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Hydraulic fluid power — Calibration of liquid automatic particle counters — Procedures used to certify the standard reference material SRM 2806

Transmissions hydrauliques — Étalonnage des compteurs automatiques de particules en suspension dans les liquides — Procédures utilisées pour certifier le matériau de référence normalisé SRM 2806

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

Attention is drawn to the possibility that some of the elements of this Technical Report may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TR 16144 was prepared by Technical Committee ISO/TC 131, *Fluid power systems*, Subcommittee SC 6, *Contamination control*.

Introduction

Solid particulates are a major contributor to wear in hydraulic systems. The fluid power industry, the aerospace industry and the military sector utilize optical automatic particle counter (APC) technologies to assess the level of hydraulic oil contamination by suspended particulate. The amount of contamination is often related to the integrity of the system and the usage of the fluid. APCs are also employed in various oil filter testing operations by the manufacturers and the users. The standard method ISO 4402[1]1) has been used for nearly 30 years to calibrate optical particle counters in terms of particle size as a function of particle concentration.

The calibration material used in ISO 4402:1991 is Air Cleaner Fine Test Dust (ACFTD) produced in the past by a division of General Motors Corporation. This material consists of a polydisperse dust having the largest number of particles, as indicated in ISO 4402:1991, with the size range of 1 µm to 80 µm diameter (particle concentration increases with decreasing diameter). There is a low concentration of particles reported to extend out to approximately 100 µm. Some problems have arisen with the use of ACFTD in such calibration procedures. Firstly, there has been ongoing concern that the particle size distribution is not accurate in the small particle size regime (< 10 µm) of the distribution[2], [3], [4], [5]. Many researchers have noted that there are more sub-10 µm particles in ACFTD than reported by ISO 4402:1991. Secondly, but not less importantly, the production of ACFTD has been discontinued by the supplier.

Thus there is a need to investigate, design and devise a new standard method (*Hydraulic fluid power — Calibration method for liquid automatic particle counters*) using a new Standard Reference Material (SRM)[6]. The National Institute of Standards and Technology (NIST) was requested to develop an SRM for use by the fluid power industry. Users will benefit from improved precision since there is a central source of only one material and increased accuracy resulting from the size characterization[7]. The new SRM, designated as SRM 2806, is composed of ISO Medium Test Dust (ISO MTD) suspended in MIL-H-5606 hydraulic fluid. The number of particles per millilitre greater than specified sizes has been determined for this material.

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1) Cancelled in 1999 and replaced by ISO 11171:1999.

Hydraulic fluid power — Calibration of liquid automatic particle counters — Procedures used to certify the standard reference material SRM 2806

1 Scope

This Technical Report describes the procedures used by the United States National Institute of Standards and Technology (NIST) for the certification of the calibration material SRM 2806, which is used in the primary calibration of liquid automatic particle counters.

SRM 2806 is a suspension of ISO MTD in hydraulic fluid with a number size distribution certified using a scanning electron microscope (SEM) and image analysis techniques.

2 Equipment and material

2.1 Test powder

2.1.1 Standard reference material SRM 2806

The particulate material used is a silica powder made from Arizona desert sand by jet milling and then air classifying to a consistent particle size distribution. Several grades with different size ranges are available and their properties are specified in ISO 12103-1[8].

The powder used to prepare SRM 2806 is an ISO 12103-A3 grade, also called ISO MTD, with supplier batch number 4390C.

2.1.2 Reference materials RM 8631 and RM 8632

Reference materials RM 8631 and RM 8632 are composed of ISO MTD and ISO ultra fine test dust lot numbers 4390C (same lot as the SRM 2806) and 4476 J, respectively. These RMs provide materials to make secondary standards used in support of ISO 11171[9] and SRM 2806[10]. The RM was received in 3,6 kg bottles. This dust was dried and spin-riffled into 147 aliquots, each of 20 mg. The material was examined for homogeneity using optical particle counters after suspension in clean oil.

2.2 Test fluid

Test fluid in which ISO MTD is suspended is a hydraulic fluid widely used worldwide for filter testing. This oil is defined in American national standards as MIL-H 5606 and in French national standards as AIR 3520, and in the NATO specification H 515.

Its physical-chemical properties are defined in annex A of ISO 16889:1999[11].

To ease particle dispersion, a small quantity (50 µg/g) of an antistatic agent is added to the oil so that its conductivity is 1 500 pS/m ± 100 pS/m.

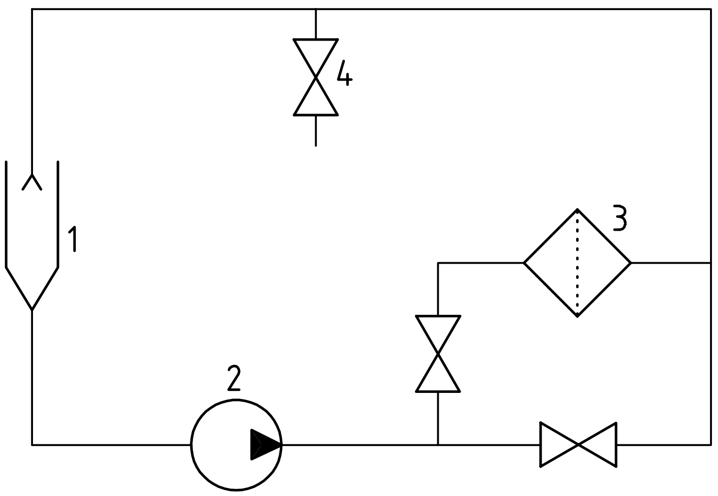
2.3 Sample preparation loop

In view of supplying worldwide demand for several years with the SRM 2806 (supplied in bottles of 400 ml), it was necessary to prepare and store a great number of bottles for further sales.

Because of the settling velocity of the larger silica grains, a special mixing loop was built with mechanical and hydraulic components which were used to eliminate grinding the powder in suspension. It was designed according to the recommendations of ISO 11943[12].

To guarantee bottle sample homogeneity, a supplementary volume of oil was necessary to allow sampling of control bottles used as described in 3.1.2.

The schematic of the sample preparation loop is given in Figure 1.



Key

1 Fluid reservoir (200 l)
2 Circulating pump
3 Clean-up filter
4 Sampling tap

Figure 1 — Schematic of calibration suspension preparation loop

2.4 Membrane preparation equipment

Particles are filtered on 25 mm diameter polycarbonate membranes, 0,2 µm pore diameter using the equipment commonly used for determining hydraulic fluid particulate contamination by gravimetry according to ISO 4405[13] or by microscopic counting according to ISO 4407[14].

2.5 Scanning electron microscope and image analyser

The scanning electron microscope used to examine particles is a JEOL 840. The images were produced by electron backscattering and collected on a MicroVax and analysed using LISPIX, a public domain image processing software developed at NIST. LISPIX currently runs on any computer.

3 Equipment validation

3.1 Sample preparation validation

3.1.1 General

Quality assurance for both production and testing was developed by a task force composed of North American members from two filter manufacturers, a particle counter manufacturer, an independent laboratory and NIST. APC measurements were made by both the independent laboratory and NIST, with NIST performing the data analysis.

