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Air quality — Bulk materials —

Part 2: Quantitative determination of asbestos by gravimetric and microscopical methods

Qualité de l'air — Matériaux solides —

Partie 2: Dosage quantitatif de l'amiante en utilisant les méthodes gravimétrique et microscopique

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Foreword

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

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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

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

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

— *Part 1: Sampling and qualitative determination of asbestos in commercial bulk materials*

— *Part 2: Quantitative determination of asbestos by gravimetric and microscopical methods*

The following part is under preparation:

— *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, 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.

Three varieties of asbestos found extensive commercial application. Chrysotile accounted for approximately 95 % of consumption, and therefore this is the variety that is encountered most frequently during analysis of samples. Amosite and crocidolite accounted for almost all of the balance, with a very small contribution from anthophyllite. Amosite was generally used as fireproofing or in thermal insulation products. Crocidolite was also used as fireproofing and thermal insulation products, but because it is highly resistant to acids, it also found application as a reinforcing fibre in acid containers such as those used for lead-acid batteries, and in some gaskets. Materials containing commercial anthophyllite are relatively rare, but it also has been used as a filler and reinforcing fibre in composite materials, and as a filtration medium. Tremolite asbestos and actinolite asbestos were not extensively used commercially, but they sometimes occur as contamination of other commercial minerals. Richterite asbestos and winchite asbestos occur at mass fractions between 0,01 % and 6 % in vermiculite formerly mined at Libby, Montana, USA. Vermiculite from this source was widely distributed and is often found as loose fill insulation and as a constituent in a range of construction materials and fireproofing.

While the asbestos mass fraction in some products can be very high and in some cases approach 100 %, in other products the mass fractions of asbestos used were significantly lower and often between 1 % and 15 %. In some ceiling tile panels, the mass fraction of asbestos used was close to 1 %. There are only a few known materials in which the asbestos mass fraction used was less than 1 %. Some adhesives, sealing compounds and fillers were manufactured in which asbestos mass fractions 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 mass fractions lower than 0,1 %.

ISO 22262-1 specifies procedures for collection of samples and qualitative analysis of commercial bulk materials for the presence of asbestos. A visual estimate of the asbestos mass fraction may also be made. While it is recognized that the accuracy and reproducibility of such estimates is very limited, for many of the types of materials being analysed these estimates are sufficient to establish that the mass fraction of asbestos in a manufactured product is, without doubt, well above any of the regulatory limits.

Because of the wide range of matrix materials into which asbestos was incorporated, microscopy alone cannot provide reliable analyses of all types of asbestos-containing materials in untreated samples. This part of ISO 22262 extends the applicability and limit of detection of microscopical analysis by the use of simple procedures such as ashing, acid treatment, sedimentation and heavy liquid density separation prior to microscopical examination.

A prerequisite for use of this part of ISO 22262 and subsequent parts of ISO 22262 is that the sample shall have been examined ISO 22262-1. ISO 22262 is for application by knowledgeable analysts who are familiar with the analytical procedures specified.[7][8][9][10]

Air quality — Bulk materials —

Part 2: Quantitative determination of asbestos by gravimetric and microscopical methods

1 Scope

This part of ISO 22262 specifies procedures for quantification of asbestos mass fractions below approximately 5 %, and quantitative determination of asbestos in vermiculite, other industrial minerals and commercial products that incorporate these minerals.

This part of ISO 22262 is applicable to the quantitative analysis of:

a) any material for which the estimate of asbestos mass fraction obtained using ISO 22262-1 is deemed to be of insufficient precision to reliably classify the regulatory status of the material, or for which it is considered necessary to obtain further evidence to demonstrate the absence of asbestos;

b) resilient floor tiles, asphaltic materials, roofing felts and any other materials in which asbestos is embedded in an organic matrix;

c) wall and ceiling plasters, with or without aggregate;

d) mineral products such as wollastonite, dolomite, calcite, talc or vermiculite, and commercial products containing these minerals.

This part of ISO 22262 is primarily intended for application to samples in which asbestos has been identified at estimated mass fractions lower than approximately 5 % by weight. It is also applicable to samples that may contain asbestos at low mass fractions incorporated into matrix material such that microscopical examination of the untreated sample is either not possible or unreliable. An annex gives recommendations for the analysis of each type of material that may contain asbestos.

It is not the intent of ISO 22262 to provide instruction in the fundamental microscopical and analytical techniques.

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 13794:1999, *Ambient air — Determination of asbestos fibres — Indirect-transfer transmission electron microscopy method*

3 Terms and definitions

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

3.1
acicular
shape shown by an extremely slender crystal with cross-sectional dimensions which are small relative to its length, i.e. needle-like

[SOURCE: ISO 13794:1999, definition 2.1]

3.2
amphibole
group of rock-forming ferromagnesium silicate minerals, closely related in crystal form and composition, and having the nominal formula: $A_{0-1}B_2C_5T_8O_{22}(OH,F,Cl)_2$, where

A = K, Na;

B = Fe^{2+}, Mn, Mg, Ca, Na;

C = Al, Cr, Ti, Fe^{3+}, Mg, Fe^{2+};

T = Si, Al, Cr, Fe^{3+}, Ti

[SOURCE: ISO 13794:1999, definition 2.2]

Note 1 to entry: In some varieties of amphibole, these elements can be partially substituted by Li, Pb, or Zn. Amphibole is characterized by a cross-linked double chain of Si-O tetrahedra with a silicon:oxygen ratio of 4:11, by columnar or fibrous prismatic crystals and by good prismatic cleavage in two directions parallel to the crystal faces and intersecting at angles of about 56° and 124°.

3.3
amphibole asbestos
amphibole in an asbestiform habit

[SOURCE: ISO 13794:1999, definition 2.3]

3.4
anisotropy
state or quality of having different properties along different axes

EXAMPLE An anisotropic transparent particle can show different refractive indices with the vibration direction of incident light.

3.5
asbestiform
specific type of mineral fibrosity in which the fibres and fibrils possess high tensile strength and flexibility

[SOURCE: ISO 13794:1999, definition 2.6]

3.6
asbestos
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

[SOURCE: ISO 13794:1999, definition 2.7]

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 (see Reference [11]), are also found in some products such as vermiculite and talc.

3.7
asbestos point
where the point coincides with an asbestos fibre in point counting

3.8
aspect ratio
ratio of length to width of a particle

[SOURCE: ISO 13794:1999, definition 2.10]

3.9
birefringence
maximum difference between refractive indices due to double refraction

3.10
chrysotile
fibrous mineral of the serpentine group which has the nominal composition:

$$Mg_3Si_2O_5(OH)_4$$

[SOURCE: ISO 13794:1999, definition 2.13]

Note 1 to entry: Most natural chrysotile deviates little from this nominal composition. In some varieties of chrysotile, minor substitution of silicon by Al^{3+} may occur. Minor substitution of magnesium by Al^{3+}, Fe^{2+}, Fe^{3+}, Ni^{2+}, Mn^{2+} and Co^{2+} may also be present. Chrysotile is the most prevalent type of asbestos.

3.11
cleavage
breaking of a mineral along one of its crystallographic directions

[SOURCE: ISO 13794:1999, definition 2.14]

3.12
cleavage fragment
fragment of a crystal that is bounded by cleavage faces

[SOURCE: ISO 13794:1999, definition 2.15]

Note 1 to entry: Crushing of non-asbestiform amphibole generally yields elongated fragments that conform to the definition of a fibre, but rarely have aspect ratios exceeding 30:1.

3.13
crossed polars
state in which the polarization directions of the polars (polarizer and analyser) are mutually perpendicular

[SOURCE: ISO 10934-1:2002, definition 2.117.2]

3.14
dispersion
variation of refractive index with wavelength of light

[SOURCE: ISO 7348:1992, definition 05.03.26]

3.15
dispersion staining
effect produced when a transparent object is immersed in a surrounding medium, the refractive index of which is equal to that of the object at a wavelength in the visible range, but which has a significantly higher optical dispersion than the object

Note 1 to entry: Only the light refracted at the edges of the object is imaged, and this gives rise to colours at the interface between the object and the surrounding medium. The particular colour is a measure of the wavelength at which the refractive index of the object and that of the medium are equal.

3.16
empty point
where the point does not coincide with any particle or fibre in point counting

3.17
energy dispersive X-ray analysis
measurement of the energies and intensities of X-rays by use of a solid-state detector and multi-channel analyser system

[SOURCE: ISO 13794:1999, definition 2.22]

3.18
fibril
single fibre of asbestos which cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearances

[SOURCE: ISO 13794:1999, definition 2.25]

3.19
fibre
elongated particle which has parallel or stepped sides

[SOURCE: ISO 13794:1999, definition 2.26]

Note 1 to entry: For the purposes of this part of ISO 22262, a fibre is defined to have an aspect ratio equal to or greater than 3:1.

3.20
fibre bundle
structure composed of parallel, smaller diameter fibres attached along their lengths

[SOURCE: ISO 13794:1999, definition 2.27]

Note 1 to entry: A fibre bundle may exhibit diverging fibres at one or both ends.

3.21
habit
characteristic crystal growth form, or combination of these forms, of a mineral, including characteristic irregularities

[SOURCE: ISO 13794:1999, definition 2.30]

3.22
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

3.23
isotropic
having the same properties in all directions

[SOURCE: ISO 14686:2003, definition 2.23]

3.24
matrix
material in a bulk sample within which fibres are dispersed

3.25
non-empty point
where a point coincides with either a particle or an asbestos fibre in point counting

3.26
point
in point counting, location on the sample where a record is made as to whether the location is occupied by a particle or an asbestos fibre, or whether the location is unoccupied

3.27
point counting
procedure in which random locations are examined on a sample to determine whether each location is occupied by a particle or an asbestos fibre, or is unoccupied, and each type of event is enumerated

3.28
polarized light
light in which the vibrations are partially or completely suppressed in certain directions at any given instant

[SOURCE: ISO 10934-1:2002, definition 2.88.1]

Note 1 to entry: The vector of vibration may describe a linear, circular or elliptical shape.

3.29
polarizer
polar placed in the light path before the object

[SOURCE: ISO 10934-1:2002, definition 2.117.4]

3.30
polar
device which selects plane-polarized light from natural light

[SOURCE: ISO 10934-1:2002, definition 2.117]

3.31
refractive Index
n
ratio of the speed of light (more exactly, the phase velocity) in a vacuum to that in a given medium

[SOURCE: ISO 10934-1:2002, definition 2.124]

3.32
serpentine
group of common rock-forming minerals having the nominal formula:

$$Mg_3Si_2O_5(OH)_4$$

[SOURCE: ISO 13794:1999, definition 2.39]

3.33
twinning
occurrence of crystals of the same species joined together at a particular mutual orientation, and such that the relative orientations are related by a definite law

[SOURCE: ISO 13794:1999, definition 2.41]

3.34
unopened fibre
large diameter asbestos fibre bundle that has not been separated into its constituent fibrils or fibres

[SOURCE: ISO 13794:1999, definition 2.42]

4 Abbreviated terms

ED	electron diffraction
EDXA	energy dispersive X-ray analysis
MEC	mixed esters of cellulose
PC	polycarbonate
PLM	polarized light microscopy
RI	refractive index
SAED	selected area electron diffraction
SEM	scanning electron microscope
TEM	transmission electron microscope

5 Determination of analytical requirements

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”, through > 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 A 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 A 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 use Annex A 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 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		

6 Range

When this part of ISO 22262 is applied to a suitably prepared sample analysed by PLM, SEM or TEM, the target range is from less than 0,001 % to 5 %. However, there is no upper limit to the concentration of asbestos that can be determined. The lower end of the range depends on the proportion of non-asbestos constituents that can be removed by gravimetric methods, and the amount of the remaining material that can be examined.

