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Foreword

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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 air*.

This second/third/... edition cancels and replaces the first/second/... edition (), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

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

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

— *Part [n]:*

— *Part [n+1]:*

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 concentrations between 0.1% and 6% in vermiculite formerly mined at Libby, Montana, U.S.A. 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 concentration in some products can be very high and in some cases approach 100%, in other products the concentrations of asbestos used were significantly lower and often between 1% and 15%. In some ceiling tile panels, the concentration of asbestos used was close to 1%. There are only a few known materials in which the asbestos concentration used was less than 1%. Some adhesives, sealing compounds and fillers were manufactured in which asbestos concentrations were lower than 1%. There are no known materials in which asbestos was intentionally added at concentrations lower than 0,1%.

In Part 1 of this standard, procedures for collection of samples and procedures for qualitative analysis of commercial bulk materials for the presence of asbestos are specified. A visual estimate of the asbestos concentration may also be made, but it is recognized that the accuracy and reproducibility of such estimates is very limited. For practical purposes, since no known commercial materials exist in which commercial asbestos was intentionally added at concentrations lower than 0,1%, Part 1 of this standard 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. Because of the wide range of matrix materials into which asbestos was incorporated, polarized light microscopy cannot provide reliable analyses 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. However, there are some classes of commercial asbestos-containing material that cannot be reliably analyzed by polarized light microscopy. The occurrence of tremolite, actinolite or richterite/winchite in a material is usually as a consequence of natural contamination of the constituents, and the detection of these minerals does not necessarily indicate that the concentration is more than 0,1% asbestos. Accordingly, classification of these minerals as asbestos-containing by the 0,1%criterion can only be achieved by quantitative analysis. Since these minerals were not specifically mined and utilized for their fibrous properties, they may occur in materials as either non-asbestiform or asbestiform analogues, or mixtures of both.

Other parts of this standard specify procedures for quantification of asbestos concentrations below approximately 5%, and quantitative determination of asbestos in vermiculite, other industrial minerals and commercial products that incorporate these minerals.

In Part 1 of this standard, the primary method for identification of asbestos is polarized light microscopy. Optionally, either scanning electron microscopy or transmission electron microscopy may be used as an alternative or confirmatory method to identify asbestos.

It is recognized that asbestos-containing materials are defined in some jurisdictions as materials containing more than 0.5% or more than 1% by weight of asbestos. Some commercially manufactured materials are known in which asbestos was intentionally added at concentrations that may have been either lower than or higher than these defined control limits. For these specific types of asbestos-containing material, it may be necessary to proceed to other parts of this standard in order to quantify the asbestos for the purpose of defining the regulatory status of the material.

This method is based on MDHS 77 [1], VDI 3866 Part 1 [2], VDI 3866 Part 4 [3], VDI 3866 Part 5 [4], AS 4964-2004 [5] and EPA/600/R-93/116 [6].

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

1 Scope

1.1 General

Part 1 of this series of standards specifies methods for sampling of bulk materials and identification of asbestos in commercial bulk materials. This series of standards is for application by knowledgeable analysts who are familiar with the analytical procedures specified [7,8,9,10]. It is not the intent of these standards to provide instruction in the fundamental analytical techniques.

Part 1 of this standard is intended for use by microscopists who are familiar with polarized light microscopy methods. The method specifies the appropriate sample preparation procedures and describes in detail the procedure for identification of asbestos by polarized light microscopy and dispersion staining. The method is suitable for most commercially manufactured asbestos-containing materials. Optionally, identification of asbestos may be carried out using scanning electron microscopy or transmission electron microscopy with energy dispersive x-ray analysis, where these facilities are available. Information is also provided on common problems, interferences and other types of fibre that may be encountered.

Part 1 of the standard is restricted to qualitative identification of asbestos in specific types of manufactured products. 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 concentration exceeding 0,1%. This method is applicable to fireproofing, thermal insulation and other manufactured products in which asbestos fibres can readily be separated from matrix materials for identification. Information is provided on simple procedures for separation of asbestos fibres from matrix materials such as asphaltic products. Detection of tremolite, actinolite or richterite/winchite does not allow any assumptions regarding the concentration, and it is necessary to discriminate between the asbestiform and non-asbestiform analogues of these minerals.

Simple analytical procedures such as polarized light microscopy are not capable of detecting or reliably identifying asbestos in some types of product, 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. It is important for the analyst to recognize the limitations of polarized light microscopy for analysis of some types of samples.

1.2 Substance determined

This International Standard specifies a number of reference methods for determination of asbestos in solid materials. The Standard consists of several parts. Part 1 of this series of standards provides a method for qualitative analysis of specific commercial products for the presence of asbestos (chrysotile, amosite, crocidolite, tremolite, actinolite, anthophyllite and richterite/winchite). Other Parts of this series of standards provide methods for analysis of specific types of commercial products for which the use of PLM on the untreated sample yields unacceptable rates of error, and quantification of asbestos in the low concentration range below approximately 5%.

1.3 Type of sample

The method defined in Part 1 of this series of standards is applicable to sampling and analysis of commercial products from which individual fibres of asbestos can be manually separated from the matrix material, either

by picking fibres from surfaces and newly fractured surfaces, or after chemical treatments, acid extraction or ashing, such that the fibres can be identified by one of the specified identification methods. Part 1 is generally applicable to asbestos-containing building materials such as fireproofing, thermal pipe and boiler insulations, asbestos cement, plasters, roofing and other similar materials.

1.4 Range

Experience from proficiency testing has shown that the range of the standard when it is applied to a suitably prepared sample in which the asbestos fibres are sufficiently large to be optically-visible using a low magnification stereo microscope is from less than 0,1% to 100%. The lower end of the range can be extended downwards by use of appropriate techniques.

1.5 Limit of detection

The limit of detection of this method is defined as the detection and identification of one fibre or fibre bundle in the amount of sample examined. The limit of detection 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 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 tailored to the nature of the sample, the limit of detection can be significantly lower than 0,01%.

2 Normative references

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

ISO xyz (all parts), *General title of the series of parts*

3 Principle

A suitable tool is used, in compliance with the relevant safety regulations, to take a sample from the material to be analyzed. The sample is then appropriately packed and labelled for transportation to the laboratory.

A representative sample of the bulk material is initially examined using a stereo-binocular microscope. Typical fibres are removed using tweezers and mounted in appropriate liquid immersion media on slides for examination by polarized light microscopy. Asbestos fibres are identified based on morphology, colour, pleochroism, and the α (lowest) and γ (highest) refractive indices qualitatively assessed using the dispersion staining technique. Detection of commercial asbestos (chrysotile, amosite, crocidolite or anthophyllite), either alone or in combination, is assumed to indicate that the asbestos is present at a concentration exceeding 0,1%. Optionally, a visual estimate of the asbestos concentration is reported in one of several broad concentration ranges. Tremolite, actinolite and richterite/winchite are identified by the same procedure, but since they are usually present as contamination of mineral products, detection of these minerals does not provide information as to their concentration. Optionally, fibres may be identified by SEM or TEM.

4 Terms and definitions

For the purposes of this document, the following apply.

4.1 Achromat
A microscope objective in which chromatic aberration is minimized for two wavelengths (one less than approximately 500 nm, and the other greater than approximately 600 nm), and spherical aberration and other aperture-dependent effects are minimized for another wavelength (usually approximately 550 nm).

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

4.3 Alpha refractive index
The lowest refractive index exhibited by a fibre, denoted by α.

4.4 Amphibole
A 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.

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^{o} and 124^{o}.

4.4 Amphibole asbestos
Amphibole in an asbestiform habit.

4.5 Analyzer
A polarizing filter used after the object (usually between the objective and the primary image plane) to determine optical effects produced by the object on the light, polarized or otherwise, with which it is illuminated.

4.6 Anisotropic
A term applied to a transparent particle having different refractive indices depending on the vibration direction of light.

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

4.8 Asbestos
A 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 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 [11], are also found in some products such as vermiculite and talc.

4.9 Aspect ratio
The ratio of length to width of a particle.

4.10 Bertrand lens
An intermediate lens that transfers an image of the back focal plane of the objective into the primary image plane.

NOTE This lens is used to view the back focal plane of the objective to adjust alignment of the dispersion staining central stop within the aperture.

4.11 Birefringence
The maximum difference between the refractive indices due to double refraction.

4.12 Chrysotile
A fibrous mineral of the serpentine group which has the nominal composition:

$Mg_3Si_2O_5(OH)_4$

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.

4.13 Cleavage
The breaking of a mineral along one of its crystallographic directions.

4.14 Cleavage fragment
A fragment of a crystal that is bounded by cleavage faces.

4.15 Crossed polars
Illumination conditions on a polarized light microscope in which the polarizer and analyzer vibration directions are perpendicular, and both are in the light path.

4.16 Dispersion
The variation of refractive index with wavelength of light.

4.17 Dispersion staining
An 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.

4.18 Energy dispersive X-ray analysis
Measurement of the energies and intensities of X-rays by use of a solid-state detector and multi-channel analyzer system.

4.19 Extinction
The condition in which an anisotropic crystal appears dark when observed between crossed polars.

NOTE This occurs when the vibration directions of the crystal are parallel to the vibration directions in the polarizer and analyzer.

4.20 Extinction angle
For a fibre, the angle between the extinction position and the position at which the length of the fibre is parallel to the polarizer or analyzer vibration directions.

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

4.22 Fibre
An elongated particle which has parallel or stepped sides. For the purposes of this International Standard, a fibre is defined to have an aspect ratio equal to or greater than 3:1.

4.23 Fibre bundle
A structure composed of parallel, smaller diameter fibres attached along their lengths.

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

4.24 Gamma refractive index
The highest refractive index exhibited by a fibre, denoted by γ.

4.25 Habit
The characteristic crystal growth form or combination of these forms of a mineral, including characteristic irregularities.

4.26 Isotropic
Description of a material that has a single refractive index.

4.27 Köhler illumination
A 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. The condenser then projects an image of an illuminated field diaphragm at the opening of the collector into the specimen plane.

4.28 Lamda zero (λ_o)
The wavelength of 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.

4.29 Matrix
The material in a bulk sample within which fibres are dispersed.

4.30 Pleochroism
The 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.

4.31 Polarized light
Light in which there is only one vibration direction.

4.32 Polarizer
A polar placed in the light path before the specimen.

4.33 Polar
A component of a microscope that permits light of only one vibration direction to be transmitted.

4.34 Refractive Index
The ratio of the speed of light in a vacuum to that in the given medium.

4.35 Retardation
The slower propagation of a wavefront in a medium of high refractive index as compared with that in a medium of low refractive index.

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

$Mg_3Si_2O_5(OH)_4$

4.37 Sign of elongation
A 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.

4.38 Temperature coefficient of refractive index
A measure of the change of refractive index of a substance with temperature.

4.39 Twinning
The 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.

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

5 Symbols and abbreviated terms

eV	-	electron volt
kV	-	kilovolt
ED	-	electron diffraction
EDXA	-	energy dispersive x-ray analysis
FWHM	-	full width, half maximum
HEPA	-	high efficiency particle absolute
MEC	-	mixed esters of cellulose
PC	-	polycarbonate
PCM	-	phase contrast optical microscopy
RI	-	refractive index
SAED	-	selected area electron diffraction
SEM	-	scanning electron microscope
TEM	-	transmission electron microscope
UICC	-	Union Internationale Contre le Cancer
n_D^{25}	-	RI of a liquid for the sodium D line (589.3 nm) and at a temperature of 25°C
$\frac{dn}{dT}$	-	change of RI of an immersion medium per °C change of temperature
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

6 Sample collection

6.1 Requirements

6.1.1 Sampling apparatus

Depending on the nature of the material to be sampled, an appropriate tool is required for collection of the sample. If the material is soft, such as thermal insulation or fireproofing, a knife or scalpel may be sufficient. In other situations, a cork borer may be used to sample all of the layers of a layered material. If the material is

hard, such as asbestos-cement, tools such as pliers, a wire cutter, hammer and chisel, or a rotating hole saw may be needed.

6.1.2 HEPA vacuum cleaner

If the material being sampled is dusty, a HEPA vacuum cleaner approved for asbestos is required to clean the area around the sampling location after collection of the sample.

6.1.3 Materials and supplies for sampling

6.1.3.1 Wetting agent

A wetting agent may be used to limit the dispersion of dust during the collection of the sample. Water, or water to which a small amount of surfactant has been added, may be applied to the surface before sampling using a spray bottle or brush.

NOTE If a sample is being collected for the purpose of product identification, it is important that no wetting agent be used, since this may result in dissolution and loss of water-soluble constituents

6.1.3.2 Filler

After collection of the sample, a minor repair may be necessary to seal the damaged area. Depending on the circumstances, spray paint, touch-up paint or plaster may be used.

6.1.3.3 Sample containers

Appropriate dust-tight containers are required for packaging the sample. Plastic bags with "zip" closures or bottles with screw caps may be used.

6.1.3.4 Labels

A method for labelling of the samples is required. Self-adhesive paper labels may be used. Alternatively, a waterproof marker may be sufficient for field use.

6.1.3.5 Dust mask

A dust mask approved for respiratory protection against airborne asbestos fibres

6.1.3.6 Light

Either a flashlight or an appropriate light source is required for collection of samples in dark locations.

6.1.3.7 Plastic bags

Plastic bags of appropriate size are required for collection of waste materials generated during sample collection.

6.1.3.8 Cleaning supplies

Cleaning materials, such as disposable paper towels and a supply of water, are required for cleaning of sampling tools to avoid cross-contamination between samples.

6.1.3.9 Location identifiers

The use of some means of identifying the precise location from which each sample is taken is recommended, since it may be necessary to re-sample the material at a later time to resolve discrepancies if they arise. A location identifier is invaluable if the sample collected is found to be not representative of the overall area, such as if the sample has been taken from a patch in a location that has been repaired. A specific colour of spray paint, or appropriate permanent labels applied to the precise location may be used.

6.2 Procedure

6.2.1 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. Exceptions from the regulations are generally permitted for some types of activity that are minimally invasive, such as the removal of material samples for analysis.

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 sampling materials suspected of containing asbestos. If the handling instructions in this clause are followed, it may be assumed that there will be no substantial release of fibres. In exceptional cases, more extensive precautions may be necessary to prevent the release of airborne fibres.

Sometimes different materials may have been applied to a surface as several layers. It is recommended that samples of all of the individual layers be collected. If a borer or hole sawing device is used to penetrate several layers, the device should be operated so that it rotates slowly. This will ensure that only coarse turnings are produced. High-speed devices are not recommended, since it is then necessary to take more complex safety precautions such as local suction and filtration to collect the dust generated.

6.2.2 Sample size requirements

6.2.2.1 General

Although only a few milligrams of sample are required for the analytical methods specified, it is necessary to take into account the homogeneity of the material, and to assure that the sample is of a sufficient size that it is representative of the material under investigation. If inspection shows that the material is finely divided and homogeneous to the unaided eye, or if the nature of the material is recognized as such from previous knowledge, a minimum sample size of approximately 5 cm^3 generally provides sufficient material for analysis.

6.2.2.2 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. 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 are 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 must be collected to ensure that the sample is representative of the material.

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

In addition to the problem of inhomogeneity, the possibility that repairs using materials from different sources may have occurred needs to be considered. For example, during renovation or repairs, some asbestos-free ceiling tiles may have been installed in a suspended ceiling, the balance of which contain asbestos, for no other reason than such ceiling tiles were readily available at the time. During repairs or rebuilding, other materials of the same appearance, but having different compositions, may have been used to repair damage to fireproofing, thermal insulation or bulkheads.

It is important to recognize that the analytical result relates only to the actual sample tested. If the sample collected is not representative, the result will not be representative of the material.

Annex A, which lists the asbestos-containing materials most frequently used, provides guidance for identifying different types of material.

6.2.2.3 Number of samples

The number of samples to be taken is dependent on the nature of the material, whether the material is homogeneous or inhomogeneous, and the size of the area under consideration. In the case of materials known from prior experience to be homogeneous, it may be sufficient to collect one sample, although collection of more than one sample provides additional confidence that the results are representative of the material being sampled. When materials are suspected to be inhomogeneous, it is necessary to collect several samples and also to ensure that each of the samples is of sufficient size. If it is intended to determine the range of asbestos content in an area of material, all of the samples must be analyzed individually. Otherwise, such samples may be combined before analysis in order to ensure that the sample analyzed represents the mean asbestos concentration of the material.

6.2.2.4 Precautions to avoid cross-contamination between samples

It is most important that precautions be taken to ensure that cross-contamination of samples does not occur. Clean all tools used for collecting samples prior to initial use and again after collection of each sample. Use a new and unused container or plastic bag for each sample, and double-bag each sample.

6.2.2.5 Selection of the sampling locations

Selection of the sampling locations depends on the type of area being sampled and on the nature of the product suspected to contain asbestos.

The material being sampled may be known to be homogeneous, for example a manufactured packing material or sheet material. Samples should be collected at locations that are as inconspicuous as possible. Edge, locations that have already been damaged, or locations behind readily detached covers are particularly suitable, provided that there are no reasons to suspect that the material in such locations is not representative.

If the material under test has a layered structure, for example in the case of multi-layer pipe insulations or multi-layer floor coverings, include all layers of the material in the collected sample. Include any coverings or adhesive layers, such as coatings or glues. Do not attempt to separate the layers under field conditions; separation of individual layers for analysis is best performed under controlled conditions in the laboratory.

If the product under test is behind a wall cladding or other covering, power sockets or light switch recesses are frequently suitable as locations for collection of material samples. If it is not possible to gain access in this manner, the claddings or coverings must be cut out in order to make it possible to take samples. These openings should be made at a location that detracts from the visual appearance as little as possible, for example behind baseboards.

6.2.2.6 Taking the samples

For many types of homogeneous material it is usually possible to collect small amounts of sample without visibly defacing the material and without incurring any significant release of airborne fibres. The amount of sample required is usually collected as follows.

(a) If the material is such that a significant release of airborne asbestos fibres may occur during collection of the sample, moisten the sampling location with water from a spray bottle, a water-soaked brush or a moist paper towel;

NOTE Water should not be used if samples are being collected in the vicinity of operating electrical equipment .

(b) if the material appears to be homogeneous, collect a sample area more than 1 cm^2 in the case of thin materials, or a volume greater than 1 cm^3 in the case of materials having a thickness of several centimetres. Remove the sample by breaking it off with pincers or preferably using a sharp cutting tool. If the material appears to be inhomogeneous, collect a sufficient amount of sample to give confidence that the volume of sample is representative of the material;

(c) place each sample in an individual dust-tight container;

(d) wipe the sampling site and the immediate surroundings, keeping them moist, or clean the area around the sample location using a vacuum cleaner with a HEPA filter;

(e) seal the exposed surface from which the sample was taken using touch-up paint, glue or other appropriate sealant;

(f) affix a permanent identification marker to the exact location from which the sample is removed.

6.2.2.7 Sample labelling

Label the sample container clearly, either using a permanent marker pen or by attaching a permanent adhesive label. Confirm that the sample label corresponds to the information on the identification marker affixed to the sampling location.

6.2.2.8 Sampling record

Make a record of the sample that contains at least the following information:

(a) full description of the sample;

(b) all details recorded on the sample label;

(c) precise description of the sampling location;

(d) building identification;

(e) identification of the room (if applicable);

(f) location in the room from which the sample was collected;

(g) the date that the sample was collected;

(h) the name of the person who collected the sample;

(i) whether the sample is a composite derived by combination of separately collected samples;

(j) whether the sample is a multi-layer sample. In the case of multi-layer samples, the positions of each of the relevant layers shall be noted.

If the sampling location is not adequately specified by the details (a) to (f) above, then, in addition,

(k) make a sketch or take a photograph (record the number of the photograph); or, record the position from which the sample was taken on a plan of the building (the drawing identification shall also be noted in the record).

(l) any other relevant data that are available with respect to the sample.

An example of a suitable sampling record is shown in Annex G.

6.2.2.9 Chain of custody

If there is any possibility that the results of sampling and analysis will be subject to litigation or legal scrutiny, it is most important that records be made of all transfers of samples between individuals, starting with the individual who collected the samples, and through to acceptance of the samples by the analyst. A chain of custody form shall be used for this purpose, on which the date of each transfer, and the name of each individual who has relinquished or accepted possession of the samples are recorded.

6.2.2.10 Storage and transport

The samples shall be packaged in dust-tight containers and a label shall be affixed to the package of samples, indicating that they may contain asbestos. Take care to ensure that unauthorized persons do not have access to the samples. There are no special requirements with respect to climate conditions for storage and transport of the samples. After the samples have been analyzed, they shall be archived for whatever period of time is specified by the individual submitting them to the analytical laboratory.

7 Sample preparation

7.1 General

It is sometimes not possible to identify asbestos in bulk materials because of interference by other constituents, because the concentration of asbestos is too low, or because the asbestos is so inhomogeneously distributed that a large amount of the sample would need to be examined in order to reliably detect and quantify asbestos. In these cases, various chemical or physical preparation methods can be used prior to the microscopic examination to remove a large proportion of the non-asbestos constituents, thus facilitating the detection of asbestos in the smaller amount of material that remains.

7.2 Removal of organic materials by ashing

Chrysotile is often difficult to detect when mixed with large amounts of cellulose, or if it is well dispersed in organic matrices such as asphalt or polyvinyl chloride. Also, some other organic fibres such as spider webs and wool have optical properties similar to those of chrysotile. Ashing of the sample at a temperature of 485°C for a period of approximately 10 hours removes the organic constituents 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.

7.3 Removal of soluble constituents by acid treatment

Matrix constituents such as calcite and gypsum often coat asbestos fibres such that their optical properties cannot be reliably examined. These constituents also often constitute a large proportion of the sample mass. Stirring of a sample in 2M hydrochloric acid for approximately 15 minutes removes many matrix constituents, and this improves the ability to identify and quantify asbestos. The acid treatment slightly reduces the refractive indices of chrysotile, and this must be accounted for when identifying chrysotile by PLM. This acid treatment does not affect the optical properties of any of the other asbestos varieties.

7.4 Sedimentation and flotation

Some materials contain large sizes of aggregate or sand that can be separated in water suspension by sedimentation or flotation. A large proportion of constituents such as vermiculite or perlite can be separated by flotation. Sand or small solid aggregate sediments in water 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.

7.5 Combination of gravimetric reduction procedures

The procedures detailed in 7.2, 7.3 and 7.4 may be combined. It is generally recommended that the procedures be used sequentially in the order given.

8 Analysis by PLM

8.1 Requirements

8.1.1 Stereo-binocular microscope

A stereo-binocular microscope is required for initial observation of samples. The examination is facilitated if the microscope has a continuous range of magnification from approximately 10x to 40x.

8.1.2 Polarized light microscope

A polarized light microscope capable of K hler (or K hler type) illumination is needed for fibre identification. The following optical accessories are necessary:

(a) a light source with blue "daylight" filter;

(b) focusing sub-stage condenser with a numerical aperture (NA) greater than that of any objective used, with a field limiting adjustable aperture;

(c) a focusing ocular with a cross-hair graticule;

(d) strain free objectives with magnifications of 4x, 10x and 40x, or similar magnifications;

(e) polarizer and removable analyzer, the vibration directions of which can be adjusted such that they are at 90 degrees to each other, and can be aligned with the cross-hair in the focusing ocular;

(f) slot between the polarizer and analyzer to allow accessory plates to be inserted at an angle of 45^{o} to the polarizer and analyzer vibration directions;

(g) removable retardation plate with approximately 550 nm retardation, with known slow and fast vibration directions;

(h) dispersion staining objective or a demonstrated functional equivalent;

(i) Bertrand lens or a focussing telescopic ocular to allow observation of the back focal plane of the objective lens;

(j) a level rotating specimen stage for which the centre of rotation can be centred relative to the optical axis of the microscope for each of the objective lenses.

