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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: $A_{0\text{-}1}B_2C_5T_8O_{22}(OH,F,Cl)_2$, 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.

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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.