7 Limit of quantification

The limit of quantification using this part of ISO 22262 is defined as the detection and identification of one fibre or fibre bundle in the amount of sample examined. The limit of quantification that can be achieved depends on:

a) the nature of the matrix of the sample;

b) the size of the asbestos fibres and bundles;

c) the use of appropriate sample preparation and matrix reduction (gravimetric) procedures;

d) the amount of time expended on examination of the sample; and,

e) the method of analysis used, PLM, SEM or TEM.

With appropriate matrix reduction procedures that are selected based on the nature of the sample, the limit of quantification can be lower than 0,001 %.

8 Principle

A known weight of the material is heated in a furnace to a temperature of 450 °C ± 10 °C to remove organic materials. Depending on the nature of the sample, the residue from the heating is treated with either hydrochloric or sulphuric acid to dissolve acid-soluble constituents. If appropriate, water sedimentation is then used to separate aggregate fragments and particles. For sensitive quantification of amphibole, some materials may require a refluxing treatment in acid, followed by a reflux treatment in sodium hydroxide. Alternatively, amphibole can be separated from many other constituents of lower densities by centrifugation in a heavy liquid. The proportion of asbestos in the residue from these treatments is then determined by appropriate PLM, SEM or TEM techniques.

9 Safety precautions

Handling asbestos is regulated by many jurisdictions, and regulations often specify a variety of procedures to ensure that individuals performing work and those in close proximity are not exposed to excessive concentrations of airborne asbestos fibres.

Care is necessary during sampling of materials that may contain asbestos, and precautions should be taken to avoid creating and inhaling airborne asbestos particles when handling materials suspected of containing asbestos. If the handling instructions in this clause are followed, it may be assumed that there is no substantial release of fibres. In exceptional cases, more extensive precautions may be necessary to prevent the release of airborne fibres.

Some of the procedures described use hazardous chemicals. These chemicals should be handled in accordance with safety requirements. Ashing of some materials also may result in discharge of toxic gases. Accordingly, the muffle furnace should be appropriately vented.

10 Apparatus

10.1 Dust extract hood. Handling and manipulation of bulk materials suspected to contain asbestos shall be performed in a suitable dust extract hood, so that neither the analyst nor the laboratory environment is exposed to airborne asbestos fibres.

10.2 Sample comminution equipment. An agate mortar and pestle, or a mill, is required for grinding of samples to suitable sizes for PLM examination.

10.3 Analytical balance, with a readability of 0,000 1 g or lower is required.

10.4 Muffle furnace, for ashing of samples to remove interfering organic constituents, a muffle furnace with a minimum temperature range up to 800 °C, with a temperature stability of ± 10 °C is required.

10.5 Slide warmer, for drying of samples and preparation of microscope slides. Alternatively, an oven may be used.

10.6 Glass filtration assembly (47 mm diameter), with 250 ml reservoir and glass frit base, with side-arm vacuum filtration flask.

10.7 Glass filtration assembly (25 mm diameter), with 15 ml reservoir and glass frit base, with side-arm vacuum filtration flask.

10.8 Side-arm vacuum flask, 1000 ml volume.

10.9 Water aspirator, or other vacuum source for filtrations.

10.10 Magnetic stirrer, for removal of acid-soluble interfering constituents, a magnetic stirrer with a glass or plastic-coated magnetic stir bar.

10.11 Glass reflux condenser system. A borosilicate glass reflux system, consisting of a 250 ml round-bottomed flask with a vertical, water-cooled borosilicate glass condenser and a mantle heater is required for treatment of samples by the sequential refluxing in acid and alkali procedure.

10.12 Centrifuge. A bench-top centrifuge is required for separation of insoluble residues during procedures including sequential refluxing in acid and alkali, or for separation of amphiboles by centrifugation in a heavy liquid.

10.13 Glass centrifuge tubes, 15 ml volume.

10.14 Sink-Float or **density bottle**. Sink-Float Standard[1)], density 2 750 kg/m^3 ± 5 kg/m^3 (2,75 g/cm^3 ± 0,005 g/cm^3) at 23 °C, for measurement of heavy liquid density. Alternatively, a 10 ml density bottle may be used.

10.15 Equipment for microscopical analysis. Appropriate microscopy equipment as specified in ISO 22262-1, for analysis of residues from the gravimetric reduction procedures.

10.16 General laboratory supplies. The following supplies and equipment, or equivalent, are required.

10.16.1 **Glassine paper sheets**, approximately 15 cm × 15 cm, for examination of samples.

10.16.2 **Scalpel holder and replacement disposable scalpel blades**.

10.16.3 **Sampling utensils, including tweezers, needles and spatulas**.

10.16.4 **Erlenmeyer flasks**, 250 ml.

10.16.5 **Crucibles**, silica or glazed porcelain, with lids.

10.16.6 **Petri dishes**.

10.16.7 **Pipettes and disposable pipette tips**, 0 µl - 1 000 µl and 0 µl - 10 µl .

10.16.8 **Disposable pipettes**.

10.16.9 **Disposable plastic beakers**, 50 ml and 1 000 ml.

10.16.10 **Borosilicate glass rods**, 5 mm diameter, approximately 20 cm in length.

10.16.11 **Polycarbonate filters**, 0,4 µm pore size, 47 mm and 25 mm diameter.

10.16.12 **MEC filters**, 0,45 µm porosity, 47 mm and 25 mm diameter.

10.16.13 **Laboratory equipment and supplies for microscopical analysis according to** ISO 22262-1.

1) Sink-Float Standard is the trade name of a product supplied by Cargille Laboratories. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

11 Reagents

11.1 Distilled or de-ionized water, filtered through a 0,22 µm porosity MEC filter.

11.2 Concentrated hydrochloric acid, reagent grade.

11.3 Concentrated sulphuric acid, reagent grade.

11.4 Glacial acetic acid, reagent grade.

11.5 Sodium hydroxide, pellets, reagent grade.

11.6 Dimethyl formamide, reagent grade.

11.7 Ethanol, denatured.

11.8 Lithium metatungstate solution, density approximately 2 950 kg/m^3.

12 Sample size and homogeneity

12.1 Sample size

Prior to analysis, it is necessary to take into account the homogeneity of the material, and to ensure that the sample is of a sufficient size that it is representative of the material under investigation. If inspection to the unaided eye and using a binocular microscope shows that the material is finely divided and homogeneous, or if the nature of the material is recognized as homogeneous from previous knowledge, a minimum sample size of approximately 5 cm^3 generally provides sufficient material for analysis.

12.2 Representative sample

A wide range of asbestos-containing materials was used in the past. Experience is very valuable in the selection of the materials to be sampled, and sampling can be facilitated by the use of all available prior knowledge about the materials or components from which the sample is being collected. For meaningful quantification, it is most important that the sample collected is representative of the composition of the product with respect to its asbestos content. Although many asbestos-containing materials may seem to be homogeneous when examined by the unaided eye, they can be quite inhomogeneous in the microscopic size range. This is particularly the case for materials such as texture coats, in which the fragments of aggregate can be significantly larger than the other constituents of the material.

In some types of material, particularly those that have been mixed at a building site, rather than a commercial product manufactured and mixed under a formulation and quality control procedure, the asbestos may not be distributed homogeneously within the material. For these types of materials, a larger sample shall be analysed to ensure that the reported asbestos mass fraction is representative of the material. It is particularly important to consider sample size requirements if the asbestos is present as natural contamination of a material, rather than as a deliberate addition made in a manufactured product.

It is recommended that a portion of the sample always be archived, because further examination of the sample is often the only way in which potential questions and discrepancies can be resolved.

13 Methods for gravimetric matrix reduction

13.1 General

The objective of gravimetric matrix reduction is to remove as much of the non-asbestos constituents of the sample being analysed as possible, so that any asbestos present represents a higher mass fraction in the final residue after the treatment. The simplest methods available are removal of organic constituents by ashing, removal of soluble constituents by dissolution in acid, and separation of larger aggregate particles by sedimentation. If the analysis is to determine amphibole varieties only, successive refluxing in acid and alkali can be effective in removing some silicate minerals, leaving any amphibole minerals unaltered. Amphibole varieties can also be separated from other constituents by density separation. Depending on the nature of the sample being analysed, any of the individual methods can be used, or a combination of several methods can be used in sequence.

13.2 Data recording

Figure 1 shows an example of a suitable form for recording of analytical data. Depending on the nature of the sample and the combination of gravimetric methods that are required for specific types of sample matrix, it may be necessary to modify the form.

BULK SAMPLE ANALYSIS: ASHING AND ACID EXTRACTION GRAVIMETRY

SAMPLE:_____ **SAMPLE NO:** _____

_____ **CRUCIBLE NO:** _____

_____ **DATE:** _____

_____ **ANALYST:** _____

INITIAL WEIGHTS			
Weight of crucible			
Weight of crucible + Sample			
Weight of sample			
ASHING			
Weight of crucible + Ash			
Weight of ash			
Weight loss during ashing			
Percent organic and water			
ACID TREATMENT			
Weight of filter			
Weight of filter + Residue			
Weight of residue			
Weight loss during acid treatment			
Percent acid-soluble materials			
Percent total residue			
PLM EXAMINATION			
Type of asbestos in residue			
Percent asbestos in residue			
Percent asbestos in sample			
Description of other components in residue			
TEM/SEM EXAMINATION			
Type of asbestos in residue			
Percent asbestos in residue			
Percent asbestos in sample			
Description of other components in residue			

COMMENTS

Effervescence:

REPORT

ASBESTOS (% in residue)

Figure 1 — Example of form for recording of gravimetric and analytical data

13.3 Selection and pre-treatment of a representative sub-sample

13.3.1 General

Prior to analysis, ensure that the sample is dry. The amount of bulk material to be used for the analysis is optional. However, it is important to take into account the results of the prior analysis using ISO 22262-1 to ensure that:

a) the sub-sample size is sufficient to ensure that the sub-sample is representative of the whole material;

b) the mass of the residue remaining after all of the proposed separation steps is sufficiently large for it to be weighed with sufficient accuracy (approximately 1 % accuracy is desirable);

c) the sub-sample is free from any paint that may be present on or in the original sample.

NOTE 1 Using this part of ISO 22262, few bulk materials result in residues lower than 2 % of the original sample weight, unless they consist of almost all organic constituents or they are gypsum or lime plasters with only trace amounts of insoluble materials.

