International Standard

ISO 6868

Workplace air — Quantitative determination of quartz and cristobalite in bulk materials by X-ray powder diffraction methods

Air des lieux de travail — Détermination quantitative du quartz et de la cristobalite dans les matériaux en vrac par des méthodes de diffraction des rayons X sur poudre

First edition
2026-08

Reference number
ISO 6868:2026(en)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

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Foreword

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Exposure to crystalline silica (CS) in the respirable fraction of inhaled airborne dust is a hazard to the health of workers in many industries. For this document, CS refers to the two most common polymorphs, quartz and cristobalite. To assess the exposure of an individual, personal samples of air are taken during a work activity, and then the amount of respirable CS is measured. In addition to the procedures for determining the airborne concentration of respirable CS in a workplace atmosphere, it can be useful, or required, to evaluate the CS content in bulk materials that have a potential risk of being respirable when they are ground or manipulated.

This document is appropriate for the analysis of material selected to be representative of a product, or soil, rock, mineral, waste, depending on the nature of the investigation. Bulk materials are generally in the solid state but can also be made by watery mixtures of insoluble matter (slurries, muds). A variety of origins, compositions, sizes and shapes, such as solid blocks, fragments, slabs, sand, powders and flours, can represent bulk materials.

The knowledge of CS content in natural rocks and soils is useful when, for example, assessing potential exposure by inhalation of airborne dust meeting the respirable convention of ISO 7708[1] in work activities that involve excavation, earth moving, and drilling plant operations. Elevated exposures to respirable CS are often found in confined spaces, such as in mining and tunnelling. Knowing the mass fraction of CS in construction materials and natural rocks or soils contributes to the characterization of potential sources of airborne dust and allows planning of risk management measures in order to reduce potential exposures to airborne dust.

The methods in this document can also be used for the analysis of samples, which have been collected for the purpose of classification and labelling of chemicals according to the Globally Harmonized System (GHS), when a CS phase is found as a “substance”, or in “mixtures” and “articles”, as defined in the GHS.

As is necessary in any good routine type analysis, especially where the results can have legal implications, it is useful to establish standard procedures for analysing the specimens. There are several methods mentioned in the literature for the determination of quartz, and many procedures can be developed, but no single procedure is applicable to all situations. Standardizing procedures for X-ray powder diffraction (XRPD) analysis of bulk materials, which are applicable to all materials, is difficult because of the variability of the matrices and their potential interferences. Therefore, the method chosen for the analysis should be carefully tested in the laboratory for the specific conditions.

The problems of processing samples containing either quartz or cristobalite, or both is essentially the same as processing samples of bulk materials in general, and the major problem is preparing a specimen that is representative of the bulk material. The potential accuracy obtainable from a sub-sample or specimen of bulk material is related to the effective number of particles interrogated by the X-ray beam, which is related to the distribution of particles sizes and their absorption coefficients.

Workplace air — Quantitative determination of quartz and cristobalite in bulk materials by X-ray powder diffraction methods

1 Scope

This document specifies three methods for quantitative measurement of crystalline silica (CS) major polymorphs (quartz and cristobalite) mass percentage content in bulk samples using X-ray powder diffraction (XRPD). This document also provides general information about the capabilities and limitations of relevance to laboratories working for routine testing.

Only X-ray diffractometers with Bragg-Brentano geometry are considered. XRPD techniques are used to characterize specimens in the form of loose powders, where the median grain size is between 1 µm and 10 µm physical diameter. Block specimens are not considered.

Although a number of methods of analysis are considered in this document, other XRPD methods of analysis can be considered if they are demonstrated to give equivalent results.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 14488, *Particulate materials — Sampling and sample splitting for the determination of particulate properties*

ISO 18158, *Workplace air — Terminology*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18158 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
block
self-adhering single piece of interconnected particles or crystallites

[SOURCE: EN 13925-1:2003, 3.2[2]]

3.2
specimen
portion of the sample in the specific form used in the diffraction instrument for a given data collection process

[SOURCE: EN 13925-1:2003, 3.3[2]]

3.3
phase
crystallographic phase
thermodynamic phase
portion of a physical system sharing a common molecular and inter-molecular structure irrespective of any subdivision by size distribution or shape

