ENVIRONMENT AGENCY

The determination of asbestos in soil and associated materials (March 2014)

Methods for the Examination of Waters and Associated Materials

This page is deliberately blank.

The determination of asbestos in soil and associated (2014)

DRAFT (March 2014)
(This draft is being developed by an SCA committee and is provided on the understanding that further development may still be required before publication is completed.)

Methods for the Examination of Waters and Associated Materials

This booklet contains a method for quantifying asbestos in soils, construction products, and associated materials. This method may be suitable for determining the presence of asbestos in soil when evaluating human health risks. Furthermore, using the procedures described in this booklet should enable laboratories to satisfy the requirements of ISO 17025 for accreditation of the method.

Whilst this booklet refers to equipment actually used, this does not endorse these products as being superior to other similar products. Equivalent equipment is available and it should be understood that resulting performance characteristics might differ when other products are used. It is left to users to evaluate these procedures in their own laboratories. Only limited performance data are presented.

Contents

The determination of asbestos in soil and associated materials

The quantification of asbestos in soil or aggregate using a gravimetric method for ACM and fibre bundles, and dispersion and fibre counting for free fibres using Phase Contrast Optical Microscopy.

NB Prior to using this quantification method, it is necessary to perform the identification of asbestos fibres or asbestos containing material (ACM) in soil or aggregate using Polarising Light Microscopy as per HSG 248. It is important to note that any laboratory offering this test commercially **must** be accredited to ISO 17025 for this method.

About this series

Introduction

This booklet is part of a series intended to provide authoritative guidance on methods of sampling and analysis for determining the quality of drinking water, ground water, river water and sea water, waste water and effluents as well as sewage sludges, sediments, soil (including contaminated soil) and biota. In addition, short reviews of the most important analytical techniques of interest to the water and sewage industries are included.

Performance of methods

Ideally, all methods should be fully validated with results from performance tests. These methods should be capable of establishing, within specified or pre-determined and acceptable limits of deviation and detection, whether or not any sample contains concentrations of parameters above those of interest.

For a method to be considered fully evaluated, individual results encompassing at least ten degrees of freedom from at least three laboratories should be reported. The specifications of performance generally relate to maximum tolerable values for total error (random and systematic errors) systematic error (bias) total standard deviation and limit of detection. Often, full evaluation is not possible and only limited performance data may be available. An indication of the status of methods is normally shown at the front of these publications on whether the method has undergone full performance testing.

In addition, good laboratory practice and analytical quality control are essential if satisfactory results are to be achieved.

Standing Committee of Analysts

The preparation of booklets within the series "Methods for the Examination of Waters and Materials" and their continuing revision is the responsibility of the Standing Committee of Analysts. This committee was established in 1972 by the Department of the Environment and is now managed by the Environment Agency. At present, there are nine working groups, each responsible for one section or aspect of water quality analysis. They are

1 General principles of sampling and accuracy of results
2 Microbiological methods
3 Empirical and physical methods
4 Metals and metalloids
5 General non-metallic substances
6 Organic impurities
7 Biological methods
8 Biodegradability and inhibition methods
9 Radiochemical methods

The actual methods and reviews are produced by smaller panels of experts in the appropriate field, in co-operation with the working group and main committee. The names of those members principally associated with this booklet are listed at the back of this booklet.

Publication of new or revised booklets will be notified to the technical press. If users wish to receive copies or advance notice of forthcoming publications, or obtain details of the index of methods then contact the Secretary on the Agency's internet web-site (www.environment-agency.gov.uk/nls) or by post.

Every effort is made to avoid errors appearing in the published text. If, however, any are found, please notify the Secretary.

Mark Gale
Secretary

October 2013

Warning to users

The analytical procedures described in this booklet should only be carried out under the proper supervision of competent, trained analysts in properly equipped laboratories.

All possible safety precautions should be followed and appropriate regulatory requirements complied with. This should include compliance with the Health and Safety at Work etc Act 1974 and all regulations made under the Act, and the Control of Substances Hazardous to Health Regulations 2002 (SI 2002/2677). Where particular or exceptional hazards exist in carrying out the procedures described in this booklet, then specific attention is noted.

Numerous publications are available giving practical details on first aid and laboratory safety. These should be consulted and be readily accessible to all analysts. Amongst such publications are; "Safe Practices in Chemical Laboratories" and "Hazards in the Chemical Laboratory", 1992, produced by the Royal Society of Chemistry; "Guidelines for Microbiological Safety", 1986, Portland Press, Colchester, produced by Member Societies of the Microbiological Consultative Committee; and "Safety Precautions, Notes for Guidance" produced by the Public Health Laboratory Service. Another useful publication is "Good Laboratory Practice" produced by the Department of Health.

Glossary

Asbestos	Complex silicate minerals including chrysotile, crocidolite, amosite and other amphiboles
AC	Asbestos Cement assumed to contain approximately 15% asbestos
ACM	Asbestos Containing Material
Bulk sample	(1)An as received sample containing coarse material such as aggregate, gravel, ballast, hardcore or similar (2)A sample of building materials such as insulation, plasterboard, roofing or similar
MMMF	Machine Made Mineral Fibres
PCOM	Phase Contrast Optical Microscopy
PLM	Polarising Light Microscopy
Respirable fibres	Fibre that meets the definition of a respirable fibre in HSG248 (>5um long, <3um width, 3:1 or higher aspect ratio)
Trace	1 or 2 fibres in an entire sample
Asbestiform	Having the form or structure of asbestos. It implies a particular kind of fibrosity in which fibres have high tensile strength and flexibility (Webster's Online Dictionary).
Asbestos cement	Cement material which is predominantly a mixture of cement and chrysotile and which when in a dry state absorbs less than 30% water by weight (CAR 2012)
Asbestos coating	A surface coating which contains asbestos for fire protection, heat insulation or sound insulation but does not include textured decorative coatings (CAR 2012)
Asbestos insulating Board (AIB)	any flat sheet, tile or building board consisting of a mixture of asbestos and other material except— (a) asbestos cement; or (b) any article of bitumen, plastic, resin or rubber which contains asbestos, and the thermal or acoustic properties of the article are incidental to its main purpose; (CAR 2012)
Asbestos insulation	any material containing asbestos which is used for thermal, acoustic or other insulation purposes (including fire protection) except— (a) asbestos cement, asbestos coating or asbestos insulating board; or (b) any article of bitumen, plastic, resin or rubber which contains asbestos and the thermal and acoustic properties of that article are incidental to its main purpose; (CAR 2012)
Asbestos-containing material (ACM)	Any material that contains asbestos above trace quantities
Aspect ratio	The ratio of the length of a fibre to its diameter.
Bonded ACM	Material where the asbestos fibres are contained in a matrix, such as resins or cement (locked into

