

行政院國家科學委員會專題研究計畫 期中進度報告

微粒沈積均勻度量測與應用(1/3)

計畫類別：個別型計畫

計畫編號：NSC93-2211-E-002-034-

執行期間：93年08月01日至94年07月31日

執行單位：國立臺灣大學公共衛生學院職業醫學與工業衛生研究所

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報告類型：精簡報告

報告附件：出席國際會議研究心得報告及發表論文

處理方式：本計畫可公開查詢

中 華 民 國 94 年 8 月 24 日

DETERMINATION OF UNIFORMITY OF FILTER DEPOSITS

ABSTRACT

Certain measurement techniques (such as the asbestos method using phase contrast microscopy) require uniform deposits of the sample on a filter. The asbestos fiber analytical methods require such uniform deposition because the analysis only observes small, randomly chosen locations on the filter. The adequate number of total counting fields and other counting protocols (such as equal distance and equivalent area) still require further study. Moreover, the classification and characterization of the uniformity (or non-uniformity) of filter deposits remains poorly defined. Sampling for asbestos and other fibers is currently conducted with a 25-mm diameter filter cassette and a 50-mm long straight tubular inlet, called a cowl.

In this study, a vibrating orifice monodisperse aerosol generator was used to generate methylene blue particles. The aerosols were dried by filtered compressed air and then neutralized by inducing a charge on the droplet stream that emerged from the vibrating orifice. An Aerodynamic Particle Sizer was used to measure the number concentration and size distribution of the generated aerosol particles. Meanwhile, the filter deposits were examined via image processing, combined with statistical methods for defining uniformity.

In order to better define uniformity and make the indicator more universal, the uniformity was defined as the exponential of the negative CV (coefficient of variation) value which was a transformation for easily understanding the uniformity of the filter deposits. The experimental results demonstrated that, when aerosol counting was performed, the equivalent area approach was superior to the equivalent distance approach.

Key Words: fiber counting, image processing, equivalent area, uniformity.

INTRODUCTION

Mass measurement for aerosol exposure is normally conducted to evaluate the extent of health hazard in the workplace and in the ambient atmosphere. However, it has been suggested that aerosol number concentration might be a better indicator for predicting the level of illness of exposed population than is the aerosol mass concentration (Peters *et al.*, 1997). For example, the number concentration of fibrous aerosols is more important than in the mass concentration. Fibrous aerosols are extremely important to industrial hygienists due to the severe potential health risks associated with inhaling such aerosols (NIOSH, 1976). Sampling of fibrous aerosols is currently conducted with a 25 mm diameter filter cassette and a 50 mm conductive cowl. The conductive cowl was employed to prevent the wearer of the sampler from easily touching the filter surface, disturbing the collected fibers, and reducing electrostatic effects. All visible sampled fibers are counted by the NIOSH 7400 method, using a phase contrast microscope (PCM).

Typically, fiber counting is performed at 400 magnification (with 10 X eyepieces, 40X objective) under conditions that satisfy OSHA requirements, including correctly adjusting the Köhler illumination using a phase contrast test slide and a Walton-Beckett graticule. The counting fields are selected randomly without looking

into the eyepieces. Counting began from either end of the wedge and progressed along a radial to the other end. Meanwhile, the “A” or “B” rules were followed for counting and determining the concentration of fibers (NIOSH, 1994). Nevertheless, these complex and lengthy procedures for counting fibers are time-consuming and require experienced microscopists.

In the analytical method, fiber counting is permitted in any area of the filter and a small portion of the membrane filter is also selected for counting. However, knowledge of the count field locations may inadvertently affect counts (NIOSH, 1994). Moreover, the random errors associated with counting fibers may limit the statistical power to detect deposition trends. Further studies are still required after considering the statistical representation of the selected area (one-fourth of the filter) and randomly chosen counting fields. Hunsaker *et al.* (1988) suggested that counting protocols should specify that counting be conducted along radial or nearly radial counting lines. Additionally, the selection of counting fields along a given counting line must not be predominantly in either the inner, middle, or outer regions of the filter. According to their study, nine counting fields in the 25 mm filters and twenty counting fields in the 37 mm filters were selected along each of six (60°) radial sectors in the middle of the rings of equivalent area, ensuring that no region of the filter was under- or over-sampled relative to another region on a per unit area basis.

Jenkins *et al.* (1992) used a fluorescent test aerosol to compare the deposition trends of five different commercially available cassette membrane filters. Comparative results revealed that in all the cassettes, deposition decreased with increasing radial distance from the filter center. The observed radial effect can contribute to both bias and random error in the membrane-filter method for analyzing airborne fibers, depending on the reason for the observed radial trends and the protocol for counting field selection. However, on-trench and off-trench positions did not significantly differ in any of the cassettes tested.

Baron and Deye (1990) recommend selecting counting fields near the filter center, because of the lower aerosol deposition density near the filter circumference compared to the center. This approach produces accurate results if the radial deposition trend is caused by reduction of fibers from air passing near the cowl walls. Moreover, their study assumes that the concentration of fibers near the center of the cowl is unaffected and is representative of the ambient concentration. However, if the radial trend results primarily from non-uniform air flow through the filter, then selecting fields to obtain a representative sample of the filter surface would be more accurate. Such an approach assumes that a very large fraction of the fibers originally present in the air sample reach the filter, although their distribution is non-uniform. However, the above studies did not compare how many counting fields are adequate or suggested other counting protocols.

The NIOSH 7400 method requires uniformly depositing fibers on the filter surface (NIOSH, 1994). However, uniform filter deposits are rare in most workplace settings. The mechanisms of particle diffusion, impaction, interception, settling, and electrostatic interaction could increase the sampling biases of the sampler inlet, and furthermore the air velocity, direction, and flow pattern in a sampler inlet can produce various degrees of sampling biases. These phenomena may cause the samplings to have different aerosol deposition patterns. For instance, while the airflow enters and passes through the cassette to the filter, the anisokinetic, anisoaxial sampling

conditions always lead the air streamlines to become turbulent or to have vortices (Vincent, 1989; Baron *et al*, 1994). Consequently, aerosols could exhibit non-uniform deposition on the filter, and this occurrence also tends to influence even more the overall precision and accuracy of the analytical process. Despite this, regardless of how many errors or potential sources of variability past studies contain, the classification and characterization of the uniformity or non-uniformity of filter deposits remains poorly defined.

