
Air quality — Bulk materials —

Part 1:

**Sampling and qualitative determination of
asbestos in commercial bulk materials**

Qualité de l'air — Matériaux solides —

*Partie 1: Échantillonnage et dosage qualitatif de l'amiante dans les
matériaux solides d'origine commerciale*

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Published in Switzerland

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Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 22262-1 was prepared by Technical Committee 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*

The following part is under preparation:

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

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Introduction

In the past, asbestos was used in a wide range of products. Three varieties of asbestos found extensive commercial application. Chrysotile accounted for approximately 95 % of consumption, and this variety is therefore likely to be encountered most frequently during the analysis of samples. Materials containing high proportions of chrysotile asbestos were used in buildings and in industry for fireproofing, thermal insulation, and acoustic insulation. Chrysotile was also used to reinforce materials to improve fracture and bending characteristics. A large proportion of the chrysotile produced was used in asbestos–cement products. These include flat sheets, tiles and corrugated sheets for roofing, pipes and open troughs for the collection of rainwater, as well as pressure pipes for supply of potable water. Chrysotile was also incorporated into products such as decorative coatings and plasters, glues, sealants and resins, floor tiles, gaskets, and road paving. In some products, chrysotile was incorporated to modify rheological properties, e.g. in the manufacture of ceiling tile panels and oil drilling muds. Long textile grade chrysotile fibre was also used to manufacture woven, spun, felted and paper products.

Amosite and crocidolite accounted for almost all of the remaining asbestos use. Amosite was widely used as fireproofing and in thermal insulation products, e.g. pipe coverings and insulating boards. Crocidolite was also used as fireproofing and in thermal insulation products, but was particularly prized because it is highly resistant to acids, flexible enough to be spun and has high tensile strength for reinforcement. Crocidolite found application as a reinforcing fibre in acid containers such as those used for lead–acid batteries, in high-performance textiles and gaskets, and was particularly important for the manufacture of high-pressure asbestos cement pipes for delivery of potable water.

Three other types of asbestos are currently regulated. Materials containing commercial anthophyllite are relatively rare, but they have also 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 some occurrences of tremolite asbestos in surfacing materials and fireproofing have been found in Japan. Tremolite asbestos and actinolite asbestos sometimes occur as contaminants of other commercial minerals. Other minerals can also occur as asbestos. For example, richterite asbestos and winchite asbestos occur at mass fractions between 0.1 % and 6 % associated with 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 materials in which asbestos was intentionally added at mass fractions lower than 0,1 %.

In this part of ISO 22262, procedures for collection of samples and qualitative analysis of commercial bulk materials for the presence of asbestos are specified. The primary method used to identify asbestos is polarized light microscopy. Because of the wide range of matrix materials into which asbestos was incorporated, polarized light microscopy cannot provide reliable analysis of all types of asbestos-containing materials in untreated samples. The applicability of polarized light microscopy can be extended by the use of simple treatments such as ashing and treatment with acid. Optionally, either scanning electron microscopy or transmission electron microscopy may be used as an alternative or confirmatory method to identify asbestos.

Although this part of ISO 22262 specifies that, optionally, a visual estimate of the asbestos mass fraction within very broad ranges may also be made, it is recognized that the accuracy and reproducibility of such estimates is very limited. Quantitative determination of the asbestos content can be needed for a number of reasons, e.g. assessment and management of the risk from asbestos materials in buildings or to comply with regulatory definitions for asbestos-containing materials. The necessity to quantify asbestos in a material depends on the maximum mass fraction that has been adopted by the jurisdiction to define an asbestos-containing material for the purpose of regulation. Definitions range from “any asbestos” to 0,1 %, 0,5 % or 1 %. For jurisdictions in which an asbestos-containing material is defined as one containing “any asbestos”, a particular problem is how to determine whether a material does not contain asbestos, since all methods have limits of detection.

For practical purposes, since no known commercial materials exist in which commercial asbestos was intentionally added at mass fractions lower than 0,1 %, this part of ISO 22262 specifies that samples be classified as asbestos-containing (i.e. containing more than 0,1 % asbestos) if either chrysotile, amosite, crocidolite or anthophyllite, or any of these varieties in combination, is detected in the analysis. When the definition of an asbestos-containing material is either 0,5 % or 1 %, depending on the nature of the product, it is often necessary to proceed to other parts of this International Standard in order to quantify the asbestos for the purpose of defining the regulatory status of the material.

The occurrence of tremolite, actinolite or richterite/winchite in a material is usually a consequence of natural contamination of the constituents, and the detection of these minerals does not necessarily indicate that the mass fraction is more than 0,1 % asbestos. Accordingly, determination of the regulatory status of these materials by any of the criteria can often be achieved only by quantitative analysis. Since these minerals were not specifically mined and utilized for their fibrous properties, they may also occur in materials as either non-asbestiform or asbestiform analogues, or as mixtures of both. Evaluation of these types of material may require a more detailed analysis.

Simple analytical procedures such as polarized light microscopy are not capable of detecting or reliably identifying asbestos in some types of commercial products containing asbestos, either because the fibres are below the resolution of optical microscopy or because the matrix material adheres too strongly to the fibres. For these types of product, it may be necessary to utilize electron microscopy.