[SOURCE: EN 13925-1:2003, 3.4[2]]

3.4
diffraction line
peak
reflection
Bragg reflection
region of the diffraction pattern containing an intensity maximum and corresponding to diffraction from a set of lattice planes

[SOURCE: EN 13925-1:2003, 3.5[2]]

4 Principle

The experimental technique used in this document to determine the diffraction pattern of a specimen is the "angular dispersive technique", where the X-rays are monochromatic and the measurement is made by scanning the diffraction angle. Diffractometers with Bragg-Brentano geometry working in reflection geometry are to be used with this document; transmission geometry is not considered.

Two types of powder specimens are considered in relation to their thickness:

— specimens of "infinite thickness";

— "thin layer" specimens deposited on filters.

NOTE "Infinite thickness" and "thin layer" are defined by the preparation steps in this document.

Phase identification is a preliminary step in every quantitative phase analysis and provides information necessary to make a decision regarding the selection of procedure for quantitative analysis. The identification of both quartz and cristobalite in an unknown specimen by XRPD is based on visual or computer-assisted comparisons of portions of its X-ray powder pattern that should include at least two main peaks, compared to the experimental patterns of quartz and cristobalite reference materials.

In quantitative phase analysis, the percentages by mass of quartz and cristobalite are determined based on the integrated intensities of one or more individual diffraction lines of each phase. These intensities are compared to the corresponding values from calibration specimens. Three methods are considered:

— external standard method, for thin layer specimens deposited on filters (comparison of the response obtained with the specimen to the response in a calibration line for quartz or cristobalite reference material, see 8.4.4);

— internal standard method, for specimens of infinite thickness (addition of a known amount of a crystalline phase material that has been shown not present in the original sample, see 8.4.5);

— spiking method, also known as standard addition method, for specimens of infinite thickness and quartz contents below approximately 20 %, depending on the matrix (addition of a known amount of quartz or cristobalite to the sample being studied, see 8.4.6).

Multiple methods applied to a single sample can be used to verify analyses. Whole powder pattern analysis and standard free methods (e.g. the Rietveld method) are not considered in this document.

5 Equipment

5.1 Diffraction system

5.1.1 Diffractometer

This document has been prepared to be used with a diffractometer employing the Bragg-Brentano parafocusing geometry, where the specimen and the receiving slit all lay on the focusing circle. The vertical θ:θ or θ:2θ configurations are most advantageous for handling powder specimens. A diffractometer generally comprises:

— goniometer;

— X-ray source that includes a line-voltage supply, a high-voltage generator and a X-ray tube; a conventional X-ray sealed tube source is often used and the most commonly used tube anode material is copper;

— incident beam optics, that can include monochromatisation or filtering, collimation and focusing or parallelism of the beam;

— specimen stage; the specimen is contained in a specimen holder, which is constructed to allow specimen rotation around the appropriate axis;

— diffracted beam optics, that can include monochromatisation or filtering, collimation and focusing or parallelism of the beam;

— detector;

— data collection system.

Procedures for instrument alignment and performance characterisation of Bragg-Brentano diffractometers are given in EN 13925-3[3].

5.1.2 Specimen holders

Several types of specimen holders can be used for diffraction analysis. The following standard circular specimen holders are recommended when the analytical methods described in this document are used.

— Specimen holder for loose powder with cavity mount; top, back or side loaded. For back-loading of powder, a specimen holder consists of a bottom plate, which supports the powder, and a ring. The diameter of the cavity to be filled generally varies between 15 mm and 30 mm, and the powder thickness is a minimum of 1 mm (for infinite thickness specimen).

— Specimen holder for 25-mm filter (for thin layer specimen), where the support base of the filter, or the filter itself, is a disc made of a metal (e.g. silver, zinc or aluminium).

5.2 Balance

A microbalance capable of weighing ±1 µg or better over the range 0 g to 5 g is required for the preparation of specimens deposited on 25-mm filters (thin layer). For the preparation of specimens of infinite thickness, by mixing of powders, the materials should be weighed in glass weighing bottles with lids; capacities of 5 ml to 15 ml are generally required, so that the total mass exceeds 5 g, therefore, the balance operational range should span up to 20 g, and an analytical sensitivity of 10 µg is sufficient. An electrostatic eliminator is needed when weighing.