3.1.2 Homogeneity testing/batch screening

An experimental sampling design was developed and implemented at NIST to measure the bottle-to-bottle homogeneity and, at the same time, to identify possible systematic errors in the instrumental measurements. In the production process, four bottles (a, b, c, d) were filled at any one time. There were 320 bottles per batch and bottles were numerically labelled sequentially from 1 (a, b, c, d), 2 (a, b, c, d), ..., to 80 (a, b, c, d) as they were produced. Selected bottles from each batch were tested for homogeneity at both the independent laboratory and NIST using APCs with extinction sensors calibrated according to ISO 4402:1991. Four bottles (a, b, c, d) were sampled and analysed from approximately the following four points in the production cycle: 5 %, 30 %, 60 %, and 95 %. Another set of four bottles that were produced directly adjacent to the first four were then analysed. For example, the first 16 bottles 5 a, 5 b, 5 c, 5 d, 25 (a, b, c, d), 50 (a, b, c, d) and 75 (a, b, c, d) were analysed in that order. Then bottles 6 (a, b, c, d), 26 (a, b, c, d), 51 (a, b, c, d), and 76 (a, b, c, d) were analysed all by the same calibrated APC. With three replicates for each bottle, this totalled 96 measurements. Each batch of 320 bottles was subjected to this procedure or a modified version of this test. A batch of material was deemed homogeneous if the coefficient of variation for the number of particles larger than 5 µm, 7 µm, 10 µm, 20 µm and 30 µm did not exceed 4 %, 4 %, 4 %, 5 % and 7 % respectively and there were no systematic variations in the batch. The cumulative particle size distribution was determined for the nominal size range of 1 µm to 80 µm particle diameter and measurements were compared for the same batch of materials.

3.1.3 Homogeneity

To provide high precision measurement capability for a user community, a standard reference material should be as homogeneous as possible. Special efforts were made to assure that this material was made with a low bottle-to-bottle variation within the batch. Within batch variability for the SRM is presented in Table 1 expressed as relative standard deviation for within batch measurements. Figure 2 shows the batch-to-batch comparison in histogram form. The histogram is composed of the mean values of the cumulative particle counts for the same volume of fluid analysed.

Table 1 — Variability found within a batch of material

Greater than size µm	Relative standard deviation %
5	1,1
10	1,3
15	2,0
20	3,8
30	6,7

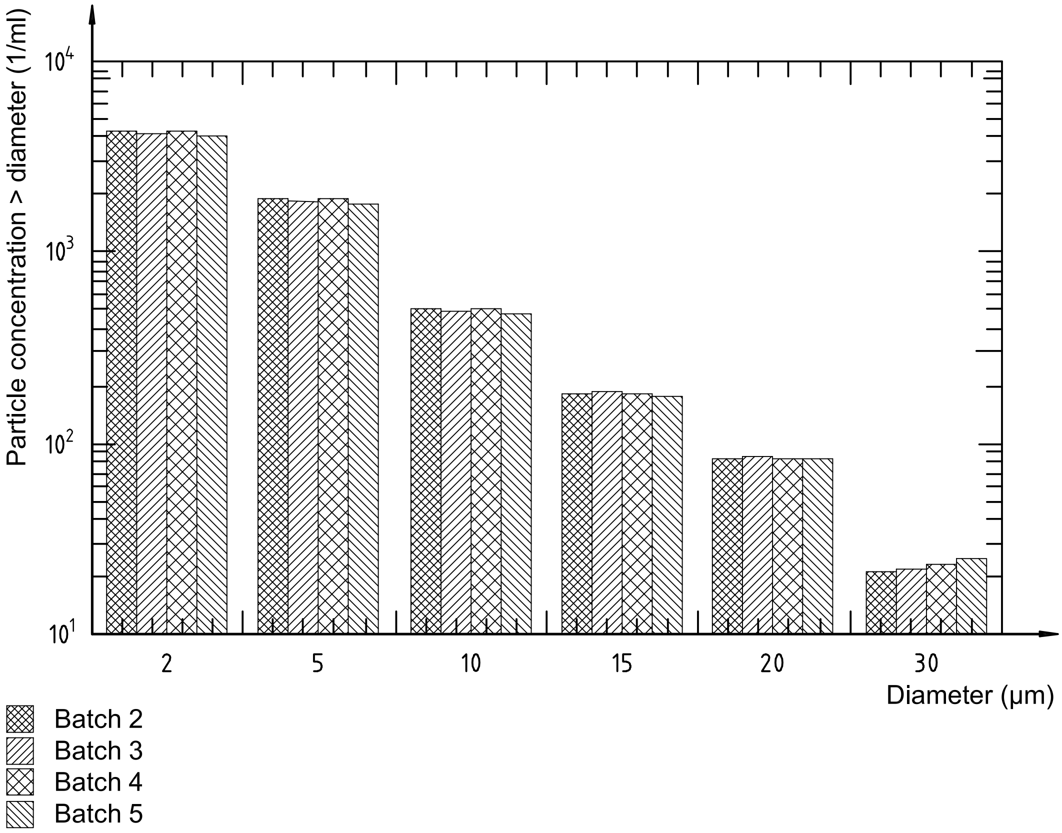


Figure 2 — Particle counts in four batches of ISO MTD to verify bottle-to-bottle homogeneity

3.2 Microscope calibration validation

3.2.1 Microscope calibration

SRM 484d, a NIST scanning electron microscope magnification standard, is mounted in the x and y direction (orthogonal) on the SEM sample stage and used in conjunction with each sample to calibrate the x-y length for particle sizing. SRM 1960, 1 µm polystyrene spheres, were examined by the same procedures used for the dust particles, in order to verify the procedure. Elemental analysis is conducted for a subset of dust particles in the filter sample using energy dispersive X-ray spectroscopy to assure, within the limits of the experiment, that only mineral dust is analysed and that other contaminating particulate material is not present.

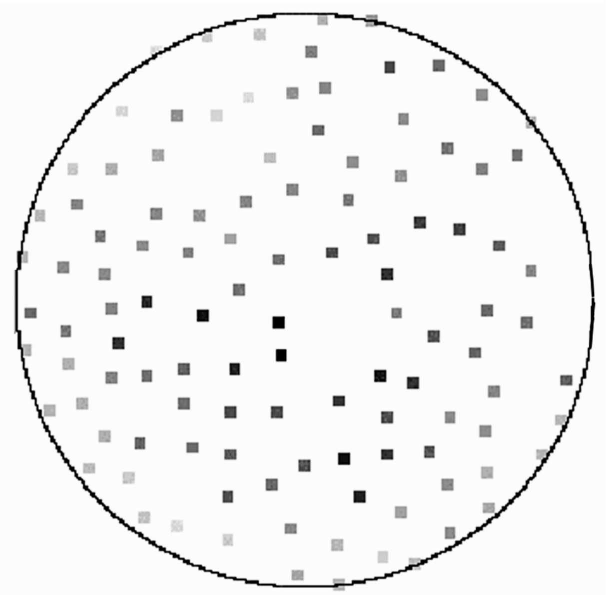
3.2.2 Traceability

Particle size is traceable to a primary measurement, optical interferometry, through the NIST SRM 484d[15]. This SRM standard was imaged for each data set collected and for each magnification. From the certified lengths and the measured number of pixels, a pixel-to-length relation was derived and was used to convert the particle images represented in pixels to area in square micrometres. Uncertainties were determined for these conversions (reported as length uncertainty). They are composed of a combination of uncertainty in the pixel determination and the reported uncertainty in the SRM[10], [15].

3.3 Membrane preparation validation

3.3.1 Sampling from the filter

One component of the total measurement uncertainty results from sampling. Analysis of a large number of fields indicated a non-uniform particle coverage on the filter. Non-uniform particle deposition was observed on many of the filters after particle separation from the hydraulic oil. Presumably, this was the result of vigorous rinsing with the clean solvent that caused particles to be preferentially relocated toward the centre of the filter. To overcome this sampling problem, the SEM was made to select randomly fields-of-view of particles on the filter surface. Figure 3 shows a schematic of a filter with the regions from which micrograph field of view images were sampled and their respective particle counts represented in grey-level format. The darkest fields contain the largest counts and are generally located in the interior region of the filter, while the low-count fields are found closer to the edge of the filter. In this case, a non-random sampling of only interior fields would give an elevated particle concentration. Note that the edge fields of view that overlap the particle-free boundary are included in the sample population. These edge regions have their area corrected by extracting the particle-free area observed in the micrographs.