8.1.3 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.

8.1.4 Sample preparation

8.1.4.1 Refractive index liquids

The majority of commercial asbestos-containing products contain only chrysotile, amosite or crocidolite, or mixtures of these three types of asbestos. Identification of these three types of asbestos can be achieved using liquids of RI 1,550, 1,680 and 1,700. The RI values of these liquids are specified for light of wavelength 589,3 nm (sodium D line) at a temperature of 25^{o}C.

For identification of tremolite, actinolite, anthophyllite and richterite/winchite, RI liquids in the range 1,605 to 1,660 are required, at intervals of 0,005.

Suitable calibrated RI liquids are commercially available, and a set of liquids with RI's from 1,500 to 1,700, at intervals of 0,005 gives sufficient range and discrimination.

If commercially available RI liquids cannot be obtained, a set of liquids sufficient for use in this Standard can be prepared [7,12] using common chemical reagents as specified in Table 1.

Table 1. Reagents for preparation of RI immersion media

Reagent	n_D^{25}	$\frac{dn}{dT}$
Glycerol triacetate	1,4277	-0,00048
Ethyl cinnamate	1,5574	-0,00048
Bromobenzene	1,5570	-0,00054
Iodobenzene	1,6173	-0,00054
1-Chloronaphthalene	1,6304	-0,00044
1-Bromonaphthalene	1,6580	-0,00045
1-Iodonaphthalene	1,7004	-0,00044
Di-iodomethane	1,7390	-0,00070

NOTE Commercially-available RI media, and the reagents in Table 1, should be used in accordance with applicable safety precautions.

Table 2 shows the mixtures of reagents required to prepare a set of RI immersion media.

Table 2. Mixtures required for RI liquids

Liquid n_D^{25}	Liquid 1	Volume Percent liquid 1	Liquid 2	Volume Percent liquid 2	$\frac{dn}{dT}$
1,545	Ethyl cinnamate	90,44	Glycerol triacetate	9,56	-0,00048
1,550	**Ethyl cinnamate**	**94,30**	**Glycerol triacetate**	**5,70**	**-0,00048**
1,555	Ethyl cinnamate	98,15	Glycerol triacetate	1,85	-0,00048
1,560	Bromobenzene	95,03	Iodobenzene	4,97	-0,00054
1,605	**Iodobenzene**	**79,60**	**Bromobenzene**	**20,40**	**-0,00054**
1,610	Iodobenzene	87,89	Bromobenzene	12,11	-0,00054
1,615	Iodobenzene	96,19	Bromobenzene	3,81	-0,00054
1,620	1-Chloronaphthalene	85,83	Bromobenzene	14,17	-0,00045
1,625	1-Chloronaphthalene	92,64	Bromobenzene	7,36	-0,00045
1,630	**1-Chloronaphthalene**	**-**		**-**	**-0,00044**
1,635	1-Bromonaphthalene	78,99	Bromobenzene	21,01	-0,00047
1,640	1-Bromonaphthalene	84,05	Bromobenzene	15,95	-0,00046
1,645	1-Bromonaphthalene	89,11	Bromobenzene	10,89	-0,00046
1,650	1-Bromonaphthalene	94,18	Bromobenzene	5,82	-0,00046
1,655	1-Bromonaphthalene	99,24	Bromobenzene	0,76	-0,00045
1,660	1-Bromonaphthalene	90,48	1-Iodonaphthalene	9,52	-0,00045
1,680	**1-Iodonaphthalene**	**54,31**	**1-Bromonaphthalene**	**45,69**	**-0,00044**
1,700	**1-Iodonaphthalene**	**-**		**-**	**-0,00044**

The three primary RI liquids for identification of chrysotile, amosite and crocidolite are indicated in Table 2 in bold type (1,550, 1,680 and 1,700). Tremolite, actinolite or anthophyllite can often be identified using only RI liquids 1,605 and 1,630, also indicated in Table 2 in bold type. Tremolite, actinolite or anthophyllite may be encountered in which the refractive indices are high because of increased iron concentration, and use of other RI liquids in Table 2 may be necessary in order to assess the refractive indices.

8.1.4.2 Asbestos reference standards

Asbestos reference standards are required. Suitable sets of standards are SRM 1866 (chrysotile, crocidolite and amosite) [13] and SRM 1867 (tremolite, actinolite and anthophyllite) [14] available from the U.S. National Institute of Standards and Technology or asbestos reference standards available from the U.K. Institute of Occupational Medicine (Chrysotile (Canada and Zimbabwe), crocidolite, amosite, tremolite, actinolite and anthophyllite) [15]. SRM 1867 tremolite and actinolite are particularly useful for qualitative discrimination between tremolite and actinolite. The International Mineralogical Association (IMA) [16,17] has specified that values of the ratio Mg/(Mg+Fe) below 0.9 are defined as tremolite, and those above 0.9 are defined as actinolite. SRM 1867 tremolite has a value of 0.84, and SRM 1867 actinolite has a value of 0.94, providing reference samples representing compositions just below and just above the IMA boundary. It is important to recognize that the IMA boundary between tremolite and actinolite is only a convention within a continuum of composition in which the iron and magnesium concentrations vary in a reciprocal manner.

Table 3. Optical properties of NIST SRM 1866 and SRM 1867 reference asbestos samples

Property	Chrysotile	Amosite	Crocidolite	Anthophyllite	Tremolite	Actinolite
Colour	White	Grey-brown	Blue	Light Brown	White	White
Pleochroism	None	Very weak	α-blue/γ-grey	None	None	None
Birefringence	Low	Medium	Low	Medium	Medium	Medium
Sign of Elongation	Positive	Positive	Negative	Positive	Positive	Positive
Extinction	Parallel	Parallel	Parallel	Parallel	16,6°	15,9°
γ	1,556	1,701	*	1,636	1,634	1,639
α	1,549	1,679	*	1,615	1,606	1,613

NOTE * For crocidolite, the NIST certificate of analysis states: "Because strong absorption in the visible light range results in anomalous dispersion characteristics that would not be useful to the analyst, no certified values of refractive index are reported for riebeckite."

Table 4. Optical properties of IOM/HSE reference asbestos samples

Property	Chrysotile (Canada)	Chrysotile (Zimbabwe)	Amosite	Crocidolite	Anthophyllite	Tremolite	Actinolite
Colour	White	White	Grey-brown	Blue	White	White	Pale green
Pleochroism	None	None	Very weak	α-blue/γ-grey	None	None	γ-green/α-grey
Birefringence	Low	Low	Medium	Low	Medium	Medium	Medium
Sign of Elongation	Positive	Positive	Positive	Negative	Positive	Positive	Positive
Extinction	Parallel	Parallel	Parallel	Parallel	Parallel	Parallel	Parallel
γ	1,552	1,552	1,692	1,696	1,624	1,632	1,652
α	1,544	1,544	1,676	1,688	1,608	1,616	1,644

8.1.4.3 Sample comminution equipment

An agate mortar and pestle is required for grinding of samples to suitable sizes for PLM examination.

8.1.4.4 Microscope slides

Microscope slides, 75 mm x 25 mm

8.1.4.5 Microscope cover glasses

Microscope cover glasses, 22 mm x 22 mm. The thickness of the cover glasses must be matched with that specified by the objective lenses. A thickness of 0,17 mm is required by many commercial objectives.

8.1.4.6 Thermometer

A thermometer is required to measure the temperature of the microscope slide preparation during observation if accurate refractive indices of asbestos fibres are to be recorded.

8.1.4.7 Alcohol or gas burner

A laboratory burner is sometimes useful for discriminating between organic fibres and asbestos fibres.

8.1.4.8 General laboratory supplies

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

(a) glassine paper sheets, approximately 15 cm x 15 cm, for examination of samples;

(b) scalpel holder and replacement disposable scalpel blades;

(c) sampling utensils, including tweezers, needles and spatulas;

(d) distilled water;

(e) concentrated hydrochloric acid, reagent grade;

(f) crucibles, silica or glazed porcelain, with lids;

(g) petri dishes;

(h) disposable pipettes;

(i) glass filtration assembly, 25 mm or 47 mm diameter;

(i) polycarbonate filters, 0,4 µm pore size, 25 mm or 47 mm diameter.

8.1.4.9 Muffle furnace (optional)

For ashing of samples to remove interfering organic constituents, a muffle furnace with a temperature range up to 500°C, with a temperature stability of ±10°C is recommended.

8.1.4.10 Magnetic stirrer (optional)

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

8.2 Qualitative analysis by PLM

8.2.1 Calibration

It is essential that the optical components of the PLM are fully understood by the analyst, and that the analyst is familiar with the alignment procedure. The alignment of the PLM shall be confirmed prior to conducting any analyses. The designs of microscopes vary, and the alignment instructions provided by the manufacturer should be followed. The critical aspects of the alignment are:

(a) the illumination source and sub-stage condenser must be adjusted such that the field limiting aperture is in focus (K hler or K hler-like illumination);

(b) the centre of rotation of the specimen stage must be aligned with the optical axis of the PLM for each of the objective lenses. This is necessary so that a particle at the centre of the field of view remains at the centre of the field of view during rotation of the stage. This condition is often achieved by centring of the rotation for one objective lens, and then laterally adjusting the position of each of the other objective lenses to align its axis with the centre of the stage rotation;

(c) the vibration directions of the polarizer and analyzer must be at 90^{o} to each other;

(d) the vibration directions of the polarizer and analyzer must accurately coincide with the directions of the cross hair in the ocular. This can be accomplished using a well-formed birefringent crystal with a known zero extinction angle. Alternatively, orientation plates consisting of an accurately mounted crystal with a fiducial line are commercially available. If the microscope has eyepieces that can be freely rotated, fix the position of the eyepiece containing the cross hair using some method such as adhesive tape;

(e) if a mechanical stage is installed on the rotating stage, the directions of the mechanical stage should be adjusted such that the zero angular position of the rotation stage corresponds to lateral motions of the mechanical stage parallel to the polarizer and analyzer directions.

On the initial set-up of the PLM, the vibration direction of the polarizer and the orientation of the vibration directions of the 550 nm retardation plate shall be determined. The vibration direction of the polarizer can be determined by examination of a slide preparation of crocidolite with the polarizer in position and the analyzer withdrawn. Under these conditions, the direction of the length of the crocidolite fibres when the dark blue pleochroism is displayed is the vibration direction of the polarizer. The orientation of the vibration directions of the 550 nm retardation plate can be determined by examination of a fibre of a known reference material such as amosite or chrysotile, and observing the change of interference colour when the retardation plate is inserted. The slow vibration direction of chrysotile or amosite is parallel to the length of the fibre. If the retardation plate adds to the retardation by the fibre, the slow vibration directions of the fibre and the retardation plate are parallel. An interference colour chart is provided in Annex B.

Before using refractive index liquids for identification of asbestos, even if certified liquids are purchased, it is recommended that the refractive indices of liquids be confirmed using reference glass samples or a refractometer. If kept tightly capped, the refractive indices of these liquids remain stable for at least 2 years. Some refractive index liquids degrade when exposed to light, therefore they should be stored in dark bottles, preferably in a dark place.

8.2.2 Sample preparation

For many samples, including fireproofing, thermal insulation and asbestos cement products, during stereomicroscope examination fibres will be observed that can be removed with tweezers. Mount typical suspected asbestos fibres on a microscope slide, and add a drop of the refractive index liquid appropriate for the suspected asbestos variety. If the suspected asbestos variety cannot be confirmed using the appropriate RI liquid, mount additional fibres from the sample on slides using RI liquids appropriate for the other asbestos varieties.

8.2.3 Sample analysis

8.2.3.1 Analytical sequence

The analytical techniques described have been shown to give reliable and reproducible results. Alternative methods can be used if equivalence in terms of detection and identification can be demonstrated. Identification of the asbestos fibres should be based on the following analytical sequence:

(a) make a preliminary visual examination of the whole of the bulk sample to assess the sample type and the required sample treatment (if any). Where possible, take a representative sub-sample at this stage for direct examination by PLM;

(b) carry out any required sample treatment to release or isolate fibres;

(c) perform a detailed and thorough search under the stereo microscope to classify the suspected fibre types present;

(d) mount representative fibres in appropriate RI liquids on microscope slides;

(e) identify the different fibrous components using PLM.

If no asbestos is detected by these procedures, prepare additional slides using random sub-samples of a few milligrams and search for thin asbestos fibres using PLM.

8.2.3.2 Preliminary examination

Examine the entire sample by the unaided eye to describe the type of material or product present, and to establish whether or not visible fibres are present. Note the nature of any matrix materials, as this may indicate the requirement for treatment of the sample. Examine the sample using the stereomicroscope. So far as possible, make an initial determination of the number of fibre types present. Record the appearance, colour and texture of the sample and any fibre types observed. For inhomogeneous or layered samples, it may be necessary to describe each separate layer or part of the sample. Sample preparation and the analysis of the sample are dependent on the quality of the initial visual examination. Also, adequate description of the appearance of the sample is important in establishing whether asbestos is present, or in which part of the sample asbestos is present.

8.2.3.3 Sample treatment

The purpose of any initial sample treatment of bulk samples is to release fibres from any matrix and to remove fine particles adhering to the fibres (both of which obscure the optical effects and hinder the identification). It is necessary to break non-friable samples (with tools if necessary) and then to examine newly-fractured edges using the stereomicroscope to observe any protruding fibres. If samples contain large pieces of hard materials, grinding of the sample may be necessary. Surfaces and edges of hard materials may be abraded to release fibres for examination. Routine procedures used for sample treatment should be fully documented. Any deviations from these procedures for particular samples should be recorded.

Dilute acetic acid or cold dilute hydrochloric acid may be used to remove calcium carbonate (limestone), calcium sulphate (gypsum) and calcium silicate, which are commonly used as binders (e.g. for insulation and asbestos boards) and fillers (e.g. in floor tiles). The removal of calcium magnesium carbonate (dolomite) will require the use of cold concentrated hydrochloric acid. Sufficient acid should be added in small aliquots for several minutes or until effervescence stops. Fibre release may be aided by stirring or by ultrasonic treatment. The sample is then filtered and repeatedly washed with water. Residual acid may degrade the fibres and affect the optical properties, and small crystals of salts may form. The sample may be rinsed with ethanol or other volatile solvents to reduce the drying time.

Organic matrices such as plastics, asphalt, resins or rubber products may require prolonged treatment in solvents to remove the matrix. An effective solvent for any particular sample type can be established only by individual testing or by foreknowledge of the type of matrix. Organic matrices may be removed by treatment in a muffle furnace at 480°C. However, heating may modify the optical properties of some of the asbestos fibres.

8.2.3.4 Stereomicroscope examination

The original samples or portions of sample that have undergone sample treatment should be examined using the stereomicroscope. For many asbestos-containing materials, asbestos fibres can be detected at magnifications within the range of the stereomicroscope. For other types of asbestos-containing material, it may not be possible to detect asbestos fibres using the stereomicroscope. The aim is to detect small fibre bundles, or individual fibres, and tentatively to assign fibre types based on their appearance. This is usually achieved by placing the sample on a piece of glassine paper or in a suitable container and carrying out a detailed search of the entire sample using needles or tweezers to separate the different fibrous components from the matrix. The appearance of these fibres is then noted. The care and vigilance with which the sample is examined at this stage are important in detecting trace quantities of asbestos. Representative fibres or fibre bundles are then selected and mounted for PLM examination.

Describe layered samples by their appearance, and note each distinct layer as a separate entity. Regulations in some jurisdications require that distinct layers are analyzed and reported separately. Other types of inhomogeneous sample will require detailed visual examination of all the different phases observed.

Asbestos is generally recognised by the fineness of its fibres, which are most often present as closely packed bundles of fibrils that will divide along their length when pressure is exerted on them with a probe or tweezers. An analyst will rapidly become familiar with characteristics such as distinctive surface lustre, flexibility and tensile strength. Initial tentative identification of suspected asbestos fibres at this stage will be confirmed or refuted by subsequent examination using PLM, SEM or TEM.

8.2.3.5 Preparation of samples for PLM examination

A tentative identification based on the stereomicroscope evaluation is used to select the most appropriate RI mounting liquid. Fibres selected must be dry and relatively free from other particulate matter. Representative fibres or fibre bundles are chosen and are placed on a clean microscope slide into a drop of RI liquid, and a clean cover glass is lowered gently onto the slide, avoiding trapping of air bubbles. The RI of the liquid selected should be 1,550 for suspected chrysotile, 1,680 for suspected amosite, 1,700 for suspected crocidolite, 1,605 for suspected tremolite or anthophyllite, and 1,630 for suspected actinolite or richterite/winchite.

If no fibres have been seen in the bulk sample using the stereomicroscope, or no asbestos fibres have been identified by PLM, then tweezers or probes should be used to take random sub-samples, after the bulk sample has undergone suitable treatment (if necessary). At least two microscope slide preparations should be made with appropriate RI liquids for examination by PLM. Any large agglomerates should be teased apart with tweezers or needles, or sheared gently between two microscope slides, to give an even distribution of particles. Selection of large particles or fibre bundles may cause tilting of the cover slip and should be avoided. The amount of sample distributed should be such that the appearance and properties of individual fibres are not obscured by other particles.

8.2.3.6 Identification of asbestos by PLM and dispersion staining

Identification of a single asbestos fibre requires the observation of the following properties in the stated observation modes:

(a) morphology – observed in all illumination conditions;

(b) colour and pleochroism – observed in plane polarized light;

(c) birefringence – observed with crossed polars;

(d) extinction characteristics – observed with crossed polars;

(e) sign of elongation – observed with crossed polars and 550 nm retardation plate inserted;

(f) refractive indices - assessed using a dispersion staining objective with polarizer only inserted.

The above order of observations facilitates the assessment of the morphological and optical properties in a logical sequence. Adjust the microscope to give K hler illumination, centre the stage, and insert the polarizer (usually adjusted to the East-West orientation below the condenser. Under these conditions, observe the morphology and colour of the selected fibre. Rotate the stage and observe whether the fibres are pleochroic. Insert the analyzer to give crossed-polars, and rotate the stage to observe birefringence and whether the extinction angle is parallel to the length of the fibre or oblique. With the polars still crossed, insert the 550 nm retardation plate and rotate the stage to determine the sign of elongation. Finally, examine the fibre under dispersion staining conditions to assess the refractive indices for parallel and normal vibration directions. This may be achieved by observing the dispersion colours at the interface between the fibre and the RI liquid. Withdraw the analyzer and the 550 nm retardation plate, increase the illumination, and insert a dispersion staining objective with a central stop in the back focal plane. Adjust the condenser aperture until the field of view becomes dark. View the back focal plane of the objective using either a Bertrand lens or a telescope ocular, and adjust the condenser alignment until the central beam is obscured by the central stop of the lens.

For fibres that exhibit parallel extinction, record the dispersion staining colours with the fibre parallel to the polarizer vibration direction and normal to the polarizer direction. If fibres exhibit oblique extinction, it is necessary to search for fibres that exhibit the maximum extinction angle. This can be achieved either by scanning the slide for such a fibre, or by rotating fibres about their axes by touching the top of the cover slip with a needle. It is only in this orientation that a monoclinic fibre exhibits the γ and the α refractive indices. When such a fibre has been located, record the dispersion staining colours with the fibre at both extinction positions.

In practice, any other sequence may be used provided that all of the required properties are observed. For example, if it is difficult to locate any suspected asbestos fibres on the prepared mount because the sample is dominated by non-asbestos fibres, or if a random sample is being searched, the sample should be scanned with the microscope in the crossed polars condition to detect the asbestos fibres. The sign of elongation may also be observed by interpretation of the observed dispersion staining colours.

The observations made of the morphology and the optical properties of the fibre are recorded. Identification is based on comparing the recorded observations on the fibres selected for analysis (and mounted in the appropriate RI liquid) against the properties of asbestos reference standards. The compositions and optical properties of commercial chrysotile, amosite and crocidolite do not vary significantly, and therefore a close match between the optical properties of the sample fibre and the asbestos standard normally will be achieved. Further representative fibres will need to be examined if the observations are inconclusive, or if more than one type of fibre was found in the stereo or PLM analysis. For tremolite, actinolite and anthophyllite, the iron concentration can vary significantly from one source to another, higher iron concentrations resulting in higher refractive indices. Examples of this variability can be seen by comparing the tremolite, actinolite and anthophyllite samples from the NIST SRM 1867 and IOM sets of reference standards, as illustrated in Annex D.

8.2.3.7 Identification of asbestos

8.2.3.7.1 Morphology

A detailed description for the morphology that is characteristic of asbestos is as follows. This morphology is characteristic of the larger fibres seen in stereomicroscope examinations and for fibres selected from bulk samples for PLM identification of fibre type.

In the light microscope, the asbestiform habit is generally recognized by the following characteristics:

(a) The presence of fibre aspect ratios in the range of 20:1 to 100:1 or higher for fibres longer than 5 µm;

(b) The capability of longitudinal splitting into very thin fibrils, generally less than 0,5 µm in width.

In addition, observation of any of the following characteristics for the fibre type under consideration provides additional confirmation that the fibres are asbestiform:

- Parallel fibres occurring in bundles;
- Fibre bundles displaying frayed ends;
- Fibres in the form of thin needles;
- Matted masses of individual fibres, and/or;
- Fibres showing curvature.

In practice, if chrysotile, crocidolite or amosite is identified in a commercial product, the assumption can safely be made that the fibres are asbestiform, and that these fibres will conform to the description above. This assumption can be made because these three types of asbestos were mined and processed to yield fibres with specific properties for intentional incorporation into products. Some anthophyllite asbestos was used in a few commercial products, but very little was mined and used commercially. However, the amphiboles tremolite, actinolite, and richterite/winchite were not generally used in commerce, and their presence in a product is more likely a consequence of naturally-occurring contamination of one or more of the major constituents. Accordingly, no assumption can be made as to whether the amphibole is asbestiform or non-asbestiform. Anthophyllite can occur as contamination of other mineral products, and in such situations no assumption can be made as to whether it is asbestiform or non-asbestiform. In some samples, these amphiboles may exhibit a mixture of morphological types and quantitative determination of the regulatory status of such samples may require a detailed study of the fibre size distribution that is beyond the scope of Part 1 of this analytical method.

In general, for Part 1 of this analytical method, the presence of either the asbestiform or the non-asbestiform analogues of tremolite, actinolite, anthophyllite or richterite/winchite can usually be specified. If all of the amphibole fibres longer than 5 µm have aspect ratios equal to or lower than 10:1, it can be concluded that the amphibole is non-asbestiform and it shall not be reported as asbestos. If any amphibole fibres longer than 5 µm, thinner than 1 µm, and with aspect ratios exceeding 20:1 are observed, at least that part of the size distribution should be considered to be asbestos. It must be recognized that some samples may still present ambiguities, and such ambiguities, when observed, shall be reported as part of the results.

