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Air quality — Bulk materials —

Part 3:
Quantitative determination of asbestos by X-ray diffraction method

Qualité de l'air — Matériaux solides —

Partie 3: Dosage quantitatif de l'amiante par la méthode de diffraction des rayons X

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Foreword

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

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 146, *Air quality*, Subcommittee SC 3, *Ambient atmospheres*.

ISO 22262 consists of the following parts, under the general title *Air quality — Bulk materials*:

- *Part 1: Sampling and qualitative determination of asbestos in commercial bulk materials*
- *Part 2: Quantitative determination of asbestos by gravimetric and microscopical methods*
- *Part 3: Quantitative determination of asbestos by X-ray diffraction method*

Introduction

In the past, asbestos was used in a wide range of products. Materials containing high proportions of asbestos were used in buildings and in industry for fireproofing, thermal insulation and acoustic insulation. Asbestos was also used to reinforce materials and to improve fracture and bending characteristics. A large proportion of the asbestos produced was used in asbestos-cement products. These include flat sheets, tiles and corrugated sheets for roofing, pipes and open troughs for collection of rainwater and pressure pipes for supply of potable water. Asbestos was also incorporated into products such as decorative coatings and plasters, glues, sealants and resins, floor tiles, gaskets and road paving. In some products, asbestos was incorporated to modify rheological properties, for example in the manufacture of ceiling tile panels and oil drilling muds.

While the asbestos concentration in some products can be very high and in some cases approaches 100 %, in other products the concentrations of asbestos used were significantly lower and often between 1 % and 15 %. In some ceiling tile panels, the concentration of asbestos used was close to 1 %. There are only a few known materials in which the asbestos concentration used was less than 1 %. Some adhesives, sealing compounds and fillers were manufactured in which asbestos concentrations were lower than 1 %. There are no known commercially manufactured materials in which any one of the common asbestos varieties (chrysotile, amosite, crocidolite or anthophyllite) was intentionally added at concentrations lower than 0,1 %.

ISO 22262-1 specifies the procedures for collection of samples and qualitative analysis of asbestos in commercial bulk materials using microscopical methods such as polarized light microscopy (PLM). ISO 22262-2 specifies the procedures for the determination of asbestos mass fractions in bulk materials by microscopical methods.

This part of ISO 22262 specifies the analytical procedures for the quantitative determination of asbestos by X-ray powder diffraction (XRD). The procedure employs a substrate standard mass absorption correction method to quantify asbestos that was previously identified by the microscopical method in ISO 22262-1. While the XRD method is useful for qualitative analysis of crystalline substances in powder samples by measurement of diffraction patterns that can be related to crystal structure, XRD analysis cannot distinguish between different morphological habits of the same mineral. Thus, XRD cannot discriminate between the asbestiform and non-asbestiform analogues of serpentine and the amphiboles. Furthermore, the primary diffraction peaks for different amphiboles lie within a very narrow range and it is not possible to quantify individual amphiboles when a mixture of amphiboles is present. Diffraction peaks appearing in XRD patterns of the asbestos-forming minerals are considered to be “possible peaks of asbestos”, assumed to represent the asbestos detected during analysis in ISO 22262-1. However, if non-asbestiform serpentine or non-asbestiform amphibole minerals are present in the sample matrix, the “possible peaks of asbestos” will represent them. Accordingly, this method is not intended for application to samples in which non-asbestiform serpentine or non-asbestiform amphibole minerals are present.

A conventional XRD method, which employs a powder sample mounted in a powder specimen holder and a scintillation counter, can quantify a crystalline material at a concentration of approximately 1 %. The XRD method using a substrate standard mass absorption correction method employed in this part of ISO 22262 can detect the diffraction peaks of chrysotile asbestos from quantities as low as 0,01 mg on a membrane filter of 2 cm^2 area [0,01 mg/filter (2 cm^2)] as shown in References [13] and [14]. The amount of sample on the filter is limited to 15 mg due to the limit of the X-ray absorption correction. In this method, gravimetric matrix reduction procedures are used to reduce the matrix constituents and interference minerals in a 100 mg comminuted sample. When the matrix reduction achieves a residual ratio of 10 % or lower, the XRD method can provide a limit of detection of 0,01 wt% and the limit of quantification can be as low as 0,03 wt%. When the matrix reduction is less effective and the residual ratio is over 10 % of the initial 100 mg sample, a sub-divided 10 mg to 15 mg sample is taken from the residual sample. In the case where none or very little of the matrix is reduced, the limit of detection can increase up to approximately 0,1 % and the limit of quantification can increase up to approximately 0,3 %. When matrix reduction achieves a residual ratio of approximately 30 % of the original weight, the limit of quantification is approximately 0,1 %. These limits of detection and quantification are further degraded if interference X-ray peaks or high background X-ray intensities from matrix materials are present.

