The ISO Method for Determination of Asbestos in Bulk Materials:

Development of ISO 22262

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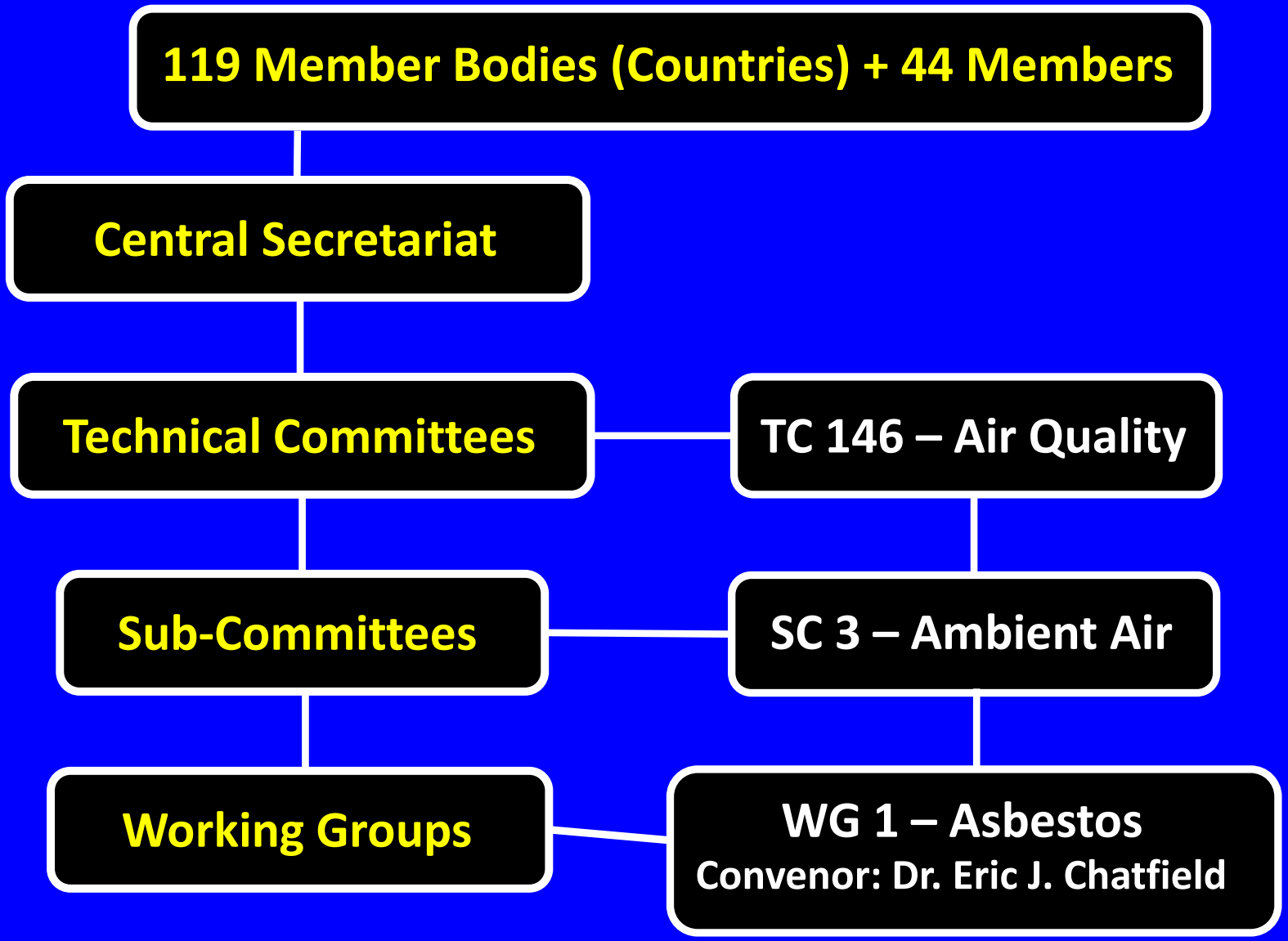
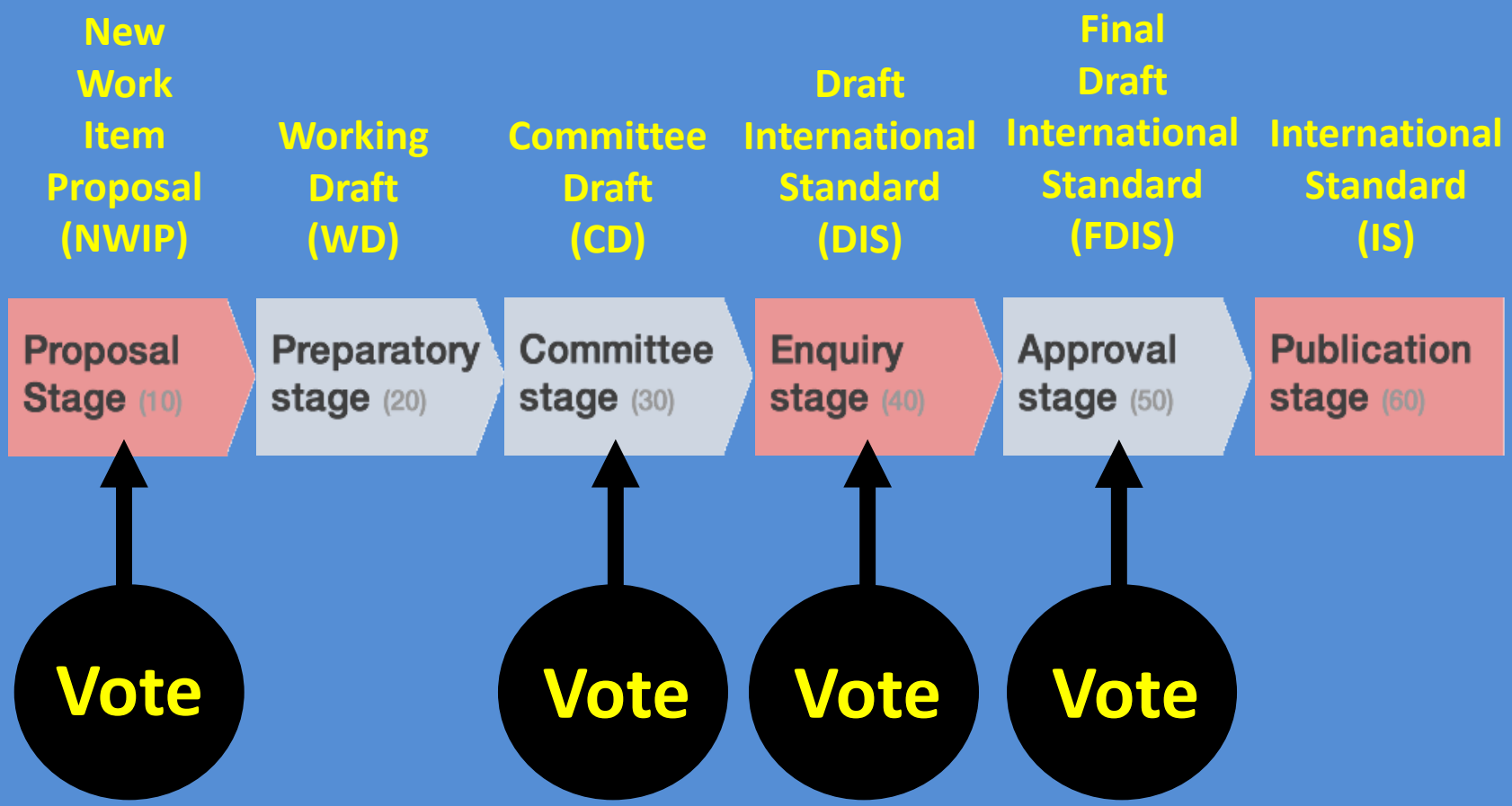
International Organization for Standardization (ISO)



Development Stages of an ISO Standard



Chronology of the Development of ISO 22262 by Working Group ISO/TC 146/SC 3/WG1

- At the 1998 meeting in Gaithersburg, a resolution was made to develop a method for determination of asbestos in bulk materials.
- At the 2000 meeting in Antalya, Turkey, a resolution was made that applicable national standards would be sent to the Convenor, who would use them as the basis to prepare a draft method for determination of asbestos in bulk materials.
- At the 2002 meeting in Toronto, a resolution was made that the WG would continue to work informally on an analytical method for determination of asbestos in bulk materials until SC 3 could arrange for the Scope of SC 3 to be expanded to include this topic.

Chronology of the Development of ISO 22262 by Working Group ISO/TC 146/SC 3/WG1 (Continued)

- At the 2003 meeting in Bautahøj, Denmark, a resolution was made to submit a new work item proposal for development of analytical methods for determination of asbestos in bulk materials.

- At the 2004 meeting in Stockholm, a resolution was made to prepare a first draft of a new Standard Method for qualitative determination of asbestos in bulk materials by the end of 2004.

- At the 2005 meeting in Burlington, a first draft of the new Standard Method was presented. Considerations such as European and U.S. positions regarding quantification of asbestos concentrations, definitions of trace and limit of detection were discussed.

Chronology of the Development of ISO 22262 by Working Group ISO/TC 146/SC 3/WG1 (Continued)

- At the 2006 meeting in West Conshohocken, a resolution was made to revise the draft of DIS 22262-1 in accordance with comments received from WG members.
- At the 2007 meeting in London, the new draft of DIS 22262-1 was discussed. It was also resolved that a preliminary draft of DIS 22261-2 (quantification of asbestos) would be prepared by the convenor and distributed to the WG members before September 2008.

Dr. Fred Wicks Dr. Norihiko Kohyama
Dr. Eric Chatfield Dr. Keiji Yada

Visit by Dr. Kohyama and Dr. Yada on 29 August 2008

Meeting with Dr. Kohyama and Dr. Yada in Toronto

- Dr. Kohyama provided a copy of JIS A 1481:2008 for submission to the ISO Working Group.
- At this meeting, the possible reactions of the ISO Working Group members to JIS A 1481:2008 were discussed, given that XRD had been rejected by most countries for identification and quantification of asbestos in bulk materials.
- Dr. Kohyama was provided with a set of 21 bulk samples of known compositions, most of which had been previously analyzed by the Environmental Protection Laboratory of Hong Kong in an inter-laboratory study.
- The purpose of these samples was to demonstrate the capability of JIS A 1481:2008 to the ISO Working Group. The results of these analyses will be summarized later.

Chronology of the Development of ISO 22262 by Working Group ISO/TC 146/SC 3/WG1 (Continued)

- During the week of 13 – 17 October 2008, the Secretary to the ISO Technical Management Board, Mr. Mike Smith, was approached by Professor Masami Tanaka. Professor Tanaka was ISO President during 2005 – 2006.
- Prof. Tanaka stated that Japanese law required that asbestos determinations be made by XRD, and wondered how such a method could be included in the ISO Standards.
- Mr. Smith contacted me to inquire if there would be any impediments to standardizing the X-ray method.
- Mr. Smith was informed that JIS A 1481:2008 was to be presented to the Working Group at the next meeting, and the Working Group would assess whether it could form a part of ISO 22262.

Chronology of the Development of ISO 22262 by Working Group ISO/TC 146/SC 3/WG1 (Continued)

- At the 2008 meeting Berlin, it was resolved that the draft of CD 22262-1 (reverted from the DIS draft) would be submitted for voting.
- Changes to the preliminary draft of CD 22262-2 were also discussed, and it was resolved that a revised draft would be prepared and sent to WG members for review before April 2009.
- It was resolved that a proposal from the Japanese delegation to add an XRD method for quantification of asbestos in bulk materials as Part 3 of ISO 22262 would be submitted for voting as a new work item.

Polarized Light Microscopy
was Selected
By the Working Group
As the Basis of ISO 22262

Why?

Polarized Light Microscopy has a Long History in Mineral Identification

- Polarized light microscopy (PLM) has been used to identify minerals based on their optical properties since the 1800's.
- PLM is still a major tool used for the identification of minerals.
- PLM has been adopted by many countries for the identification of asbestos fibres in building materials.
- The introduction of the dispersion staining objective as an accessory to the PLM made identification of asbestos fibres straightforward and requires a minimum of training.

Why Polarized Light Microscopy was Selected As the Basis of ISO 22262

1) It is rapid. For many materials, results can be obtained in a few minutes.

2) It is inexpensive. The only major equipment requirement is a polarized light microscope.

3) The PLM method identifies asbestos unequivocally.

4) The PLM method is very sensitive. In the absence of matrix interferences, concentrations as low as 2 ppm can be detected.

5) It meets the ISO requirement that the method can be widely used.

6) At the beginning of the ISO project in 1998, there was already more than 20 years' experience in many countries in the use of polarized light microscopy (PLM) for identification and quantification of asbestos in building materials.

