

## **Annex: Information on the measurement method(s) for asbestos and difficulties measuring asbestos in relation to a low occupational exposure limit value (OELV) or in a dusty (work) environment in view of lowering the European OELV for asbestos.**

### **Introduction**

The European Parliament recently submitted a proposal to lower the European occupational exposure limit value (OELV) for asbestos from 100,000 fibres/m<sup>3</sup> to 1,000 (asbestos) fibres/m<sup>3</sup>. Discussions are now underway at the European level about the feasibility of lowering this European OELV. An important part of this discussion concerns the different measurement and analytical methods that are used in Europe for determining (asbestos) fibre concentrations.

After advice of the Health Council of The Netherlands on the exposure-risk relationship<sup>1</sup> of environmental and occupational exposure to asbestos,<sup>2</sup> and advice on the feasibility of The Netherlands Socio-Economic Council, in the Netherlands currently the national OELV for asbestos (both chrysotile and amphibole asbestos) is 2,000 asbestos fibres/m<sup>3</sup>. The Dutch legislation does not specify the measurement and analytical technique to be used when determining occupational exposure to asbestos, but in practice scanning electron microscopy (SEM) in combination with Energy Dispersive X-ray analysis (EDXA) is used according to the method as specified in standard ISO 14966.<sup>3</sup> Transmission Electron Microscopy (TEM) and Phase Contrast Microscopy (PCM) are not used in the Netherlands on a regular basis for analysis of asbestos exposure.

The Netherlands Ministry of Social Affairs and Employability has asked TNO to provide information and an evaluation of relevant topics in this discussion, based on common practice in the Netherlands.

Measuring exposure to asbestos, analysing samples and comparing it to an occupational exposure limit value (OELV) is not a straightforward and easy task. In this evaluation a comparison of analytical methods is first described. Next, an explanation is given on the sampling of asbestos to measure exposure and compare to an OELV for compliance, as is standardised in EN 689. It is important to realise that sampling method, number of measurements, and the analytical method used are interconnected to arrive at a specific limit of detection and have an effect on the determination of compliance to the OELV. Factors of influence in the sampling method are air flow through the sampling pump, filter use and duration of sampling. The practical implementation of the analytical method also influences the limit of detection. Several issues and difficulties are elaborated and some developments in innovation presented.

---

<sup>1</sup> The Health Council of The Netherlands calculates the risks for a predefined risk level of  $1 \times 10^{-4}$  per year (acceptable level) and  $1 \times 10^{-6}$  per year (target level).

<sup>2</sup> Health Council of the Netherlands. Asbestos: Risks of environmental and occupational exposure. The Hague: Health Council of the Netherlands, 2010; publication no. 2010/10.

<sup>3</sup> ISO 14966:2019 en. Ambient air - Determination of numerical concentration of inorganic fibrous particles - Scanning electron microscopy method. 1 December 2019.

## Comparison of analytical methods for asbestos

The information as provided below is based on the general technical specifications of the three most commonly used analytical techniques for measuring exposure to asbestos (Tromp & Tempelman, 2016).<sup>4</sup> In Table 1 below the information about the three most commonly used methods for measuring asbestos is summarized.

**Table 1:** Characteristics of commonly used analytical methods for measuring asbestos

Parameter	PCM	SEM (EDXA)	TEM (SAED & EDXA)
Filter	Membrane filter of mixed esters of cellulose or cellulose nitrate, pore size 0.8 to 1.2 $\mu\text{m}$	Gold-coated capillary-pore polycarbonate filters, maximum nominal pore size 0.8 $\mu\text{m}$	Membrane filter of mixed esters or cellulose, capillary-pore polycarbonate filter
Distinguish between chrysotile and amphibole asbestos fibres and other fibres	No	Yes	Yes
Lower limit of visibility (resolution) *	0.2 - 0.25 $\mu\text{m}$	Approx. 0.2 $\mu\text{m}$ (regular SEM) Approx. 0.1 $\mu\text{m}$ (high-resolution SEM)	Approx. 0.01 $\mu\text{m}$
Limit of detection **	approx. 2,000 fibres/ $\text{m}^3$ ***	approx. 100 - 200 fibres/ $\text{m}^3$	approx. 1,000 fibres/ $\text{m}^3$
Preparation of samples	Simple	Very simple	Complex and time consuming
Counting rules	WHO (>5 $\mu\text{m}$ length and <3 $\mu\text{m}$ width; aspect ratio 3:1)	ISO 14966: 2019 (>5 $\mu\text{m}$ length and <3 $\mu\text{m}$ width; aspect ratio 3:1)	ISO 10312 / ISO 13794 (aspect ratio 5:1, minimum length 0.5 $\mu\text{m}$ )