	a matrix; e.g. asbestos- cement, vinyl tiles). If in reasonable condition, the release of respirable fibres from bonded ACMs in soils is likely to be low. HSG64 (Appendix 2) gives a table of ACMs in buildings, listed in order of ease of fibre release.
Cement-bonded Asbestos	Collective term for materials containing asbestos in a cement matrix, including high density (e.g. asbestos cement) and low density (e.g. asbestos insulating board) materials.
Control limit	A concentration of asbestos fibres in the atmosphere when measured in accordance with the 1997 WHO recommended method, or by a method giving equivalent results to that method approved by the HSE of 0.1 f/ml of air (100,000 fibres/m3) averaged over a continuous period of 4 hours.
Environmental (exposure)	Exposures that result from background concentrations of asbestos in air (*i.e.* exposures other than occupational or para-occupational exposures)
Exposure scenario	Detailed description of all events likely to result in human exposure to airborne asbestos fibres from soil, and the factors and considerations that influence these potential exposures
Fibre	A particle that is 5 um or longer, with a length-to-width aspect ratio of 3 to 1 or longer.
Fibril	A fine filament or fibre approximately 150 Angstroms in diameter (Naumann and Dresher, 1966)
Friable ACM	Friable ACM release asbestos fibres easily. Friable ACM may be crumbled or pulverised or reduced to powder by hand pressure when dry (e.g. pipe insulation, sprayed insulation, millboard and bonded ACMs not in reasonable condition). Disturbance of these materials can generate large quantities of 'respirable' fibres (OSHA)
Interferences	Fibrous substances which, if present, may interfere with asbestos analysis. Some common fibres are (HSG248 para A3.14): natural organic fibres (such as cotton and hair), synthetic organic fibres (such as aramid, polyester and rayon), man-made mineral fibres (for example, mineral wool and glass fibre), and naturally occurring mineral 'fibres' (such as wollastonite and diatom fragments) and according to OSHA: Fibreglass; Anhydrite; Plant Fibres; Perlite Veins; Gypsum; Membrane Structures; Sponge Spicules; Microorganisms. The use of electron microscopy or optical tests such as polarized light, and dispersion staining may be used to differentiate these materials from asbestos when necessary.
Joint and several Liability	Where in law two or more parties can be sued for causing the same damage. Each party can be liable for a part of the damage,[jointly with others] or the whole amount of the damage

	alone[severally liable]
Latency period	Period between exposure and the onset of asbestos-related disease (*i.e.* first appearance of symptoms or diagnosis)
Occupational exposure	Exposures that occur directly as a result of work activities
Para-occupational exposure	Exposure which occurs in household members who live with an occupationally exposed worker, but who are not themselves occupationally exposed. Such exposure might occur, for example, when laundering contaminated clothing.
Peritoneum	Self-lubricating membrane lining of the lower digestive tract and abdominal cavity
Pleura	Self-lubricating membrane lining of the lung and chest cavity
Prescribed disease	A disease arising from a person's occupation and not a risk common to everybody
Respirable fibres	Respirable fibres are very small fibres (i.e. <3 um diameter, usually longer than 5 um and have aspect ratios of at least 3:1) that can be inhaled into the lower regions of the lung and are generally acknowledged to be most important predictor of hazard and risk for cancers of the lung.
Conceptual site model	A diagrammatic and tabular representation of the characteristics of the site shows the possible relationships between contaminants, pathways and receptors as well as relevant uncertainties.
Trace	HSG 248 refers to 'trace asbestos identified' where "1 or 2 fibres are seen and identified as asbestos" (HSE 2005 p. 16)

The determination of asbestos in soil and associated materials

1. Introduction

Asbestos is a known carcinogen and over 4000 deaths a year are attributed to asbestos related diseases. Most of the current legislation relates to workplace protection, clearance of buildings, demolition etc, and there is currently no specific legislation or guidance relating to asbestos in soil. It is now apparent that many brownfield sites are contaminated with asbestos to some degree and this is a cause for concern as many soils and construction/demolition materials submitted to laboratories are not requested for asbestos analysis.

Asbestos is not one compound, but consists of a group of naturally occurring mineral silicates: chrysotile (white), existing as a fibrous serpentine form, and the amphiboles such as crocidolite (blue), amosite (brown) and the asbestos forms of actinolite, anthophyllite and tremolite. Because of their excellent fire retardant properties, these materials were used extensively in the construction and manufacturing industries up until 1999, when chrysotile was finally banned. Chrysotile is the most common form, and is less carcinogenic than crocidolite, amosite or any of the other amphiboles.

Asbestos containing material (ACM) and/or free fibres are found in many sites where construction or demolition has taken place, and may not be visible to the naked eye on a preliminary site inspection. The presence of free fibres represents a much greater hazard and risk to human health than asbestos bound up in cement, tiles, bitumenised products or other material. If the soil is wet, then there is little chance of airborne fibres being released, but when dry, there is a significant risk of release. A study performed by Addison *et al* in 1988 demonstrated that soils containing as little as 0.001% asbestos could release fibres at a concentration exceeding the control limit of 0.06 fibres/ml. Soil can be tracked back into buildings on shoes and clothes, adhere to vehicles and tyres on site, and surface soil can be windblown.

It is crucial that a contract review procedure is followed when clients request asbestos analysis, in order to determine the end use of the data. It may be for classification of material as hazardous waste (> 0.1%), or for human health risk assessment, whereby a much lower reporting limit is required. Currently, this is assumed to be 0.001%, but there is no ratification of this number by any regulator.

Laboratories previously offered a gross visual screen to determine if asbestos containing material was present in the sample, but this would only cover pieces of ACM and bundles of fibres, and would not include small fragments or free fibres. UKAS no longer recognise this method as robust and it should not now be offered by any laboratory. Samples must be inspected under a stereomicroscope to determine the presence of potential ACM and fibres, and if detected, analysis can then be performed using gravimetric and fibre dispersion/counting methods as appropriate, to reliably quantify asbestos in the samples.