5.3 Reference materials

5.3.1 Quartz and cristobalite

Pure quartz and cristobalite reference materials with a median size of particles in the range 1 µm to 10 µm, preferably smaller than 5 µm, and similar to the median size of particles in the specimens, are to be used.

Reference materials can be prepared in the laboratory from pure quartz and cristobalite crystals, if the correct particle size is achieved and phase purity is characterized.

NOTE United States National Institute of Science and Technology (NIST) have developed Standard Reference Materials (SRM's) for respirable quartz (1878 series) and respirable cristobalite (1879 series) with certified phase purity.[1)]

5.3.2 Internal standard

For the internal standard method, the chosen mineral phase should have a fully resolved diffraction line, not overlapping any lines of the CS phases, and free from microabsorption (i.e. be of very small particle size or have an absorption similar to the specimen) and extinction. Hence, it is essential that internal standard powder crystallite size be in the range 1 µm to 10 µm, preferably smaller than 5 µm, and similar to the median size of particles in the specimens. Internal standard, quartz and cristobalite reference materials are generally prepared in the laboratory from pure crystals milled until the optimum size range is reached. A sufficient amount of each reference material should be prepared, tested, stored and added to the specimens for analyses.

NOTE 1 The definition of "fully resolved" line is beyond the scope of this document. The experience of the analyst can guide in assessing and addressing this aspect of the analysis.

NOTE 2 Grain size can be measured by using one of the techniques described in 7.3 NOTE 4, and purity characterized by XRPD.

NOTE 3 Corundum is a mineral phase often used as an internal standard. NIST SRM 676a is a corundum structure-alumina powder intended primarily for use as an internal standard for quantitative determinations by XRPD.[2)]

5.3.3 XRD drift correction disc

An aluminium or silicon polycrystalline disc or another suitable robust material, should be used to correct for the drift in XRD radiation intensity over time.

NOTE The sintered alumina disc NIST SRM 1976b is frequently used.[3)]

5.4 Equipment for the preparation of specimens of infinite thickness

5.4.1 Laboratory drying oven

For ovens of the forced-draft type, the air circulation should not be so strong that any transport of particles can take place.

5.4.2 Masonry hammer, jaw crusher or similar device

Initial processing is often needed to reduce the material to the required size for milling. Fractionation (e.g. use of a masonry hammer or jaw crusher) of the material into smaller pieces can be performed dry, although processing involving a grinding rather than a crushing action should be performed 'wet' to reduce the potential for a deterioration of crystalline structure.

5.4.3 Laboratory mill

Several types of mechanical grinding devices, driven by a motor, can produce the required grinding action by freely moving milling pieces (i.e. balls and rods) in a cylindrical grinding chamber completely encapsulated.

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1) This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the products named. Equivalent products may be used if they can be shown to lead to the same results.

2) This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

3) This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

The device rotates at a given speed, and size reduction is achieved by impact and shear between the milling pieces, whose effectiveness is improved by vibration. Chamber and milling pieces made of the same material should be used. Hard materials such as tungsten carbide, silicon nitride, zirconium oxide and corundum are recommended, while agate should be avoided because of potential contamination. Grinding chambers can typically have volumes between 80 ml and 500 ml, and the material filling can generally range between 15 % and 50 % of the volume (the instruction of the manufacturer should be followed). A so-called "micronizing" mill can also be used. This mill generally uses corundum cylindrical grinding elements in a small airtight polypropylene jar that handle a sample volume of 4 ml (or 10 g). Wet grinding (water, 2-propanol or cyclohexane) is the preferred grinding method for a micronizing mill.

NOTE The minimum quantity of material that can be ground is an important parameter. For example, if a 250 ml grinding chamber is used, a minimum filling of 30 ml is generally suggested. This minimum volume corresponds to a mass of approximately 40 g for a fine sand sample of average density (2,6 g cm^{-3}). Therefore, if the sample mass is less than 40 g, it cannot be ground in the described chamber.