Phase Contrast Microscopy (PCM), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Energy Dispersive X-ray analysis (EDXA), Selected Area Electron Diffraction (SAED), World Health Organisation (WHO), International Organisation for Standardisation (ISO).

\* Resolution: thinnest visible fibre with standard microscope settings for fibre counting.

\*\* Limit of detection: the lowest concentration that can be measured with 90% certainty (based on Poisson distribution) with standard microscope settings for fibre counting.

\*\*\* The limit of detection of PCM is, in theory, approximately 2,000 fibres/ $\text{m}^3$ , but in practice measuring asbestos fibre concentrations lower than approximately 5,000 fibres/ $\text{m}^3$  is not suitable due to the presence of other fibres.

### Phase contrast microscopy (PCM)

With a pump a known amount of air is drawn through a filter (membrane filter, cellulose ester). The filter is placed on a slide and partially dissolved with warm acetone vapor, making it transparent. After applying a drop of glycerol triacetate (refractive index 1.43) a cover slip is applied on the filter. After about 10 minutes, the prepared filter can be viewed under the Phase Contrast Microscope. By default, 100 graticule image fields are searched for fibres, after which the number of fibres per (complete) filter

<sup>4</sup> Tromp PC, Tempelman J. Bepaling van de vezelconcentratie in de lucht na asbestsanering. Onderzoek naar de geschiktheid van licht- en elektronenmicroscopische bepalingsmethoden bij de eindcontrole na een asbestsanering. TNO rapport TNO 2016 R10496-A versie, 13 juni 2016.

is calculated. The fibre concentration is determined by dividing the number of fibres per filter by the sampled volume of air.

With PCM it is NOT possible to distinguish between asbestos fibres and non-asbestos fibres, the concentration applies to the total concentration of fibrous particles. This means that in addition to asbestos also mineral wool, paper, skin flakes, plant residues, textiles, etc. As a result, the concentration of fibres as counted with PCM will often be higher than the concentration of asbestos fibres counted with SEM or TEM. This applies in particular to (remediation) activities of materials that also contain many other fibrous materials in the matrix (for example floor tarpaulin and cardboard). On the other hand, the limited resolution of PCM can also lead to an underestimation of the asbestos concentration in air. This applies in particular to the asbestos types chrysotile and crocidolite, which may contain fibres thinner than 0.20 µm.

In theory, the limit of detection of PCM is approximately 2,000 fibres/m<sup>3</sup>, but in practice, PCM is not suitable for measuring asbestos fibre concentrations lower than approximately 5,000 fibres/m<sup>3</sup>. As PCM is a non-specific method, the limit of detection is limited by the “background noise” of (non-asbestos) organic and non-organic fibrous components that are always present in the air. Especially at low asbestos fibre concentrations, the presence of other fibrous material plays a major role.

#### Pros PCM:

- Fast (preparation and analysis);
- Cheap;
- Transportable (analysis can be performed at the measuring location).

#### Cons PCM:

- No identification of fibres, non-specific;
- High limit of detection, in theory 2,000 and in practice 5,000 fibres/m<sup>3</sup>;
- Thin fibres (diameter <0.20 - 0.25 µm) are not detected.

