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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

Reference number
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Foreword

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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.

b) The sample is ground and sieved through a 250 µm mesh sieve to prepare a comminuted sample. It is necessary to grind the sample in this way in order to obtain high quality data by XRD analysis. Depending on the matrices, such as organic components, it may be necessary to ash the sample before grinding.

c) A sub-sample of 100 mg is taken from the comminuted sample and treated with formic acid and/or ashing and the residue is filtered onto a membrane filter, such as a PTFE-coated glass fibre filter, a silver filter or a polyvinylchloride (PVC) filter. After weighing, if the residual ratio is lower than 15 %, the filtered residual sample is used for the quantitative analysis of asbestos by the XRD method. Prior to the quantitative analysis, a qualitative XRD scan is recommended to examine the diffraction intensities of any asbestos minerals and also to check whether peaks from any other minerals in the matrix interfere with them.

NOTE For some lagging samples composed of calcium silicates, a chemical treatment using formic acid is sometimes not effective and more than 15 % residue remains after the treatment. In this case, an alkali treatment using 20 % sodium hydroxide solution is effective to reduce the matrix. The procedure is described in 10.3.

d) When the residue from the matrix reduction by ashing and treatment with formic acid or alkali exceeds 15 %, take a sub-residual sample of 10 mg to 15 mg from the residual sample and transfer it onto a filter. The maximum amount of sample on a membrane filter is limited to 15 mg due to limitations of the mass absorption correction.

e) When matrix reduction of a 100 mg comminuted sample yields a residual sample of 10 mg (residual ratio: 10 %), the XRD method can provide a limit of detection (LOD) of approximately 0,01 % and a limit of quantification (LOQ) of approximately 0,03 %. When the residual ratio is higher, the LOD can increase up to 0,1 % and the LOQ can increase up to 0,3 %. A LOQ of approximately 0,1 % is achieved when matrix reduction results in a residual ratio of approximately 30 %. 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.

9.3 Preparation of working curve and measurement

Measure the diffraction intensity from the substrate standard metallic plate (zinc, aluminium, etc.), to which is attached a blank filter (the same type of filter to be used for the preparation of the working curves).

Asbestos standards (12.2, Note 1) in the range between 0,05 mg and 5 mg are deposited on the filters and the each filter is placed on a substrate standard zinc plate. The diffraction intensities from zinc and asbestos are then measured for each filter.

The diffraction intensity of a zinc plate to which a filter containing asbestos is attached will be attenuated compared with that of zinc plate to which a blank filter is attached. From this attenuation ratio, the correction factor, K_f, is calculated using Formula (B.1). The absorption-corrected diffraction intensity of the asbestos is calculated using Formula (B.2).

The working curve is obtained by plotting the weights of asbestos in the range between 0,05 mg/filter (2 cm^2) and 5 mg/filter (2 cm^2) on the abscissa and the respective absorption-corrected diffraction intensities of the asbestos on the ordinate.

For the quantitative measurement of asbestos in a sample, the weight of asbestos (mg) corresponding to the observed diffraction intensity can be obtained by comparison of the absorption-corrected diffraction intensity of the asbestos in the sample with the diffraction intensity shown in the working curve. The asbestos mass fraction (%) is then calculated from the ratio of the weight of asbestos and the weight of the original sample, i.e. 100 mg.

9.4 Interference minerals

Before quantitative analysis, it is necessary to investigate whether any interference minerals are present from which diffraction peaks appear at or near those of chrysotile or amphibole (see Table 2). Precise checks of diffraction patterns for potentially overlapping peaks can establish the existence of these interference minerals. When a diffraction peak from an interference mineral appears at or near that used for determination of asbestos, chemical treatment (using acid and/or alkali) may effectively dissolve those interference minerals and also reduce the matrix. When interference minerals cannot be removed effectively by such treatment, it will be necessary to use a secondary diffraction peak of asbestos for the quantification (see 11.1 and 11.2). When a secondary peak is employed for quantitative analysis, the quantification limit of the measurement is sometimes degraded.

10 Preparation of comminuted sample

The various sample preparation procedures described in ISO 22262-2 for the quantification of asbestos by PLM can also be used for this XRD method.

Comminution of asbestos-containing materials can generate airborne asbestos fibres and is therefore hazardous. Accordingly, the preparation of samples shall be carried out in a HEPA-filtered containment apparatus.

10.1 Preparation of comminuted sample from original sample

Prepare the comminuted samples from the original samples as follows.

a) Original samples analysed by the qualitative analysis in ISO 22262-1 are pulverized into powder. The comminution procedure shall be as follows.

   1) If the original sample is hard, scrape material from the side surface using a knife or other appropriate tool. Obtain approximately 0,5 g of sub-sample before putting it in a pulverizer.

   2) For the comminution, use a mortar (porcelain mortar, agate mortar or alumina mortar, etc.), Wiley mill, an ultra-centrifugal cutter, a vibrating mill, a ball mill or other appropriate pulverizing device.

   3) For some resilient materials, such as asphaltic and vinyl materials, ashing at a temperature lower than 450 °C for a period of approximately 10 h is effective for removal of the organic constituents (see ISO 22262-2:2014, 6.2). After ashing, the sample is milled using an agate mortar or a vibrating mill.

b) Take care to avoid excessive comminution.

c) A small portion of the comminuted sample is passed through a 250 µm mesh sieve. Additional comminution and sieving is carried out until the entire comminuted sample has been passed through the sieve. To facilitate sieving, wet sieving using water or isopropyl alcohol has been found to be effective. All of the sieved sub-samples are combined to comprise the comminuted sample.

10.2 Heat treatment of comminuted samples that contain organic constituents

The analyst shall refer to the method described in ISO 22262-2:2014, 13.4.

The treatment of a comminuted sample having organic constituents shall be as follows.

a) Place each of the three randomly-selected sub-samples of about 0,2 g each into a pre-weighed crucible (m_c).

b) Weigh the crucible with the combined sample (m_a).

c) Place the crucible in a muffle furnace set at a temperature of (450 ± 10) °C and heat for approximately 10 h.

d) After heating, leave the crucible and contents to cool in clean conditions and weigh (m_b). Then calculate the weight loss ratio (r) according to Formula (1):

$$r = \frac{m_b - m_c}{m_a - m_c} = \frac{m}{m_0} \quad (1)$$

where

r is the weight loss ratio;

m_a is the weight of the original sample and crucible before heating (g);

m_b is the weight of the original sample and crucible after heating (g);

m_c is the weight of the crucible (g);

m_0 is the weight of the original sample before heating (g);

m is the weight of the original sample after heating (g).

10.3 Pretreatment for preparation of residual samples

The preparation of residual samples for quantitative determination of asbestos shall be carried out as follows (refer to the method described in ISO 22262-2:2014, 12.3).

a) Weigh a 25 mm diameter PTFE-coated glass fibre filter (m_1) (hereafter referred to as "filter") and mount it on a substrate standard metal plate (zinc, aluminium, etc.) in the goniometer of the X-ray diffractometer and measure the diffraction intensity of the metal through the filter.

b) Weigh 100 mg of the comminuted sample precisely (to the nearest 0,000 1 g) (m_2: weighed value of comminuted sample) and put it in a conical flask. Add 20 ml of 20 % formic acid and 40 ml of distilled water and disperse the sample in solution by using an ultrasonic cleaner for about 2 min.

c) Put each flask into a water bath at a temperature of 30 °C and shake the flask for 12 min.

d) Filter the sonicated sample on to a pre-weighed filter using a vacuum filtration apparatus.

e) After drying the filter with the residual sample at about 80 °C, weigh the filter (m_3). The difference between m_3 and m_1 is the mass of the residual sample.

f) The residual ratio (%) is calculated using Formula (2):

$$[(m_3 - m_1)/m_2] \times 100 \quad (2)$$

When the residual ratio exceeds 15 %, a sub-residual sample shall be prepared in accordance with 10.4.

A treatment with alkali is sometimes needed to reduce matrices in building materials consisting of calcium silicates (e.g. lagging). The procedure is as follows.

a) Weigh the filter (m_1) and measure the diffraction intensity of the base metal plate as described in 10.3 a).

b) Weigh approximately 100 mg of the comminuted sample consisting of calcium silicate lagging (m_2) and place it in a 100 ml conical flask. Add 60 ml of 20 % sodium hydroxide solution and heat it to reduce the volume to approximately 50 ml. Allow the flask and contents to cool to room temperature.

c) Filter the suspension on to a pre-weighed filter using a vacuum filtration apparatus. After the filtration, wash the residues on the filter using 20 % formic acid and water.

d) After drying, weigh the filter (m_3). The difference ($m_3 - m_1$) is the weight of the residual sample and the residual ratio (%) is calculated using Formula (3):

$$[(m_3 - m_1)/m_2] \times 100 \quad (3)$$

NOTE 1 Thin PTFE-coated glass fibre filters are frequently used for the measurement of airborne dust in working environments. This type of filter rarely absorbs moisture, it provides excellent weight stability. Glass fibre filters produce a large and broad X-ray background at around 20° to 22° at 2θ for the Cu Kα line, while the diagnostic diffraction peaks of asbestos appear at around 10° to 12° and around 28° to 30° at 2θ where the X-ray background is very low. In addition, PTFE-coated glass fibre filters are extremely thin and highly transparent to X-rays.

NOTE 2 In this XRD measurement method, the residual sample after the matrix reduction treatment is filtered onto a PTFE-coated glass fibre filter and the filter is dried before weighing. The filter sample is mounted directly in the goniometer of the X-ray diffractometer. PTFE-coated glass fibre filters are convenient for measuring both the weights of residual samples and the intensities of diffraction peaks from substrate standard metals and asbestos.

Other membrane filters such as silver, polyvinylchloride (PVC) or mixed esters of cellulose (MEC) may also be used.

10.4 Preparation of sub-residual samples

For residual samples exceeding 15 mg, prepare sub-residual samples as follows.

a) Take a sub-residual sample of 10 mg to 15 mg from the residual sample and disperse it in distilled water using the ultrasonic cleaner.

b) Using a vacuum filtration apparatus, filter the sonicated sub-residual sample onto a pre-weighed PTFE-coated glass fibre filter and dry the filter at about 80 °C.

c) After drying, weigh the filter and subtract the weight of the blank filter to obtain the mass of the sub-residual sample (m_3: weight of the sub-residual sample).

11 Diffraction peaks for analysis of asbestos and of interference materials

11.1 Diffraction peaks for quantitative analysis of asbestos

The diffraction peaks used for the quantification of asbestos are indicated in Table 2, in which mineral names related to asbestos, the International Center for Diffraction Data (ICDD) file numbers, d-spacing (Å), diffraction angle for CuKα X-rays, intensity (I/I_1) and reflection indices (hkl) are shown. Each diffraction peak has a small fluctuation in the angle (2θ) and the intensity (I/I_1) in actual samples. Whenever possible, the main diffraction peaks at 12,1° for chrysotile and at 10,4° to 10,7° for amphiboles are used for quantitative analysis. When the peak of a matrix mineral interferes with the main peak from chrysotile or amphibole, the secondary diffraction peaks at 24,4° for chrysotile and 28,4° to 29,2° for amphiboles are used.

When a sample contains two or more kinds of asbestos, special procedures are required. The varieties of asbestos present are known after completion of the analysis by ISO 22262-1. For mixtures of chrysotile and any variety of amphibole asbestos, the individual concentrations can be measured. For mixtures of different varieties of amphibole asbestos, there are calibration difficulties. If the mixture consists of amosite and crocidolite, the calibrations are sufficiently similar that the diffraction peaks appearing near 10,5° or 28,6° can be interpreted as the sum of the mass fraction of the two kinds of amphibole asbestos. For mixtures of amphiboles with differing calibrations, it is not possible to quantify the results.