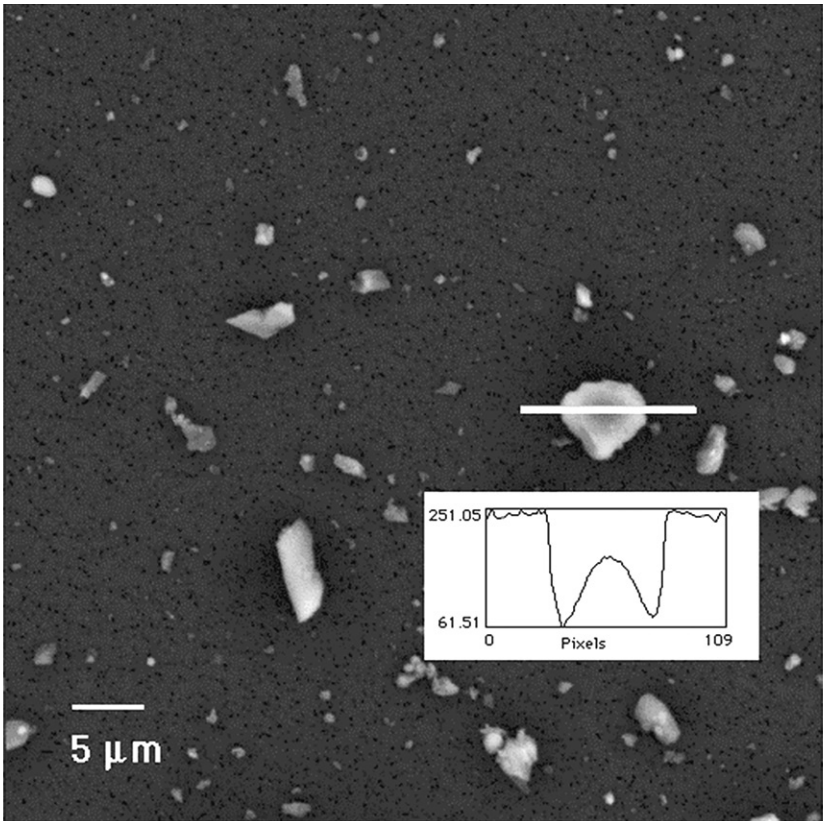


NOTE The darker squares correspond to fields with the highest number of particles counted and the lighter squares indicate low particle coverage.

Figure 3 — Schematic of a filter surface showing the location from which fields were sampled and micrographs obtained

3.3.2 Image analysis

The image analysis was carried out on the original images; none of the features of the particles was altered or enhanced. Thresholding was accomplished visually for each image to maximize the particle thresholded area without introducing background pixels or false particles into the analysis. Once thresholded, the software determined the number of pixels comprising the particles, i.e. the areas. Each magnification that spans a portion of the particle size distribution is analysed separately. The particles have brighter edge regions when compared to the particle interiors as illustrated by Figure 4. The pixel scan across the horizontal line is shown as an insert in the figure. This is observed in electron micrographs because the scattering beam electrons can more readily escape from the particle edges and be detected whereas electrons penetrating the central part of the particle have less probability of detection[16]. Since the edges are bright, they are almost always above threshold and included in the particle. The software “fills” all hollow particles, i.e. particles having pixels below threshold in their central region. Thus, the critical step in determining accurate particle area is to identify the particle boundary or edge. There is a complication in that the true particle edge can never be known to be better than a pixel width. Edge determinations for linear microstructures by electron microscopy for metrology are discussed at length[17]. To minimize the area uncertainty, particles need to be represented by a large number of pixels. For example, a 1 µm sphere is represented by approximately 270 pixels at 3 300 × magnification. One would not want to analyse the same particle at 300 × where the particle would be represented by only 2 or 3 pixels. The lowest number of pixels used in the design analysis is, in most cases, between 13 and 50 pixels.



NOTE The background filter has a grey level of 251 and the edges have a grey level of 60 to 70 while the centre of the particle is approximately 150 to 180. This image was obtained with NIH Image, a public domain software package, and has an inverted grey level assignment with respect to Lispix.

Figure 4 — SEM image of ISO medium test dust showing the particle edge brightening

3.3.3 Projected area diameter of stable oriented particles

The polycarbonate filter material used in this analysis has a planar surface. Consequently, microscopic images are collected for particles laying flat on a planar surface. Particles should settle in their most stable configuration on the filter and thus should exhibit on average their largest projected area. In the APC application, the material is normally measured with the particles suspended in random orientation or oriented with respect to the fluid flow. The values for the particles characterized are in terms of their projected area diameter found from their most stable orientation.

3.4 Membrane and SRM 2806 stability testing

SRM 2806 will be tested at 6-month intervals to ensure that the particle size distribution is not changing with time. APCs calibrated to standard polystyrene spheres will be used for this stability monitoring. A historical record of the size distribution will be made from the time a batch of material arrives at NIST until it is sold. Spot microscopy checks will be performed on selected bottles as necessary if the APC measurements indicate any changes in the material.

4 Test procedure

4.1 Calibration suspension preparation SRM 2806

The SRM material was produced from ISO 12103-A3 medium test dust suspended in hydraulic oil at known concentrations. The ISO MTD, lot number 4390C, specified in ISO 12103 was suspended at a concentration of

2,8 mg of dust per litre of MIL-H-5606 hydraulic fluid. The material was produced in 320-bottle batches, each bottle containing approximately 400 ml of the suspension.

4.2 Membrane preparation

Before microscopy on the individual particles could be performed, particles had to be separated from the hydraulic fluid by filtration. All the apparatus associated with the procedure was carefully cleaned with demineralized particle free water (determined by APC measurements) and rinsed with heptane solvent that was prefiltered by 0,2 µm pore membrane filter. Filtered heptane was used as the clean solvent to remove the hydraulic oil from the filter and to wash the filtering apparatus. The filtrations were performed in a class 100 cleanroom to minimize possible contamination by ambient airborne particles. Polycarbonate filters (25 mm diameter with 0,2 µm pore size) were used to filter the particles from the oil. These filters have high collection efficiency for particles greater than 0,2 µm and provide a smooth, planar surface for electron microscopy[18], [19].

The procedure, adapted from an existing Society of Automotive Engineers (SAE) Method[20] entailed producing three filters per sample for analysis:

1) the filter, solvent and apparatus blank were obtained by flushing several hundred millilitres of filtered solvent through a clean filter while at the same time washing the walls of the filter funnel;

2) filtering a known volume (at standard temperature) of SRM 2806 hydraulic oil suspension through a second filter followed by funnel wall washing;

3) extensively washing the funnel down with filtered solvent onto a third clean filter.

The first procedure provided assessment of the cleanliness of the blank filter material, of the filtering apparatus, of the filtered solvent and of the overall sample processing. Then, in the second procedure, particles in individual bottles of SRM 2806 were resuspended by sonication, mechanical shaking and then resonication. Following resuspension and mixing, 10 ml or 30 ml volumes were carefully pipetted from the bottle and flushed through the filter using the prefiltered solvent. The walls of the funnel and pipette were extensively flushed with solvent. Finally, for the third procedure a new clean filter was installed and the same funnel was washed down again to ensure that all particles had been removed from the walls. All three filters were examined by electron microscopy and the measurement results obtained from them are used in the data analysis.