The type of sample matrix is also important, and laboratories should be required to provide validation data for different types of soil, aggregate, and ballast in order to receive accreditation for each matrix.

Other information

General comments on sampling, storage and subsampling:

This document will not comment on sampling procedures, other than to stress the importance of taking representative samples from site, and to ensure samples are individually double sealed and clearly labelled as potentially containing asbestos.

Samples do not require refrigeration.

General comments on analysis

It is important that at least approximately 1 kg of sample is submitted for the presence of asbestos (see section 2 of the method), and not used for any other analysis.

The whole as received sample should be screened visually for ACMs and fibres, with a dried subsample examined by stereomicroscopy (x20 - x40).

Prior to quantifying asbestos in soil, samples must be examined to identify the presence of asbestos. If none is detected, then the sample will be reported as 'No asbestos detected' (NAD).

Staff must receive and complete the internal training module and obtain the P401 Identification of asbestos in bulk samples (PLM) certificate before performing any analysis.

General usage classifies asbestos analysis into three stages:

Stage 1: The determination of presence or absence, followed by identification of asbestos as detailed in HSG 248*
Stage 2: The removal of ACM and fibre bundles with gravimetric analysis to determine percent by weight
Stage 3: The dispersion and collection of free fibres followed by fibre counting and measurement

*Laboratories must obtain UKAS accreditation to ISO 17025 for identification of asbestos before this method can be offered commercially.

2 Hazards and safety precautions

Risk of exposure to airborne asbestos fibres by inhalation, and skin contact of reagents used in the method.

Asbestos is a Class 1 carcinogen and great care should be taken to avoid inhalation of fibres. All samples received in the laboratory should be handled in safety cabinets with appropriate fume extraction and filtration. Internal asbestos

air tests should be performed monthly. It is important to note that other chemical hazards may be present in the soil, which will not be removed by the HEPA filters (e.g. organic vapours), so laboratories must ensure they comply with all relevant H & S regulations such as the H & S at Work Act.

3 References

HSG 248 Asbestos: The Analysis Guide for Sampling, Analysis and Clearance Procedures. 2005.

McCrone W.C., Asbestos Identification (Second Edition), The McCrone Research Institute, 1987.

LAB 30, Application of ISO/IEC17025 for Asbestos Sampling and Testing, UKAS, Edition 2, April 2008.

Davies, L. S.T., et al. 1996. HSE Contract Research Report No. 83/1996, Development and validation of an analytical method to determine the amount of asbestos in soils and loose aggregates. IOM, Edinburgh

MDHS 87

MDHS 89

The quantification of asbestos in soils and associated materials using a gravimetric method for ACM and fibre bundles, and dispersion and fibre counting for free fibres using Phase Contrast Optical Microscopy, including calculation of the concentration of Potentially Respirable Fibres.

Scope

This method describes the quantification of asbestos in soil, construction or similar associated materials. The asbestos may be in the form of ACM, fibre bundles, or individual (free) fibres, and this method seeks to address as wide a range of materials as possible by weighing the fragments of ACM and fibre bundles, and expressing their presumed asbestos content as a percentage, and also isolating the free fibres (dispersion) and then trapping these on filter papers to be measured and counted. These can also then be expressed as a percentage of the sample. The sum of the two results provides a quantitative measure of the asbestos in the sample expressed on a dry weight basis.

1 Performance Characteristics of the Method

1.1	Substances determined	Asbestos: chrysotile, crocidolite, amosite, and the asbestos forms of actinolite, anthophyllite and tremolite
1.2	Type of sample	Soil, aggregate, ballast, and construction materials
1.3	Basis of method	Visible fragments of ACM and fibre bundles are removed and determined gravimetrically, with free fibres dispersed, filtered, and measured and counted using PCOM. The sum of the two results is calculated as % by weight of the original dried sample
1.4	Range of application	Gravimetric: 100 - 0.1% Free fibres: 0.1- 0.001%
1.5	Calibration curve	Not applicable
1.6	Standard deviation	Gravimetric: Free fibres:
1.7	Limits of detection	Gravimetric: 0.01% Free fibres: 0.00001% ??
1.8	Bias	Gravimetric: Free fibres:

2 Principle

2.1 Gravimetric analysis – For 1 kg or less, the entire sample is weighed using a calibrated balance, and examined inside a safety cabinet. The dry weight of the sample is also determined. Any items that may potentially contain asbestos are removed from the sample for identification. Suspect ACMs and fibres are examined using stereomicroscopy

and Polarised Light Microscopy as described in HSG 248. Items confirmed as containing asbestos are removed, weighed using a calibrated balance, and the mass percentage ACM/fibre content of the sample is calculated. The asbestos content of the sample is then calculated based on the typical asbestos content of the specific ACMs found

2.2 Free fibre analysis - following the gravimetric method, representative subsample, of the residue is weighed into a conical flask, and water added in the ratio of 1:200 solid to liquid (volume dependent on sample type). The sample/solution is mixed vigorously for a minimum of 30 seconds to ensure complete dispersion, allowed to settle for 10 seconds, and then a known quantity is filtered through a cellulose-ester filter paper. The filter is then placed onto a microscope slide, allowed to dry, and then cleared and fixed using the acetone/triacetin method described in HSG 248. The slides are then evaluated using PCOM. From the number and size of the asbestos fibres observed on the slides the mass percentage of asbestos in the sample is estimated.

The sum of both ACM/visible fibres and free fibres is reported as the % asbestos content in the original sample on a dry weight basis.

3 Interferences

Non-asbestos fibres may be present in the sample. These may be of mineral or organic origin and care must be taken to avoid misidentification. Where possible fibres are identified as asbestos using the modified PCOM. When counting free fibres, if it is not possible to determine the fibre as non-asbestos, the fibre is presumed to be either chrysotile or amphibole based on the morphology of the fibre. Fibres which are clearly not identified as asbestos are not included in the count.

Clay matrices or oily samples may cause problems with the dispersion method.

4 Sample handling

All handling and examination of samples for asbestos, and the opening of all containers, should be conducted in a safety cabinet with appropriate extraction and HEPA filters. All staff must wear appropriate PPE. Care should be taken to avoid any risk of cross contamination, particularly airborne.

The client should provide the laboratory with an individual asbestos sample which may require quantitative analysis. Samples for quantification of asbestos should be not be used for testing by other departments within the laboratory, as any removal of material will compromise the accuracy of the results.