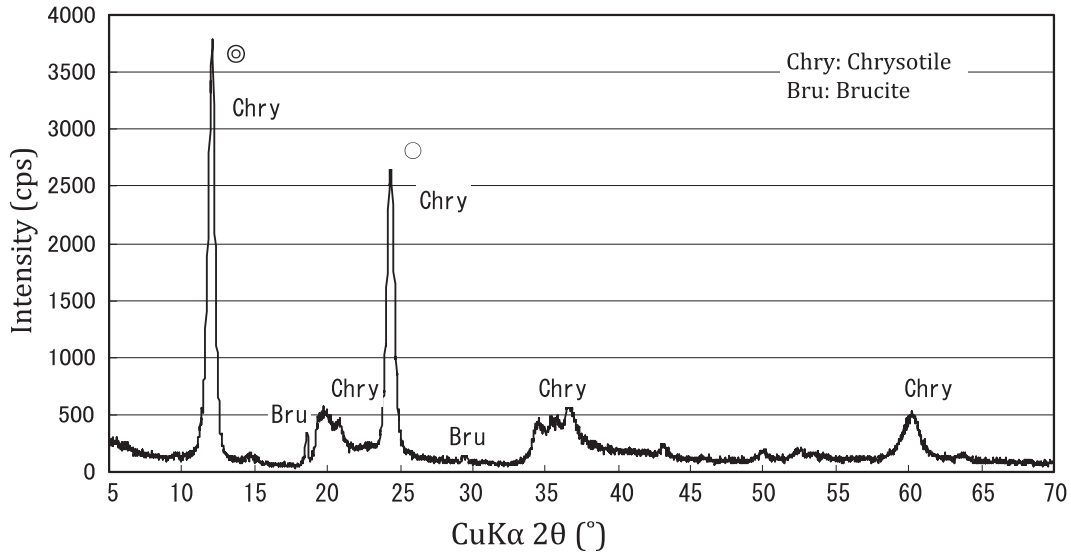
Table 2 — Diffraction peaks used for quantitative analysis of asbestos

	Minerals related to asbestos	ICDD File No.	d (Å)	2θ[a]	I/I_1	hkl	d (Å)	2θ[a]	I/I_1	hkl
			Primary diagnostic peak				Secondary diagnostic peak			
Serpentine	Chrysotile $2Mc_1$	21–543	7,31	12,1	100	002	3,65	24,4	70	004
Amphiboles	Grunerite (Amosite)	17–725	8,33	10,6	100	110	3,07	29,0	80	060,310
	Riebeckite (Crocidolite)	19–1061	8,4	10,5	100	110	3,12	28,6	55	310
	Tremolite	13–437	8,38	10,5	100	110	3,12	28,6	100	310
	Actinolite	25–157	8,47	10,4	70	110	3,143	28,4	70	310
							2,959	30,1	70	–151
	Anthophyllite	9–455	8,26	10,7	55	210	3,05	29,2	100	610,501

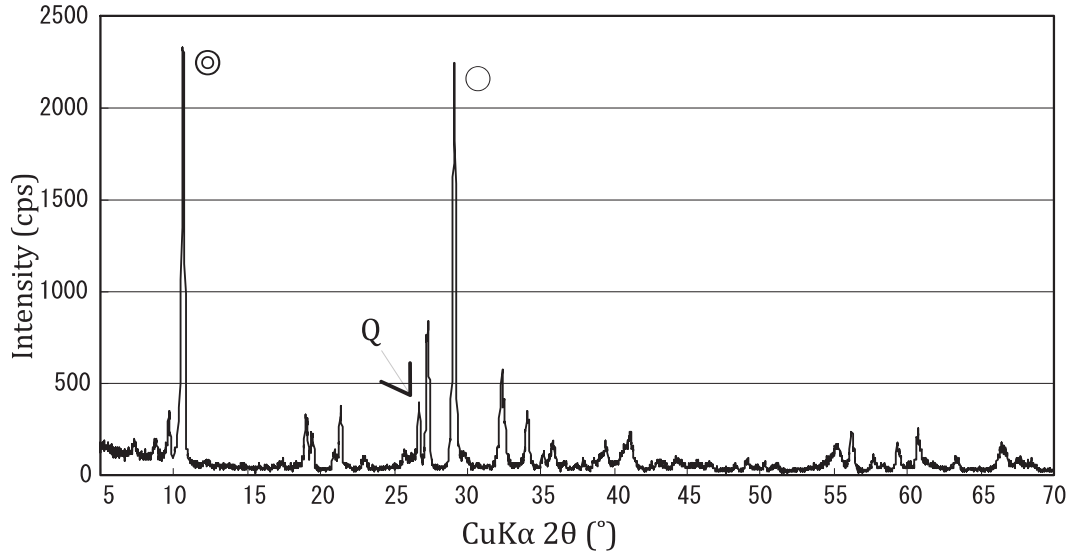
[a] Copper target, Kα line.

ICDD File No.: Diffraction data compiled by Joint Committee on Powder Diffraction Standards-International Center for Diffraction Data, Mineral Powder Diffraction File Data Book 1980.

The diffraction patterns of reference asbestos samples are shown in Figure 1 a) to e). With the exception of a quartz peak in Figure 1 b) (amosite) and Figure 1 c) (crocidolite), all diffraction peaks in Figure 1 b) to e) are attributed to those of the amphibole asbestos of each type. The diffraction peaks used for quantification are marked by a double circle (the main peak) and a single circle (the secondary peak). Tremolite and actinolite are members of a solid solution series, the composition of which varies with the concentration of iron. The XRD pattern obtained from an unknown sample, therefore, may differ from the pattern exhibited by each end-member mineral shown in the ICDD/JCPDS file. Therefore, tremolite and actinolite are categorized as tremolite/actinolite except when the pattern fits either that of tremolite or actinolite as listed in the ICDD/JCPDS file.

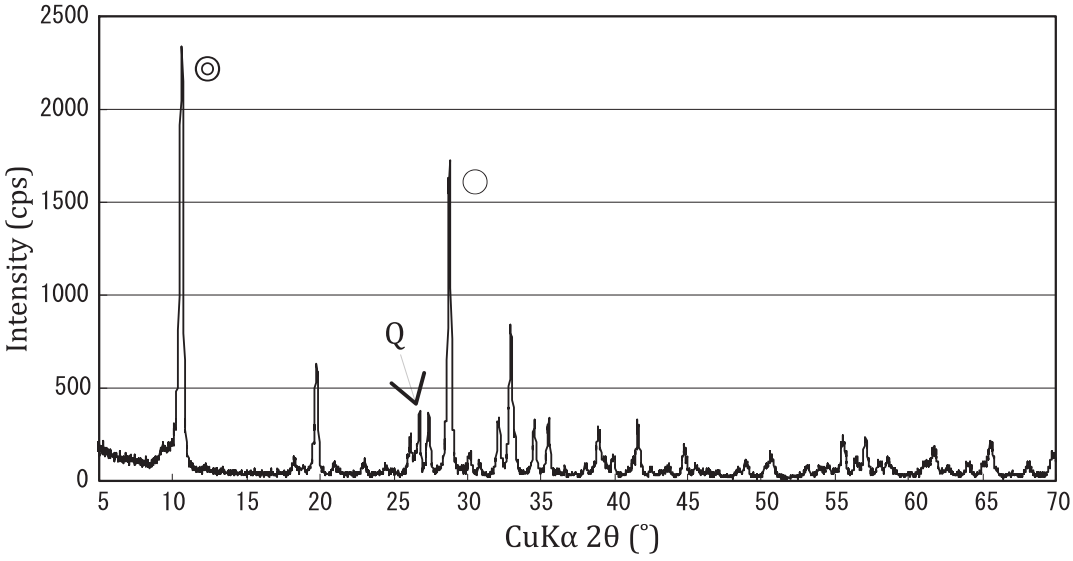


a) Chrysotile (JAWE 111; Coalinga mine, USA)



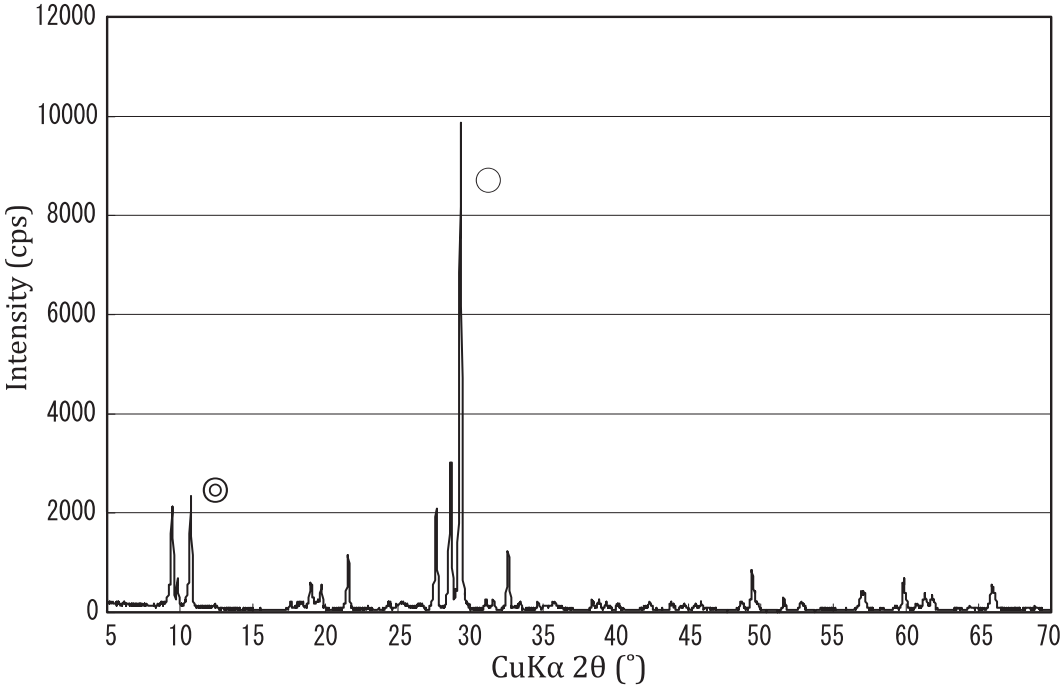
b) Amosite (JAWE 211; Transvaal mine, South Africa)

All diffraction peaks except the quartz peak (Q) in Figure 1 b) are attributed to those of amosite.



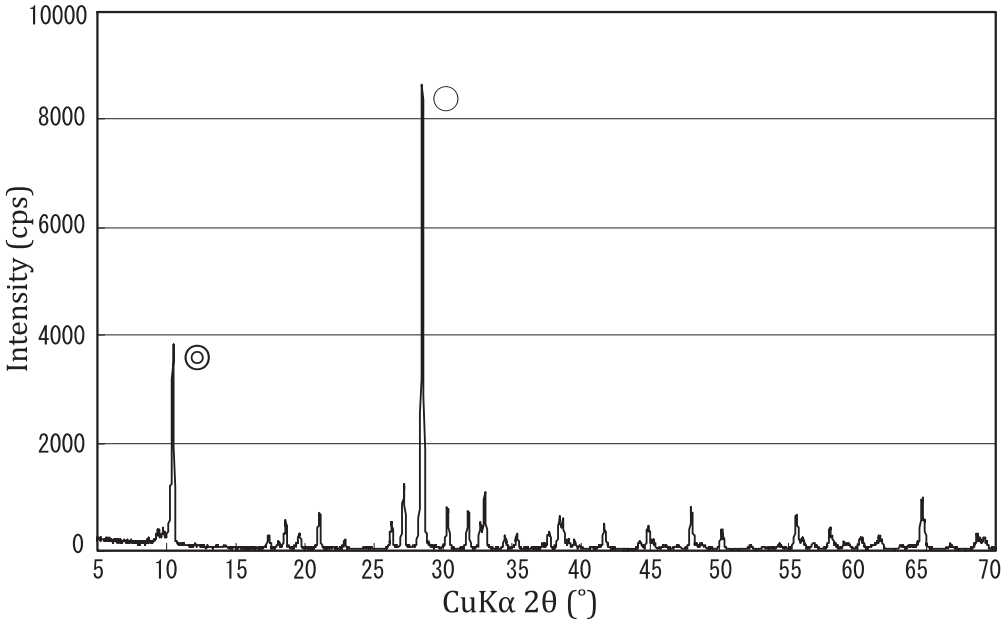
c) Crocidolite (JAWE 311; Transvaal mine, South Africa)

All diffraction peaks except the quartz peak (Q) in Figure 1 c) are attributed to those of crocidolite.



d) Tremolite asbestos (JAWE 411; Yamaga mine, Japan)

All diffraction peaks that appeared in Figure 1 d) are attributed to those of tremolite.



e) Anthophyllite asbestos (JAWE 511; a mine in Afghanistan)

All diffraction peaks that appeared in Figure 1 e) are attributed to those of anthophyllite.

Figure 1 — XRD patterns of typical asbestos

11.2 Interference minerals

11.2.1 Possible interference minerals

When a diffraction peak of a matrix material overlaps or adjoins the first diffraction peak of asbestos, the secondary peak of asbestos should be used. Therefore, before quantitative analysis, the XRD pattern of the sample should be examined to determine if any diffraction peaks of interference minerals overlap or adjoin with the diagnostic diffraction peaks of asbestos. When a diffraction peak from a matrix mineral overlaps the main peak of chrysotile (12,1°) or amphibole asbestos (10,4° to 10,7°), the secondary diffraction peak of 24,4° for chrysotile or of 28,4° to 29,2° for amphibole asbestos is used for quantitative analysis, using a residual or sub-residual sample. When chrysotile and chlorite co-exist in a sample, the use of the secondary diffraction peak of chrysotile is recommended for quantification as chlorite does not interfere with this peak. The use of secondary asbestos peaks for quantification can lead to higher limits of quantification than for primary diffraction peaks.