4.3 Membrane examination and particle counting

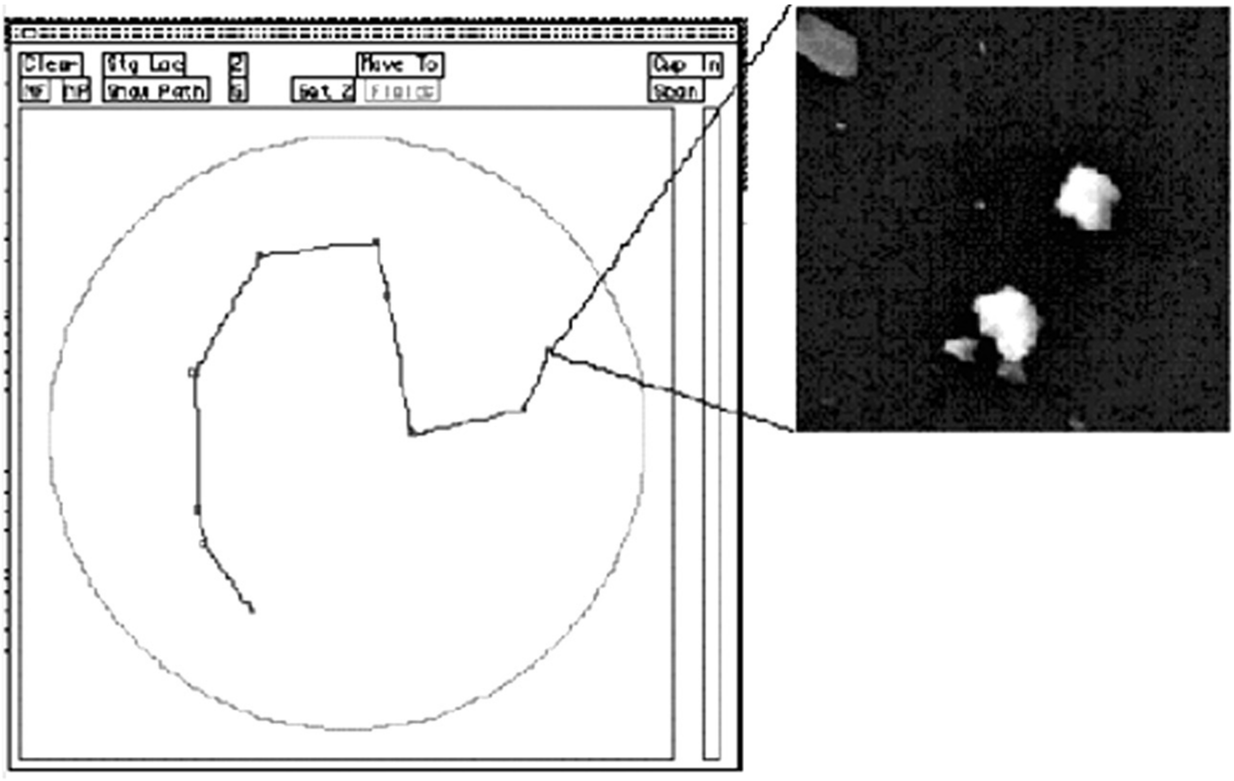
4.3.1 Sampling microscopic fields of view

Sampling is one of the most important elements of the procedure to properly characterize the particulate material. Collecting an accurate representative sample is critical. If sampling is incorrectly done, size distribution analysis can be biased due to a number of problems that are a function of filtering the material. An example of this is large or small particle segregation and nonuniform particle coverage. The sampling process is constrained in that the entire filter cannot be sampled due to time and image storing limitations. Thus a subset of the filter area has to be carefully chosen to provide a representative sample. One way to accomplish this task is random sampling from all possible regions on the sample-containing filter where there are filtered particles. Practically, this means the sampling has to be conducted on the filter in the entire circular region defined by the funnel placement including the regions where the filter is in contact with the funnel edge.

4.3.2 Scanning electron microscopy

The scanning electron microscope (SEM) is used in this work to collect images of the particles that are in turn used to attain the primary particle size/count metrology necessary for this SRM. The SEM is used because it is well suited for imaging particles in the range of particle size of concern, 1 µm to 100 µm. Backscatter electron imaging provides the maximum grey level contrast for subsequent processing of the digital images. The technique is well established[21], [22], [23], [24], 25]. The entire 25 mm diameter polycarbonate filter containing the particle sample is gold-coated using a low temperature plasma source and subsequently mounted onto the sample stage in the SEM. The SEM is computer controlled for automated sample stage movement and image collection.

The software selects a true set of random fields-of-view to be collected and computes the most efficient route to scan across the sample surface. The stage then steps through the sequence, the sample is brought to optical focus, and backscatter electron 12-bit images are collected and stored for each field. This is illustrated in Figure 5 where a random field selection from a circular filter surface is simulated and one of the associated electron images is “grabbed” by the interfaced computer. The gain of the SEM was adjusted for each filter and each magnification to assure the particles have as high a possible contrast over the filter substrate with a dynamic range of 256 without saturation. Gain settings were adjusted to include all features of the filter and particles. The grey level value for the filter (background peak in the histogram) was a nominal 20 to 30 with particle grey levels ranging from greater than 40 to 50 out of 256. All images are archived on two CD-ROMs so that a permanent record of the data is available. Magnifications of 100 ×, 300 ×, 500 ×, 1 500 × and 3 300 × are used to interrogate and span the particle size range of interest. These magnifications correspond to fields of view with areas of approximately 0,8 mm^2, 0,09 mm^2, 0,03 mm^2, 0,004 mm^2 and 0,0008 mm^2. Figure 6 presents four typical micrograph images of the ISO medium test dust on a polycarbonate filter at the four magnifications, Figure 6 a): 3 300 ×, Figure 6 b): 1 500 ×, Figure 6 c): 500 × and Figure 6 d): 100 ×. These micrographs show the number of particles and relative size and shape of the dust material.



NOTE Software randomly selects fields of view and collects micrograph images. Shown here is a 3 300 × image of a dust particle. The analysis path is independent of the random field selection.

Figure 5 — Automated SEM image collection (3 300 ×)