Why Polarized Light Microscopy was Selected as the Basis of ISO 22262 (Continued)

6) By 1998, identification of asbestos by PLM and dispersion staining was known to be reliable when performed by an analyst with the proper training and experience.

7) A 1-week intensive course was sufficient to train an analyst to perform asbestos identification by PLM and dispersion staining.

8) Training courses on asbestos identification by PLM and dispersion staining had been available in the U.K. since 1969 and the U.S.A. since 1973. Thousands of analysts had been trained.

9) The first book for training of analysts to identify asbestos by PLM and dispersion staining was published in 1980 by Dr. Walter McCrone of the McCrone Research Institute.

10) A second edition, re-named "Asbestos Identification", was published in 1987.

Consideration by the ISO Working Group of Other Available Instrumental Techniques for the Qualitative Examination of Bulk Building Materials for Asbestos

1) Scanning electron microscopy SEM) with energy dispersive x-ray analysis (EDXA) and Transmission electron microscopy (TEM) with EDXA and electron diffraction were considered useful for identification of fibres.

2) Infra-red analysis was rejected because of its limitations of sensitivity and lack of specificity.

3) X-ray diffraction was rejected primarily because it does not discriminate between asbestos and non-asbestiform amphiboles. It was also considered to be subject to unacceptable interferences when used to analyze real building materials.

Published Analytical Methods Formed the Basis of ISO 22262

- In 1994, three analytical methods had been published.
- All were based on the use of polarized light microscopy (PLM):
  - United States Environmental Protection Agency (1982): Interim Method for the Determination of Asbestos in Bulk Insulation Samples. EPA Report EPA-600/M4-82-02
  - United States Environmental Protection Agency (1993): Test Method, Method for the Determination of Asbestos in Bulk Building Materials. EPA Report EPA-600/R-93/116.
  - Health and Safety Executive (1994): Asbestos in Bulk Materials, MDHS 77 (Now HSG 248)
- In 2004, Standards Australia published an analytical method. This was also based on PLM.

Vote on DIS 22262-1, 2010-04-09

Result of voting

P-Members voting: 10 in favour out of 11 = 91 % (requirement >= 66.66%)

(P-Members having abstained are not counted in this vote.)

Member bodies voting: 1 negative votes out of 13 = 8 % (requirement <= 25%)

Approved

The negative vote was cast by the U.S.A., relating to discrimination between amphibole asbestos and non-asbestiform amphibole.

This negative vote was resolved at the 2010 meeting in Maui by incorporating a minor change.

INTERNATIONAL STANDARD

ISO 22262-1

First edition
2012-07-01

Air quality — Bulk materials —

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

Qualité de l'air — Matériaux solides —

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

ISO 22262-1 Was Published in 2012

Objective of and Requirements for the Analysis

- **To establish the regulatory status of a material**
  - **Does the asbestos concentration exceed that defined by a regulatory body as constituting an "asbestos-containing material (ACM)"?**

- **Depending on the jurisdiction, a regulated ACM is defined as:**
  1) **A material containing any detectable asbestos;**
  2) **> 0,1% asbestos;**
  3) **> 0,5% asbestos;**
  4) **> 1% asbestos.**

Objective of and Requirements for the Analysis (Continued)

- **Once it is known that the asbestos concentration in a building material exceeds the applicable regulatory limit, further knowledge about the actual concentration of asbestos does not change the regulatory action in any way.**

- **For asbestos concentrations that are obviously much higher than the regulatory limit, visual estimation of the asbestos concentration by PLM is sufficient for the purpose of the analysis.**

- **The only region where precise quantification of the asbestos concentration is important is between "none detected" and 1%, i.e. at concentrations close to the applicable regulatory limit.**

For many types of asbestos-containing material, PLM examination of the untreated material is sufficient

1) Commercial asbestos-containing building materials (ACM) almost always have asbestos fibres sufficiently large that they can be removed using tweezers.

2) Identification of large asbestos fibres by PLM is very rapid and straightforward.

3) After identification of the asbestos, a random sample of the material is examined by PLM.

4) For many types of ACM, it is intuitively obvious that the asbestos concentration far exceeds any of the regulatory standards.

5) Pulverization of the material is not necessary, and, if performed, makes it more difficult to identify asbestos.

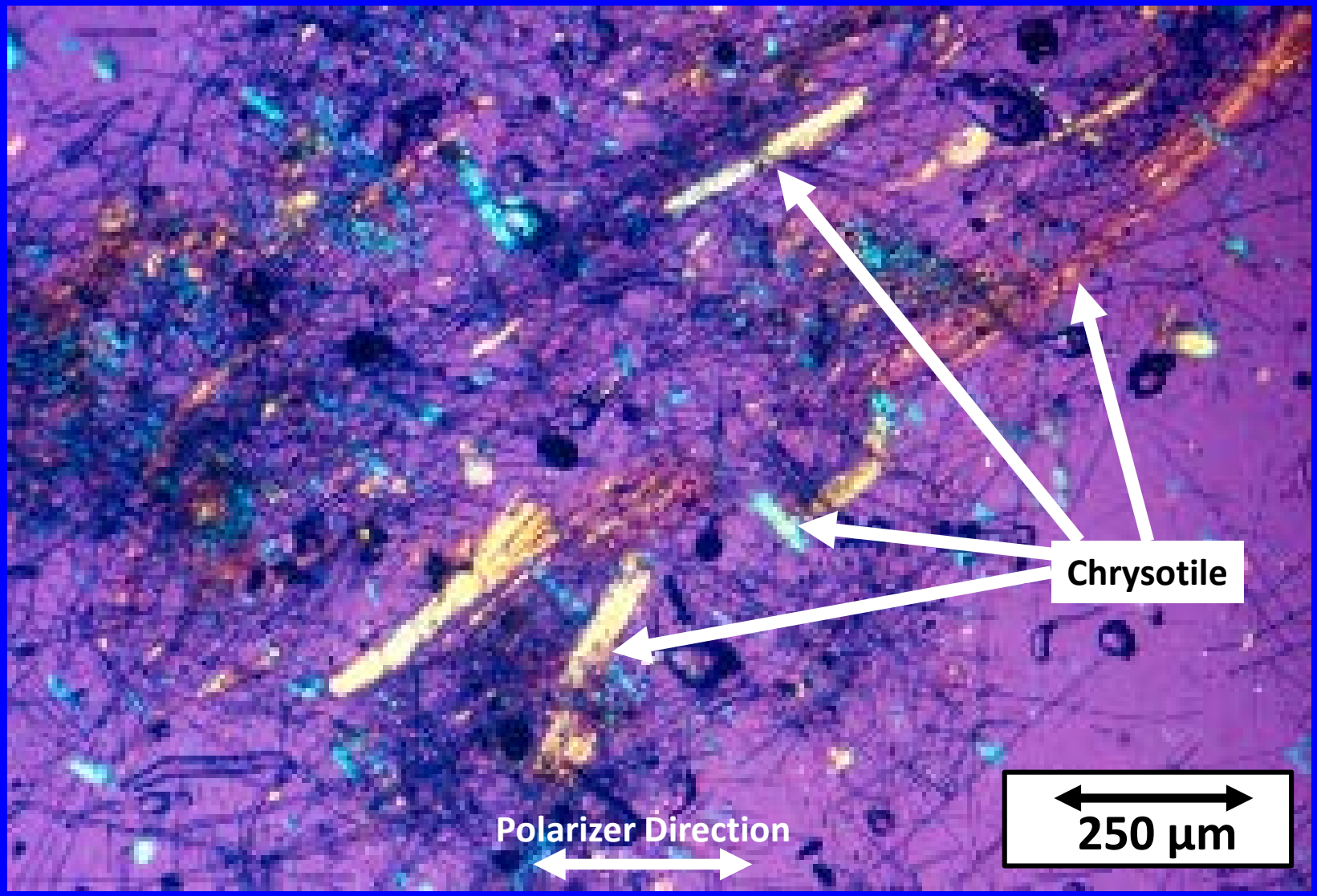
Identification of Asbestos in Fireproofing, Thermal Insulation and Cement Products by Polarized Light Microscopy

- For materials such as fireproofing, thermal insulation, cement boards and cement pipes, there is either a large proportion of asbestos or no added asbestos. This type of sample can be examined directly by PLM and dispersion staining. No sample preparation prior to PLM observation is required.

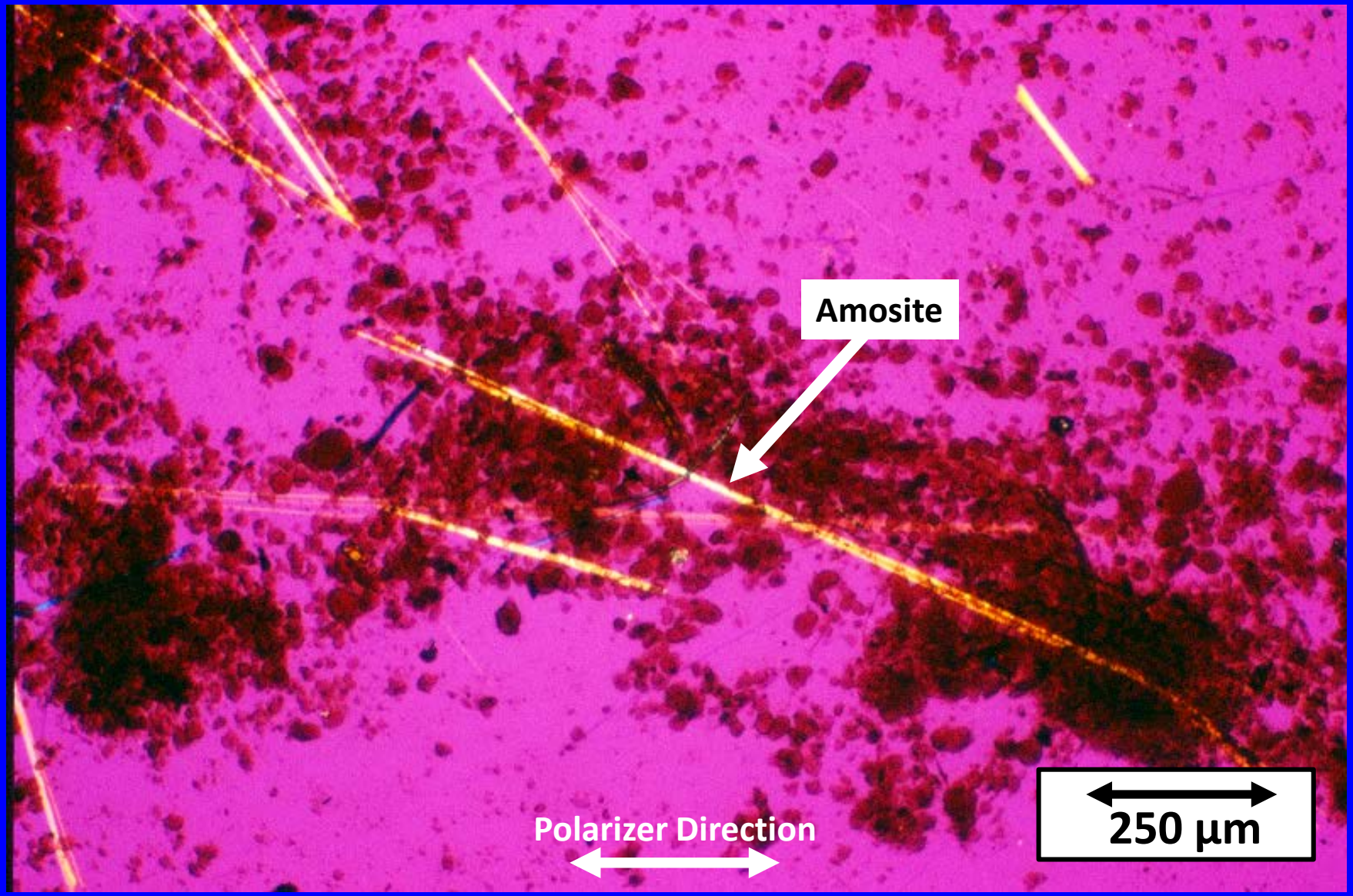
- Classification of such materials as either asbestos-containing (ACM) or containing no detectable asbestos can often be achieved after 5 - 15 minutes of observation on the PLM.