Baron and Shulman (1987) compared the Magiscan 2 Image Analysis System (M-2) with the manual counting method. The M-2 operated two or three times faster, and required less skill and training to use than conventional manual counting technique. However, it could not count the thinnest fibers visible by light microscopy or the fibers out of the operator-selected focal plane. Also, it sometimes counted non-fibrous particles, and over counted by breaking fibers into segments. Furthermore, the M-2 method cost approximately 5 times conventional NIOSH 7400 method at that time. Chen and Baron (1996) showed that fibers may exhibit somewhat different aerodynamic behavior from compact particles, and that the aerodynamic diameter of a fiber depends on its orientation. When fibers settle in still air, even when oriented vertically (parallel to the motion) or horizontally, the fiber aerodynamic diameter is approximately three times its physical diameter. Furthermore, Baron *et al.*(1994) noted that although their experiment had been performed with spherical or compact particles, fibers might become aligned in shear flow fields or in electrostatic fields and their behavior under gravitational, inertial, or electrostatic forces will resemble that of compact particles with the same aerodynamic diameter and charge level. Consequently, the results of using compact particles could be applied to the counting of fibers or other airborne particles, provided they were evaluated carefully before implementation.

This investigation aimed to determine the adequate number of total counting fields and statistically compare three counting protocols (equal distance, equivalent area, NIOSH 7400 method). The characteristics of uniform or non-uniform filter deposits were also calculated and defined via convenient statistical analysis. Additionally, to overcome the time-consuming work of fiber counting, image-processing techniques were employed in place of traditional counting methods.

EXPERIMENTAL METHODS

System setup

To investigate the uniformity of the aerosol deposit on the filter, a vibrating orifice monodisperse aerosol generator (VOMAG; model 3450, TSI Inc., St. Paul, MN) was used to generate the desired particles with a count median diameter (CMD) of 3 μm and geometric standard deviation (GSD) of about 1.05. To facilitate continuous operation of the generator for more than a few days consecutively, a pressure supply system for the liquid and a large solution reservoir was added to the VOMAG. The volumetric concentration of the solution being generated was selected to produce the desired CMD after the solvent evaporated from the droplets. Methylene blue (MB) was chosen as the challenge aerosol, because, in addition to stronger contrast to the background filter, the MB deposit can be examined visually for uniformity after exposure to the water vapor. The aerosols were dried by the filtered compressed air and then neutralized by inducing a charge equal and opposite to that of the spray charge, whereby a voltage applied to the dispersion-air-orifice plate induced a charge

on the droplet stream emerging from the vibrating orifice (Reischl *et al.*, 1977) as presented in Fig.1. After that, the aerosols were passed through an aluminum honeycomb flow straightener. An aerosol electrometer (model 3068, TSI Inc.) was used to confirm the neutralization of particle charges in the testing chamber. Meanwhile, the aerosol particle size and concentration were measured by an aerodynamic particle sizer (APS, model 3320, TSI, Inc.).

An asbestos filter sampler with a conductive cowl (Millipore Corp., Bedford, Mass.) and a 25 mm, 0.8 μm pore size mixed cellulose ester (MCE) filter were used in the sampling. The MB particles were sampled on the surface of the filter, after being generated from the VOMAG. The filter with MB particle deposits were then examined under 400X of phase contrast microscope. Three counting protocols: equal distance (along the radial line), equivalent area (of the annular ring), and NIOSH 7400 method were adopted and 10~200 counting fields were selected manually. The counting results of three methods were then compared correspondingly using the same filter. Meanwhile, a digital camera (Flexcam[®], model 999 0006-NPSCTA, VideoLab, Inc. Minneapolis, MN) with resolution set at 300*300 dpi (dots per inch) captured each selected field (as shown in the flow chart of Fig.2). The captured image files were decolorized and converted into 256-grayscale bitmap (BMP) files, and then changed into two extremes (black and white) after the threshold value was manually set, as shown in Fig.3. The MATLAB[®] language and image processing toolbox were programmed to count the fraction of black pixels (particles) compared to the total number of pixels in each counting field.

In the image-processing step, the procedure of changing the grayscale BMP files into black and white, a threshold value should be properly set. Here, the black pixels increased with increasing threshold value, and could cause the coverage of particles to be overestimated. However, the coverage of particles was underestimated when the threshold value was set too low. Consequently, the proper threshold value was determined by trial and error, and was verified by comparing the coverage with the area calculated by particle number, since monodisperse aerosols were used in the present study. From Fig.4, the threshold value was set at 36. As the figure showed, when the value was below 36, the particle coverage was underestimated, but when the value was above 36, the particle coverage was overestimated.

The counting method of equal distance was along each of the radial lines, and each counting field was selected at 0.5~5 mm intervals. The maximum number of radial lines was four, and maximum counting fields were 200 with minimum intervals of 0.5mm. The equivalent area counting was similar to that using the equal distance method. Along each of the four (90°) radial sectors, twenty counting fields from the 25 mm filters were selected in the middle of rings of equivalent area. The maximum number of radial sectors was thirty-two (11.25°), and the maximum number of counting fields was 160. Compared to the above equal distance and equivalent area methods, the NIOSH 7400 method randomly select any sample of one-fourth of the filter and any counting fields of the filter quarter.

To avoid heavy overlapping of aerosol deposits, the sampling time was set at two minutes and the sampling flow rate was set at 5 L/min. In addition, to avoid unnecessary interference from electrostatic attraction, the sampler was electrically

grounded during testing. The final step of the image analysis used the statistical methods, as discussed below.

Statistical analysis

Following the image processing, all the data of the filter deposits can be separated into two: the black pixels (particles) and the whole pixels in each field. The “coverage” could be used to indicate the ratio of the field area occupied by the particles, and define as:

$$C = \frac{P}{F} * 100\% \quad (1)$$

C: coverage (fraction of total viewing area occupied by particles).

P: pixels of all particles in a counting field.

F: total pixels in a counting field.

Notice that the particle number in a counting field can be used to replace the coverage, if the deposited particles are monodisperse, and vice versa.