Minerals that show a diffraction peak near the diffraction angle of 12,1° (2θ) of chrysotile are the serpentine minerals lizardite and antigorite, kaolin minerals including halloysite, chlorite (clinochlore), sepiolite, vermiculite, synthetic minerals of gypsum ($CaSO_4$) and hydrated calcium silicates such as xonotlite or tobermorite, as shown in Table 3. The diffraction angle of 12,1° for chrysotile is slightly different from the peaks of gypsum (11,7°), xonotlite (12,7°), kaolinite (12,4°), chlorite (12,4°) and sepiolite (11,8°). Depending on the relative mass fractions of chrysotile and the interference mineral, the peak from chrysotile cannot always be discriminated from those originating from these minerals when they are present. Vermiculite used in building materials has usually been heated at a temperature of about 700 °C or more, causing the basal spacing of 14,5 A to be reduced to 10 A, therefore the peak at 7,15 A (004) from vermiculite is no longer present.

There appear to be no diffraction peaks of minerals showing angles similar to 10,4° to 10,7° (2θ) of the main peaks of amphibole, but the diffraction peaks of calcite (29,4°), xonotlite (29,0°) and tobermorite (29,0°) appear near the secondary peaks of amphibole. Therefore, it is better to use the main diffraction peaks near 10,5° (2θ) for the quantitative analysis of amphibole.

Table 3 — Diffraction peaks of quantitative analysis of asbestos and possible interference minerals in building products

	Minerals	ICDD File No.	d (Å)	2θ[a]	I/I_1	hkl	d (Å)	2θ[a]	I/I_1	hkl
Asbestos	Chrysotile $2Mc_1$	21–543	7,31	12,1	100	002	3,65	24,4	70	004
Minerals[b]	Gypsum	21–816	7,61	11,6	45	020	3,67	24,2	80	002
	Xonotlite	23–125	6,98	12,7	36	001	3,61	24,6	80	002+
	Lizardite	18–779	7,4	11,9	100	001	3,58	24,9	80	002
	Antigorite	21–963	7,29	12,1	100	001	3,62	24,6	60	002
	Kaolinite 1T	14–164	7,17	12,4	100	001	3,57	24,9	100	004
	Halloysite 7A	29–1487	7,3	12,1	65	001				
	Clinochlore IIb	7–160	7,14	12,4	80	002	3,56	24,9	25	008
	Sepiolite	26–1226	7,53	11,8	25	130				
	Vermiculite	16–613	7,15	12,4	15	004				
Asbestos	Grunerite (Amosite)	17–725	8,33	10,6	100	110	3,07	29,0	80	060,310
	Riebeckite (Crocidolite)	19–1061	8,4	10,5	100	110	3,12	28,6	55	310
	Tremolite	13–437	8,38	10,5	100	110	3,12	28,6	100	310
	Actinolite	25–157	8,47	10,4	70	110	3,143	28,4	70	310
							2,959	30,1	70	–151
	Anthophyllite	9–455	8,26	10,7	55	210	3,05	29,2	100	610,501
Minerals[b]	Calcite	5–585					3,035	29,4	100	104
	Talc	13–558					3,12	28,6	100	006
	Tobermorite	19–1364					3,08	29,0	100	220
							2,98	30,0	65	222
	Xonotlite	23–125					3,08	29,0	65	110

[a] Copper target, Kα line.

[b] Possible interference minerals of chrysotile and amphibole asbestos, respectively.

ICDD File No.: Diffraction data compiled by Joint Committee on Powder Diffraction Standards-International Center for Diffraction Data, Mineral Powder Diffraction File Data Book 1980.

11.2.2 Mass reduction treatments for dissolving interference minerals

The gravimetric matrix reduction methods described in ISO 22262-2 are useful to remove some interference minerals in addition to some of the matrix of a sample. Ashing or chemical digestion using formic acid or alkali is sometimes effective to incinerate or dissolve interference minerals in the matrix. As described in ISO 22262-2, if the quantitative analysis is to determine amphibole varieties only, successive refluxing in acid and alkali can effectively remove interference minerals, including some silicates, leaving any amphibole minerals unaltered. An example showing these effects is shown in Annex D.

12 Quantitative analysis by XRD employing substrate standard mass absorption correction

12.1 General

The diffraction intensity of a crystalline substance can be measured using either the peak height or the peak area (integral intensity). Sometimes the peak heights of diffraction peaks from asbestos vary depending on the source of the asbestos, but the variation of the integral intensities is small. For this

analytical method, commercially-available standard asbestos samples are suitable for preparation of calibration filters. It is also important to record the identities of the reference standards used in the analysis because the weight of asbestos on the sample filter is obtained by comparing the diffraction intensity obtained from the sample filter with that obtained from the filter prepared from the particular asbestos standard.

12.2 Preparation of working curve

For samples presumed to contain asbestos mass fractions higher than approximately 1 %, prepare the working curve in accordance with working curve I. For samples presumed to contain asbestos mass fractions lower than approximately 1 %, prepare the working curve in accordance with working curve II.

Working curves shall have a correlation coefficient of at least 0,99.

NOTE 1 Standard asbestos samples are available from several organizations including the following: JAWE [Explanation of JAWE standard substances (in Japanese), 1986; Tokyo, Japan], NIST (Office of Standard Reference Materials, Gaithersburg, MD 20889, USA), HSE (Tylee BE, Davies LST, Addison J (1996) Asbestos reference standards — Made available for analysts. Ann Occup Hyg 40, 711–714). UICC reference standards are also available from several commercial sources.

NOTE 2 Substrate standard metal plates such as zinc, aluminium, etc., are commercially available.

12.2.1 Preparation of working curve I

Working curve method I will be used to analyse samples that are estimated according to 9.2 c) to contain relatively high mass fractions of asbestos (>1 %). Prepare working curve I as follows.

a) Weigh blank filters and measure the diffraction intensities from substrate standard metals on which the blank filters are mounted for preparation of the working curve.

b) Weigh accurately (to the nearest 0,000 01 g) 0,1 mg, 0,5 mg, 1,0 mg, 3,0 mg and 5,0 mg (these are reference values) of the asbestos reference standard material and put them in five separate conical flasks. Add 20 µl, 100 µl, 200 µl, 600 µl and 1,0 ml of 20 % formic acid and 40 µl, 200 µl, 400 µl, 1 ml and 2 ml of distilled water or isopropyl alcohol to these conical flasks using micro-pipettes and treat them in an ultrasonic cleaner for at least 1 min.

c) Place each flask into the water bath at a temperature of 30 °C for 12 min, periodically shaking the flask.

d) Filter each of the suspensions onto a 25 mm diameter PTFE-coated glass fibre filter using vacuum filtration equipment. Dry the filters and weigh to the nearest 0,000 01 g. Use these filters as the standard samples to derive working curve I.

e) Mount each standard filter sample in the X-ray diffractometer and measure the diffraction intensities of the metallic substrate standard plate and the relevant asbestos peaks. Plot the working curve in accordance with the calculation of the substrate standard mass absorption correction described in Annex B. For the substrate plate, metals such as zinc or aluminium may be used.

12.2.2 Preparation of working curve II

Working curve method II will be used to analyse samples that are estimated according to 9.2 c) to contain relatively low mass fractions of asbestos (>1 %). Prepare working curve I as follows.

a) Weigh blank filters and measure the diffraction intensities from substrate standard metals on which the blank filters are mounted for preparation of the working curve.

b) Weigh accurately approximately 10 mg of the asbestos standard sample and place it in a 500 ml beaker with 100 ml isopropyl alcohol. Place the beaker in an ultrasonic bath for at least 1 min to disperse the asbestos. Transfer the asbestos suspension to a 1 000 ml volumetric flask and make up to a volume of 1 000 ml by addition of isopropyl alcohol. The concentration of this suspension corresponds to

0,01 mg of asbestos per ml. If desired, a suspension with the same concentration of asbestos can be prepared by dispersing 5 mg of asbestos in isopropyl alcohol in a 500 ml volumetric flask.

c) Using a pipette, take aliquots of 1 ml, 3 ml, 5 ml, 10 ml, 30 ml, 50 ml and 100 ml of the original suspension and put each into a conical flask.

When taking each aliquot, shake the volumetric flask containing the original suspension vigorously and immediately withdraw the aliquot using a pipette.

If the original suspension is left to stand for an extended period, the asbestos may flocculate. It is very difficult to re-disperse the flocs of asbestos and it is therefore necessary to prepare a new suspension.

d) Add 10 µl, 20 µl, 60 µl, 100 µl and 200 µl of formic acid to each aliquot of suspension, respectively, and treat in the ultrasonic bath for at least 1 min.

e) Place each flask into the water bath at a temperature of 30 °C for 12 min, periodically shaking the flask.

f) Filter each of the suspensions onto a 25 mm diameter PTFE-coated glass fibre filter using vacuum filtration equipment. Dry the filters and weigh to the nearest 0,000 01 g. Use these filters as the standard samples to derive working curve II.

If the volume of suspension to be filtered is less than 10 ml, add isopropyl alcohol to increase the volume to at least 10 ml. This is to ensure that a uniform deposit is obtained on the filter.

g) Mount each standard filter sample in the X-ray diffractometer and measure the diffraction intensities of the metallic substrate standard plate and the relevant asbestos peaks. Plot the working curve in accordance with the calculation of the substrate standard mass absorption correction described in Annex B. For the substrate plate, metals such as zinc or aluminium may be used.

12.3 Procedure for quantitative analysis

Carry out the quantitative analysis as follows.

a) Mount the residual sample or sub-residual sample as prepared in 10.3 and 10.4 on the sample stand in the goniometer of the X-ray diffractometer.

b) Measure the diffraction intensities of the metal (zinc, aluminium, etc.) from the substrate standard metal plate and asbestos in the filter sample under the same conditions as those used for the preparation of the working curve. Apply the mass absorption correction to the diffraction intensities according to the substrate standard mass absorption correction procedure described in Annex B.

c) Calculate the mass of asbestos from the working curve prepared in 12.2 and the asbestos mass fraction (%).

d) Repeat the procedures in a) to c) on at least three residual or sub-residual analytical samples and report the average value as the asbestos mass fraction (%) for the bulk material.

12.4 Calculation of asbestos mass fraction

Calculate the asbestos mass fraction (%) in the comminuted sample according to 12.4.1 or 12.4.2.

12.4.1 Calculation of asbestos mass fraction from a residual sample

Calculate the asbestos mass fraction in each residual sample using Formula (4). Calculate the average asbestos mass fraction for at least three residual samples using Formula (5).

When the procedure in 10.2 is used to prepare the residual sample from a comminuted sample that contains an organic component, correct the calculated mass fraction by the weight loss ratio, *r*.

$$w_{\mathrm{i}} = \frac{A_{\mathrm{s}}}{m_1} \times r \times 100 \tag{4}$$

$$w = \frac{w_1 + w_2 + w_3}{3} \tag{5}$$

where

w_{i} is the asbestos mass fraction in each comminuted sample (%) (w_1, w_2 and w_3);

A_{s} is the asbestos mass in each residual sample derived from the relevant working curve (mg);

m_1 is the weight of the comminuted sample (mg);

R is the weight loss ratio. In the case of no weight loss: $r = 1$;

w is the asbestos mass fraction in the original sample (%).

12.4.2 Calculation of the asbestos mass fraction from a sub-residual sample

Calculate the asbestos mass fraction from one sub-residual sample using Formula (6). Calculate the asbestos mass fraction for three sub-residual samples using Formula (5).

In the case of the sub-residual sample prepared by reducing the mass of a residual sample under the heating conditions as indicated in 10.2, it shall be corrected by the weight loss ratio, *r*:

$$w_{\mathrm{r}} = \frac{A_{\mathrm{s1}} \times \left(m_2 / m_3\right)}{m_1} \times r \times 100 \tag{6}$$

where

w_{r} is the asbestos mass fraction of each comminuted sample in the case of heating (%);

A_{s1} is the mass of asbestos in the sub-residual sample derived from the relevant working curve (mg);

m_1 is the weight of the comminuted sample (mg);

m_2 is the weight of the residual sample (mg);

m_3 is the weight of the sub-residual sample (mg);

r is the weight loss ratio. In the case of no weight loss: $r = 1$.