The XRD method specified in this part of ISO 22262 is based on NIOSH 9000-1/7[16], NIOSH 7500-1/10[17], EPA/600/R-93/116[18] and JIS A 1481-3.[19]

Air quality — Bulk materials —

Part 3:
Quantitative determination of asbestos by X-ray diffraction method

1 Scope

This part of ISO 22262 is primarily intended for quantitative analysis of samples in which asbestos has been identified at estimated mass fractions lower than approximately 5 % by weight.

This part of ISO 22262 extends the applicability and limit of detection of quantitative analysis by the use of simple procedures of ashing and/or acid treatment prior to XRD quantification.

This part of ISO 22262 is applicable to the asbestos-containing materials identified in ISO 22262-1. The following are examples of sample matrices:

a) any building materials in which asbestos was detected by the analysis in ISO 22262-1;

b) resilient floor tiles, asphaltic materials, roofing felts and any other materials in which asbestos is embedded in an organic matrix and in which asbestos was detected when using ISO 22262-1;

c) wall and ceiling plasters, with or without aggregate, in which asbestos was detected when using ISO 22262-1.

If non-asbestiform serpentine or non-asbestiform amphibole minerals are included in the matrix, the XRD peaks that are assumed to be “possible peaks of asbestos” will represent these minerals. This method is not for application to natural minerals that may contain asbestos or any products that incorporate such natural minerals. This method is intended only for application to building material samples that contain deliberately added commercial grade asbestos including tremolite asbestos.

This part of ISO 22262 is intended for use by analysts who are familiar with X-ray diffraction methods and the other analytical procedures specified in the References [5] and [6]. It is not the intention of this part of ISO 22262 to provide basic instruction in the fundamental analytical procedures.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 22262-1:2012, *Air quality — Bulk materials — Part 1: Sampling and qualitative determination of asbestos in commercial bulk materials*

ISO 22262-2:2014, *Air quality — Bulk materials — Part 2: Quantitative determination of asbestos by gravimetric and microscopical methods*

3 Terms and definitions

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

3.1
asbestiform
specific type of mineral fibrosity in which the fibres and fibrils possess high tensile strength and flexibility

[SOURCE: ISO 13794:1999, 2.6]

3.2
asbestos
term applied to a group of silicate minerals belonging to the serpentine and amphibole groups which have crystallized in the asbestiform habit, causing them to be easily separated into long, thin, flexible, strong fibres when crushed or processed

Note 1 to entry: The Chemical Abstracts Service Registry Numbers of the most common asbestos varieties are: chrysotile (12001–29–5), crocidolite (12001–28–4), grunerite asbestos (Amosite) (12172–73–5), anthophyllite asbestos (77536–67–5), tremolite asbestos (77536–68–6) and actinolite asbestos (77536–66–4). Other varieties of asbestiform amphibole, such as richterite asbestos and winchite asbestos[20], are also found in some products such as vermiculite and talc.

[SOURCE: ISO 13794:1999, 2.7, modified]

3.3
comminuted sample
analytical sample prepared by comminution and sieving of the original sample

3.4
gravimetric matrix reduction
procedure in which constituents of a material are selectively dissolved or otherwise separated, leaving a residue in which any asbestos present in the original material is concentrated

[SOURCE: ISO 22262-2:2014, 3.22]

3.5
integral intensity
peak area count (integral count) of a designated XRD peak after subtracting the background area

3.6
limit of detection
weight of asbestos on a filtered sample which produces a detectable XRD peak under the measurement conditions shown in Annex A

Note 1 to entry: Expressed as a percentage mass fraction of the original sample.

3.7
limit of quantification
weight of asbestos on a filtered sample for which the integral intensity of the XRD peak can be measured

Note 1 to entry: Expressed as a percentage mass fraction of the original sample. Limit of quantification is conventionally expressed as three times the limit of detection.