NOTE 2 Use of a higher starting weight ensures that the effects of inhomogeneities in the distribution of asbestos in the original material are reduced. These inhomogeneities generally become more serious at lower asbestos mass fractions in the original material, and particularly when any asbestos present is a natural contaminant.

The appropriate weight for the sub-sample and any pre-treatment required depends on the nature of the sample.

13.3.2 Plasters without aggregate

These materials consist primarily of gypsum, calcium carbonate and calcium hydroxide, sometimes with starch, and if they contain asbestos, it is usually homogeneously distributed. Use a minimum sub-sample weight of approximately 0,1 g. Sub-samples of up to 1 g can be analysed satisfactorily. Grind the sub-sample to a powder using a mortar and pestle.

13.3.3 Plasters with aggregate

These plasters consist primarily of gypsum, calcium carbonate, calcium hydroxide, large stone aggregate, and sand, often with animal hair or jute fibres, and sometimes low mass fractions of chrysotile. If there appears to be no large stone aggregate, use a minimum sub-sample of approximately 0,5 g. If large stone aggregate (2 mm to 5 mm in dimension) is present, a minimum sub-sample weight of approximately 5 g is necessary. Lightly grind the sub-sample using a mortar and pestle to break up the aggregates of particles and fragments so that subsequent ashing and chemical treatments will be efficient. Since separation of major proportions of the non-asbestos components by sedimentation is an essential feature in the analysis of this type of sample, it is important to not reduce the particle sizes any more than absolutely necessary. Sub-samples from samples received as pulverized material should be taken from the original sample by cone and quarter methods.

13.3.4 Cements with and without aggregate

Portland cement plasters present serious problems in later parts of the procedure, so the sub-sample weight shall be limited to no greater than 1 g. These materials are very hard. Grind the material to separate the aggregates of particles and fragments.

13.3.5 Floor tiles

Use a clean scalpel to shave off thin layers of the material. Take the shavings from the full cross-section of the material, by cutting perpendicular to the plane of the sheet, avoiding any adhesive, if present, on the under-side, and also avoiding any sealant or debris on the upper surface. This ensures that the

sample is representative of all of the layers in the material if it is a layered structure. Use a minimum sub-sample weight of approximately 0,3 g of shavings.

13.3.6 Asphaltic materials without aggregate

Select a sub-sample that represents the entire thickness of the asphaltic material. A minimum sub-sample weight of approximately 0,5 g is recommended

13.3.7 Asphaltic materials with aggregate

Any asbestos present in these materials is incorporated in the asphaltic constituent, and so the reported asbestos mass fraction from the analysis is critically dependent on the relative amounts of asphalt and aggregate in the sub-sample. If the aggregate size does not exceed approximately 1 cm, use a minimum sub-sample weight of approximately 10 g. Alternatively, quantification of asbestos may be based on the weight of the asphaltic constituent only, which can be obtained from the weight loss observed during ashing. In this case, a lower sub-sample weight may be used. However, whether this approach is acceptable depends on the precise language of the applicable regulatory control limit.

NOTE As guidance for calculating the sub-sample weight required, this type of material generally contains approximately 5 % of asphalt.

13.3.8 Caulkings, mastics, putties, groutings, wall joint compounds

These materials are manufactured products prepared under quality control, and they can be assumed to be homogeneous. With the exception of mastics, a minimum sub-sample weight of approximately 0,3 g is recommended. Where possible, higher sub-sample weights should be used for mastics, since if they do not contain asbestos, the residue after ashing may be insufficient for accurate weighing.

13.3.9 Cellulosic materials

Use a minimum sub-sample weight of approximately 0,5 g.

13.3.10 Texture coats

Use a minimum sub-sample weight of approximately 0,5 g. Texture coats are frequently covered by a thick coat of paint, and it is often difficult to obtain sufficient material for analysis.

13.4 Removal of organic materials by ashing

13.4.1 General

If it is known from prior experience that the organic content of the material is not significant and does not compromise the analysis, then the ashing step may be omitted. In this case, the sub-sample is weighed and taken directly to the acid dissolution step.

Some materials contain starch, which can compromise filtration of the final residue; it is therefore recommended that all previously uncharacterized samples be ashed.

Ashing of the sample at a temperature of 450 °C for a period of more than 4 h removes the organic constituents from many materials with very little effect on the optical properties of chrysotile. Although the colour and optical properties of amosite and crocidolite are altered by this oxidation treatment, many of the fibres can often still be identified by PLM. The optical properties of tremolite, actinolite, anthophyllite and richterite/winchite are almost unaffected by this treatment. The heat treatment does not affect the composition of any of the asbestos varieties, and they can all be identified by electron microscopy after the treatment.

Use a muffle furnace temperature of 450 °C for routine work. Removal of organic materials from some matrices may require a longer period of heating up to 10 h. If necessary, the temperature of the muffle

furnace may be increased to 485 °C. The temperature shall not exceed 500 °C, because chrysotile begins to degrade above this temperature.

Take appropriate precautions to prevent exposure to toxic fumes that are released when samples containing polyvinyl chloride or asphaltic materials are treated in the muffle furnace.

13.4.2 Procedure

Label a glazed porcelain or fused silica crucible with a heat-resistant marker, and weigh it. Place the sub-sample of the bulk material into the crucible, and weigh it again to obtain the weight of the sub-sample. Cover the crucible with a crucible lid and place it in a muffle furnace at a temperature of 450 °C ± 10 °C for a minimum period of 4 h. Remove the crucible from the muffle furnace and allow it to cool to room temperature. Remove the crucible lid and weigh the crucible with its contents. If inspection of the contents of the crucible indicates that the removal of organic materials is incomplete, return the crucible to the muffle furnace for additional treatment.

13.5 Acid treatment and sedimentation procedures

13.5.1 General

Matrix constituents such as calcite, gypsum and mineral wool are soluble in hydrochloric acid. These constituents also often constitute a large proportion of the sample mass. Stirring a sample in 2 mol/l hydrochloric acid for 15 min removes many such matrix constituents, and this improves the ability to identify and quantify asbestos. The acid treatment slightly reduces the refractive indices of chrysotile, and this has to be considered when identifying chrysotile by PLM. This acid treatment does not affect the optical properties of any of the other asbestos varieties.

NOTE Other acids such as acetic acid or formic acid are effective in dissolving gypsum and calcite, but they are not effective in removing other materials such as mineral wool or dolomite.

After acid treatment, in some materials large sizes of aggregate or sand remain that can be separated from the acid suspension by sedimentation or flotation. A large proportion of constituents such as exfoliated vermiculite or expanded perlite can be separated by flotation. Sand or aggregate fragments sediment from aqueous suspension very much more rapidly than most of the asbestos, and in some samples a large proportion of the sand or aggregate can be separated from the fraction that contains any asbestos.

Samples that contain acid-soluble constituents, with or without insoluble aggregate, are appropriate for the procedure in 13.5.2. Resilient floor tile is a special case because the frequent presence of dolomite as a major constituent requires the use of concentrated hydrochloric acid. The procedure for resilient floor tile is specified in 13.5.3.

13.5.2 Procedure for acid treatment of samples containing soluble constituents, with or without insoluble aggregate

13.5.2.1 Dissolve the acid-soluble and water-soluble components

Transfer the ashed contents of the crucible to a 200 ml Erlenmeyer (conical) flask. Add 100 ml of 2 mol/l hydrochloric acid and a polytetrafluoroethylene (PTFE)-coated magnetic stirring bar. Place on a magnetic stirrer for a period of 15 min.

Ensure that excess acid is used, so that all acid-soluble materials will be dissolved.

13.5.2.2 Weigh a polycarbonate filter and set up filtration systems

Weigh a 47 mm diameter, 0,4 µm pore size polycarbonate filter, and place it into a labelled Petri dish. Assemble a 47 mm diameter vacuum filtration system and install the pre-weighed, 47 mm diameter, 0,4 µm pore size polycarbonate filter. Assemble a 25 mm diameter vacuum filtration system and,

depending on the proposed type of subsequent analysis, install either a 0,45 µm nominal porosity MEC filter for PLM point counting or a 0,4 µm pore size PC filter with a 5 µm MEC back-up diffuser filter for SEM or TEM examination.

If the sub-sample being analysed contains Portland cement, filtration of the final residue may be very slow. If Portland cement is known to be present, use a PC filter of 0,8 µm pore size for the filtration.

13.5.2.3 Separate the sedimented, floating and suspended particles

This step is critical and requires some manual dexterity. The objective is to keep the asbestos fibres suspended in the liquid, while allowing the heavy components such as sand and aggregate to settle out, or light fractions such as expanded perlite or exfoliated vermiculite to float.

a) If there appears to be no floating perlite or vermiculite, and there also appears to be no sediment of large aggregate particles, proceed directly to 13.5.2.4.

b) If floating perlite or vermiculite is present, before proceeding further, add distilled water until the floating fraction is brought to the rim of the Erlenmeyer flask. Using a clean spatula, remove as much of the floating fraction as possible from the surface of the liquid, and place it in a pre-weighed 50 mm plastic Petri dish. Place the Petri dish on a slide warmer and allow the material to dry.

c) If Step b) was necessary, allow the flask to stand for several minutes and then pour approximately 50 % of the supernatant liquid into a clean 1 000 ml beaker. If step b) was not necessary, proceed directly to d)

d) Swirl the liquid in the flask with a circular motion to re-suspend the settled particles. Hesitate until the larger particles have settled and then pour most of the supernatant liquid into the beaker. Add 150 ml of distilled water to the Erlenmeyer flask and repeat the sedimentation and removal of supernatant liquid. Repeat again with an additional 150 ml of distilled water.

e) Using a wash bottle with distilled water, wash out the sedimented material from the Erlenmeyer flask into a 100 mm Petri dish. Decant the water into the 1 000 ml beaker, and then place the Petri dish on to a slide warmer and allow the sediment to dry. Drying may be accelerated by rinsing the sediment with a small volume of ethanol, and decanting the ethanol into the 1 000 ml beaker.

f) Add distilled water to the beaker to bring the volume to a known value (700 ml is usually satisfactory).

13.5.2.4 Prepare MEC or PC filters to be used for point counting, SEM or TEM examination

Thoroughly disperse the particulate in the suspension in the 1 000 ml beaker (or the Erlenmeyer flask if there are no large aggregate fragments visible) by stirring and blowing air through a pipette into the suspension. Withdraw an aliquot for filtration through the 25 mm diameter MEC or PC filter. The particulate loading on the filter shall be suitable for the type of analysis proposed. In general, aliquots of between 0,5 ml and 4 ml have been found to be suitable for PLM point counting, but lower aliquots are usually necessary to obtain appropriate particulate loadings for SEM or TEM examination. Before filtration, dilute each aliquot to a volume of more than 5 ml with distilled water to ensure a uniform particulate deposit on the filter. Prepare a minimum of four filters from four separate aliquots. Place each filter into a labelled Petri dish and allow the filters to dry on the slide warmer.