Soil samples should be collected in 1 kg tubs or heavy duty polythene bags. Samples should be individually double sealed and labelled as 'Potential asbestos', and not used for other testing.

No chemical preservation or refrigeration is required.

5 Reagents

5.1 10% Hydrochloric acid

5.2 Acetone

5.3 Triacetin

No reagents are required for the gravimetric section of the analysis.

6 Apparatus

Gravimetric

6.1 Balances capable of weighing to 2 and 5 decimal places

6.2 Disposable gloves

6.3 Metal spatula

6.4 Controlled environment cabinet fitted with high efficiency HEPA filtered extraction Units.

The cabinet extractor units and face velocities are tested monthly to ensure the linear velocity is >0.5m/s and DOP tested every six months to check filter efficiency.

6.5 Sample bags

Free fibres

6.7 Balance capable of weighing to 2 decimal places

6.8 Analytical balance capable of weighing to 4 decimal places

6.9 Blunt nose forceps

6.10 Mixed cellulose-ester filter papers, 25mm diameter, pore size of 0.8µm

6.11 Straight-sided filter apparatus

6.12 Filtration collar

6.13 Vacuum pump

6.14 Auto pipette capable of pipetting 0.25ml

6.15 1ml Pasteur pipette

6.16 Acetone hot block/vaporiser

6.17 Microscope slides and coverslips

6.18 5 ml Syringe

6.19 1000 ml conical flask

6.20 Metal spatula

6.21 Phase Contrast Microscope with Polariser/Analyser and Red Tint Plate

6.22 NPL Test Slide

6.23 Stage Micrometer slide

6.24 Graticule – need spec

6.25 Tally Counter

6.26 Coarse filters (for waste disposal)

6.27 47mm diameter 0.8 µm filters (for waste disposal)

6.28 Oven for drying to a temperature between 40^{o}C and 110^{o}C

7 Analytical Procedure

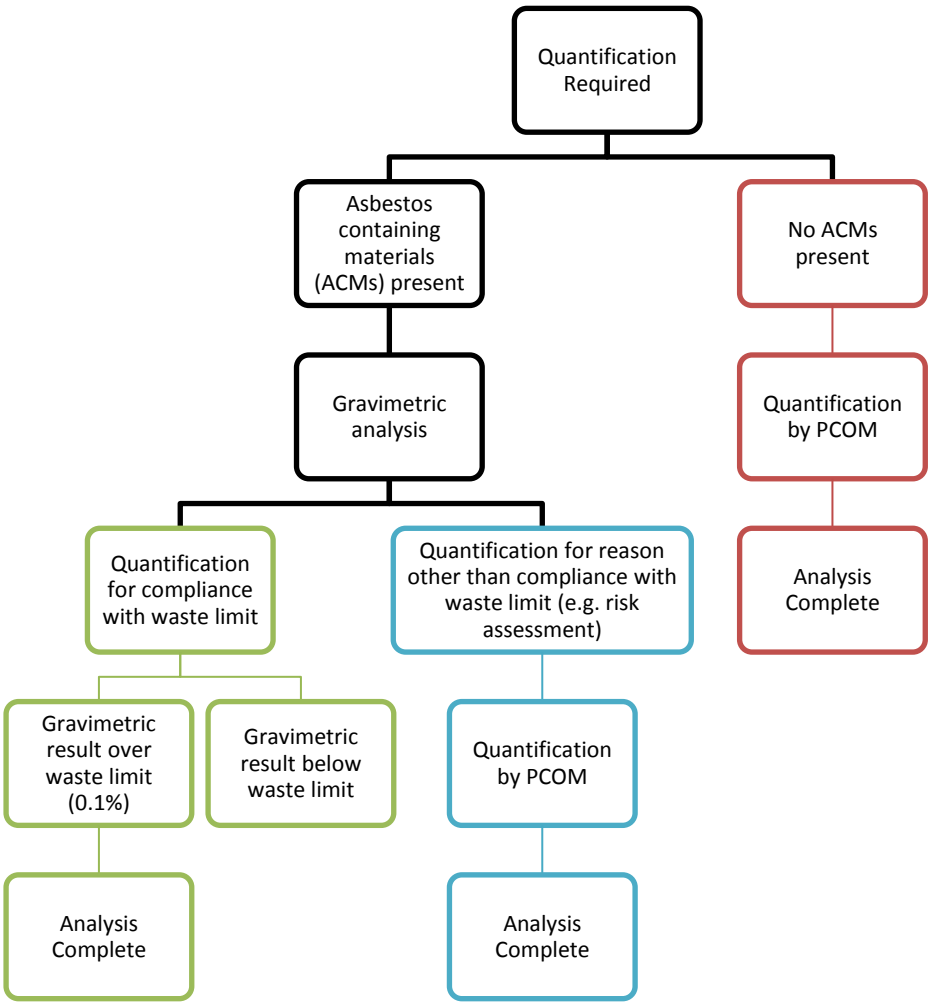
Step	Procedure	Notes

The SCA format uses this three column approach, but there is no point in doing this until we have agreed on the text. HD

The client requirements should be established before analysis begins. WM2 states that finding even one small piece of ACM in a sample will consign the whole load as hazardous. In addition, for waste classification and carriage, it is important if the asbestos is bound or not.

The following flow chart may be used to determine the analysis required on each sample.

Extra Step?



7.1 Sample preparation

7.1.1 All preparations must take place within the Safety Cabinets under full extraction.

7.1.2 The whole, as received, sample should be spread across a clean tray and evaluated visually for the presence of ACM and fibres, which should be removed for further examination using PLM, according to HSG 248 for identification.

If asbestos is identified, the fragments and fibres removed for ID should be retained and returned to the sample for quantification by gravimetric analysis as in 7.2.

7.1.3 deleted

7.2 Gravimetric analysis

7.2.1 Inside a safety cabinet, empty the sample into a suitable weighed tray, reweigh, and record the weight.

7.2.2 For soil samples, manually remove components such as brick, concrete or pebbles of > 10 mm (approximate) in size. Place these components in a weighed dish, re-weigh and record the weight.

For samples which produce < 20 g of fines are not suitable for this method.

7.2.3 From the residual sample, remove a representative subsample of approximately 20 - 100 g to a weighed dish, re-weigh, and dry in an oven between 40°C and 110°C. Record the weight after drying to calculate the moisture content of the residual sample. If < 50 g, use the whole of the residual sample.

