

# Scanning Electron Microscope Fibre Counting Scheme Information Book for Participants



## Issue Status

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13	30.01.20	12	Participant Responsibilities	JS

**Note:** Latest issue supersedes all previous issues.

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## 1. Purpose of the Scheme

The SEMS Proficiency Testing Scheme is intended to assess and improve the performance of laboratories evaluating filters for airborne inorganic fibre concentrations. The scheme provides an externally verified indication of performance, which analysts and laboratories can use to satisfy themselves and their clients that their results are of appropriate quality.

SEMS has been organised primarily for European laboratories evaluating airborne fibre samples by Scanning Electron Microscopy (SEM) using the ISO 14966 or VDI 3492 methods. However, it is open to any laboratory with appropriate facilities.

The scheme will also be useful for accreditation bodies to ensure laboratories are operating to the appropriate quality and to demonstrate consistency between laboratories.

The scheme is intended to be self-regulatory and self-financing, so that initiatives for improving analytical performance are taken by the individual laboratory and not imposed by an outside body. As the scheme cannot ensure day-to-day quality, each laboratory must devise its own internal system, which will take into account other factors, not included in the scheme such as sampling location, sampling method and test method, any of which may affect the test result.

## 2. Management of the Scheme

Management and administration of the scheme is carried out by the Science Division of the UK Government's Health and Safety Executive (HSE). Four other laboratories currently assist with validation of samples.

Participation in this scheme does not constitute recognition or approval of a laboratory by HSE or the steering committee.

## 3. Overview

Four loaded filter samples are distributed to participating laboratories in each Round. Laboratories have approximately one month to analyse the samples and return the results to the PT Team. It is recommended that the samples are included as part of the normal routine workload of the laboratory. Access to the online data entry for submitting results will be closed at midnight on the deadline date. Following the deadline, results from participants are processed by the PT Team and the individual and group reports are then produced. The individual and group reports are released to participants within approximately 1 month after the results deadline. The performance of a laboratory is not formally assessed, although some indication is given in relation to the results of the other participating laboratories. A copy of the Group Summary will be published on the website.

## 4. Membership

SEMS is open to any laboratory involved in the evaluation of airborne asbestos samples by SEM. The annual membership fee covers one round of 4 samples. The planned sample dispatch date is detailed on the scheme schedule and the website. The membership fee is non-returnable.

Current participants are required to submit a subscription form via the PT Online Data Entry System before the end of each financial year. It is the participants' responsibility to ensure all details are filled in correctly on the subscription form and that payment is promptly made in full. **Failure to pay scheme fees may result in samples and reports being withheld, and ultimately exclusion from scheme participation.**

## 5. Operation of the Scheme

### 5.1 Samples

Samples are dispatched to participating laboratories by courier service according to the schedule published on the website (<http://www.hsl.gov.uk/proficiency-testing-schemes>). Participants should contact the PT Team if they have not received their samples within 10 days of the published date.

SEMS samples are pre-gold coated polycarbonate filters loaded with known densities of asbestos and/or other inorganic fibre types. All filter samples are dispatched in labelled plastic sampling cowls, sealed in a plastic bag. It is recommended that the sample cowls be opened in a suitable environment (fume cupboard/asbestos handling cabinet). The samples are produced in batches and it is intended that they be analysed once by the laboratory and are **not** returned to the PT Team afterwards. The laboratory may retain the samples and use them for training or QC purposes.

The samples are all artificially produced in the laboratory and may contain one or more inorganic fibre types and include non-fibrous matrix material. Fibre densities will normally be between 10 and 100 fibres mm<sup>-2</sup> but may occasionally be outside these limits.

Samples are produced in batches such that each sample in any given batch will have an equivalent fibre density. At least 10% of each batch is validated by independent validation laboratories, prior to incorporation into the scheme to ensure suitability (see Appendix 2).

### 5.2 Analysis

There are two recommended analytical methods for this scheme:

- The ISO 14966 (2002) method: Ambient air – Determination of numerical concentration of inorganic fibrous particles – Scanning electron microscopy method. **This method does not currently include the WHO fibre counting rules (see below for more details).**
- The VDI 3492 (2013) method; Measurement of inorganic fibrous particles – Scanning electron microscopy method. This method uses the WHO counting rules.