12.5 Lower limits of detection and quantitative determination for the working curve

Make 10 replicate measurements of the integral diffraction intensity of asbestos using the minimum amount of standard sample, 0,01 mg/filter(2 cm^2) to 0,1 mg/filter(2 cm^2), prepared at the time of preparation of the working curve. Calculate the standard deviation of the integral diffraction intensity. Calculate the LOD of the working curve from the gradient of the working curve using Formula (7). Calculate the LOQ of the working curve from the gradient of the working curve using Formula (8).

$$w_{\mathrm{k}} = \frac{\sigma / a}{m_1} \times 100 \tag{7}$$

$$w_t = \frac{3\sigma/a}{m_1} \times 100 \quad (8)$$

where

w_k is the limit of detection (%);

w_t is the limit of quantification (%);

σ is the standard deviation of the integral diffraction intensity;

a is the gradient of the working curve;

m_1 is the weight of the comminuted sample (100 mg).

12.6 Evaluation of uncertainty of XRD measurement

The uncertainty associated with the determination of asbestos by this XRD method is a combination of the following:

a) sample preparation variability;

b) variation of the diffraction intensity due to instrumental performance;

c) diffraction angle;

d) sample conditions;

e) analytical technique.

The uncertainty of this method is evaluated using interlaboratory comparison tests. The (combined) standard uncertainty, $u(y)$, of the result of a measurement, y, and the expanded uncertainty, $U_{0,95}(y)$, of a result of measurement y on a specified level of confidence 0,95 can be calculated in accordance with ISO 20988 which consists of a type A evaluation method described by GUM 98-3. However, it is not exclusive of the basic method of estimation of uncertainty described by GUM 98-3. Examples of evaluation of the uncertainty are shown in Annex E.

13 Test report

The test report shall include the following or at least items a) to j):

a) the date that the sample was received at the analytical laboratory;

b) a reference to this part of ISO 22262, i.e. ISO 22262-3;

c) the identification of the sample, preferably a sample number or identification code. Include the sampling location if it is known by the analyst;

d) the date of the analysis;

e) the identity of the analyst;

f) the variety or varieties of asbestos detected and the mass fraction of each asbestos variety in percentage. If there is more than one variety of amphibole present, report mass fractions in accordance with 11.1;

g) individually for each sample, the residual ratio and the limit of detection for the specific procedure used;

h) individually for each sample and each asbestos variety reported, the limit of quantification for the specific procedure used;

i) any procedure used not specified in this part of ISO 22262 or regarded as an optional procedure;

j) a summary of all applicable specimen preparation details.

Items k) to n) shall be recorded in the laboratory data, but the extent to which they are included as part of the test report is optional:

k) the method used for sample comminution;

l) a record of all weight measurements and observations made during the gravimetric procedures including the weight percentage loss during heating, the weight percent loss during acid treatment and the weight percentage residue;

m) the instrumental measurement conditions used for quantitative analysis;

n) a record of the X-ray scans.

Annex A
(normative)

X-ray diffractometer parameters for quantitative analysis of asbestos

A.1 General

This Annex specifies the operating conditions for the X-ray diffractometer and the measurement parameters for the quantitative analysis of asbestos.

A.2 Measurement conditions for quantitative analysis

Use the conditions listed in Table A.1 for quantitative analysis. Using a rotating sample table for the quantitative determination, measure the diffraction intensity (integral intensity) of asbestos, ensuring that the measurement represents 2 000 counts or more after subtraction of the background.

Table A.1 — Conditions of X-ray diffractometer for quantitative analysis

Setting item		Measurement condition
X-ray counter cathode		Copper (Cu, Kα)
Tube voltage (kV)		40 to 45
Tube current (mA)		30 to 45
Monochromatic colouring (removal of K_β ray)		Ni-filter or graphite monochromator
Detector		SC[a] counter or SCA[b] detector
Intensity measurement (counts)		For the measurement of the diffraction intensity, the peak area (integral intensity) is measured after subtracting the background. Sufficient counts should be obtained for the quantitative determination of low levels of asbestos, e.g. 2 000. counts or more.
Time constant (s)		1
Scan rate (°/min)	Continuous scanning (°/min)	1/8 to 1/16
	Step scanning	0,02° × 10 s to 0,02° × 20 s
Divergence slit (°)		1
Scattering slit (°)		1
Receiving slit (mm)		0,3
Scanning range (°, 2θ)		The range includes of about 2° to 3° (2θ) between the lower and higher angle of a quantitative diffraction peak.

[a] Scintillation.

[b] Semiconductor array.

A.3 Preparation of working curves and lower limit of quantification

Standard asbestos minerals of known weight were dispersed in water and filtered onto glass fibre filters, which were dried and used as standard asbestos filters for preparation of working curves. The diffraction intensities of asbestos on these filters were measured using the conditions given in A.2 and these were plotted against the weights of asbestos to yield the working curves. Examples of working

curves for chrysotile and amosite are shown in Figures A.1, A.2, A.5 and Figures A.3, A.4 and A.6, respectively.