7.2.4 Carefully examine the sample and remove any ACM and fibre bundles and transfer to a weighed dish for each type of visually similar ACM. Re-weigh the dish and use the weights to calculate the weights of ACM.

7.2.5 The residual fraction should be retained for further detailed analysis, as required.

7.2.6 The approximate mass percentage of asbestos in the sample resulting from the ACM is given by the formula:

$$\sum \left(\left(\frac{A}{S} \times 100 \right) \times \left(\frac{C}{100} \right) \right)$$

Where A = the weight of each type of ACM
S = sample weight
C = the asbestos content of each ACM based on the 'worst case' value given in HSG264

7.3 Detailed gravimetric analysis

A representative weighed portion (if possible 20 g +) of the fines should be selected for detailed examination under stereo binocular microscope. During this examination it may be

possible to hand pick and weigh small fragments of ACM and/or asbestos fibre bundles that were not identified during the initial examination of the bulk sample. If such materials are recovered these should be transferred to suitable containers and weighed on a five place (at least) analytical balance. It is necessary to use a more accurate weighing instrument for this stage of the analysis due to the small weights of material that are likely to be involved. Even using a relatively sensitive weighing machine it is not practical to attempt to establish weights of asbestos recovered from a soil sample below 1mg.

Results for this stage of the analysis are calculated by expressing the weight of any asbestos recovered as a percentage of the weight of the sub-sample selected for detailed analysis. 1mg of asbestos recovered from 20g of fines represents 0.005%.
This percentage can then be applied to the total weight of the fines in order to estimate the total mass of asbestos in this fraction of the sample, and then adjusted back to the original sample.
If during detailed analysis of the sub-sample, asbestos fibres are detected but there are either too few and/or they are too fine to hand pick and weigh then these should be quantified using the fibre counting/sizing method by PCOM.

It is recommended in HSG 248 that the start and finish times for this method should be at least 15 minutes apart, dependant on the sample type.

7.4 Free fibre analysis using PCOM

7.4.1 Weigh between 1 and 5 g of the residual material from 7.3 into a suitably sized conical flask, and record the weight.

7.4.2 Add water in a ratio 1:200 solid:liquid (depending upon the sample matrix).

7.4.3 Vigorously mix the sample for a minimum of 30 seconds until the sample is completely dispersed.

7.4.4 Leave the sample to stand for 10 seconds to allow the denser material to settle.

7.4.5 Set up the filter assemblies with 25 mm diameter, 0.8 micron pore size filter papers, using blunt nose tweezers.

7.4.6 Label each slide with the appropriate sample identification.

7.4.7 After the 10 seconds settling period, using a calibrated autopipette, take a 1 ml aliquot from the mixture, from approximately 3 cm below the surface, and deposit it onto the filter. The volume removed may need to be less than 1 ml, depending on the 'muddiness' of the solution.

7.4.8 Add approximately 5 ml of water to the filter to mix the aliquot and ensure even distribution on the filter. Filter this mixture using the vacuum pump.

7.4.9 Remove the filter from the filter assembly and place onto the labelled microscope slide.

7.4.10 Allow the filters to dry, ensuring the filters do not curl, before clearing and fixing using the acetone / triacetin method described in section 7.4.11.

7.4.11 Clearing and fixing microscope slides

Acetone vapour is highly flammable and slightly toxic. Wear gloves for this stage to prevent acetone vapour coming into contact with skin

7.4.11.1 Ensure the acetone vaporiser is on, checking that it is at the correct temperature.

7.4.11.2 Position the filter paper on the microscope slide underneath the outlet of the acetone vaporiser and inject acetone slowly into the hot block so that the acetone emerges in a steady stream over the filter.

7.4.11.3 Place the cleared filter onto the hot block for a few seconds to allow any excess acetone to evaporate.

7.4.11.4 Place a drop or two of triacetin onto a clean coverslip using a micropipette or other suitable dropper, invert the slide and lower the filter onto the coverslip.

7.4.11.5 Place the mounted slide onto the hot block until the triacetin has cleared, and then evaluate the filter using phase contrast optical microscopy (PCOM).

7.4.12 Evaluation by PCOM

7.4.12.1 The microscope must be adjusted and used in accordance with the manufacturers instructions, and the analyst must check its performance at the beginning of each counting session (or more frequently if any adjustments have been made):

7.4.12.2 Measure the diameter of the Graticule- this should be 100 +/-3.

7.4.12.3 Remove the stage micrometer and replace it with the HSE test slide.

7.4.12.4 Centre and focus the test slide using phase contrast microscopy

7.4.12.5 Check using the Bertrand lens that the phase rings are concentric and centred. Adjust if necessary.

7.4.12.6 Check and re-adjust the field iris and condenser height at the working magnification to obtain köhler illumination.

7.4.12.7 Check that a minimum of band 5 is visible by traversing from the most to the least visible.

7.4.12.8 The focus and condenser focus will need readjustment before each filter is evaluated.

7.4.12.9 200 graticule areas on the slide must be counted, although counting can stop if the analyst reaches 100 fibres, provided at least 20 fields have been evaluated, or after 200 fibres have been counted, regardless of the number of graticules counted.

NB A maximum number of 8 samples per day per analyst is permitted.

If any of the slides are uncountable for any reason, for example if the analyst tried to clear the filter before it was fully dry, then extra aliquots should be taken from the mixture to replace the unusable slides.

If the slides are uncountable due to excessive particle loading, then a new set of slides should be prepared.

7.4.13 PCOM analysis

7.4.13.1 Place one of the slides to be evaluated onto the microscope stage.

7.4.13.2 Ensure all the appropriate information is recorded for the sample (mass of sample, volume of mixture, diameter of graticule and the weight of the weights of all the subsamples used and materials removed).

7.4.13.3 Graticule areas for counting must be chosen at random to avoid bias and to be representative of the exposed filter area. Fields lying within 4mm of the filter edge should not be counted. Fields should be rejected if a filter grid line obstructs all or part of the field of view, or if more than half of the field is obscured by large particles.

7.4.13.4 Where possible, each fibre observed must be classified as amphibole, chrysotile, or non-asbestos using the extinction and sign of elongation characteristics, as described in MDHS 87. Straight or gently curved fibres which cannot be confirmed as non-asbestos (e.g. because they are too fine) should be assumed to be amphibole asbestos, while curled fibres should be assumed to be chrysotile.