8.2.3.7.2 Colour and Pleochroism

Colour and pleochroism are observed using plane polarized light. Pleochroism is a diagnostic property in the identification of crocidolite. Crocidolite has a strong absorption, which gives a dark blue colour when the fibres are parallel to the polarizer vibration direction, changing to pale blue or grey when the fibres are perpendicular to the polarizer vibration direction. This is illustrated in Figures D.13 and D.14 in Annex D. Pleochroism in amosite may occur after heating, or occasionally in unheated fibres, depending on the Fe/Mg ratio of the mineral. Chrysotile shows little colour contrast and no pleochroism in plane polarized light. Depending on the iron concentration, actinolite may exhibit a green colour when the fibres are parallel to the polarizer vibration direction, changing to a grey or yellowish colour when the fibres are perpendicular to the polarizer direction. Pleochroism in the IOM actinolite reference sample is illustrated in Figures D.43 and D.44 of Annex D.

8.2.3.7.3 Birefringence

When a particle with more than one refractive index is observed between crossed polars with its planes of vibration at 45° to those of the polarizer, interference colours are observed against the dark background. For asbestos these interference colours depend on the fibre thickness, the birefringence and on the degree of randomness of the fibril orientation about the fibre axis.

Between crossed polars, an asbestos fibre aligned at 45° to the polarizer vibration direction should be clearly visible. Chrysotile has a low birefringence and gives a grey colour for thin fibres, and a white colour or higher first (or even second) order colours for thick fibres. Crocidolite has a low birefringence and anomalous interference colours from grey to pale blue. Amosite has moderate birefringence, giving white interference colours for thin fibres and higher first or second order colours for thick fibres. Tremolite, actinolite and anthophyllite and richterite/winchite similarly exhibit moderate birefringence. Fibres with a variable thickness, for example with wedge shaped cross-sections, will show parallel bands of colour along their lengths, representing lower interference colours for progressively thinner sections. Examples are shown in Annex D.

Isotropic materials have zero birefringence, and therefore do not exhibit interference colours. Between crossed polars, isotropic materials such as man-made vitreous fibres are almost invisible, but, depending on the difference between their refractive index and that of the immersion liquid, will often be seen easily with the 550 nm retardation plate in position, or with slightly uncrossed polars. Interference colours can be used to distinguish asbestos from some natural organic fibres, which may show non-uniform interference along the fibre length and also incomplete extinction.

8.2.3.7.4 Extinction angle

As the microscope stage is rotated through 360°, an asbestos fibre viewed between crossed polars will disappear from view or "extinguish" at four positions each 90° apart, while at an angle of 45° to an extinction position interference colours should be visible. Many fibres, including asbestos, generally show complete extinction when parallel to the vibration directions of the polarizer or the analyzer. Chrysotile, amosite, crocidolite and anthophyllite each show parallel extinction when the fibre is parallel to the vibration direction of the polarizer or analyzer. Tremolite, actinolite and richterite/winchite may exhibit parallel extinction or oblique extinction, depending on the orientation of the fibre and the crystalline nature of the fibre. Highly asbestiform fibres of these amphiboles may show parallel extinction at all axial orientations. Other fibres of high aspect ratio may show oblique extinction, and axial rotation of the fibre by touching the cover glass of the slide with a needle will allow the maximum extinction angle to be determined. Tremolite and some low-iron actinolite fibres that exhibit only parallel extinction cannot easily be discriminated from anthophyllite. However, it is unlikely that **all** of the tremolite or actinolite fibres in a sample would exhibit parallel extinction, and observation of some with oblique extinction angles would confirm the identity of the mineral, with the presumption that parallel extinction fibres with otherwise similar properties are the same mineral species. In these cases, reliable discrimination between anthophyllite and either tremolite or actinolite may only be possible by examination of the compositions of the fibres by SEM or TEM.

8.2.3.7.5 Sign of elongation

The sign of elongation describes the relationship between the length of the fibre and the optical properties. For asbestos fibres the two available vibration directions are parallel to the long axis and perpendicular to it. If the high refractive index vibration direction is parallel to the long axis, then the fibre is described as positive; if the low refractive index vibration direction is parallel to the long axis the fibre is described as negative. Between crossed polars, with the 550 nm retardation plate inserted at 45° to the polarizer and analyzer vibration directions, the sign of elongation can be determined by observing the colours of fibres that previously had given grey or white first order interference colours between crossed polars. For a retardation plate with

the slow direction (usually marked) in the North East-South West direction, the first order colours observed are as follows:

Positive fibre - blue-green with fibre North East – South West

- orange-yellow with fibre North West – South East

Negative fibre - orange-yellow with fibre North East – South West

- blue-green with fibre North West – South East

Crocidolite is the **only** asbestos type that has a negative sign of elongation. However, exposure to heat of about 300°C or higher may result in a reversal of the sign of elongation of crocidolite to positive. In such cases, however, the thermal history of the fibre is usually indicated by a change of colour.

8.2.3.7.6 Refractive indices

The refractive indices of an asbestos fibre are assessed by mounting a clean separated fibre in a liquid of known refractive index and orienting it either parallel or perpendicular to the polarizer vibration direction. One or more observations are conducted to determine whether the refractive index of the fibre is higher than, lower than or equal to, that of the immersion liquid.

Use the dispersion staining objective to view fibres mounted in a liquid with refractive index close to that of the fibre, so that dispersion staining colours can be observed. When dealing with an unknown sample, the observations (a) to (e) below can be used to help choose a suitable refractive index liquid such that the refractive index of the fibre and the liquid are sufficiently close that dispersion staining colours are produced.

Differences in dispersion between particles and liquids mean that, even though the refractive indices match at one wavelength, they may be quite different at others. This leads to colour effects at the particle-liquid interface when fibres are observed in matching refractive index liquids using white light. In practice, it is easiest to observe small bright particles and colours against a black background; these conditions are achieved with a central stop in the back focal plane of the objective when used with an axial beam of light produced by the condenser iris. The colours observed at the particle-liquid interface depend on the precise wavelength at which the refractive index of the liquid and that of the fibres match. When the match of refractive index is at a wavelength of 589,3 nm (the D line of sodium), the colour at the particle-liquid interface is a deep blue-magenta. For central stop dispersion staining, the colour observed indicates how close, and in which direction, the refractive index of the particle differs from that of the immersion medium:

(a) Fibre refractive index >> Liquid refractive index : White

(b) Fibre refractive index > Liquid refractive index: Purple-red/Orange/Yellow

(c) Fibre refractive index = Liquid refractive index : Deep blue/magenta

(d) Fibre refractive index < Liquid refractive index : Blue/Blue-green

(e) Fibre refractive index << Liquid refractive index : White

Different colours are observed when the fibre is oriented parallel or perpendicular to the polarizer vibration direction, arising from the different refractive indices of asbestos fibres in the two perpendicular directions relative to the polarizer vibration direction. Recording of the predominant colours is used to characterize the refractive indices of the fibres. Identification of chrysotile, amosite and crocidolite can be performed with a dispersion staining objective using three high dispersion liquids having the refractive index values 1,550 for chrysotile, 1,680 for amosite and 1,700 for crocidolite. In practice, for commercial chrysotile, because of variations in the fibre composition according to the source, a small range of fibre refractive indices and dispersion staining colours may be encountered. The refractive indices of commercial amosite and crocidolite do not vary significantly. For the purpose of Part 1 of this International Standard, the three refractive index liquids adequately cover the observed range of refractive indices for chrysotile, amosite and crocidolite from all known major commercial sources. Crocidolite from Bolivia is an exception, in that the refractive indices are lower than those from other sources of crocidolite. However, Bolivian crocidolite is very rare in commerce. Should Bolivian crocidolite be encountered, it can be readily recognized on the basis of its fibrous morphology, negative sign of elongation and blue/grey pleochroism.

Identification of tremolite, actinolite and anthophyllite can often be performed using a dispersion staining objective using liquids of refractive index values 1,605 and 1,630. Tremolite or actinolite should be suspected if some of the fibres exhibit oblique extinction, and the γ index observed parallel to the extinction position can be used to define whether the fibre is tremolite or actinolite. If it is important to discriminate between tremolite and actinolite, classify fibres as tremolite if the γ index is estimated to be equal to or lower than 1,637 and as actinolite if the γ index is estimated to be higher than 1,637.

Some sources of talc contain fibres that can be mistaken for anthophyllite. These fibres have intergrowths of both the anthophyllite and talc crystal structures. The fibres exhibit refractive indices that are lower than those of anthophyllite and intermediate between those of talc and anthophyllite. If this type of fibre is present, examine the sample in a liquid of refractive index 1,615. If no γ indices are observed that are higher than 1,615, classify the fibres as talc. Classify any fibres with γ indices equal to or exceeding 1,615 as anthophyllite.

Identification of richterite/winchite asbestos is difficult by PLM alone. Richterite/winchite should be suspected if the sample also contains vermiculite or talc. Attempts to identify richterite/winchite by PLM alone will usually result in classification of the fibres as actinolite, and such an error may be important for regulatory interpretation. Where richterite/winchite is suspected, and the fibres exhibit properties similar to those of actinolite, it is recommended that the fibres be identified by either SEM or TEM.

Annex C shows dispersion staining charts for the α and γ refractive indices of chrysotile, amosite, crocidolite, tremolite, actinolite, anthophyllite and richterite/winchite in the appropriate refractive index liquids. Chrysotile exhibits a small range of refractive indices, depending on the source. For each of the types of asbestos, an acceptable range of colour for the α and γ dispersion staining colours is indicated, representing the observed range in minerals from commercial sources. For chrysotile, it is also important to establish that the λ_o values for the parallel and normal orientations with respect to the polarizer vibration direction do not differ by more than 100 nm in recognition of its low birefringence. For chrysotile, although there is a range of refractive indices depending on the source, studies have shown that the two indices vary in an approximately parallel manner. Annex D (Figures D.1 and D.2) shows examples of chrysotile mounted in 1,550 refractive index liquid, as viewed between crossed polars with the 550 nm retardation plate inserted. Note the fibrillar, wavy appearance, and the blue-green colour in the North East direction, changing to an orange colour when the fibres are rotation into the North West direction, showing that the fibres have a positive sign of elongation. Annex D also shows an example of chrysotile as viewed under dispersion staining conditions (Figures D.3 and D.4), showing magenta for fibres parallel to the vibration direction of the polarizer and blue for fibres normal to the vibration direction of the polarizer. However, it must be considered that the colours exhibited in the two directions will vary, depending on the source of the chrysotile and any heating or acid treatment. Nevertheless, any variation applies to both the α and γ refractive indices, and the difference between the two (birefringence) remains nearly constant.

Annex D shows examples of amosite mounted in 1,680 refractive index liquid, as viewed between crossed polars with the 550 nm retardation plate inserted (Figures D.5 and D.6). The thin fibres exibit a blue green colour in the North East direction, changing to an orange colour when the fibres are rotation into the North West direction, showing that the fibres have a positive sign of elongation. Because of the higher birefringence of amosite, some of the thicker fibres exhibit first and second order interference colours that can be compared with the interference colour chart in Annex B. Annex D also shows amosite viewed under dispersion staining conditions (Figures D.7 and D.8), with a gold colour for fibres parallel to the vibration direction of the polarizer and blue for fibres normal to the vibration direction of the polarizer. Except for heated amosite, these colours vary only slightly for amosite from different sources. The behaviour of heated amosite is also illustrated for the two fibre orientations in Figures D.9 and D.10. Heated amosite exhibits significantly higher refractive indices, and dark brown - light brown pleochroism respectively for fibres parallel and normal to the polarizer vibration directions.

Annex D shows examples of crocidolite mounted in 1,700 refractive index liquid as viewed between crossed polars with the 550 nm retardation plate inserted (Figures D.11 and D.12). The fibres exibit a yellow-orange colour in the North East direction, changing to an blue colour when the fibres are rotation into the North West direction, showing that the fibres have a negative sign of elongation. The birefringence of crocidolite is very low, and so the dispersion staining colours for fibres parallel and normal to the polarizer vibration direction are not very different. However, a lighter blue is discernable for the parallel direction, indicating that the lower refractive index is parallel to the length of the fibre (Figures D.13 and D.14). The blue – grey pleochroism of crocidolite is also shown in Figures D.15 and D.16. The behaviour of heated crocidolite is also illustrated for the two fibre orientations in Figures D.17 and D.18. Heated amosite exhibits dark brown - light brown pleochroism respectively for fibres parallel and normal to the polarizer vibration directions. For heated crocidolite such as that illustrated, the sign of elongation would be positive, and in this condition electron

microscopy with energy dispersive x-ray analysis would be necessary to discriminate between crocidolite and amosite.

Annex D shows examples of NIST SRM 1867 tremolite mounted in 1,605 RI liquid, as viewed between crossed polars with the 550 nm retardation plate inserted (Figures D.19 and D.20). The thin fibres exibit a blue green colour in the North East direction, changing to an orange colour when the fibres are rotated into the North West direction, showing that the fibres have a positive sign of elongation. Because of the moderate birefringence of tremolite, some of the thicker fibres can exhibit first and second order interference colours that can be compared with the interference colour chart in Annex B. Annex D also shows NIST SRM 1867 tremolite viewed under dispersion staining conditions (Figures D.21 and D.22), with a yellow colour for fibres parallel to the extinction position closest to the vibration direction of the polarizer and dark blue for fibres at the other extinction position. The dark blue colour of the fibre in Figure D.22 and the magnitude of the extinction angle indicate that this fibre is presenting the α refractive index at this orientation. Figures D.23 to D.26 show NIST SRM 1867 tremolite in 1.625 RI liquid, which is intermediate between the γ and α indices of the fibres. Additional examples are shown of the IOM Reference tremolite in 1.605 RI liquid in Figures D.35 to D.38. This tremolite exhibits parallel extinction.

Annex D shows examples of NIST SRM 1867 actinolite mounted in 1,630 RI index liquid, as viewed between crossed polars with the 550 nm retardation plate inserted (Figures D.27 and D.28). The thin fibres exibit a blue-green colour in the North East direction, changing to an orange colour when the fibres are rotation into the North West direction, showing that the fibres have a positive sign of elongation. Because of the moderate birefringence of tremolite, some of the thicker fibres can exhibit first and second order interference colours that can be compared with the interference colour chart in Annex B. Annex D also shows NIST SRM 1867 tremolite viewed under dispersion staining conditions (Figures D.29 and D.30), with a purple-red colour for fibres parallel to the extinction position closest to the vibration direction of the polarizer and light blue for fibres at the other extinction position. Additional examples are shown of the IOM Reference actinolite in 1,640 RI liquid in Figures D.39 to D.44. The IOM actinolite is considerably more asbestiform than the NIST SRM 1867 actinolite, exhibits parallel extinction, and it also exhibits pleochroism as illustrated in Figures D.43 and D.44.

Annex D shows examples of NIST SRM 1867 anthophyllite mounted in 1,605 refractive index liquid, as viewed between crossed polars with the 550 nm retardation plate inserted (Figures D.31 and D.32). The thin fibres exibit a blue-green colour in the North East direction, changing to an orange colour when the fibres are rotation into the North West direction, showing that the fibres have a positive sign of elongation. Because of the moderate birefringence of anthophyllite, some of the thicker fibres can exhibit first and second order interference colours that can be compared with the interference colour chart in Annex B. Annex D also shows anthophyllite viewed under dispersion staining conditions (Figures D.33 and D.34), with blue-purple colours for fibres parallel to the vibration direction of the polarizer and light blue for fibres normal to the vibration direction of the polarizer. Figure D.33 shows some fibres that exhibit purple dispersion staining colours. This indicates that the refractive index in that orientation is higher than 1.630, representing the γ index. Other fibres exhibit a blue colour, which indicates that the refractive index in the particular axial orientation is lower than 1.630. This is probably a result of intergrowths of talc in the fibre bundle, since all fibres in this orientation relative to the polarizer direction should exhibit only the γ index. Additional examples are shown of the IOM Reference anthophyllite in 1,605 refractive index liquid in Figures D.45 to D.48.

Annex D shows examples of richterite/winchite mounted in 1,630 refractive index liquid, as viewed between crossed polars with the 550 nm retardation plate inserted (Figures D.49 and D.50). The thin fibres exibit a blue-green colour in the North East direction, changing to an orange colour when the fibres are rotation into the North West direction, showing that the fibres have a positive sign of elongation. Because of the moderate birefringence, some of the thicker fibres exhibit first and second order interference colours that can be compared with the interference colour chart in Annex B. Annex D also shows richterite/winchite viewed under dispersion staining conditions (Figures D.51 and D.52), with a purple colour for fibres parallel to the extinction position closest to the vibration direction of the polarizer and blue for fibres at the other extinction position. Regardless of the highly asbestiform appearance of this sample, the fibres exhibit oblique extinction.

8.2.4 Interferences

8.2.4.1 Heated Asbestos.

Changes occur to asbestos when it is heated. Therefore, care should be taken if sample preparation involves heating the asbestos-containing material. Even short exposure of crocidolite to temperatures of 300°C to 500°C may cause colour changes, and increases in both refractive index and the birefringence. For crocidolite, the changes with heating are: the sign of elongation reverses and the colour changes through grey

then yellow to orange-brown; pleochroism is suppressed at the grey coloration stage, but re-appears on further heating. For amosite the sign of elongation remains positive but the colour changes through yellow to a dark brown, and pleochroism is observed. Thus, heat-degraded crocidolite and amosite cannot be distinguished from each other by light microscopy after exposure to temperatures above about 500°C. The refractive indices of chrysotile increase after significant exposure to temperatures of about 600°C or greater: the birefringence decreases, and in a few cases the sign of elongation changes to negative and the fibres become pale brown. The alteration of asbestos by heat is dependent upon both the duration and the temperature of exposure. Prolonged exposure to high temperatures can result in complete degradation but with judicious sampling unaffected fibres often can be detected in peripheral locations or in debris that became detached during installation. However in extreme situations analytical electron microscopy may be required to aid identification. Examples of heated amosite and crocidolite in plane polarized light are shown in Annex D.

8.2.4.2 Leached chrysotile

Exposure of chrysotile to acidic aqueous media may result in reduction of the refractive indices as a consequence of leaching of magnesium from the crystal structure. Progressive leaching also results in reduction of the birefringence, and ultimately the fibre becomes isotropic. In addition to the action of mineral acids used in some of the procedures in this standard, leaching may also occur in chrysotile exposed to aggressive water (water with only low concentrations of dissolved calcium and magnesium, and with low values of pH). Leached chrysotile may be encountered on the surfaces of chrysotile cement products such as roofing materials after long periods of exposure to rain.

8.2.4.3 Fibres with morphological and/or optical properties similar to those of asbestos

Most of the fibres discussed in the following paragraphs occur infrequently in samples presented for analysis. However, analysts need to be aware of their existence and distinguishing characteristics in PLM. There are five types of fibre that can resemble chrysotile. Some mineral fibres can also superficially resemble amphiboles.

Polyethylene is the most important of the interfering fibres because it is used as an asbestos substitute. Shredded polyethylene resembles chrysotile. In RI liquid 1,550 the dispersion staining colours are within the range for those of chrysotile, although experienced analysts will observe morphological differences and de-saturation of the blue colour perpendicular to the fibres because of the low refractive index in this direction. The birefringence is also higher than that of chrysotile. If polyethylene is suspected, the melting of fibres on a hot plate or in a flame will readily distinguish them from chrysotile.

Fibres from leather have low birefringence and similar dispersion staining colours to chrysotile. At magnifications below 100x, they appear to have similar morphology to that of chrysotile, but they usually exhibit clearly-visible, uniform fibrils. Individual chrysotile fibrils are too small to be seen by PLM, although uniform bundles of fibrils are visible. In most instances the differences between chrysotile and leather can be detected during stereomicroscope examination. If leather is suspected as being present, the sample may be ashed at 400°C to remove it, and then the residual ash can be re-examined for identification of asbestos. Care should be taken to not allow the sample temperature to rise above 500°C.

Macerated aramid fibres may appear to have morphology similar to that of chrysotile, but these fibres can be recognized by their extremely high birefringence showing high order white interference colours. When mounted in refractive index liquid 1,550 the refractive indices are clearly inconsistent with those of chrysotile.

Spider web and natural organic fibres such as cellulose and feathers have refractive indices close to those of chrysotile and show similar interference colours between crossed polars. In a sample with little non-fibrous material, the morphology of these fibres can be readily distinguished from that of chrysotile. However, in samples containing significant particulate material, sometimes only a small portion of the fibre can be observed due to obscuration by the particles and this can lead to misidentification. These fibres can be removed by ashing the sample or exposing individual fibres to a flame.

Talc fibres are thin ribbons that may sometimes be recognized by characteristic morphological twists. For the refractive index parallel to the fibre length, they have a value in the range 1,589 to 1,600, resulting in a pale yellow dispersion staining colour when immersed in refractive index liquid 1,550. The other two refractive indices of talc are in the ranges 1,539 to 1,550 and 1,589 to 1,600, and with a dispersion staining objective, blue and pale yellow colours perpendicular to the fibre are observed in refractive index liquid 1,550 at different orientations as the fibre is "rolled". It is important to demonstrate that the γ index of any straight fibres that do

not exhibit ribbon-like morphology is lower than 1,615, in order to exclude the possibility that the fibres are anthophyllite

Fibrous brucite normally consists of straight white to pale brown fibres but brucite lacks the tensile strength of asbestos. It is brittle and is soluble in acid. Brucite has a negative sign of elongation, which reverses to positive when heated. Sometimes brucite fibres may appear to be isotropic. It is distinguished from chrysotile by its refractive indices. In central stop dispersion staining brucite yields colours of yellow to pale yellow in refractive index liquid 1,550.

Superficially, fibrous wollastonite can be mistaken for tremolite. Fibrous wollastonite has an acicular morphology, is very brittle, and is white in appearance and slowly soluble in acid. After treatment for a short time (for example 15 minutes) in 10% hydrochloric acid, the fibres exhibit etched areas. Wollastonite always displays a non-zero extinction angle. The refractive index almost parallel to the fibre is in the range 1,628 to 1,650. The other two refractive indices are in the ranges 1,626 to 1,640, and 1,631 to 1,653, and are observed across the fibre, at different orientations as the fibre is rolled. A distinctive feature is that the refractive index with the length of the fibre almost parallel to the polarizer vibration direction is intermediate between the two refractive indices observed at the different orientations across the fibre as the fibre is rolled. Examination of many fibres with crossed polars and with the 550 nm retardation plate inserted will show most as having a positive sign of elongation, and fibres in other orientations will appear to have a negative sign of elongation. Gentle pressure on the cover slip with a needle can be used to rotate a fibre and show it to change from a positive to a negative sign of elongation as it is rolled into a different axial orientation.

Diatomaceous earth may exhibit acicular fragments with the appearance of fibres. However, these fibres have a low refractive index of approximately 1,42 and are readily distinguished from asbestos fibres using dispersion staining. Also, there is usually characteristic morphology that can be recognized when the material is examined at magnifications around 500x.

8.2.4.4 Identification of Other Sample Components

A laboratory conducting routine analysis selectively removes fibres for examination and ignores the majority of the non-asbestos materials. The composition of many asbestos products is relatively uniform during the manufacture and a wider knowledge of these non-asbestos materials can be helpful in recognizing many common products or formulations. Because of this, the analyst should become familiar with the information in Annex A.