Asbestos Identification by ISO 22262-1

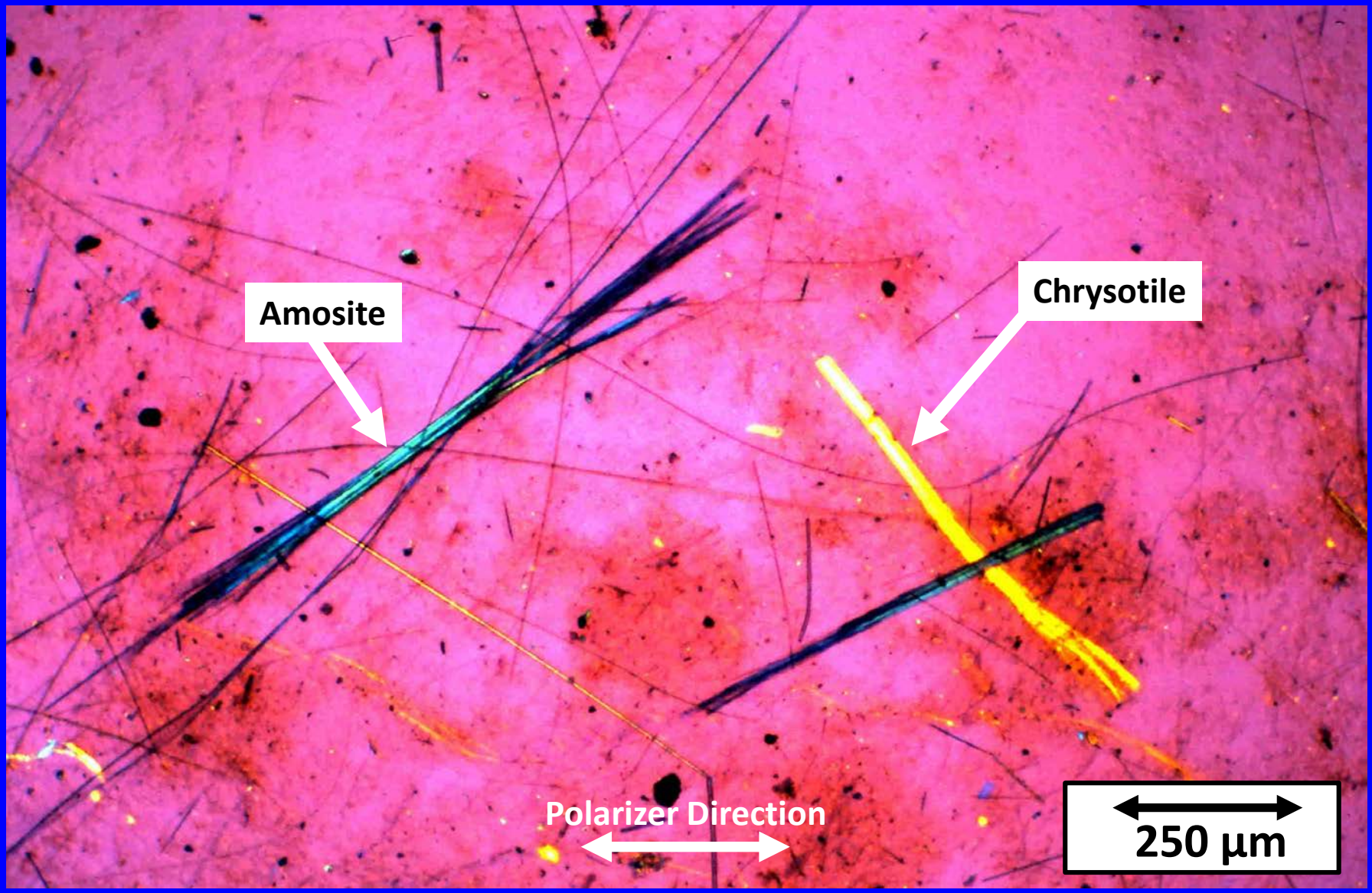
- Examine the sample under a stereo-binocular microscope for the presence of fibres. Select suspected asbestos fibres for identification.
- Prepare a slide on which the suspected asbestos fibres are immersed in the appropriate R.I. liquid for the suspected variety of asbestos.
- Observe:
  - the colour of the fibres;
  - the birefringence;
  - the sign of elongation;
  - pleochroism (parallel and perpendicular to the fibre length).
- Record the parallel λ_o and the perpendicular λ_o.
- Check that the two values of λ_o are within the specified acceptable range for the suspected mineral.
- Optionally, SEM-EDXA or TEM-EDXA may be used.



PLM Micrograph of Sprayed Fireproofing in a liquid of R.I. 1.550. The Sample Contains Mineral Wool and 20% Chrysotile.

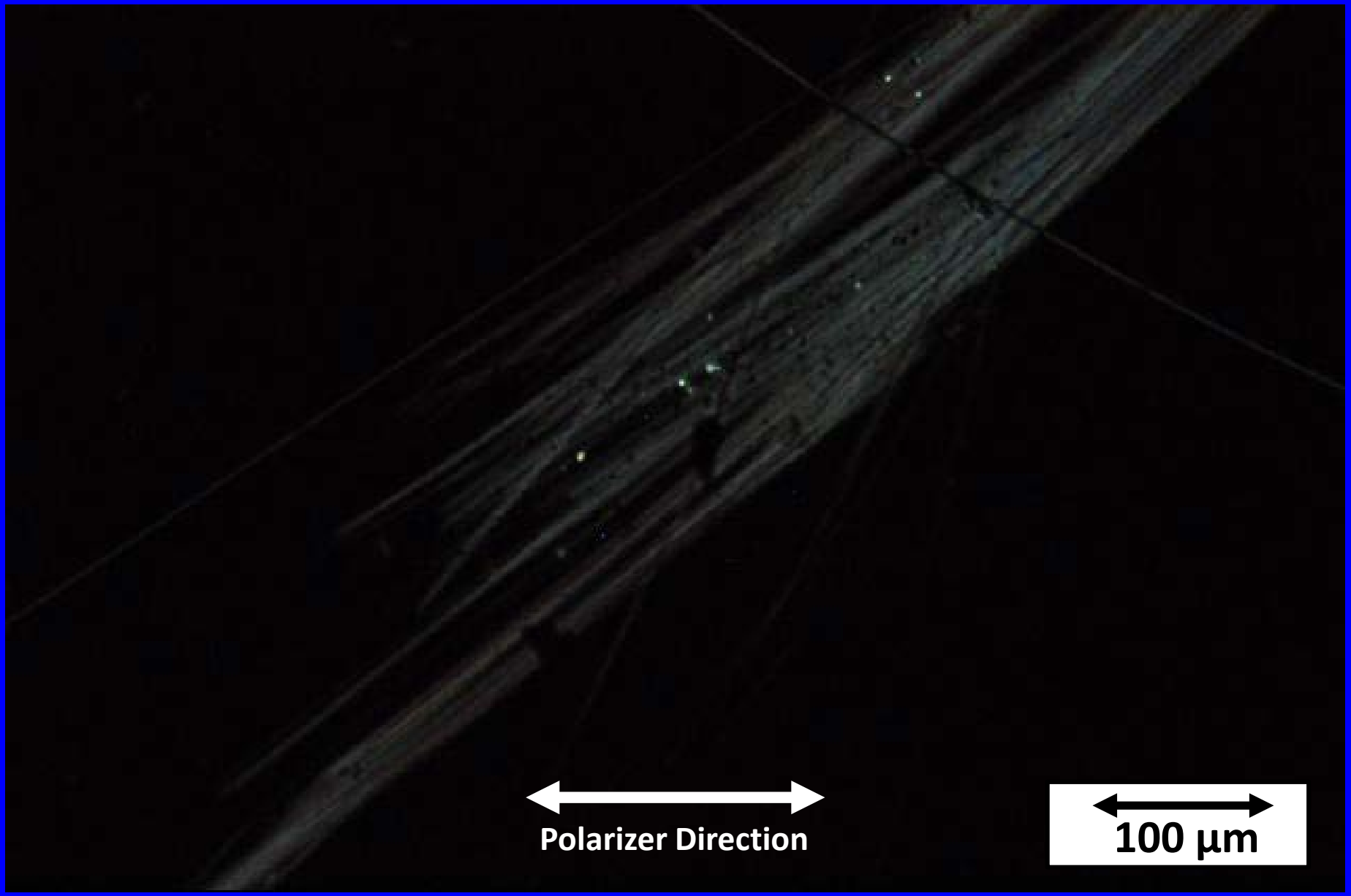


**PLM Micrograph of Thermal Insulation in a Liquid of R.I. 1.680.
The Sample Contains Hydromagnesite and 15% Asbestos (Amosite).**

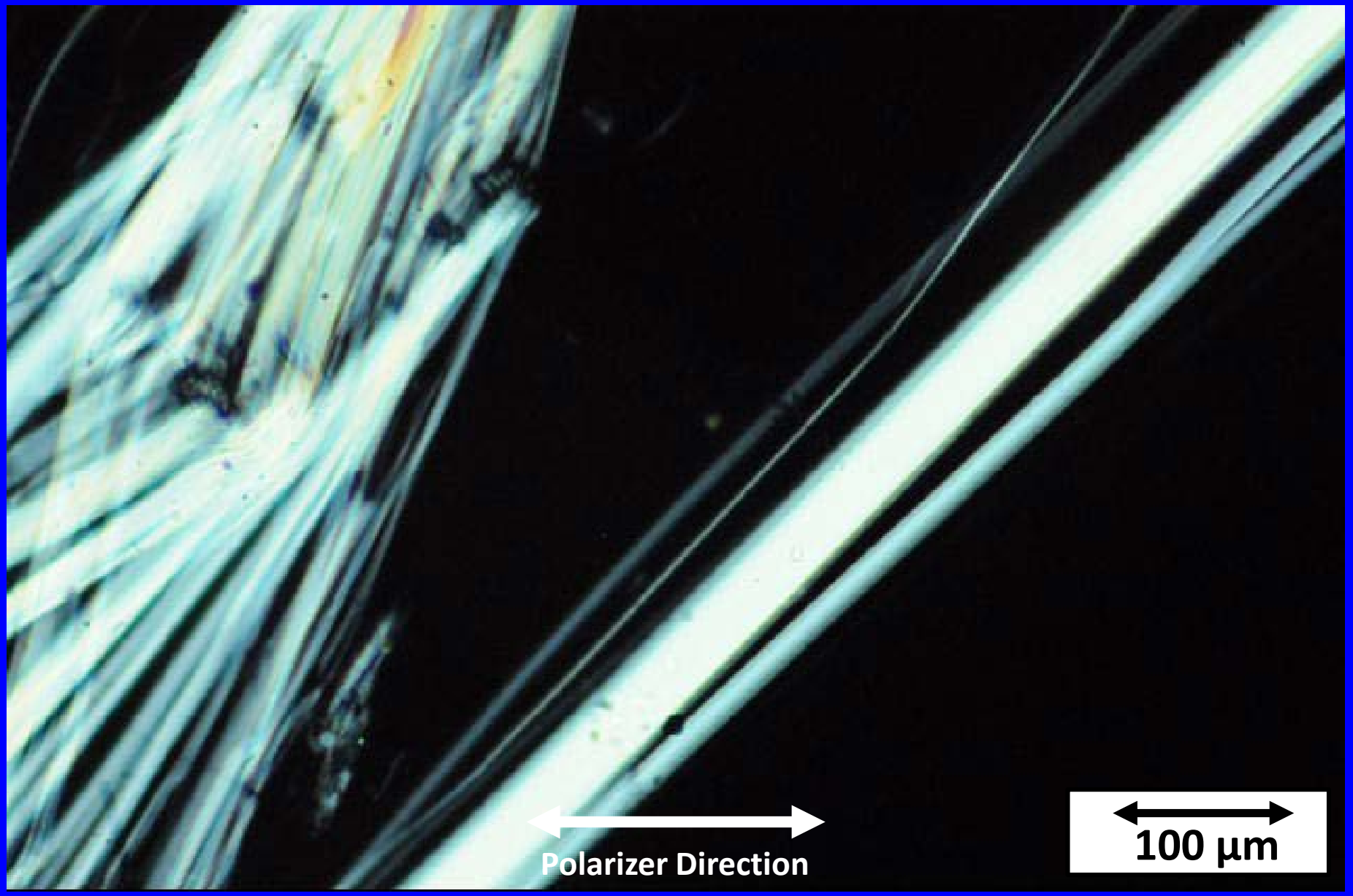


PLM Micrograph of Thermal Insulation in a Liquid of R.I. 1.550. The Sample Contains Hydromagnesite and 15% Asbestos (Amosite and Chrysotile).