### **Scanning Electron Microscopy (SEM)**

With a pump a known amount of air is drawn through a filter (polycarbonate core pore filter with a gold coating). The filter is then applied directly to a SEM table/stub and can be viewed in the SEM without further preparation. By default, 100 image fields are scanned at 2,000x magnification. An element spectrum of the fibres found is recorded with EDXA. On the basis of this spectrum, asbestos fibres can be identified, after which the number of asbestos fibres per filter is calculated. The fibre concentration is determined by dividing the number of asbestos fibres per filter by the sampled volume of air. The method is described in detail in the standard ISO 14966. Transportable SEM equipment (table top) is available, and can be set up in a measuring vehicle.

#### Pros SEM:

- Identification of asbestos fibres with EDXA;
- Low limit of detection possible (100-200 fibres/m<sup>3</sup>);
- Easy preparation (low probability of errors during preparation);
- Good performance characteristics (validation, proficiency testing within EU).

#### Cons SEM:

- Extensive facilities required to make a SEM transportable;
- Much more expensive than analysis with PCM;

- Requires a higher level of education from the analyst.

### **Transmission Electron Microscopy (TEM) - direct and indirect method**

With a pump a known amount of air is drawn through a filter (direct method: polycarbonate core pore filter without gold coating; indirect method: cellulose ester membrane filter). With the direct method, a carbon coating is evaporated under high vacuum. The filter is dissolved in e.g. chloroform, after which a carbon fleece containing the particles is placed on a gauze with a diameter of 3 mm. The thin carbon fleece is transparent to the electron beam (80-100 kV) and suitable for viewing in the TEM at 10,000 to 40,000x magnification. With the indirect method, the filter is first incinerated to remove the filter and all organic components (pre-concentration). After possible further concentration (acid treatment), the ash residue is again ultrasonically suspended in water and filtered over a core pore filter. Otherwise, the procedure is identical to that of the direct method. An element spectrum of fibres found is recorded with EDXA. This spectrum can be used to identify asbestos fibres. As a second analysis technique, a TEM can use Selected Area Electron Diffraction (SAED). The specific crystal structure with which asbestos fibres can be unambiguously identified can be determined based on the diffraction pattern. After counting a known surface area, the number of asbestos fibres per filter can be calculated. The fibre concentration is determined by dividing the number of asbestos fibres per filter by the sampled volume of air. The method is described in detail in the standards ISO 10312 (TEM direct method)<sup>5</sup> and ISO 13794 (TEM indirect method)<sup>6</sup>.

#### Pros TEM:

- Identification of asbestos fibres with EDXA and/or SAED;
- Also thinnest ( $\approx 0.02 \mu\text{m}$ ) asbestos fibres are visible.

#### Cons TEM:

- Not transportable
- Considerably more expensive than analysis with SEM or PCM;
- Sample preparation is complex (many actions), high risk of errors or contamination of the sample;
- Limit of detection relatively high (1,000-2,000 fibres/m<sup>3</sup>);
- Performance characteristics less than SEM due to small sample size and sample preparation;
- Application of SAED requires professional knowledge at an academic level (mineralogy / crystallography).

## **Comparison of analytical methods for asbestos in order to demonstrate compliance with OELV**

Checking for compliance of exposure with an OELV is not straightforward, it requires a number of exposure measurements, with a specific certainty and statistical test, and is also dependent on the analytical method used. This is explained in this section with an introduction in the exposure measurement strategy, and summarized in table 2.

---

<sup>5</sup> ISO 10312:2019 en. Ambient air - Determination of asbestos fibres - Direct transfer transmission electron microscopy method. 1 October 2019.

<sup>6</sup> ISO 13794:2019 en. Ambient air - Determination of asbestos fibres - Indirect-transfer transmission electron microscopy method. 1 October 2019.

In the European standard EN 689 a strategy to perform representative measurements of exposure by inhalation to chemical agents in order to demonstrate the compliance with OEL values (OELVs) is specified.<sup>7</sup> In this standard it is described how measurement results (measured concentration levels) should be compared with an OELV, which depends on the number of measurements collected for a similar exposure group (SEG).