**Please note that the following rules will be used for this scheme:**

- **When fibres are in contact with non-fibrous particles, the particles should be ignored. If fibre ends are concealed by particles, only the visible parts of the fibre are assessed.**
- **Fibres in a bundle are counted individually if they can be distinguished from one another.**
- **A fibre bundle is assessed as a single fibre if the individual fibres cannot be distinguished from one another, and if the complete fibre bundle complies with the definition of a countable fibre. A countable fibre has length >5µm, width <3µm and aspect ratio >3:1.**
- **Count 100 inorganic fibres or an area representing approximately 1mm<sup>2</sup> of the sample. The minimum area searched should be at least 0.25mm<sup>2</sup>.**
- **An on-screen magnification of between 2000 – 2500x**

Other methods may be used if they can be shown to give equivalent results. Laboratories should always report which method they have used and document any significant differences from the ISO 14966 and VDI 3492 methods.

## 5.3 Reporting by the Laboratory

Participating laboratories have approximately 1 month to analyse the samples and report their results. Laboratories must return results for all four samples. Laboratories reporting fewer than four results in any round may not have their results processed. Participants are required to report the following:

- The number of **countable fibres** (as defined above) counted for each fibre type identified (amphibole, chrysotile and other inorganic). The presence of organic fibres should not be reported.
- The number of fields of view searched
- The fibre density for each fibre type identified in fibres mm<sup>-2</sup>
- The screen area of the SEM used in mm<sup>2</sup>
- The on-screen magnification used – note this may not be the same as the magnification used by the camera.
- The manufacturer, model and age (approximate) of the microscope used.
- The number of fibres with length <5µm may also be reported. However, this is not used in the assessment of results.

**Please note that the on-line data entry system does not recognise the “,” symbol as a decimal point and that the “.” symbol must be used.**

Up to three individual results may be submitted for each sample.

The laboratory should retain copies of worksheets completed during the analysis – these may be required for scrutiny in future rounds.

The fibre density is calculated as follows:

$$d = \frac{f}{Fa}$$

Where: d = the fibre density (in fibres per mm<sup>2</sup>)  
f = the number of fibres counted  
F = the number of fields evaluated  
a = the area of a single field (mm<sup>2</sup>)

## 5.4 Some Practical Suggestions

### 5.4.1 Filter Preparation

If filters are mounted on carbon pads, this may influence any subsequent evaluation carried out at a later date (as the contrast may change considerably). It is therefore recommended that only part of the filter is prepared, leaving the remainder spare, if required.

### 5.4.2 Fibre Identification

Countable fibres should be generally classified as serpentine (chrysotile), amphibole asbestos or other inorganic fibres; you may also classify the asbestos fibres more precisely (chrysotile, amosite, actinolite, anthophyllite, crocidolite and tremolite). However, this additional fibre discrimination is not currently assessed.

## 5.5 Processing of Results

Results (i.e. the information listed in 5.3 above) should be submitted via the PT on-line data entry system (PT ODES). Laboratories may submit up to three results for each sample. “One result” may include total

fibre density plus densities for amphibole asbestos, chrysotile and other inorganic fibres as appropriate. Data is processed as received and cannot be changed after the closing date of each round. Results will not normally be accepted after the required deadline date. Participants may change their results up to the date of the deadline.

Laboratories receive feedback based on the results for each sample per round.

### **Data Analysis**

Data analysis is based upon the total asbestos fibre density (amphibole & chrysotile) derived from fibre numbers counted and the area of the filter searched. The distribution of fibres on a filter derived from airborne sampling is normally described as being Poisson-distributed. For Poisson-distributed counts, the variance (standard deviation squared) is equal to the mean. However, in practice the variation may be larger due to differences in sample production, laboratories and individual microscopists. A comparison of the observed standard deviations with the expected standard deviations (expected under Poisson distribution) show that the observed variation is larger than that expected, and it is difficult to quantify how much of this may be due to differences in sample production, and how much is due to differences between labs/microscopists.

The data will be compared against the criteria used in the UK phase contrast fibre counting proficiency testing scheme RICE. Details of the analysis used can be found in Appendix 1.

### **5.6 Complaints and Appeals**

Complaints **must** be in writing and will be forwarded to and considered by the Fibres Proficiency Testing Steering Committee (FPTSC). The complaint will be acknowledged and the participant informed of receipt and date for consideration. The decision of the FPTSC will be sent to the participant as soon as possible.