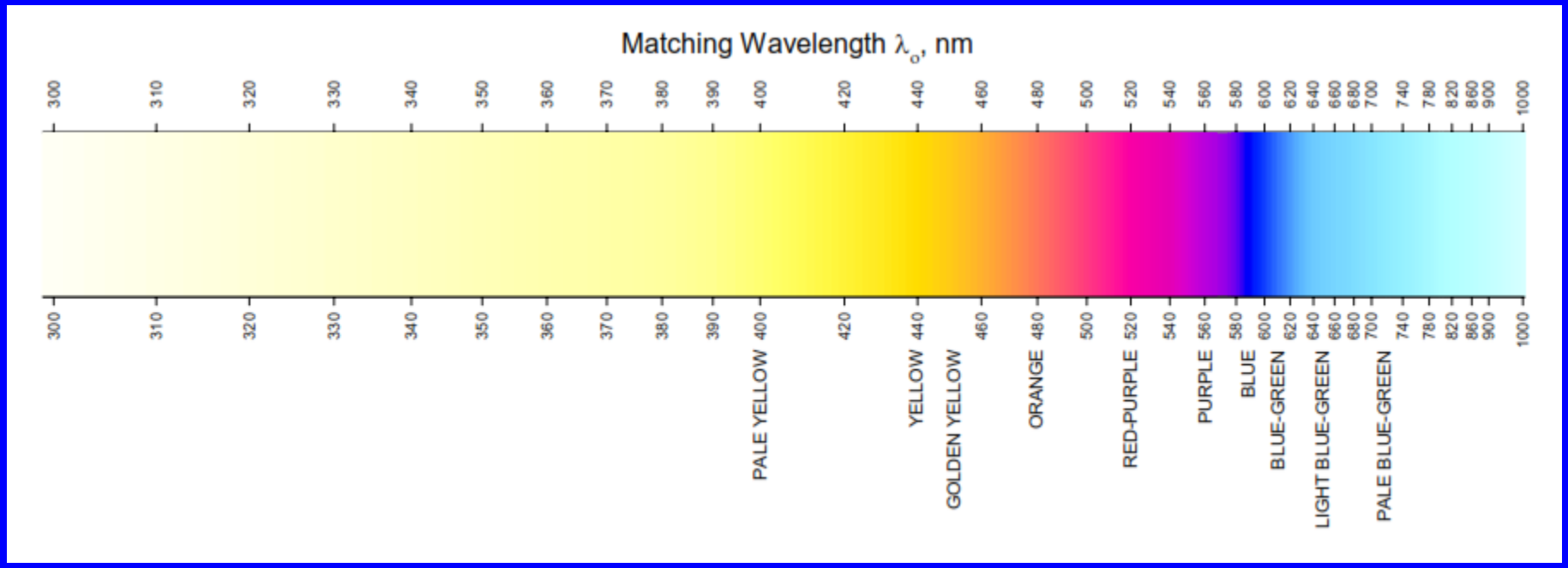
Identification of Amosite by PLM – Dispersion Staining



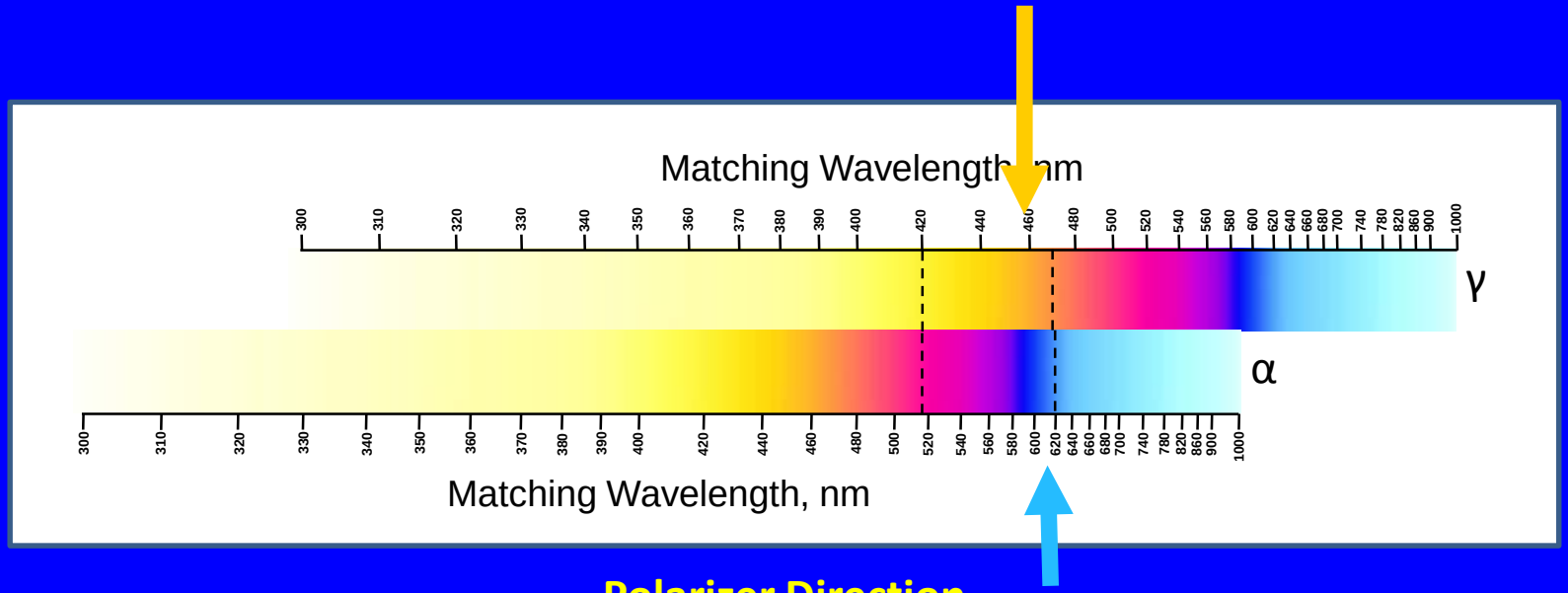
Identification of Crocidolite by PLM – Dispersion Staining



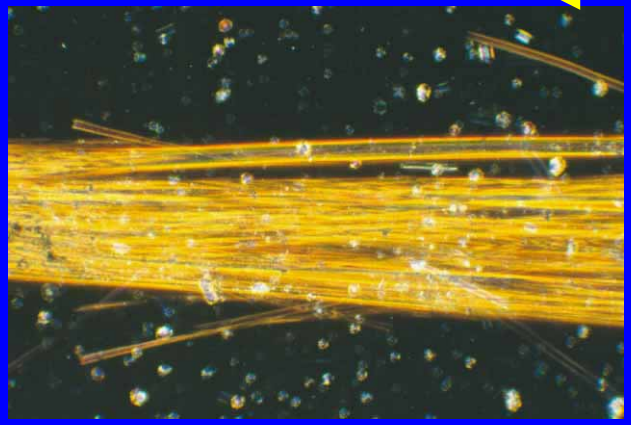
Identification of Chrysotile by PLM – Dispersion Staining



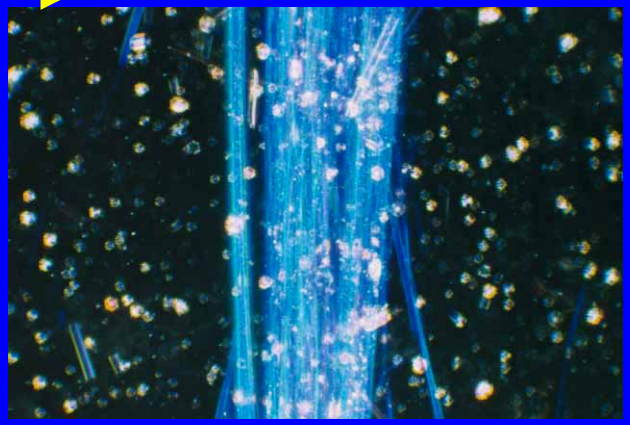
An accurate dispersion staining chart was produced for ISO 22262-1



Polarizer Direction

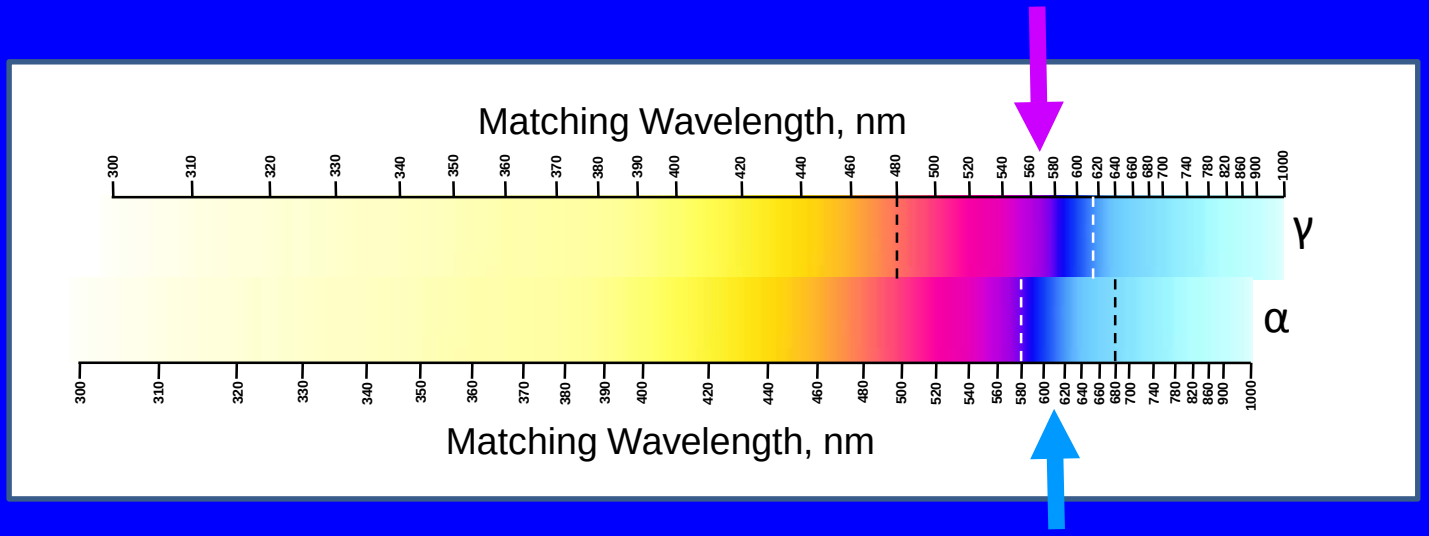


γ (Parallel)

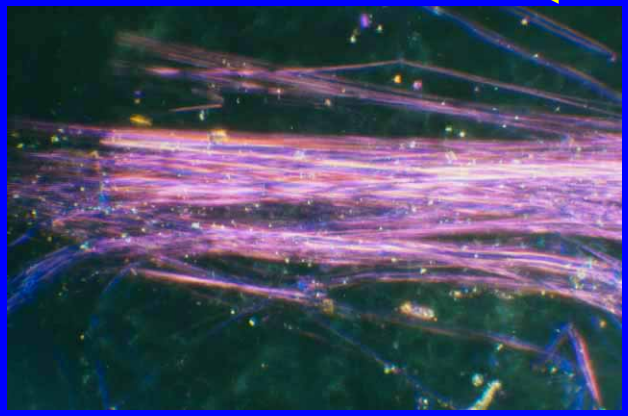


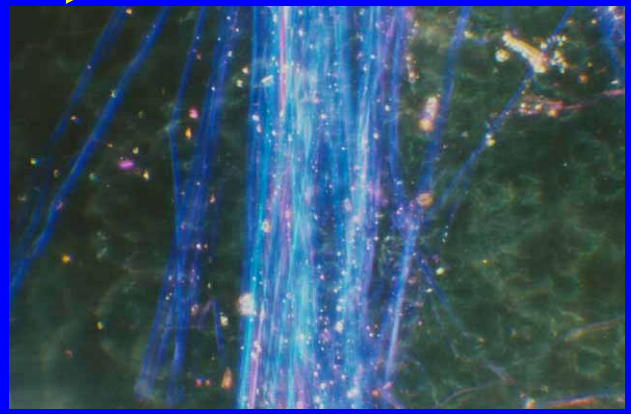
α (Perpendicular)

Dispersion Staining Colours for Amosite



Polarizer Direction





γ (Parallel) **α (Perpendicular)**

Dispersion Staining Colours for Chrysotile

There have been claims that polarized light microscopy is not capable of measurements below 1% asbestos.

This is not correct.

Limit of Detection for Polarized Light Microscopy

1) Assume that 1 mg of the sample is placed on the microscope slide.

2) Assume that all particles are visible and separate.

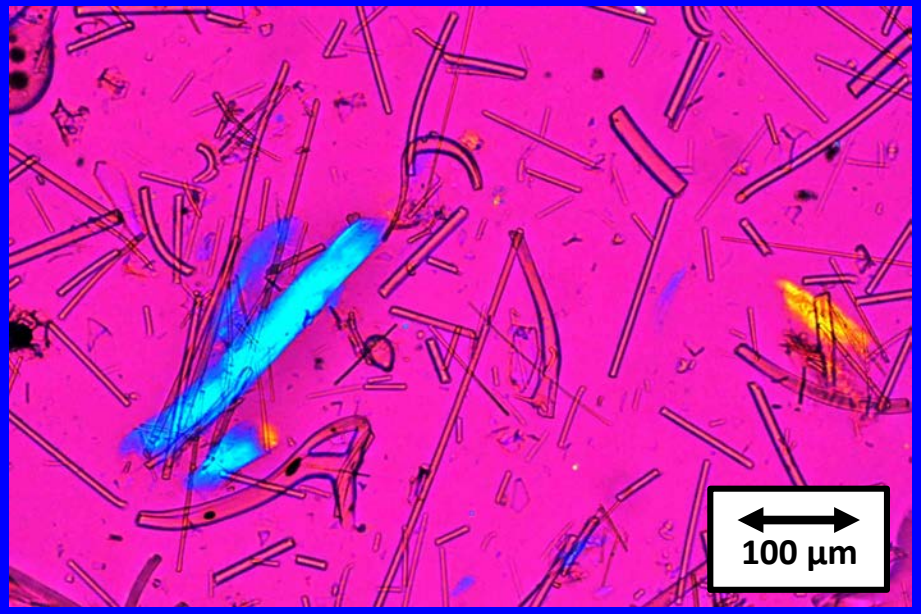
3) Assume that a single asbestos fibre 100 µm long and 2 µm diameter is on the microscope slide.