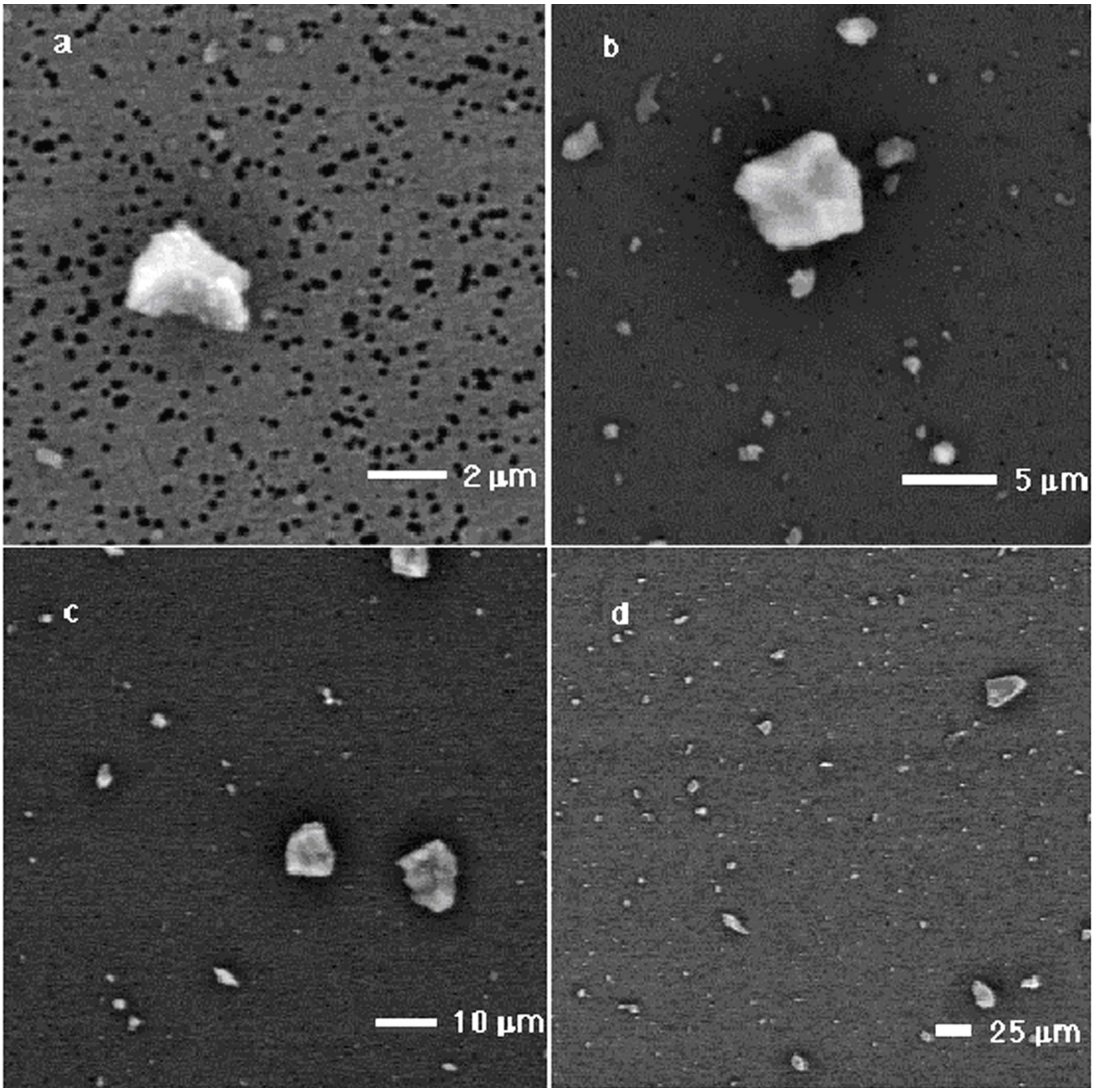
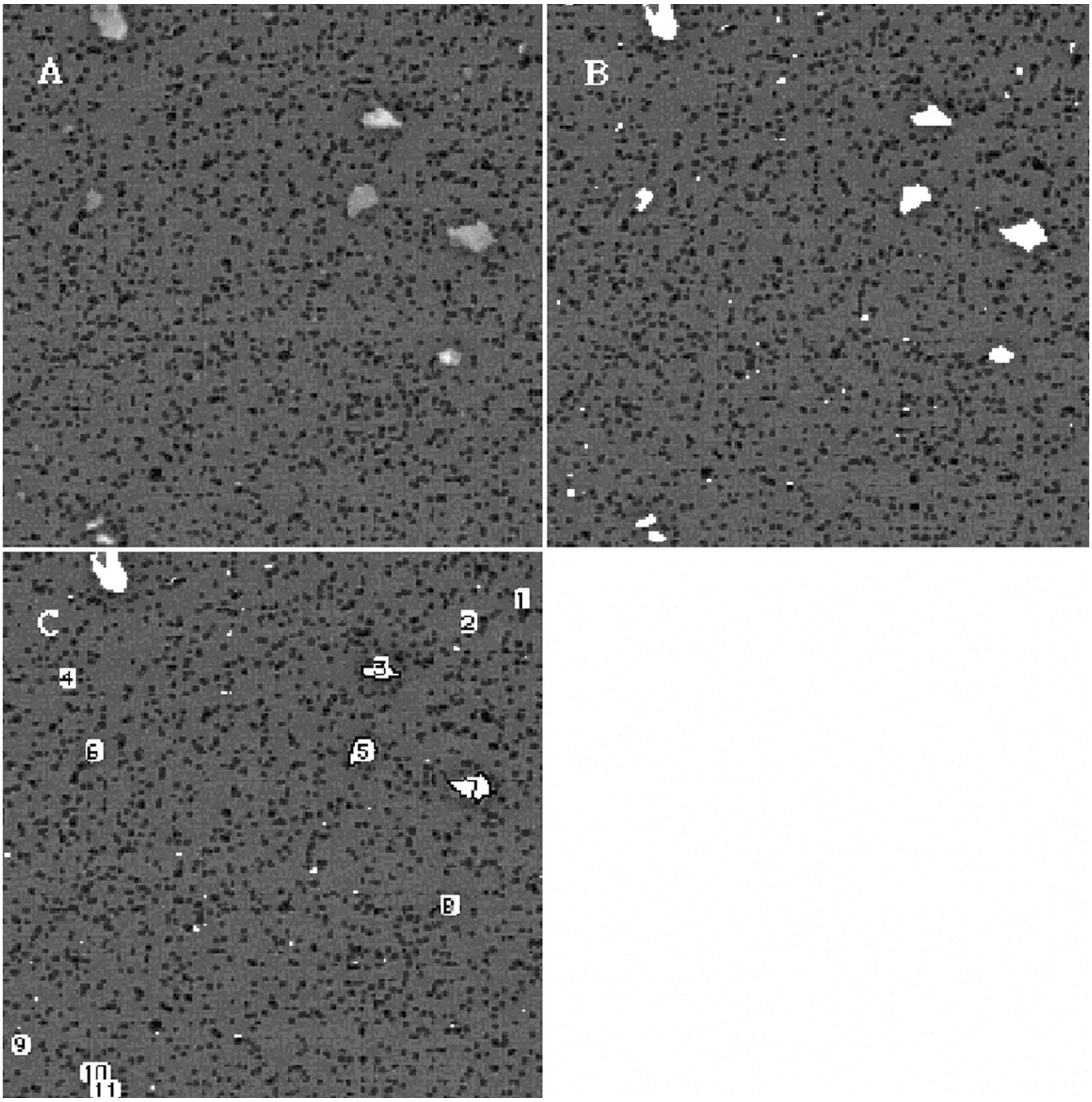


Figure 6 — Typical SEM micrographs of ISO medium dust obtained at the four magnifications: a) 3 300 ×, b) 1 500 ×, c) 500 ×, and d) 100 ×

4.3.3 Image analysis for particle size and number determination

All the particle information is contained in electron micrograph images taken of particles residing on the filter surface. The SEM micrograph images are composed of digitized 12-bit grey level squares commonly referred to as pixels. These images are reduced to 8-bit images by dropping the lowest bit information. Each image contains 512 × 512 pixels. For each magnification, the pixel dimension is determined by SRM 484d. The dust, being composed of mineral particles, has high image contrast due both to the particle topography and to the fact that the average elemental atomic number is larger than the substrate filter material that is composed of carbon and hydrogen. The particles appear as “light” objects because of an increase in electron backscatter yield compared to the filter. The pixels that define the particles' size and shape are identified by grey-level thresholding[26]. Thresholding is done by the analyst for each image, and normally a minimum pixel area object limit is set. For example, one setting would allow particles to be counted if they are composed of more than 5 pixels. After the image is thresholded, the total pixel area for each particle is computed as well as the longest chord length (maximum particle diameter) and particle perimeter. By summing the number of thresholded objects, the particle number for that field of view at the specified magnification is determined from the image. An example of a SEM raw image (A) and thresholded regions (B) is shown in Figure 7. In Figure 7 c), certain particles are accepted for the data set (shown numbered) because their total pixel area exceeds a discrimination level set by the analyst and those that fall below are not counted.