7.4.13.5 If an asbestos fibre is deemed ‘countable’, the fibre must be sized using the graticule, and the measurements recorded. A respirable fibre is defined as an amphibole or chrysotile fibre which is 5µm or more in length, and has an aspect ratio of greater than 3:1. Fibres should be counted regardless of their contact with other particles, and all fibres should be counted.

7.4.13.6 Fibre dimensions for each fibre should be recorded to the nearest 5µm for length, and 0.5µm for diameter. Only those parts of the fibre which lie inside the graticule area should be measured. If one end of the fibre is in the field, but the other end is outside, record half the length of the fibre.

7.4.13.7 Analysts should keep track of the number of fields counted using a tally counter.

7.4.13.8 On completion of the sample, the total number of graticule areas evaluated (normally 200) should be recorded.

For a full explanation of counting rules, and examples please see Appendix 1

7.4.14 Calculation of results

The overall mass percentage of asbestos is given by the formula:

$$\left(\frac{A\,W\,(\sum V\rho^{A}\,+\,\sum V\rho^{C})}{aNqS}\times 100\right)\times F$$

Where:
ρ^{A} = average density of amphibole fibres (3.3 x 10^{-6} µg µm^{-3})
ρ^{c} = density of chrysotile (2.5 x 10^{-6} µg µm^{-3})
V = volume of fibre (µm^{3})
W = volume of mixture (ml)
A = area of filter
S = Weight of soil in suspension (µg)
a = area of graticule (mm^{2})
N = number of graticules evaluated
q = Vol aliquot on filter (ml)
F = Material removed correction factor

The material removed correction factor (F) is calculated using the following equation:

$$F=\frac{(T-g)}{T}$$

Where:
T = total dry weight of sample
g = mass of material removed fraction of sample

The purpose of this correction factor is to adjust the result to take into account large non-asbestos items within the sample such as stones which were too large to be analysed by PCOM and which would therefore bias the result.

7.4.15 Potentially Respirable Fibres (optional)

To produce the result, each fibre counted during quantification is checked to see whether it conforms to the definition of a respirable fibre as defined in HSG 248, that is, greater than 5µm in length, narrower than 3 µm in width, and with an aspect ratio of greater than 3:1.

The number of potentially respirable fibres is calculated by first calculating the number of fibres per ml of the mixture using the following equation:

$$Fibres\ per\ ml=\frac{1000\times N\times F^{2}}{V\times n\ \times G^{2}}$$

Where:
N = the number of respirable fibres counted
F= the filter diameter
V = the volume of the aliquot
n = the number of graticules counted
G = the graticule area

The number of fibres per ml of mixture is then converted to the number of fibres per gram of sample using the following equation:

$$Fibres\ per\ g=Fibres\ per\ ml\times\left(\frac{V}{S}\right)$$

Where:
V = the volume of the mixture (ml)

S = the mass of the sample in the mixture (g)

7.5 Reporting

Reports should include the following information, as a minimum:

- Total mass % of asbestos
- Gravimetric (ACM) % of asbestos
- Free fibres % of asbestos
- Asbestos type (from PLM analysis), if required
- Respirable fibres, if required
- Any anomalies or problems, e.g. clay or chrysotile clumping

7.6 Quality Control

QC schemes should comply with LAB 30 and HSG 248 – Technical Bulletin 1 from LAB 30 now incorporates asbestos in soils.

Analysts performing quantitative analysis should participate in their internal laboratory quantification QC scheme.

Filters for fibre counting must be checked by performing blank counts prior to use. Checked batches of filters will have the assigned batch number and the initials of the analyst performing the blank count on the base of each box of 25 filters. See appendix 1 for more information.

7.6.1 Gravimetric QC scheme

The Quality Manager should retain a selection of asbestos containing materials of a known weight, which, on a monthly basis, will be used to spike two soil samples which will then be issued to the laboratory for analysis by each authorised analyst.

The results should fall within the margin of error calculated for the method.

7.6.2 PCOM QC Scheme

The HSL has recently set up a PT scheme for quantifying asbestos in soil, and the laboratory should participate in this scheme. This scheme is known as the Asbestos in Soils Scheme (AISS), and this covers both identification and quantification.

In addition, the laboratory should establish a fibre counting QC scheme based on the recommended internal QC scheme for asbestos air testing.

The Quality Manager should retain as selection of slides at a range of concentrations and fibre types.

Each slide in the QC scheme should be validated as suitable for use by repeated counting (a minimum of 10 counts per slide) to gain a laboratory reference value (LRV). The LRV is the average result (mass of asbestos per mm^2) for all of the counts on the slide.

Each month the Quality Manager should issue a QC round, consisting of 4 slides at a mixture of fibre densities representative of typical samples encountered by the laboratory , for example 2 low density slides, 1 medium density slide and 1 high density slide.

The analyst should use a 'QC Counting' spreadsheet to record the data for each QC round. On completion the spreadsheet should be saved and emailed to the Quality Manager.

For each QC round, the analysts' performance value (PV) should be calculated for each slide by comparing the analyst results to the LRV for that particular slide using the following formula:

$$PV = \frac{Analyst\ Result - LRV}{ESD}$$

Where:
Analyst Result = the mass/mm^2 calculated by the analyst for the slide
LRV = the Laboratory Reference Value for the slide (mass/mm^2)
ESD = the Expected Standard Deviation for the slide

The rolling modulised (ignoring +/- signs) mean of the previous six performance values should be also be calculated.

Each individual counter's performance should be checked after the completion of each QC round against the following criteria:

- No individual performance value to be outside the range 0f -2.0 - +2.0
- A running modulised mean for an individual counter, of the last 6 performance values to be maintained at <1.0
- At least 80% of the performance values from the last four months to lie between -1.5 and +1.5

Failure of a counter to meet the acceptance criteria must result in appropriate action by the Quality Manager.

A counter returning a performance value outside the range -2.0 - +2.0 should be required to recount the slide as soon as possible, and the counter should be temporarily withdrawn from the authorised list (without amendment to the list of authorised counters). If the recount allows the counter's performance to be assessed as satisfactory then no specific further action is necessary. If the recount still leaves the analyst outside the acceptable criteria then action must be taken to identify and correct the problem. If retraining takes place, the counter should be required to count 6 reference slides, all of which must meet the acceptance criteria (performance value and mean modulised performance value for the six slides) in order to be returned to the authorised list. If it is anticipated that this process will take more than one week to complete, then the counter should be formally removed from the authorised list.