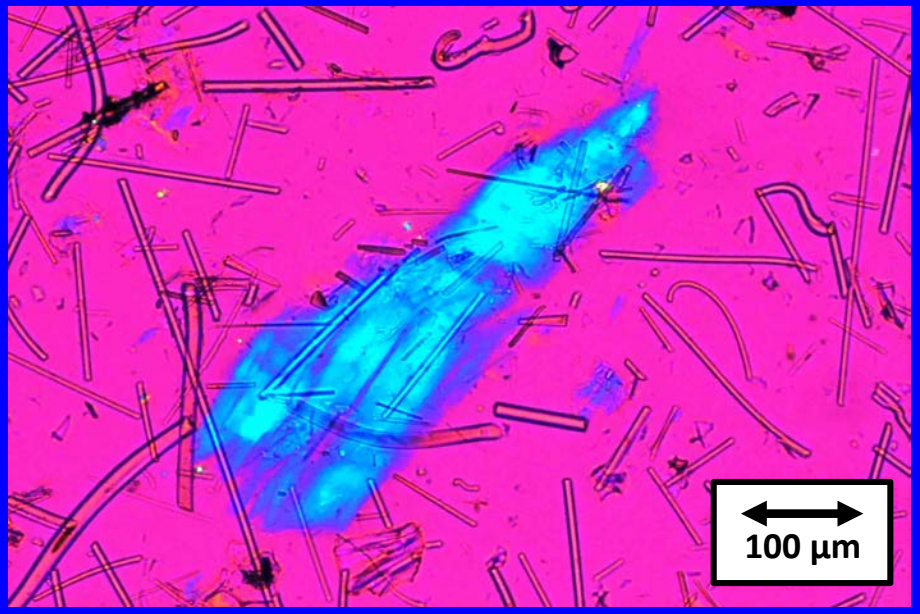
4) This gives a theoretical limit of detection of approximately 0.0001% or 1 part per million.

5) Matrix obscuration can degrade this LOD, but an LOD significantly lower than 0,01% is achieved by use of appropriate gravimetric reduction methods prior to microscopical examination.

Detection of Coalinga (Calidria®) Chrysotile

- Coalinga (Calidria) chrysotile was mined near King City, California.
- Large amounts of this chrysotile were exported to Japan.
- Coalinga chrysotile was used in:
  - Resilient floor tiles;
  - Ceiling tiles;
  - Extruded cement products;
  - Bakelite.
- There have been claims that Coalinga chrysotile cannot be detected by PLM. These claims are incorrect.
- This chrysotile has unique morphology. All fibres are shorter than 30 µm, but aggregates and bundles can be detected and identified by PLM.
- Products containing this chrysotile have been found in Japan.





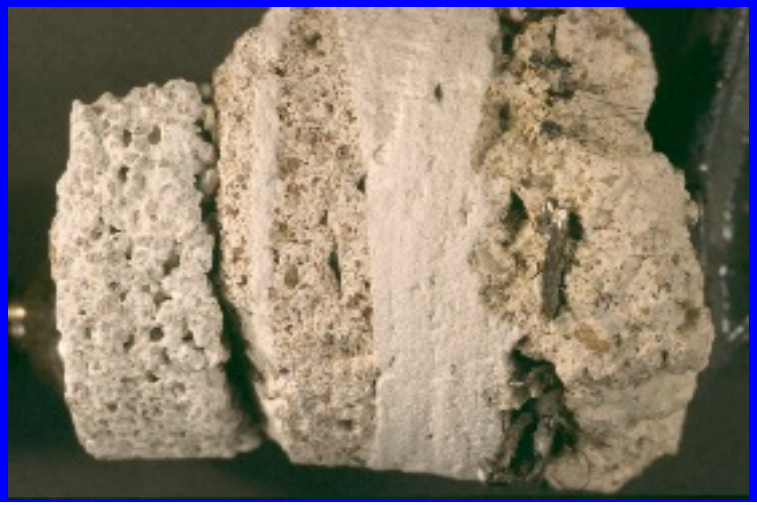
Polarizer Direction

Sample mounted in 1.550 R.I. liquid and examined by PLM – dispersion staining

PLM Micrograph of Coalinga Chrysotile in a Ceiling Tile

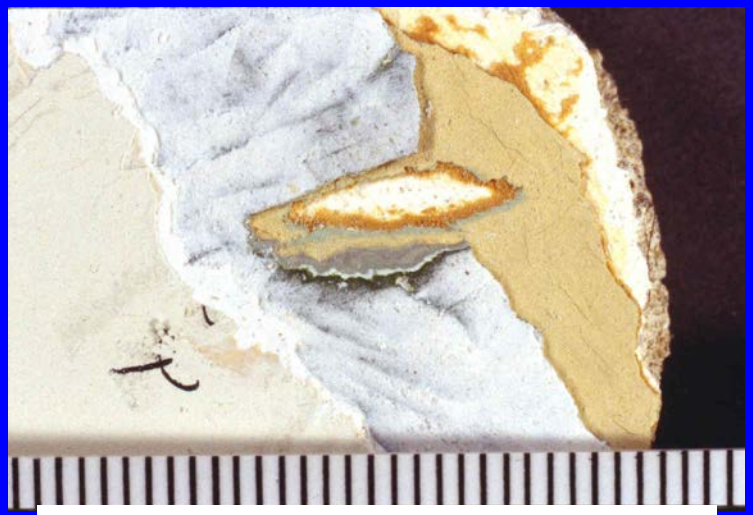
Materials That Require Quantification

- **Quantitative determination of asbestos in materials estimated by ISO 22262-1 to contain less than 5% asbestos.**
- **Quantitative analysis of materials with difficult matrices, such as plasters and cements that have aggregate as a constituent.**
- **Quantitative analysis of mineral products such as vermiculite and talc.**



5 cm Diameter Plaster Core, 6 Layers

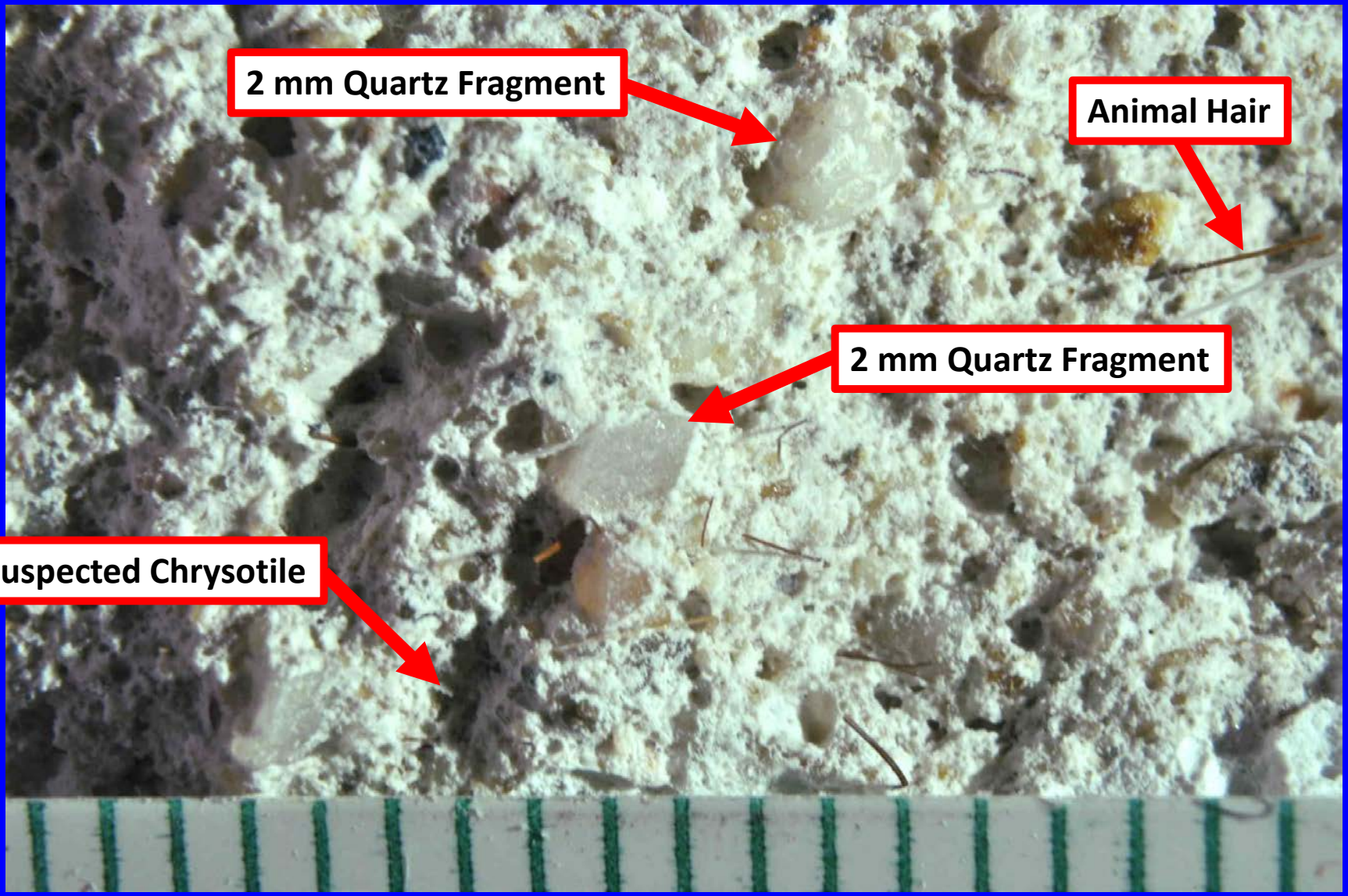
Asphalt Roofing Material



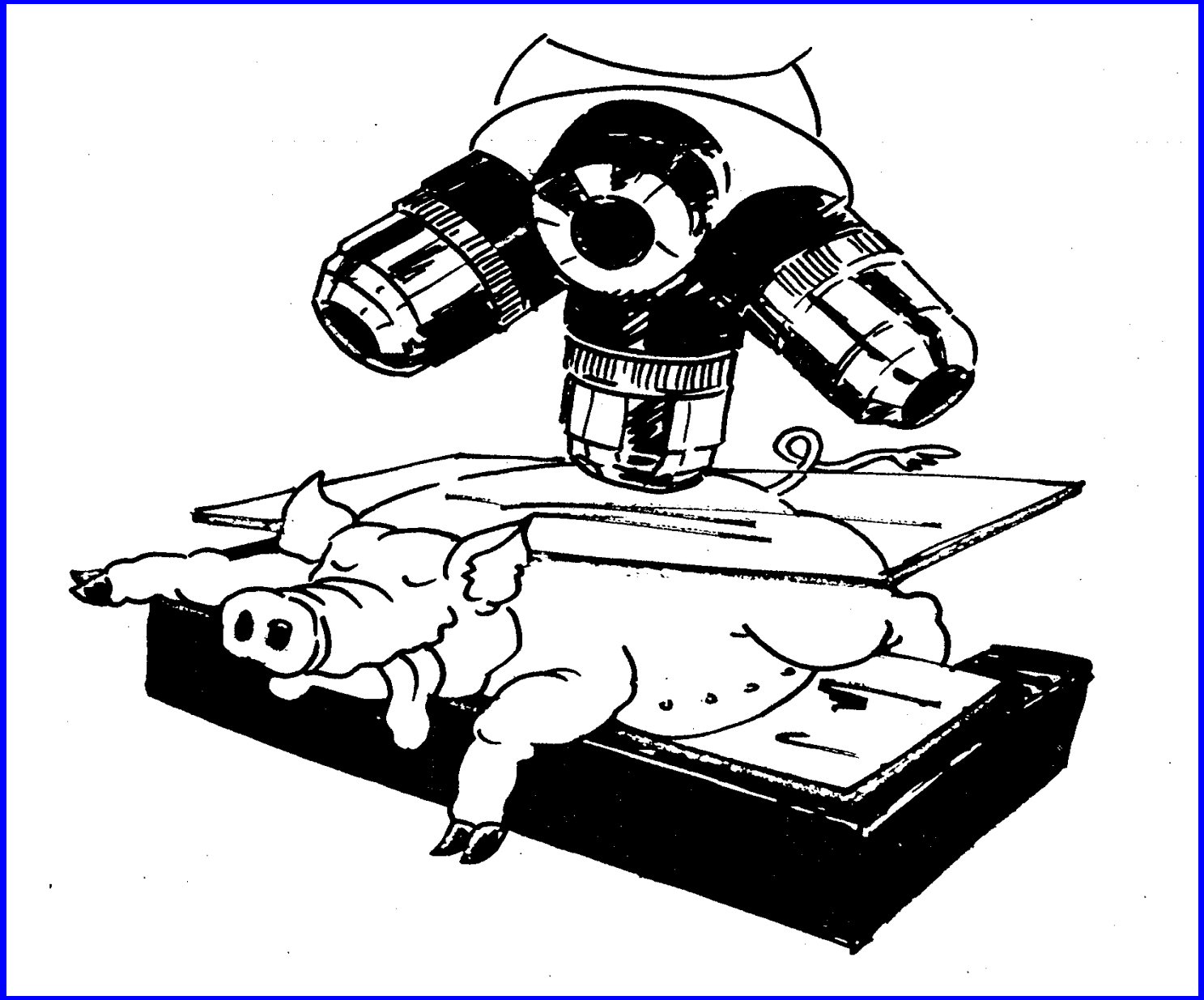
Skim Coat Between Paint Layers

Resilient (Vinyl) Floor Tiles

Typical Sample Types Analyzed Using ISO 22262-2



Plaster Containing Sand, Hair and Chrysotile
Does it Contain More than 0,1% Chrysotile?