13.5.2.5 Filter the balance of the suspension

Filter the balance of the suspension through the 0,4 µm or 0,8 µm pore size polycarbonate filter. Remove the polycarbonate filter from the filtration system and place it into its labelled Petri dish. Place the Petri dish on to the slide warmer and allow the filter to dry.

13.5.2.6 Weigh the separated fractions

Before weighing the sedimented material and the polycarbonate filter with the deposit, examine them using a stereo-microscope. Using forceps, remove any very large fibre bundles visible in the sedimented

material and place them into a pre-weighed container (a small formed piece of aluminium foil is satisfactory). If the filter deposit exhibits any large particles, which would make sub-sampling of the filter deposit unrepresentative, remove large non-asbestos material and transfer it to the sedimented material, and remove any large asbestos fibres and add them to the pre-weighed container with any fibres removed from the sedimented material. Transfer the sedimented material to a pre-weighed 50 mm plastic Petri dish. Weigh the floated material (if applicable), the sedimented material, the manually picked fibres and the filter with the final residue deposit. Proceed to Clause 14.

13.5.3 Procedure for resilient floor tile

13.5.3.1 Treat the residual ash with concentrated hydrochloric acid

Weigh a 47 mm diameter, 0,4 µm pore size polycarbonate capillary-pore filter, and place it in a labelled Petri dish. Set up a 47 mm diameter filtration assembly, using a vacuum flask and water aspirator. Install the polycarbonate filter in the filtration assembly.

Transfer the residual ash from the crucible to an agate mortar, add 0,5 ml of freshly distilled water, and grind the material to disperse it. Slowly add 2 ml of concentrated hydrochloric acid. Calcite, dolomite and ankerite will dissolve with evolution of carbon dioxide. Calcite will dissolve very rapidly, but any dolomite or ankerite will dissolve slowly. If evolution of gas is observed to be slow, continue to grind the material in the mortar at intervals in order to facilitate more rapid dissolution. Complete dissolution of dolomite or ankerite may require 15 min or longer, but the residual ash shall not be exposed to the concentrated acid for longer than 20 min. When no more evolution of gas is observed, immediately dilute the suspension with 10 ml of freshly distilled, filtered water.

13.5.3.2 Filter the suspension

Apply the vacuum to the filtration assembly, pour the suspension into the filter reservoir, and rinse the mortar and pestle into the reservoir using about 50 ml of freshly distilled water. Allow the filtration to proceed to completion. Rinse the mortar and pestle and filtration reservoir with approximately 10 ml of distilled water, and allow the filtration to proceed to completion. Repeat the rinsing operation a second time in order to ensure that the entire residue has been transferred and that all of the hydrochloric acid has been washed out of the filtered deposit.

13.5.3.3 Dry and weigh the polycarbonate filter

Remove the polycarbonate filter and transfer it to the labelled Petri dish. A cellulose pad should be placed in the bottom of the dish to prevent the polycarbonate filter from sticking to the base of the Petri dish as it dries. The filtration assembly should be washed immediately, so that residual traces of chrysotile are not allowed to dry on the unit.

Allow the polycarbonate filter to dry, using a heat lamp or a slide warmer. The filter may curl during drying. Allow the polycarbonate filter with the deposit to cool down to room temperature and weigh it.

13.5.4 Examination of materials for amphibole fibres

13.5.4.1 General

Vermiculite, wollastonite, attapulgite and sepiolite represent a group of materials that can be dissolved using sequential refluxing in acid and alkali.[24] Boiling the product in either hydrochloric or sulphuric acid removes cations such as magnesium and calcium, leaving a residue of silica gel. Boiling of this residue in sodium hydroxide dissolves the silica gel, leaving any amphibole minerals almost unaltered.

For analysis of wollastonite, it is necessary to use hydrochloric acid.

NOTE Routine measurement of the amphibole mass fraction in vermiculite and vermiculite-containing materials is often more conveniently made by the methods specified in Clause 15.

13.5.4.2 Select a representative sub-sample

Weigh a porcelain or silica crucible. Select up to approximately 2 g of the material, and place it into the crucible and weigh again.

13.5.4.3 Heat treat the sub-sample

Place the crucible into a muffle furnace, maintained at a temperature of 600 °C ± 10 °C for a minimum period of 10 h. Remove the crucible from the muffle furnace, and allow it to cool down to room temperature.

13.5.4.4 Reflux the heat-treated sub-sample in acid

Transfer the contents of the crucible to the 250 ml round-bottomed flask which is part of the reflux assembly. Add 80 ml of either 2 mol/l sulphuric acid or 2 mol/l hydrochloric acid, and boil the suspension for 1 h. Place a piece of unglazed porcelain in the flask to reduce violent bumping during boiling. Allow the flask to cool down to room temperature.

13.5.4.5 Centrifuge and wash the residue

Transfer the contents of the flask to centrifuge tubes, and centrifuge at a minimum speed of 2 800 rpm for 10 min. Decant the supernatant liquid from the centrifuge tube, and disperse the centrifugate in filtered, distilled water. Centrifuge the suspension again. Repeat the washing procedure a second time.

13.5.4.6 Reflux the residue in sodium hydroxide

Transfer the centrifugate to the 250 ml flask of the reflux assembly, and add approximately 80 ml of 4 mol/l sodium hydroxide. Reflux the suspension for 1 h.

13.5.4.7 Separate the final residue

Transfer the contents of the flask to centrifuge tubes, and centrifuge for 10 min. Wash the centrifugate twice as described in 13.5.4.5. Using ethanol, transfer the centrifugate to a pre-weighed polystyrene Petri dish. Place the Petri dish on a slide warmer at 60 °C and allow the ethanol to evaporate. Weigh the Petri dish containing the centrifugate to obtain the weight of the final residue.

13.5.4.8 Identify and Quantify amphibole in the final residue

Use the procedures specified in ISO 22262-1 to identify any asbestos, if present, in the final residue. If amphibole is detected, it can be quantified by PLM, SEM or TEM as specified in Clause 14.

14 Procedures for quantification of asbestos in the final residue from gravimetric matrix reduction

14.1 General

After application of quantitative gravimetric analytical procedures, it is usually necessary to quantify the asbestos in the final residue. This residue is generally in the form of a thick layer of fine particulate material on the surface of a polycarbonate filter. In some preparations, filters with particulate material loadings suitable for PLM point counting may also have been prepared from the final residue. Asbestos in the final residue from gravimetric reduction procedures is quantified by one of the following methods. The appropriate procedure depends on the nature of the material. The procedures below are designed to achieve a sufficiently reliable value for the asbestos mass fraction for comparison with regulatory limits, at minimum effort and expense.

14.2 Examination of the residue on the filter and selection of the appropriate procedure

Examine the deposit on the polycarbonate filter using the stereo-binocular microscope. It is sometimes possible to see large fibre bundles that can be picked from the deposit and identified by PLM (taking into consideration the fact that the refractive indices will have been reduced by exposure to the hydrochloric acid). If possible, select fibres and identify them using the methods specified in ISO 22262-1. If additional documentation on the fibre identification is required, either SEM or TEM methods as specified in ISO 22262-1 may be used. Depending on the nature of the final residue and the applicable control limits, select one of the following methods for quantification of any asbestos present.

14.2.1 Gravimetric measurements alone

Some materials have compositions such that after ashing, or a combination of ashing and acid treatment, the final residue consists almost completely of asbestos. For these types of material, it is only necessary to identify the asbestos using the procedures specified in ISO 22262-1, and the mass fraction of asbestos in the original sample can be calculated from the gravimetric data alone. In some cases, the final residue may represent such a small proportion of the original sample that, even if the residue were 100 % asbestos, the mass fraction could not exceed the applicable regulatory limit. If this situation occurs, a decision should be made as to whether it is necessary to proceed further, since the asbestos mass fraction can be reported as lower than the particular regulatory limit. Further quantification of asbestos in the residue, or confirmation of the absence of asbestos can be achieved by any of the following methods.

14.2.2 Visual estimation by PLM, SEM or TEM observation

Visual estimation of the mass fraction of asbestos fibres in a matrix of other materials has been demonstrated to consistently yield an over-estimate of the proportion of asbestos. In some cases, taking account of the residue mass fraction after application of the gravimetric procedures, on the basis of a visual examination of the residue it can be intuitively obvious that the proportion of asbestos in the residue is negligible, and that a control limit cannot be exceeded. Such a visual estimate for the purpose of demonstrating compliance with a control limit may be carried out using PLM, SEM or TEM. Because visual estimation is known to yield over-estimates of the asbestos mass fraction, do not use visual estimation to demonstrate that a control limit is exceeded. For this purpose and other situations in which visual estimation is not appropriate, use the point counting procedure specified in 14.2.3 or the mass counting procedure specified in 14.2.4.

NOTE For the specific case of floor tile, if asbestos can be reliably identified by PLM in the residue, the analysis may be terminated, if desired, because such residues generally consist almost entirely of chrysotile. If chrysotile is identified in a floor tile, it is generally present at substantial mass fractions that exceed any of the regulatory limits. However, because of the interference by pigments such as titanium dioxide, and the possibility that chrysotile from the Coalinga, California deposit is a constituent, the PLM procedure is not reliable for confirming that asbestos is not present or for determining the mass fraction of asbestos in the residue. For quantification of chrysotile, or for confirmation that asbestos is not present, it is necessary to examine the residue by either SEM or TEM using the procedures specified in 14.2.2, 14.2.3 or 14.2.4 .

14.2.2.1 Preparation of SEM or TEM specimens

Additional analytical steps other than identification of any asbestos detected in the residue may or may not be necessary. If the residue weight, assuming it to consist of 100 % asbestos, represents less than the regulatory asbestos mass fraction limit, then the necessity of proceeding further should be considered, because the asbestos mass fraction could not possibly exceed the regulatory limit. For the situation where a visual estimate of the amount of asbestos in the residue is likely to be sufficiently precise to establish the regulatory status of the material, a drop-mount SEM or TEM procedure is the optimum way of identifying asbestos and estimating its proportion in the final residue. This is also the optimum procedure to demonstrate the absence of asbestos. Drop mounts for SEM or TEM are prepared by dispersing some of the residue in ethanol, and evaporating a small drop of the residue on a carbon or beryllium planchette mounted on a specimen stub for SEM observation, or a carbon-coated grid for TEM observation.

Cut out approximately 1 cm^2 of the polycarbonate filter and deposit, and transfer this to a disposable plastic beaker. It will often be found that the deposit can easily be separated from the filter. Add 5 ml of ethanol, and treat in an ultrasonic bath for 1 min to remove most of the deposit into suspension. Pipette 3 µl of the suspension, using a disposable-tip micropipette, onto the top of an SEM stub or a carbon-coated grid under a heat lamp. Turn on the heat lamp, and allow the drop of suspension to dry on the stub or the grid. When it is dried, transfer the specimen to a labelled Petri dish.

