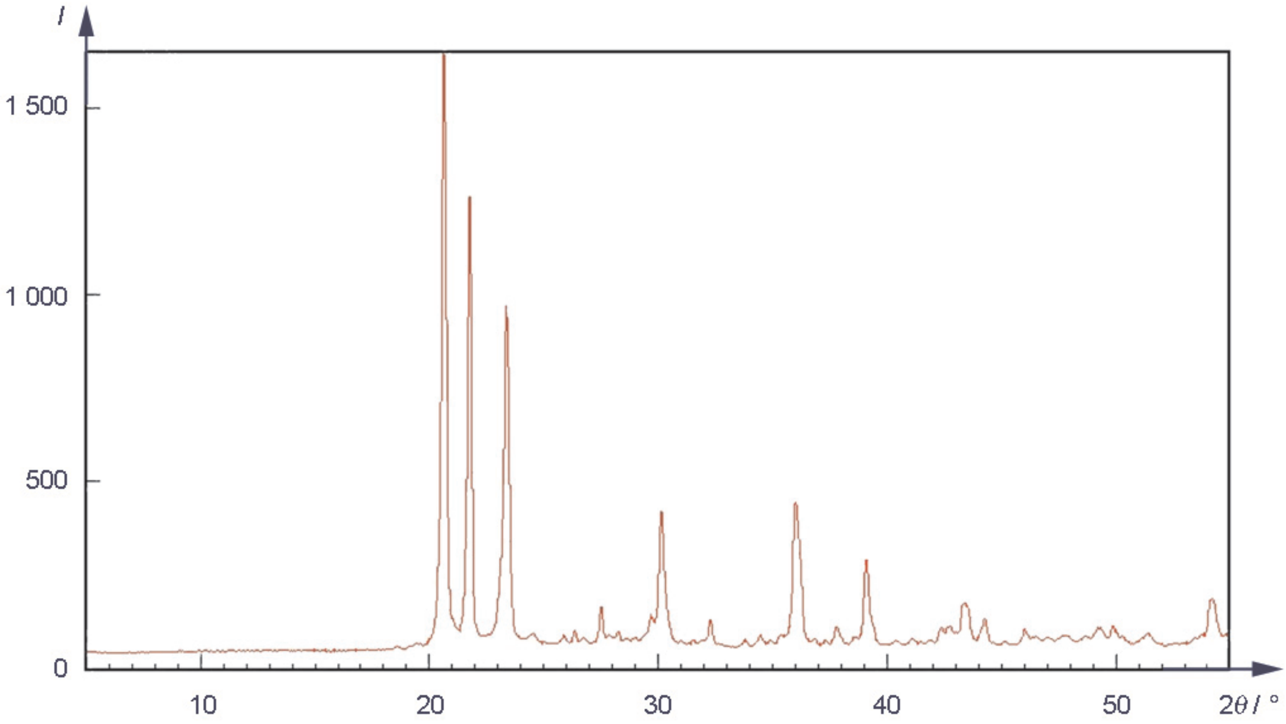


Key

2θ peak position

I intensity/counts

Figure B.1 — An X-ray diffraction scan of crystalline α-cristobalite

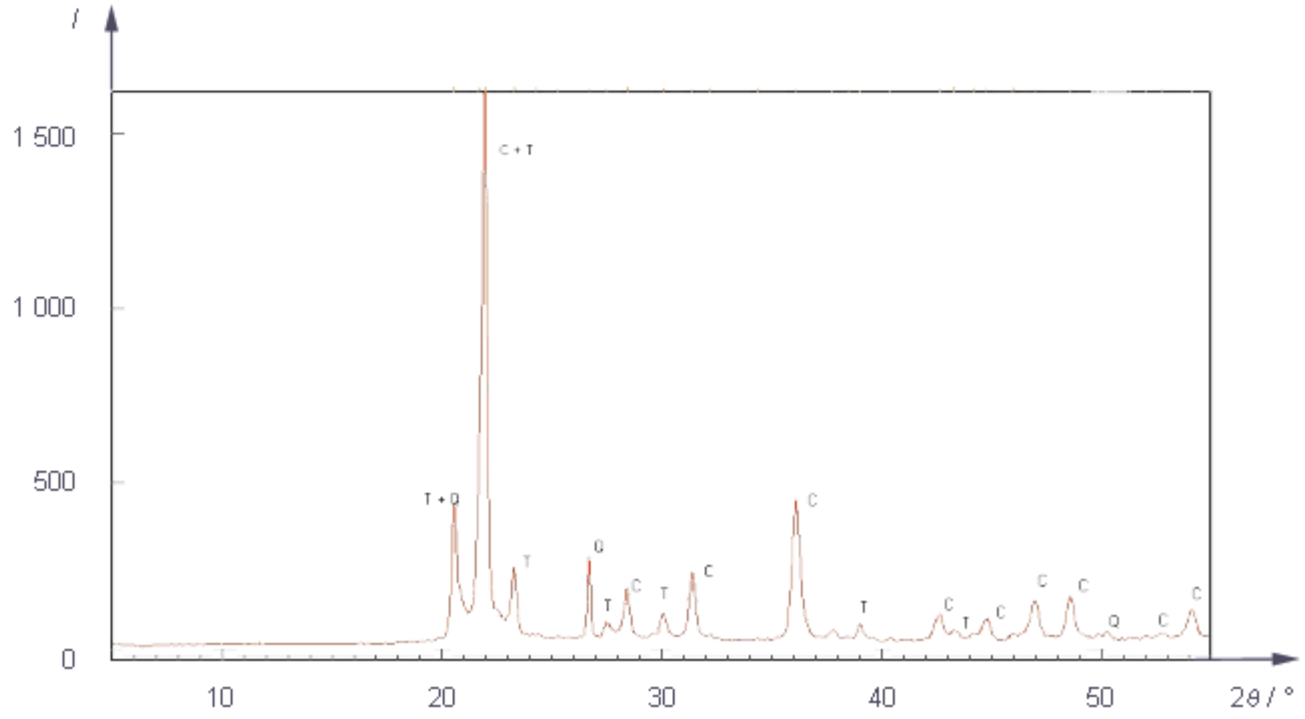


Key

2θ peak position

I intensity/counts