3.8
matrix
materials in a bulk sample within which fibres are dispersed

[SOURCE: ISO 22262-1:2012, 2.36, modified]

3.9
original sample
sample taken from a building material product which was analysed using ISO 22262-1

3.10
residual ratio
reduction ratio (percent) achieved by gravimetric matrix reduction of the comminuted sample

3.11
residual sample
analytical sample remaining after treatment with formic acid or other appropriate treatment to remove matrix constituents

3.12
standard uncertainty
uncertainty of the result of a measurement expressed as a standard deviation

[SOURCE: ISO 20988:2007, 3.2]

3.13
sub-residual sample
analytical sub-sample taken from the residual sample for analysis when the residual ratio exceeds 15 %

4 Range

This part of ISO 22262 is for application to building material samples and the target range for the mass fraction of asbestos is from 0,1 % to 5 %. In this method, gravimetric matrix reduction procedures are used to reduce the matrix constituents and interference minerals in a 100 mg comminuted sample. There is no upper limitation for quantification; this XRD method can quantify asbestos up to 100 %. The lower end of the range depends on the residual ratio obtained by the matrix reduction methods. When the matrix reduction method is not effective and 100 % of the sample remains, the limit of detection (LOD) is 0,1 % and the limit of quantification (LOQ) is 0,3 %. When lower values of the residual ratio can be achieved, the LOD and LOQ can decrease to 0,01 % or smaller. However, the LOD and LOQ that can be obtained during analyses of actual building materials also depends on the X-ray peak selected for analysis and whether interference X-ray peaks or high-intensity background from matrix materials are present.

5 Limit of quantification

This XRD method can detect 0,01 mg asbestos in a 10 mg sample on a filter of area 2 cm^2 and quantify 0,03 mg asbestos in a 10 mg sample on a filter of area 2 cm^2. The maximum sample weight for which the X-ray absorption can be corrected is approximately 15 mg on a filter of area 2 cm^2. In this method, gravimetric matrix reduction procedures are used to reduce the matrix constituents and interference minerals in a 100 mg comminuted sample. When a 100 mg sample containing 0,01 mg asbestos is reduced by matrix reduction to 10 mg (residual ratio: 10 %), the XRD method can detect the 0,01 mg asbestos on the filter. Consequently, 0,01 % asbestos in the original sample of 100 mg can be detected. In this case, the LOD and LOQ of the XRD method are approximately 0,01 % and 0,03 %, respectively. When the matrix reduction methods are less effective and more than 15 mg of the sample remains, the LOD and LOQ can increase up to 0,1 % and 0,3 %, respectively. An LOQ of approximately 0,1 % is achieved when the sample is reduced to approximately 30 % by the matrix reduction methods.

The LOD and LOQ that can be achieved during analysis of actual building materials depend on the following:

a) the type of asbestos being analysed;

b) whether a secondary peak is being measured because the primary peak has overlapping peaks;

c) the differences between the source of asbestos in the sample and that of the standard asbestos reference material used to derive the working curve;

d) the extent to which gravimetric matrix reduction can remove matrix materials;

e) the presence of interference X-ray peaks or high backgrounds from matrix materials;

f) the power of the X-ray generator and the type of X-ray detector used.

6 Symbols and abbreviated terms

m_1	weight of comminuted sample at the time of X-ray quantitative analysis (mg)
m_2	weight of residual sample at the time of X-ray quantitative analysis (mg)
m_3	weight of sub-residual sample at the time of X-ray quantitative analysis (mg)
A_s	weight of asbestos in the residual sample, derived from working curve (mg)
w_i	asbestos mass fraction of one analytical sample (%)
w_r	asbestos mass fraction of one sub-residual sample (%)
w	asbestos mass fraction in building material products or other products (%)
r	weight loss ratio after heat treatment for a sample containing organic constituents
V	integral X-ray diffraction intensity in counts
s_i	standard deviation of integral X-ray diffraction intensity of i times
a	gradient of working curve
w_k	lower limit of detection of asbestos mass fraction (%)
w_t	lower limit of determination of asbestos mass fraction (%)
XRD	X-ray powder diffraction

7 Requirements for quantification

A prerequisite for use of this part of ISO 22262 is that the sample shall have been examined using ISO 22262-1.