**Table 2:** Overview of analytical techniques that can be used depending on the limit values used for compliance testing based on number of exposure measurements collected and OELV

Type of compliance test (EN 689)	Number of exposure measurements collected	Limit values for compliance testing		
		OELV 2,000 f/m <sup>3</sup>	OELV 10,000 f/m <sup>3</sup>	OELV 100,000 f/m <sup>3</sup>
Preliminary test	3 measurements (10% OELV)	200	1,000	10,000
	4 measurements (15% OELV)	300	1,500	15,000
	5 measurements (20% OELV)	400	2,000	20,000
Statistical test	≥6 measurements (100% OELV)	2,000	10,000	10,000
Green: all analytical techniques can be applied (PCM, SEM, TEM)				
Blue: only EM techniques can be applied (SEM, TEM)				
Yellow: only SEM can be applied				

When only a limited number of measurements (3-5) is collected the possible degree of variation in occurring exposure levels can only be mapped to a limited extent. In that case a preliminary comparison test is performed in which a 'safety factor' is applied. When 6 or more measurements are collected a statistical test can be performed to compare exposure levels of the SEG with the OELV. In this statistical test variation in exposure is taken into account. This statistical test determines, with at least 70% confidence, whether less than 5% of the exposures in the SEG exceed the OELV.

In the case of a preliminary test the following comparison is performed:

- If all results are below 0,1-0,2x the OELV it is considered the OELV is not exceeded (compliance):
  - o 0.10 OELV for a set of 3 exposure measurements;
  - o 0.15 OELV for a set of 4 exposure measurements;
  - o 0.20 OELV for a set of 5 exposure measurements;
- If one of the results is higher than the OELV, it is considered that the OELV is exceeded (non-compliance);
- If all the results are below the OELV and one result is above 0,1 OELV (set of three results) or 0,15 OELV (set of four results) or 0,2 OELV (set of five results) it is not possible to conclude on compliance with the OELV (No-decision). In this situation additional exposure measurements shall be carried out (requiring at least a total of six measurements) in order to apply the statistical test based on the calculation of the confidence interval of the probability of exceeding the OELV.

Based on the methods of comparison with an OELV (depending on the number of measurements available) and the characteristics of the different analytical techniques as described above (depending

<sup>7</sup> EN 689 :2018+C1:2019 en. Workplace exposure - Measurement of exposure by inhalation to chemical agents - Strategy for testing compliance with occupational exposure limit values.

mainly on the limit of detection), an overview is given of which analytical techniques can be applied when different OELVs (100,000, 10,000 and 2,000 fibres/m<sup>3</sup>) are considered (see Table 2).

Measurement results based on analysis with **PCM** can be used for compliance testing in case of the current European OELV of 100,000 fibres/m<sup>3</sup>, but due to the relatively high limit of detection can only be used for compliance testing with an OELV of 10,000 fibres/m<sup>3</sup> if 6 or more measurements are collected and thus the statistical test can be performed. In case of an OELV of 2,000 fibres/m<sup>3</sup> PCM cannot be used.

Measurement results based on analysis with **TEM** can be used for compliance testing in case of the current European OELV of 100,000 fibres/m<sup>3</sup> or an OELV of 10,000 fibres/m<sup>3</sup>. However, due to the still relatively high limit of detection, TEM can only be used for compliance testing with an OELV of 2,000 fibres/m<sup>3</sup> if 6 or more measurements are collected and thus the statistical test can be performed.

Measurement results based on analysis with **SEM** can be used for compliance testing in case of OELVs of 100,000 fibres/m<sup>3</sup>, 10,000 fibres/m<sup>3</sup> or 2,000 fibres/m<sup>3</sup>.

## **Possible issues with regard to the limit of detection for analysis with SEM in view of comparing measurement results with OELV, at low exposure activities**

The limit of detection of the SEM method with regard to exposure measurements depends on several factors in the 'chain of events'. Starting with the use and settings of equipment to sample (such as air flow, filter types) until the actual analysis with SEM (counting rules). This is elaborated in this section.

In the standard for an analytical technique generally criteria are described that must be met to be able to apply that technique in a reliable way, including counting rules and sampling requirements. As in the Netherlands only SEM is being used for measuring exposure to asbestos, and TNO mainly has practical experience with SEM, this evaluation is limited to SEM according to standard ISO 14966.