14.2.2.2 Estimation of the proportion of asbestos

Examine the specimen in the SEM or TEM. Estimate the proportion of asbestos, taking account of the entire sample in the SEM, or by examination of several representative grid openings on a TEM specimen. If the examination is intended to confirm that asbestos is present, ensure that a sufficient amount of particulate material is examined to give confidence in this determination with respect to the applicable control limit.

NOTE Specifically for resilient floor tile, it will often be found that the final residue from this procedure consists of between 70 % and 95 % chrysotile asbestos with titanium dioxide pigment particles. Other types of floor tile will yield residues in which chrysotile is a smaller proportion of the total residue, or in which a low mass fraction of amphibole or other types of mineral fibres or particles can be found. Some rare floor tiles contain major proportions of tremolite and/or anthophyllite. In addition to chrysotile, the residues from floor tiles may contain non-asbestiform tremolite, anthophyllite, talc, antigorite, kaolinite, iron oxide, barium sulfate, mica, quartz or other mineral species.

14.2.2.3 Calculation of the asbestos mass fraction in the original material

Calculate the mass fraction percent asbestos in the original sub-sample using the following relationship:

$$C = \frac{R \times P}{W}$$

where

C is the mass fraction of asbestos in the original sub-sample;

R is the weight of residue after ashing and acid treatment, in g;

W is the weight of original sub-sample, in g;

P is the mass fraction of asbestos in the residue derived from TEM or SEM examination.

14.2.3 Point counting by PLM or SEM

Conventional point counting determines the relative projected areas occupied by different particle species on a microscope slide. The integrated relative volumes of different particle species can be calculated from a conventional point count, but **only** if the particles are all of the same thickness. If the densities of the various particle species are known, the relative weights of the different particle species can be calculated. However, conventional point counting does not produce correct results when applied to the determination of the proportion of asbestos in a mixture of particles with a wide range of different thicknesses and different densities. The result also clearly depends on the extent of any sample preparation. The percentage area measurements provided by conventional point counting are not suitable for calculation of the relative weight mass fractions of asbestos and other particles.

The gravimetric matrix reduction procedures have the overall effect of removing low density materials and restricting the range of particle sizes in the final residue. Computer modelling studies of the application of point counting to logarithmic-normal particle diameter and fibre diameter distributions have shown that the majority of the integrated volume of a particle or fibre species is contributed by a minority of particles and fibres with the largest diameters. In practice, after gravimetric matrix reduction according to this part of ISO 22262, the volume, and therefore most of the weight, of particles in a logarithmic-normal size distribution can be accounted for by considering only those particles larger

than approximately 10 % of the largest particle detected. The mass point counting protocol used in this method, **used with the specified point counting criteria**, is based on these observations.

14.2.3.1 Preparation of specimens for point counting

14.2.3.1.1 Preparation of slides for PLM point counting

Microscope slides for point counting can be prepared either by clearing the MEC filters, or from the residue collected on the polycarbonate filter. In practice, it has been found more satisfactory to use the MEC filters for the point counting, because it is sometimes difficult to re-disperse material removed from the polycarbonate filter, particularly if the residue on the filter contains substantial quantities of chrysotile. Prepare microscope slides for point counting according to a) or b).

The refractive index liquid to be used for point counting shall be selected such that all particle species present as significant proportions of the weight are visible (i.e. their refractive indices do not closely match that of the liquid). For the majority of samples containing chrysotile, amosite or crocidolite, a liquid of refractive index 1,605 has been found to be suitable, but occasionally it is found that a major particle species present cannot be reliably seen in this liquid.

a) Place 100 µl of a mixture of 35 % dimethylformamide, 15 % glacial acetic acid and 50 % distilled water on a clean microscope slide. Gently lower the edge of one of the MEC filters so that it touches the liquid, and lay it down on to the liquid so that there are no bubbles underneath it. Absorb any excess liquid from the slide using the edge of a paper towel. Repeat the procedure with the other MEC filters. Place the slides on the slide warmer at a temperature between 65 °C and 70 °C for a period of 10 min. Remove them from the slide warmer. Place 30 µl of glycerol triacetate on each of the filters, and apply cover slips. The slides are ready for point counting.

   It is important to confirm that all particle species present as significant proportions of the weight are visible under the illumination conditions used for point counting. If it is found that any major particle species has refractive indices such that the particles are not visible, an alternative method of preparing the slides shall be used.

b) In many cases, the filtered deposit can be separated from the polycarbonate filter by curling back the filter using forceps. Grinding of the residue on the polycarbonate filter from the gravimetric reduction procedure should not be necessary, because liberation of the fibres from the matrix material during the ashing, acid dissolution and the subsequent filtration should have resulted in a relatively uniform and homogeneous deposit on the polycarbonate filter, and any large particles should have already been removed in the flotation, sedimentation and manual picking of large particles and large fibre bundles. A representative sub-sample of the deposit can then be broken off using forceps and placed on to a clean 75 mm × 25 mm microscope slide. In other cases, the deposit may not be self-cohesive, and it is then necessary to cut a representative area from the filter and then, using the edge of a scalpel blade, to scrape the deposit off the filter on to the microscope slide. After placing the sub-sample of the residue on to the microscope slide, apply one drop of the refractive index liquid to it. Using a second microscope slide, hold it parallel to the first one, but rotated at 90° to it, and bring the two slides into contact with the refractive index liquid touching both slides. Press the slides together lightly, and rub them backwards and forwards parallel to each other in a rapid shearing motion. The liquid can then be moved back to the middle of the slide using the edge of the second slide, and the process repeated several times. This procedure usually produces a uniform dispersion of particles, and a 22 mm × 22 mm cover slip should then be placed on the first slide. If the particle density is too high, the process can be repeated after the addition of more refractive index liquid, and some of the suspension can be removed and used to prepare additional slides. With practice, the amount of residue necessary to produce slides of acceptable particle density can be judged reliably. In some samples, the particles and fibres tend to re-aggregate after dispersal, but this does not appear to affect the point counting procedure. The number of slides to be prepared depends on the degree of matrix reduction achieved and the mass fraction of asbestos to be measured. Use a minimum of two slides.

14.2.3.1.2 Preparation of PC filters for SEM point counting

Using a scalpel, cut a 1 cm square of the filter and mount it on an SEM stub using double-sided adhesive tape. Take care to not disturb particulates on the filter during the cutting operation. Apply graphite paint to the edges of the filter. Evaporate a film of carbon on to the surface of the filter.

14.2.3.2 Microscope adjustment for PLM point counting

Use of the correct illumination conditions and checking of particle visibility are critical to the validity of the point count.

a) Set up the PLM with crossed polars and a 530 nm plate in position.

b) Select an objective to provide a total magnification of approximately ×100.

c) Adjust the illumination system to provide the maximum illumination, and close the sub-stage aperture as much as possible to provide maximum contrast.

d) Use an eyepiece containing a cross-hair with scale divisions on at least one of the directions.

e) Examine one of the prepared slides to determine if all of the particle species have sufficient contrast under the illumination conditions. Pay particular attention to isotropic particle species. If all particle species can readily be seen, proceed with the point count. If any one of the particle species exhibits insufficient contrast, select an alternative refractive index liquid and prepare new slides from the final residue deposit on the polycarbonate filter.

14.2.3.3 Microscope conditions for SEM point counting

Use a magnification of ×100 for the point count. A transparent overlay can be used over the display of the SEM with either one index point or several randomly positioned index points over the area of the display.

14.2.3.4 Point counting

The following point counting criteria ensure that the result is based on statistically-valid numbers of particles and fibres, and that the sizes of the particles and fibres included in the measurements are those which contribute the majority of the weight.

a) Scan all of the slides or SEM specimens to estimate the approximate diameters of the largest particle and the largest fibre.

b) Carry out the point count, recording:

   i) for particles, only those points which occur on particles exceeding 10 % of the diameter of the largest particle detected in the initial scan; and,

   (ii) for each species of fibre, only those points which occur on such fibres whose widths exceed 20 % of the width of the largest fibre of that species detected in the initial scan.

c) When a point falls on a region where a particle and fibre of sufficient diameter are overlapped, record one point for each.

d) When a point falls on an overlapped region of two fibres of sufficient diameter, record as two points.

e) When a point falls on an overlapped region of two particles of sufficient diameter, record as two points.

f) When a point falls on one segment of a split fibre, record it only if the diameter of the segment under the point meets the minimum 20 % diameter criterion specified in b).

g) Distribute the non-empty points over all of the available samples.

h) Continue the point count until a minimum of 20 asbestos points have been recorded, or until the stopping point, appropriate for the particular control limit, of either 13 × *P*, 26 × *P*, or 130 × *P* non-empty points have been recorded, where *P* is the weight of the final residue on the polycarbonate filter expressed as a percentage of the original sample weight. Regardless of the above, use a minimum of two slides and count a minimum of 100 non-empty points.

The statistical reliability of a point count for determination of asbestos depends on the number of asbestos points, **not** on the total non-empty points examined. Accordingly for comparison against a regulatory standard of 0,1 % asbestos by weight, continue point counting until either a minimum of 20 asbestos points, or the **equivalent** of 13 000 non-empty points have been accumulated. For comparison against a regulatory standard of 0,5 % asbestos by weight, continue point counting until either a minimum of 20 asbestos points, or the **equivalent** of 2 600 non-empty points have been accumulated. For comparison against a regulatory standard of 1 % asbestos by weight, continue point counting until either a minimum of 20 asbestos points, or the **equivalent** of 1 300 non-empty points have been accumulated. For any of these regulatory standards, the **actual** number of non-empty points that are required depends on the degree of matrix reduction achieved, assuming that fewer than 20 asbestos points are encountered during the point count. For example, if 90 % of the mass of non-asbestos constituents is removed by matrix reduction, leaving a residue of 10 % of the original weight, the required number of non-empty points is reduced by a factor of 10, assuming that the 20 asbestos point stopping rule is not encountered first. The point count may be terminated before 20 asbestos points are encountered if the resulting confidence limits permit determination of compliance.

14.2.3.5 Calculation of results

The mass fraction percentage of asbestos is calculated from the formula:

$$C = \frac{100}{W} \times \left(M + R \times \frac{A}{N} \right)$$

where

W is the weight of original sub-sample, in g;

M is the weight of manually-picked asbestos, in g;

R is the weight of final residue on PC filter, in g;

A is the number of asbestos points counted;

N is the number of non-empty points counted.

The 2-sided upper and lower 95 % confidence limits should also be calculated.

14.2.4 Determination of asbestos weight mass fraction from fibre measurements made by PLM, SEM or TEM

14.2.4.1 General

Some materials such as talc cannot be selectively removed from asbestos by chemical dissolution. Depending on the variety of asbestos that is present, it may be possible to separate the asbestos by density separation, but for talc, separations by this method are often incomplete. The optimum approach is to prepare samples for either SEM or TEM analysis, and to quantify any asbestos by a fibre count to determine mass. This procedure can also be used for the determination of chrysotile in finely-divided materials.

14.2.4.2 Dispersion of the material in filtered, distilled water

Weigh a sub-sample of 0,1 g to 1 g, and disperse in 20 ml of ethanol. Transfer the suspension to a 1 000 ml glass beaker, and dilute to 800 ml with filtered, distilled water.

