

ENVR 116L Aerosol Technology Laboratory

LABORATORY MANUAL

Professor David Leith
Department of Environmental Sciences and Engineering
University of North Carolina at Chapel Hill
School of Public Health

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ENVR 116L
Aerosol Technology Laboratory
Spring 2006

Instructor: David Leith Office 0032 Hooker, Phone 966-3851
Home Phone 929-6176
Baity Lab Director: Maryanne Boundy Baity Laboratory, Phone 966-7337
Home Phone 967-8564

Class Meetings: Mondays 11-12 Hooker 0033

- 16 January **Sizing Polydisperse Aerosols of Dry Particles**
Leader - Todd
(read instructions and do lab at your leisure)
- 23 January *Discussion*: Article #1
Leader - Maryanne
- 30 January *Discussion*: Sizing Polydisperse Aerosols
- Scanning Electron Microscopy**
Leader - Javier
(read instructions; bring samples to EM lab for analysis)
Visit EM Lab, Dr. Bob Bagnell – Monday afternoon
- 6 February **Report Due**: Sizing Polydisperse Aerosols
Discussion: Article #2
Leader - Tausha
- 13 February **Isokinetic Sampling**
Leader - Tausha
Discussion: Scanning Electron Microscopy
- 20 February **Report Due**: Scanning Electron Microscopy
Discussion: Article #3
Leader - Todd
- 27 February **Cascade Impactors**
Leader - Javier
Discussion: Isokinetic Sampling
- 6 March **Report Due**: Isokinetic Sampling
Discussion of Article #4
Leader - David

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- 13 March *Spring Break*
- 20 March **Class Project**
Discussion: Cascade Impactors
Discussion: Class Project
- 27 March **Report Due:** Cascade Impactors
Discussion: Class Project
Visit Lab of Professor Tony Hickey, UNC Pharmacy School
- 3 April *Discussion:* Class Project
Visit EPA Human Exposure Facility
- 10 April *Discussion:* Class Project
Visit: Research Triangle Institute, RTP
- 17 April *Discussion:* Class Project
- 24 April **Report Due:** Class Project

SIZING POLYDISPERSE AEROSOLS OF DRY PARTICLES

Background

In the lecture course, ENVR 116 Aerosol Technology, you conducted a lab using the optical microscope to measure the size distribution of particles. You should review your lab notes for that lab, particularly the parts that concern calibration and use of the Porton graticule and the filar micrometer. The techniques used in your previous lab are simple but lead to ineffective use of a microscopist's time. In this lab we will investigate how to obtain information in a more efficient way.

Determining the size distribution of an aerosol is always important. More problems in aerosol physics concern particle size than any other characteristic. In this lab we will measure size by taking a sample of the particles in an aerosol on a membrane filter and analyzing that filter with an optical microscope. We will also measure size by passing a sample of material through a cascade of sieves.

Preparing a Microscope Slide

Particles can be collected on a membrane filter, then analyzed to determine their size distribution using a microscope fitted with a Porton graticule. The graticule must first be calibrated using the technique we used in the previous lab.

Filter mounting solution can be immersion oil found in bottles that breed like flies in cabinets that hold microscope equipment. You may also choose to use "membrane filter mounting solution", made from a 1:1 mixture of dimethyl phthalate and diethyl oxylate into which new scrap membrane filter has dissolved in proportion 1 part mixture to 0.05 parts filter by weight. This solution will produce a slide that is stable for several hours to several days or more.

Whatever mounting solution you use, check to be sure that your solution does not dissolve the particles you wish to analyze. You can reassure yourself that particles do not dissolve by focusing on a small particle, measuring its size, then letting the microscope stand for a few minutes before measuring the size of the same particle again. The most accurate way to measure size for this task is using the filar micrometer. If particle size changes, the particle may be dissolving. Dissolution of particles in the mounting solution is a more important problem than is generally recognized.

Cut a sector from the filter and place it, dust side up, on a drop of mounting solution that has been smeared to the shape of the sector on a cleaned glass slide. Because the filter has a refractive index of 1.515, the same as that of glass and of the mounting solution, the filter “disappears” as the solution is drawn into the filter pores by capillary action. The particles on the filter will still be visible unless they also have a refractive index of about 1.515 in which case they become transparent also. Some minerals do have a refractive index of about this value. When the filter is transparent, place a cleaned cover slip on top by placing one edge on the edge of the slide, then allowing the other edge to fall downward like a trap door that closes gently.