Figure B.2 — An X-ray diffraction scan of crystalline tridymite



Key

2θ peak position
I intensity/counts
C cristobalite
Q quartz
T tridymite

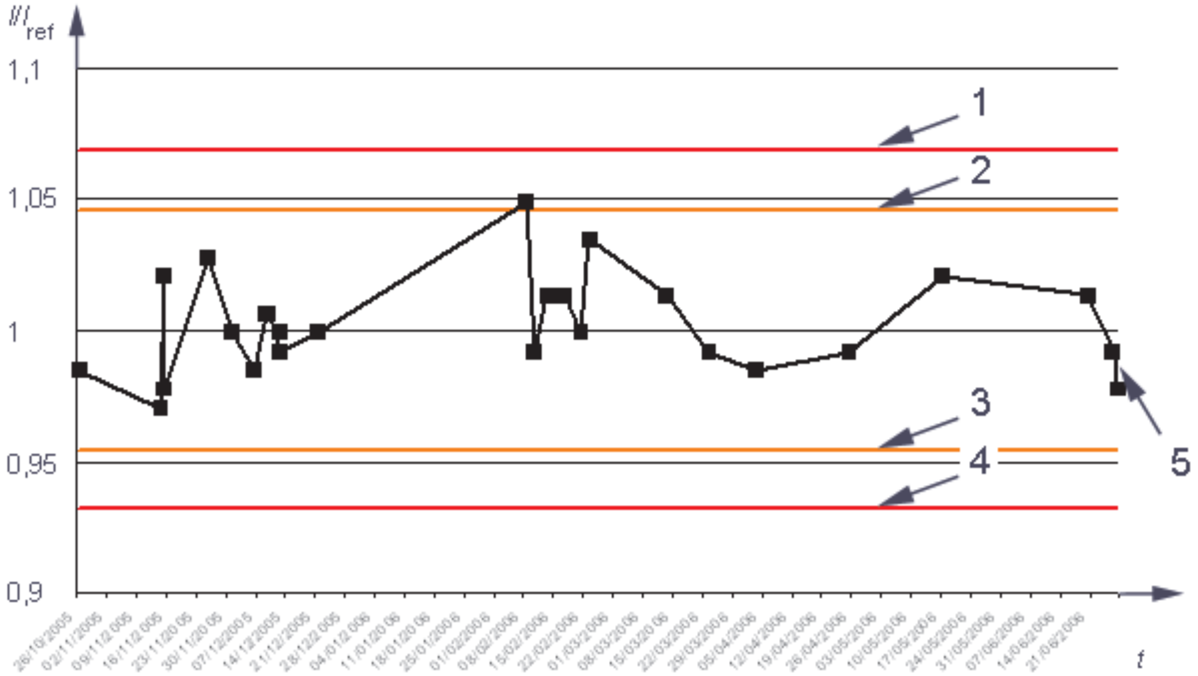
NOTE The cristobalite in this example came from an industrial source and contained a trace amount of quartz.