NOTE Talc is sometimes difficult to disperse in water, because some of the particles appear to be hydrophobic. Dispersion is more readily achieved in ethanol.

14.2.4.3 Preparation of filters for SEM or TEM analysis

Use PC filters of 0,2 µm pore size to prepare analytical filters for SEM or TEM analysis. The volume of the aqueous dispersion to be filtered depends on either the particulate mass fraction or the asbestos fibre mass fraction in the material. The volume of the aqueous dispersion required to produce an analytical filter with a suitable particulate or fibre loading for analysis often cannot be predicted, and it is usually necessary to prepare several analytical filters corresponding to filtration of different aliquots. Uniform deposits of particulate on the analytical filters cannot be ensured if liquid volumes smaller than 5 ml are filtered using 25 mm diameter filtration systems (199 mm^2 active area); accordingly, where it is required to filter volumes smaller than 5 ml, the aliquot shall be diluted with freshly distilled water to a volume exceeding 5 ml.

Pour the aliquot of the dispersion into the filtration reservoir, and apply the vacuum. If the volume of the aliquot is larger than the capacity of the filtration reservoir, do not allow the level of liquid in the reservoir to fall below 5 cm depth before the remaining volume is added. Failure to observe this precaution may result in disturbance of the filtered particulate and non-uniform deposition of particulate.

With the vacuum still applied, unclamp the filtration assembly and remove the filtration reservoir. Using clean tweezers, remove the analytical filter and transfer it to a Petri dish. Allow the filter to dry before placing the cover on the Petri dish.

14.2.4.4 Preparation of specimens for SEM or TEM observation

Prepare SEM specimens by mounting a portion of the analytical filter on a SEM specimen stub. Carbon-coat the surface of the filter in a vacuum evaporator. Prepare TEM specimens from the PC filters using the procedures specified in ISO 13794.

14.2.4.5 Fibre counting to determine the mass of asbestos

The mass of asbestos in the residue from gravimetric matrix reduction can be determined by counting and measuring the dimensions of asbestos fibres on a suitably prepared specimen of the residue. The mass of asbestos is then calculated by summing the volumes of all of the fibres detected in the count, and assuming a density for the variety of asbestos involved. However, in order to measure the integrated mass with reliable statistical precision, it is necessary to adopt a fibre counting strategy that allows large fibre and fibre bundles that contribute most to the mass fraction to be counted with greater statistical reliability. The number of fibres and bundles to be counted in order to achieve a reliable estimate of the mass fraction depends primarily on the range of the diameter distribution. The mass fraction measurement is most sensitive to fibres and bundles of large diameters, which are generally statistically infrequent relative to the smaller fibres and bundles. If the diameter distribution is narrow, such as that found in a dispersion of chrysotile fibrils, the mass fraction can be measured with approximately the same precision as that of the numerical mass fraction. If the diameter distribution is broad, mass fractions calculated from SEM or TEM examinations to determine numerical mass fractions are statistically unreliable. The strategy specified below is designed to give greater statistical significance to the large fibres and bundles, which contribute most to the mass fraction.

Initially, establish the largest width of asbestos fibre or bundle that can be detected on the grid by a cursory survey of the specimen, at a reduced magnification. Calculate the volume of this large fibre or bundle. Adjust the magnification of the SEM or TEM to a value such that a width of 1 mm on the display or fluorescent screen corresponds to approximately 10 % of the width of the previously-selected large structure. Carry out a routine SEM or TEM examination at this magnification, terminating the examination at the end of a defined area on which the integrated volume of all fibres and bundles

recorded is at least ten times the volume of the originally-selected fibre or bundle. For asbestos fibres which intercept the edge of the field of view on the SEM, or the grid bars on the TEM, measure only the visible parts of the fibres and bundles within the field of view or grid opening, for the purposes of calculation of the mass fraction of asbestos.

14.2.4.6 Calculation of the mass fraction percentage of asbestos

Calculate the mass fraction percentage of asbestos fibres and bundles using the relationship:

$$M = 10^{-13} \times \frac{G \times D \times A \times S}{E \times F \times T} \times \sum_{j=1}^{j=n} \left(W_j^2 \times L_j \right)$$

where

M is the mass fraction of asbestos in percent;

G is the geometric cross-section shape factor;

A is the area of analytical filter in mm^2;

D is the density of asbestos in kg/m^3;

S is the volume of suspension in ml;

W_j is the width of the jth structure in µm;

L_j is the length of the jth structure in µm;

E is the area of analytical filter examined in mm^2;

F is the equivalent volume of suspension filtered in ml;

T is the weight of original sub-sample in g.

In the above relationship, assume:

a) the value for G to be 0,7854 for chrysotile, and 0,5 for the amphiboles; and,

b) the following values for the densities of the asbestos varieties: chrysotile 2 550 kg/m^3, crocidolite 3 370 kg/m^3, amosite 3 430 kg/m^3, anthophyllite 3 000 kg/m^3, tremolite 3 000 kg/m^3, actinolite 3 100 kg/m^3, richterite/winchite 3 050 kg/m^3. The densities of the asbestos varieties vary with composition, but the values specified are sufficiently accurate for the purposes of this calculation.

15 Determination of asbestiform amphibole in vermiculite

15.1 General

The special characteristics of vermiculite and the wide range of vermiculite-containing products are such that a different approach to quantitative analysis is required. Accordingly, analysis of vermiculite and vermiculite-containing products are considered separately.

Many deposits of vermiculite contain low mass fractions of amphibole minerals, and some of these fragments of amphibole may have chemical compositions that are within the ranges of the regulated amphibole asbestos species. However, these amphibole fragments are usually present in a non-asbestiform crystal habit. Some of the amphibole may be present in an asbestiform crystalline habit, conforming to the conventional definition of asbestos. Chrysotile has not been detected in any commercial vermiculite, although some vermiculite contains scrolls which are short and exhibit morphological features superficially similar to those of chrysotile. Scrolls of lizardite that are morphologically similar and compositionally identical to chrysotile have also been detected in one source of vermiculite.

However, both types of scroll can be discriminated from chrysotile by TEM methods. In exfoliated vermiculite, chrysotile would be unlikely to survive the exfoliation process, since the temperatures used for exfoliation exceed those at which chrysotile is converted to forsterite.

This method can be used to determine the weight percent asbestiform amphibole for vermiculite ore concentrate, exfoliated vermiculite, loose fill vermiculite attic insulation, vermiculite block fill insulation, and materials containing vermiculite, including horticultural vermiculite, potting soil, slow release horticultural fertilizers, sprayed vermiculite-containing fireproofing materials and vermiculite-containing lightweight concrete.

15.2 Required size of sample for analysis

It is most important to recognize that reliable and reproducible results cannot be obtained by analysis of small samples of vermiculite. Any amphibole particles present in vermiculite are usually much fewer in number than the flakes of vermiculite, and if only a small sample size is analysed the number of amphibole particles included in the sample will be small and often unrepresentative. If asbestiform amphibole fibres are present in vermiculite, the fibres and fibre bundles usually have a large size spectrum ranging from a few micrometres in length up to the size of the vermiculite flakes themselves.

Table 2 gives approximate weights of vermiculite that should be used for the initial sub-sample. For products containing vermiculite, a visual estimate of the proportion of vermiculite in the product should be made and the starting weights in Table 1 should be proportionately increased.

Table 2 — Minimum sub-sample weights of vermiculite for analysis

Size of vermiculite flakes mm	Minimum starting weight for analysis g
< 2	2
> 2 to < 5	10
> 5	50

The sub-sample shall be obtained from the original sample by the cone and quarter method. On a clean surface, such as a sheet of aluminium foil, form the sample into a cone. Use a thin flat sheet of metal or rigid plastic to divide the cone into two parts, vertically from the apex. Form either of the two fractions into a cone, and repeat the procedure until one of the separate fractions is of a suitable weight for analysis.

15.3 Sample pre-treatment

15.3.1 Exfoliated vermiculite, loose fill vermiculite attic insulation and horticultural vermiculite

Before analysis, vermiculite insulation samples originating from attics may require manual removal of foreign materials such as other types of insulation, wood or cement fragments. Otherwise, exfoliated vermiculite, as marketed for application as loose attic fill attic insulation or for horticultural use, requires no pre-treatment before analysis.

For this measurement, a known weight of the exfoliated vermiculite is first suspended in filtered distilled water in a beaker. The size of beaker required, and the volume of water used, depends on the size grade of the vermiculite. If the flake size of the vermiculite is greater than approximately 5 mm, as is the case for these categories of product, a 1 000 ml beaker with 800 ml of water is satisfactory. Using a spoon, place a portion of the exfoliated vermiculite sub-sample into the beaker, and immerse the vermiculite several times by pushing it under the surface using the spoon. Remove the floating vermiculite and discard it. Continue to wash portions of the vermiculite in this manner until all of the sub-sample has been treated. Carefully remove all fragments of floating vermiculite from the surface of the water, and allow the suspension to settle for 1 min. Decant the supernatant liquid to a second beaker. Use ethanol to wash the sediment from the first beaker into a pre-weighed Petri dish and dry the sediment by placing

the Petri dish on a slide warmer at a temperature of 60 °C. When the sediment is dry, weigh the dish to obtain the weight of the sediment. Proceed to 15.4.2.

15.3.2 Vermiculite block fill insulation

Vermiculite block fill insulation may have been treated with asphalt. Asphalt can be detected by examination of the sample using a stereo binocular microscope. If asphalt is present, weigh the sub-sample and transfer it to a fused quartz tray or other suitable open container for ashing. Place the sub-sample in a muffle furnace operating at a temperature of 450 °C ± 10 °C for a minimum period of 10 h. Weigh the residual ash, and calculate the percentage as a proportion of the original dried sub-sample. Use the flotation method specified in 15.3.1 to separate the majority of the vermiculite. Weigh the sediment and proceed to 15.4.2.

15.3.3 Vermiculite ore concentrate

Samples of vermiculite ore concentrate shall be exfoliated prior to analysis. Set the temperature of the muffle furnace to 800 °C, and place a fused silica tray into the furnace. Have available a large glass or metal container available to receive the exfoliated vermiculite. A sheet of aluminium foil, formed into a container, has been found satisfactory. Using crucible tongs, remove the silica tray from the muffle furnace. The tray will be at a red heat. Sprinkle a small amount of the vermiculite sample into the silica tray. Return the silica tray to the muffle furnace, close the furnace door for approximately 15 s to complete the exfoliation. Remove the silica tray from the muffle furnace and pour the exfoliated vermiculite into the container. Repeat this procedure as many times as necessary, until sufficient vermiculite for the analysis has been exfoliated. Weigh the exfoliated material and use the flotation method specified in 15.3.1 to separate the majority of the vermiculite. Weigh the sediment and proceed to either 15.4.2 or 15.4.3, depending on the size grade of the vermiculite.