Stratified Counting

In the previous lab we discussed counting and sizing particles using the Porton graticule in the optical microscope. This process is tedious. The technique of stratified counting can help you count particles in a time-efficient way.

You will probably find that your sample has many more small particles than large ones. Because the precision of particle counting is related to the square root of the number of particles counted, the precision of your results will vary with particle size. That is, all else being equal, your results will be more precise for small particles than for large particles because you will count many small particles and few large ones.

Precision in particle counting is especially important for the larger particles if you wish to transform your results from a count to a mass size distribution as we will discuss later. Stratified counting allows you to concentrate your efforts on particles that are present infrequently with the result that the precision of your analysis is more uniform across all particle sizes. The textbook by Hinds has a discussion of this topic; see pages 406-408.

To count using the stratified technique, first size *all* particles in one or more fields to get a rough understanding of the particle size distribution. For subsequent fields, count *only particles of those sizes rarely seen*. Analysis of these subsequent fields will go quickly. You may decide to stop counting additional particles in a size range if your cumulative total of particles in that size range exceeds a number such as 20, 30, or 40, depending on the overall precision necessary for your analysis.

As an example of this process consider the table below. For the first two fields, particles of all sizes were counted. Because many small particles were present, only particles larger than Porton size 2 were counted in subsequent fields. After the fourth field, only particles larger than Porton size 5 were counted.

Analysis of data from a stratified count is done using *the average number of particles per field, from the fields for which particles were actually counted*. In this way we obtain an unbiased estimate of the particle concentration for particles of each size present.

A key assumption in stratified counting is that the size distribution of particles in all fields is the same. This assumption may not always be met; for example, fields near the center of a filter

may be enriched in large particles if a closed-face filter holder with a small inlet is used. In this case, several fields can be combined in such a way that *together* they provide an unbiased estimate of the particle size distribution across the entire filter. To select such a set of fields for a circular filter, pick field locations in a way analogous to the way a pitot tube is located when making a velocity traverse in a ventilation duct. The appropriate row entry for the stratified counting table is then the sum of the data for these combined fields.

Example: Stratified Data

Field	Porton Number									
	1	2	3	4	5	6	7	8	9	10
A	24	16	8	10	5	3	7	2	0	0
B	31	20	16	6	9	4	2	2	2	0
C	--	--	10	9	10	9	3	5	1	0
D	--	--	4	2	7	4	0	1	4	1
E	--	--	--	--	--	3	2	2	1	0
F	--	--	--	--	--	5	3	0	0	0
Avg. per Field	27.5	18.0	9.5	6.75	7.75	4.67	2.83	2.00	1.33	0.17

Converting Count Distributions to Mass Distributions

At times one may need to convert a size distribution measured by count to a size distribution expressed on a mass basis. If one is interested in a mass-based distribution, the size distribution measurement should ideally be made on a mass basis: by sieving, or by using a cascade impactor. Nevertheless, in circumstances where insufficient particle mass is available, where sampling times must be short, or where an automatic instrument is used that counts particles rather than measures their mass, only a count-based sample may be feasible. In such cases, the only recourse is to take a count-based sample and convert it to a mass-based sample by equation.

The Hatch-Choate equations, found in many textbooks including the one we used in the lecture course, can be used to convert count-based data to data expressed on another basis such as surface or mass. For the conversion from count to mass,

$$mmd = cmd \exp(3 \ln^2 \sigma_g)$$

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where mmd is the mass median diameter, cmd is count median diameter, and σ_g is the geometric standard deviation.

One must bear firmly in mind that this equation assumes the dust has a perfect log-normal distribution. Deviation from perfect log-normality will lead to errors in the conversion. The importance of the error will increase with increasing polydispersity of the dust (non log-normality in a dust with apparent σ_g of 3 is greater than for a dust with apparent σ_g of 2; there is no error if the dust is monodisperse, $\sigma_g=1$, because for this situation mmd = cmd).

A direct conversion technique that does not depend on assumptions about the distribution of the dust is outlined below. This technique will work for log-normally distributed dusts and dusts that are not log-normally distributed.