With the coverage of each counting field calculated, the coefficient of variation (CV) could be used to indicate the variation of coverage among counting fields. The CV was given as follows:

$$CV = \frac{\sigma}{\mu} \quad (2)$$

CV: coefficient of variation.

σ : standard deviation of coverage of the particle area.

μ : the mean coverage of the particle area.

When the value of CV decreased, the variation of coverage among counting fields was small, indicating that each counting field had a similar particle deposition rate. However, the variation in coverage among counting fields increased with the value of CV. So it would be difficult to determine whether a deposition patterns was uniform or not by using the value of CV alone. In order to better define uniformity and make the indicator more universal, the uniformity was defined as the following equation.

$$U = e^{-CV} \quad (3)$$

U: uniformity of filter deposits.

The exponential of the negative CV value was a transformation to easily understand the uniformity of the filter deposits. According to Eq.3, the more uniform is the filter deposits, the closer the uniformity value approaches 1.0. Meanwhile, the uniformity value approaching 0 is the case of least uniform.

The image processing method is a tedious and time consuming procedure since there are still many occasions in need of human decision. Therefore, the standard

error (SE) of CV was evaluated by the Bootstrap re-sampling method (Mooney and Duval, 1993; Hall, 1992; Efron and Tibshirani, 1993). The Bootstrap method requires renumbering all the selected counting fields and randomly re-sampling the counting fields. For every re-sampling the CV of the coverage (of the re-sampled counting fields) is recalculated. The bootstrap method can then be expressed as follows.

$$\begin{array}{llll}
 n_1^{(1)} & n_2^{(1)} & \dots & n_k^{(1)} \rightarrow CV^{(1)} \\
 n_1^{(2)} & n_2^{(2)} & \dots & n_k^{(2)} \rightarrow CV^{(2)} \\
 n_1^{(3)} & n_2^{(3)} & \dots & n_k^{(3)} \rightarrow CV^{(3)} \\
 n_1^{(4)} & n_2^{(4)} & \dots & n_k^{(4)} \rightarrow CV^{(4)} \\
 & \cdot & & \\
 & \cdot & & \\
 & \cdot & & \\
 & \cdot & & \\
 n_1^{(b)} & n_2^{(b)} & \dots & n_k^{(b)} \rightarrow CV^{(b)}
 \end{array}$$

$$SE(\widehat{CV}) = \sqrt{\frac{1}{B-1} \sum_{b=1}^B (CV^{(b)} - \overline{CV})^2} \quad (4)$$

$SE(\widehat{CV})$: standard error of the CV.

\overline{CV} : mean of CV.

n_k^b : a random sample from the population of k particle coverage.

b : bootstrap sample size (in the study was 100).

Using the macro function of Microsoft[®] Excel, the random selection of the counting fields was accomplished. Following the same technique, the uniformity could be evaluated for its standard error using the bootstrap method.

$$SE(\widehat{U}) = \sqrt{\frac{1}{B-1} \sum_{b=1}^B (U^{(b)} - \overline{U})^2} \quad (5)$$

$SE(\widehat{U})$: standard error of U.

\overline{U} : mean of U.

RESULTS AND DISCUSSIONS

As illustrated in the upper plot of Fig.5, the four radial lines that separated the 25 mm filter into eight (45°) radial sectors were selected as the counting lines. Since the minimum counting interval with the equal distance method was set at 0.5 mm along a radial line, the maximum number of counting fields was 50. Pooling the counting data from all of the observation fields on the four radial lines (following the counting sequence), the particle coverage pattern was shown in the lower portion of Fig.5. It can be seen that the particle coverage was lower near the edge of the filter, possibly because of the boundary effect of the airflow passing through the straight tubular cowl. Except for at the edge, particle coverage was relatively constant throughout the filter. However, some counting field had a higher standard deviation around the mean coverage, probably due to non-uniform filter deposits, or formation of the multiplets during aerosol generation and sampling. Particle overlapping may occur but was

minimized to less than 5 % according to the calculated cross-sectional area of the deposited particles.

The equal distance method was designed to representatively evaluate the whole filter for aerosol deposits, since the radial lines separate the entire filter into numerous equal sectors. However, the equal distance method apparently placed too much emphasis on the central portion of the filter because all the counting radial lines pass through the center of the filter, as shown in Fig.6. If not all four diameters were counted, the CV value might vary from 0.25 (B line) to 0.65 (A+B lines), depending on the total number of the counting field. In general, the CV decreased with increasing total counting field, as expected. The CV value approached 0.4 and tended to be constant when the counting fields exceeded 100. The standard error was calculated using Eq.4. The CV value of three diameters (A, B, and C) was lower than that of the total 200 counting field, indicating that D lines must have a higher degree of non-uniformity, as shown in Fig.6.

The equivalent area counting method, as used in this study, should provide more representative results in evaluating the whole filter than the equal distance method, because same weighting was given to all the rings of identical surface area. However, as indicated in the lower portion of Fig.7, the CV value decreased with increased total counting fields. Meanwhile, the CV value approached a constant of 0.4 when the counting fields exceeded 100. Furthermore, the standard error decreased with increasing counting fields.

Since the conventional NIOSH 7400 method was designed to randomly select any quarter of the filter for fiber counting, the CV values of counting fields on two filter quarters (A and B) are presented and compared in Fig.8 as a function of total counting field. As indicated by the fluctuation of the CV curves, the different parts of the filter apparently had different CV trends. The CV curves became more constant while the counting fields exceeded 100. Nevertheless, more counting fields need to be counted to prove that this behavior is typical. The NIOSH 7400 method seemed to be less statistically representative than the other two methods, based on the uniformity of filter deposits produced in the present study.

The uniformity (U) was used to describe the distribution of aerosol deposits on the filter, as shown in Figure 9. The uniformity curves of the three methods were synchronous to their CV curves. As stated above, the more uniform are the filter deposits, the closer the uniformity value approached 1.0. In the equal distance method (the upper plot of Fig.9), a few radial lines had good uniformity in a particular set of counting fields. However, these could be purely coincidental. In all three plots, the standard errors decreased with increasing counting fields, because of higher number counts.