5.4.4 Other equipment

5.4.4.1 Mortar and pestle for hand grinding and mixing. They are manufactured out of many different materials, but hard mortar types such as boron carbide, are recommended, while agate can introduce contamination and should be avoided.

5.4.4.2 Preparation kit for mounting the powder in the specimen holder. The kit generally comprises a specimen "preparation table" onto which a specimen holder ring is clamped, a powder press block, and a spatula to remove the surplus powder (see 7.6).

5.4.4.3 Stainless steel test sieves used to verify fragments size before grinding them in the laboratory mill; a receiving pan is used to collect the passing material.

NOTE Fragment sizes of less than 10 mm can generally be milled, but a smaller initial size improves the result of grinding. The use of a test sieve with aperture size between 2,0 mm to 2,8 mm is suggested when a ball or rod mill is used. If a "micronizing" mill is used, the sample can be passed through a 0,4 mm aperture sieve prior to grinding.

5.5 Equipment for the preparation of thin thickness specimens

5.5.1 Filters for analysis

Filters shall be of a 25 mm diameter. The filter types generally used for the XRPD analysis of CS, and their advantages and disadvantages, are listed in Table 1. These filter materials generally do not interfere with the measurement of the major reflections of quartz (101), (100), and (112), and cristobalite (101), (200 and 112), and (102). In any case, batches of filters should be tested to detect potential interferences, because impurities can be introduced during the filter manufacturing process. These tests are also useful to verify the background fluctuation levels, since they have an effect on the readability of diffraction peaks, worsening the limit of detection for CS. Silver filters exhibit the least variability and lowest background levels and thus are useful in situations where low limits of detection are required. However, cristobalite combined reflections (200 and 112) can sometimes be difficult to measure because they are located in the tail at the side of silver reflection (100). Silver and mixed esters of cellulose filters are rigid and easy to handle when weighing and loading in the sample holder. PVC and especially polycarbonate filters are flexible and require careful handling. A silver filter used for analysis allows an effective correction for absorption (see 8.4.4.3). When an organic filter with high or medium transparency to X-ray radiation (see Table 1) is used for analysis, an effective correction for absorption can still be carried out by measuring the intensity of a reflection of the underlying metallic (e.g. silver, zinc or aluminium) support base.

Table 1 — Examples of filters for CS analysis

Filter material and pore size[a]	Interfering peak						Background fluctuation index[b]		Mass absorption correction
	Quartz			Cristobalite			Quartz		Filter transparency index[c]
	101	100	112	101	200	102	101	100	
Silver (Ag) 0,45 µm	no	no	no	no	yes	no	1	1	1
Polyvinyl chloride (PVC) 5 µm	no	no	no	no	yes [d]	no	1,5	1	0,72
Mixed esters of cellulose (MCE) 0,8 µm	no	no	no	no	yes [d]	no	2,4	1,7	0,78
Polycarbonate (PC) 0,4 µm	no	no	no	no	yes [d]	no	1,3	2,2	0,98
Polytetrafluoroethylene (PTFE) 0,5 µm	no	yes	no	yes	yes [d]	no	2,5	ND	0,74

[a] The analyst should be aware that for any filter pore size the recovery will possibly not be complete.

[b] Background fluctuation index $I_{BF} = F_{B,filter} / F_{B,Ag}$ where $F_{B,Ag}$ and $F_{B,filter}$ are the standard deviations of XRD intensities measured in the ranges of primary (Qz1) and secondary (Qz2) quartz peak profiles (Cu anode) on a blank silver filter, and on a blank filter of the material under testing placed on a silver filter, respectively. $I_{BF} = 1$ is used as a reference for silver filters.

[c] Filter transparency index $I_{FT} = I_{filter,Ag} / I_{filterAg,Ag}$, where $I_{filterAg,Ag}$ and $I_{filter,Ag}$ are the XRD intensities measured in the range of silver primary peak profile (Cu anode) on a blank silver filter, and on a blank filter of the material under testing placed on a silver filter, respectively. $I_{FT} = 1$ is used as a reference for silver filters.

[d] Interference is due to the silver filter placed under the filter of the material under testing.