If the participant wishes to appeal following a sample query investigation, then this also must be in writing and should be sent to the PT Team administrator. The appeal will then be considered at the next meeting of the FPTSC or during a convening of FPTSC members by email/teleconference at the discretion of the FPTSC chairperson. The participant will be informed of the outcome by the chairperson of the FPTSC.

### **5.7 Technical Queries**

Queries of a non-administrative nature (which may affect a laboratory's participation or performance in the scheme) must be submitted by email and should be sent to the email address given in Appendix 3.

Any query regarding round scores for must be sent to the PT Team within 20 working days of the date of issue of round reports. Queries received after the next round has been dispatched will not be accepted. All queries will be dealt with on an individual basis and will be considered and acted upon by the PT Team. If the PT Team is unable to resolve the query it will be forwarded to the FPTSC. For example some of the different categories of queries are outlined below:

#### **a) Assigned Reference Values**

Any query concerning the assigned reference value(s) of a sample after the results have been published, should be sent in the appropriate format as outlined above. The PT team will carry out initial investigations following receipt of written queries. Appeals against penalty scores are investigated in the first instance by the PT team and a response sent in writing by email to the participant. This investigation may require the analysed and/or unanalysed portions of the sample to be returned to the PT Team at the participant's own cost.

Any decisions regarding participant scores will be sent to the relevant participant(s) as appropriate. Any appeals will be brought before the FPTSC.

b) Accreditation

Any query regarding participant accreditation should be taken up with the participants appropriate accreditation body.

c) Extensions

Extensions will not normally be considered. If samples are dispatched later than published, then the same approximately 25 working day reporting period will be used and all participants will be contacted and given the new deadline date.

d) Change of Address

It is the participants' responsibility to provide the PT Team with an up-to-date receipt location/address and contact details and to inform whoever receives their samples when PT samples are expected. If participants wish to change their contact or company details, they should contact the PT Team who will issue a 'Change of Contact Details Form', which must be fully completed and returned. Where necessary, participants should ensure any required paperwork is in place to allow transit of samples through customs. Any additional charges incurred from couriers will be passed onto the participant. If samples are returned to the PT Team by the courier and have to be re-dispatched to a different address to that given on the order form or on a 'Change of Contact Details Form', then a charge may be levied.

e) Return of Samples

Any samples returned to the PT Team for investigation regarding asbestos content, contamination or penalty scores must be sent via a courier, at the participants own expense. It is strongly recommended that any courier used is licensed to carry hazardous materials. Samples returned for investigation will be dispatched back to participants with the next available round of SEMS samples. The process is summarised below;

(i) Contact PT Team

(ii) Form sent to participant to complete

(iii) Courier sample & Request Form back to the PT Team

(iv) Investigation undertaken

(v) Outcome delivered

(vi) Sample returned to participant

f) Re-issue of Reports

The Individual & Group Report will be available to download on the on-line PT data entry system.

## 6. Records and Confidentiality

Paper and computer records are maintained and include information on laboratory name, contact name, postal address, e-mail address, laboratory performance and telephone. It is the responsibility of the participant to notify the PT Team of any changes to details.

Results are submitted to the scheme organisers in confidence and are not revealed to a third party except where required to do so by law, for example in association with a criminal prosecution.

A list of participants in the SEMS PT scheme may be published on the group report. Laboratories wishing to be excluded from this list must inform the PT Team in writing.

## 7. Collusion and Falsification of Results

Laboratories must not submit results which are false reflections of the performances of those laboratories' individual analysts, whether by collusion within or between laboratories, or by any other means. Falsification of results is regarded as an extremely serious matter. The following disciplinary procedures may be applied whenever there is clear documentary evidence of falsification of results or improper collusion.

- The relevant accreditation body will be informed
- The laboratory will revert to unclassified
- The membership of the laboratory may be suspended, subject to review by FPTSC.

### Reinstatement after suspension for falsifying results

Laboratories must satisfy UKAS, or the relevant accreditation body, that appropriate corrective and preventive actions have been put in place to prevent recurrence. Evidence must be supplied to the PT Team, which will be reviewed by FPTSC.

Upon reinstatement, the laboratory will be treated as a new participant (i.e. they will be categorised as unclassified until the relevant numbers of rounds have been completed).

## 8. Advertising by Participants

It is recommended that any publicity and advertising material should describe the laboratory as an "SEM PT Scheme participant". Participants may wish to describe their level of performance but should state the year in which they achieved this status.