Assessment of the third criteria (At least 80% of the performance values from the last four months to lie between -1.5 and +1.5) should be flexible, to allow the Quality Manager the opportunity to interpret the reasons for failing to comply. It should be remembered that the possibility of 'rogue' results when dealing with low density slides is quite high, especially where slides containing chrysotile are concerned. The QC scheme should not result in analysts requiring constant retraining.

When an individual fails to meet any of the criteria laid down, all observations and corrective actions must be documented. Where the defined criteria are overruled (see previous paragraph), then justification for this action must also be documented.

Each week, the Quality Manager will issue 1 slide which is in the process of being validated. The analysts' counts on the unvalidated slide should be used as data for the validation of that slide.

For full information on the QC scheme see Appendix 1.

7.7 WASTE DISPOSAL

7.7.1 After examination, the remaining subsamples should be double bagged within the controlled environment cabinet.

7.7.2 Following analysis by PCOM, the residual mixture used should be filtered first through a coarse filter to remove large particles. The filtrate should then be filtered through a 0.8µm filter before being disposed of.

7.7.3 Slides will be kept for 6 months, after which they will be disposed of as asbestos waste.

7.7.3 Residual subsamples should be stored in a controlled area and retained for a minimum of six months.

7.7.4 All washings should be filtered and the filters disposed of as asbestos waste.

7.7.5 All waste samples, filter papers and Petri dishes should be placed in disposal bags inside the cabinets.

7.7.6 All waste should be disposed of in a red bag clearly marked as ASBESTOS WASTE. This should be sealed and placed inside a clear bag similarly marked.

7.7.7 This bag should be transported to a suitable waste disposal site with the appropriate documentation and disposed of as asbestos waste.

When waste is removed from the laboratory, a special waste Section 17 document must be completed or collected by a licensed transportation contractor with a copy retained by the laboratory.

Address for correspondence

However well procedures may be tested, there is always the possibility of discovering hitherto unknown problems. Analysts with such information are requested to contact the Secretary of the Standing Committee of Analysts at the address given below. In addition, if users wish to receive advanced notice of forthcoming publications, please contact the Secretary.

Secretary
Standing Committee of Analysts (National Laboratory Service)
Environment Agency
56 Town Green Street
Rothley
Leicestershire
LE7 7NW
www.environment-agency.gov.uk/nls

Environment Agency
Standing Committee of Analysts

Members assisting with this booklet

Rob Blackburn	ATAC and ARCA
Chris Caird	ALS
Steve Clark	IOM
Hazel Davidson	DETS
Laurie Davies	HSL
Steve Forster	IEG Technologies
Paul Gribble	Alcontrol
Phil Helier	Chemtest
Matt Holt	REC
John Leeson	DETS
Tim Platt	Exova
Clare Stone	I2
Rhodri Williams	Alcontrol
David Wood	SAL

Appendix 1

Fibre Counting Rules

Selecting fields for evaluation

Graticule areas for counting must be chosen at random to avoid bias and to representative of the exposed filter area. Fields lying within 4mm of the filter edge should not be counted. Fields should be rejected if a filter grid line obstructs all or part of the field of view, or if more than half of the field is obscured by large particles.

To help prevent re-counting fibres, it is helpful to use the mechanical stage to move across the stage in straight lines.

When evaluating each field, it may be helpful to examine each quarter graticule area individually, focusing up and down to ensure that all fibres are observed.

Counting fibres

A countable fibre is as a fibre which is 5µm or more in length, and has an aspect ratio of greater than 3:1. Fibres should be counted regardless of their contact with other particles.

Where possible, each countable fibre observed must be classified as amphibole, chrysotile, or non-asbestos using the extinction and sign of elongation characteristics, as described in DETS 082 and HSG 248. Straight or gently curved fibres which cannot be confirmed as non-asbestos should be assumed to be amphibole asbestos, while curled fibres should be assumed to be chrysotile.

If a fibre is deemed 'countable', and it decided that the fibre should be classed as asbestos, then the fibre must be sized using the graticule and rotating stage, and the measurements entered directly onto the excel spreadsheet.

Fibre dimensions for each fibre should be recorded to the nearest 5µm for length, and 0.5µm for diameter.

Only those parts of the fibre which are inside the graticule should be measured.

Analysts should keep track of the number of fields counted using a tally counter.

Counting can stop if the analyst reaches 100 fibres, provided at least 20 fields have been evaluated, or on reaching 200 fibres, regardless of the number of graticules counted.

On completion of the sample, the total number of graticule areas evaluated should be entered onto the spreadsheet.

A fibre which is split should be sized according to the length of longest individual branch.

The width of split fibres should be recorded as the narrowest width where all of the fibres are together (i.e. a place along the length of the fibre before it began to split), even if this point of measurement is outside of the graticule area.

Chrysotile fibres are very difficult to measure, especially lengthways. They should be estimated using any straight sections of fibre available.

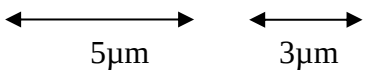
A fibre which crosses the middle of the entire graticule area should be recorded as 100µm long, even it not straight.

Individual branches of split fibres should not be counted individually as this will tend to greatly overestimate the mass of fibres present, split fibres should be assessed as one fibre.

Very fine fibres, which are clearly finer than 0.5µm, should still be recorded as 0.5µm wide.

Examples of measuring fibres and fibre counting rules are shown below:

Fibre definitions

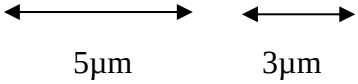


1 countable fibre

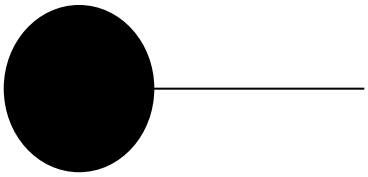
Not countable (too short)

1 Countable fibre (length measured along curve)

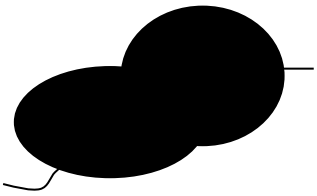
1 Countable fibre



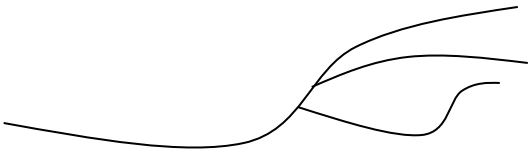
Not countable (aspect ratio <3:1)