Quantification of asbestos beyond the estimate of mass fraction achieved using ISO 22262-1 may not be necessary, depending on the applicable regulatory limit for definition of an asbestos-containing material, the variety of asbestos identified and whether the sample can be recognized as a manufactured product. Common regulatory definitions of asbestos-containing materials range from “presence of any asbestos” to >0,1 %, >0,5 % to >1 % by mass fraction of one or more of the regulated asbestos varieties. For many bulk samples analysed using ISO 22262-1, it is intuitively obvious to an experienced analyst that the asbestos mass fraction far exceeds these mass fraction limits. In the case of these types of samples, an experienced analyst can also confidently determine that the asbestos mass fraction is well below these regulatory limits. More precise quantification of asbestos in these types of samples is unnecessary, since a more precise and significantly more expensive determination of the asbestos mass fraction will neither change the regulatory status of the asbestos-containing material nor any subsequent decisions concerning its treatment. Annex C shows a tabulation of most asbestos-containing materials, the variety of asbestos used in these materials and the range of asbestos mass fraction that may be present. Annex C also indicates whether, in general, the estimate of asbestos mass fraction provided by the use of ISO 22262-1 is sufficient to establish the regulatory status of the material or whether quantification of asbestos by this part of ISO 22262 is necessary. The analyst should refer to Annex C for guidance on the probable asbestos mass fractions in specific classes of product and the optimum analytical procedure to obtain a reliable result.

Asbestos was never deliberately incorporated for any functional purpose into commercially manufactured asbestos-containing materials at mass fractions lower than 0,1 %. Accordingly, if any one or more of the commercial asbestos varieties (chrysotile, amosite, crocidolite or anthophyllite) is detected in a manufactured product, the assumption can be made that asbestos is present in the product at a mass fraction exceeding 0,1 %. Therefore, if the regulatory definition of an asbestos-containing material in a jurisdiction is either "presence of any asbestos" or greater than 0,1 %, then detection of one or more of the commercial asbestos varieties in a recognizable manufactured product automatically defines the regulatory status of the material. If the regulatory definition is either 0,5 % or 1 % and the mass fraction of asbestos is estimated to be lower than approximately 5 %, then more precise quantification is necessary to guarantee the regulatory status of the material.

Detection of tremolite, actinolite or richterite/winchite in a material does not allow any assumptions to be made regarding the asbestos mass fraction because these asbestos varieties were, in general, not deliberately added to the products. Rather, they generally occur as accessory minerals in some of the constituents used to manufacture products. Since the non-asbestiform analogues of the amphiboles are not generally regulated, it is also necessary to discriminate between the asbestiform and non-asbestiform analogues of these minerals. When present, these amphibole minerals often occur as mixtures of the two analogues in industrial minerals.

It is not possible to specify a single analytical procedure for all types of material that may contain asbestos because the range of matrices in which the asbestos may be embedded is very diverse. Some materials are amenable to gravimetric matrix reduction and some are not.

The requirements for quantification beyond that achieved in ISO 22262-1 are summarized in Table 1.

Table 1 — Summary of requirements for quantification of asbestos in bulk samples

Type of material	**Regulatory control limit**			
	"Any asbestos"	**Mass fraction >0,1 %**	**Mass fraction >0,5 %**	**Mass fraction >1 %**
Commercially manufactured product	If any commercial asbestos variety is detected, no further quantification is required.		If asbestos is detected at an estimated mass fraction of <5 %, more precise quantification is required to establish the regulatory status of the material.	
Other materials	If any variety of asbestos is detected, no further quantification is required.	If asbestos is detected at an estimated mass fraction of <5 %, more precise quantification is required to establish the regulatory status of the material.		

8 Apparatus and reagents

8.1 Apparatus

8.1.1 Sample comminution equipment

An agate mortar and pestle, or a mill, is required for grinding of samples to suitable sizes for XRD measurement. This equipment shall be used in a negative pressure HEPA-filtered dust hood with a minimum face velocity of 0,4 m/s.

8.1.2 Negative pressure, HEPA-filtered dust hood

A HEPA-filtered dust hood with a minimum face velocity of 0,4 m/s is required to accommodate equipment for comminution of samples.

8.1.3 Analytical balance

An analytical balance with a readability of 0,000 01 g (0,01 mg) or lower is required.

8.1.4 Muffle furnace

For ashing of samples to remove interference organic constituents, a muffle furnace with a minimum temperature of 500 °C, and a temperature stability of ±10 °C, is required.

8.1.5 Ultrasonic cleaner

An ultrasonic cleaner is required for dispersion of residual samples before carrying out the filtration process.

8.1.6 Glass filtration assembly (25 mm diameter)

A glass filtration assembly with a vacuum filtration flask is required.