CONCLUSIONS

Three different methods (equal distance, equivalent area, and random on quarter) of counting aerosol deposits on the filter were performed and compared. The consistency of these counting protocols was examined using the coefficient of variation (CV), which could also be converted to uniformity (U) through an exponential transformation. The advantage of adopting U is that it has a clear lower limit of 0 (least uniform) and upper boundary of 1.0 (most uniform). Using a

MATLAB package and its image processing toolbox facilitated the determination of the CVs of the counting results. The variation of the counting results decreased with increasing number of counting fields, and this was true for all three counting protocols. Generally speaking, the value of uniformity approached a constant (0.65 in this work) when the counting fields exceeded 100.

In theory, the equivalent area method should be the most accurate (representative or non-biased) and precise counting protocol, because it treats all surface area equally. The equal distance method has the tendency to over-emphasize the central portion of the filter, because all counting lines cut through the center. The random on quarter method might lead to less representative results if the distribution of aerosol deposits is extremely distorted. There are other ways to obtain representative results, such as the randomly-ordered method, which is also unbiased. However, it might require more counting fields to yield accurate results (Leith and First, 1976).

Factors that might contribute to the non-uniformity of aerosol deposition on filter such as sampling orientation, sampling flow, aerosol size distribution, particle charge and polarity, sampler charge and polarity, and the configuration of the samplers, are all likely to affect the aerosol deposition patterns on the filter, and therefore, change the variation of the counting results.

ACKNOWLEDGMENTS

This study was supported by the National Science Council of Taiwan, through grant NSC 93-2211-E-002-034.

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FIGURE CAPTION LIST

- Figure 1: The schematic diagram of system setup.
- Figure 2: The flow chart of image processing and particle counting.
- Figure 3: The demonstration of image processing and coverage determination (particle counting).
- Figure 4: The determination of threshold value.
- Figure 5: The particle coverage profile on the MCE filter following equal distance counting method.
- Figure 6: The CV versus various counting lines and total counting fields using equal distance counting method.
- Figure 7: The CV as a function of total counting fields using equivalent area counting method.
- Figure 8: The CV as a function of total counting fields using NIOSH 7400 counting method.
- Figure 9: The uniformity versus total counting fields using (from top) equal distance, equivalent area and NIOSH 7400 counting methods.

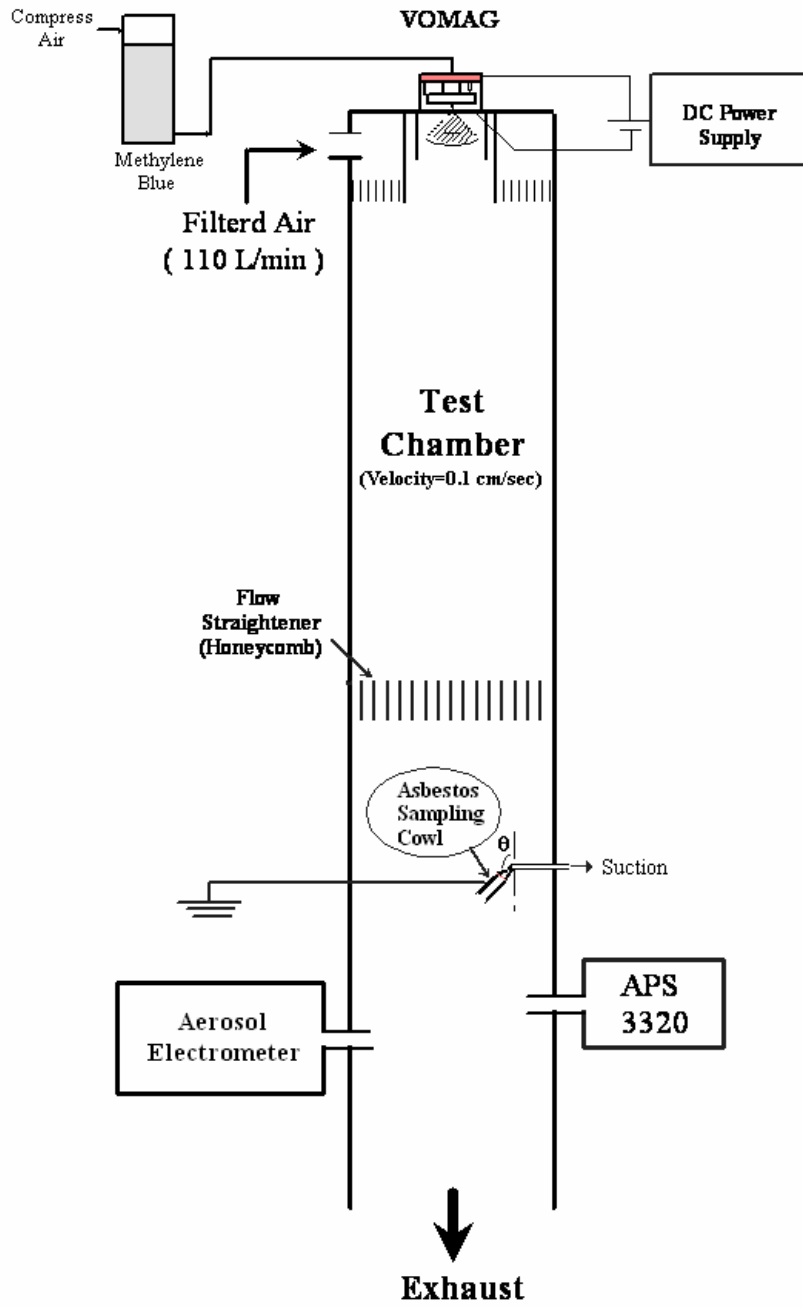


Figure 1: The schematic of diagram system setup.