The mass of n_i particles, assumed to be spheres, with diameter d_i can be calculated from

$$\text{mass}_i = n_i (\pi d_i^3/6) \rho_p .$$

The total mass of all spheres in the distribution, mass_t , is

$$\text{mass}_t = \sum [n_i (\pi d_i^3/6) \rho_p] ,$$

so that the mass fraction of particles with size i is

$$\text{mass fraction of particles with size } i = [\text{mass}_i / \text{mass}_t] = n_i d_i^3 / \sum [n_i d_i^3]$$

after canceling common constants and assuming density does not change with particle diameter.

An example of conversion from count to mass frequency distributions for the data above taken from a stratified counting procedure is in the table below. Note that the diameter taken as characteristic of a size range is the mean of the upper and lower bounds for the particle sizes in that range. Count frequency is given by $[\text{count}_i / \sum \text{count}_i]$ whereas cumulative count frequency up to size "n" is given by

$$\sum_1^n \text{count}_i / \sum \text{count}_i .$$

Compare the data in the second to last column of the table directly above with the count data in the preceding table, taken from measurements using the microscope. Note that the single particle counted in Porton range 10 accounts for nearly 15% of the total mass. This shows the necessity of obtaining a representative sample of the largest particles present using stratified

counting. Only a few counts in these large sizes can have a great effect on a mass distribution determined from particle counts.

Example: Conversion of Count to Mass
 Frequency Distributions

Port	Size Range	Count				Mass		
		d_i	n_i	Freq.	Cum.	$n_i d_i^3$	Freq.	Cum.
1	<1 μm	0.5	27.5	0.342	0.342	3.4	0.0004	0.0004
2	1.0 to 1.4	1.2	18.0	0.224	0.566	31.1	0.0038	0.0042
3	1.4 to 2.0	1.7	9.5	0.118	0.684	46.7	0.0057	0.0099
4	2.0 to 2.8	2.4	6.75	0.084	0.768	93.3	0.0114	0.0213
5	2.8 to 4.0	3.4	7.75	0.096	0.864	304.6	0.0371	0.0584
6	4.0 to 5.6	4.8	4.67	0.058	0.922	516.5	0.0630	0.1214
7	5.6 to 8.0	6.8	2.83	0.035	0.957	889.8	0.1085	0.2299
8	8.0 to 11.2	9.6	2.00	0.025	0.982	1769.5	0.2157	0.4456
9	11.2 to 16.0	13.6	1.33	0.017	0.999	3345.6	0.4078	0.8534
10	16.0 to 22.4	19.2	0.17	0.002	1.001	1203.2	0.1467	1.0001
Total			80.50	1.001		8203.7	1.0001	

Sieving

Sometimes the size distribution of the dust parent to an aerosol can be analyzed using sieves. This parent dust probably does not have the same size distribution as the aerosol particles because particles may be generated unevenly with respect to size; that is, smaller particles in the parent dust may be easier or harder to aerosolize than larger particles. The best methods for determining the size distribution of aerosol particles require analysis of the aerosol itself; however, sieving the parent dust is an easy way to estimate the sizes of particles that *might* be present in an aerosol.

Sieves with different mesh sizes can be arranged in a column such that the mesh openings become smaller as one descends through the column. At the bottom is a pan that catches dust small enough to pass through all the sieves.

Woven-wire sieves and the Ro-Tap Sifter

Woven-wire sieves are effective for coarse dusts, generally larger than 50 to 100 μm in diameter. They are less effective for smaller dusts because small particles remain stuck to surfaces easily. A 400 mesh sieve, with openings of 37 μm , is the finest mesh readily available. Smaller mesh sieves are available, but are made by a different process than woven wire as discussed below.

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When fine, insoluble dusts are to be analyzed, wet sieving is appropriate. Remove the pan, and use a water stream through the top sieve to wash the dust to its appropriate level. Sieves can be removed from the top of the column as washing becomes complete. The analysis is finished when no more fine dust can be seen coming through the bottom sieve. With this technique, the sieves must be dried before the dust on them can be weighed. The mass of dust smaller than the bottom sieve is determined by difference.

Place a weighed sample of dust in the uppermost sieve, then put a lid over this sieve. Tape the joints between sieves to keep dust from working through these joints as the sieves are shaken. Then place the sieve stack on the automatic shaker.