## 9. Quality Control and Training Products

A laboratory may wish to purchase extra sets of samples, for example, for training purposes or to add to their internal quality control scheme. If requested, these samples may be supplied in sets of four. The PT Team are also able to provide a wide range of asbestos quality control and training products, information on which can be found on the website.



# Annex 1 - Data Analysis and Derivation of Assigned Reference Values

## Regular Inter-laboratory Counting Exchange (RICE) Criteria

Where  $R$  is the reference value – in this case the **Median** value of laboratory results

**High density slides** ( $R > 63.7$  fibres.  $\text{mm}^{-2}$ )

Target band A:  $> 0.65R$  to  $< 1.55R$

Target band B:  $> 0.50R$  to  $0.65R$  [band -B] and  $> 1.55R$  to  $2.00R$  [band +B]

Target band C:  $< 0.50R$  [band -C] and  $> 2.00R$  [band +C]

**Low density slides** ( $R \leq 63.7$  fibres.  $\text{mm}^{-2}$ )\*

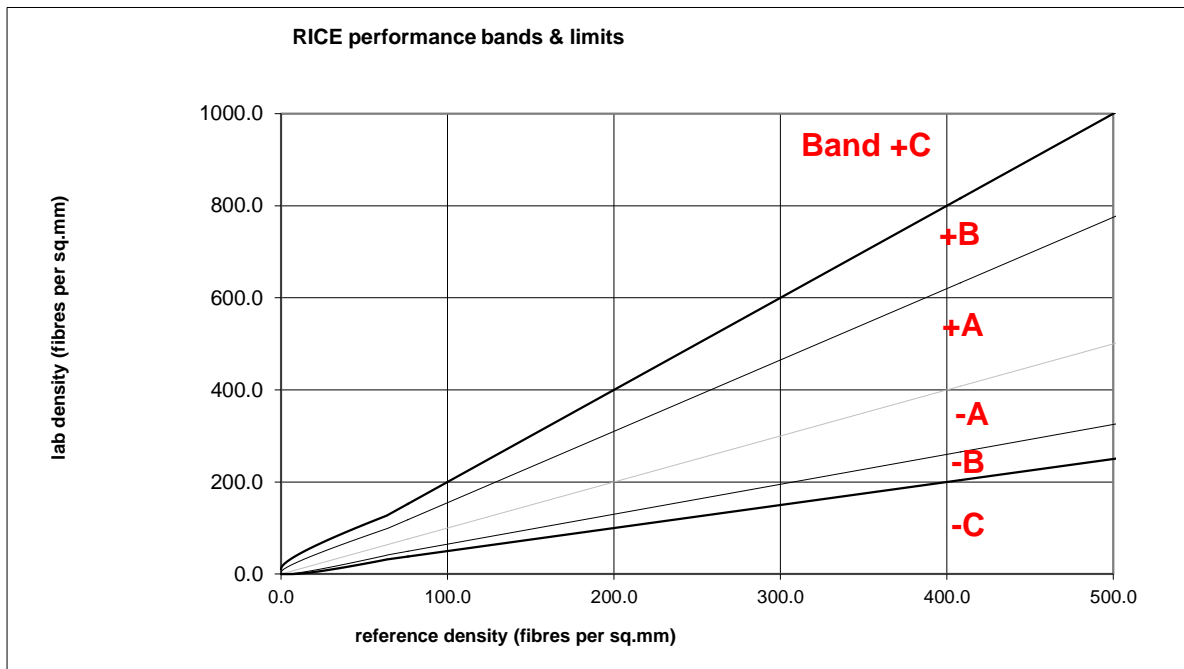
Target band A:  $(\sqrt{R-1.57})^2$  to  $(\sqrt{R+1.96})^2$  [band A]

Target band B:  $< (\sqrt{R-2.34})^2$  to  $(\sqrt{R-1.57})^2$  [band -B]  
 $> (\sqrt{R+1.96})^2$  to  $(\sqrt{R+3.30})^2$  [band +B]

Target band C:  $< (\sqrt{R-2.34})^2$  [band -C]  
 $> (\sqrt{R+3.30})^2$  [band +C]

\* For samples less than  $5.5$  fibres. $\text{mm}^{-2}$  the lower limit is set to zero when the component within the brackets  $(\sqrt{R-n})$  is less than zero.