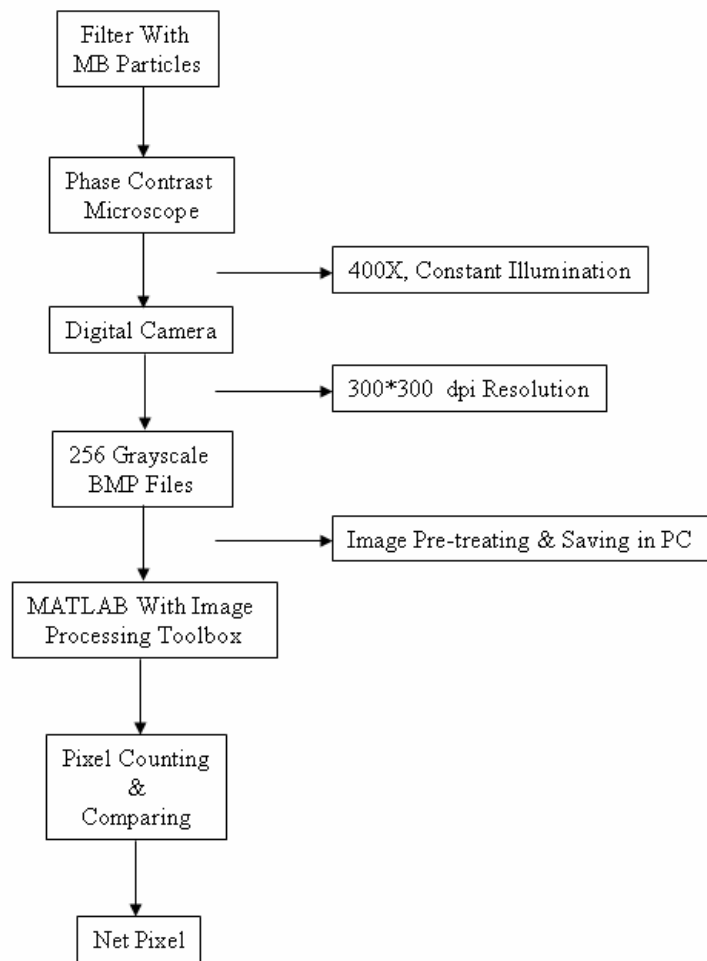


Figure 2: The flow chart of image processing and particle counting.

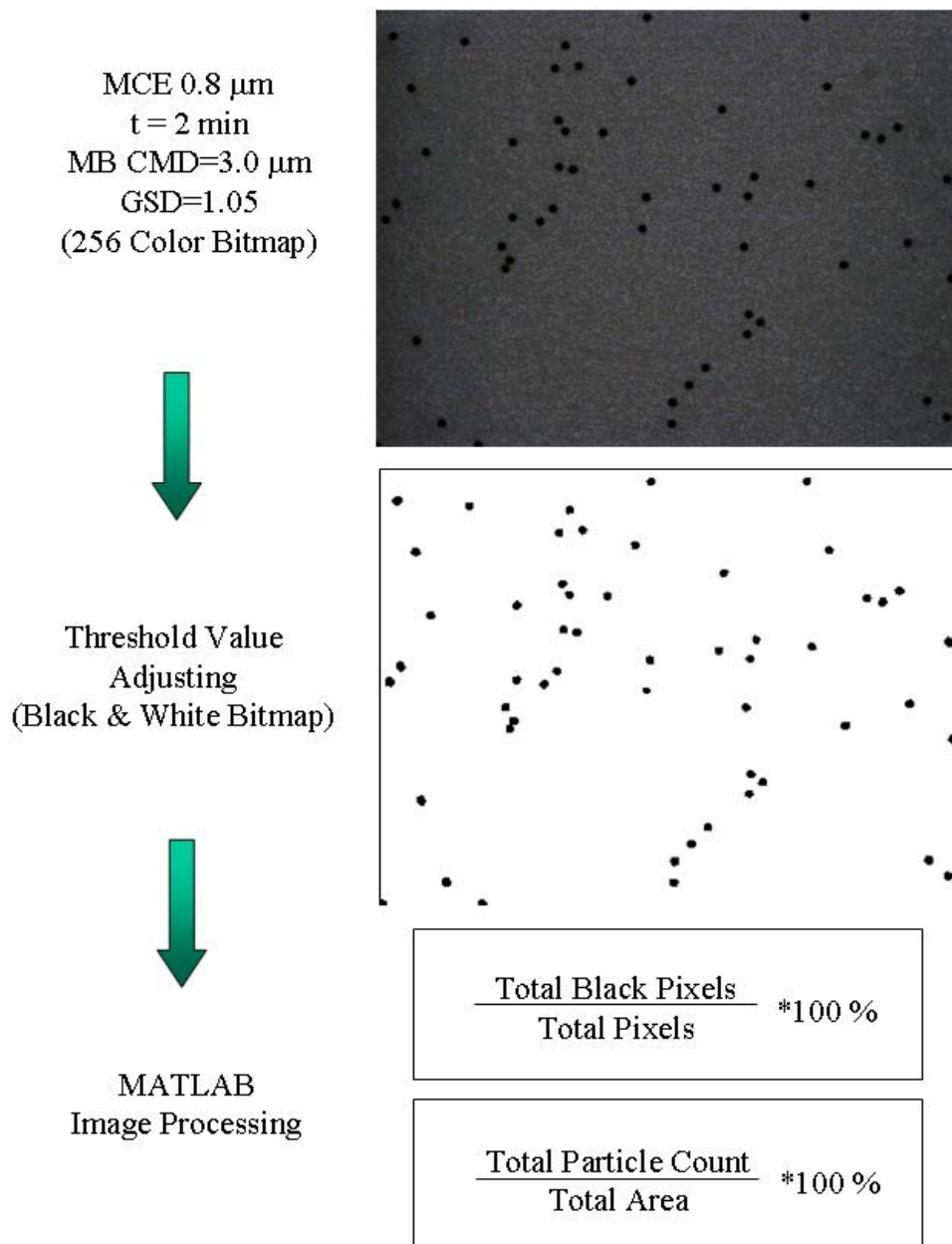


Figure 3: The demonstration of image processing and coverage determination (particle counting).