For Some Types of Plaster Samples, Attempts to Prepare Microscope Slides for PLM are Generally Unsuccessful

Vote on DIS 22262-2, 2013-02-09

Result of voting
P-Members voting: 10 in favour out of 10 = 100 % (requirement >= 66.66%)
(P-Members having abstained are not counted in this vote.)
Member bodies voting: 0 negative votes out of 13 = 0 % (requirement <= 25%)
Approved

ISO/DIS 22262-2 proceeded to FDIS voting on 2014-07-07.

INTERNATIONAL STANDARD

ISO 22262-2

First edition
2014-09-01

Air quality — Bulk materials —

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

Qualité de l'air — Matériaux solides —

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

ISO 22262-2 Was Published in 2014

Requirements for Quantification of Asbestos

Type of Material	Regulatory Control Limit			
	"Any Asbestos"	>0.1%	>0.5%	>1%
Commercially Manufactured Product	If any commercial asbestos variety is present, no further quantification required		If asbestos concentration is estimated as <5%, more precise quantification is required	
Other Materials	If any variety of asbestos is detected, no further quantification is required	If asbestos concentration is estimated as <5%, more precise quantification is required		

In ISO 22262-2
The Sample Matrix and Type of Asbestos Define the Optimum Analytical Procedure

- **Because of the wide range of sample matrices in building materials, it is not possible to write a step-by-step method that applies to all types of sample.**
- **ISO 22262-2 specifies a number of gravimetric and microscopical techniques that can be selected and combined to provide the optimum analytical procedure for the particular sample matrix.**
- **For the majority of matrix types, a limit of quantification (LOQ) of 0,001% or lower can be achieved.**

ISO 22262-2 – Gravimetric Reduction Methods

1) Ashing to remove organic materials.
2) Removal of acid-soluble materials.
3) Removal of light materials that float on water.
4) Removal of heavy aggregates by sedimentation.
5) Removal of magnetic particles.
6) For determination of amphibole only, successive refluxing in 2M hydrochloric acid and 4M sodium hydroxide.
7) Heavy liquid centrifugation for amphibole separation.

ISO 22262-2 Procedures for Quantification of Asbestos in the Residue from Gravimetric Reduction

1) Gravimetry alone.
2) Gravimetry plus visual estimation.
3) Gravimetry plus optical point counting.
4) Gravimetry plus SEM or TEM fibre measurements.

ISO 22262-2 Guidance for Analysis of Sprayed Asbestos-Containing Materials

APPLICATIONS

Contour-following fire-resistant coating of steel structures

Coating of ceilings and walls for noise protection

Sealing of pipe, duct and cable feed-throughs

TYPICAL COMPOSITIONS

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

RECOMMENDATION FOR ANALYSIS

Estimate of asbestos concentration by PLM according to Part 1 is sufficient

ISO 22262-2 Guidance for Analysis of Sprayed Decorative Coatings (Texture Coats)

APPLICATIONS

Coating of ceilings and walls to provide a textured surface which masks irregularities

TYPICAL COMPOSITIONS

Chrysotile up to 5%

Some compositions may also contain tremolite

RECOMMENDATION FOR ANALYSIS

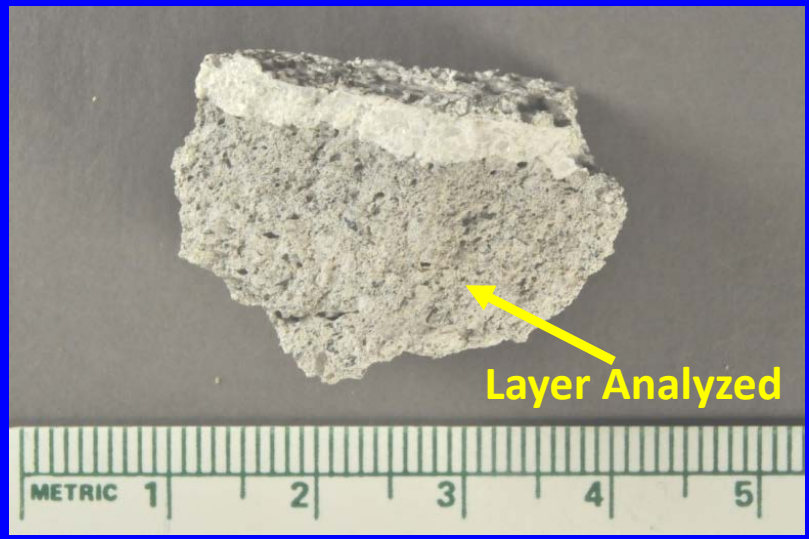
Ashing, treatment with 2M hydrochloric acid and separation of aggregate by flotation or sedimentation is recommended

Quantification of asbestos in the residue can be by PLM point counting, SEM or TEM methods

ASHING, SEDIMENTATION AND ACID EXTRACTION GRAVIMETRY

SAMPLE:	[redacted]	SAMPLE NO:	[redacted]
	Sample RJC140221b, Stucco-Bag	DATE:	2014-03-26
	Grey Cementitious Base Layer	ANALYST:	EJC

INITIAL WEIGHTS			COMMENTS
Weight of Weighing Dish	18.80000		
Weight of Weighing Dish + Sample	20.74570		400 point count of residue is equivalent to a 7200 point count of the original sample.
Weight of Sample	1.94570		
ASHING			
Weight of Crucible	18.80000		
Weight of Crucible + Ash	20.68050		
Weight of Ash	1.88050		
Weight Loss During Ashing	0.06520		
Percent Organic Materials and Water	3.35098		
SEDIMENTATION/FLOTATION			
Weight of Sediment/Floats Container	3.84240		
Weight of Container + Sediment/Floats	5.04510		
Weight of Sediment/Floats	1.20270		
Percent Sediment/Floats	61.81323		
ACID TREATMENT			
Weight of Filter	0.01600		
Weight of Filter + Residue (After Hand-Picking)	0.12470		
Weight of Residue (After Hand-Picking)	0.10870		
Percent Acid-Soluble Materials	29.24911		
Percent Residue (After Hand-Picking)	5.58668		
ASBESTOS	Chrysotile		
Weight of Container for Hand-Picked Asbestos	0.00000		
Weight of Container + Hand-Picked Asbestos	0.00000		
Weight of Hand-Picked Asbestos	0.00000	0.00000	0.00000
Percent Hand-Picked Asbestos	0.00000	0.00000	0.00000
Point Count			
Total Points Counted	400	400	400
Asbestos Points Counted	9		
Point Count Lower 95% Confidence Limit	4.1150	0.0000	0.0000
Point Count Upper 95% Confidence Limit	17.0850	2.9900	2.9900
Asbestos in Sample (Weight Percent)	0.1257	0.0000	0.0000
Lower 95% Weight Concentration Limit	0.0575	0.0000	0.0000
Upper 95% Weight Concentration Limit	0.2386	0.0418	0.0418



Organic = 3,4%
Sediment = 61,8%
Acid Soluble = 29,2%
Residue = 5,6%
Points Counted = 400
Chrysotile Points = 9

Chrysotile Mean Concentration = 0,13%
95% Confidence Limits = (0,05 – 0,24)%
Regulated Control Limit = 0,5%

Gravimetric Analysis Combined With PLM Point Counting Is Very Sensitive

Quantification of Asbestos by X-ray Diffraction

- The Japanese delegation prepared a working draft (WD 22262-3) of a method for quantification of asbestos by x-ray diffraction, based on JIS A 1481:2008.
- It was resolved at the 2009 Working Group meeting in Atlanta to submit this working draft to the TC 146/SC 3 secretariat for voting as a new work item.
- Results for the 21 samples submitted for analysis by the x-ray diffraction method were distributed to the Working Group members.

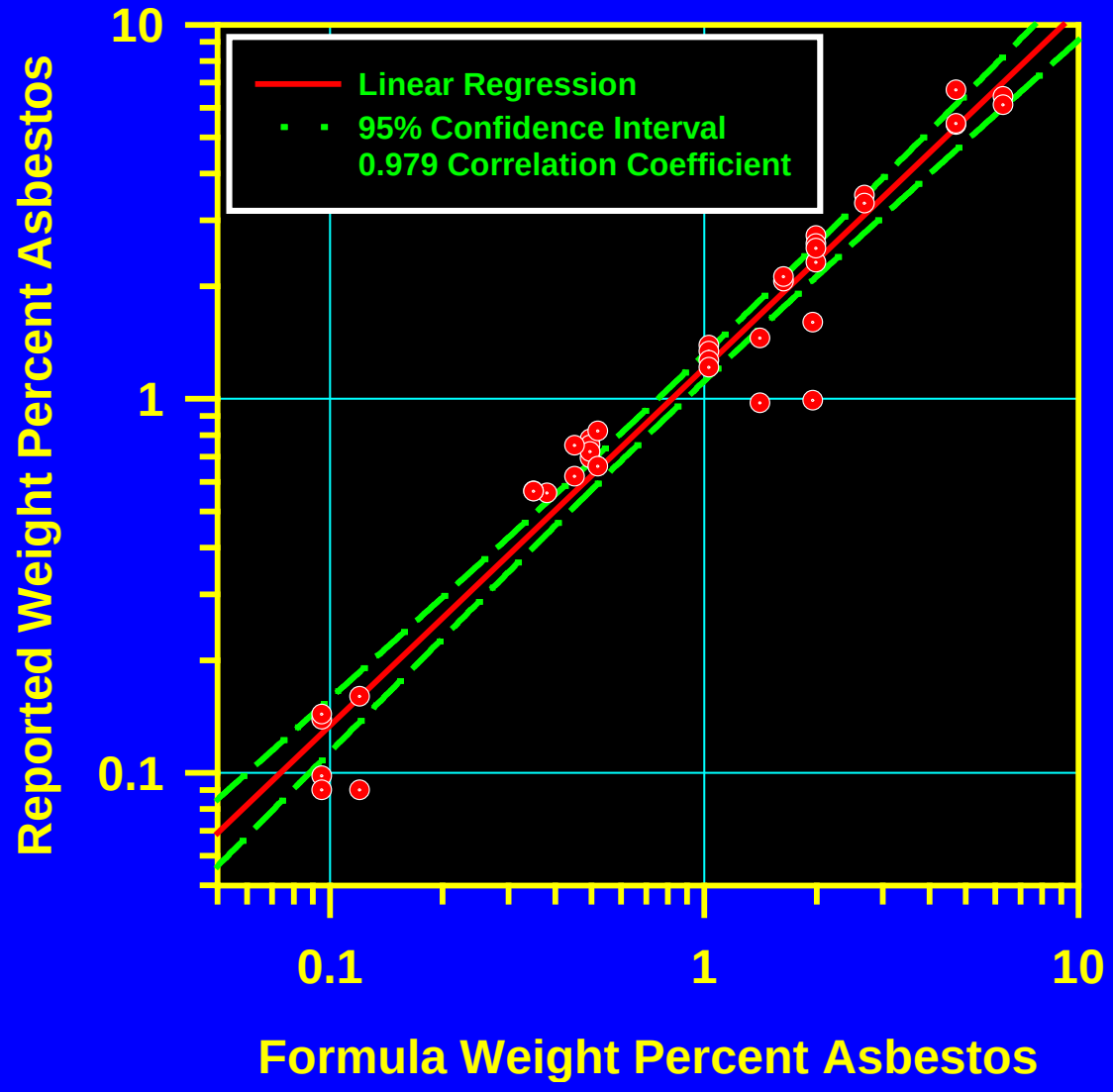
Samples Provided to Demonstrate to the ISO Working Group the Capability of the X-ray Diffraction and PCM Dispersion Staining Method

1) 21 samples provided.