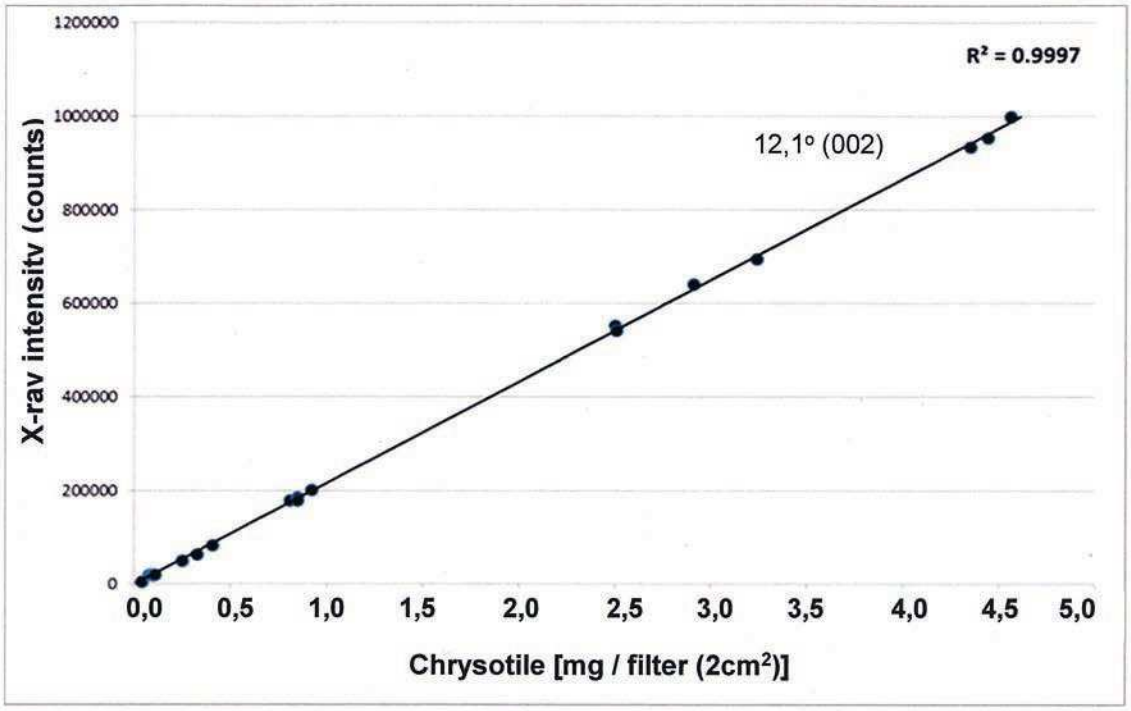


Figure A.1 — Example of working curve I for chrysotile

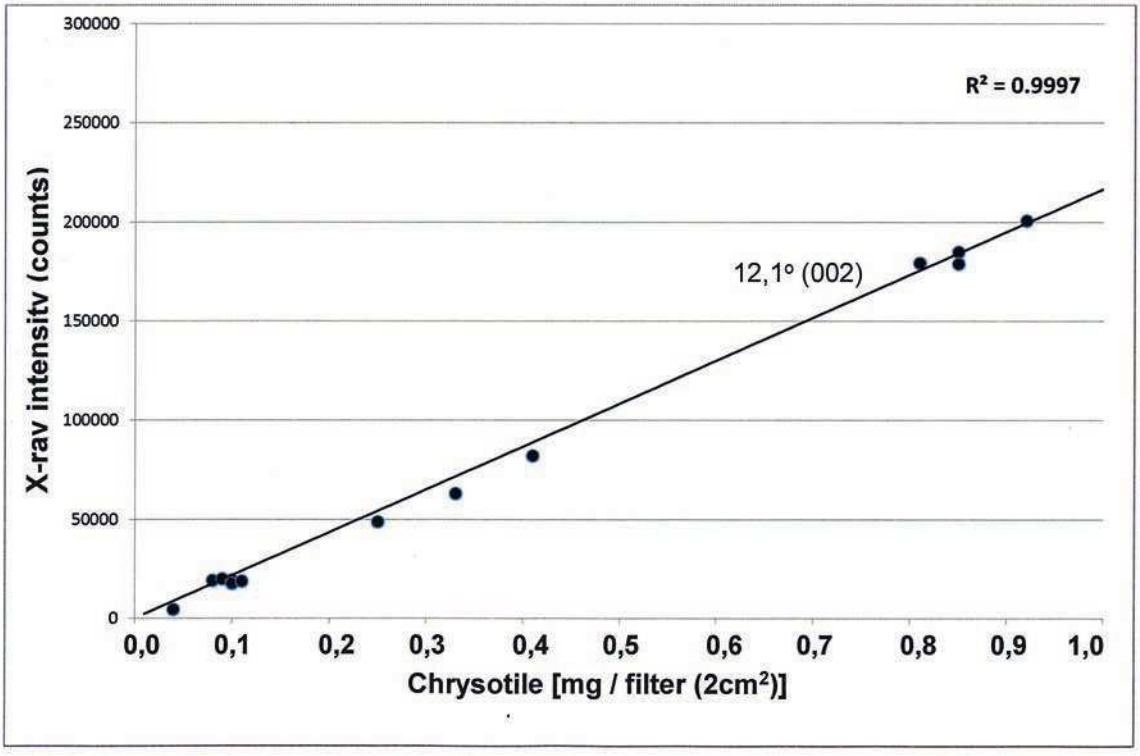


Figure A.2 — Example of working curve I for chrysotile

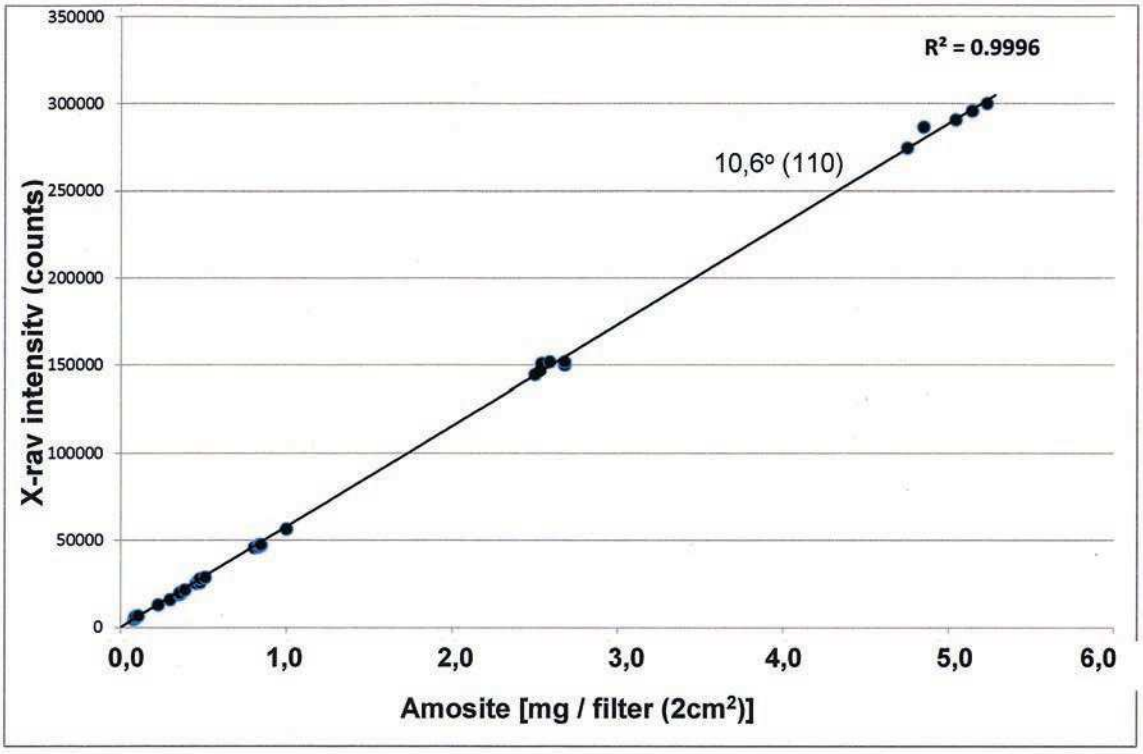


Figure A.3 — Example of working curve I for amosite

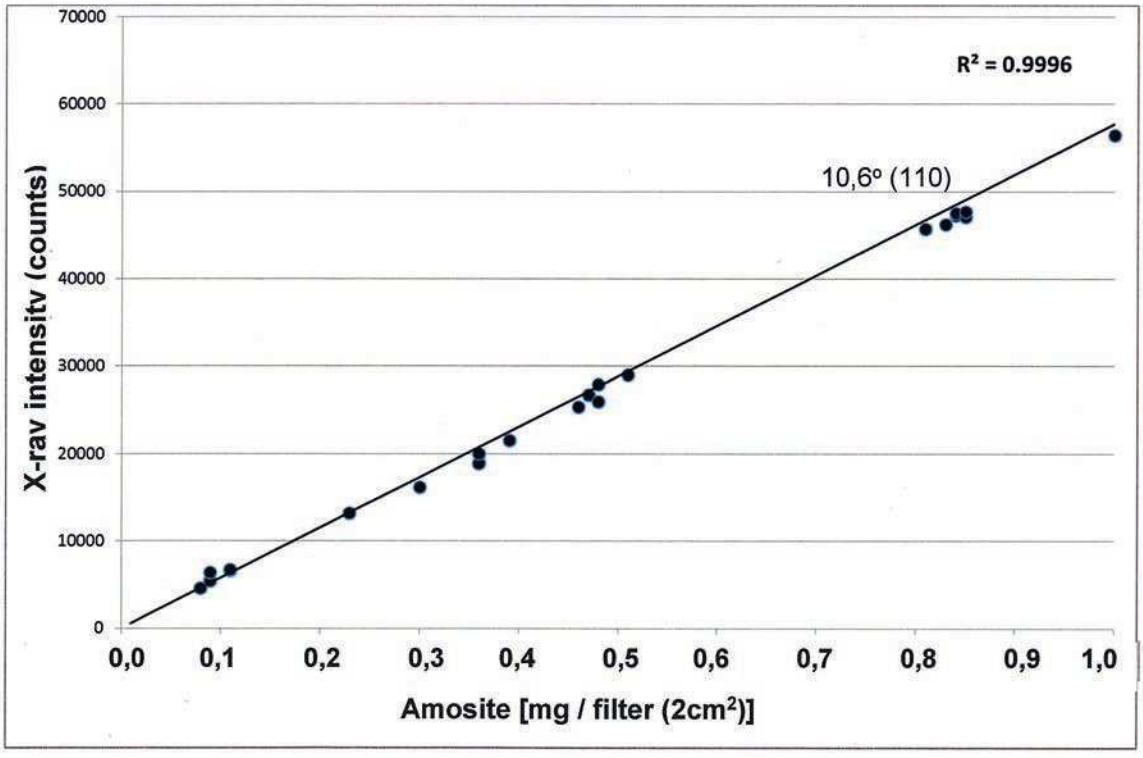


Figure A.4 — Example of working curve I for amosite

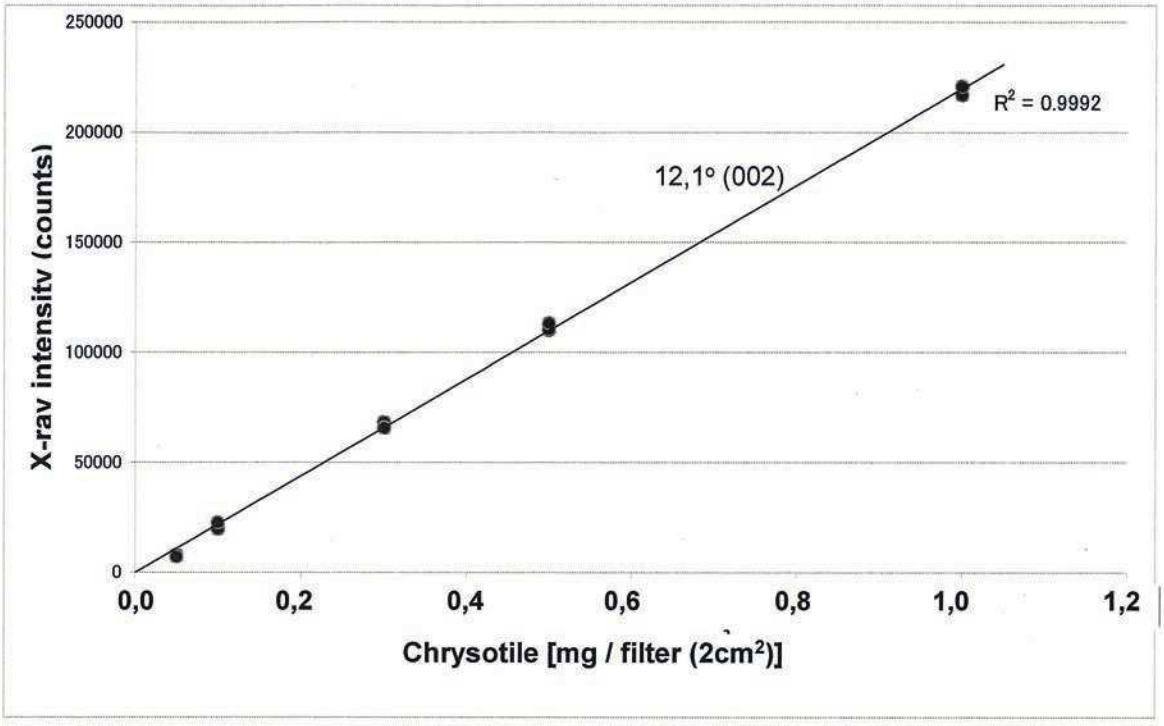


Figure A.5 — Example of working curve II for chrysotile

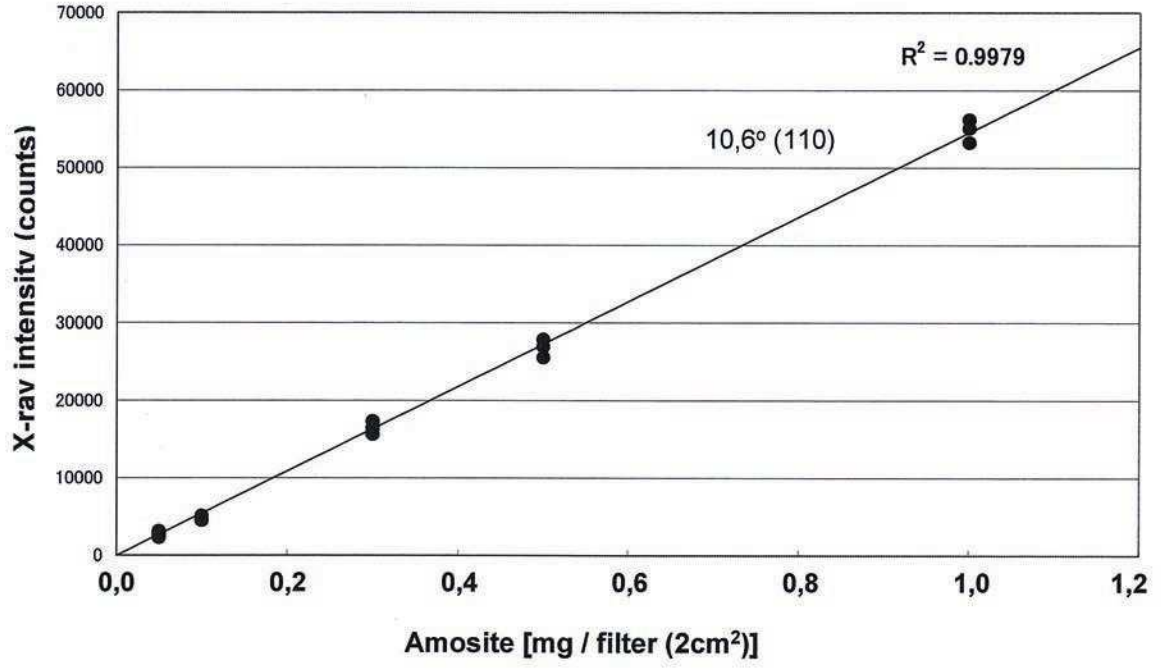


Figure A.6 — Example of working curve II for amosite

Annex B
(normative)

Substrate standard mass absorption correction for asbestos quantification

B.1 General

This Annex specifies the procedure for measurement and calculation of the mass fraction of asbestos by the XRD method using a substrate standard mass absorption correction.

B.2 Procedure for measurement and calculation

a) Measure the diffraction intensity from the substrate standard metallic plate (I^0_{Zn}) (Zn is for zinc; aluminium, silver, etc., can also be used) with blank filters attached. The filters should be the same filters as those to be used for preparation of the working curve.

b) Place a filter on which the asbestos was deposited (in the range between 0,01 mg and 5 mg) on the substrate standard zinc plate and measure the diffraction intensities from the zinc plate (I_{Zn}) and the asbestos (I_a). Repeat these measurements for each of the asbestos standard filters.

c) The diffraction intensity from the zinc plate (I_{Zn}) with a filter loaded with asbestos will be reduced compared with that when a blank filter is used. The correction factor K_f is calculated from this reduction ratio T (= I_{Zn} / I^0_{Zn}) using Formula (B.1).

$$K_f = \frac{-R_\theta \ln T}{1 - T^{R_\theta}} \quad \text{(B.1)}$$

where

R_θ is (= $\sin\theta_{Zn}$ / $\sin\theta_a$);

($\sin\theta_{Zn}$) is the ratio of diffraction angles of the zinc plate;

($\sin\theta_a$) is the ratio of diffraction angles of the asbestos;

ln is the natural logarithm.

d) The diffraction intensity, corrected for absorption (I) is calculated using Formula (B.2)

$$I = I_a \times K_f \quad \text{(B.2)}$$

where

I_a is the intensity of the diffraction peak of asbestos.

e) The asbestos weight in a sample can be obtained by comparing the corrected diffraction intensity with the working curve. The working curve is prepared by plotting the asbestos weight of the working curve in the range between 0,01 mg/filter (2 cm^2) and about 5 mg/filter (2 cm^2) on the abscissa and the respective intensity of the diffraction peak, corrected for absorption, on the ordinate.

Annex C
(informative)

Types of commercial asbestos-containing materials and optimum analytical procedures

The properties of asbestos such as non-flammability, chemical stability and high strength have led worldwide to a broad use of this mineral in the building and industrial sectors. Asbestos cement products, asbestos-containing lightweight panels and fire-prevention panels, asbestos packings and asbestos cloths, asbestos boards, asbestos foams, asbestos-containing fireproofing and acoustic and decorative plasters (sprayed asbestos), asbestos-containing compositions for trowel application and putties are the most important uses. In addition, there is also a variety of products to which asbestos fibres were frequently added in smaller mass fractions, for example paints for protective coatings, adhesives, plastic sheets and tiles.

Table C.1 shows the most important asbestos-containing materials with examples of their applications and the typical asbestos mass fractions that have been observed in these materials. In exceptional cases, asbestos mass fractions deviating from those quoted may have been used.

Table C.1 also shows the optimum analytical procedure for each of the different varieties of material.

Table C.1 — Optimum analytical procedures for asbestos-containing materials

Product	Examples of application	Typical asbestos type and mass fraction if asbestos is present	Analysis in accordance with	Optimum analytical procedure
Asbestos cement flat boards	— Roof claddings — Sidings — Banister elements — Windowsills — Staircases — Partition walls — Support for cable runs — In small sizes as slates and shingles in the roofing and siding sectors	10 % to 12 % chrysotile, sometimes also <5 % crocidolite or amosite in addition to chrysotile	ISO 22262-1	An estimate of asbestos mass fraction from analysis according to ISO 22262-1 is usually sufficient, since in these types of material, asbestos is either present at mass fractions significantly exceeding legislated control limits or asbestos is not present in the formulation.
Asbestos cement corrugated sheets	— Roof claddings — Perimeter insulation — Sidings in the industrial sector	10 % to 12 % chrysotile, sometimes also with <5 % crocidolite in addition to chrysotile	ISO 22262-1	An estimate of asbestos mass fraction from analysis according to ISO 22262-1 is usually sufficient, since in these types of material, asbestos is either present at mass fractions significantly exceeding legislated control limits or asbestos is not present in the formulation.
Asbestos cement pipes/ ducts	— Drinking water and wastewater pipes — Service pipes — Inlet air and exhaust air ducts — Cable shafts	10 % to 15 % chrysotile, drinking water pipe also up to 5 % crocidolite or amosite in addition to chrysotile	ISO 22262-1	An estimate of asbestos mass fraction from analysis according to ISO 22262-1 is usually sufficient, since in these types of material, asbestos is either present at mass fractions significantly exceeding legislated control limits or asbestos is not present in the formulation.
Asbestos cement mouldings	— Standard ashtrays — Flower boxes — Garden articles — Sculptures	10 % to 12 % chrysotile	ISO 22262-1	An estimate of asbestos mass fraction from analysis according to ISO 22262-1 is usually sufficient, since in these types of material, asbestos is either present at mass fractions significantly exceeding legislated control limits or asbestos is not present in the formulation.