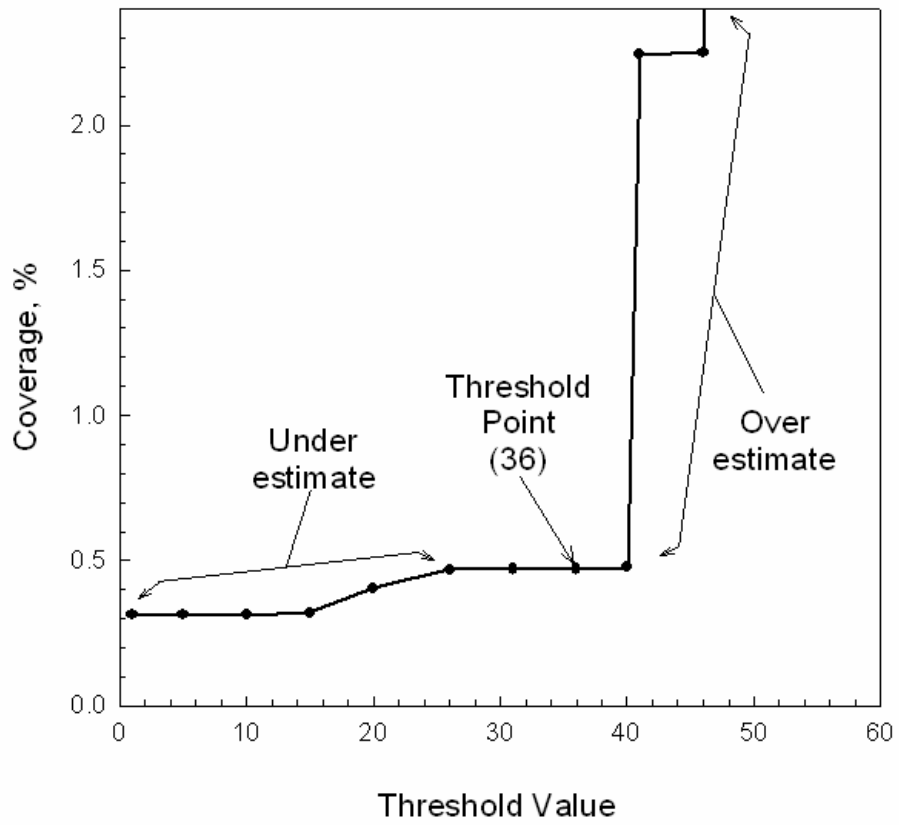


Figure 4: The determination of threshold value.

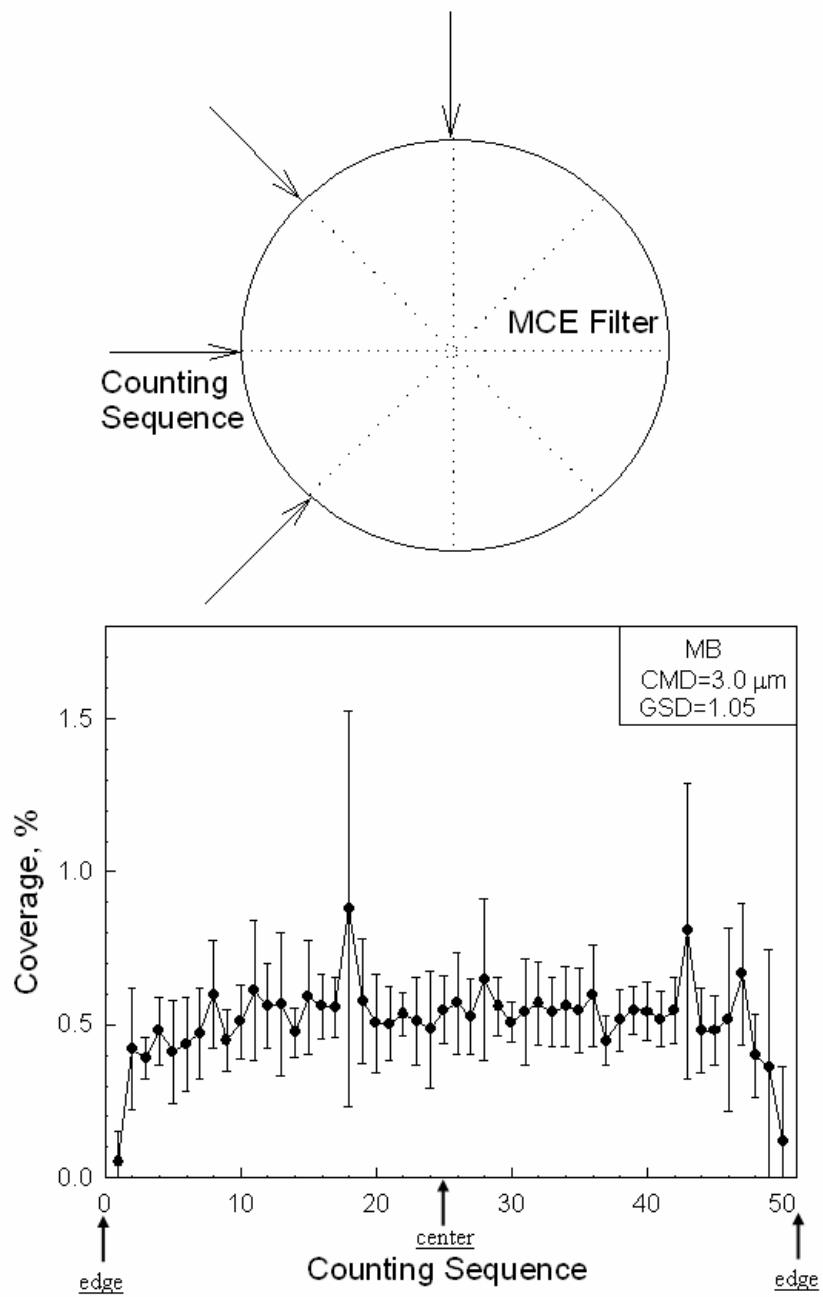


Figure 5: The particle coverage profile on the MCE filter following equal distance counting method.