For a list of parts of this International Standard, see the Foreword.

The method specified in this part of ISO 22262 is based on MDHS 77,^[11] VDI 3866 Part 1,^[13] VDI 3866 Part 4,^[14] VDI 3866 Part 5,^[15] AS 4964-2004,^[8] EPA/600/R-93/116,^[10] and NF X46-020:2008.^[12]

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Air quality — Bulk materials — Part 1: Sampling and qualitative determination of asbestos in commercial bulk materials

IMPORTANT — The electronic file of this document contains colours which are considered to be useful for the correct understanding of the document. Users should therefore consider printing this document using a colour printer.

1 Scope

This part of ISO 22262 specifies methods for sampling bulk materials and identification of asbestos in commercial bulk materials. This part of ISO 22262 specifies appropriate sample preparation procedures and describes in detail the procedure for identification of asbestos by polarized light microscopy and dispersion staining.

This part of ISO 22262 also specifies simple procedures for separation of asbestos fibres from matrix materials such as asphalt, cement, and plastics products. Optionally, identification of asbestos can be carried out using scanning electron microscopy or transmission electron microscopy with energy dispersive X-ray analysis. Information is also provided on common analytical problems, interferences and other types of fibre that may be encountered in the analysis.

This part of ISO 22262 is applicable to qualitative identification of asbestos in specific types of manufactured asbestos-containing products and commercial minerals. This part of ISO 22262 is applicable to the analysis of fireproofing, thermal insulation, and other manufactured products or minerals in which asbestos fibres can readily be separated from matrix materials for identification.

NOTE This part of ISO 22262 is intended for use by microscopists who are familiar with polarized light microscopy methods and the other analytical procedures specified (References [16]–[19]). It is not the intention of this part of ISO 22262 to provide instruction in the fundamental analytical techniques.

2 Terms and definitions

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

2.1

achromat

microscope objective in which chromatic aberration is corrected for two wavelengths and spherical aberration and other aperture-dependent defects are minimized for one other wavelength (usually about 550 nm)

EXAMPLE One wavelength less than about 500 nm, the other greater than about 600 nm.

NOTE This term does not imply any degree of correction for curvature of image field; coma and astigmatism are minimized for wavelengths within the achromatic range.

[ISO 10934-1:2002,^[3] 2.6]

2.2

acicular

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

[ISO 13794:1999,^[4] 2.1]

2.3

alpha refractive index

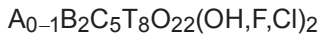
α

lowest refractive index exhibited by a fibre

2.4

amphibole

group of rock-forming ferromagnesium silicate minerals, closely related in crystal form and composition, and having the nominal formula:



where

A is K, Na

B is Fe²⁺, Mn, Mg, Ca, Na

C is Al, Cr, Ti, Fe³⁺, Mg, Fe²⁺

T is Si, Al, Cr, Fe³⁺, Ti

NOTE 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°.

[ISO 13794:1999,^[4] 2.2]

2.5

amphibole asbestos

amphibole in an asbestiform habit

[ISO 13794:1999,^[4] 2.3]

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2.6

analyser

polar used after the object to determine optical effects produced by the object on the light, polarized or otherwise, with which it is illuminated

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NOTE It is usually positioned between the objective and the primary image plane.

[ISO 10934-1:2002,^[3] 2.117.1]

2.7

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.

2.8

asbestiform

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

[ISO 13794:1999,^[4] 2.6]

2.9

asbestos

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

NOTE 1 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).

[ISO 13794:1999,^[4] 2.7]

NOTE 2 Other varieties of asbestiform amphibole, such as richterite asbestos and winchite asbestos (Reference [20]), are also found in some products such as vermiculite and talc.

2.10

aspect ratio

ratio of length to width of a particle

[ISO 13794:1999,^[4] 2.10]

2.11

Bertrand lens

intermediate lens which transfers an image of the back focal plane of the objective into the primary image plane

NOTE The Bertrand lens is used for conoscopic observation in polarized light microscopy and for adjustment of the microscope illuminating system, especially in phase-contrast and modulation-contrast microscopy.

[ISO 10934-1:2002,^[3] 2.87.2]

2.12

birefringence

quantitative expression of the maximum difference in refractive index due to double refraction

[ISO 10934-1:2002,^[3] 2.16]

2.13

camera length

equivalent projection length between the specimen and its electron diffraction pattern, in the absence of lens action

[ISO 13794:1999,^[4] 2.12]

2.14

chrysotile

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



NOTE 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.

[ISO 13794:1999,^[4] 2.13]

2.15

cleavage

breaking of a mineral along one of its crystallographic directions

[ISO 13794:1999,^[4] 2.14]

2.16

cleavage fragment

fragment of a crystal that is bounded by cleavage faces

NOTE 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.

2.17

crossed polars

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

[ISO 10934-1:2002,^[3] 2.117.2]

2.18

***d*-spacing**

distance between identical adjacent and parallel planes of atoms in a crystal

[ISO 13794:1999,^[4] 2.18]

2.19

dispersion

variation of refractive index with wavelength of light

[ISO 7348:1992,^[1] 05.03.26]

2.20

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 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.