15.3.4 Vermiculite-containing horticultural products

Vermiculite represents only a fraction of the weight of potting soil and similar horticultural products. The other components are generally organic, with some plant nutrients. Water may also represent a significant proportion of the weight. The results of the analysis should generally be expressed in terms of dry weight, because the water content may vary depending on the storage conditions. Weigh a container (a disposable container formed from aluminium foil is suitable), place the sub-sample in the container and weigh again. Dry the sub-sample for a minimum period of 10 h, either on a slide warmer or in an oven at a temperature of 60 °C, and then re-weigh. Calculate the weight of water evaporated from the sample.

Transfer the sub-sample to a fused quartz tray or other suitable open container for ashing at 450 °C. Place the sub-sample in a muffle furnace operating at a temperature of 450 °C ± 10 °C for a minimum period of 10 h. Weigh the residual ash, and calculate the percentage as a proportion of the original dried sub-sample. Use the flotation method specified in 15.3.1 to separate the majority of the vermiculite. Weigh the sediment, and proceed to 15.4.2 or 15.4.3, depending on the size grade of the vermiculite.

15.3.5 Sprayed vermiculite-containing fireproofing

Vermiculite-based insulation and fireproofing products often contain as much as 60 % of other materials such as gypsum, cellulose, glass fibre and calcium carbonate, and also possibly chrysotile. If the fireproofing contains chrysotile, analysis according to ISO 22262-1 is usually sufficient to establish its regulatory status. Continue with this gravimetric method if there is a requirement to quantify asbestiform amphibole.

Constituents such as gypsum and calcium carbonate can be removed by treatment in 2 mol/l hydrochloric acid, without affecting the vermiculite. Gently crush 3 g to 4 g of the product into fragments of about 0,5 cm dimension, using a mortar and pestle. Crushing is intended to facilitate ashing or organic constituents such as cellulose and the dissolution of the carbonates and gypsum by hydrochloric acid. Place the crushed material into a pre-weighed porcelain crucible and weigh to obtain the weight of the sub-sample. Place the crucible with lid into the muffle furnace, and ash the sample at 450 °C for a

minimum period of 10 h. Remove the crucible with lid from the muffle furnace, allow it to cool to room temperature, and weigh the crucible without the lid.

Transfer the ashed sub-sample to a 250 ml Erlenmeyer conical flask, add a stirring bar, and also add 100 ml of 2 mol/l hydrochloric acid. Add an additional 50 ml of water. Place the flask on a magnetic stirrer, and stir for 15 min. Hold a magnet close to the outside of the flask to attract the stirrer bar, and slide the magnet upwards until the stirrer bar can be removed. Add water to the Erlenmeyer flask until the meniscus is at the top of the flask. Allow a few minutes for any floating material to come to the surface. Using a spatula, remove and discard as much of the floating material as possible.

Set up a glass filtration assembly with a pre-weighed 47 mm, 0,8 µm pore size polycarbonate filter and connect it to a water aspirator. Transfer the ashed and acid-treated sub-sample from the Erlenmeyer conical flask to the filter reservoir. Rinse the flask with water and add it to the filter reservoir. After the filtration is completed, rinse the filtered material by addition of 20 ml of water, and allow the filtration to complete. Repeat the rinsing operation two additional times, to ensure that soluble materials have been removed from the residue. Dry the filter and residue on a slide warmer. Weigh the filter and residue. Proceed to 15.4.3.

15.4 Separation of amphibole and measurement of the amphibole mass fraction

15.4.1 General

Two methods are available for determination of the amphibole mass fraction in sediments or residues from the procedures in 15.3. If the size grade of the vermiculite is such that any fibre bundles of asbestiform amphibole are sufficiently large, they can be manually picked out from the sediment or residue and weighed. For smaller size grades of vermiculite, this is not feasible and further concentration of the amphibole by centrifugation in a heavy liquid is necessary, followed by point counting of the centrifugate.

15.4.2 Manual separation of amphibole fibre bundles and weighing

Collect separated fibrous amphibole fragments in a pre-weighed Petri dish, and, after all of the sediment has been examined, weigh the Petri dish to obtain the weight of fibrous amphibole. Calculate the percentage of fibrous amphibole as a proportion of the original sub-sample weight. Select representative fragments of fibrous amphibole and identify them according to the procedures specified in ISO 22262-1.

15.4.3 Separation of amphibole by centrifugation in a heavy liquid

Transfer the residue or sediment from the procedures of 15.3 to a centrifuge tube. For sprayed fireproofing samples, there is usually too much residue to process in one 15 ml centrifuge tube, and it is necessary to divide the residue between two centrifuge tubes or use a larger centrifuge tube.

Prepare sufficient liquid of density 2 750 kg/m^3 to complete the number of analyses to be performed. Adjust the density of the lithium metatungstate solution to 2 750 kg/m^3 by addition of filtered distilled water. The density is most conveniently monitored by use of a Sink-Float standard. Place the Sink-Float in a glass beaker, and add the required volume of heavy liquid to it. Progressively add water, stirring well after each addition, until the Sink-Float begins to sink in the liquid. Adjust the density with either water or more lithium metatungstate until the Sink-Float is suspended in the liquid, and rises to the surface only slowly.

Add approximately 10 ml of density-adjusted heavy liquid to each of the centrifuge tubes. Using a 5 mm diameter glass rod, disperse the residue in the liquid by macerating the solids between the inside of the centrifuge tube and the rod. Add sufficient heavy liquid to bring the liquid level up to approximately 2 cm from the top of each centrifuge tube. Disperse the solids throughout the liquid using the glass rod.

The required time for centrifugation depends on the dimensions and rotation speed of the particular centrifuge in use. The majority of the weight of amphibole in vermiculite is present as relatively large fragments, and a centrifuge time sufficient for sedimentation of all particles larger than 5 µm diameter and with a minimum density of 3 000 kg/m^3 is used. Annex B gives the information to calculate the

required centrifugation time for centrifuges with different dimensions and rotation speeds. Place the centrifuge tubes in opposite positions in the centrifuge, and operate the centrifuge at maximum speed for the calculated time. The time required for the dimensions of the centrifuge and rotation speed as shown in the example in Annex B, is 5 min. After centrifuging, remove each of the tubes from the centrifuge. There will be a small pellet of solids at the bottom of each tube, and in general a much larger amount of floating material. Using the glass rod, macerate the floating fraction against the inside of each tube, and re-disperse the material in the heavy liquid, without disturbing the pellet of solids at the bottom of the tube. Centrifuge the tubes for a further 5 min. Repeat the dispersal and centrifugation one more time.

Using a small spatula, remove as much of the floating material as possible from the top of each centrifuge tube. Aspirate as much of the heavy liquid as possible from each tube, taking care to not disturb the pellet of particulate material at the bottom. There will be some residual floating material adhering to the upper parts of the centrifuge tubes. Remove this adhering material from the upper parts of the centrifuge tubes using a piece of paper towel wrapped around a small spatula or glass rod. It is necessary to wet the paper towel with water to facilitate the removal of the adhering material.

Direct a jet of water from a wash bottle at the pellet of particulate in each centrifuge tube to disperse the pellet in the water. Disperse the particles in a minimum of 5 ml of water. Centrifuge the tubes for 1 min. Remove the tubes and pour out the liquid, taking care to not disturb the pellets at the bottom of the tubes. Repeat the washing and centrifugation operations two additional times to remove traces of the heavy liquid. After the final centrifugation, pour out the liquid from each tube and use a jet of ethanol to wash the pellets of particulate material into a pre-weighed 50 mm diameter plastic Petri dish. Place the Petri dish on a slide warmer and allow the ethanol to evaporate to dryness. After drying, weigh the Petri dish and contents.

Identify any asbestiform amphibole in the centrifugate according to the procedures specified in ISO 22262-1. Quantify any asbestiform amphibole in the centrifugate by the point counting procedures specified in 14.2.3.

15.4.4 Confirmation of the absence of asbestos in the vermiculite sub-sample

Filter a minimum of 50 ml of the supernatant liquid from 15.3.1 on to a 47 mm, 0,4 µm pore size polycarbonate filter. Allow the filter to dry. Prepare SEM or TEM drop mounts according to 14.2.2.1, and examine a sufficient amount of the particulate material to give confidence that asbestos, if present, is at a mass fraction lower than the applicable control limit.

16 Determination of asbestos in talc

16.1 General

Some sources of talc contain either chrysotile or amphibole as contaminants. The most common amphiboles that may be associated with talc are tremolite and anthophyllite. Talc is insoluble in acids, and therefore the gravimetric matrix reduction methods described in Clause 13 are not effective.

16.2 Determination of chrysotile in talc

Although separation of chrysotile from talc by centrifugation in a heavy liquid is theoretically possible, in general it is not a practical technique. Determination of the chrysotile mass fraction by TEM, using 14.2.4, is the optimum analytical procedure.

16.3 Determination of amphibole in talc

Centrifugation in a heavy liquid can permit separation of amphibole from talc, because the published density range for talc is 2 580 kg/m^3 to 2 830 kg/m^3, and the minimum density for the amphibole asbestos varieties is 3 000 kg/m^3. Separation of amphibole from talc is therefore possible using a liquid of density 2 850 kg/m^3. Calculate the required centrifugation time according to Annex B. Use the centrifugation method described in 15.4.3. Identify any asbestiform amphibole in the centrifugate according to the

procedures specified in ISO 22262-1. Quantify any asbestiform amphibole in the centrifugate by the point counting procedures specified in 14.2.3.

NOTE The centrifugation time determines the smallest particle size that will be included in the centrifugate. This limitation is intended to be considered when this method is used.

If it is required to include all fibre sizes in the measurement, determination of the mass fraction by TEM, using 14.2.4, is the optimum analytical procedure.

17 Determination of compliance with legislative control limits

17.1 General

Compliance with a regulatory limit is generally considered acceptable if the upper 95 % confidence limit of the measured mass fraction is lower than the legislative control limit. Conversely, if the lower 95 % confidence limit of the measured mass fraction is higher than the regulatory limit, the regulation is deemed to have been contravened. Accordingly, the statistical reliability and accuracy of measured asbestos mass fractions should always be considered when making a determination of compliance or non-compliance with a legislative control limit. Measurements of asbestos mass fraction in bulk materials often exhibit great variability, some of which may originate in the fundamental lack of homogeneity in the sample, and some being a consequence of errors in the measurement method.

The limit of detection for each analysis is relevant to the determination of compliance and shall be reported. For example, a measurement of 0,2 % with a limit of detection of 0,1 % is of questionable value in determining that the measurement is out of compliance with a control limit of 0,1 %. However, this measurement is likely to be satisfactory for comparison with a control limit of 1 %.

17.2 Gravimetry alone

If the final residue consists almost completely of asbestos, the calculated asbestos mass fraction depends only on the precision of the weight measurements. This is the optimum situation, since, assuming that the starting sub-sample was sufficiently large to be representative, the reliability of a particular measurement is controlled only by the accuracy of the measured weights. The magnitude of potential errors that could occur in the gravimetric procedure, combined with the potential weighing errors, should be evaluated in terms of proximity of the measured mass fraction to the control limit.