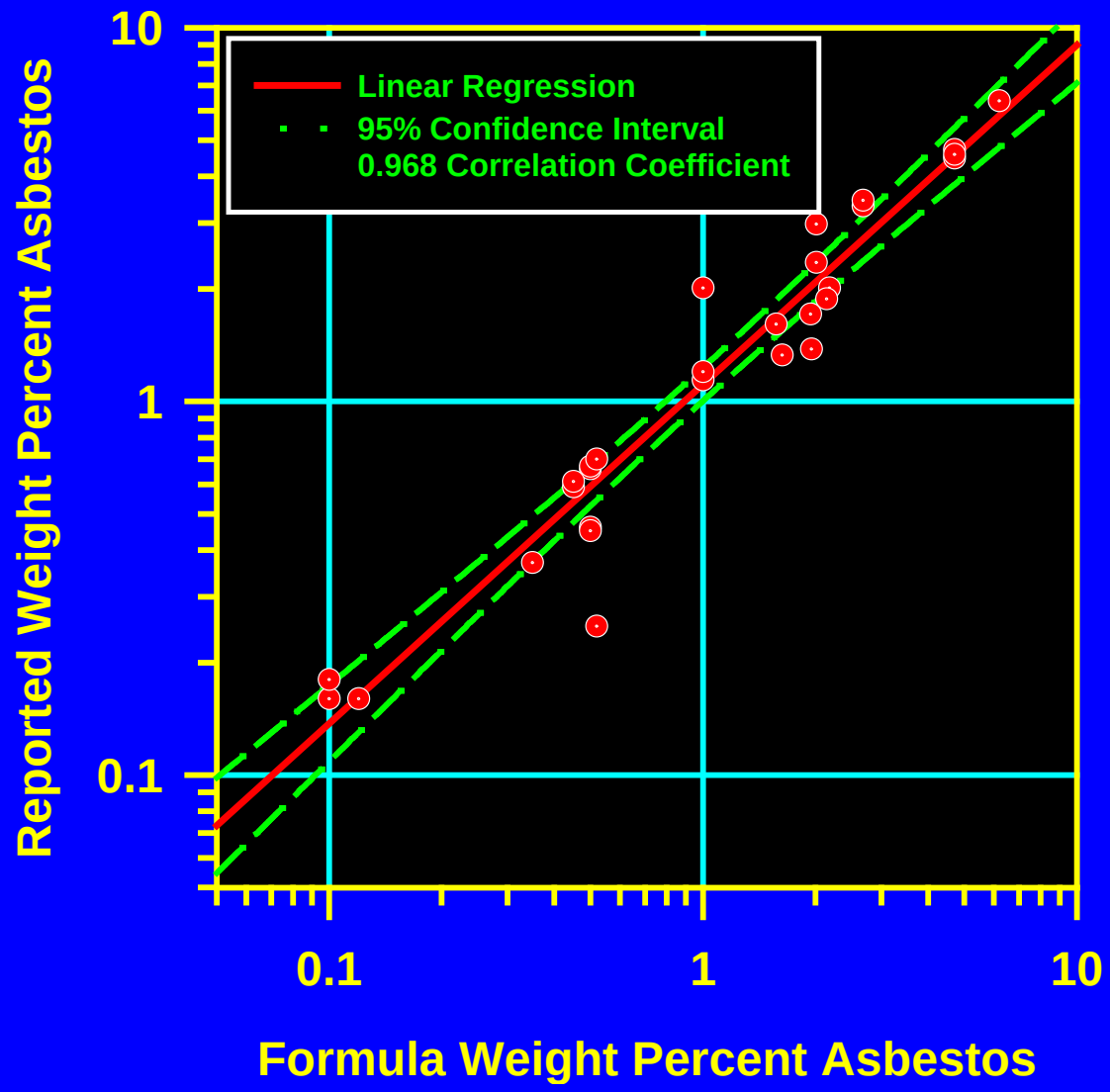
2) None of the samples had asbestos concentrations greater than approximately 5%.

3) Most of the samples had matrices that necessitated the use of gravimetric reduction.

4) All positive samples had previously been analyzed in a blind inter-laboratory study between the Environmental Protection Laboratory of Hong Kong and an analyst at Chatfield Technical Consulting Limited.



Results of Plaster Analyses by Chatfield Technical Consulting Limited



Results of Plaster Analyses by the Environmental Protection Laboratory of Hong Kong

Summary of Results Reported from the X-ray Diffraction and PCM Dispersion Staining Method

1) 15 of 21 final results reported would be considered satisfactory, in that asbestos was reported above 0,1% when asbestos was present in the sample.

2) 5 of 21 samples which had asbestos concentrations between 0,12% and 3,41% were reported as no asbestos detected (false negatives).

3) In 9 of 21 samples, qualitative X-ray diffraction reported asbestos types that were not present (false positives).

4) Three samples contained Coalinga chrysotile, which is the same as the chrysotile standard in JIS A 1481. Concentrations of 0,60% and 3,41% were reported as not detected, and a discrepancy between optical and x-ray results caused a concentration of 4,56% to be reported as >0,1%.

5) PCM with dispersion staining optical microscopy was consistently used to discount the results obtained using qualitative x-ray diffraction.

Analytical Result from XRD - PCM Dispersion Staining Method

Sample	XRD – PCM Dispersion Staining Results			Actual Concentration
	Qualitative Analysis		Quantitative Analysis	
	XRD	PCM-DS	Concentration	
CTC 12	Amosite Crocidolite Tremolite/ Actinolite	None	None	Chrysotile 3,41%

Sample	Constituents	Weight Percent
CTC 12	RG-144 Chrysotile (Coalinga)	3,41
	Gypsum	67,23
	Kaolinite	9,34
	Talc	17,20
	Titanium Dioxide	2,82

Comments by the X-Ray Laboratory on the Analysis of Sample CTC 12

1) Qualitative analysis showed XRD peaks at 10,5° and 12,4°, reported as Amosite, crocidolite and tremolite/actinolite.

2) PCM – DS count – no fibres with dispersion colours were observed in 3000 particles.

3) Quantitative analysis using 12,4° peak gave a result of 21,7% chrysotile.

4) Because of the high concentration result of 21,7%, it was considered "difficult to believe that the 12,4° material peak is chrysotile".

5) The sample was reported as "No Asbestos".

The actual concentration in Sample CTC 12 was 3,41% Coalinga chrysotile, which is the same Coalinga chrysotile that is specified as the reference standard in JIS A 1481.

Analytical Result from XRD - PCM Dispersion Staining Method

Sample	XRD – PCM Dispersion Staining Results			Actual Concentration
	Qualitative Analysis		Quantitative Analysis	
	XRD	PCM-DS	Concentration	
CTC 08	Chrysotile Tremolite/ Actinolite	None	None	Tremolite 0,30%

Sample	Constituents	Weight Percent
CTC 08	Tremolite Asbestos	0,30
	Asphalt with Glass Fibre and Cellulose	68,81
	Sand	15,45
	Calcium Carbonate	15,44

Dispersion Staining Colours for PCM-DS Fibre Count Specified in JIS A 1481:2008

Table 2 Dispersion colour of asbestos

Kind of asbestos	Refractive index $n_D^{25\,°C}$	Dispersion colour
Chrysotile	1.550 [b]	Reddish purple to blue
Amosite	1.680 [b]	Pink
	1.700	Blue
Crocidolite	1.680 [b]	Orange to reddish brown
	1.690 [b]	Pink
	1.700	Blue
Tremolite/ actinolite [a]	1.605	Golden yellow
	1.620 [b]	Reddish purple
	1.640	Blue
Anthophylite	1.605	Golden yellow
	1.618 [b]	Reddish purple
	1.640	Blue

Notes [a] Since it is difficult to discriminate between tremolite and actinolite by the dispersion colour of every refractive index, they are regarded as the same kind in analysis.

[b] It is the refractive index indicating the sensitive colour of asbestos respectively.

Comparison of Asbestos Fibre Identification Procedures Specified in WD 22262-3 and ISO 22262-1

WD 22262-3:2011 (JIS A 1481:2008)

1) "Dispersion Colour"

For chrysotile: Reddish Purple to blue

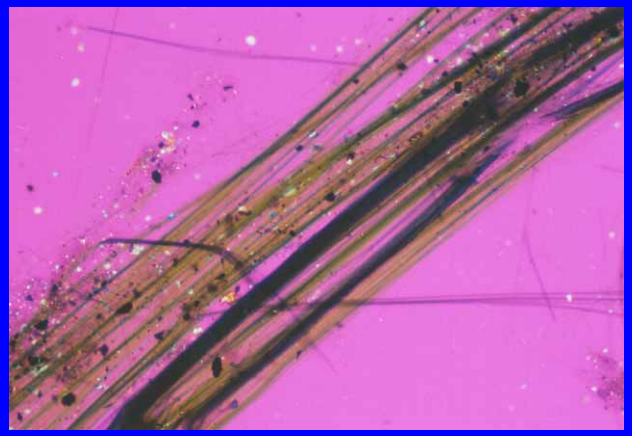
No requirement to determine any other optical properties of chrysotile, or even if the fibres are birefringent.

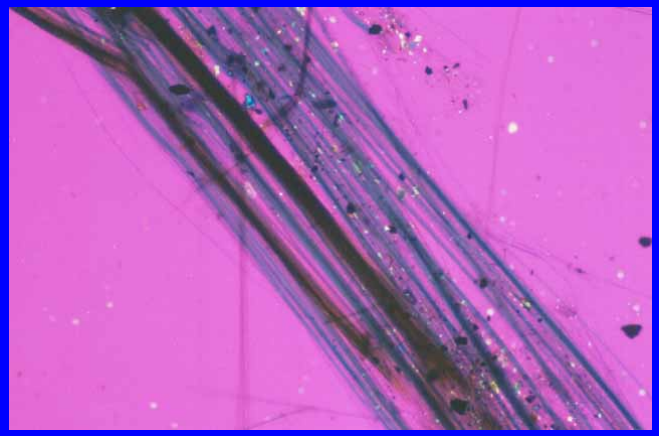
Many fibres other than chrysotile exhibit this range of dispersion colours. Comminution also inhibits identification of fibres by DS.

ISO 22262-1:2012

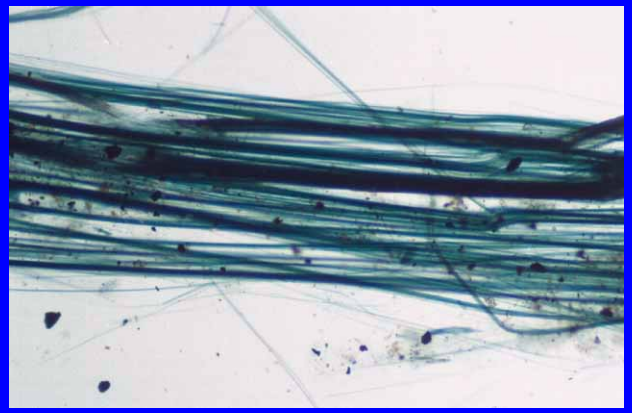
1) Colour of the fibres
2) Birefringence
3) Sign of elongation
4) Pleochroism
5) Parallel DS match λ_o
6) Perpendicular DS match λ_o