Public domain computer software, MacLispix, developed at NIST, and NIH Image, is used to carry out the image analysis[27], [28]. The relevant information derived from the images is the following: particle area, longest diameter (chord), particle perimeter, number of detected objects, location of the field of view on the filter and location of each particle thresholded. Since the micrograph images are archived, additional analyses can be carried out if required.



NOTE (A) is the original image, (B) is the grey-level thresholded image and (C) shows particles collected and rejected. The small particles were not accepted because of low pixel count discrimination and one large particle was rejected because it touches the image boundary.

Figure 7 — Steps involved in image processing a 3 300 × SEM image of ISO medium dust

5 Data processing

The data were analysed by two independent methods (design and model). The design approach is a straightforward engineering calculation that assumes uniform area distribution derived from correct experimental design and procedure. The model method, as the name suggests, uses the data to develop a model that represents the particle coverage of the filter and assumes a form for the particle distribution[29]. The basis of the analysis in both cases is the raw individual particle area data extracted by image analysis from the image fields and represented in terms of pixels for each SEM magnification. Particle-pixel values can range from six to 1 000 for the largest particles present. For each SEM magnification, the particle area data is put into histogram form to give frequency of occurrence, np, for each particle area possibility. If a particle touches the field edge, it is not analysed. This correction eliminates undesirable fractional particle areas from the data set, but would bias the size distribution toward smaller particle sizes if a correction were not made to the histogram. The frequency of occurrence is corrected to account for particles touching the edge of the field of view and consequently not counted. The correction scales np by a field of view area correction factor (an effective field of view area). Details on the derivation of this correction are contained elsewhere[21]. Since large particles are more likely to touch the edge, they are sampled from a smaller effective area. The ratio of the effective area, A_{eff}, to the field area, A_{f}, is

$$A_{\mathrm{eff}} / A_{\mathrm{f}} = 1 - 2d / L + d^2 / L^2$$

where

d is the particle projected circular equivalent diameter [$d = 2(\text{area}/\pi)^{1/2}$], and

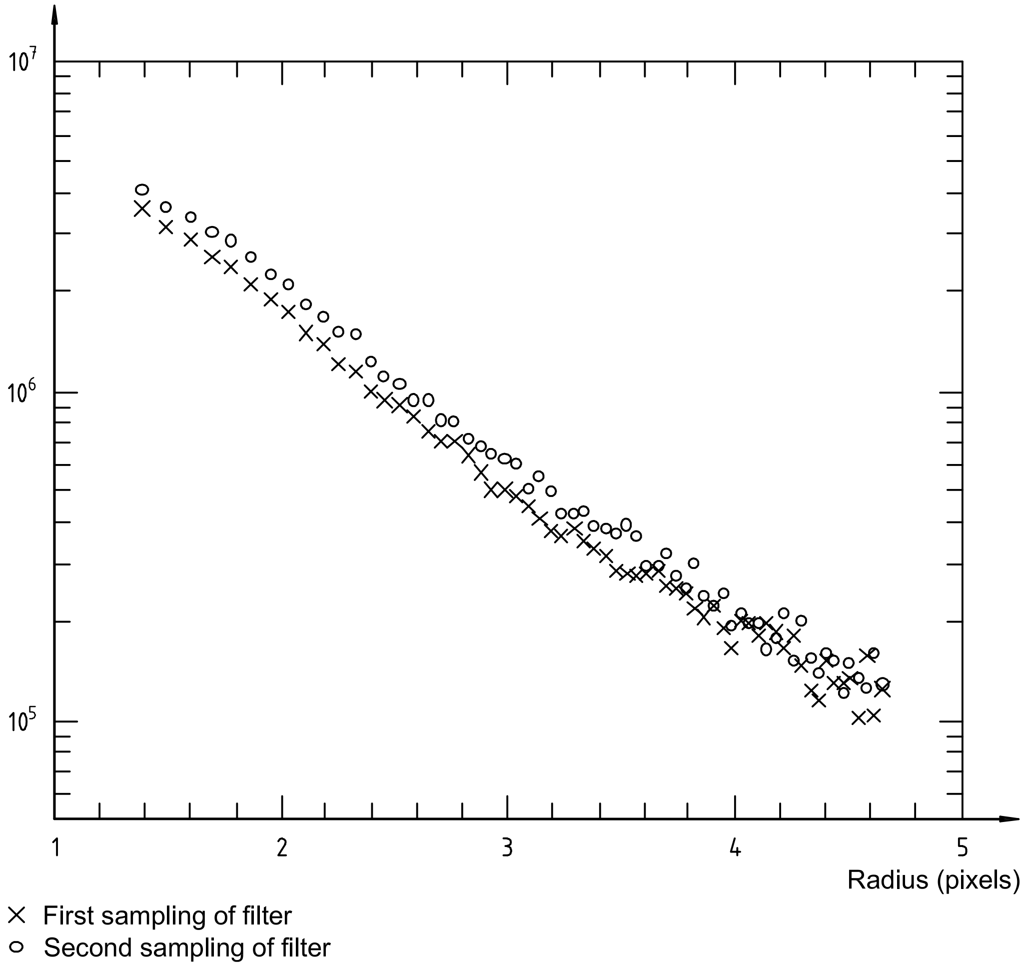
L is the dimension of the field.

This correction is minor for small particles and increases as the particle size increases. The measured size of the particle is dependent upon pixel-to-length conversion for that particular magnification. The diameter in pixels is transformed to micrometres through the pixel to micrometre conversion factor obtained for the given magnification using SRM 484d. The number of particles on the filter sampled per millilitre is calculated from measuring the area covered by the sampled fields of view (corrected for partial fields), the area of the filter and the volume of fluid filtered.

The design approach employs a straightforward summing of the particles. The total number of particles sampled on the filter is obtained by multiplying the field corrected number of particles experimentally observed by the ratio of the total filter area to the field of view area. The model approach derives the particle number density on the filter from a kriging model calculation[29]. Kriging, an analysis method used in the mining industry, was applied to model the number of particles collected on the filter from the discrete fields-of-view data. This analysis results in a smaller sampling uncertainty when compared to the analogous design result. The actual total number of particles on the filter as determined by both methods was found to be similar (within 3 %). The design result was larger.

To more accurately represent the area of the particles, images were collected over five SEM magnifications, and data from four of the magnifications were used in this analysis. The entire cumulative number size distribution is constructed by connecting the data obtained from the segmented distributions that are traceable to their specific magnifications. There are imperfect matchings for the cumulative size distributions found for each separate magnification. For example, the cumulative distribution for 1 500 × does not exactly match the cumulative segment for 500 × and the same is true for the other lower magnifications. One way to understand the reason for this offset is to assume an invariant continuous cumulative distribution of particle sizes and to allow the size offsets appearing from one magnification to another as a result of very slight mismatches in the size scale from magnification to magnification. Mismatch in the concentration scale is less likely because the same filter is sampled albeit in different filter locations, but randomly in both cases and with a sufficient number of fields and particles to represent the filter. The evidence for this size-shift model is that good matches for cumulative data can be obtained by slight shifts in one or the other size scale. The reason for these mismatches is not entirely known, but there are some strong indications that problems arise from both the SEM image collection and the image analysis. For a given filter sample, each magnification constitutes a data set of SEM images, obtained at optimized microscope conditions for the sample and magnification. The next magnification on the same sample will have slightly different gain settings and a slightly different grey level image histogram. Each set of images has slightly different characteristics, such as the location of the background grey level peak that is adjusted during the microscopy to obtain the optimal particle-to-filter contrast required for the analysis. Empirically, the image grey level histograms across magnifications shows some variation. The grey level for the filter substrate is not exactly the same and there is often “speckling” of the substrate that affects the thresholding. The uncertainty across magnifications is further complicated by the image analysis thresholding process. Thresholding is most relevant with respect to obtaining accurate particle areas[30]. The image analysis was standardized as much as possible, but full automation of the process was not possible; there were human judgements made for each image to determine the threshold value.