9 Analysis by SEM

9.1 Requirements

9.1.1 Scanning electron microscope

A scanning electron microscope, with an accelerating voltage of at least 20 kV, is required.

9.1.2 Energy dispersive x-ray system

The SEM shall be equipped with an energy dispersive X-ray analyzer capable of achieving a resolution better than 170 eV (FWHM) on the MnKα peak. The performance of an individual combination of SEM and solid state X-ray detector is dependent on a number of geometrical factors. The x-ray detector must be capable of detecting sodium in crocidolite, in order to permit discrimination between crocidolite and amosite.

9.1.3 Vacuum coating unit

A vacuum coating unit capable of producing a vacuum better than 0,013 Pa shall be used for vacuum deposition of carbon on the SEM specimens. A sample holder is required which will allow the SEM specimens to be continuously rotated and tilted during the coating procedure.

9.2 Calibration

For the purposes of this method, calibration consists of obtaining EDXA spectra from reference samples of chrysotile, amosite, crocidolite, tremolite, actinolite, anthophyllite and richterite/winchite. The chemical

compositions of commercial chrysotile, amosite, crocidolite and anthophyllite do not vary substantially, and comparison of unknown EDXA spectra with those from the three reference asbestos samples constitutes sufficient identification for Part 1 of this analytical method. For most purposes, it is not necessary to discriminate between tremolite and actinolite, since the compositional boundary between them is a matter of convention. When it is necessary to discriminate between tremolite and actinolite, the NIST SRM 1867 tremolite and actinolite samples are particularly useful since these samples have compositions just below and just above the boundary defined by the International Mineralogical Association. In some applications the magnesium may be partially leached from chrysotile, leading to a chemical composition that approaches that of talc. In order to facilitate the discrimination between chrysotile and talc or anthophyllite, it is recommended that an EDXA spectrum also be obtained from a known sample of talc. Use this spectrum to define the upper limit of the magnesium concentration in talc. Examples of EDXA spectra obtained on the NIST SRM 1866 and SRM 1867 samples, the IOM reference asbestos samples, Bolivian crocidolite and richterite/winchite are illustrated in Annex E. For positive identification, reference EDXA spectra from asbestos standards similar to those shown in Annex E should be recorded using the specific combination of SEM and EDXA detector, since the geometries and detector efficiencies vary between different instruments.

9.3 Sample preparation

Select representative fibres, either from the original bulk sample or from the residue remaining after treatment according to procedures in Clause 7. Mount these fibres either directly on a graphite SEM stub or on double-sided adhesive tape on an SEM stub. Place the SEM stub in the vacuum coating unit and evaporate a thin film of carbon on to the surface of the fibres.

9.4 Qualitative analysis by SEM

9.4.1 Sample analysis

The SEM stub with the unknown fibres is examined at a low magnification in the SEM, and EDXA spectra are acquired from regions of the fibres that are clear of other attached particles. The EDXA spectra are compared with the reference spectra.

9.4.1.1 Chrysotile

Classify a fibre as chrysotile if:

(a) the Mg and Si peaks are clear, and comparable in Mg/Si ratio with that of the reference; and,

(b) any Fe, Mn and Al peaks are small.

NOTE Depending on the composition of adjacent or attached particles, other peaks such as Ca or Cl may also be visible.

NOTE Anthophyllite and talc both yield EDXA spectra that conform to this specification, but the Mg/Si peak height ratio for these minerals is lower than that for chrysotile. In order to avoid erroneous classification of talc or anthophyllite as chrysotile, it is important to take account of the Mg/Si peak height ratio, and to calibrate the EDXA detector using known samples of chrysotile and talc.

9.4.1.2 Amosite

Classify a fibre as amosite if:

(a) the Mg, Si and Fe peaks are comparable in ratio to those of the reference amosite;

(b) no statistically significant peaks from Na or Al are present; and,

(c) the Mn peak, if present, is small.

NOTE Depending on any adjacent or attached particles, other peaks such as Ca or Cl may also be visible.

9.4.1.3 Crocidolite

Classify a fibre as crocidolite if:

(a) the Na, Si and Fe peaks are comparable in ratio with those of the reference crocidolite; and,

(b) any peak from Mg is small, and no peaks from Al or Mn are visible.

NOTE Depending on the adjacent or attached particles, other peaks such as Ca or Cl may also be visible.

NOTE If a large peak from Mg is present, the fibre may be magnesio-riebeckite. Bolivian crocidolite is the only known commercial source, although this variety of crocidolite may occur as contamination of other minerals.

9.4.1.4 Tremolite

Classify a fibre as tremolite if:

(a) the Mg, Si, Ca and Fe peaks are comparable in ratio to those of reference tremolite;

(b) no statistically significant peaks from Na or Al are present; and,

(c) the Mn peak, if present, is small.

NOTE Depending on any adjacent or attached particles, other peaks such as S or Cl may also be visible.

9.4.1.5 Actinolite

Classify a fibre as actinolite if:

(a) the Mg, Si and Fe peaks are comparable in ratio to those of the reference actinolite;

(b) no statistically significant peaks from Na or Al are present; and,

(c) the Mn peak, if present, is small.

NOTE Depending on any adjacent or attached particles, other peaks such as S or Cl may also be visible.

9.4.1.6 Anthophyllite

Classify a fibre as anthophyllite if:

(a) the fibre is straight and exhibits no evidence of a ribbon-like structure;

(b) the Mg and Si peaks are comparable in ratio to those of the reference anthophyllite. Anthophyllite from some sources may not exhibit a peak from Fe, although in commercial anthophyllite a peak from Fe will probably be observed;

(c) no statistically significant peaks from Na or Al are present; and,

(d) the Mn peak, if present, is small.

NOTE Depending on any adjacent or attached particles, other peaks such as Ca, S or Cl may also be visible.

9.4.1.7 Sodic-calcic amphibole asbestos (richterite/winchite)

Classify a fibre as sodic-calcic amphibole if:

(a) the spectrum is similar to that of actinolite or tremolite, but the Ca peak is substantially smaller and a Na peak is present. A K peak may also be present;

(b) no statistically significant peak from Al is present; and,

(c) the Mn peak, if present, is small.

NOTE Depending on any adjacent or attached particles, other peaks such as S or Cl may also be visible.

10 Analysis by TEM

10.1 Requirements

10.1.1 Transmission electron microscope

A TEM operating at an accelerating potential of 80-120 kV is required. The TEM shall have an illumination and condenser lens system capable of forming an electron probe smaller than 250 nm in diameter.

10.1.2 Energy dispersive X-ray analyzer

The TEM shall be equipped with an energy dispersive X-ray analyzer capable of achieving a resolution better than 170 eV (FWHM) on the MnKα peak. Since the performance of individual combinations of TEM and EDXA equipment is dependent on a number of geometrical factors, the required performance of the combination of the TEM and X-ray analyzer is specified in terms of the measured X-ray intensity obtained from a fibre of small diameter, using a known electron beam diameter. Solid-state X-ray detectors are least sensitive in the low energy region, and so measurement of sodium in crocidolite is the primary performance criterion. It is important that the combination of electron microscope and X-ray analyzer shall yield, under routine analytical conditions, a peak from sodium that allows discrimination between the spectra from crocidolite and amosite.

10.1.3 Vacuum coating unit

If carbon coated specimen grids are not available, a vacuum coating unit capable of producing a vacuum better than 0,013 Pa shall be used for vacuum deposition of carbon for preparation of carbon coated grids.

10.1.4 Calibration grids for EDXA

TEM specimen grids prepared from dispersions of chrysotile, amosite, crocidolite, tremolite, actinolite, anthophyllite, richterite/winchite and talc are required for calibration of the EDXA system. It is recommended that gold or nickel grids be used to facilitate detection of sodium.

10.1.5 Disposable tip micropipettes

Disposable tip micropipettes, capable of transferring a volume of approximately 3 µL to a carbon coated TEM specimen grid.

10.2 Calibration

For the purposes of this method, calibration consists of obtaining EDXA spectra from reference samples of chrysotile, amosite, crocidolite, tremolite, actinolite, anthophyllite and richterite/winchite. The chemical compositions of commercial chrysotile, amosite, crocidolite and anthophyllite do not vary substantially, and comparison of unknown EDXA spectra with those from the three reference asbestos samples constitutes sufficient identification for Part 1 of this analytical method. For most purposes, it is not necessary to discriminate between tremolite and actinolite, since the compositional boundary between them is a matter of convention. When it is necessary to discriminate between tremolite and actinolite, the NIST SRM 1867 tremolite and actinolite samples are particularly useful since these samples have compositions just below and just above the boundary defined by the International Mineralogical Association. In some applications the magnesium may be partially leached from chrysotile, leading to a chemical composition that approaches that of talc. In order to facilitate the discrimination between chrysotile and talc or anthophyllite, it is recommended that an EDXA spectrum also be obtained from a known sample of talc. Use this spectrum to define the upper limit of the magnesium concentration in talc. Examples of EDXA spectra obtained on the NIST SRM 1866 and SRM 1867 samples, the IOM reference asbestos samples, Bolivian crocidolite and richterite/winchite are illustrated in Annex F. For positive identification, reference EDXA spectra from asbestos standards similar to those shown in Annex F should be recorded using the specific combination of TEM and EDXA detector, since the geometries and detector efficiencies vary between different instruments.

10.3 Sample preparation

Remove representative fibres from the sample, and place them in an agate mortar and pestle. Add approximately 1 mL of ethanol, and grind the fibres with the pestle until they are well dispersed in the ethanol. Set up a laboratory stand and clamp, and use it to hold a pair of fine-point tweezers that are supporting a carbon coated TEM specimen grid, with the carbon side facing upwards. Using a disposable tip micropipette, drop a 3 µL volume of the ethanol dispersion on to the grid, and allow it to dry. Drying is faster if the grid is held under a heat lamp. When dry, the TEM grid is ready for examination.

10.4 Qualitative analysis by TEM

10.4.1 Chrysotile

The morphological structure of chrysotile as seen in the TEM is characteristic, and with experience, can be recognized readily. However, a few other minerals have similar appearance, and morphological observation by itself is inadequate for most samples.

Classify a fibre as chrysotile if:

(a) the Mg and Si peaks are clear, and comparable in Mg/Si ratio with that of reference chrysotile; and,

(c) any Fe, Mn and Al peaks are small.

NOTE Depending on the composition of adjacent or attached particles, other peaks such as Ca or Cl may also be visible.

NOTE Anthophyllite and talc both yield EDXA spectra that conform to this specification, but the Mg/Si peak height ratio for these minerals is lower than that for chrysotile. In order to avoid erroneous classification of talc or anthophyllite as chrysotile, it is important to take account of the Mg/Si peak height ratio, and to calibrate the EDXA detector using known samples of chrysotile and talc.

10.4.2 Amosite

Classify a fibre as amosite if:

(a) the Mg, Si and Fe peaks are comparable in ratio to those of the reference amosite; and,

(b) no statistically significant peaks from Na or Al are present; and,

(d) the Mn peak, if present, is small.

NOTE Depending on any adjacent or attached particles, other peaks such as Ca or Cl may also be visible.

10.4.3 Crocidolite

Classify a fibre as crocidolite if:

(a) the Na, Si and Fe peaks are comparable in ratio with those of the reference crocidolite;

(b) no statistically significant peak from Al is present; and,

(c) any peak from Mg is small, and no Mn peak is visible.

NOTE Depending on the adjacent or attached particles, other peaks such as Ca or Cl may also be visible.

NOTE If a large peak from Mg, is present, the fibre may be magnesio-riebeckite. Bolivian crocidolite is the only known commercial source, although this variety of crocidolite may occur as contamination of other minerals.

10.4.4 Tremolite

Classify a fibre as tremolite if:

(a) the Mg, Ca and Fe peaks are comparable in ratio with those of the reference tremolite;

(b) no statistically significant peak from Al is present; and,

(c) any peak from either Na or K is small.

NOTE Depending on the adjacent or attached particles, other peaks may occur. It is important to obtain the EDXA spectra from clean areas of the fibre, since distortion of peak heights by contributions from attached particles may compromise the identification.

10.4.5 Actinolite

Classify a fibre as actinolite if:

(a) the Mg, Si and Fe peaks are comparable in ratio to those of the reference actinolite;

(b) no statistically significant peaks from Na or Al are present; and,

(c) the Mn peak, if present, is small.

NOTE Depending on any adjacent or attached particles, other peaks such as S or Cl may also be visible.

10.4.6 Anthophyllite

Classify a fibre as anthophyllite if:

(a) the fibre is straight and exhibits no evidence of a ribbon-like structure;

(b) the Mg and Si peaks are comparable in ratio to those of the reference anthophyllite. Anthophyllite from some sources may not exhibit a peak from Fe, although in commercial anthophyllite a peak from Fe will probably be observed;

(c) no statistically significant peaks from Na or Al are present; and,

(d) the Mn peak, if present, is small.

NOTE Depending on any adjacent or attached particles, other peaks such as Ca, S or Cl may also be visible.

10.4.7 Sodic-calcic amphibole asbestos (richterite/winchite)

Classify a fibre as sodic-calcic amphibole if:

(a) the spectrum is similar to that of actinolite or tremolite, but the Ca peak is substantially smaller and a Na peak is present. A K peak may also be present;

(b) no statistically significant peak from Al is present; and,

(c) the Mn peak, if present, is small.

NOTE Depending on any adjacent or attached particles, other peaks such as S or Cl may also be visible.

11 Test report

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

(a) reference to this International Standard and the applicable part of the Standard;

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

(c) the date of the analysis;

(d) identity of the analyst;

(e) all applicable specimen preparation details;

(f) any procedure used not specified in this International Standard or regarded as an optional procedure;

(g) the variety or varieties of asbestos detected;

(h) the analytical method used to identify the asbestos.

The following items (i) to (k) shall be recorded in the laboratory data, but the extent to which they are included as part of the test report is optional:

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

(j) the estimated concentration(s) of the asbestos varieties detected in ranges as follows:

None detected

0,1% to 5%

5% to 50%

50% to 100%;

(k) 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 H.

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Annex A
(normative)

Types of commercial asbestos-containing material

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 concentrations, for example paints for protective coatings, adhesives, plastic sheets and tiles.

Table A1 gives the most important asbestos-containing materials with examples of their applications and the typical asbestos concentrations. In exceptional cases, asbestos concentrations deviating from those quoted may have been used. Table A.2 lists products that can contain asbestos.

Table A1. Asbestos-containing materials; examples of use and typical asbestos content

Product	Examples of application	Typical asbestos concentration
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
Asbestos cement corrugated sheets	– Roof claddings – Perimeter insulation – Sidings in the industrial sector	10 - 12% chrysotile, sometimes also with some manufacturers < 5% crocidolite
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
Asbestos cement mouldings	– Standard ashtrays – Flower boxes – Garden articles – Sculptures	10 – 12% chrysotile

Product	Examples of application	Typical asbestos concentration
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
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
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.

Product	Examples of application	Typical asbestos concentration
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
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
Asbestos foams	– Infilling (sealing) of movement joints – Seals at fire shutters and fire barriers	approximately 50 % chrysotile
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

Product	Examples of application	Typical asbestos concentration
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
Asbestos-containing floorings	– Reinforcement in flexible sheets – Rot-resistant support layer as underlay of cushion vinyl flooring materials–	Chrysotile 10 - 20% Chrysotile 80 - 100%
Asphalt or vinyl asbestos floor tiles	– Reinforcement	Asphalt tiles up to 35% chrysotile, vinyl tiles up to 20% chrysotile.
Rubberized asbestos seals	– Gaskets for pipe flanges	Chrysotile 50 - 90%
Asbestos-containing friction products	Brake linings Brake bands Clutch linings	Chrysotile 10 - 70%

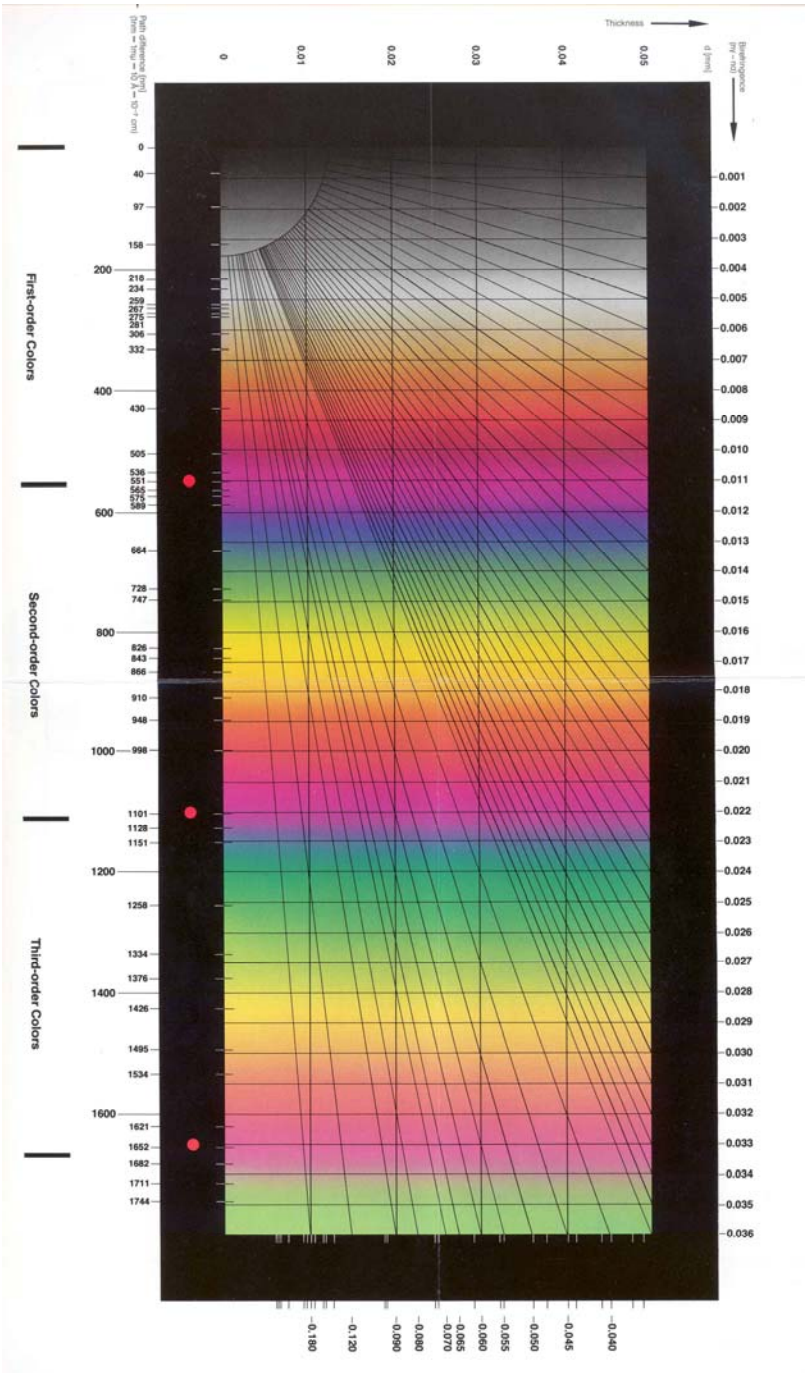
Table A2. Materials that can contain asbestos; examples of application and typical asbestos concentration

Product	Examples of application	Approximate asbestos concentration
Filter media	– Air filters – Liquid filters – Sterile and aseptic filters – Clarifying sheets – Diaphragms for chloralkali electrolysis process	95% chrysotile, rarely amosite
Talc (asbestos content dependent on deposit)	– Release agents for electric cables, rubber products – Release agents in the confectionery industry – Tailor's chalk – Paper manufacture – Medicine, cosmetics	chrysotile and/or actinolite/tremolite
Vermiculite	– Attic and wall cavity insulation – Fireproofing – Horticultural products	Depends on the source of the vermiculite. Vermiculite from Montana, U.S.A. may contain up to 6% of a mixture of amphibole asbestos types
Industrial minerals including wollastonite, sepiolite, mica, dolomite	Ceramics manufacture Plastics fillers Surfacing materials and joint compounds Ceiling tiles	Depends on the source of the mineral. May contain up to 7% tremolite/actinolite.
Surfacings	– Road construction	1%

Product	Examples of application	Approximate asbestos concentration
Chemical products for construction and other products*)	– Bitumen, roofing and sealing sheets	3%
	– Sealing putties	2%
	– Glazing putties	4%
	– Bituminous coatings	30%
	– Fillers and sealers	25%
	– Jointing compounds	5%
	– Paints	9%
	– Glues	4%
	– Fire retardants	10%
	– Sub-floor protection	4%

Annex B
(normative)

Interference colour chart



Annex C
(normative)

Dispersion staining charts

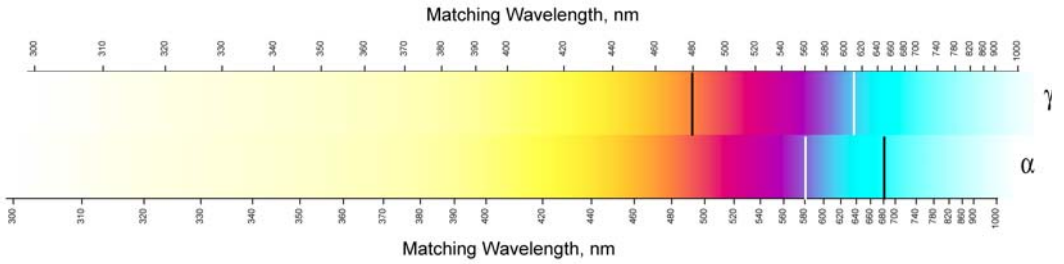


Figure C.1 — Dispersion staining chart for chrysotile in 1.550 RI liquid.

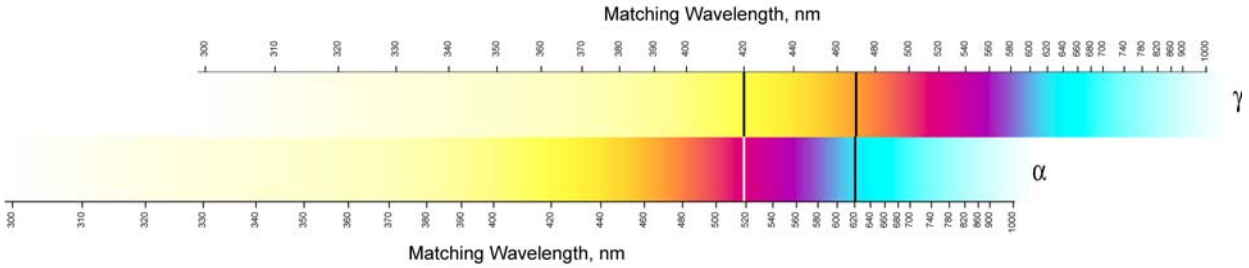


Figure C.2 — Dispersion staining chart for amosite in 1.680 RI liquid.