17.3 Gravimetry combined with visual estimation

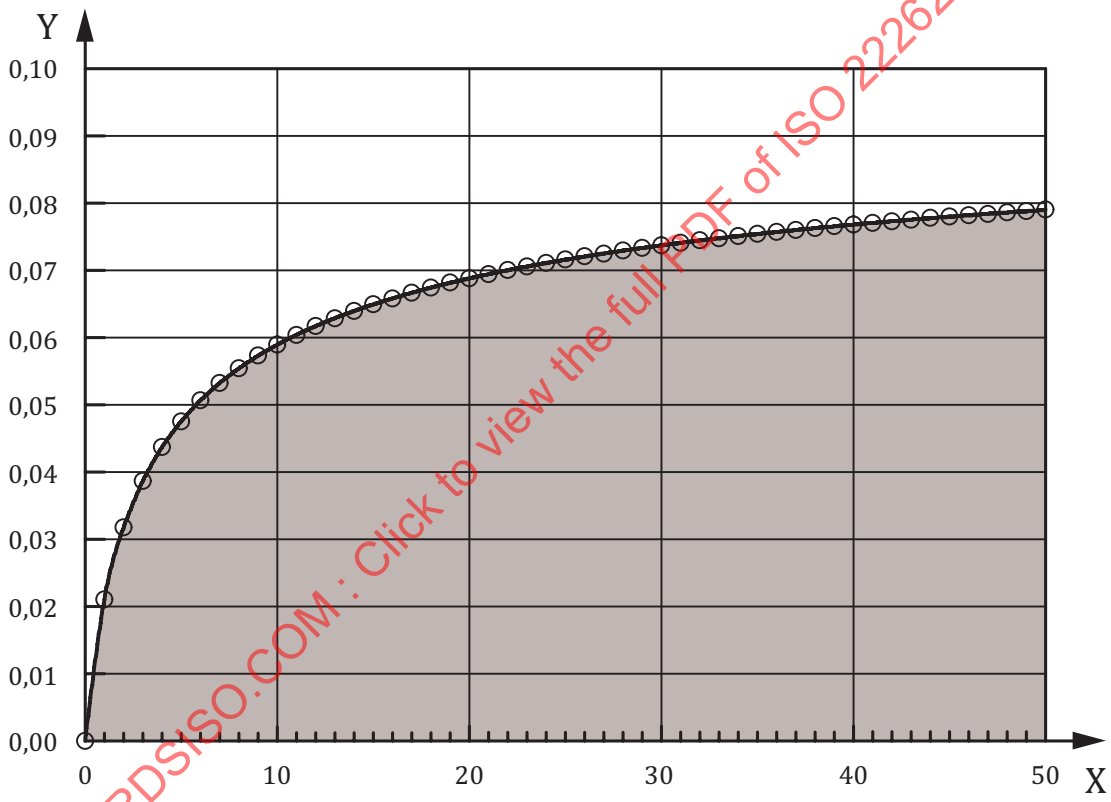
The weight percentage represented by the final residue from gravimetric matrix reduction is the maximum possible value for the asbestos mass fraction. Even a superficial visual estimation made by PLM, SEM or TEM may provide sufficient information to allow a confident statement about compliance or non-compliance with a specific control limit. In particular, observation of a SEM specimen or TEM specimen grids prepared from the final residue may show that asbestos cannot be detected. Experience has shown that, in a homogenized material such as the final residue from gravimetric matrix reduction, mass fractions of chrysotile as low as 0,001 % can be readily detected by TEM examination of simple drop mounts prepared from the final residue. For many samples, PLM, SEM or TEM examination of simple drop mounts prepared from the final residue can provide sufficient information to demonstrate compliance. In other cases, these simple procedures are not sufficiently reliable.

17.4 Gravimetry combined with point counting

The weight percentage of the final residue from gravimetric matrix reduction represents the maximum possible asbestos mass fraction. Point counting, as specified in 14.2.3, is then used to determine the proportion of asbestos in the final residue. For comparison with a legislative control limit, the calculated asbestos mass fraction is compared with the one-sided 95 % upper confidence limit for the number of asbestos points recorded. Figure 2 shows the maximum calculated mean mass fraction of asbestos for which the upper 95 % confidence limit is lower than 0,1 %. For a measurement of asbestos mass

fraction to be in compliance with this control limit, the measurement shall occur within the shaded area of Figure 2. If it does not, then it is necessary to perform additional point counting until the data point occurs in the shaded area. However, when calculated asbestos mass fractions are too close to the relevant control limit (> 0,8 %, > 0,4 % or > 0,08 %), it becomes impractical, on a scientific basis, to demonstrate compliance. For a control limit of 1 %, the values on the ordinate should be multiplied by 10, and for a control limit of 0,5 %, the values on the ordinate should be multiplied by 5. Otherwise, the procedure is identical.

In order to confirm that a measured asbestos mass fraction contravenes a 0,1 % control limit, the point counting data should be compared with the one-sided 95 % lower confidence limit for the number of asbestos points recorded. Figure 3 shows the minimum calculated mean mass fraction of asbestos for which the lower 95 % confidence limit is higher than 0,1 %. Non-compliance with a 0,1 % control limit is confirmed at 95 % confidence if the measurement occurs within the shaded area of Figure 3. If the measured asbestos mass fraction is too close to the control limit, it may not be possible to demonstrate non-compliance.

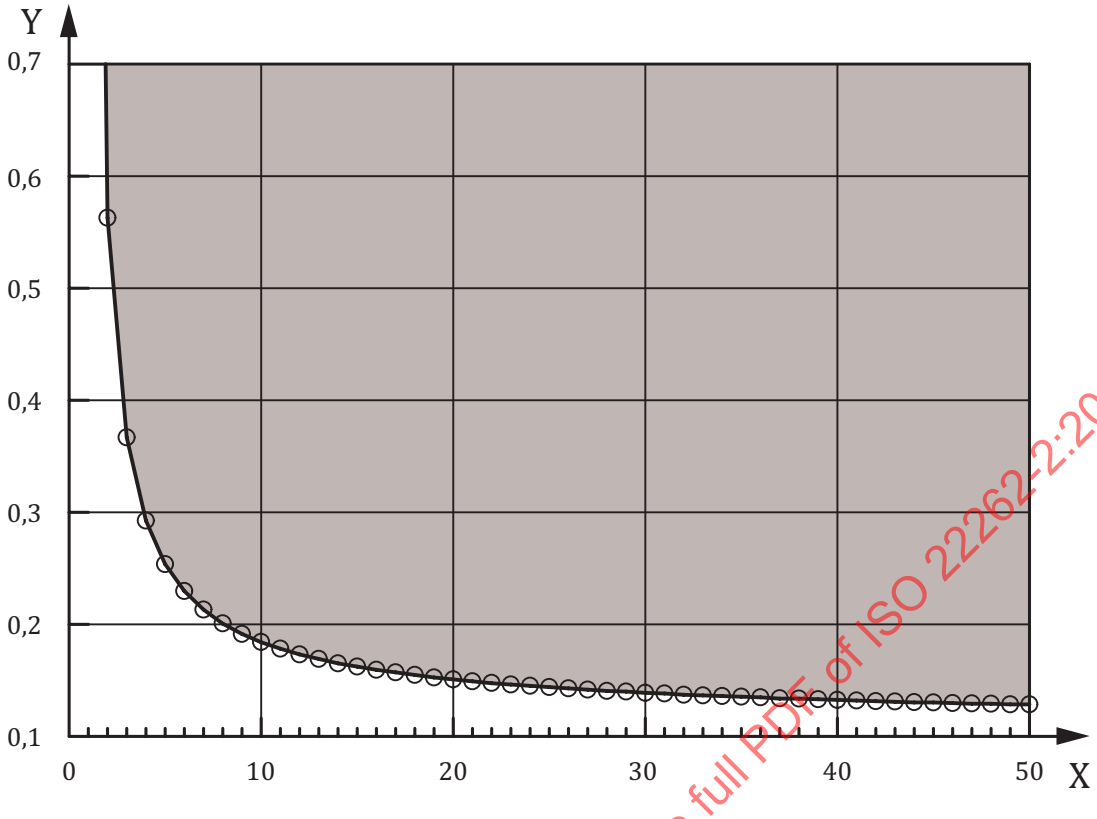


Key

X number of asbestos points

Y calculated mean asbestos mass fraction, %

Figure 2 — Maximum calculated mean mass fraction of asbestos for which the 95 % upper confidence limit of the mass fraction is lower than 0,1 % for a given number of asbestos points



Key

X number of asbestos points

Y calculated mean asbestos mass fraction, %

Figure 3 — Minimum calculated mean mass fraction of asbestos for which the 95% lower confidence limit of the mass fraction is higher than 0,1% for a given number of asbestos points

17.5 Quantitative SEM or TEM fibre counting

The result of quantitative determination of mass from SEM or TEM fibre counts using the mass counting protocol is subject to statistical variability. Although the mass counting protocol requires that the largest fibre contribute no more than approximately 10 % of the total mass, consider the sensitivity of the final result to Poisson variability of the largest particles when determining compliance with a legislated control limit.

18 Test report

The test report shall include at least items a) to i) as follows:

a) a reference to this part of ISO 22262 (i.e. ISO 22262-2) and the applicable clause/subclause;

b) the identification of the sample, including the location (if known by the analyst);

c) the date of the analysis;

d) the identity of the analyst;

e) the variety or varieties of asbestos detected and the mass fraction of each asbestos variety in percent;

f) individually for each sample, an estimate of the limit of quantification for the specific procedure used;

g) the analytical method used to identify the asbestos;

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

i) a summary of all applicable specimen preparation details.

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

j) a record of all weight measurements and observations made during the gravimetric procedures;

k) the observations made to confirm the identification of the asbestos varieties reported, including any optional procedures;

l) the variety or varieties of any non-asbestos fibres detected, and the observations made which allowed these fibres to be discriminated from asbestos fibres;

An example of a suitable format for the test report is shown in Annex C.

Annex A
(normative)

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

Table A.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 - 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 - 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 - 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.

Table A.1 *(continued)*

Product	Examples of application	Typical asbestos type and mass fraction if asbestos is present	Analysis in accordance with	Optimum analytical procedure
Asbestos cement mouldings	– Standard ashtrays – Flower boxes – Garden articles – Sculptures	10 - 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.
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 - 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 A.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 % - 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 millboards	– 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 - 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.
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.

Table A.1 *(continued)*

Product	Examples of application	Typical asbestos type and mass fraction if asbestos is present	Analysis in accordance with	Optimum analytical procedure
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 feed-throughs through walls required to be fire resistant – Encasing of ventilation ducts	40 - 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	Ashing, treatment with 2 mol/l hydrochloric acid, and separation of aggregate by flotation or sedimentation is the optimum procedure. 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	Ashing and treatment with 2 mol/l hydrochloric acid is the optimum procedure. 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.
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	Ashing and treatment with 2 mol/l hydrochloric acid is the optimum procedure. 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 - 20 % Chrysotile 80 - 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.