8.1.7 General laboratory supplies

The following supplies and equipment, or equivalent, are required:

a) glassine paper sheets, approximately 10 cm × 10 cm, for examination of the original samples and the comminuted samples;

b) disposable aluminium or plastic weighing cups, approximately 3 cm to 5 cm in diameter;

c) sampling utensils, including tweezers, needles and others;

d) conical beakers, 50 ml;

e) beakers, 500 ml;

f) volumetric flasks, 1 000 ml;

g) petri dishes;

h) disposable pipettes, 20 µl, 100 µl, 200 µl, 400 µl, 600 µl, 1 ml and 2 ml;

i) polytetrafluoroethylene (PFTE)-coated glass fibre filters, 25 mm diameter.

8.1.8 X-ray diffractometer

An X-ray powder diffractometer using Bragg-Brentano (para-focusing) geometry is required and equipped as follows:

a) copper target X-ray tube, with a power of 1,6 kW or higher;

b) sample spinner to improve particle statistics;

c) nickel filter, graphite monochromator or other X-ray optics with similar or better energy resolution to obtain a monochromatic X-ray beam (CuKα line);

d) high-efficiency position-sensitive X-ray detector, e.g. position-sensitive semiconductor detector.

NOTE Due to the low detection limits required, older type scintillation or proportional counters are not recommended.

8.2 Reagents

8.2.1 Dust-free distilled water.

8.2.2 Concentrated formic acid, reagent grade.

8.2.3 Sodium hydroxide pellets, reagent grade.

8.2.4 Isopropyl alcohol, reagent grade.

9 Quantitative XRD method and principle

9.1 Quantitative XRD methods using an external standard

Since the intensity of an XRD peak depends on the amount of a crystalline substance in a sample, the mass fraction of a crystalline substance can be determined by measurement of the diffraction intensity. However, as the diffraction intensity is influenced not only by the mass fraction of the crystalline materials but also by the absorption of the X-rays by the sample itself, the measured intensity should be corrected for this absorption to enable quantification. The well-known quantitative methods for correcting the absorption in XRD of powder samples are the internal standard method and the standard addition method.[5][6] The analytical accuracies of these methods are high for most substances; however, for fibrous particles, such as asbestos, accuracy can suffer because the fibre orientation varies greatly and this affects the diffraction intensity. An external standard method using a substrate standard was developed for small samples of powder[7] and it was simplified by mounting the sample on a copper foil and measuring the diffraction intensity from the foil with and without the sample in place. A correction factor can then be calculated from the observed attenuation of the diffraction peak from the copper foil.[8] Another method, using a silver membrane filter which replaced the copper foil, was developed.[9] Then, a technique was developed in which a sample of airborne particulate is collected on a polycarbonate filter and the particulate is re-deposited on a silver membrane filter.[10][11][12] It was recognized that a fibrous sample deposited on a thin filter yields a stable and reproducible diffraction intensity due to the fact that fibres are oriented parallel to the filter surface.[13][14] A thin filter asbestos sample is placed on a substrate metal plate and the diffraction intensities from both asbestos on the filter and the substrate metal plate can be measured because a thin filter does not significantly absorb X-rays. The technique using a membrane filter of mixed esters of cellulose and a substrate zinc plate was developed for measurement of airborne quartz samples.[15] These XRD methods using a substrate metal filter or a thin filter on a substrate metal plate are employed by various organizations for quantitative analysis of asbestos and crystalline silica.[16][17][18][19] The substrate standard mass absorption correction method is employed in this part of ISO 22262.

9.2 Summary of the quantitative method

The XRD method specified in this part of ISO 22262 is applicable to the quantitative analysis of asbestos in asbestos-containing samples as identified by ISO 22262-1. The observed diffraction intensities of all crystalline substances in a sample are attenuated as a result of X-ray absorption by the sample matrix. The attenuation of the diffraction intensities from a crystalline substance can be corrected using a correction factor, based on the reduction of the diffraction intensity of the substrate standard material, as shown in Annex B. The diffraction intensities from asbestos in the working curves shown in Annex A are those that have been corrected for the attenuation due to X-ray absorption. The weight of material on a filter (2 cm^2) should be less than 15 mg.[13][14] For weights up to 15 mg, under optimum conditions, the diffraction intensity (integral intensity) of asbestos can be measured to as low as 0,01 mg/filter (2 cm^2),[13][14] provided that interference X-ray peaks or high background from matrix materials are not present. For the quantification of asbestos in bulk materials by this part of ISO 22262, gravimetric matrix reduction methods are used to remove as much as possible of the matrix constituents of the sample, so that any asbestos is concentrated to a higher mass fraction in the final residual sample.

A summary of the quantitative procedure is as follows.

a) An appropriate sub-sample (0,5 g or more) is taken from the original sample, in which asbestos has already been identified by ISO 22262-1.