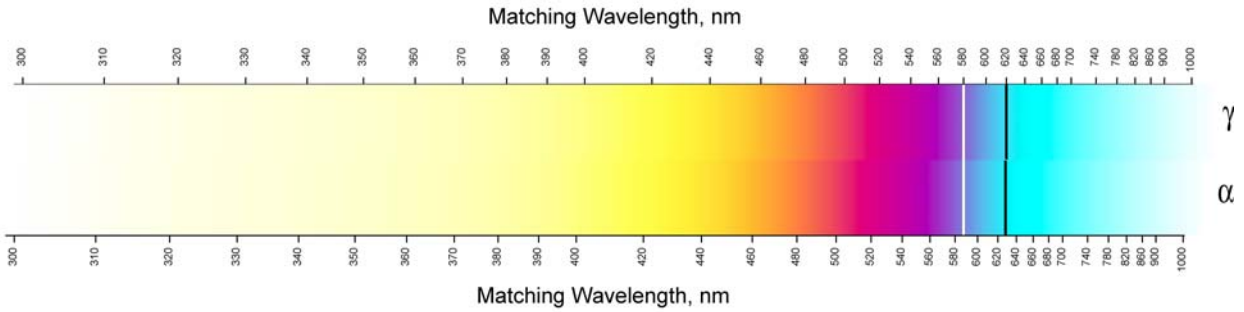


Figure C.3 — Dispersion staining chart for crocidolite in 1.700 RI liquid

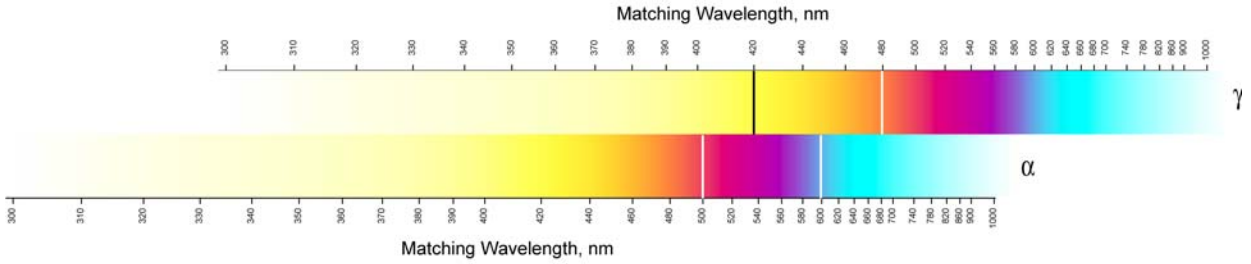


Figure C.4 — Dispersion staining chart for tremolite in 1.605 RI liquid.

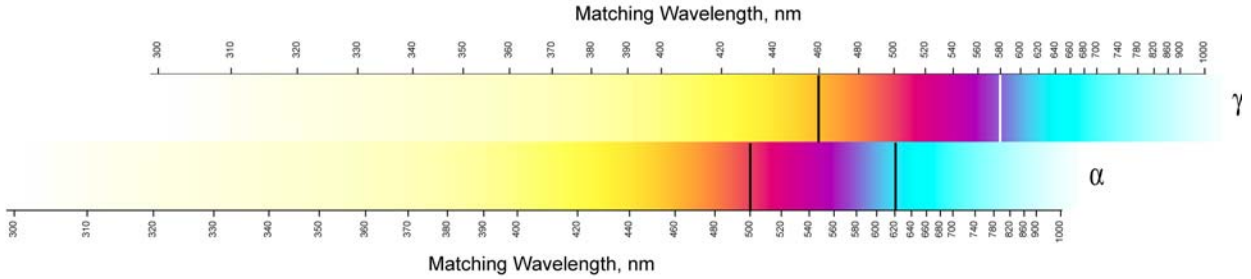


Figure C.5 — Dispersion staining chart for actinolite in 1.630 RI liquid.

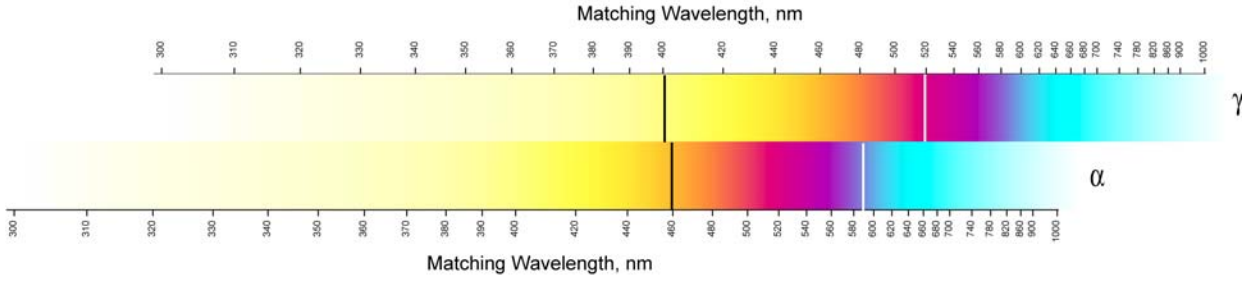


Figure C.6 — Dispersion staining chart for anthophyllite in 1.605 RI liquid.

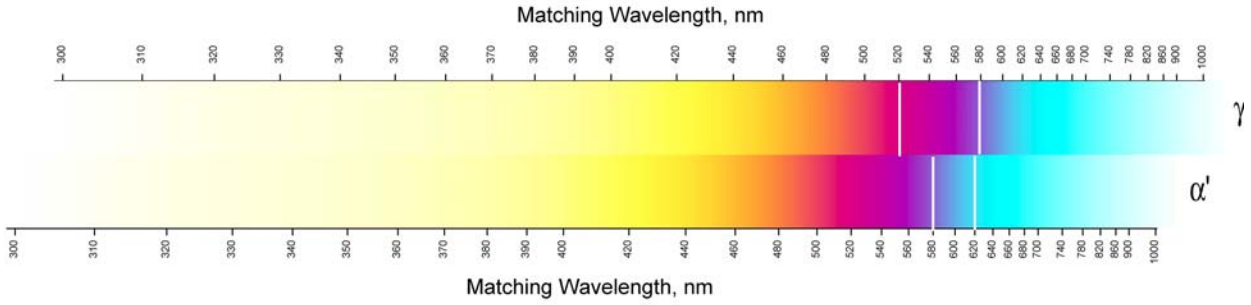


Figure C.7 — Dispersion staining chart for richterite/winchite asbestos in 1.630 RI liquid.

Annex D
(normative)

Identification of asbestos by PLM and dispersion staining in commercial asbestos-containing materials

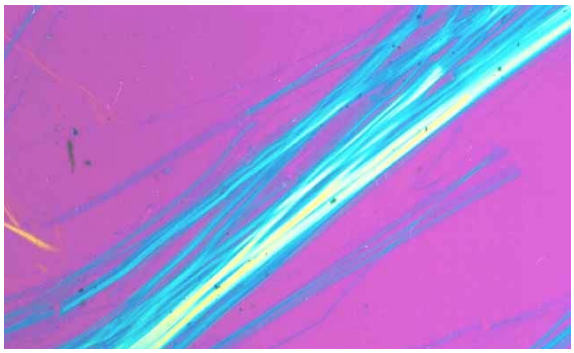


Figure D.1 — PLM micrograph of NIST SRM 1866 chrysotile in 1.550 RI liquid, crossed polars with 550 nm retardation plate

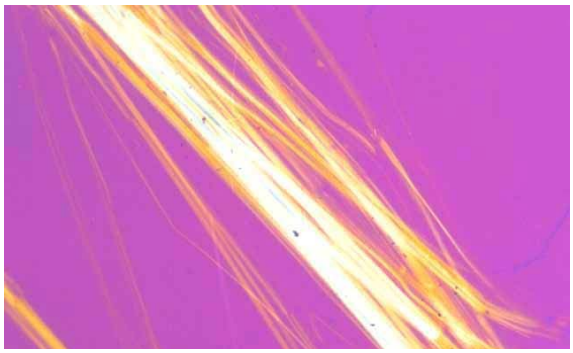


Figure D.2 — PLM micrograph of NIST SRM 1866 chrysotile in 1.550 RI liquid, crossed polars with 550 nm retardation plate

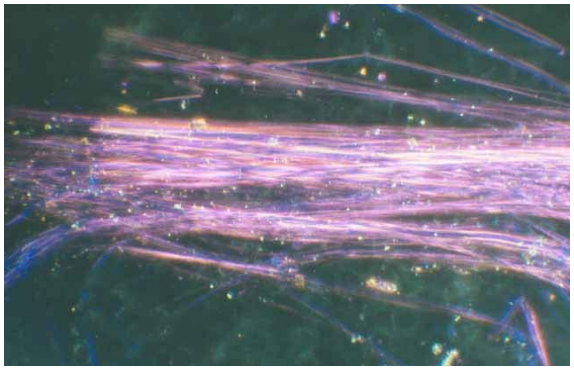


Figure D.3 — NIST SRM 1866 chrysotile in 1.550 RI liquid viewed in dispersion staining, fibre length parallel to polarizer vibration direction

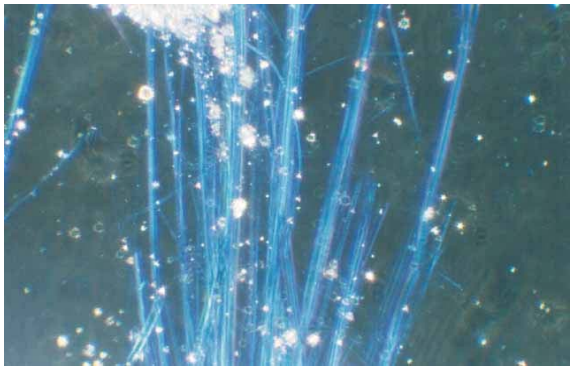


Figure D.4 — NIST SRM 1866 chrysotile in 1.550 RI liquid viewed in dispersion staining, fibre length normal to polarizer vibration direction

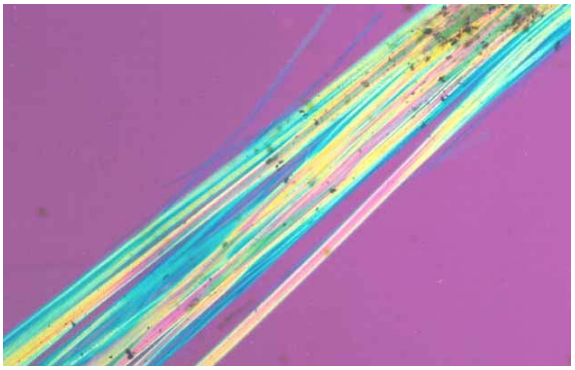


Figure D.5 — PLM micrograph of NIST SRM 1866 amosite in 1.680 RI liquid, crossed polars with 550 nm retardation plate.

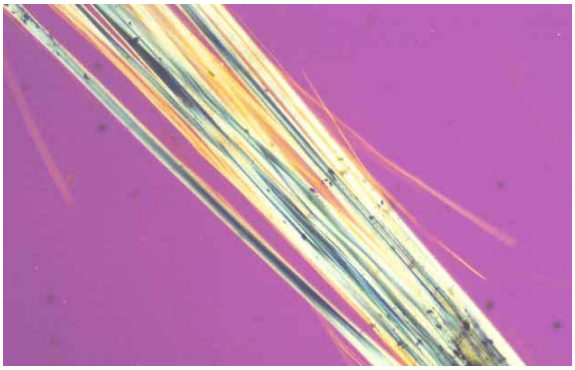


Figure D.6 — PLM micrograph of NIST SRM 1866 amosite in1.680 RI liquid, crossed polars with 550 nm retardation plate

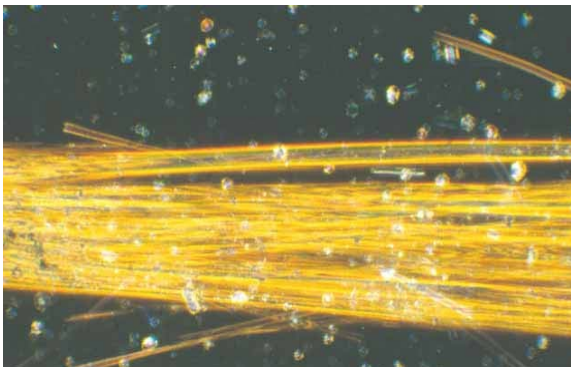


Figure D.7 — NIST SRM 1866 amosite in 1.680 RI liquid viewed in dispersion staining, fibre length parallel to polarizer vibration direction.

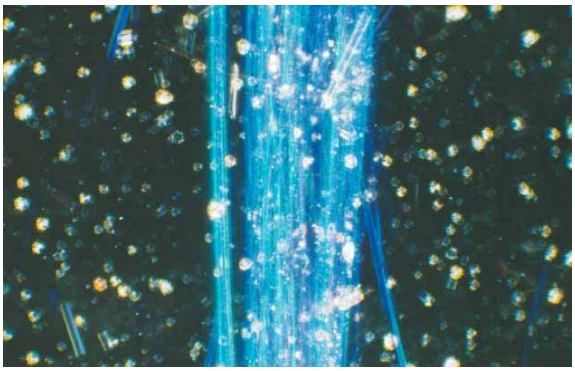


Figure D.8 — NIST SRM 1866 amosite in 1.680 RI liquid viewed in dispersion staining, fibre length normal to polarizer vibration direction.

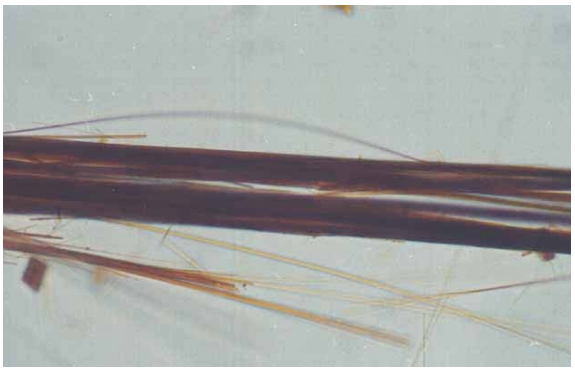


Figure D.9 — Heated amosite in 1.680 RI liquid viewed in plane polarized light, fibre length parallel to polarizer vibration direction.

Figure D.10 — Heated amosite in 1.680 RI liquid viewed in plane polarized light, fibre length parallel to polarizer vibration direction.

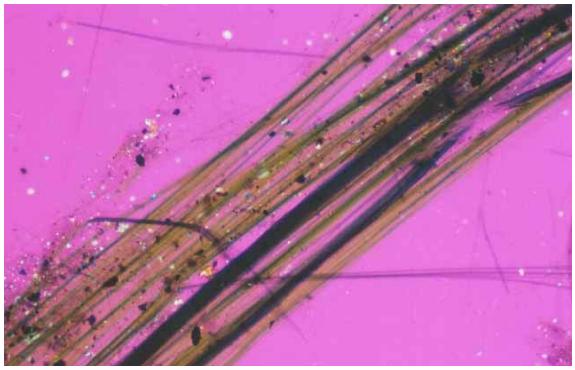


Figure D.11 — PLM micrograph of NIST SRM 1866 crocidolite in 1.700 RI liquid, crossed polars with 550 nm retardation plate

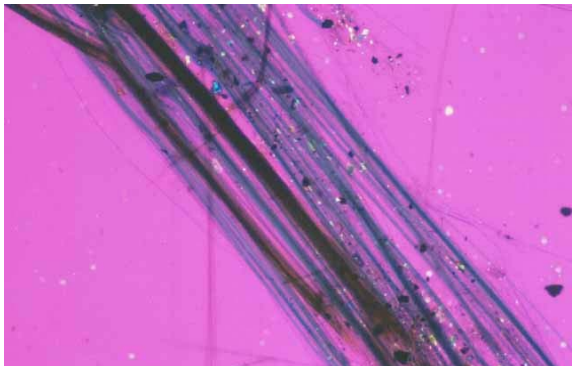


Figure D.12 — PLM micrograph of NIST SRM 1866 crocidolite in 1.700 RI liquid, crossed polars with 550 nm retardation plate

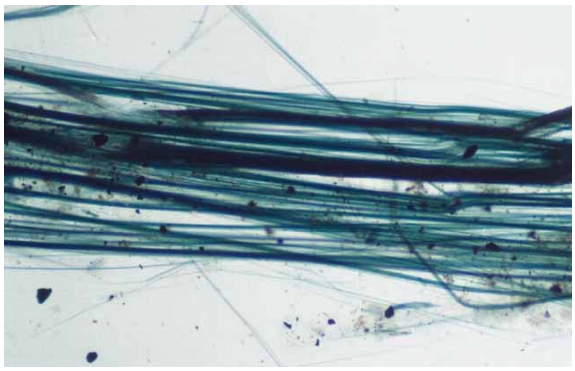


Figure D.13 — NIST SRM 1866 crocidolite in 1.700 RI liquid in plane polarized light. Fibres parallel to polarizer vibration direction.

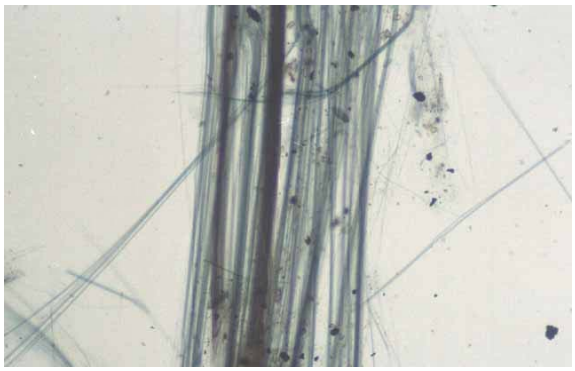


Figure D.14 — NIST SRM 1866 crocidolite in 1.700 RI liquid in plane polarized light. Fibres normal to polarizer vibration direction.

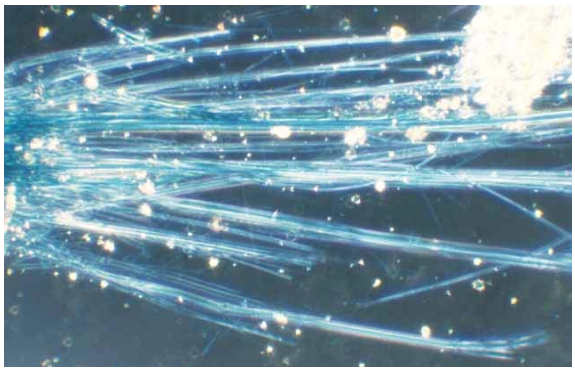


Figure D.15 — NIST SRM 1866 crocidolite in 1.700 RI liquid. Dispersion staining, fibre lengths parallel to polarizer vibration direction.

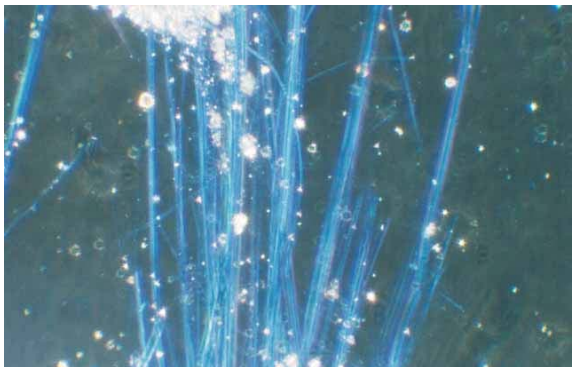


Figure D.16 — NIST SRM 1866 crocidolite in 1.700 RI liquid. Dispersion staining, fibre lengths normal to polarizer vibration direction.

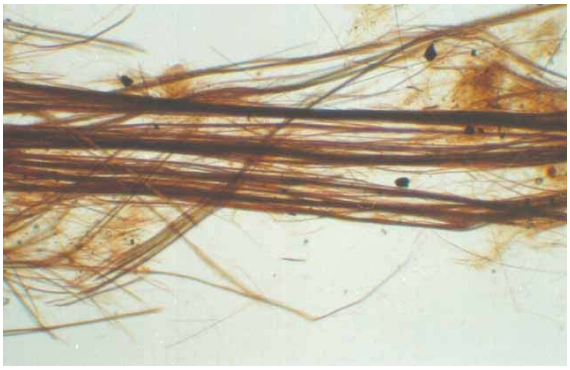


Figure D.17 — Heated crocidolite viewed in plane polarized light. Fibre length parallel to polarizer vibration direction.

Figure D.18 — Heated crocidolite viewed in plane polarized light. Fibre length normal to polarizer vibration direction.

Figure D.19 — PLM micrograph of NIST SRM 1867 tremolite in 1.605 RI liquid, crossed polars with 550 nm retardation plate.

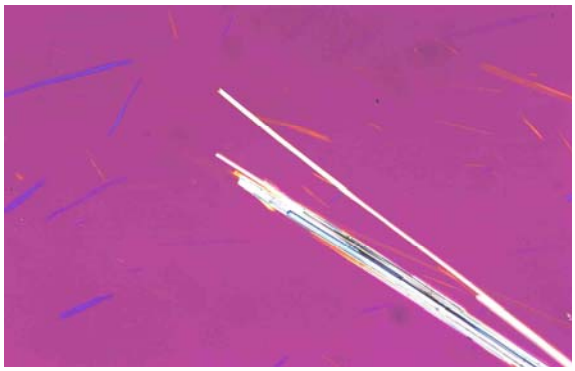


Figure D.20 — PLM micrograph of NIST SRM 1867 tremolite in 1.605 RI liquid, crossed polars with 550 nm retardation plate.

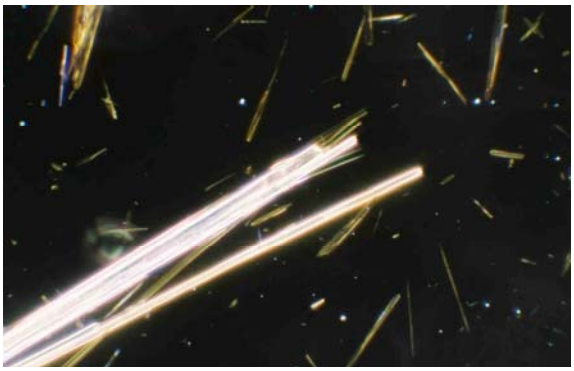


Figure D.21 — SRM 1867 1867 tremolite in 1.605 RI liquid viewed in dispersion staining, fibres at extinction position.

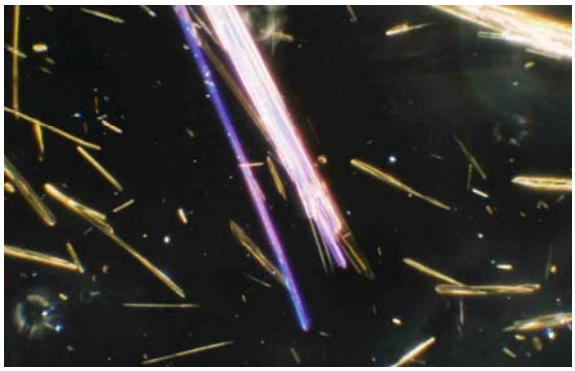


Figure D.22 — SRM 1867 tremolite in 1.605 RI liquid viewed in dispersion staining, fibres at extinction position.

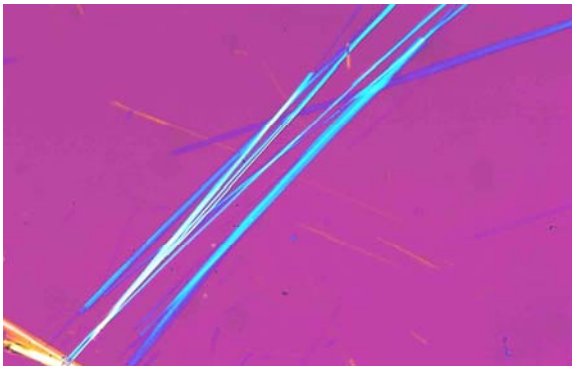


Figure D.23 — PLM micrograph of NIST SRM 1867 tremolite in 1.625 RI liquid, crossed polars with 550 nm retardation plate.