An analytical result always consists of a nominal value with a lower and upper confidence limit. For ALL microscopic techniques that are used to analyse the amount of asbestos on a filter, a certain number of image fields is being searched by an analyst, after which that result is extrapolated to the filter as a whole.<sup>8</sup> This extrapolation introduces some degree of uncertainty, which is expressed as the 90% confidence interval based on the Poisson distribution around the nominal value. The accuracy of the analysis amongst others depends on the number of image fields of the filter being searched and the volume of air that is drawn through the filter during sampling. These factors also determine the limit of detection of a particular analysis. The volume of air is in turn dependent on the flow (litres of air per minute) of the sampling pump and the sampling duration (minutes). The higher the number of image fields that is counted and/or the higher the volume of air that is drawn through the filter, the lower the limit of detection will be and the smaller the confidence interval around a nominal value. A summary is given in Table 3 and explained in detail below.

---

<sup>8</sup> The effective surface area of a 25 mm filter is 314 mm<sup>2</sup> based on an effective diameter of 20 mm. In practice most often disposable plastic samplers are used, with an effective diameter of 22 mm, resulting in an effective surface area of a 25 mm filter is 380 mm<sup>2</sup>. However, TNO also uses reusable aluminum samplers, with an effective diameter of 20 mm.

**Table 3:** Applicability of SEM for testing compliance with two OELVs (10,000 and 2,000 fibres/m<sup>3</sup>) depending on the limit of detection as defined by sampling characteristics (flow and sampling duration) and the surface area of the filter that is analysed

Sampling characteristics			Limits of detection (in f/m <sup>3</sup> ) with varying surface areas (in mm <sup>2</sup> ) being analysed							
Flow (L/min)	Sampling duration (hours)	Volume air (m <sup>3</sup> )	OELV 2,000 f/m <sup>3</sup>				OELV 10,000 f/m <sup>3</sup>			
			1 mm <sup>2</sup> *	2.5 mm <sup>2</sup> **	5 mm <sup>2</sup>	10 mm <sup>2</sup>	1 mm <sup>2</sup> *	2.5 mm <sup>2</sup> **	5 mm <sup>2</sup>	10 mm <sup>2</sup>
8	1	0.48	2400	950	480	240	2400	950	480	240
	2	0.96	1200	480	240	120	1200	480	240	120
	4	1.92	590	240	120	60	590	240	120	60
	8	3.84	290	120	60	30	290	120	60	30
4	2	0.48	2400	950	480	240	2400	950	480	240
	4	0.96	1200	480	240	120	1200	480	240	120
	8	1.92	590	240	120	60	590	240	120	60
2	2	0.24	4700	1900	950	480	4700	1900	950	480
	4	0.48	2400	950	480	240	2400	950	480	240
	8	0.96	1200	480	240	120	1200	480	240	120
Green: Both preliminary (3-5 measurements) and statistical test (≥ 6 measurements) for compliance with OELV possible (based on EN 689)										
Orange: Only statistical test (≥ 6 measurements) for compliance with OELV possible (based on EN 689)										
Red: No testing of compliance with OELV possible (based on EN 689)										

\* Minimal filter surface area to be analysed as recommended in ISO 14966

\*\* Minimal filter surface area commonly applied by TNO

With regard to the counting rules, ISO 14966 recommends that a minimum of 1 mm<sup>2</sup> of the surface area of the filter (0.3% of the filter) is analysed. In addition, it is prescribed that the maximum surface area of an image field is 0.025 mm<sup>2</sup>, a minimum of 50 image fields or 50 fibres should be counted, and that the minimum analysed filter area is 0.25 mm<sup>2</sup>. It is always possible to customise the analytical method counting more image fields to meet specific requirements. However, it should be noted that as a general rule of thumb it is recommended that in the context of limiting the analysis uncertainty, a maximum of 1,000 image fields is counted per filter. The counting of image fields is performed by an analyst, and the more image fields that need to be searched, the more room for error due to e.g. fatigue exists.