Figure B.3 — The combined X-ray diffraction scans of cristobalite, quartz and tridymite

Annex C
(informative)

Example of quality control charting for respirable crystalline silica

It is useful to record or plot the result obtained by each individual X-ray reflection or IR absorbance used in analysis because differences in background or peak position when analysing the same sample may produce an unexpected result on one of the reflections/absorbances and bias an average value. The example in Figure C.1 shows the variation of the measurements of the primary XRD 26,6° 2θ peak on a sample containing about 100 µg of α-quartz. Similar plots can be created for IR analysis. The ratio between the results obtained and the value of the quality control material is plotted against the date of analysis. The value of the quality control material can be a theoretical or known value determined from the mass loaded on to a filter or an assigned or participant value from a proficiency-testing sample. If these values are used, the ratio of the result obtained with the assigned value may also provide an indication of potential bias. The ratio rather than absolute difference from the quality control value is often plotted so that samples with slightly different values can be included on the same chart. When using XRD, membrane or PVC filters become dry and brittle after repeated measurement. A slight trend towards larger values is not unexpected when measuring RCS with X-rays on these types of filter materials.



Key

t	date of analysis
I/I_{ref}	ratio of result and target value
1	upper action limit
2	upper control limit
3	lower control limit
4	lower action limit
5	quality control ratio value

Figure C.1 — A quality control chart for X-ray diffraction measurement

Annex D
(informative)

Estimation of expanded uncertainty for measurements of respirable crystalline silica

D.1 Uncertainty estimation

Uncertainty is quoted on measurement reports so that occupational health professionals can assess the reliability of a measurement value when interpreting data. If the exposure limit value (LV) is within the stated uncertainty of the result, further measurements may have to be taken to increase confidence that the LV is not exceeded. Knowing the expected uncertainty of the measurement is especially important if LVs are established at masses that approach the LOQ of the measurement methods, since the relative precision of measurements decreases as the amount of analyte measured is reduced. It is possible that very low LVs for RCS will be established in many countries because there is no measurable exposure level where there is no risk of developing silicosis. Performance requirements, such as EN 482 and Chapter E of the *NIOSH manual of analytical methods* (Reference [9]), describe the maximum allowable uncertainty or inaccuracy of occupational hygiene measurement methods in terms of relative error. EN 482 states that the expanded uncertainty of a measurement, including sampling, should be $\leqslant 50$ % in the concentration range 0,1 to 0,499 times the LV and $\leqslant 30$ % in the concentration range 0,5 to 2 times the LV. NIOSH have a method accuracy requirement for laboratory tests of ± 25 %, which may be expanded to ± 35 % for accuracy determined using field comparison of methods. Therefore, it is important that, when measurements are taken to assess compliance with an LV, information about the expanded uncertainty or variability of RCS measurements is available and that the method selected is demonstrated to meet the performance requirements of EN 482 or NIOSH at the appropriate occupational exposure limit. This annex provides guidance about how to calculate the uncertainty of RCS measurements to comply with ISO/IEC Guide 98-3[4] and gives an example of the uncertainty associated with a reported value. Other approaches may still be valid if it can be demonstrated they provide a “realistic” assessment of the variability of results.