If no automatic shaker is available, you can shake the sieve stack by hand; however, this work is surprisingly hard and is tedious as well. In addition, results are often difficult to replicate because of variations in shaking technique. Shake the sieves with a swirling motion, striking the pan firmly against a hard surface from time to time, to cause each particle to descend to the sieve it cannot pass through.

The following instructions are taken from the manual for operation of our Ro-Tap sieve shaker. This device is the industry standard for sieve analysis. The Ro-Tap costs about \$1,200, not including the sieves. Each sieve costs about \$45 to \$60, for meshes larger than about 70 μm . Sieves with meshes down to 38 μm cost over \$100 per sieve. A 38 μm sieve costs \$168 in the Fisher catalog.

Size of Sample to be Tested

“In determining the size or weight of a sample we must consider the type of material, its screenability, and the range of particle sizes present. For example, in making a sieve analysis of a material representing a feed to a screen or a product from a crusher in which the particle range is very wide, a large sample of from 500 up to 1000 grams may be required. If the material to be tested is a finely ground product, a sample of 25 to 100 grams should be used.

“There is a natural tendency to use too large a sample in the test. This is incorrect as in general the smaller the sample (properly taken) the more consistent the results. To obtain an accurate sieve test every particle must present itself to the screen openings for rejection or for passing through to the next finer sieve. If there are six or seven sieves in the nest, a fine mesh particle must repeat this operation six or seven times. If the sieves are overloaded, the fine mesh particle may never get a chance to get to its proper sieve. However, the sample should be large enough so that the first sieve retains enough particles to be representative.

“The general rule in determining the size of a sample is that it be limited in weight so that no sieve in the series used in the analysis be overloaded. Overloading is most likely to occur in making analyses on closely graded materials where the range of particle size is confined to close limits. In this case the size of sample should be determined by the

capacity, without overloading, of the sieve retaining the largest amount of the sample. Overloading of the sieves results in unreliable data as blinding of the meshes occurs on the heavily loaded sieve. As an aid in determining the size of the sample, the following procedure is suggested.

“Accurately split out on a sample splitter samples of varying weights--as for example, 25, 50, 100, 150, and 200 grams of the material. Then run these various samples on the sieves selected for a period of, say, five minutes. A comparison of these results will definitely show the correct size of sample to use.

“For example, if the 100 gram sample shows approximately the same results in percentages retained and passing through the sieves as the 50 gram sample, whereas the 150 gram sample shows less material through the finest sieve, this would be an indication that a 100 gram sample would be satisfactory for tests.

“Near-mesh particles are those having dimensions that are close to the sieve opening. To secure analyses that are accurate, it is essential that the sieves be lightly loaded so that each of these near-mesh particles can be presented to the sieve opening many times and thus allow maximum opportunity for accurate classification.

Length of Sieving Time

“The time required for sieving in the Ro-Tap testing sieve shaker is dependent upon the type of test desired. For example, in many instances for plant control operations a 3 to 5 minute test on a free sieving material is sufficient to give the desired data, whereas on more difficult materials sieving time of from 10 to 30 minutes is justified. If sieve tests are made for the purpose of determining whether the material meets with definite specifications, a longer period of sieving may be established. All interested parties, however, should agree and follow a standardized method as in only this way will their tests be comparable.

“In determining the length of sieving time necessary it is suggested that three or four samples be cut out on a sample splitter to the weight which has been previously proven as satisfactory. One of these samples could then be sieved for five minutes, one for 10 minutes, another for 15, and a fourth for 20 minutes. After tabulating these results by percentages, the length of time necessary to stabilize the sieving action will be readily apparent. A satisfactory "end point" is considered to have been reached when an additional period of sieving time fails to change the results on any of the sieves used in the analysis by more than 0.5% to 1.0%. In reporting sieve tests, calculations carried to 0.1% are the limit of accuracy justified except in very unusual cases.

Weighing the Sample

“After completion of the agitation of the sieves the entire nest of sieves should be brought to the weighing station for recording of the analysis. Weighing should always be done by grams and a balance having at least a capacity of 500 grams with a sensitivity of 0.10 gram is desirable. The total weight of the material retained on the various sieves and in the pan should be very close to the weight of the original sample. The (absolute) weight of the sample or the (absolute) weight of the material retained on each sieve is never used for comparative purposes, but all results are expressed by the percentage of the total sample retained or passed through a particular sieve.