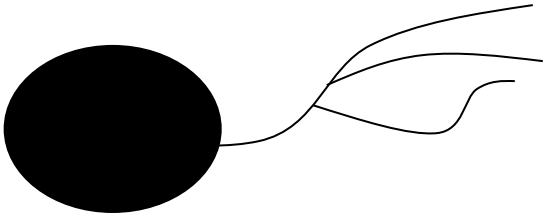
1 countable fibre (particle ignored)



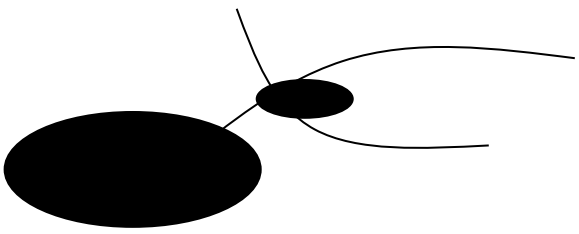
Not countable (visible parts of fibre too short)



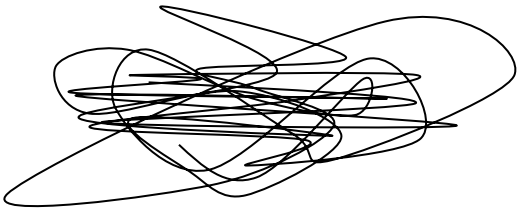
1 Countable fibre, width assessed where all fibres are together



1 countable fibre (particle ignored)

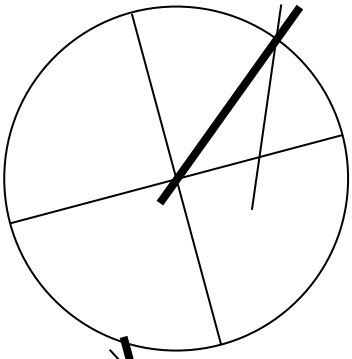


2 countable fibres



No countable fibres distinguishable, and whole clump >5µm wide

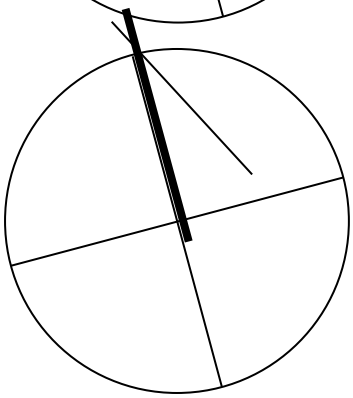
Measuring fibres



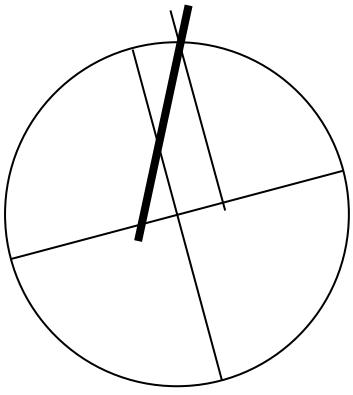
Two large fibres can be seen in this field. In order to assess their size, they should be compared to the measurements on the graticule.

The graticule has two guides, one with increments of 5µm and one with increments of 3µm

The 5µm guide should be used to measure the length, and the 3µm guide used to measure the width.

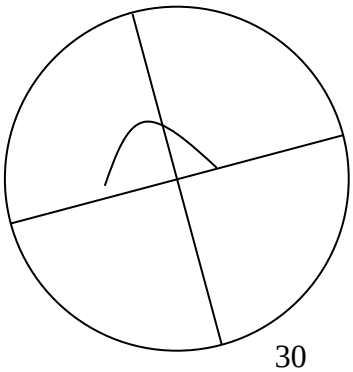


By rotating the stage, fibres can be lined up with the rulers, as in this diagram, making it easier to measure the dimensions.

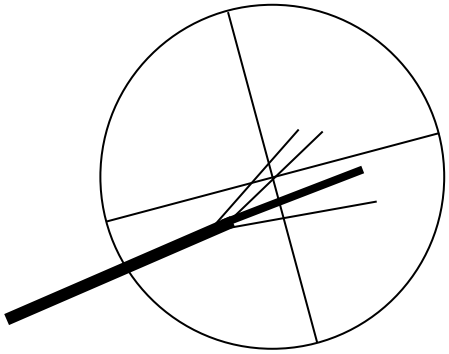


After measuring the first fibre, the stage should be rotated again to measure the second fibre.

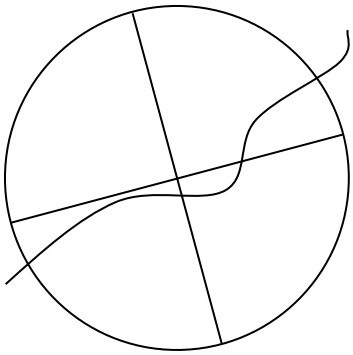
This fibre does not lie directly on the guide, but by aligning it parallel to the guide the length can be estimated with reasonable accuracy.



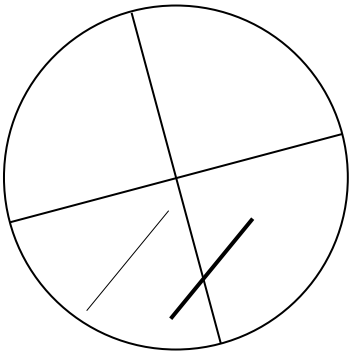
Measure curved fibres by assessing the lengths of the straighter parts of the fibre



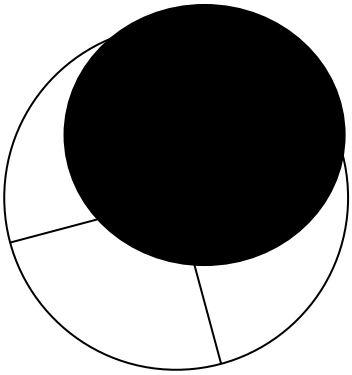
Measure the length of split fibres as the length of the longest part, and the width as the narrowest part of the fibre where all the branches are still attached to the main fibre, even is this point is outside of the graticule area.



Curved fibres passing right through the graticule should be recorded as 100µm long.



Both the fibres in this field are less than 0.5 µm in width, and they should both be recorded as 0.5µm, despite one fibre being obviously much finer than the other.



If more than half of the field is obscured by particles then the field should not be counted