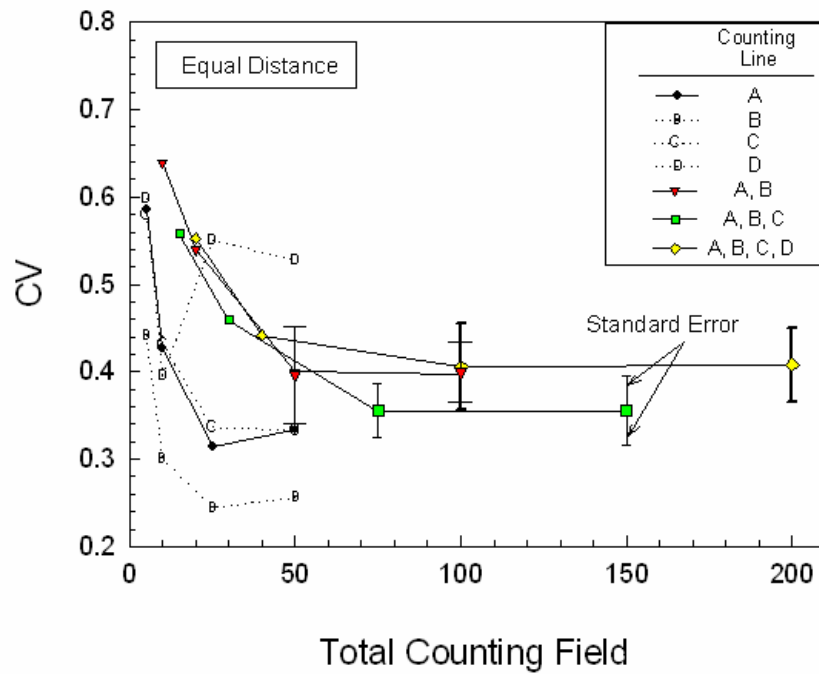
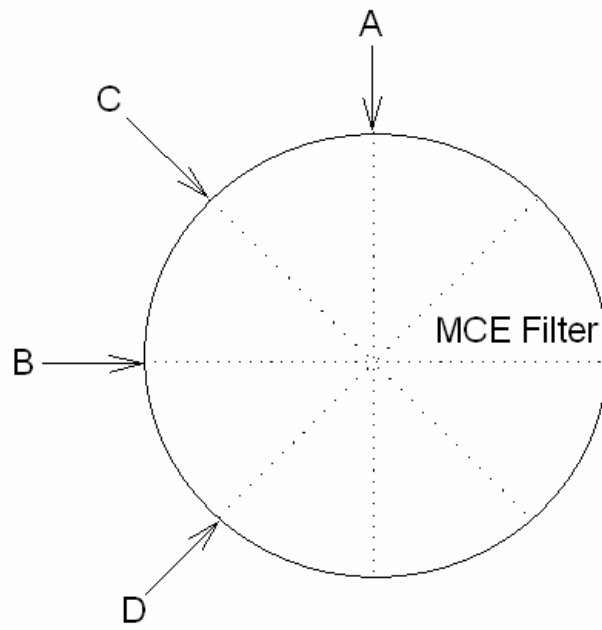


Figure 6: The CV versus various counting lines and total counting fields using equal distance counting method.

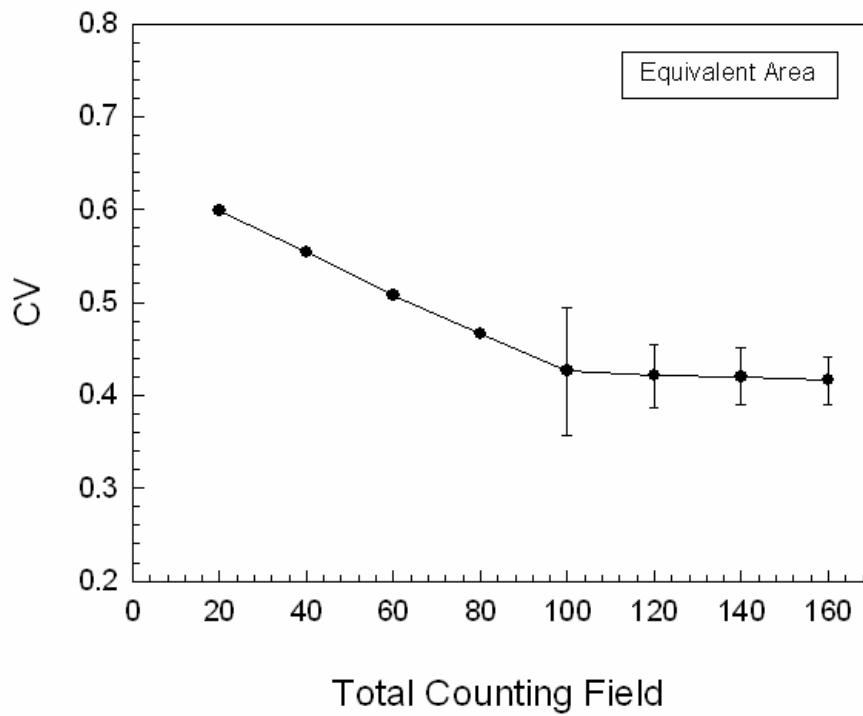
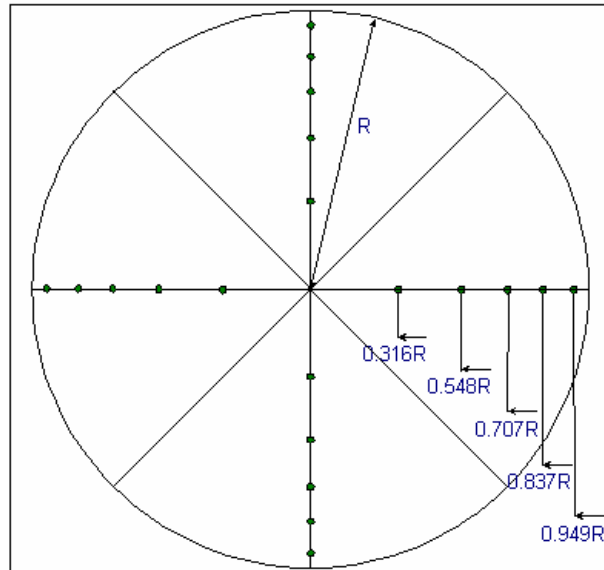
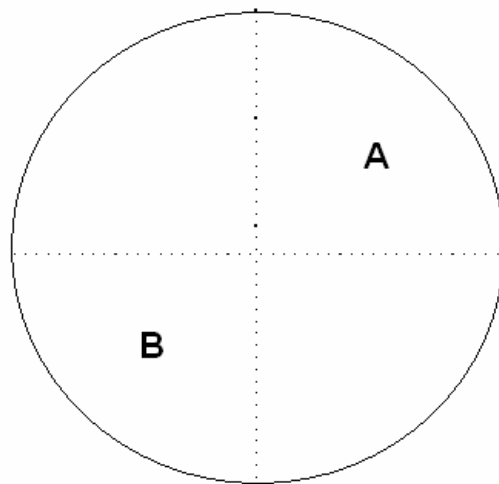


Figure 7: The CV as a function of total counting fields using equivalent area counting method.