Table C.1 *(continued)*

Product	Examples of application	Typical asbestos type and mass fraction if asbestos is present	Analysis in accordance with	Optimum analytical procedure
Asbestos containing lightweight building boards or fire-resistant panels	— Sealing of openings in walls required to be fire resistant, fire-protection encasement of ventilation ducts, cable ducts and cable shafts — Fire closures in walls required to be fire resistant (fire shutters, fire barriers) — Fire-protection encasements — Smoke-removal ducts — Insert in fire-resistant doors and gates — Substructure of luminaries (lighting fixtures)	Approximately 15 % chrysotile and approximately 15 % amosite	ISO 22262-1	An estimate of asbestos mass fraction from analysis according to ISO 22262-1 is usually sufficient, since in these types of material asbestos is either present at mass fractions significantly exceeding legislated control limits or asbestos is not present in the formulation.
Asbestos-containing lightweight building boards or fire-resistant panels	— Lining fire-hazard rooms — Partition walls, partition surfaces, doors — Sanitary modules — Support and beam encasements — Smoke aprons — Fire locks	Up to 50 % chrysotile, sometimes up to 35 % amosite	ISO 22262-1	An estimate of asbestos mass fraction from analysis according to ISO 22262-1 is usually sufficient, since in these types of material, asbestos is either present at mass fractions significantly exceeding legislated control limits or asbestos is not present in the formulation.
Asbestos-containing pipe and boiler insulations	— Corrugated paper pipe insulation — 85 % magnesia block and pipe insulation — Calcium silicate block and pipe insulation	30 % to 100 % chrysotile, total of 15 % asbestos, may be chrysotile, amosite or crocidolite, or any mixture of two or more	ISO 22262-1	An estimate of asbestos mass fraction from analysis according to ISO 22262-1 is usually sufficient, since in these types of material, asbestos is either present at mass fractions significantly exceeding legislated control limits or asbestos is not present in the formulation.

Table C.1 *(continued)*

Product	Examples of application	Typical asbestos type and mass fraction if asbestos is present	Analysis in accordance with	Optimum analytical procedure
Asbestos packing, asbestos cloth	— Seals or sealing strips on lightweight walls required to be fire resistant (at ceiling, floor, joints between elements, wall terminations) — Seals on pipe and duct feed-throughs in walls and ceilings — Seals between flanges of ventilation ducts — Seals on fire-resistant glazing, shelter doors, chimney soot doors — Seals and insulation on heat-generation systems, hot pipes and hot valves — Fire blankets — Heat-resistant clothing, heat-resistant gloves — Lining of pipe clips for hot water, steam and sprinkler pipes — Lamp wicks — Mantles for gas lamps	Predominantly chrysotile (80 % to 100 %); for acid-resistant applications crocidolite	ISO 22262-1	An estimate of asbestos mass fraction from analysis according to ISO 22262-1 is usually sufficient, since in these types of material, asbestos is either present at mass fractions significantly exceeding legislated control limits or asbestos is not present in the formulation.
Asbestos mill-boards	— Sealing strips on lightweight walls required to be fire resistant (at ceiling, floor, joints between elements, wall terminations) — Substructure of luminaries (lighting fixtures) — Bottom coating of wooden windowsills over radiators	80 % to 100 % chrysotile	ISO 22262-1	An estimate of asbestos mass fraction from analysis according to ISO 22262-1 is usually sufficient, since in these types of material, asbestos is either present at mass fractions significantly exceeding legislated control limits or asbestos is not present in the formulation.

Table C.1 *(continued)*

Product	Examples of application	Typical asbestos type and mass fraction if asbestos is present	Analysis in accordance with	Optimum analytical procedure
Asbestos foams	— Infilling (sealing) of movement joints — Seals at fire shutters and fire barriers	Approximately 50 % chrysotile	ISO 22262-1	An estimate of asbestos mass fraction from analysis according to ISO 22262-1 is usually sufficient, since in these types of material, asbestos is either present at mass fractions significantly exceeding legislated control limits or asbestos is not present in the formulation.
Sprayed asbestos	— Contour-following fire-resistant coating of steel structures — Coating of ceilings and walls in music auditoria, theatres, churches, garages, industrial rooms (for noise protection) — Sealing off openings for cable, pipe and duct feedthroughs through walls required to be fire resistant — Encasing of ventilation ducts	40 % to 70 % of chrysotile, crocidolite or amosite, also mixtures of mineral wool with either 20 % amosite or up to 30 % chrysotile. Other mixtures include 15 % chrysotile with either perlite or vermiculite, and gypsum.	ISO 22262-1	An estimate of asbestos mass fraction from analysis according to ISO 22262-1 is usually sufficient, since in these types of material, asbestos is either present at mass fractions significantly exceeding legislated control limits or asbestos is not present in the formulation.
Sprayed decorative coatings (texture coats)	— Coating of ceilings and walls to provide a textured surface which masks irregularities	Chrysotile, up to 5 %. Some constituents may also contain tremolite.	ISO 22262-2 ISO 22262-3	Ashing, treatment with 2 mol/l hydrochloric acid and separation of aggregate by flotation or sedimentation are the optimum procedures. Quantification can be either with PLM point counting, SEM or TEM.
Gypsum wallboard joint compounds	— Provides smooth joint between adjacent panels	Chrysotile, up to 5 %. Some constituents may also contain low mass fractions of tremolite.	ISO 22262-2 ISO 22262-3	Ashing and treatment with 2 mol/l hydrochloric acid are the optimum procedures. Quantification can be either with PLM point counting, SEM or TEM. Absence of asbestos is most effectively demonstrated by TEM examination of the residue using the drop mounting technique.

Table C.1 *(continued)*

Product	Examples of application	Typical asbestos type and mass fraction if asbestos is present	Analysis in accordance with	Optimum analytical procedure
Asbestos-containing troweled-on compositions and putty	— Grouting of prefabricated concrete components — Sealing of movement joints — Pipe feed-throughs through walls and ceilings — Door casings of fire-resistant doors — Anti-drumming coatings (car preservation) — Coating of underwater structures — Baseboard coating on house walls	Up to 20 % chrysotile	ISO 22262-2 ISO 22262-3	Ashing and treatment with 2 mol/l hydrochloric acid are the optimum procedures. PLM examination of the residue is usually sufficient. In some cases, PLM point counting may be necessary. Absence of asbestos is most effectively demonstrated by TEM examination of the residue using the drop mounting technique.
Asbestos-containing floorings	— Reinforcement in flexible sheets — Rot-resistant support layer as underlay of cushion vinyl flooring materials	Chrysotile 10 % to 20 % chrysotile 80 % to 100 %	ISO 22262-1	An estimate of asbestos mass fraction from analysis according to ISO 22262-1 is usually sufficient, since in these types of material, asbestos is either present at mass fractions significantly exceeding legislated control limits or asbestos is not present in the formulation.
Asphalt or vinyl asbestos floor tiles	— Reinforcement	Asphalt tiles up to 35 % chrysotile, vinyl tiles up to 20 % chrysotile	ISO 22262-2 ISO 22262-3	In some floor tiles, substantial asbestos can be observed at a fracture surface and identified according to ISO 22262-1. Most floor tiles are best analysed by ashing and treatment with concentrated (11,3 mol/l) hydrochloric acid, followed by SEM or TEM examination using the drop mount technique.
Rubberized asbestos seals	— Gaskets for pipe flanges	Chrysotile 50 % to 90 %	ISO 22262-1	An estimate of asbestos mass fraction from analysis according to ISO 22262-1 is usually sufficient, since in these types of material asbestos is either present at mass fractions significantly exceeding legislated control limits or asbestos is not present in the formulation.

Table C.1 *(continued)*

Product	Examples of application	Typical asbestos type and mass fraction if asbestos is present	Analysis in accordance with	Optimum analytical procedure
Asbestos-containing friction products	— Brake linings — Brake bands — Clutch linings	Chrysotile 10 % to 70 %	ISO 22262-1	An estimate of asbestos mass fraction from analysis according to ISO 22262-1 is usually sufficient, since in these types of material, asbestos is either present at mass fractions significantly exceeding legislated control limits or asbestos is not present in the formulation.
Acid-resistant containers	— Lead-acid battery boxes — Drums for acid	Crocidolite 10 % to 50 %	ISO 22262-1	Substantial asbestos can often be observed at a fracture surface and identified according to ISO 22262-1. Otherwise, ashing of a sub-sample is the optimum procedure, followed by examination according to ISO 22262-1.
Filter media	— Air filters — Liquid filters — Sterile and aseptic filters — Clarifying sheets — Diaphragms for chloralkali electrolysis process	95 % chrysotile, rarely amosite	ISO 22262-1	An estimate of asbestos mass fraction from analysis according to ISO 22262-1 is usually sufficient, since in these types of material, asbestos is either present at mass fractions significantly exceeding legislated control limits or asbestos is not present in the formulation.

Table C.1 *(continued)*

Product	Examples of application	Typical asbestos type and mass fraction if asbestos is present	Analysis in accordance with	Optimum analytical procedure
Talc (asbestos content dependent on deposit)	— Release agents for electric cables, rubber products — Release agents in the confectionery industry — Tailor's chalk — Paper manufacture — Medicine, cosmetics	Chrysotile and/or actinolite/tremolite	ISO 22262-2	Talc is not amenable to gravimetric matrix reduction methods. For chrysotile, preparation of TEM specimens from the untreated material is the optimum procedure, followed by examination by the mass counting procedure. For amphibole, either centrifugation in heavy liquid, followed by evaluation of the centrifugate by microscopy, or preparation of TEM specimens from the untreated material is the optimum procedure, followed by examination using the mass counting procedure.
Vermiculite (exfoliated)	— Loose-fill attic and wall cavity insulation — Supply to horticultural applications	Depends on the source of the vermiculite. Vermiculite that originated from a former mine in Montana, USA may contain up to 6 % of a mixture of asbestiform amphibole types. Current sources of vermiculite may contain no detectable amphibole or low concentrations of amphibole. Depending on the source of the vermiculite, some of any amphibole that is present may have asbestiform morphology.	ISO 22262-2	Remove the vermiculite by flotation on water. Manually pick out any asbestiform amphibole from the sediment, identify it according to ISO 22262-1, and weigh to obtain the weight percentage. If manual removal of asbestiform amphibole is not feasible because the particle sizes are too small, use the heavy liquid centrifugation technique to separate the amphibole. Alternatively, sequential refluxing in 2 mol/l hydrochloric acid and 4 mol/l sodium hydroxide may be used to dissolve most of the vermiculite. Examine the residue by PLM, SEM or TEM according to ISO 22262-1. Further quantification may be carried out using the SEM or TEM mass counting procedure.

Table C.1 *(continued)*

Product	Examples of application	Typical asbestos type and mass fraction if asbestos is present	Analysis in accordance with	Optimum analytical procedure
Vermiculite concentrate	— Supply to exfoliation plants	Amphibole <<1 %	ISO 22262-2	Exfoliate in a muffle furnace at a temperature of 800 °C. Analyse as for exfoliated vermiculite.
Vermiculite-containing gypsum wallboard	— Fire-resistant wallboard containing vermiculite concentrate	Amphibole << 1 %	ISO 22262-2	Dissolve acid-soluble materials by treatment with 2 mol/l hydrochloric acid. Exfoliate in a muffle furnace at a temperature of 800 °C. Analyse residue as for exfoliated vermiculite.
Vermiculite-containing horticultural products	— Potting soil — Fertilizer	Amphibole <<1 %	ISO 22262-2	Remove organic materials by ashing. Dissolve acid-soluble materials by treatment with 2 mol/l hydrochloric acid. Remove any floating vermiculite. Examine the residue from these treatments by PLM, SEM or TEM. Alternatively, centrifuge the residue from these treatments in heavy liquid and examine the centrifugate for asbestiform amphibole by PLM, SEM or TEM.
Vermiculite-containing fireproofing	— Spray applied on structural steel and decks	Early fireproofing could contain approximately 15 % chrysotile and up to approximately 1 % of amphibole. Later formulations contain no chrysotile and generally much less than 1 % amphibole.	ISO 22262-2	Remove any organic materials by ashing. Remove acid-soluble materials such as gypsum and carbonates by treatment with 2 mol/l hydrochloric acid. Separate any floating vermiculite. Centrifuge the residue from these treatments in heavy liquid and examine the centrifugate for asbestiform amphibole by PLM, SEM or TEM.
Vermiculite-containing lightweight concrete	— Building decks	Amphibole <<1 %	ISO 22262-2	Treat the sample with 2 mol/l hydrochloric acid. Separate any floating vermiculite. Screen out any large fragments of aggregate. Examine the residue for asbestiform amphibole by PLM, SEM or TEM. Amphibole can be concentrated further, if necessary, by heavy liquid centrifugation.