To measure exposure to asbestos of workers during a regular working day, generally a sampling duration of 6-8 hours is applied. However, in case the work with asbestos is repetitive and/or only takes a short amount of time, the sampling duration can be shorter. The flow of a pump depends on the type of pump that is used. The portable pumps that are generally used to measure personal exposure to substances commonly provide a flow of 2 L/min. There are special portable personal pumps available that can provide a flow up to 4-6 L/min, but these are not often used. In addition high volume non-portable (stationary) pumps are available that operate at a flow of 8 L/min. In the Dutch

standard NEN 2939<sup>9</sup> for measuring personal exposure to asbestos fibres a limit of detection of minimal 10% of the OELV (200 fibres/m<sup>3</sup>) is recommended, and it is prescribed that a minimum of 100 image fields are counted. In addition, for short-term tasks a minimal sampling duration of 1 hour is recommended. In the Netherlands, high volume non-portable pumps are most often used in exposure studies, to be able to reach a flow of 8 L/min, especially in case of short-term tasks. In that case a long flexible hose is mounted on the pump for the worker to be able to perform his/her tasks without being hindered by the pump.

In case of low exposure activities, the expected exposure to asbestos is assumed to be (far) below the OELV. To be able to compare the results of exposure measurements during low exposure activities with an OELV, it is very important that the limit of detection of the analysis is lower than the OELV. Analysis of a filter with a result below the limit of detection means that no fibres were detected on the part of the filter that was analysed. During the analysis of a filter with SEM only a relatively small part of the filter is actually analysed; if no fibres are detected during the analysis with SEM, the upper 95 % confidence limit is 2.99 fibres (according to ISO 14966). As mentioned before, the limit of detection of analysis with SEM depends on the volume that is drawn over the filter and the surface area of the filter that is being analysed. In Table 3 an overview is given of the limit of detection based on different combinations of flow (related to the different types of pumps that are used) and sampling duration as well as different surface areas of the filter being analysed. In addition, the colours indicate whether the limit of detection resulting from a particular combination of sampling characteristics and surface area analysed is suitable for comparison with two different OELVs (10,000 and 2,000 fibres/m<sup>3</sup>) in accordance with the principles of NEN 689.

When an OELV of 10,000 fibres/m<sup>3</sup> is considered, the limits of detections that can be achieved based on the different combinations of sampling characteristics and the recommended minimal surface area to be analysed as stated in ISO 14966 (1 mm<sup>2</sup>) are almost always low enough to compare with the OELV. The only exception is situations in which a personal pump is applied in combination with a short sampling duration. If in those cases the exposure levels are below the limit of detection at least 6 measurements should be collected to be able to compare the measurements results with the OELV.

When an OELV of 2,000 fibres/m<sup>3</sup> is considered, in many cases a considerable number of image fields needs to be analysed to make sure that the limit of detection is low enough. The recommended minimal surface area to be analysed as stated in ISO 14966 (1 mm<sup>2</sup>) will not be sufficient in many cases to be able to perform a (preliminary) test. In case a portable pump is used, with a flow of 2-4 L/min, in many cases a long sampling duration and/or a considerable analytical effort is necessary to be able to compare the measurement results with the OELV.

Thus, the choice of the limit of detection of an analysis is crucial when comparing measurement results with an OELV, and more or less determines the sampling and analytical efforts that should be made. In the Netherlands the limit of detection for exposure measurements is 200 fibres/m<sup>3</sup>. As is shown in Table 4, when that limit of detection is taken into account (which depends on the volume of air that is sampled could mean that a considerable amount of image fields need to be analysed), counting a few asbestos fibres during the analysis will not result in exceedance of the OELV right away. However, it

---

<sup>9</sup> NEN 2939: 2<sup>nd</sup> draft. Workplace air - Measurement of respirable asbestos fibre concentrations of workers handling asbestos or asbestos containing materials, using scanning electron microscopy and X-ray micro analysis [will soon become available].

should be noted that in practice when asbestos fibres are found during the analysis the number of image fields that is actually counted is often reduced, which will result in a higher degree of uncertainty in the measured asbestos fibre concentration.<sup>10</sup>

**Table 4:** Overview of effect of number of counted fibres on testing compliance with OELV of 2,000 fibres/m<sup>3</sup>, based on a limit of detection of 200 fibres/m<sup>3</sup> (10% of OELV) and a minimum of 100 image fields analysed, as recommended in NEN 2939