The plot below shows the positions of the performance limits in relation to the reference counts up to reference density  $500$  fibres per  $\text{mm}^2$ .



## Annex 2

### Sample Production and Validation

All samples used in the scheme are produced in the laboratory using a range of real and “manufactured” asbestos containing materials. As a result, most samples will include some non-fibrous matrix material.

A modified dustiness-testing drum is used to generate an aerosol of fibrous particles using a range of carefully selected asbestos containing materials or in some cases pure asbestos. Samples are produced on pre-gold coated 25mm polycarbonate filters loaded into air sampling cassettes, which in turn are connected to a multi-port sampling device or “sputnik”. The sputnik is connected directly to the dustiness tester and the airborne dust cloud is sampled at around 1.8 litres per minute. The sampling time and rotation speed of the dustiness drum are adjusted to produce the required fibre density.

Sample densities will typically range between 10-100 fibres  $\text{mm}^{-2}$ . However, occasionally these limits will be exceeded.

The target fibre types will include amphibole asbestos, chrysotile and other inorganic fibres. Complex mixtures of fibre types will not normally be used.

The “reference” laboratories will validate approximately 10% of each sample batch.

## Annex 3

### Contacts and Advice

For all SEMS enquiries please contact:

By email: [proficiency.testing@hse.gov.uk](mailto:proficiency.testing@hse.gov.uk)

By Telephone: 00 44 (0) 203 028 3382

By Post:

Proficiency Testing Team

HSE Science & Research Centre

Harpur Hill

Buxton

Derbyshire

SK17 9JN

SEMS Website page:

<http://www.hsl.gov.uk/proficiency-testing-schemes/sem-fibre-counting-scheme>

PT on-line data entry (PT ODES) web link;

<https://xnet.hsl.gov.uk/aims/>

## Annex 4

### Responsibilities, Terms & Conditions

HSE will operate the Proficiency Testing (PT) schemes in accordance with its obligations as set out in the relevant Scheme Information Book for Participants, available on our website.

Participants must abide by the rules and responsibilities of the scheme set out in the information book and in particular as detailed below.

Participants should comply with the HSE's standard conditions of business, a copy of which is available upon request.

### Participant Responsibilities

Participant responsibilities are outlined below:

- Your PT Laboratory Number **must be quoted in all correspondence**.
- All queries should be directed to the proficiency testing email address and the PT Team will respond as soon as possible.
- To ensure participants are kept up to date with important information it is the participants' responsibility to inform the PT Team of any changes to contact details.
- Participants must ensure that any required paperwork / permits are in place to allow samples through national border controls / customs (additional charges from the courier will be passed on to participant should there be further fees payable). Failure to do so may result in delays or detainment of sample shipment and hence participation in the round.
- Participants must use the Proficiency Testing Online Data Entry System (PT ODES) to subscribe to the scheme.
- Samples **will not** be despatched until full payment has been received.
- The preferred method of payment is via Worldpay (online credit/ debit card system). A full VAT receipt is available immediately after the payment has been processed.
- If an invoice is required, the participant needs to allow 4-6 weeks for it to be issued and must pay, in full, within 30 days of the invoice date. An invoice shall not be regarded as paid until funds (GBP) are received into a UK sterling bank account operated by Health and Safety Executive.
- It is the participants' responsibility to ensure that scheme round results are submitted by the defined deadline dates. Participants must have access to certain mandatory IT facilities such as Microsoft Word and Excel, an email account and a web access facility in order to participate.
- Participants must submit round results via the PT ODES for each scheme. The PT Team reserves the right to refuse results submitted on reporting formats alternative to the online data entry system provided.
- Transcription errors and / or errors in reporting results in the correct format by participants is considered by the PT Team to be part of the proficiency testing assessment process. As such, the PT Team will not amend or correct any submitted results if requested. Participants can however, change their results, provided that any amendments are made by the close of the round and the before the predefined reporting deadline.

- For a copy of HSE's standard conditions of business, or for any queries regarding AIMS please email [proficiency.testing@hse.gov.uk](mailto:proficiency.testing@hse.gov.uk) or visit the website at <http://www.hsl.gov.uk/proficiency-testing-schemes>
- For urgent queries, please call 00 44 (0) 203 028 3382. Please remember to quote the allocated PT laboratory number in all correspondence.