Cleaning the Sieves

“Using a spare bottom pan the material retained on a sieve should be dumped into the pan and the sieve inverted and placed over the pan. Then a soft brass wire brush or nylon bristle brush is used to gently brush the underside of the sieve using a circular motion, being careful not to exert too much pressure against the wire cloth. In most every case virtually all the near mesh particles imbedded in the meshes can be removed by this dry brushing process. The sieve can then be raised from the pan and the side of the frame tapped by the handle of the brush to clean the remaining.

Reference Information

“The American Society for Testing and Materials has available a publication on test sieving (STP-447). W.S. Tyler, Inc, 8200 Tyler Blvd, Mentor, Ohio 44060 has available a bulletin of Testing Sieves and their Uses (Bulletin No. 53). For any specific assistance in the proper use of the Ro-Tap and the testing sieves please contact the Laboratory Equipment Division of W.S. Tyler.”

Further Notes on Sieving

We have found that removing particles embedded in the finest sieves can be difficult. Be careful not to be too forceful with a brush while trying to remove these particles because the fine sieves are expensive and fragile. We have found some success in removing imbedded particles by placing the sieve in an ultrasonic bath for a few minutes. The whole sieve will not fit in the bath; however, half of it will fit. After the first half is cleaned, rotate the sieve to treat the second half.

In addition, we have found most success when sieving a total amount of dust that is somewhat less than the guidelines above. That is, we have generally used 10 to 50 grams of material to start with, rather than 25 to 200.

Plotting Size Distributions

We may wish to plot a size distribution by frequency, or to plot a cumulative size distribution. Both frequency data and cumulative data are in the table above, in which conversion from count to mass by calculation was discussed. Frequency for a count distribution refers to the number or fraction of total particles counted in a certain size range; similarly, frequency for a mass distribution refers to the number or fraction of total mass found, for particles in a certain size range.

Frequency Distributions The number of particles found in a certain size range depends in part on the width of the size range. For example, refer to the second table above. This table shows that $0.342 + 0.566 = 0.908$ or 90.8% of the particles by count are in the first two Porton size ranges. Had we defined size ranges differently, by using one size range for particles from 0 to $1.4 \mu\text{m}$ in diameter instead of two size ranges, the number of particles in that size range would be different.

To account for the width of the size ranges, particle counts can be divided by the width of the associated size intervals to give a “normalized” count per interval. For example, the normalized count for the first size range in the table above would be $27.5/(1-0) = 27.5$ particles/ μm , $18.0/(1.4-1) = 45.0$ particles/ μm for the second interval, etc. Similar calculations for the frequency by mass would give $0.0004/(1-0) = 0.0004$ for the first interval, $0.0038/(1.4 - 1.0) = 0.0095$ for the second interval, etc.

Frequency distribution are properly plotted as normalized values on the vertical axis against the *midpoint* of the size interval, d_i , on the horizontal axis. The midpoint size is used because it best characterizes all particles within the interval it represents. For example, a plot of the frequency distribution by count for the un-normalized data above would have (27.5,0.5) and (18,1.2) as its first two points; similarly, a frequency distribution by mass for these un-normalized data would have (0.0004,0.5) and (0.0038,1.2) as its first two points. A plot of the *normalized* frequency distribution by count would have (27.5, 0.5) and (45,1.2) as its first two data points. The normalized plot would ordinarily be used because it represents the data in a less biased way as discussed above.

Cumulative Distributions Cumulative distributions by count or mass represent the cumulative number or mass of particles less than the *upper bound size* for each size range. The values on one axis for these plots are always count or mass fractions although count or mass percentages can also be used. For example, the first two points for the cumulative count distribution for the data above would be (0.342,1.0) and (0.566,1.4) whereas for the cumulative mass distribution the first two points would be (0.0004,1.0) and (0.0042,1.4). Note that the upper bound size is used because the cumulative distribution represents the fraction of particles smaller than a stated size. For the first size range, for example, the fraction of particles plotted represents the fraction of all particles smaller than the upper bound size of the first size interval.