Flow (L/min)	Sampling duration (hours)	Volume (m <sup>3</sup> )	Filter area (and number of image fields *) to be analysed to obtain LOD of 200 fibres/m <sup>3</sup>	Number of counted fibres and resulting fibre concentration (fibres/m <sup>3</sup> ) **						
				0	1	3	5	10	20	30
8	1	0.48	10 mm <sup>2</sup> (800 image fields)	< 200	66	200	330	660	1300	2000
8	2	0.96	5 mm <sup>2</sup> (400 image fields)							
8	4	1.92	2,5 mm <sup>2</sup> (200 image fields)							
8	8	3.84	1,2 mm <sup>2</sup> (100 image fields)							
Green: compliance with OELV of 2,000 fibres/m <sup>3</sup>										
Red: non-compliance with OELV of 2,000 fibres/m <sup>3</sup>										

\* When using microscope settings commonly applied by TNO for fibre counting.

\*\* Counting 0 fibres means that the analytical result is < LOD, from 1 counted fibre a quantification is allowed.

## Possible difficulties when measuring asbestos in dusty (work) environments

The presence of high fibre concentrations and/or high dust concentrations can lead to overloaded filters. For example, when high-energy techniques such as milling or sanding are used, the probability of dust formation is high, especially when no control measures are applied. It is advisable to be aware of the possibility of overloaded filters in such circumstances, and adapt the measurement strategy accordingly (see suggestions below).

According to ISO 14966 a filter is overloaded if more than one-eighth (12.5%) of the filter surface is loaded with dust particles. This leads to poorly visible fibres and overlapping fibres, which prevents reliable microscopic counting. That is why overloaded filters are rejected for analysis and are not included as a quantitative measurement result. Overloading of filters can be prevented by reducing the flow rate and/or measuring time. This method leads to a decrease in measurement sensitivity, which can be partly compensated for by counting more image fields and/or combining the results of several filters taken simultaneously in the same space (weighted average).

It is also possible to perform a qualitative screening of the overloaded filter, in order to obtain an indication of the presence of asbestos fibres. In such a qualitative screening, using SEM/EDXA, a comparable number of image fields is analysed as would be done if the filter had not been overloaded. For each image field it is determined whether asbestos fibres are present, to be able to determine whether or not asbestos fibres are present on the filter as a whole. Because analysis of an overloaded filter may lead to an underestimation of the actual asbestos fibre concentration (due to the overload not all fibres are clearly visible, e.g. because they lie on top of each other, or under other (dust) particles), the result of such an analysis cannot be used for compliance testing.

<sup>10</sup> ISO 14966 states that the analysis can be stopped if 50 fibres are counted, taking into account that the minimal surface area of 0.025 mm<sup>2</sup> is analysed.

Another possibility to prevent overloaded filters is to take a serial measurement in which successive shorter measurements (using multiple filters) are combined into a collective sample. In this case it is important to apply for each of the short duration measurements a comparable flow and sample duration. During the analysis of each of the filters a comparable number of image fields is counted, after which these are combined into one measurement result. Using bigger filters (for instance 47 mm instead of 25 mm) would also decrease the probability of overloading of filters.

In case a filter is overloaded, it is also possible to apply an indirect method (comparable as is used for TEM). In case organic dust is present on the filter, plasma ashing can be applied to the filter to remove all organic components (pre-concentration). In case inorganic dust is present on the filter, the dust on the filter can be resuspended in water to dilute the concentration, after which part of the solution is filtrated over a filter again to be analysed. In case salts are present on the filter these can be 'rinsed' from the filter with a diluted hydrochloric acid solution. It should be noted that to be able to apply such indirect methods, the preparation of the filters for analysis is often complex, as many actions are needed, due to which there is a high risk of errors or contamination of the sample.