5.5.2 Other equipment

5.5.2.1 Fume cupboard, to contain dusts, vapours and gases.

5.5.2.2 Beakers, latex gloves, tongs and powder spatulas.

5.5.2.3 Ultrasonic bath sonicator for breaking up the agglomerated particles of samples contained in beakers.

5.5.2.4 Magnetic stirrer, with thermally insulated top, to ensure homogeneous distribution of particles in the 2-propanol suspensions contained in volumetric flasks.

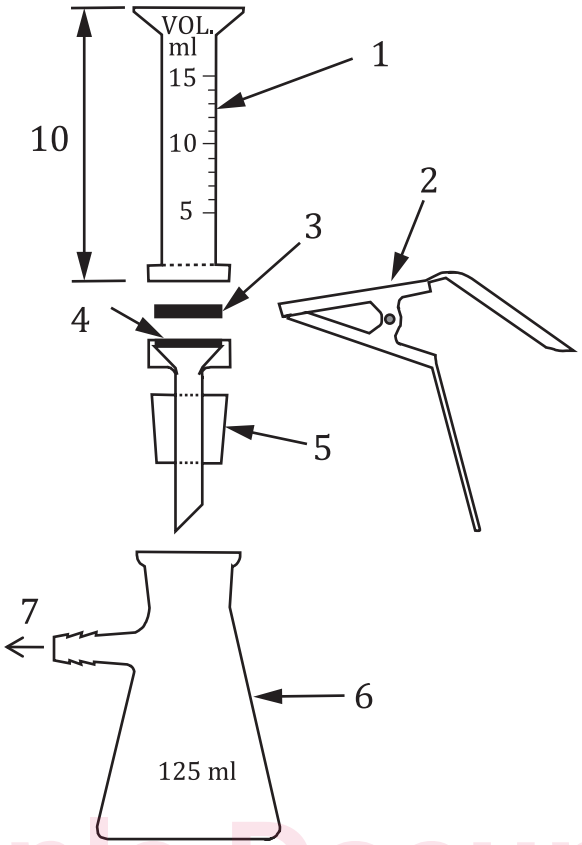
5.5.2.5 Volumetric pipettes, for the desired aliquots.

5.5.2.6 Volumetric flasks, with polyethylene stoppers for the preparation of 2-propanol suspensions of calibration powders.

5.5.2.7 Pump, to generate vacuum and rapidly filter the suspension.

5.5.2.8 Vacuum filtration assembly. A typical assembly with a side arm vacuum flask, with a 25-mm filter holder is shown in Figure 1. When in use, the flask is clamped to a ring stand to prevent it from falling over, and is connected to the pump with vacuum tubing.

Dimensions in centimetres



Key

1	glass funnel
2	spring clamp
3	25-mm filter
4	vacuum base with sintered disc
5	rubber stopper
6	receiver flask
7	to vacuum pump

Figure 1 — Example of a filtration apparatus suitable for powder deposition on filters

5.6 Equipment for sample treatment and reagents

5.6.1 Furnace

A furnace capable of operating up to 1 000 °C can be required to remove interfering substances (see Annex C). The material is placed into platinum or glazed ceramic crucibles with lids.

5.6.2 Reagents

5.6.2.1 Deionised water.

5.6.2.2 2-propanol.

5.6.2.3 2 M HCl,of a volume fraction of 16,4 % (carbonate dissolution).

5.6.2.4 Glacial acetic acid, of a volume fraction of 10 % solution (carbonate dissolution).

6 Sampling

The sample from bulk material submitted for analysis should be as far as possible representative of the whole material. Sampling of particulate materials shall be performed in accordance with ISO 14488. Depending on its size and physical state, this sample shall be stored in an appropriate sample bag or container, and an identification label will be attached allowing proper tracking to the source.

The minimum quantity of bulk material to be collected should be defined before the sampling is performed. Contact the analytical laboratory to determine the quantity needed, based on discussion of the method chosen for the analysis (see NOTE in 5.4.3) and the purpose of the results.

As much information on the sample as possible should be provided to the analytical laboratory.

NOTE For example, it can be indicated how the sample was collected, what silica polymorphs and possible interference it can contain, how the sample was formed, including temperature and pressure if applicable. This information can be based on previous analyses or a knowledge of the sample and process involved. Whether the sample contains natural, unreacted raw materials or whether the sample has been processed (chemically, thermally or otherwise), this information is important as it suggests the phases that will possibly be present (see Annex B).