D.2 Uncertainty of measurement

This method of calculating the uncertainty requires the analyst to examine the analytical procedure and identify each individual component that may add to the variability of a measurement. Estimates of the precision of each component are then calculated as a standard deviation or relative error and combined. According to ISO/IEC Guide 98-3[4], the n single components of uncertainty of type A and the m components of type B uncertainty can be estimated or calculated and then properly added to obtain a combined uncertainty, $u_{(\mathrm{c})}$. The final expanded uncertainty, U, is calculated at the end, multiplying $u_{(\mathrm{c})}$ by an approximate coverage factor, $k = 2$, if the degrees of freedom are at least 30.

$$u_{(\mathrm{c})A} = \sqrt{\sum_1^n u_{sA}^2 + \sum_1^n u_{aA}^2} \tag{D.1}$$

$$u_{(\mathrm{c})B} = \sqrt{\sum_1^m u_{sB}^2 + \sum_1^m u_{aB}^2} \tag{D.2}$$

$$u_{(c)} = \sqrt{u(c)_A^2 + u(c)_B^2} \qquad U = 2\,u_{(c)} \tag{D.3}$$

where

u_{sA} is the sampling uncertainty of type A;

u_{sB} is the sampling uncertainty of type B;

u_{aA} is the analysis uncertainty of type A;

u_{aB} is the analysis uncertainty of type B.

The single components of the uncertainty of sampling can mainly be those linked to:

a) volume of sampled air;

b) efficiency of the sampler;

c) transportation and storage of samples.

The single components of the uncertainty of analysis can mainly be those linked to:

1) analytical variation;

2) correction for the recovery or bias of the method;

3) instrumental drift.

Clauses D.3 and D.4 give guidelines on how laboratories can quantify each of these components.

D.3 Sampling

The analyst can only assess the variation of the measurement itself rather than the sampling environment. The precise characterisation of the sampling environment is the responsibility of the individual supervising the collection of the sample, who is not always the same person that measures the sample. If the purpose of the uncertainty estimate is to assess if the method complies with EN 482, then it should include the estimate of the contribution from the sampling procedure directly influencing the measurement. Many of these sampling components are not easily estimated within a laboratory and it is common practice, in these circumstances, to include a value taken from published standards (see guidelines in EN 482).

The sampling components and typical values associated with the sampling process and how they are determined are shown in Table D.1.

The dust on the filter material should not suffer any degradation or loss if it is stored under normal laboratory conditions. Sample loss occurs if pressure is applied against the sample surface or if the sample is too heavily loaded ($>$ 6 mg for 25 mm diameter filters). At most normal loadings ($<$ 4 mg), the respirable dust is safely contained on the filter and is not lost if the sample is handled with care. The filter material degrades if the sampling environment is particularly humid. In most circumstances, transportation and storage can be ignored for crystalline silica measurement uncertainty estimation.

D.4 Analysis

The processes in D.4.1 to D.4.3 are associated with the analysis. A summary of typical values for each process is shown in Table D.2.

Table D.1 — Summary of uncertainty contributions and typical values for RCS sampling

Variable	Determination method	Typical values
Master flow meter calibration (bubble flow meter)	Test certificate	0,6 %
Pump rate reading	10 repeat measurements	1,9 %
Sampling time	Estimation from EN 482 assuming 4 h sampling period	0,24 %
Sampling instrument		
Calibration of sampler test system	Listed as a typical value in EN 13205	1 % to 2 %
Estimation of sampled concentration	Listed as a typical value in EN 13205	1 % to 2 %
Bias relative to sampling convention	Listed as a typical value in EN 13205	5 % to 10 % for well optimised samplers 10 % to 25 % otherwise
Individual sampler variability	Listed as a typical value in EN 13205	3 % to 7 %
Excursion from nominal flow rate	Listed as a typical value in EN 13205	2 % to 5 % if the concentration is calculated from the nominal flow rate 5 % to 9 % if the concentration is calculated from the average flow rate
Sampling uncertainty taking mid range values listed in EN 13205		11,8 % from average flow rate 10,1 % from nominal flow rate

NOTE Some countries recommend using the nominal flow rate for cyclones or impactors as the sampling instrument is able to self-compensate in terms of the mass, because the separation efficiency is dependent on the pump performance.

Table D.2 — Summary of uncertainty contributions and typical values for RCS analysis

Variable	Determination method	Typical values[a] Method A	Typical values[a] Method B
Certified standard	From certification	1,4 % to 0,4 %	1,4 % to 0,4 %
Analytical precision	Within laboratory reproducibility	9 %	
Instrumental precision	Repeat measurements		5 %
Calibration	Residual error		7 %
Recovery/method bias	Recovery tests/proficiency test results	See D.4.2	See D.4.2
Instrument drift	Variation of differences	2 %	2 %
Analysis uncertainty		9 %	9 %

[a] For a 100 µg sample

Method A — Uncertainty estimation from an intermediate reproducibility exercise where the test samples are included in the whole analytical procedure including recovery

Method B — Uncertainty estimation from a repeatability exercise where calibration is traceable to primary standards.