Thresholds were consistently set to maximize the particle area without incorporating the substrate, i.e. filter domain pixels. The fact that each magnification had slightly different characteristics possibly translated to slight size shifts in the different segments of the size distribution. Evidence is presented for a duplicate analysis on the same filter sample that naturally contains an identical particle population. The same fields were not selected, but all other sampling and image analysis protocols were followed exactly. The data is presented in Figure 8. The size distributions are slightly shifted. On further analysis the histograms of the grey levels were found to be similar but not identical, and there were significant differences in the grey level values of the substrate background pixels. This, once again, is most likely due to gain setting differences in the microscopy.



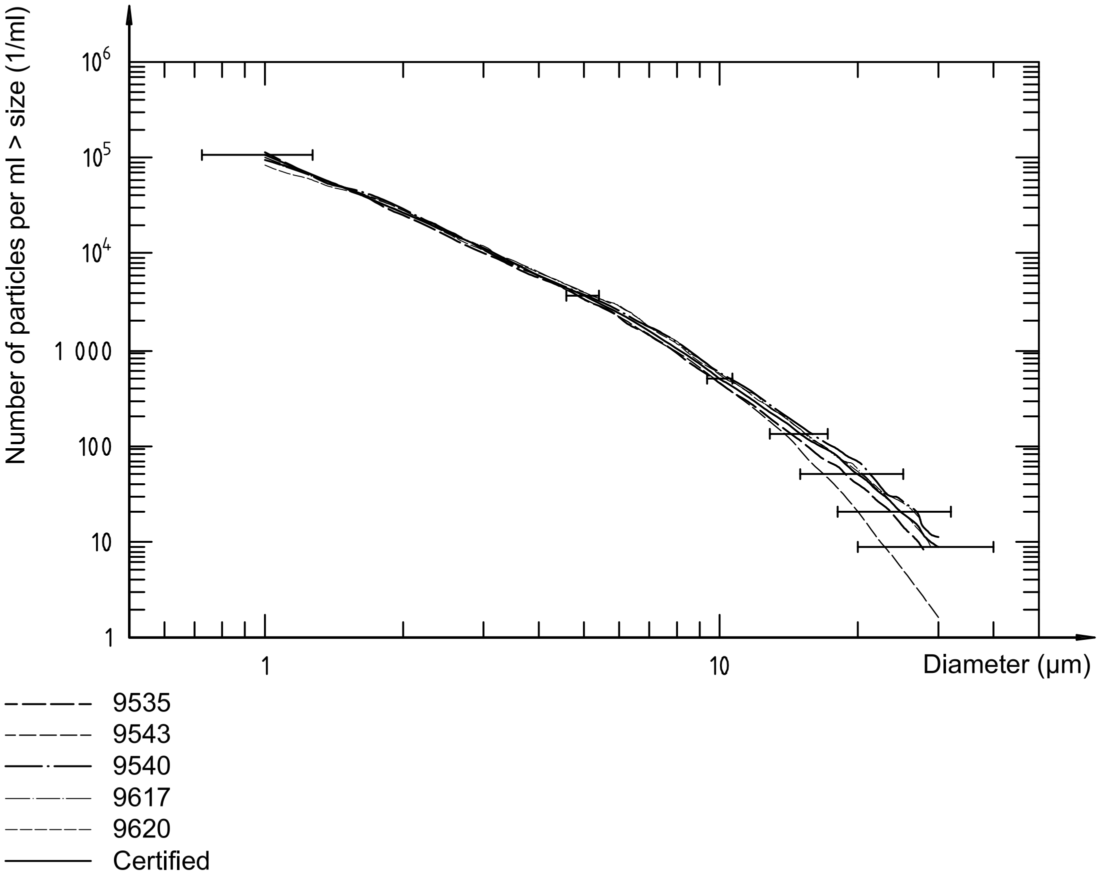
NOTE SEM imaging process optimized in each case. All procedures were duplicated as exactly as possible, but identical fields were not analysed.

Figure 8 — Size distribution derived from two SEM samplings of the same filter at a single magnification, 500 ×

The cumulative size distribution is obtained by summing the number of particles found in each size across all sizes. For the design approach, the cumulative sums of each segment of the distribution obtained at the separate magnifications are spliced together to form the cumulative distribution over the entire size range of 1 µm to 50 µm. In each case, the splicing is done by taking the higher magnification segment as reference and shifting the lower magnification segment. The test for an optimal fit is by differencing the number of counts in the higher magnification to that of the data derived from the lower magnification for 20 to 30 size classes and minimizing the remainders.

This guarantees segment overlap not at just one size, but an optimized melding of the data. The model approach optimized the integral of the particles from the size region of interest to infinity for both magnifications. In both design and model approaches, the size shift was less than 1 µm between segments.

A summary of the cumulative size distribution obtained by both model and design approaches and the associated measurement uncertainties are presented in Table 2. The design results for five filters and the model values are also shown in Figure 9, where the error bars correspond to twice the total uncertainty.



NOTE The error bars correspond to twice the total uncertainty (k = 2) reported for the model values in Table 2.

Figure 9 — Plot summarizing the cumulative number size distribution obtained for five separate filter analyses (9535, 9543, ...) compared to the certified values

Table 2 — Model (certified) and design particle concentration data as related to particle size and associated uncertainties