2.21

electron diffraction

technique in electron microscopy by which the crystal structure of a specimen is examined

[ISO 13794:1999,^[4] 2.19]

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2.22

electron scattering power

extent to which a thin layer of substance scatters impinging electrons from their original directions

[ISO 13794:1999,^[4] 2.20]

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2.23

energy dispersive X-ray analysis

EDXA

measurement of the energies and intensities of X-rays by use of a solid-state detector and multichannel analyser system

[ISO 13794:1999,^[4] 2.22]

2.24

eucentric

condition in which the area of interest of an object is placed on a tilting axis, at the intersection of the electron beam with that axis, and is in the plane of focus

[ISO 13794:1999,^[4] 2.23]

2.25

extinction

condition in which an optically anisotropic object appears dark when observed between crossed polars

[ISO 10934-1:2002,^[3] 2.51]

NOTE Extinction occurs when the vibration directions of the crystal are parallel to the vibration directions in the polarizer and analyser.

2.26

extinction angle

angle between the extinction position and the position at which the length of a fibre is parallel to the polarizer or analyser vibration directions

2.27**fibril**

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

[ISO 13794:1999,^[4] 2.25]

2.28**fibre**

elongated particle which has parallel or stepped sides

[ISO 13794:1999,^[4] 2.26]

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

2.29**fibre bundle**

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

NOTE A fibre bundle may exhibit diverging fibres at one or both ends.

[ISO 13794:1999,^[4] 2.27]

2.30**gamma refractive index**

γ

highest refractive index exhibited by a fibre

2.31**habit**

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

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[ISO 13794:1999,^[4] 2.30]

2.32**high-efficiency particulate air filter****HEPA**

filter that is at least 99,97 % efficient by volume on 0,3 μm particles

[ISO 14952-1:2003,^[6] 2.13]

2.33**isotropic**

having the same properties in all directions

[ISO 14686:2003,^[5] 2.23]

2.34**Köhler illumination**

method of illuminating specimens in which an image of the illumination source is projected by a collector into the plane of the aperture diaphragm in the front focal plane of the condenser, which then projects an image of an illuminated field diaphragm at the opening of the collector into the specimen plane

2.35**lamda zero**

λ_0

matching wavelength corresponding to the dispersion staining colour shown by a particle in an immersion medium

NOTE At this wavelength, the particle and the immersion medium have the same refractive index.

2.36
matrix

material in a laboratory sample within which fibres are dispersed

2.37
Miller index

set of either three or four integer numbers used to specify the orientation of a crystallographic plane in relation to the crystal axes

[ISO 13794:1999,^[4] 2.33]

2.38
pleochroism

property of an optically anisotropic medium by which it exhibits different brightness and/or colour for different directions of light propagation, or for different vibrations, on account of variation in selective spectral absorption of transmitted light

2.39
polarized light

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

NOTE The vector of vibration may describe a linear, circular or elliptical shape.

[ISO 10934-1:2002,^[3] 2.88.1]

2.40
polarizer

polar placed in the light path before the object

[ISO 10934-1:2002,^[3] 2.117.4]

2.41
polar

device which selects plane-polarized light from natural light

[ISO 10934-1:2002,^[3] 2.117]

2.42
refractive index

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

[ISO 10934-1:2002,^[3] 2.124]

2.43
retardation

difference in optical path length expressed in wavelengths, length units or phase angles between two mutually perpendicular plane-polarized waves

[ISO 10934-1:2002,^[3] 2.128]

2.44
selected area electron diffraction

technique in electron microscopy in which the crystal structure of a small area of a sample is examined

[ISO 13794:1999,^[4] 2.38]

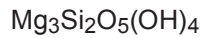
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2.45**serpentine**

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



[ISO 13794:1999,^[4] 2.39]

2.46**sign of elongation**

description of the directions of the high and low refractive indices in a fibre

NOTE The fibre is described as positive when the higher refractive index is parallel to the length of the fibre, and negative when the lower refractive index is parallel to the length of the fibre.

2.47**temperature coefficient of refractive index**

measure of the change of refractive index of a substance with temperature

2.48**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

[ISO 13794:1999,^[4] 2.41]

2.49**unopened fibre**

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

[ISO 13794:1999,^[4] 2.42]

2.50**zone-axis**

line or crystallographic direction through the centre of a crystal which is parallel to the intersection edges of the crystal faces defining the crystal zone

[ISO 13794:1999,^[4] 2.43]

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3 Symbols and abbreviated terms

$\frac{dn}{dT}$	change of RI of an immersion medium per degree Celsius change of temperature
n_D^{25}	RI of a liquid for the sodium D line (589,3 nm) and at a temperature of 25 °C
α	lowest RI of an anisotropic particle
β	intermediate RI of an anisotropic particle
γ	highest RI of an anisotropic particle
λ_0	wavelength at which the RI of a particle is equal to the RI of the liquid in which it is immersed
ED	electron diffraction
EDXA	energy dispersive X-ray analysis
FWHM	full width, half maximum