D.4.1 Analytical precision

D.4.1.1 Method A — Determining analytical variation from intermediate reproducibility

This component comprises both the instrumental and calibration error and can be determined from a within-laboratory exercise; measuring replicate samples at different measurement levels across the analysis range on different days and calculating the reproducibility using ANOVA. The relationship between reproducibility and the mass measured should be characterised across the analytical range and the equation used in the calculation of the reported uncertainty since the precision is not expected to be constant at all measurement levels. It is difficult to accurately produce replicate samples using aerosol deposition methods and, in these circumstances, laboratories may choose to calculate the uncertainty from repeatability and calibration data (Method B) or use within-laboratory precision results from interlaboratory comparisons (subject to certain considerations). This procedure can be used when laboratories are using methods that are able to produce replicate test samples. Alternatively those laboratories using direct on-filter methods may prepare pairs of test samples with similar loadings, normalise the results obtained for each pair with their average and calculate the reproducibility of the normalised results.

D.4.1.2 Method B — Determining analytical precision from repeatability data

The components described in D.4.1.2.1 to D.4.1.2.3 should be considered for RCS analysis.

D.4.1.2.1 Instrumental precision

This precision is derived by the repeated measurement of samples loaded with different masses of crystalline silica within the expected analysis range. The relationship between precision and mass measured should be characterised and used in the calculation of the reported uncertainty. In XRD analysis, instrumental precision may vary depending on the diffraction peak or peaks selected for reporting and this should be taken into consideration.

D.4.1.2.2 Calibration precision

The uncertainty of weighing and volumetric procedures involved in making the calibration test samples are small when using methods where the test samples are prepared by pipetting known aliquots of suspension, such as NIOSH Method 7500[8]. However, in a method where the test samples are prepared by the repeated weighing of filters, the error can be as much as ± 6 % in a well-controlled environment. If weighing is used to prepare the calibration test samples, then this factor should be carefully controlled because it may become an important contribution to the uncertainty of the calibration line.

The uncertainty of the calibration line is determined from the square root of the sum of squares of the residual errors. This is the sum of the differences in the value of the mass on the standard compared with the value calculated from the intensity obtained when measuring the standard and calibration coefficients. The uncertainty of the calibration line, u_c, is given by Equation (D.4) (see Eurachem/CITAC Guide CG 4[13]).

$$u_\text{c} = \frac{s_\text{c, res err}}{b_\text{c}} \sqrt{\frac{1}{n_\text{p}} + \frac{1}{n_\text{c}} + \frac{\left(m_\text{obs} - \bar{m}\right)^2}{\sum\left(m_i - \bar{m}\right)^2}} \tag{D.4}$$

where

b_c is the calibration slope co-efficient;

$s_\text{c,res err}$ is the standard deviation of the residual error of the calibration line;

n_c is the number of calibration standards;

n_p is the number of measurements;

m_obs is the observed value;

$\bar{m}$ is the average mass of the calibration standards;

m_i is the mass of calibration standard i.

where

$$s_{\text{c, res err}} = \sqrt{\frac{\sum\left(I_{\text{pred}} - I_i\right)^2}{\left(n_{\text{c}} - 2\right)}} \tag{D.5}$$

in which

I_i is the intensity of calibration standard i;

I_{pred} is the observed value of the intensity.

The root mean square (RMS), $s_{\text{c,res err}}$, should be calculated taking into account the degrees of freedom ($n_{\text{c}} - 2$).

The term

$$\frac{\left(m_{\text{pred}} - \bar{m}\right)^2}{\sum\left(m_i - \bar{m}\right)^2} \tag{D.6}$$

allows for an increase in uncertainty at the extreme masses of the calibration and is almost zero in the centre of the calibration range.

The reported masses of most measurements of RCS are at the lower end of the calibration range so the formula should not be simplified. The value obtained from the calculation is in terms of mass and has to be compared with the predicted mass to obtain a relative value to use in the expanded uncertainty calculation. If the amount of analyte on the calibration test samples is traceable to a primary standard, the residual error also includes an estimate of method bias.