Table C.1 *(continued)*

Product	Examples of application	Typical asbestos type and mass fraction if asbestos is present	Analysis in accordance with	Optimum analytical procedure
Industrial minerals: wollastonite, sepiolite, attapulgite	— Ceramics manufacture — Plastics fillers — Surfacing materials and joint compounds — Ceiling tiles — Drilling muds (attapulgite)	Depends on the source of the mineral. May contain several percentage of tremolite or actinolite.	ISO 22262-2	Sequential refluxing in 2 mol/l hydrochloric acid and 4M sodium hydroxide. Examine the residue by PLM, SEM or TEM according to ISO 22262-1. Further quantification may be carried out using the SEM or TEM mass counting procedure.
Industrial minerals: calcite, dolomite and gypsum	— Manufacture of building materials — Industrial uses	Depends on the source of the mineral. Carbonate minerals may contain several percentage of tremolite or actinolite.	ISO 22262-2	Dissolve in excess hydrochloric acid. Examine the residue by PLM, SEM or TEM according to ISO 22262-1. Further quantification may be carried out using the SEM or TEM mass counting procedure.
Industrial minerals, mica	— Ceramics manufacture — Manufacture of building materials	Depends on the source of the mineral. May contain tremolite or actinolite.	ISO 22262-2	Many industrial minerals, including mica, are not amenable to any gravimetric matrix reduction methods. Preparation of TEM specimens from the untreated material is the optimum procedure, followed by the SEM or TEM mass counting procedure.
Asphalt surfacings	— Road construction	Chrysotile, generally ≤1 %	ISO 22262-2 ISO 22262-3	Chrysotile, if present, is in the asphaltic component. Ash a representative sample and examine the residue by either PLM, SEM or TEM according to ISO 22262-1. Alternatively, separate a sub-sample of the asphaltic component and ash it. Quantitative point counting by PLM or mass counting by SEM or TEM may be necessary. Absence of asbestos may be confirmed by SEM or TEM examination of a drop mount.

Table C.1 *(continued)*

Product	Examples of application	Typical asbestos type and mass fraction if asbestos is present	Analysis in accordance with	Optimum analytical procedure
Wall and ceiling plasters	— Interior wall and ceiling coatings, with or without aggregate and fibres such as animal hair or jute — Exterior stuccos	Chrysotile Generally, locally mixed and inhomogeneous. May be any mass fraction up to approximately 3 %	ISO 22262-2 ISO 22262-3	Chrysotile, when present, is often inhomogeneously distributed. Start with a sub-sample of approximately 3 g or more, depending on the size of any aggregate. Ash the sub-sample, treat with 2 mol/l hydrochloric acid and separate the aggregate by sedimentation. Examine the residue by point counting or confirm the absence of asbestos by SEM or TEM examination of the residue.
Drilling muds	— Oil exploration, rock drilling	Chrysotile Often the chrysotile is very fine and short, sometimes originating from Coalinga, California. May contain up to 100 % chrysotile.	ISO 22262-2 ISO 22262-3	Disperse in water and prepare TEM specimens by the drop mount method. If chrysotile is present, it will be at a substantial weight mass fraction.
Chemical products for construction and other products	— Bitumen, roofing and sealing sheets — Sealing putties — Glazing putties — Bituminous coatings — Fillers and sealers — Jointing compounds — Paints — Glues — Fire retardants — Subfloor protection	Chrysotile up to 30 % Chrysotile up to 2 % Chrysotile up to 4 % Chrysotile up to 30 % Chrysotile up to 25 % Chrysotile up to 5 % Chrysotile up to 9 % Chrysotile up to 4 % Chrysotile up to 10 % Chrysotile up to 4 %	ISO 22262-2 ISO 22262-3	If substantial chrysotile cannot be observed using ISO 22262-1, ash the sample. In some cases, the residual ash will contain substantial mass fractions of chrysotile and it may be possible to terminate the analysis. If other constituents are present in the ash, further matrix reduction may be possible by treatment with hydrochloric acid. Examine the residue using the procedures specified in ISO 22262-1. The absence of asbestos can be confirmed by examination of the residue by SEM or TEM.

Annex D
(informative)

Effects of matrix reduction methods

The gravimetric matrix reduction methods described in ISO 22262-2 and in this part of ISO 22262 are effective not only to reduce the matrices of samples but also to remove some interference minerals in bulk building materials.

Examples that illustrate the effects of matrix reduction are shown in Figures D.1 to D.3. The examples shown are two samples of sprayed rock wool and an asbestos-cement flat board.

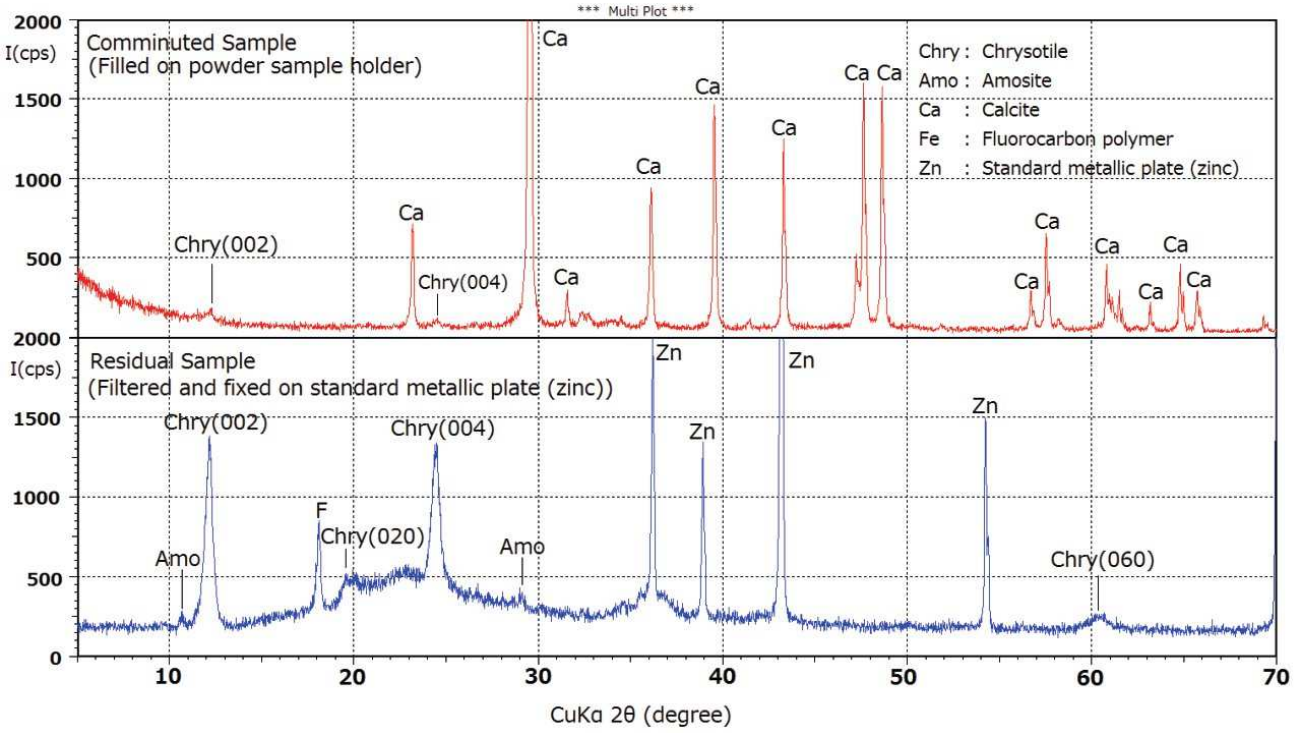


Figure D.1 — X-ray diffraction patterns of sprayed rock wool

The upper chart is for the untreated comminuted sample obtained using a conventional XRD measurement with a pressed powder sample holder.

The lower chart is for the residual sample (14 mg) obtained after formic acid treatment of a 100 mg comminuted sample (residual ratio: 14 %) and filtered on PTFE-coated glass fibre filters, then mounted on a zinc plate.

As shown in the lower chart, X-ray peaks from calcite were removed, the “possible peaks of chrysotile” were enhanced and small “possible peaks of amosite” became visible after matrix reduction using formic acid.

The chrysotile and amosite were quantified as 2,7 % and 0,2 %, respectively.

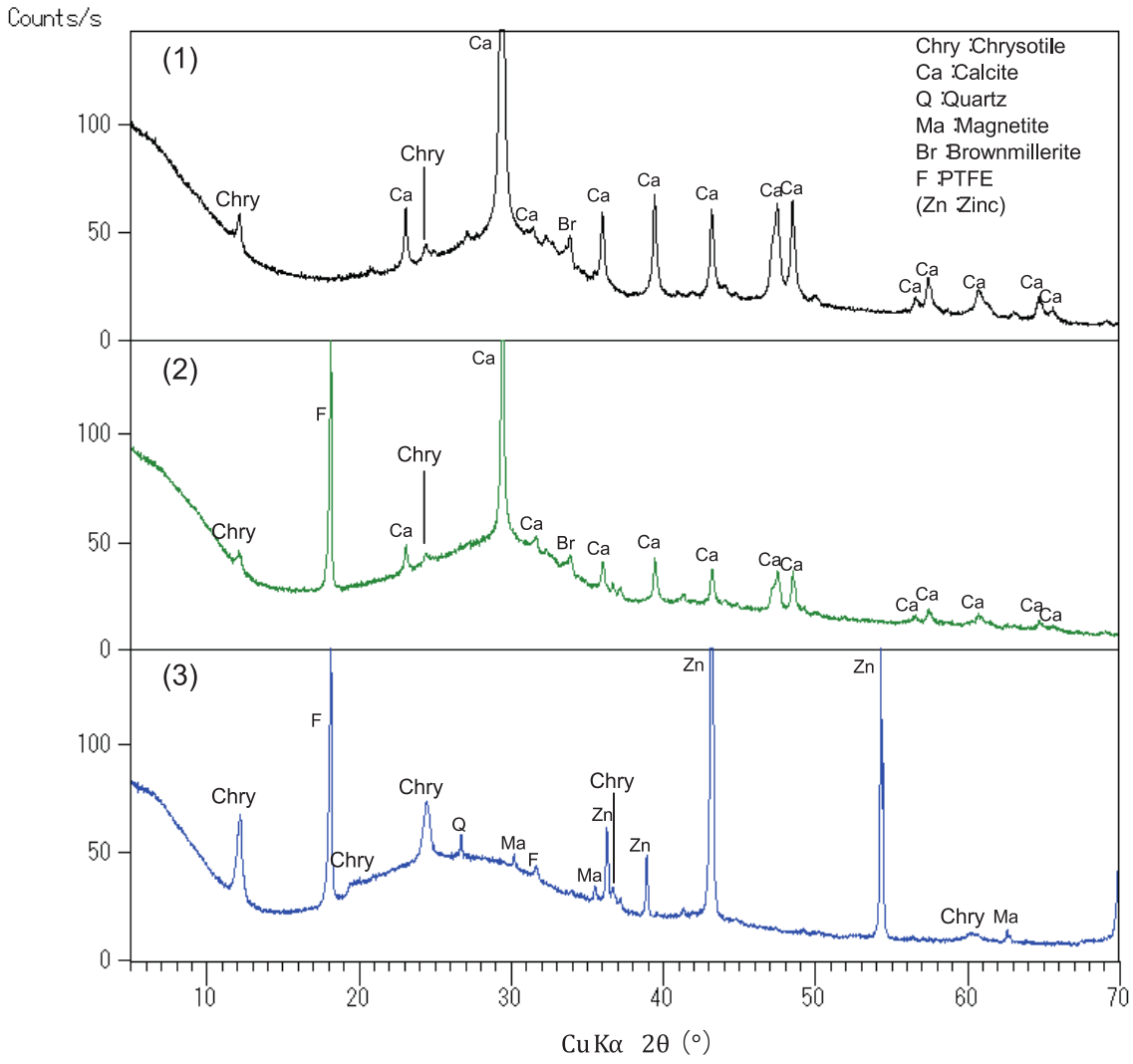


Figure D.2 — X-ray diffraction patterns of sprayed rock wool

Chart (1) shows a comminuted sample mounted in a conventional powder sample holder.