MCE Filter

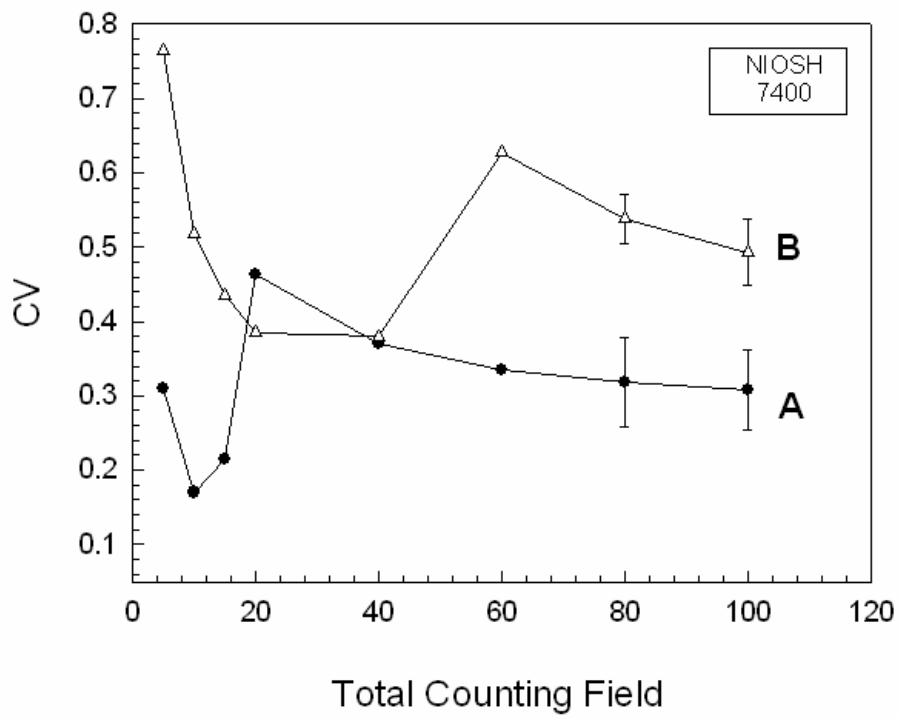


Figure 8: The CV as a function of total counting fields using NIOSH 7400 counting method.

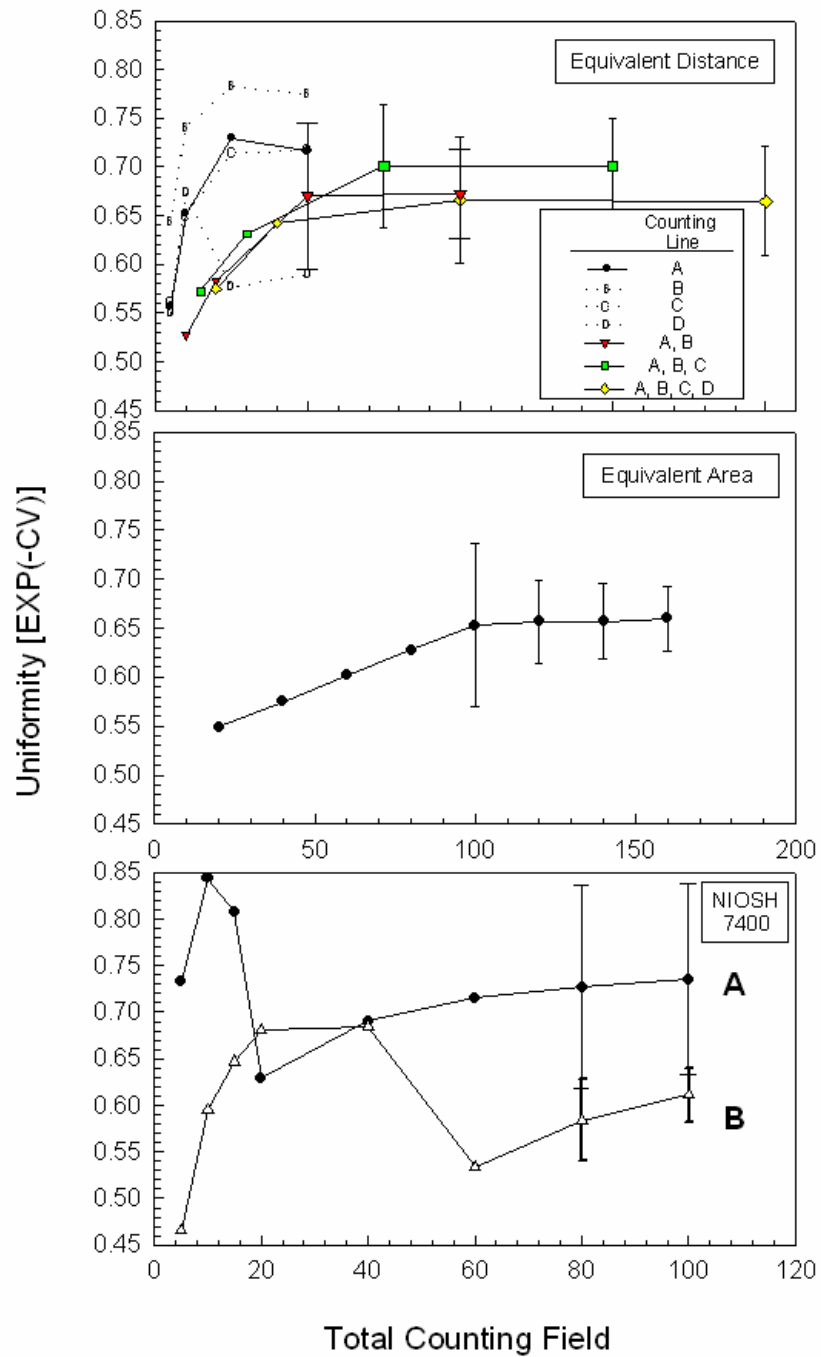


Figure 9: The uniformity versus total counting fields using (from top to down) equal distance, equivalent area and NIOSH 7400 counting methods.