Table D.3 is an example of the XRD calibration line of the primary quartz peak at 26,6°.

In the example given in Table D.3, the uncertainty in the calibration line is small in terms of micrograms but in terms of relative error it makes an impact on the expanded uncertainty when measuring masses of 25 µg or less (see Table D.4).

D.4.2 Method bias

D.4.2.1 Calibration dust standard

Either the calibration or the reported results should be corrected for the known crystallinity of the dust standard used for calibration to prevent potential bias. The certified value for crystallinity has a known variability that can be included in the uncertainty estimation. However, the variability of the certified value for α-quartz is likely to be very small (0,4 % to 1,4 %) and does not influence the expanded estimate.

D.4.2.2 Recovery

This is a major source of potential bias. A separate estimate for this is not needed if the samples used in the intermediate precision exercise are involved in the whole analytical process, otherwise it should be determined separately by measuring the mass recovered from loaded filters included in the sample preparation process. It is important to gain an estimate of the loss of analyte at the LV and half the LV for full and half-shift sampling. The reader should be aware that some procedures might not permit half-shift sampling.

D.4.2.3 Certified reference material

The method bias can be determined through the use of a suitable CRM or by participation in proficiency testing programmes. The availability of a suitable CRM or laboratory reference material on filters is very limited. If a CRM is used the reported uncertainty of the method bias u_{mb} should be a combination of the uncertainty of the assigned value and the variation of the error of the measurement results compared with the assigned value of the CRM.

$$u_{\text{mb}} = \sqrt{u_{\text{CRM}}^2 + s_m^2} \tag{D.7}$$

where

u_{CRM} is the uncertainty of the CRM taken from the certificate;

s_m is the standard deviation of measurements on the certified reference value.

Table D.3 — Example for an X-ray diffraction calibration

Calibration points $-m_i$ µg	$(m_i - \bar{m})$	$(m_i - \bar{m})^2$	**Intensity** I_i counts/s	I_{pred} **from calibration formula**	**Difference** $I_{\text{pred}} - I_i$	$\left(I_{\text{pred}} - I_i\right)^2$
74	–97,17	9 442,01	8,438	9,336	0,898	0,806
31	–140,17	19 647,63	3,622	3,561	–0,061	0,004
145	–26,17	684,87	7,87	18,871	1,001	1,002
155	–16,17	261,47	20,21	20,214	0,004	0,000
143	–28,17	793,55	19,00	18,603	–0,397	0,158
146	–25,17	633,53	19,49	19,005	–0,485	0,235
347	175,83	30 916,19	44,97	46,000	1,030	1,060
214	42,83	1834,41	28,45	28,138	–0,312	0,097
264	92,83	8 617,41	35,12	34,853	–0,267	0,071
212	40,83	1 667,09	28,64	27,869	–0,771	0,594
173	1,83	3,35	24,31	22,632	–1,679	2,817
150	–21,17	448,17	18,42	19,543	1,123	1,260
	Sum	74 949,67			Sum	8,11
From equation of regression line						
	8,11	Sum of differences squared of $\sum\left(I_{\text{pred}} - I_i\right)^2$				
	0,900	RMS, $s_{\text{c,res err}}$				
	0,134 3	Slope, b, from the equation				
	6,704	$s_{\text{c,res err}}/b$				
	171,17	Average calibration point mass, $\bar{m}$				
	74 949,67	Sum of squares $\sum(m_i - \bar{m})^2$				

Table D.4 — Predicted uncertainties in the RCS analysis due to the XRD calibration

Mass measured m_{pred}	$\left(m_{\text{pred}} - \bar{m}\right)^2$	**Uncertainty** u_{c} µg	**Relative uncertainty** %
25	21 364,69	7,842	31,37
50	14 682,17	7,582	15,16
100	5 065,17	7,192	7,19
150	448,17	6,997	4,66
300	16 597,17	7,658	2,55

D.4.2.4 Proficiency testing schemes

Alternatively, long-term participants in proficiency testing schemes can obtain estimates of method bias from their results. To estimate the uncertainty of the method bias value, the standard deviation of differences of the laboratories' results from the values assigned by the proficiency-testing provider should be calculated. To provide a realistic estimate, loadings nearest the EL or towards the lower third of the analytical range supplied by the proficiency testing provider should be considered in preference to those at the higher end of the analytical range. The variation in laboratories' results should be combined with the uncertainty of the assigned value.

$$u_{\text{mb}} = \sqrt{s_{\text{PT}}^2 + \sigma_m^2} \tag{D.8}$$

where

σ_m is the standard deviation of measurements from the proficiency testing assigned value;

s_{PT} is the uncertainty of the assigned values.