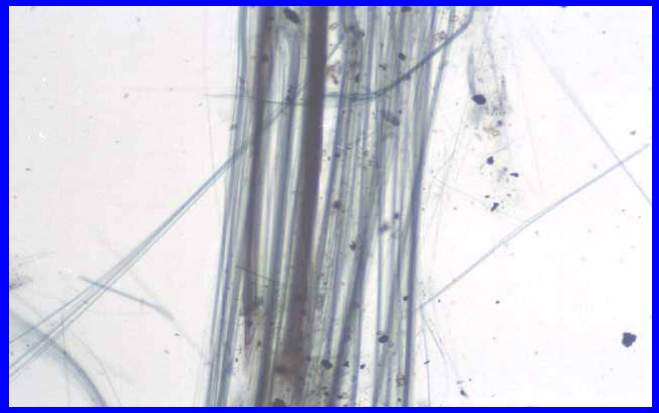
Negative Sign of Elongation





Blue – Grey Pleochroism





Polarizer Direction

Identification of Crocidolite
Important Observations Required by ISO 22262-1

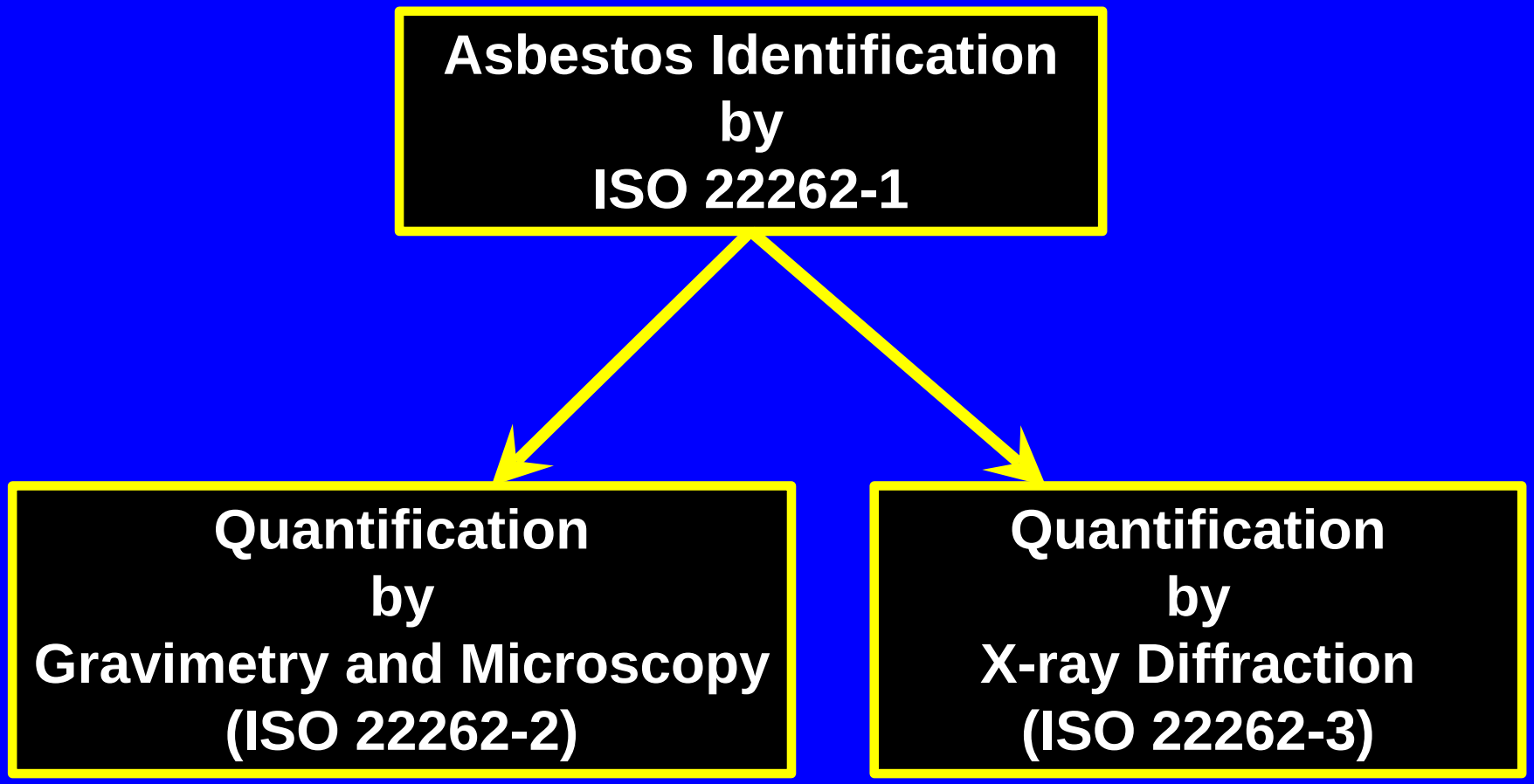
ISO Working Draft WD 22262-3 based on JIS A 1481:2008 was rejected by the Working Group in Vienna on 2011-09-25

- **The resolutions of the Working Group:**
  - **"Based on the reviews by technical experts, however, Working Group 1 recommends to SC3 that work on the present proposal be discontinued".**
  - **"At the present stage the WG regards the method as insufficiently developed for an ISO Standard for reliable detection and quantification of lower levels of asbestos in variable matrices".**
  - **"If technical problems are resolved it is recommended that a new work item could be considered".**

Reasons for Rejection of the WD 22262-3 Draft that was Based on JIS A 1481:2008

- The PCM-dispersion staining optical microscopy procedure specified in JIS A 1481:2008 and WD 22262-3 for identification of asbestos fibres was scientifically unacceptable to the Working Group.
- The Working Group also considered that there were numerous other technical deficiencies in the draft, including:
  1) There was no discussion of discrimination between asbestos and non-asbestiform analogues.
  2) The effect of interferences by minerals commonly present in building materials was not included.
  3) Initial pulverization of the sample inhibits the ability to identify asbestos fibres by optical microscopy methods.
  4) The overall method for detection and identification of asbestos fibres was unnecessarily complex and appeared to be unreliable.

Working Group Meeting, Vienna, 2011-09-26
New Proposal by the Japanese Delegation



A New Work Item Proposed on 2011-09-26 by the Japanese Delegation was Accepted by ISO/TC 146/SC 3/WG1

- **"WG1 requires that ISO 22262-1 will be used normatively prior to any subsequent quantification by microscopy or XRD".**
- **"WG1 understands that XRD quantifies mineral species, without regard to whether they are asbestiform or non-asbestiform".**
- **"WG1 understands that the new draft will include a full discussion of the limitations of the XRD method, including interferences and variability of calibration standards".**
- **"Determination of the asbestos content must depend on the microscopical determination of asbestos fibres as already identified in ISO 22262-1".**

Proposed ISO 22262-3

1) The method is based on gravimetric reduction using ashing and formic acid treatment, followed by x-ray diffraction.

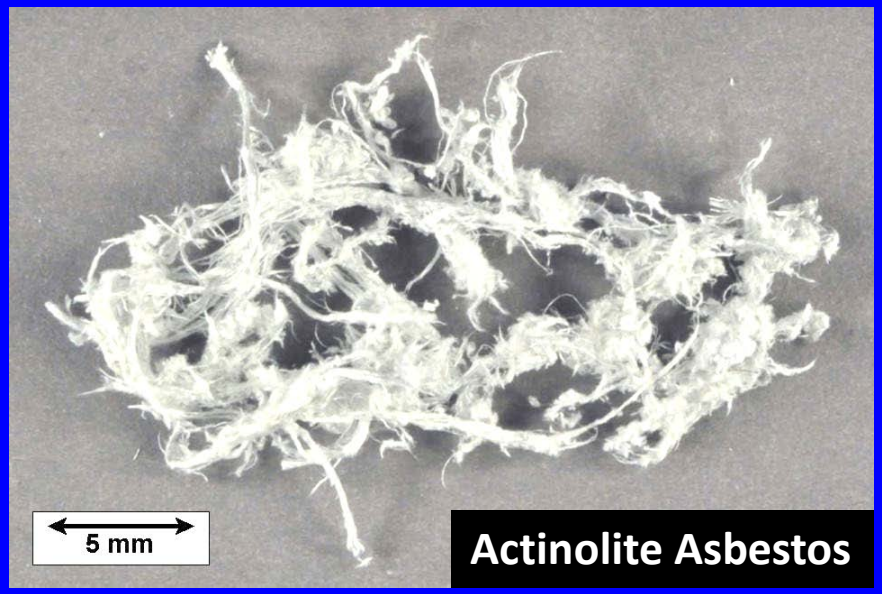
2) The Limit of Detection (LOD) and the Limit of Quantification (LOQ) have been determined for chrysotile in a non-interfering matrix (feldspar).

   - If, for example, the residue from gravimetric reduction is 10% of the original weight, the LOD is 0,01% and the limit of quantification LOQ is 0,03%.

   - If gravimetric reduction is not effective, the LOD is 0,1% and the LOQ is 0,3%.

3) The actual LOD and LOQ that can be achieved depend on a number of sample-dependent factors, such as interfering peaks and use of secondary peaks for measurement.

4) There is no discrimination between amphibole asbestos and non-asbestiform amphibole.



X-ray Diffraction Determines the Concentration of the Mineral Species.
It Does Not Specifically Determine Asbestos.

Vote on DIS 22262-3, 2015-08-11

Result of voting

P-Members voting: 8 in favour out of 11 = 73 % (requirement >= 66.66%)

(P-Members having abstained are not counted in this vote.)

Member bodies voting: 3 negative votes out of 12 = 25 % (requirement <= 25%)

Approved

Negative votes were cast by the U.S.A., the U.K. and Canada.
The negative votes cited a number of unresolved technical issues.

All remaining technical issues were resolved at the 2015 meeting in Delft by incorporating technical changes.

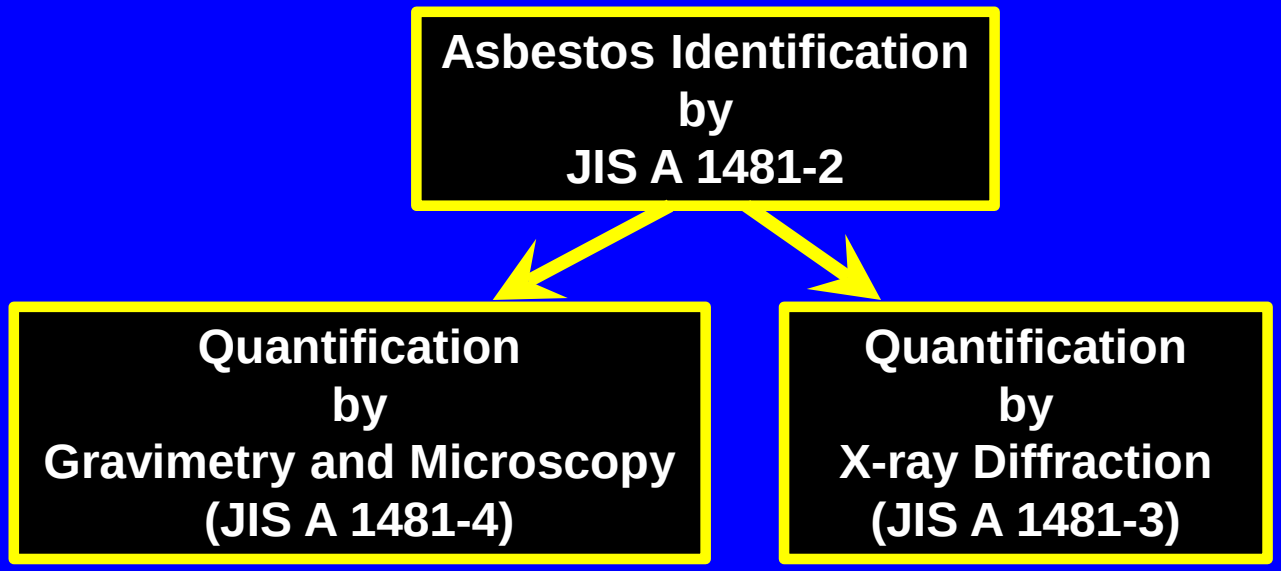
The document was also extensively edited prior to FDIS voting.

Concerns Regarding the Comparability of ISO 22262 with JIS A 1481 Were Clarified by the Japanese Delegation at the ISO/TC 146/SC 3 Plenary meeting in Burlington on 2016-09-29

Number	JIS Title	Assurances Given by the Japanese Delegation
JIS A 1481-1:2016	Air quality – Bulk materials - Part 1: Sampling and qualitative determination of asbestos in commercial bulk materials	It is identical to ISO 22262-1:2012
JIS A 1481-2:2016	Determination of asbestos in building material products – Part 2: Sampling and qualitative analysis for judgement of existence of containing asbestos.	
JIS A 1481-3:2016	Determination of asbestos in building material products - Part 3: Quantitative analysis of containing asbestos by x-ray diffraction method	It is identical to FDIS 22262-3 (Now ISO 22262-3:2016)
JIS A 1481-4:2016	Air quality – Bulk materials – Part 4: Quantitative determination of asbestos by gravimetric and microscopical methods	It is identical to ISO 22262-2:2014

The Asbestos Identification procedure in JIS A 1481-2 (XRD PLM-DS) was rejected by ISO/TC 146/SC 3/WG1 in 2011

Use of any National Standard is a domestic matter of no concern to ISO.



It is important to recognize that analyses conducted according to the above flow chart are not consistent or compliant with ISO 22262.

INTERNATIONAL STANDARD

ISO 22262-3

First edition
2016-10-31

Air quality — Bulk materials —

Part 3: Quantitative determination of asbestos by X-ray diffraction method

Qualité de l'air — Matériaux solides —

Partie 3: Dosage quantitatif de l'amiante par la méthode de diffraction des rayons X

ISO 22262-3 Was Published in 2016

All 3 Parts of ISO 22262 Have Now Been Published

- **Identification of asbestos by ISO 22262-1 is a mandatory requirement of the ISO standard.**
- **Depending on the regulatory limit, quantification of asbestos beyond the estimate obtained using ISO 22262-1, may not be necessary.**
- **Following examination by ISO 22262-1, any necessary further quantification of asbestos may be performed using either the gravimetric and microscopical methods of ISO 22262-2 or the gravimetric and x-ray diffraction methods of ISO 22262-3.**

Trillium grandiflorum