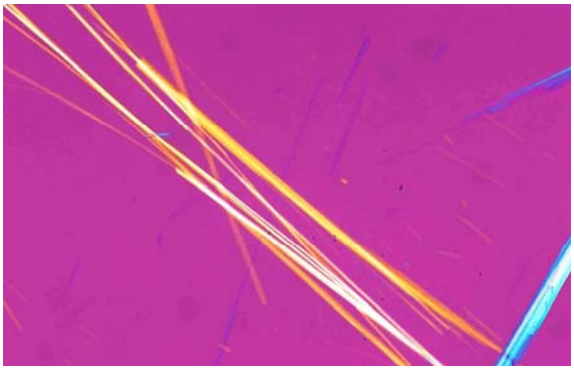


Figure D.24 — PLM micrograph of NIST SRM 1867 tremolite in 1.625 RI liquid, crossed polars with 550 nm retardation plate.

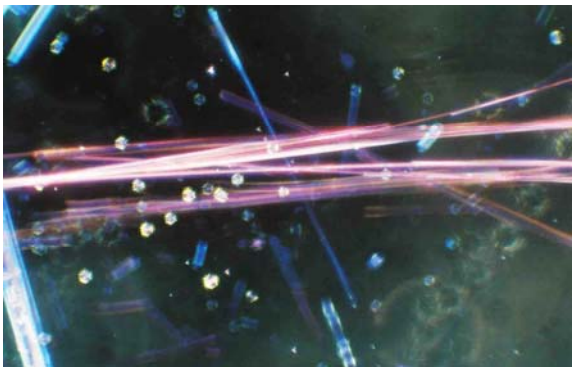


Figure D.25 — NIST SRM 1867 tremolite in 1.625 RI liquid viewed in dispersion staining. Fibres at extinction position.

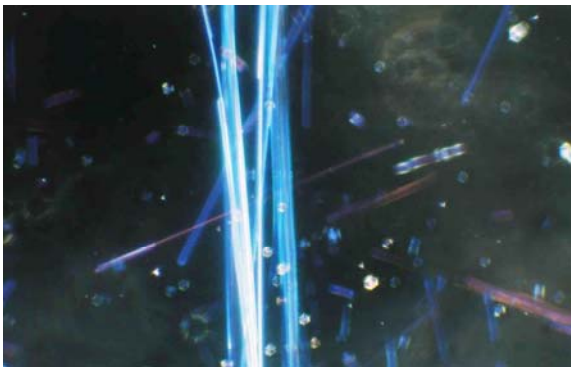


Figure D.26 — NIST SRM 1867 tremolite in 1.625 RI liquid viewed in dispersion staining. Fibres at extinction position.

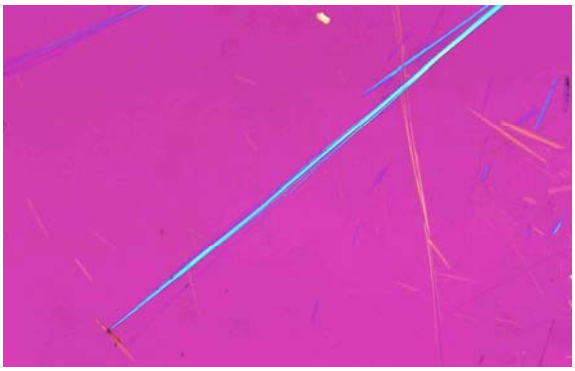


Figure D.27 — PLM micrograph of NIST SRM 1867 actinolite in 1.630 RI liquid, crossed polars with 550 nm retardation plate.

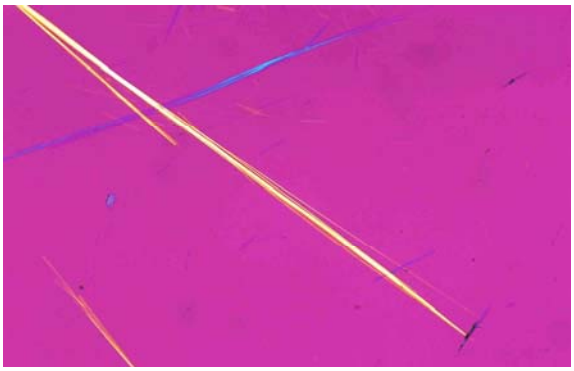


Figure D.28 — PLM micrograph of NIST SRM 1867 actinolite in 1.630 RI liquid, crossed polars with 550 nm retardation plate.

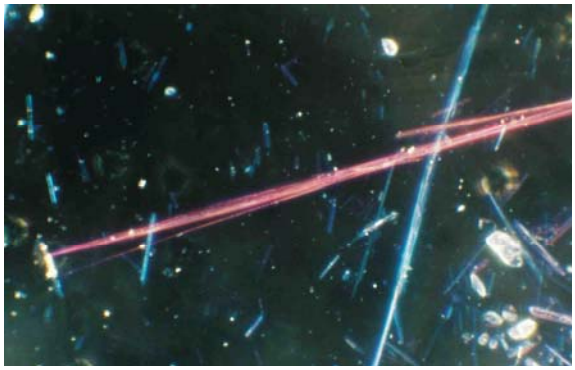


Figure D.29 — NIST SRM 1867 actinolite in 1.630 RI liquid viewed in dispersion staining. Purple fibre at extinction position.

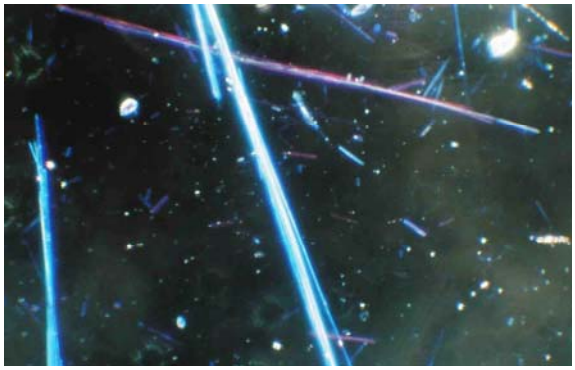


Figure D.30 — NIST SRM 1867 actinolite in 1.630 RI liquid viewed in dispersion staining. Light blue fibre at extinction position.

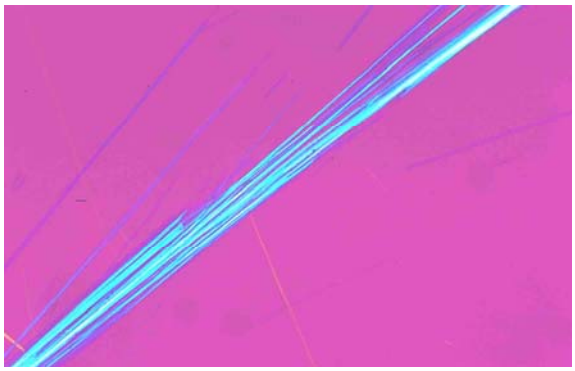


Figure D.31 — PLM micrograph of NIST SRM 1867 anthophyllite in 1.630 RI liquid, crossed polars with 550 nm retardation plate.

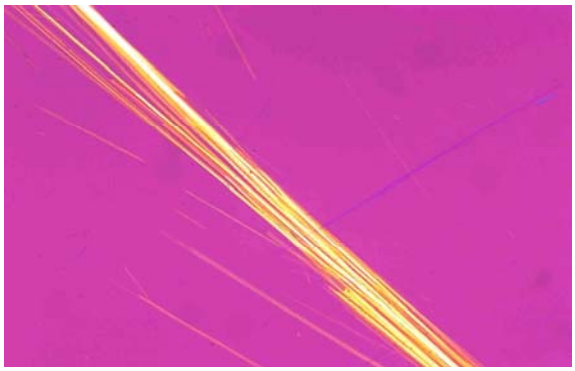


Figure D.32 — PLM micrograph of NIST SRM 1867 anthophyllite in 1.630 RI liquid, crossed polars with 550 nm retardation plate.

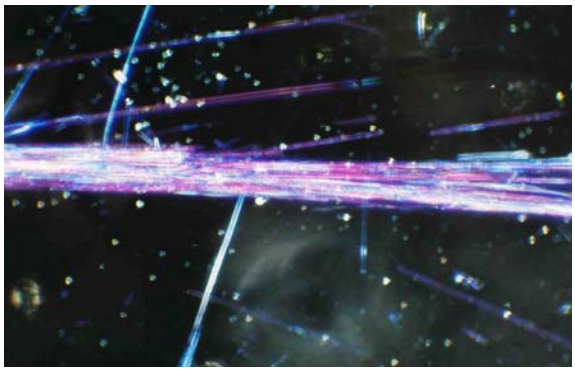


Figure D.33 — NIST SRM 1867 anthophyllite in 1.630 RI liquid viewed in dispersion staining, fibre lengths parallel to polarizer vibration direction.

Figure D.34 — NIST SRM 1867 anthophyllite in 1.630 RI liquid viewed in dispersion staining, fibre lengths normal to polarizer vibration direction.

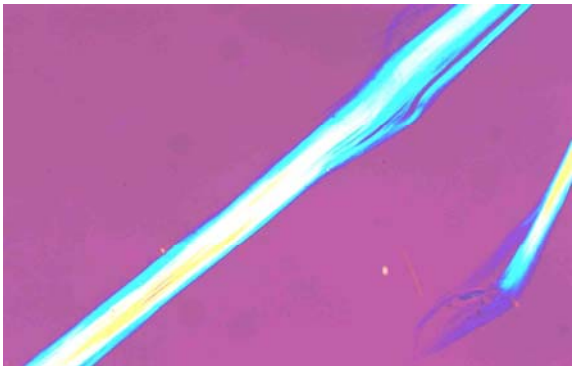


Figure D.35 — PLM micrograph of IOM tremolite in 1.605 RI liquid, crossed polars with 550 nm retardation plate.

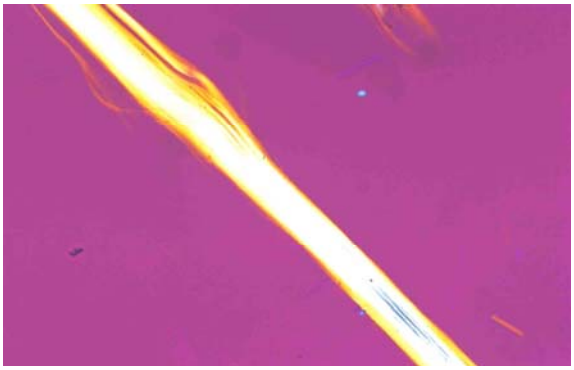


Figure D.36 — PLM micrograph of IOM tremolite in 1.605 RI liquid, crossed polars with 550 nm retardation plate.

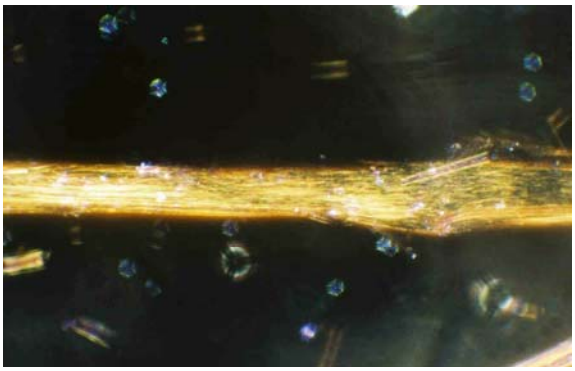


Figure D.37 — IOM tremolite in 1.605 RI liquid viewed in dispersion staining, fibre lengths parallel to polarizer vibration direction.

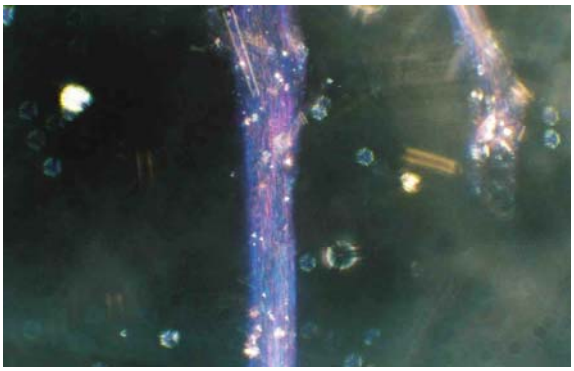


Figure D.38 — IOM tremolite in 1.605 RI liquid viewed in dispersion staining, fibre lengths normal to polarizer vibration direction.

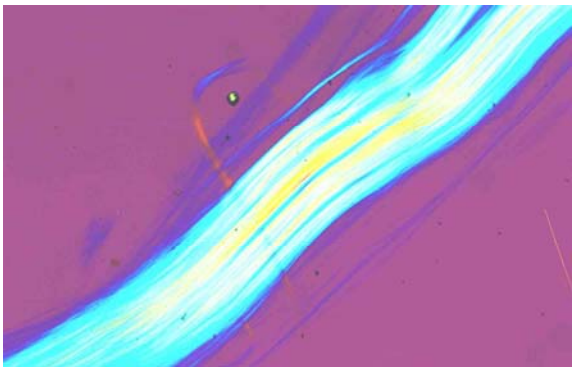


Figure D.39 — PLM micrograph of IOM actinolite in 1.640 RI liquid, crossed polars with 550 nm retardation plate.

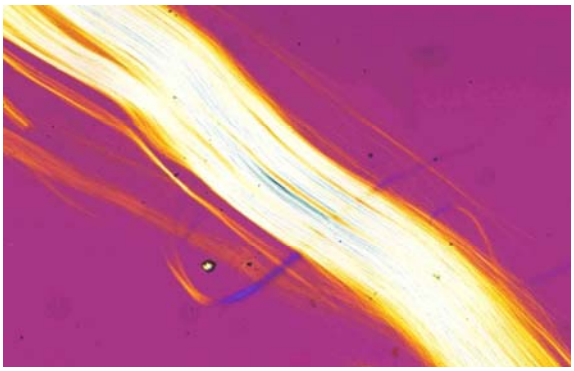


Figure D.40 — PLM micrograph of IOM actinolite in 1.640 RI liquid, crossed polars with 550 nm retardation plate.

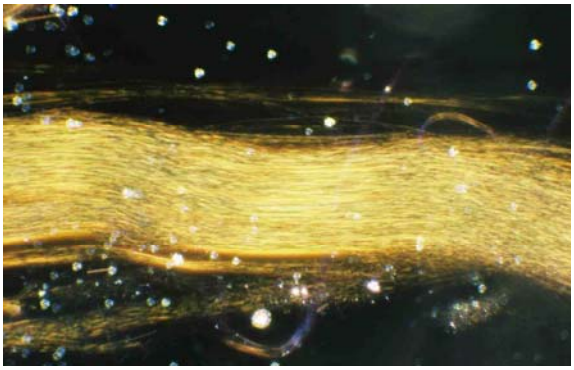


Figure D.41 — IOM actinolite in 1.640 RI liquid viewed in dispersion staining, fibre lengths parallel to polarizer vibration direction.

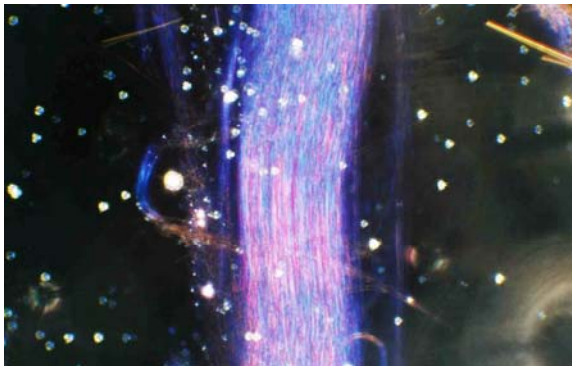


Figure D.42 — IOM actinolite in 1.640 RI liquid viewed in dispersion staining, fibre lengths normal to polarizer vibration direction.

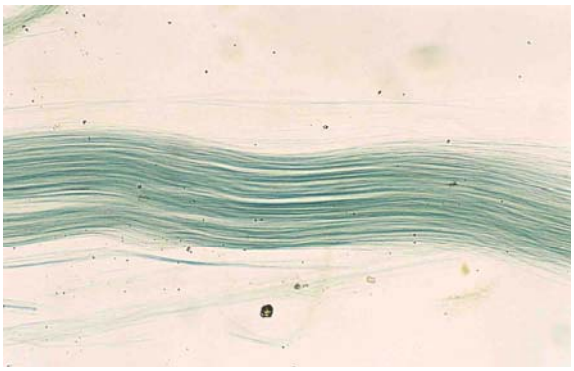


Figure D.43 — IOM actinolite in 1.640 RI liquid in plane polarized light, fibres parallel to polarizer vibration direction.

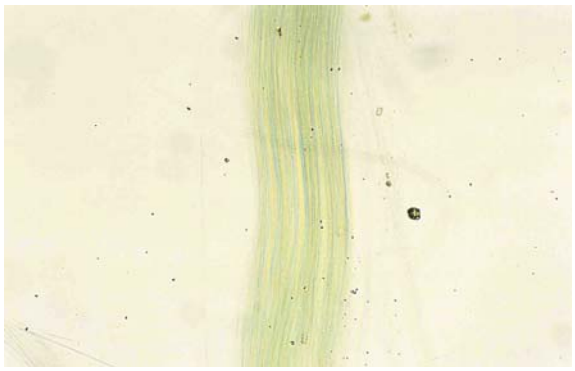


Figure D.44 — IOM actinolite in 1.640 RI liquid in plane polarized light, fibres normal to polarizer vibration direction.

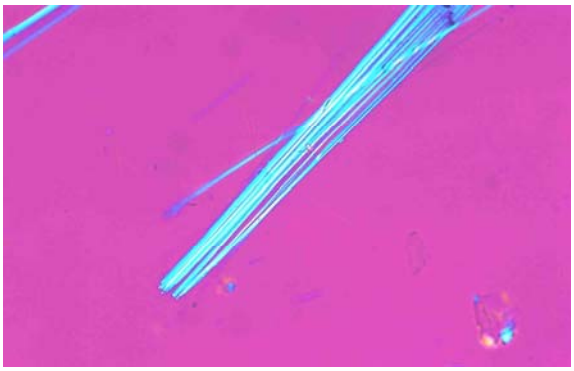


Figure D.45 — PLM micrograph of IOM anthophyllite in 1.605 RI liquid, crossed polars with 550 nm retardation plate.

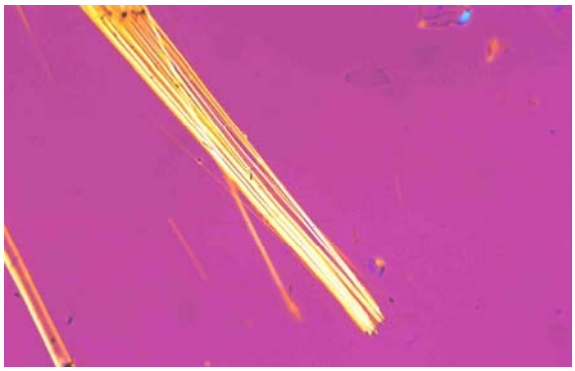


Figure D.46 — PLM micrograph of IOM anthophyllite in 1.605 RI liquid, crossed polars with 550 nm retardation plate.

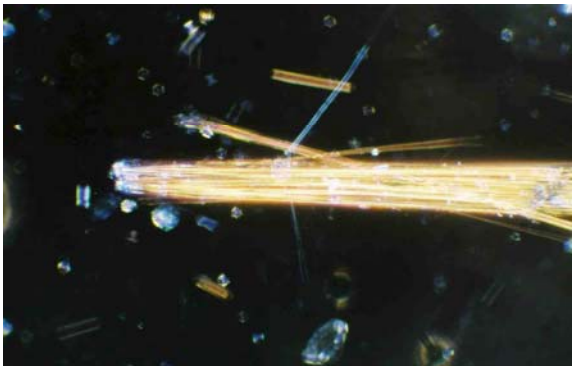


Figure D.47 — IOM anthophyllite in 1.605 RI liquid viewed in dispersion staining, fibre lengths parallel to polarizer vibration direction.

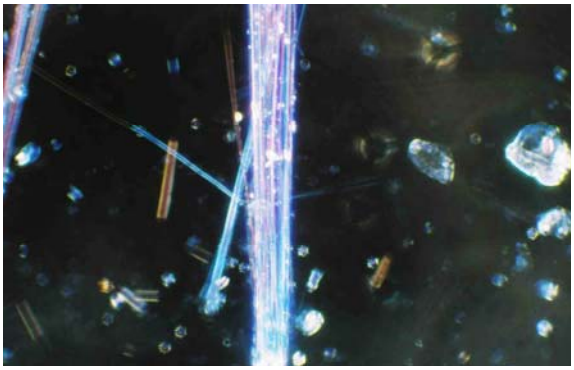


Figure D.48 — IOM anthophyllite in 1.605 RI liquid viewed in dispersion staining, fibre lengths normal to polarizer vibration direction.

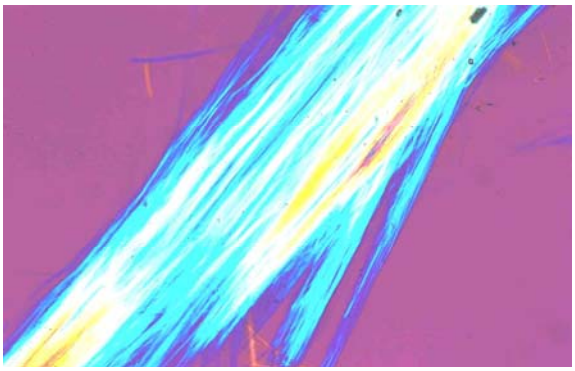


Figure D.49 — PLM micrograph of richterite/winchite asbestos in 1.630 RI liquid, crossed polars with 550 nm retardation plate.

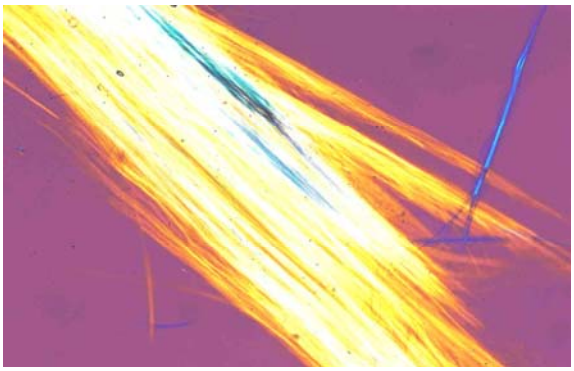


Figure D.50 — PLM micrograph of richterite/winchite asbestos in 1.630 RI liquid, crossed polars with 550 nm retardation plate.

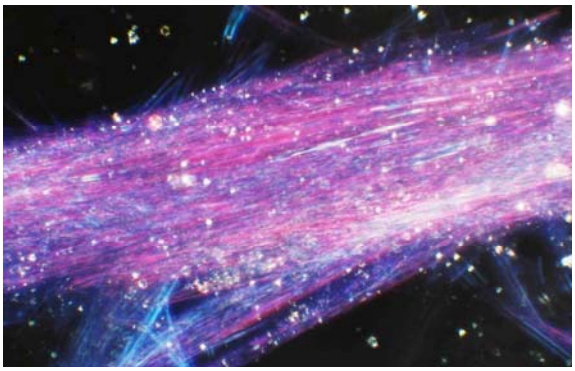


Figure D.51 — Richterite/winchite asbestos in 1.630 RI liquid viewed in dispersion staining, fibres at extinction position.