If the results have legal implications, standard procedures for collecting and processing, the samples can be required, and the desired minimum levels (fraction percentages) for quartz and cristobalite quantitative determinations should be specified to the laboratory.

7 Specimen preparation

7.1 General

The preparation of the specimen is one of the more critical steps in quantitative XRPD analysis, and the success of the analysis largely depends on the correct preparation for the specimen being analysed. In the analytical laboratory, the bulk sample is reduced to a loose powder and a specimen is prepared for analysis. Care must be taken to ensure that the physical state and composition of the prepared specimen be as close as possible to that of the original sample. Steps taken to homogenize and sub-sample the sample further for analysis should follow procedures in ISO 14488 and should be documented.

Although it is desirable to obtain the required information with the least amount of sample treatment, the specimen preparation can involve a number of steps such as:

— crushing a block of material;

— grinding the crushed material;

— treating the powder to remove interferences;

— drying the powder if it is wet;

— depositing the powder on a filter (thin layer specimen) or mounting it in a specimen holder (specimen of infinite thickness).

Two types of specimen preparations are included in this document:

— specimen of “infinite” thickness, mounted in a specimen holder;

— “thin” layer specimen, deposited on a filter.

A specimen is said to have infinite thickness when its thickness is greater than the beam penetration depth, i.e. according to EN 13925-2,[4] when the diffracted intensity is at least 99,9 % of the maximum attainable by

increasing the specimen thickness. This infinite thickness, t_{infinite}, can be calculated (expressed in centimetres) as shown in Formula (1):

$$t_{\text{infinite}} \geq \frac{3,45 \cdot \sin\theta}{\mu' \cdot \rho'} \quad (1)$$

where

ρ' is the specimen density (g cm^{-3});

μ' is the weighted sum of the mass attenuation coefficients (or mass absorption coefficients, cm^2 g^{-1}) of the constituent elements of the material;

θ is the X-ray diffraction angle (°).

The formula is valid for a "solid" specimen and the effective thickness can be obtained by dividing t_{infinite} by the granular volume of the specimen (generally >70 %, porosity <30 %) when minimized by good specimen packing. In practice, the specimen thickness equals the thickness of the specimen holder (see 5.1.2) in which it is mounted, that is always much greater than the beam penetration depth.

NOTE Hence, the effective infinite thickness of a specimen is dependent on porosity and the mass attenuation coefficients of the phases, since absorption affects the beam penetration depth. Figure 2 a) shows the range of possible penetration depths of CuKα at diffraction angles from 20° to 60° 2θ in a wide range of μ' values, for specimens with 30 % porosity. For a specimen made of a pure quartz powder (ρ'= 2,65 g cm^{-3}, μ'= 34 cm^2 g^{-1}), an effective infinite thickness t = 0,012 61 cm is calculated for the diffraction primary peak (θ = 13,33°) if a Cu anode is used.

The deposition of a powder on a filter to produce a "thin" layer specimen, and its analysis by XRPD according to an external standard method, is a procedure derived from the analysis of airborne dusts collected on membrane filters (e.g. see ISO 16258-2[5]). This methodology involves the construction of a calibration curve by measuring the intensity of a diffraction peak from specimens of pure quartz/cristobalite with known masses deposited on filters. The analytical approach assumes that the diffracted intensity is a linear function of the irradiated specimen mass, after a correction for the X-ray absorption (due to the powder load thickness). This correction is calculated by measuring the transmittance of the specimen (see D.1.1.3) and is more accurate when a small increase in powder load produces a great decrease of transmittance, i.e. when the powder load is low. This document considers acceptable a load on the filter that produces a change in the slope of the curve transmittance versus load (mg) lower than 15 %. The field of acceptable loads for specimens within a wide range of mass attenuation coefficients is shown in Figure 2 b), for deposition diameters of 1,5 cm and 2,0 cm.

According to AFNOR NF X 43-295[6], the maximum load of powder on a 25-mm filter achievable without any losses from the filter due to its manipulation should not exceed 10 mg, but this is greater than the recommended maximum mass for quantification [see Figure 2 b)].