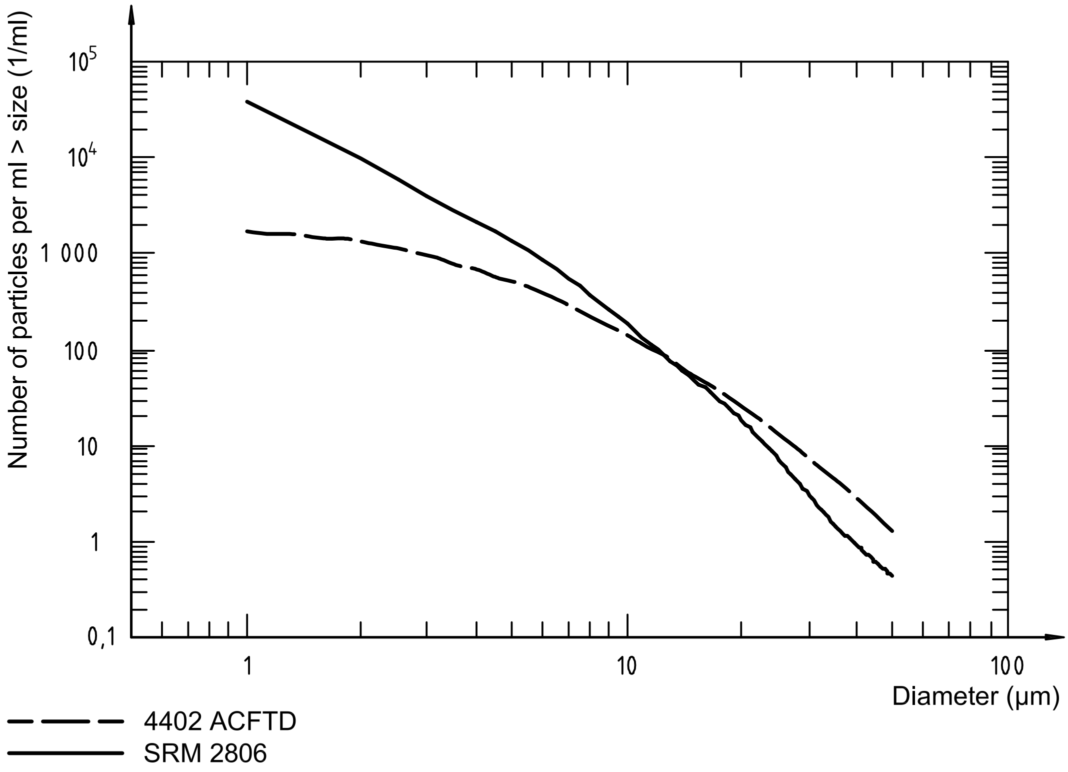
Model particle concentration	Design particle concentration	Projected area diameter[a]	Uncertainty					Combined standard uncertainty	Expanded uncertainty[b]
			of sampling	of volume	of length	of digitization	of size fractionation	U_c	U
1/ml	1/ml	µm	µm	µm	µm	µm	µm	µm	µm
108400	108 960	1	0,004	0,004	0,000 6	0,127	0,051	0,137	0,274
27035	28 094	2	0,006	0,006	0,000 03	0,123	0,049	0,133	0,265
11209	10 918	3	0,009	0,010	0,000 5	0,122	0,059	0,136	0,272
6095	6 506,4	4	0,011	0,013	0,002	0,121	0,094	0,154	0,308
3737	3 860,7	5	0,012	0,015	0,004	0,121	0,167	0,207	0,414
2395	2 918,8	6	0,016	0,016	0,006	0,131	0,246	0,280	0,560
1573	1 814,3	7	0,019	0,017	0,009	0,131	0,236	0,271	0,542
1055	1 220,0	8	0,024	0,018	0,013	0,131	0,210	0,250	0,499
725,8	810,21	9	0,031	0,019	0,017	0,131	0,235	0,272	0,544
513,7	570,90	10	0,041	0,021	0,021	0,131	0,289	0,322	0,643
374,6	423,84	11	0,052	0,023	0,025	0,131	0,351	0,380	0,759
281,0	314,86	12	0,065	0,025	0,029	0,131	0,467	0,490	0,981
216,4	238,97	13	0,081	0,027	0,031	0,131	0,616	0,637	1,273
170,4	190,38	14	0,101	0,030	0,033	0,131	0,810	0,828	1,655
136,8	147,12	15	0,123	0,032	0,033	0,131	1,090	1,106	2,211
111,3	119,62	16	0,146	0,034	0,033	0,131	1,190	1,207	2,414
91,33	97,156	17	0,168	0,035	0,033	0,131	1,439	1,455	2,910
75,29	79,509	18	0,187	0,036	0,034	0,131	1,706	1,722	3,444
62,17	65,359	19	0,204	0,036	0,036	0,131	2,036	2,050	4,101
51,35	53,621	20	0,219	0,036	0,041	0,131	2,509	2,522	5,045
42,40	45,411	21	0,235	0,036	0,047	0,131	2,473	2,488	4,976
35,01	37,919	22	0,255	0,036	0,056	0,131	2,738	2,754	5,508
28,95	31,885	23	0,281	0,037	0,068	0,131	2,953	2,970	5,941
23,99	24,973	24	0,314	0,038	0,081	0,131	3,267	3,286	6,573
19,95	20,272	25	0,356	0,039	0,097	0,131	3,450	3,472	6,945
16,66	16,925	26	0,408	0,040	0,115	0,131	3,603	3,631	7,261
13,98	13,817	27	0,468	0,041	0,135	0,131	3,983	4,015	8,030
11,80	11,202	28	0,539	0,043	0,158	0,131	4,144	4,184	8,369
10,02	8,703 6	29	0,618	0,045	0,184	0,131	4,247	4,298	8,595
8,568	8,328 1	30	0,706	0,047	0,212	0,131	4,914	4,971	9,943

[a] The particle diameter was determined for the dust particles in their most mechanically stable orientation and thus the particle area approaches on average the largest possible value. Literature values for the ratio of mean projected diameters for random and for stably oriented quartz particles (common to ISO MTD) is approximately 0,8.

[b] Each combined standard uncertainty is the square root of the sum-of-the-squares of the standard uncertainty derived or estimated from the measurement process. The expanded uncertainty in the projected area diameters are twice (coverage factor $k = 2$) the combined standard uncertainties. In cases where all uncertainties are type A (uncertainties evaluated by statistical means), then $k = 2$ level defines a confidence interval of approximately 95 %[31].

Errors in the measurement that are related to particle concentration and particle size are expressed in terms of a single parameter, particle size, for convenience of the user community. Uncertainties are present due to particle counting and sampling (Poisson statistics), length determination, digitized pixel representation, fluid volume sampling and size fractionation due to fluid transfer. The uncertainty due to length contributes to the total uncertainty and enters directly into the particle size and indirectly in terms of the field of view area. The length uncertainty (2 % relative) comes from the uncertainty in the measurements necessary to convert digitized image pixels to length by a traceable SRM length standard and the random and systematic error reported in the SRM 484d (1 % of pitch length measured). The size of each field of view contributes to uncertainties in the particle concentration since the number of particles measured is scaled by the ratio of the total filter area to the measured field of view area. This field size is also traceable to SRM 484d. Uncertainty in the length conversion operates in two opposing fashions on the size distribution. An increase in the length conversion, $K_L = K + \delta$, increases the particle size found in the images and at the same time decreases the number concentration. The uncertainty in fluid volume (from pipetting) was conservatively taken to be 2 % of the volume sampled and translates directly into affecting the number concentration. The particle area is determined in terms of the number of pixels having a grey level above the set threshold. The particle edge pixels are the most questionable. The true particle edge lies somewhere within the outer edge set of pixels and cannot be known to better than these edge pixels. The uncertainty is taken to be ± 0,5 pixel (59 nm) on each edge or one full pixel uncertainty for the particle diameter. Digitization uncertainty was calculated for the smallest particles where the magnification (pixels/µm) was the largest. This minimizes the error of the true particle size; as the pixel size approaches zero, the particles are ideally represented and there is no ambiguity with regard to the edge location. All subsequent magnifications are referenced to the highest magnification uncertainty. Size fractionation results from transferring the fluid to the filter and is an estimated uncertainty based on the variability found in the data contained in Figure 9.

Results in Figure 10 show the ISO 4402 data for ACFTD and the SEM results for SRM 2806 ISO MTD. The SRM data are expressed as equivalent spherical diameters while the size in ISO 4402 is expressed as the longest chord. The SRM 2806 data has been scaled by a factor of 2,8 (concentration: 2,8 mg/l) to facilitate direct comparison of the two results at the 1 mg/l concentration levels. The work on the ISO MTD indicates that the size distribution is similar to that found by Masbaum on ACFTD; both Masbaum and others[2], [3], [4] and SRM data indicate the presence of more small (< 10 µm) particles than identified by ISO 4402.



NOTE All values referenced to suspensions with 1 mg/l of material.

Figure 10 — Plot of ISO 4402 ACFTD values and SRM 2806 ISO MTD measurements

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