If the uncertainties of the assigned values are not provided by the proficiency testing provider, they can be estimated from the between-laboratory standard deviation and number of participants using Equation (D.9)

$$s_{\text{PT}} = \frac{s_{\text{T}}}{\sqrt{n}} \tag{D.9}$$

where

s_{T} is the average between-laboratory standard deviation of participants results obtained from literature or from proficiency testing reports;

n is the average number of participants involved in the rounds.

However, in RCS analysis, a between-laboratory standard deviation of about 15 % can reasonably be achieved at about 50 µg. If 15 laboratories are involved in the scheme, σ_m would have to be about 5 % or less before s_{PT} would contribute more than 1 % to the method bias uncertainty.

D.4.3 Instrumental drift

Instrumental drift is calculated from the variation of the differences of the value of a monitor measured before and after the analyses of samples. For IR instruments, the calibration is not expected to change during the analysis of a typical set of samples and for most XRD instruments the change in monitor is often 2 % or less. However, the instrumental drift can be poorer on older types of instrument, when the IR calibration has not been checked for some time or when the intensity of the X-ray tube is substantially reduced from its initial value.

D.5 Uncertainty estimated from the Horwitz equation

The Horwitz equation for between-laboratory precision data is proposed as a method to estimate the uncertainty RCS measurements may achieve. The Horwitz equation is an empirical expectation for the median spread of results based on data obtained from a wide range of collaborative testing programmes in the Association of Official Analytical Chemists in the USA. Collaborative testing and proficiency testing may differ in the precision achieved because proficiency testing often permits different methodologies. The Horwitz equation for the coefficient of variation (CV), C_V, as a percentage, in this International Standard is expressed as

$$C_V = A\, m^B \tag{D.10}$$

where

A, B are constants;

m is the concentration level, expressed as the mass of the analyte sampled.

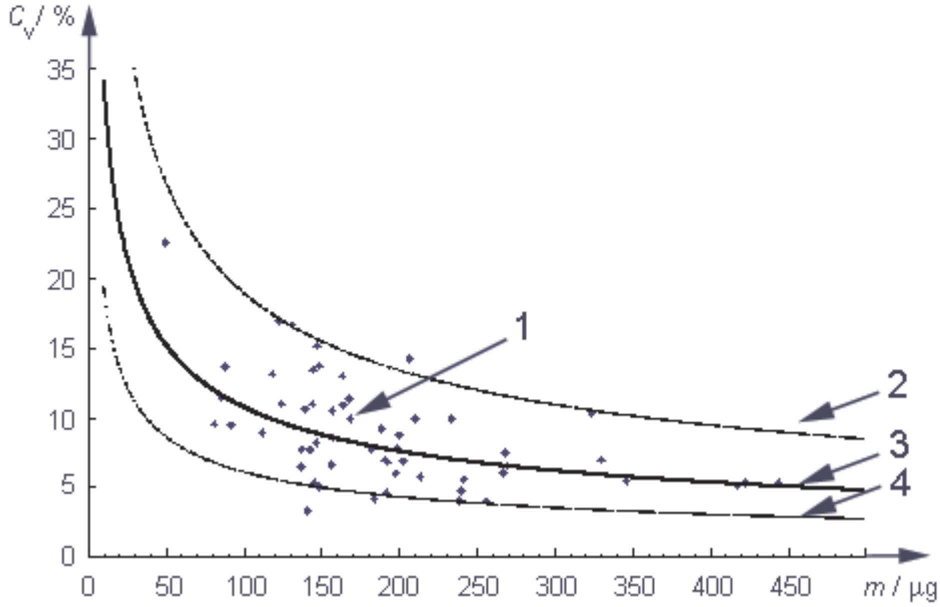
Reference [20] suggests the least-squares fitting of a linearised form of the Horwitz equation as a useful tool to explore analytical systems. The Horwitz equation is linearised by taking the logarithm of both sides.

$$\log C_V = B \log m + \log A \tag{D.11}$$

The linearised equation has the added convenience of allowing the use of the analytical unit of measurement rather than the mass fraction required by the standard approach using the Horwitz equation; if the constants in the equation are consistent with the analytical unit of measure used to derive them, the equation returns the median CV value. When the constants A and B are fitted to data from the United Kingdom proficiency testing programme the Workplace Analysis Scheme for Proficiency (WASP) (WASP rounds 36 to 49, 70) the resulting empirical Horwitz equation is:

$$C_V = 109{,}01\, m^{-0{,}503\,6} \tag{D.12}$$

where m is the mass, in micrograms. See Figure D.1.