Chart (2) shows a 10 mg comminuted sample filtered on a PTFE-coated glass fibre filter and mounted on glass plate.

Chart (3) shows a 10 mg sub-residual sample taken from the residual sample (16 mg) after formic acid treatment of a 100 mg comminuted sample (residual ratio: 16 %), filtered on a PTFE-coated glass fibre filter, then mounted on a zinc plate.

As shown in chart (3), calcite and brownmillerite in a sprayed rock wool were dissolved and “possible chrysotile peaks” were enhanced by application of the matrix reduction method using formic acid. Chrysotile was quantified as 0,7 %.

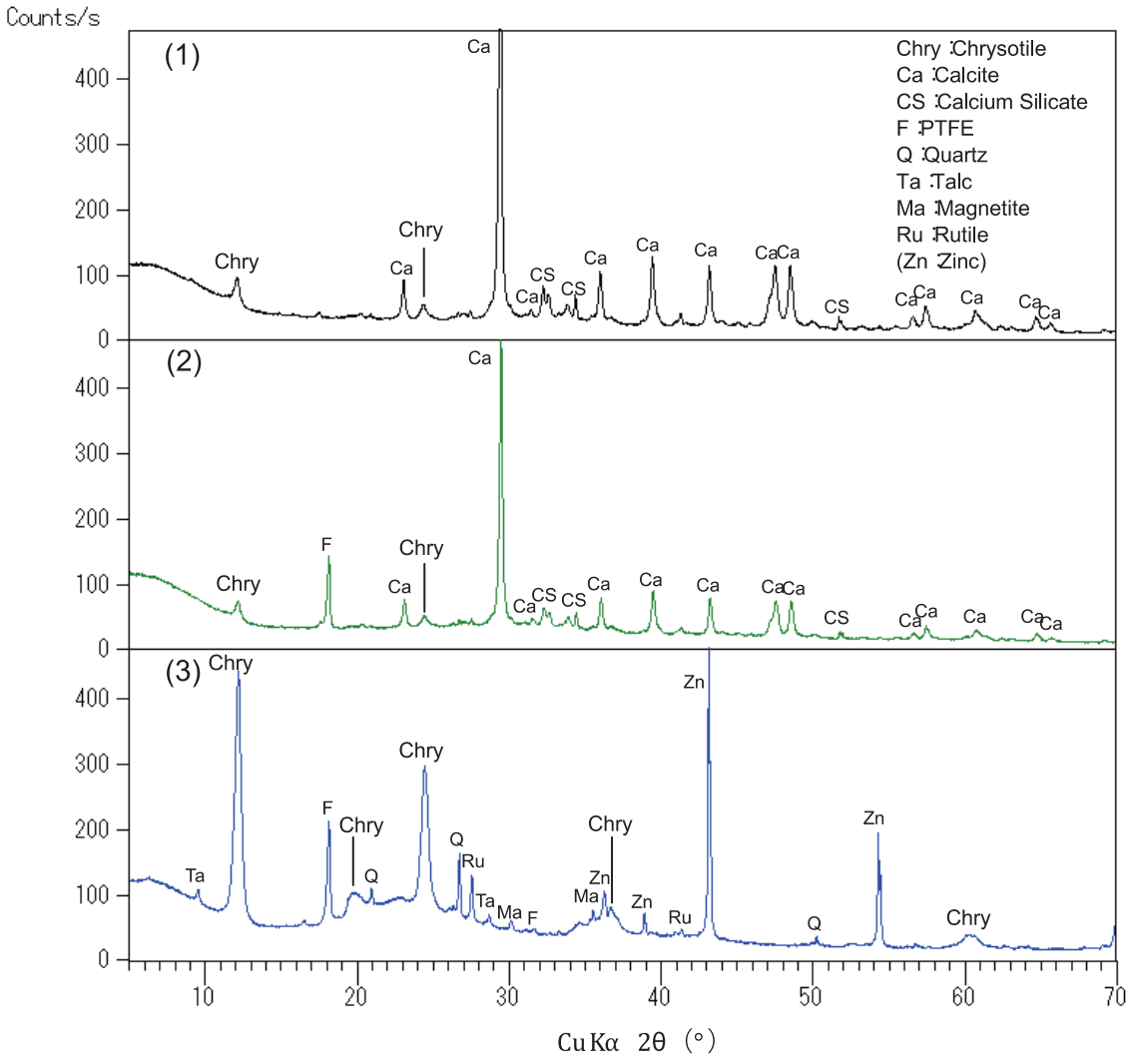


Figure D.3 — X-ray diffraction patterns of asbestos cement flat board

Chart (1) shows a comminuted sample mounted in a conventional powder sample holder.

Chart (2) shows a 10 mg comminuted sample filtered on a PTFE-coated glass fibre filter and mounted on a glass plate.

Chart (3) shows a 10 mg sub-residual sample taken from the residual sample (16 mg) after formic acid treatment of a 100 mg comminuted sample (residual ratio: 16 %), filtered on a PTFE-coated glass fibre filter, then mounted on a zinc plate.

As shown in chart (3), calcite and calcium silicate in a cement slate board were dissolved and "possible chrysotile peaks" were enhanced by application of the matrix reduction method using formic acid. Chrysotile was quantified as 5,8 %.

Annex E
(informative)

Range of typical detection limits and evaluation of uncertainty of quantitative XRD measurements by XRD method

E.1 Detection limits for asbestos

Table E.1 — Typical detection limits[1][13][14][15]

Chrysotile		
XRD peak (reflection)	12,1° (002)	24,3° (004)
Range (µg/cm^2)[a]	2,5 µg to 5 µg	5 µg to 10 µg
Amphibole asbestos		
XRD peak (reflection)	Ca 10,5° (110) (210[b])	Ca 28,4 to 29,0° (310[c])
Range (µg/cm^2)	2 µg to 5 µg	2,5 µg to 5 µg

[a] The effective area of filter is 2 cm^2.

[b] For anthophyllite.

[c] Varies depending on asbestos types such as (330) for crocidolite and (610) for anthophyllite.

E.2 Uncertainty estimation

Uncertainty estimation using the measured values from interlaboratory tests is described, which is in accordance with the experimental design type A7 in ISO 20988:2007.

E.2.1 Statistical analysis method for uncertainty

The instructions for the calculation of the uncertainty for this method are shown in Table E.2, which refers to ISO 20988:2007, B.9.

Table E.2 — Elements of uncertainty estimation for the interlaboratory comparison of identical systems

Element	Instruction
Problem specification	
Uncertainty parameter	Standard uncertainty on the level of y = 0,1 mg/10 mg to 2,0 mg/10 mg amount of chrysotile in a matrix. Expanded 95 % uncertainty on a level of y = 0,1 mg/10 mg to 2,0 mg/10 mg amount of chrysotile in a matrix.
Experimental design	Interlaboratory comparison of identical measurement system. An unknown, but fixed test sample of chrysotile is observed N = 5 times by K = 3 laboratories each using a single measuring system of the same type.
Input data	Series of observation $y(i,j)$ with j = 1 to N = 5 and i = 1 to K = 3
Reference values	$\overline{\overline{y}} = \sqrt{\frac{1}{K \times N}\sum_{i=1}^{K}\sum_{j=1}^{N} y(i,j)}$
Addition information	Reference value $\overline{\overline{y}}$ is treated as an unbiased estimate of the true value of the measurement.
Representativeness	The series of observations provided represent variations between the laboratories participating in the interlaboratory comparison. Each participating laboratory independently calibrated the XRD system in use before the test.
Data treatment	
Model equation	$y(k,j) = \overline{\overline{y}} + a(i) + e(i,j)$ with residual deviation $e(k,j) = y(i,j) - \overline{y(k)}$ laboratory bias $a(i) = \overline{y(\iota)} - \overline{\overline{y}}$ laboratory means $\overline{y(\iota)} = \frac{1}{N}\sum_{j=1}^{N} y(i,j)$
Variance equation	$\mathrm{var}(y) = u^2(\overline{\overline{y}}) + u^2(a) + u^2(e)$
Repeatability standard deviation	$u(e) = s_r = \sqrt{\frac{1}{K}\sum_{i=1}^{K} s^2(k)}$
Laboratory standard deviation	$s_L = \sqrt{\frac{1}{N-1}\sum_{j=1}^{N}\left(y(k,j) - \overline{y(k)}\right)^2}$
Interlaboratory variation	$u(a) = \sqrt{\frac{1}{K}\sum_{i=1}^{K}\left(\overline{y(k)} - \overline{\overline{y}}\right)^2}$
Standard uncertainty of reference value	$u(\overline{\overline{y}}) = \sqrt{\frac{1}{K}u^2(a)}$

Table E.2 *(continued)*

Element	Instruction
Results of uncertainty analysis	
Standard uncertainty of y	$u(y) = \sqrt{\frac{1}{K-1}\sum_{K=1}^{K}\left(\overline{y(\iota)} - \overline{\overline{y}}\right)^2 + s_r^2(y)}$
Number of degrees of freedom	$v \cong K - 1$ since $u(y) \cong u(a)$
Coverage factor	$k_{0,95}$ $k = 2,0$
Expanded uncertainty of y	$U(y) = k_{0,95} \cdot u(y)$
Range of application	$\min(y) \leq y \leq \max(y)$

E.2.2 Results of interlaboratory tests using test samples and evaluation of the uncertainty of the measurement

Test samples were prepared by mixing known amounts of chrysotile with feldspar (microcline) powder as the matrix. The microcline matrix chosen for the test samples was selected to simulate the X-ray mass absorption coefficient of that of the matrix that generally remains after matrix reduction of actual bulk materials. When the matrix reduction method is applied to building materials, some constituents, such as rock-wool, cement, calcium carbonate, etc., are dissolved by the formic acid treatment. The main residual materials would be quartz, silicate minerals and some amorphous materials. The X-ray mass absorption coefficients of these residual materials are smaller than those of cement or calcium carbonate, which were dissolved, and are comparable with those of silicate minerals. Therefore, feldspar (microcline) powder was selected as a suitable matrix to represent these silicate minerals.

Aliquots of 0,1 mg, 0,2 mg, 0,5 mg, 1,0 mg and 2,0 mg of chrysotile were mixed with 10 mg microcline powder, respectively, and these mixtures were transferred to the filters to be used for the XRD measurements. For these samples, matrix reduction was not used, in order to simulate the situation in which matrix reduction method is not effective and 100 % of the sample remains, i.e. the residual ratio is 100 %. These five test samples were each measured five times by three laboratories. The statistical method for calculation of uncertainty and the results of the interlaboratory tests are shown in Tables E.3 and E.4.

Table E.3 — Results of measurements of the test samples by the XRD

Level of concentration (j) (mg/10 mg)	Laboratories (i)		
	1	2	3
0,1	0,11	0,07	0,12
	0,09	0,08	0,10
	0,10	0,08	0,10
	0,11	0,08	0,12
	0,09	0,06	0,11
0,2	0,21	0,17	0,18
	0,18	0,16	0,16
	0,24	0,17	0,17
	0,21	0,16	0,16
	0,26	0,18	0,18

Table E.3 *(continued)*

Level of concentration (j) (mg/10 mg)	Laboratories (i)		
	1	2	3
0,5	0,59	0,47	0,45
	0,59	0,47	0,46
	0,54	0,43	0,44
	0,63	0,48	0,46
	0,62	0,45	0,45
1,0	1,11	0,84	0,93
	1,13	0,92	0,98
	1,18	0,96	1,03
	1,14	0,89	0,97
	1,23	0,95	1,05
2,0	2,09	1,73	1,84
	2,26	1,77	1,85
	2,17	1,85	1,95
	2,44	1,96	2,04
	2,23	1,78	1,84

Table E.4 — Results of estimated standard uncertainties by the measurement of test samples

Concentration level (i)	Mean value (mg/10 mg)	Repeatability standard deviation (mg/10 mg)	Laboratory variation (mg/10 mg)	Standard uncertainty $u(i)$ (mg/10 mg)	Expanded uncertainty (i) (mg/10 mg)
1	0,09	0,010	0,015	0,020 (20 %)	0,040 (44 %)
2	0,19	0,019	0,024	0,034 (18 %)	0,068 (36 %)
3	0,50	0,024	0,065	0,079 (16 %)	0,158 (31 %)
4	1,02	0,048	0,102	0,128 (13 %)	0,255 (25 %)
5	1,99	0,105	0,181	0,234 (12 %)	0,468 (24 %)

The standard uncertainty is estimated as 12 % to 20 % within the range of the mass fraction from 0,1 mg/10 mg (1 %) to 2 mg/10 mg (20 %). The expanded 95 % uncertainty is from 24 % to 44 %, within the range of the mass fraction from 0,1 mg/10 mg (1 %) to 2 mg/10 mg (20 %).

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