## **Innovation in measuring and/or analytical methods**

Below a few developments with regard to measuring and/or analysing asbestos are described. In principle, each of these methods could be applied if it is demonstrated that they perform at least as well as the techniques that are currently used and are stated in the various standards. If new techniques are introduced, it is important to arrange a form of regular quality control for these techniques, comparable to the proficiency tests that are organized for analysis with SEM and PCM, and in which accredited laboratories participate.

### **Innovations with regard to PCM**

In the case of PCM, the limit of detection could be lowered by increasing the sampled volume of air (by measuring longer and/or using a higher flow rate) and/or by counting more image fields. In practice PCM is not suitable for measuring asbestos fibre concentrations lower than approximately 5,000 fibres/m<sup>3</sup> due to the presence of organic and non-organic fibrous components in the air. However, it is possible to improve the resolution of the PCM by using better objectives (lenses), in which case the resolution of SEM can be approached. The use of these specific lenses require additional skills of the analyst. Also, often more image fields will have to be counted in order to arrive at a comparable accuracy.

Tests are being done (particularly in Japan) using fluorescence microscopy in combination with PCM (FCM/FM) (Nishimura et al., 2016<sup>11</sup>; Cai et al., 2021<sup>12</sup>). With this technique fibres are fluorescently coloured by means of a protein and then counted by a computer. The advantage of fluorescence microscopy is that a distinction can be made between asbestos fibres and other fibres, so that the non-asbestiform fibres no longer interfere with the limit of detection. This means that a limit of detection of less than 2,000-5,000 fibres/m<sup>3</sup> would become possible. FCM/FM shows promising results as a fast

---

<sup>11</sup> Nishimura T, Alexandrov M, Ishida T, Hirota R, Ikeda T, Sekiguchi K, Kuroda A. Differential counting of asbestos using phase contrast and fluorescence microscopy. *Ann. Occup. Hyg.* 2016; 60 (9): 1104-1115.

<sup>12</sup> Cai C, Nishimura T, Hwang J, Hu X-M, Kuroda A. Asbestos Detection with Fluorescence Microscopy Images and Deep Learning. *Sensors* 2021; 21: 4582 (<https://doi.org/10.3390/s21134582>).

and selective microscopic technique for determining the concentration of asbestos fibres. However, the method is not yet operational and more research is needed on the performance characteristics of this technique.

### **Portable pumps operating at a higher flow rate**

To measure personal exposure to any substance, portable pumps are preferably used that hinder the employee as little as possible in carrying out their work. At the moment the standard portable pumps operate at a relatively low flow rate, which has an effect on the limit of detection that can be reached, especially when sampling duration is shorter. There are pump suppliers who state that they can supply portable pumps that can be used at a flow rate around 8 L/min. However, this type of pumps need to be tested to check whether they perform correctly in practical situations.

Research into suitable equipment (pumps, filters and sampling heads) that can be used to measure personal exposure to asbestos at higher flow rates, making sure that the collected samples can be analysed with the desired accuracy, would be of added value.

### **Sensors / direct-reading instruments**

Sensors or direct-reading instruments could be used as an alternative for microscopic analysis. With these techniques exposure can be assessed in real-time, without subsequent analysis in a laboratory. In principle results are more reliable because they no longer depend on individual analysts.

Various suppliers work on the development of on-site fibre monitors to directly (real-time) measure the (asbestos) fibre concentration. Examples are the Real-Time Fiber Monitor Model 7400AD from MSP, the Asbestos Alert Monitor from Alert Technology BV, and the Fibrous Aerosol Monitor Model FM-7400 from MIE. However, these monitors are often not (yet) specific enough for asbestos fibres, and their limit of detection is often rather high (>10,000 fibres/m<sup>3</sup>). In addition, at the moment these monitors are also too big to be able to use them as portable measuring devices that can be attached to a worker.

### **Automatic counting**

Several parties are working on the automatic counting of (asbestos) fibres at the moment. This research focusses on different aspects, like recognition of empty image fields and recognition of fibre dimensions. Automatic counting would make it possible to analyse a larger portion of the filter without added uncertainty in the analysis due to errors of the analyst. This in turn would make it possible to measure lower asbestos fibre concentrations (lower limits of detection). However, these techniques are not yet operational, and (more) research is needed on the performance characteristics of these techniques.