Key

C_V coefficient of variation $C_V = 109{,}01\ m^{-0{,}503\,6}$

m mass

1 interlaboratory standard deviation of results from WASP participants using direct on-filter analysis methods

2 upper 97,5 % confidence level

3 linearised Horwitz curve

4 lower 97,5 % confidence level

Figure D.1 — Coefficient of variation, as a percentage, predicted using the coefficients obtained from the linearised Horwitz equation for results from laboratories using direct on-filter analysis methods

The two-sided 90 % confidence interval is calculated based on the standard error in the estimate (SEE), $(s/\sqrt{n_p})_{\log m,\ \log C_V}$ obtained from the linearised equation. For these data, $(s/\sqrt{n_p})_{\log m,\ \log C_V} = 0{,}150\,2$

$$C_V = 109{,}01(m^{-0{,}503\,6}) \times 10^{1{,}645(0{,}150\,2)} \quad \text{upper 5 \% boundary} \tag{D.13}$$

$$C_V = 136{,}34(m^{-0{,}503\,6})/10^{1{,}645(0{,}150\,2)} \quad \text{lower 5 \% boundary} \tag{D.14}$$

The empirical Horwitz equation fits data from WASP quite well, but the extrapolated curve can be sensitive to outliers at high and low masses and Horwitz-like equations are known to easily overestimate the CV at low concentrations. However, the derived empirical equation is similar to the Poisson counting error formula, familiar to XRD measurements, where the counting error is the primary contributor to the analytical error

$$C_V = 100\ K\ m^{-0{,}5} \tag{D.15}$$

where K is a constant that includes the statistical sampling of the calibration slopes of the laboratories involved in the study. The close similarity to the Poisson error prediction indicates that the equation fitted to the WASP data may be useful within the analytical range where the background noise is a minor consideration. Other work has observed that about 120 ppb (ng/g) the measured CV of results tends to fall in the range 20 % to 25 % even though the Horwitz equation may predict higher values. Within-laboratory CV is often one-half to two-thirds the between-laboratory precision measured in collaborative tests. Therefore, when the between-laboratory CVs are close to about 22 % (about 25 µg based on the fitted Horwitz-like equation) the within-laboratory CVs are approaching their LOQ.

D.6 Proficiency test results

Proficiency testing results can contribute to the estimation of uncertainty and can also be used to assess if the uncertainty estimates obtained are valid. There are however several considerations.

a) The lowest mass in the analytical range of a proficiency-testing sample is often higher than the masses measured on routine samples.

b) Extrapolation of the standard deviation of measurements below the lowest proficiency test sample mass is not recommended unless additional precision tests can verify the predicted relationship.

D.7 Reporting

It is best practice to report the expanded uncertainty with the analysis result in terms of mass. If the sampling volume is known this can be reported in terms of mg/m^3. For RCS analysis, many laboratories may not have control over the calibration of the sampling apparatus used to collect the samples, so it may not be appropriate to report a value combining the uncertainties from sampling and analysis in these circumstances. When appropriate, the uncertainty statement should indicate whether an estimate has been included for the uncertainty associated with the sampling apparatus or give an indication of the magnitude of this uncertainty separately. If laboratories have used the same procedure detailed in this International Standard they may wish to indicate that many of the estimates for the sampling instrument were obtained from international standards.

In RCS measurements, the uncertainty is usually determined by taking measurements on filter samples with a pure crystalline silica dust standard. In samples with significant interferences, the uncertainty reported is smaller than the true range of analysis results. Possible interferences should be noted in the report and if appropriate, the laboratory should add a statement about how the reported uncertainty is affected.

The reported results should be corrected for the percentage crystalline silica in the powder used to prepare the calibration test samples.

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