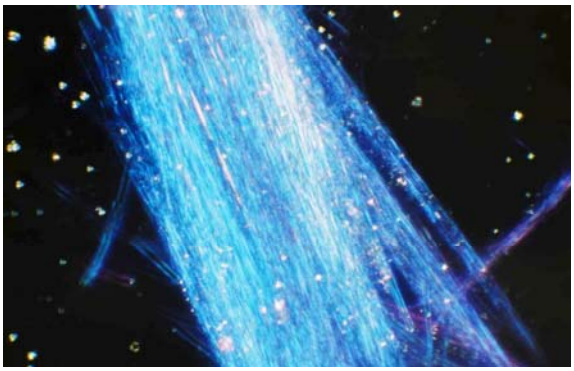


Figure D.52 — Richterite/winchite asbestos in 1.630 RI liquid viewed in dispersion staining, fibres at extinction position.

Annex E
(normative)

Identification of asbestos by SEM in commercial asbestos-containing materials

The following are examples of EDXA spectra collected on an SEM operating at 15 kV and using a silicon solid-state detector with a beryllium window. The SEM specimens were prepared by mounting representative fibre bundles from NIST SRM 1866, NIST SRM 1867 and the IOM reference asbestos varieties on adhesive tape on SEM specimen stubs. All specimens were carbon coated in a vacuum evaporator.

Prior to use of this Standard, obtain calibration spectra from the reference standards, using the actual accelerating voltage and the specific x-ray detector.

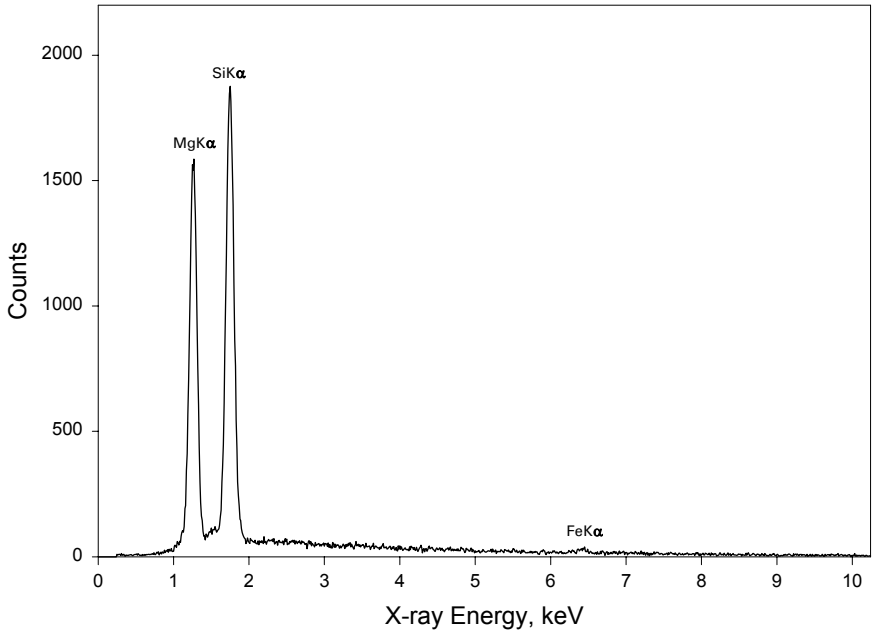


Figure E.1 — Energy dispersive x-ray spectrum obtained from NIST SRM 1866 chrysotile.

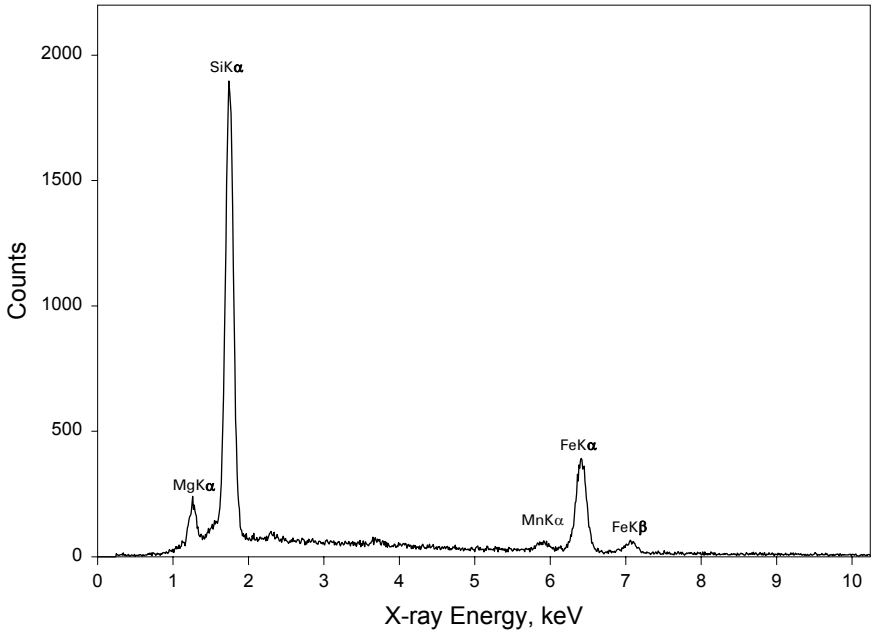


Figure E.2 — Energy dispersive x-ray spectrum obtained from NIST SRM 1866 amosite.

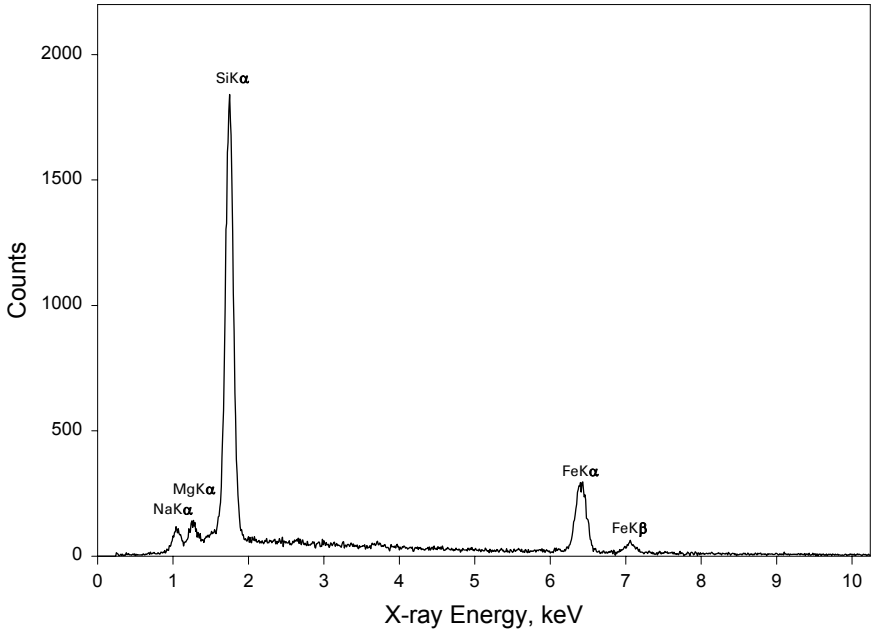


Figure E.3 — Energy dispersive x-ray spectrum obtained from NIST SRM 1866 crocidolite.

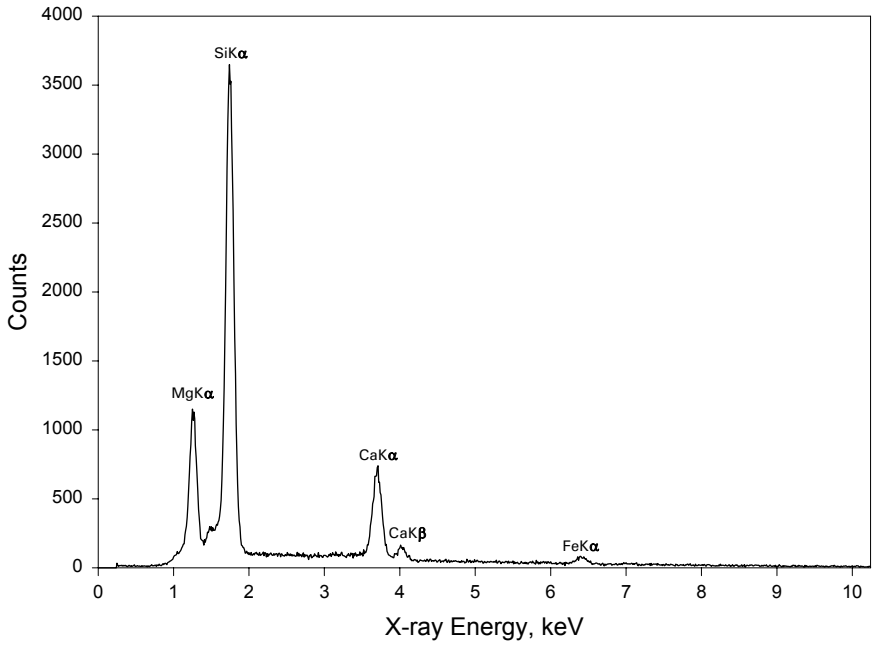


Figure E.4 — Energy dispersive x-ray spectrum obtained from NIST SRM 1867 tremolite.

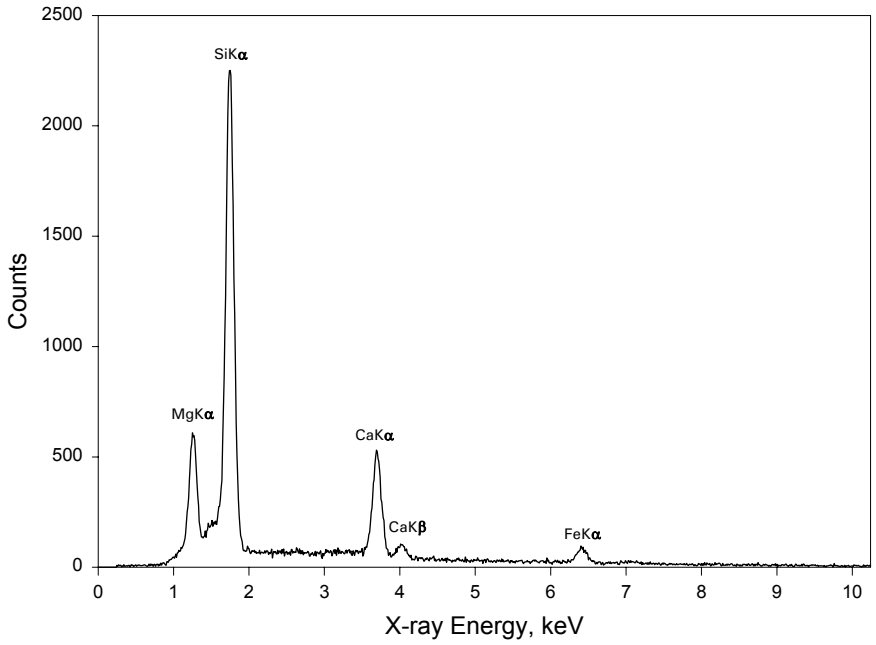


Figure E.5 — Energy dispersive x-ray spectrum obtained from NIST SRM 1867 actinolite.

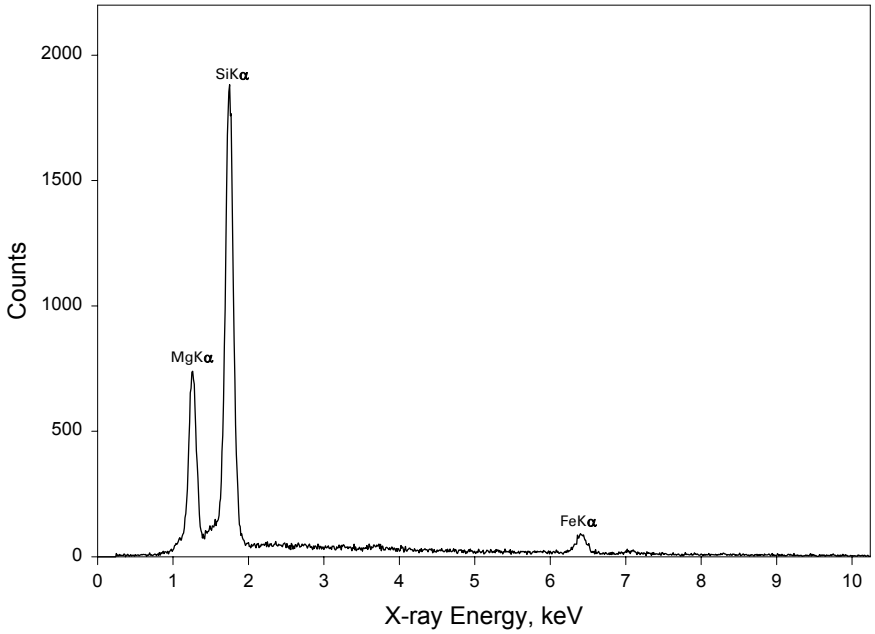


Figure E.6 — Energy dispersive x-ray spectrum obtained from NIST SRM 1867 anthophyllite.

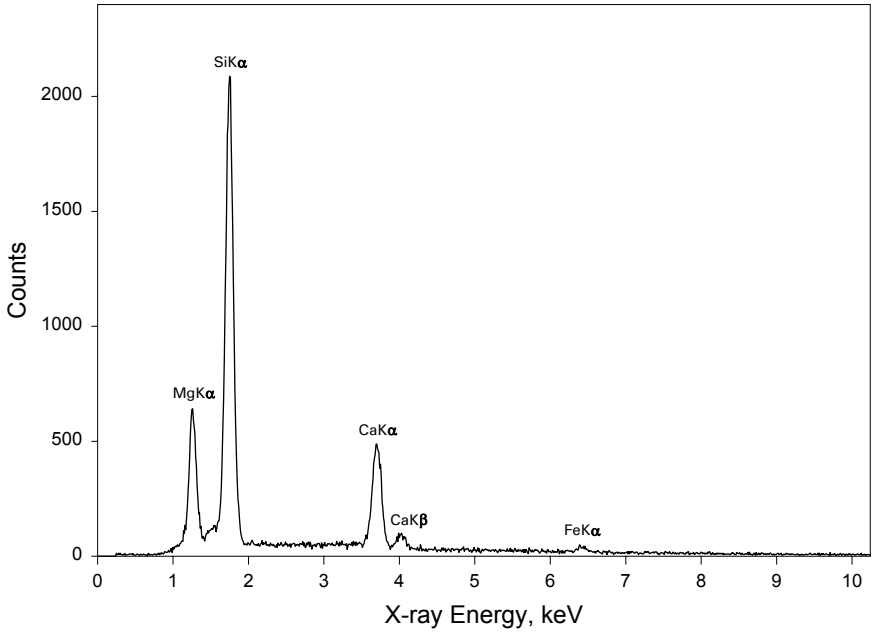


Figure E.7 — Energy dispersive x-ray spectrum obtained from IOM tremolite.

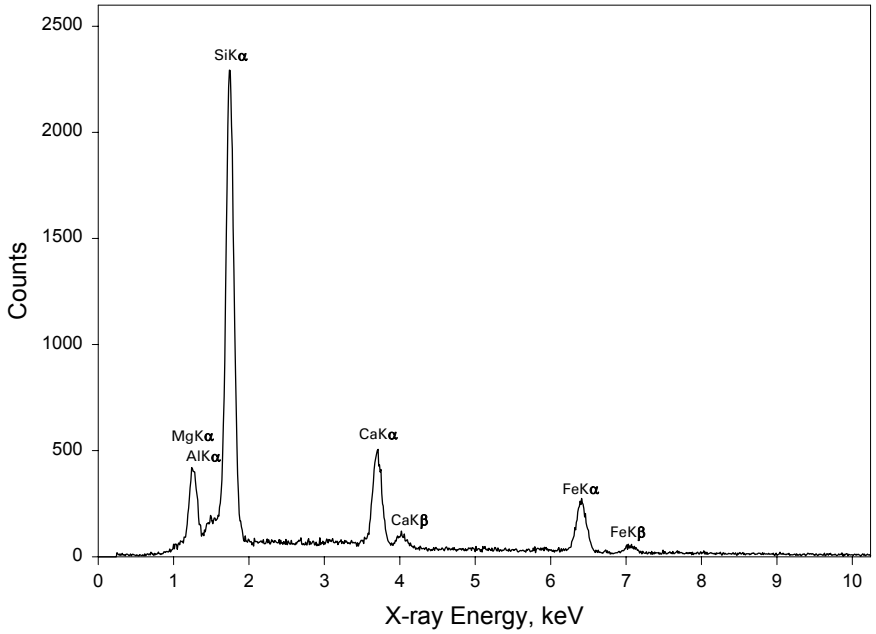


Figure E.8 — Energy dispersive spectrum obtained from IOM actinolite.

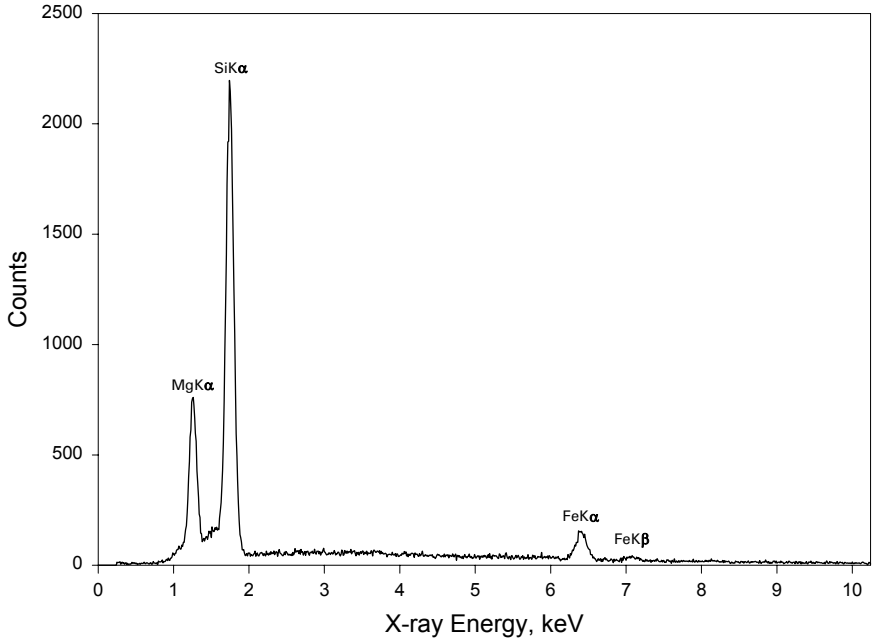


Figure E.9 — Energy dispersive x-ray spectrum obtained from IOM anthophyllite.

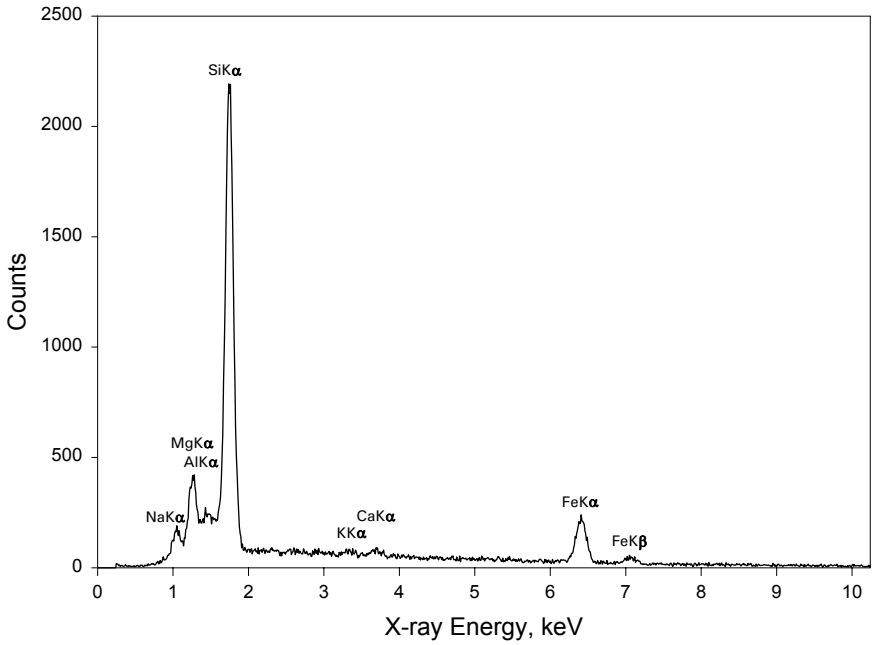


Figure E.10 — Energy dispersive x-ray spectrum obtained from Bolivian crocidolite.

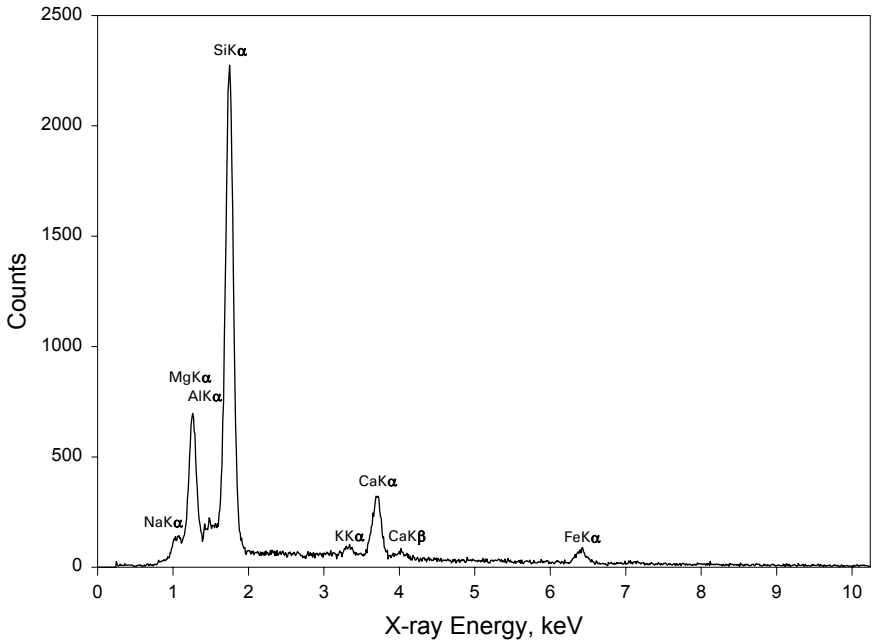


Figure E.11 — Energy dispersive x-ray spectrum obtained from richterite/winchite.

Annex F
(normative)

Identification of asbestos by TEM in commercial asbestos-containing materials

The following are examples of EDXA spectra collected on a TEM operating at 80 kV and using a silicon solid state detector with a beryllium window. The TEM specimens were prepared by the micropipette method from NIST SRM 1866, NIST SRM 1867 and IOM reference asbestos varieties. All specimens were prepared using gold grids in order to avoid interference in detection of the sodium kα peak by the copper Lα peak which would partially overlap the sodium peak if copper specimen grids were used.

Prior to use of this Standard, obtain calibration spectra from the reference standards, using the actual accelerating voltage and the specific x-ray detector.

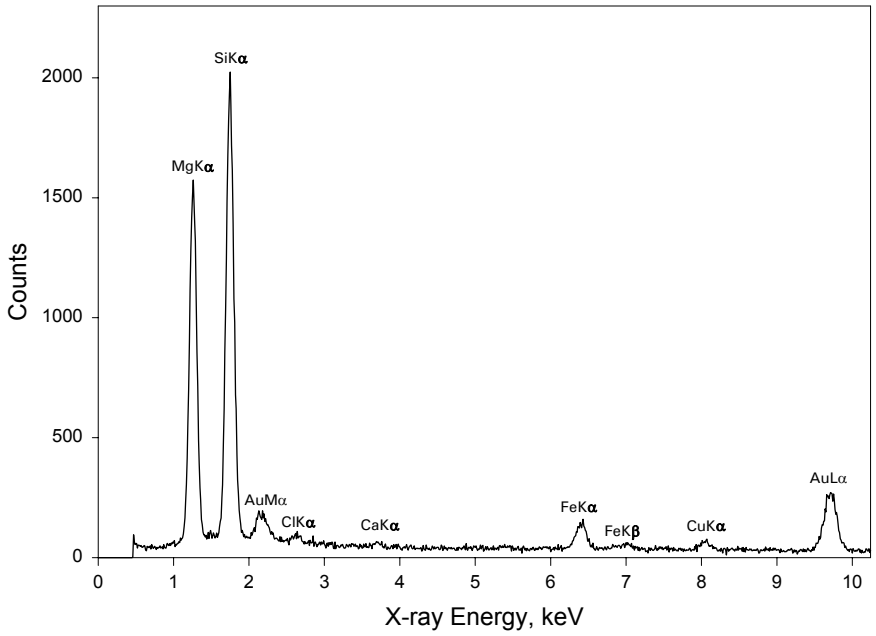


Figure F.1 — Energy dispersive x-ray spectrum obtained from NIST SRM 1866 chrysotile. The gold and small copper peaks originate from the gold specimen grid.

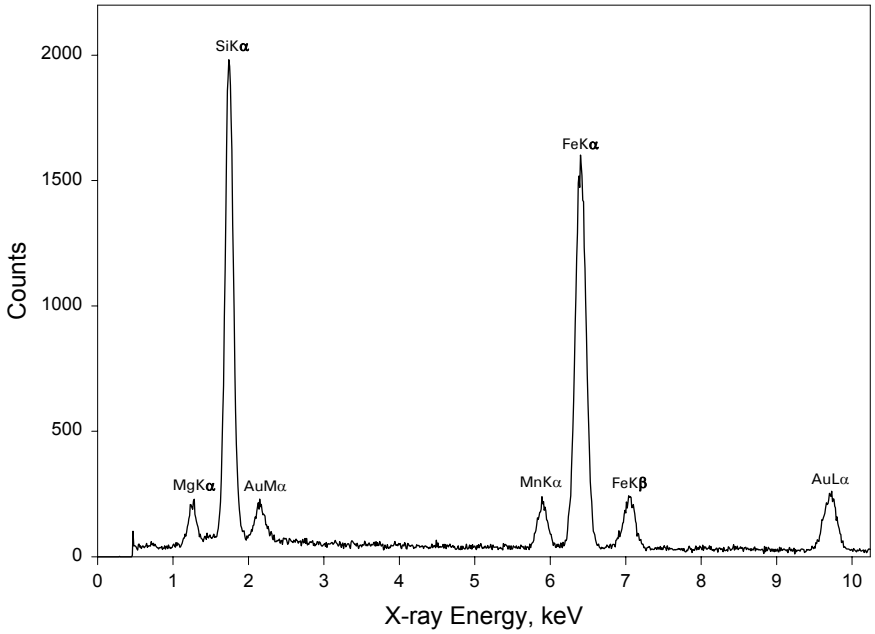


Figure F.2 — Energy dispersive x-ray spectrum obtained from NIST SRM 1866 amosite. The gold peaks originate from the gold specimen grid.

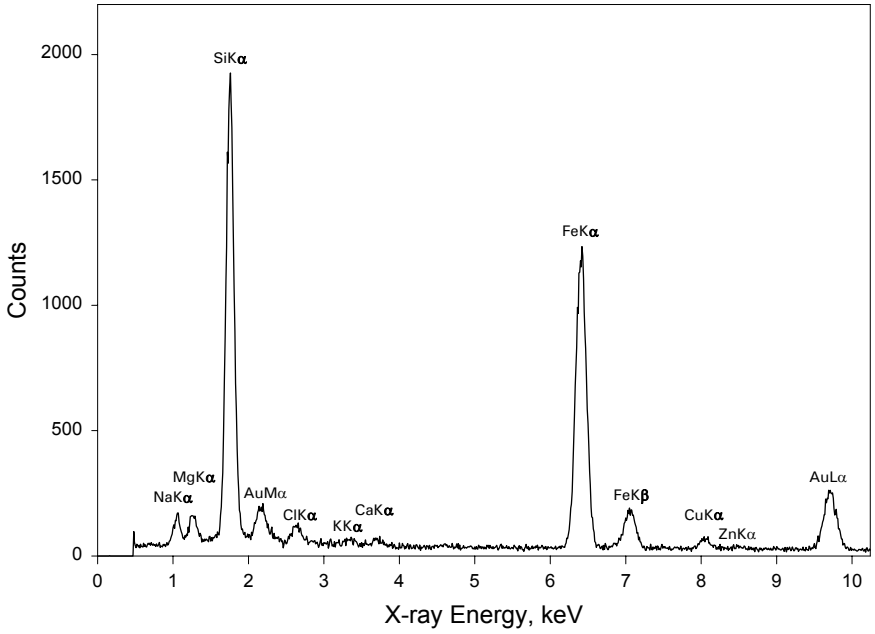


Figure F.3 — Energy dispersive x-ray spectrum obtained from NIST SRM 1866 crocidolite. The gold and small copper peaks originate from the gold specimen grid.

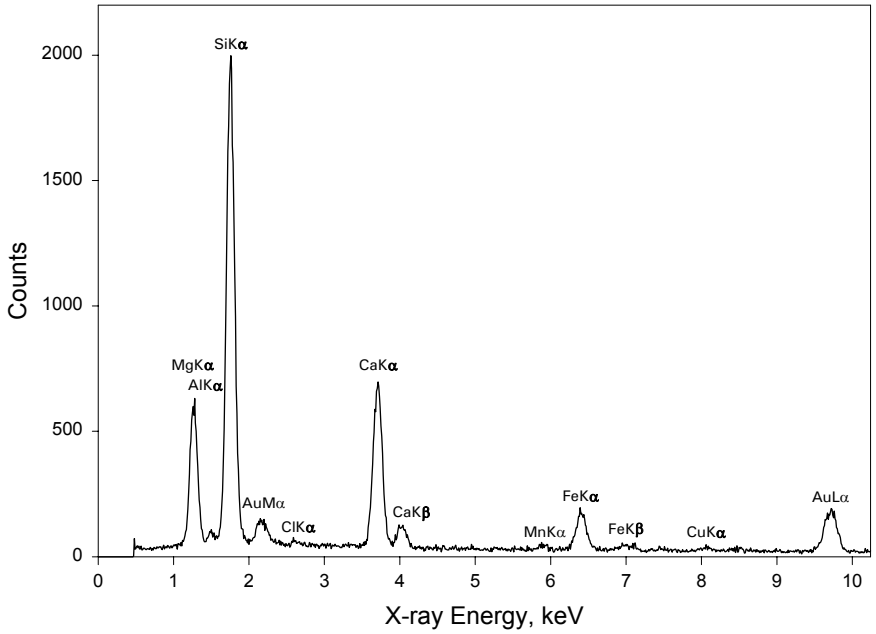


Figure F.4 — Energy dispersive x-ray spectrum obtained from NIST SRM 1867 tremolite. The gold and small copper peaks originate from the gold specimen grid.

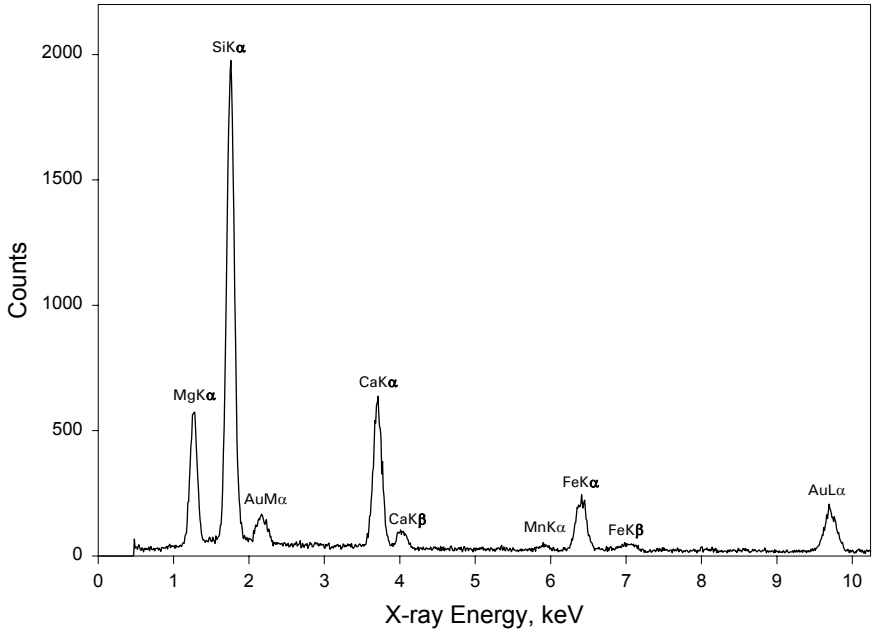


Figure F.5 — Energy dispersive x-ray spectrum obtained from NIST SRM 1867 actinolite. The gold peaks originate from the gold specimen grid.

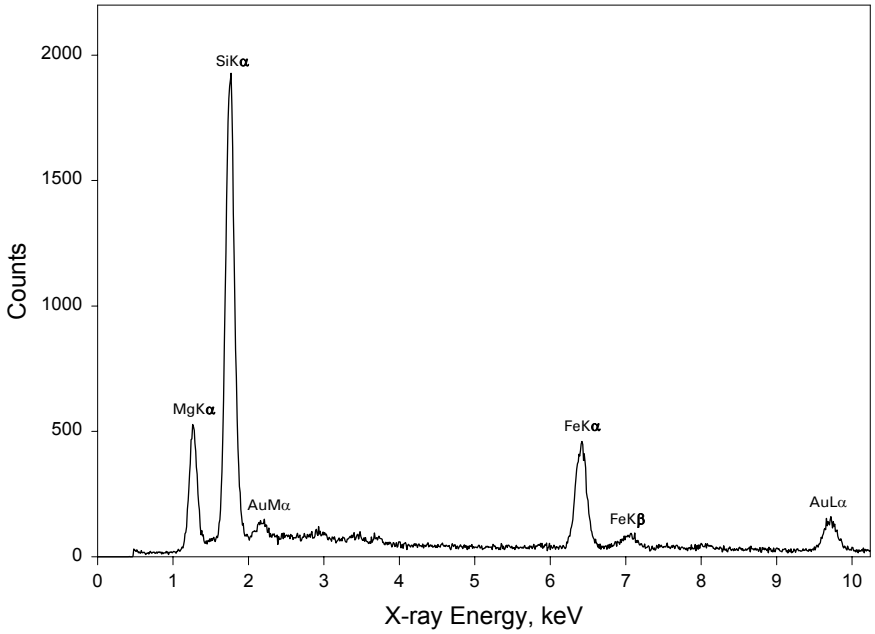


Figure F.6 — Energy dispersive x-ray spectrum obtained from NIST SRM 1867 anthophyllite. The gold peaks originate from the gold specimen grid.

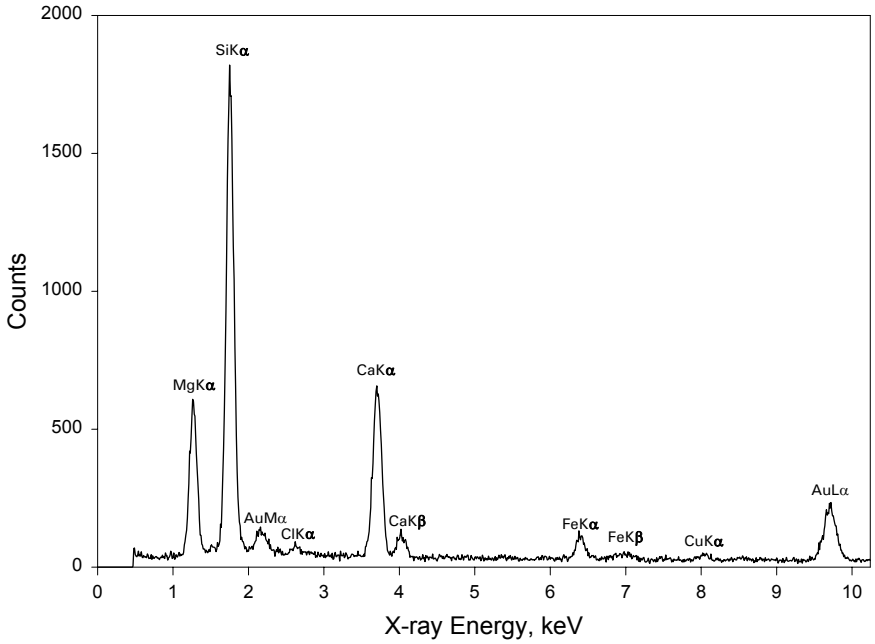


Figure F.7 — Energy dispersive x-ray spectrum obtained from IOM tremolite. The gold and small copper peaks originate from the gold specimen grid.

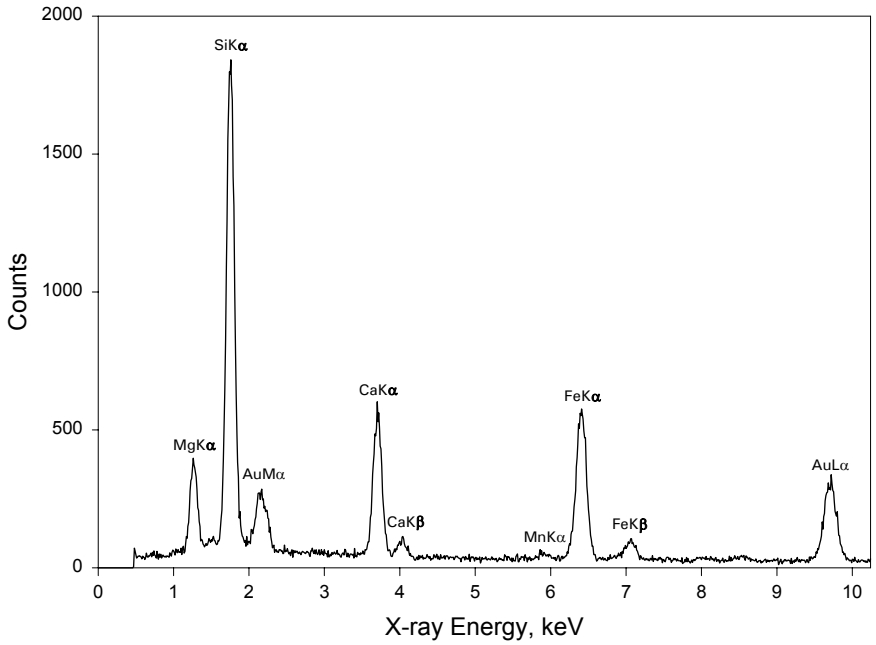


Figure F.8 — Energy dispersive x-ray spectrum obtained from IOM actinolite. The gold peaks originate from the the gold specimen grid.

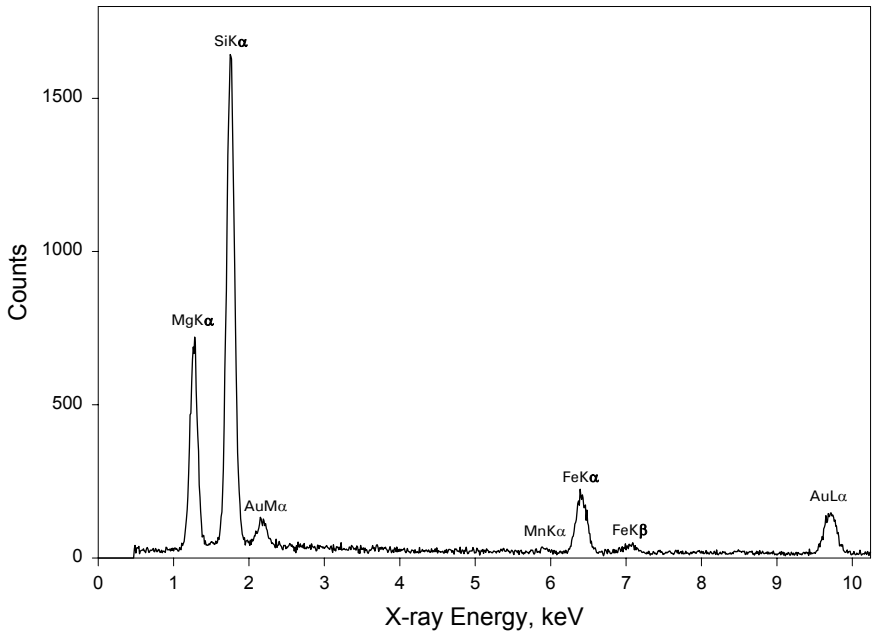


Figure F.9 — Energy dispersive x-ray spectrum obtained from IOM anthophyllite. The gold peaks originate from the gold specimen grid.

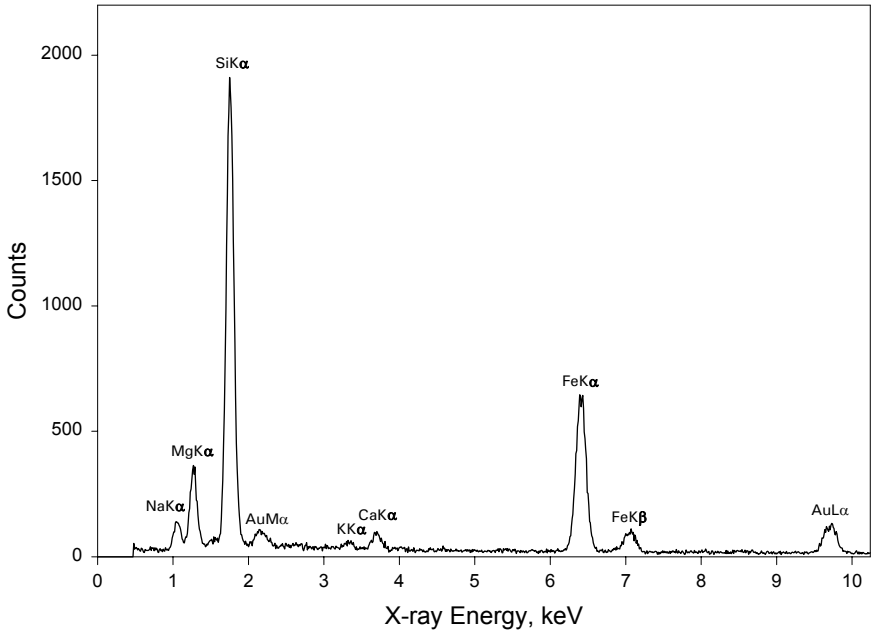


Figure F.10 — Energy dispersive x-ray spectrum obtained from Bolivian crocidolite. The gold peaks originate from the gold specimen grid.

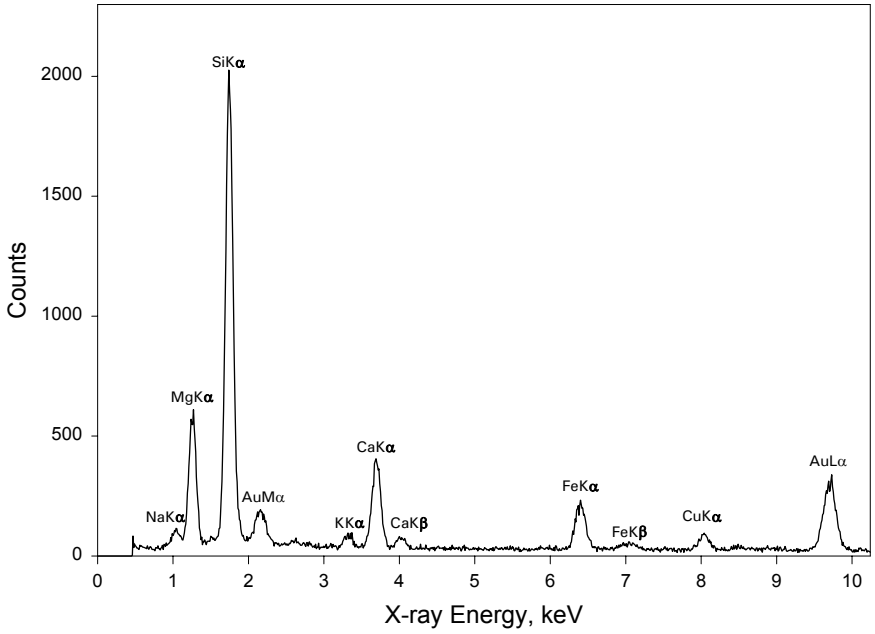


Figure F.11 — Energy dispersive x-ray spectrum obtained from richterite/winchite asbestos. The gold and small copper peaks originate from the gold specimen grid.

Annex G
(informative)

Example of sampling record

Date:	Samples taken by:
Building and Location:	

Room:		Sample identification:
Sampling Location:		
Reference:	Plan No:	Position in plan:
Sketch No:		Photo No:
Sample details:		
Comments:		

Annex H
(informative)

Example of analysis report

ANALYSIS OF BULK MATERIALS FOR ASBESTOS BY ISO 22262 PART 1

Date of Analysis:			
Analyst:		**Signature:**	
Notes:	Part 1 of ISO 22262 refers to qualitative analysis of commercial products for asbestos. In this method, polarized light microscopy with dispersion staining is the default procedure for identification of asbestos. If the sample characteristics required the use of either of the optional electron microscope methods to identify asbestos, the method used is indicated. If accurate quantification of asbestos concentration in the range below approximately 5% by weight is required for the purpose of determining the regulatory status of an asbestos-containing material, use the appropriate other parts of the Standard.		

Sample	Asbestos	Estimated asbestos concentration	Non-asbestos fibres	Comments
Sample 20050411-1 Pipe covering Grey corrugated paper	Chrysotile	5% - 50%	Cellulose Brucite	Sample ashed to remove interfering materials.
Sample 20050412-3 Pipe Covering White fibrous material	Amosite Chrysotile	5% - 50% 0,1% - 5%	None	
Sample 20050412-4 Fireproofing from beam Blue fibrous material	Crocidolite	50% - 100%	None	
Sample 20050413-1 Pipe covering Off-white fibrous material	None detected	0%	Mineral wool	
Sample 20050413-2 Plaster White material	Tremolite	0.1% - 5%	None	
Sample 20050413-3 Ceiling tile Grey fibrous material	Chrysotile	0,1% - 5%	Mineral wool Cellulose	Chrysotile too fine to identify by PLM. Chrysotile identified by TEM method.