Plotting Cumulative Distributions on Log-Probability Paper The course website has a link to log probability paper that you can use to plot cumulative size distributions manually. Excel is an

excellent spreadsheet for processing data from microscopes and sieves; unfortunately, the Excel plotting routine cannot directly plot data using log-probability axes. I have written an Excel spreadsheet to get around this limitation and will put it onto the course website. This spreadsheet was developed to display data taken with a cascade impactor (see lab later in this course). The spreadsheet also determines and plots other parameters as well, including the best-fit log-probability line, the mass median diameter and geometric standard deviation, and other parameters that may be of interest such as the cumulative percentage by mass smaller than 2.5 μm and smaller than 10 μm .

Procedure

The purposes of this lab are: (1) to determine the size distribution of a dust by count using the optical microscope, (2) to measure the openings of a 400 mesh sieve, (3) to determine the size distribution of the same dust by mass using a sieve analysis, and (4) to convert the optical distribution by count into a distribution by mass mathematically, allowing direct comparison of the optical analysis with the sieve analysis.

Procedure for Microscopy

Take a small bulk sample of coarse Arizona road dust from its container, and disperse it on a microscope slide. One way to do this is to immerse the end of a straightened paper clip or a toothpick in the dust, then rub the wire or toothpick in a droplet of immersion oil that has been placed on a glass slide. Continue to rub the wire in the oil using a swirling motion until the dust is well-dispersed in the liquid. This is known as the “rubbing out” technique. Then cover the sample with a glass cover slip and examine it under the optical microscope. You may have to make several preparations until you obtain one that has a “reasonable” density of particles.

Using a stratified counting technique, determine the cumulative size distribution by count for this sample. Plot the cumulative distribution data on log-probability paper. Determine the count median diameter, d_{50} . Force a straight line through the data and estimate a geometric standard deviation. Comment on whether the data appear to follow a log-normal distribution.

Procedure for Sieve Analysis - Ro-Tap Sifter

Place the 400 mesh sieve on the stage of the optical microscope equipped with the filar micrometer. Focus on an opening in the sieve. Measure the distance between parallel wires, and the distance from corner to corner for the square sieve opening. Repeat for several square sieve openings.

Assemble the sieve assembly and tape the joints between adjacent sieves to prevent dust leakage. Weigh out four samples of coarse Arizona road dust: 5, 10, 20 and 40 grams. Sieve each for five minutes, then weigh the amount of dust collected on each sieve and in the pan. In this way, determine the proper mass of this dust to use for an analysis. Weigh out a second amount of dust of the mass you have selected, then sieve this sample for 20 minutes. Weigh the

amount of dust collected in each sieve and in the pan, then report the cumulative size distribution by mass for this dust.

Analysis

Do the following conversions and plots. Be ready to discuss your findings by Monday, 30 January.

1. Convert the size distribution by count measured with the microscope to a size distribution by mass. Do this using the Hatch-Choate equation based on the count median diameter and your estimate of the geometric standard deviation. In addition, do this using the direct count-to-mass calculation technique described above.
2. Compare the size of the opening in the 400 mesh sieve that you measured with the filar micrometer with the nominal opening size marked on the sieve. What is the best way to characterize the size of the sieve opening?
3. How much Arizona road dust should be used in a sieve analysis using the Ro-Tap sieve shaker? How does size distribution vary with mass sieved from your tests, if sieving time is five minutes? How do the results from the five minute tests compare with the 20-minute test for the dust mass you decide is proper.
4. Plot the cumulative distributions calculated by Hatch-Choate and calculated from the direct conversion procedure from the microscope analysis along with the cumulative distribution by mass as measured by the sieve analysis on the same piece of log-probability graph paper.
5. How well do the cumulative distributions by mass from the Hatch-Choate and directly converted count data compare with the distribution by mass from the sieves? What do you think accounts for any discrepancies? Which size distribution by mass do you believe represents most accurately the size distribution by mass of the aerosol? What leads you to this conclusion?

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SCANNING ELECTRON MICROSCOPY

Background

Although the scanning electron microscope (SEM) and the optical microscope can both be used to count and size particles of interest to aerosol scientists, the two instruments differ in their methods of operation and in the characteristics of the images obtained. The chief advantages of the scanning electron microscope are:

1. Markedly increased depth of field compared to the optical microscope at the same magnification, so that the SEM image appears more three-dimensional,
2. Significantly smaller limit of resolution than the optical microscope, so that smaller particles can be resolved.

Disadvantages of the scanning electron microscope are:

1. The sample must be prepared and examined in a vacuum so that volatile particles cannot be examined with this technique,
2. The scanning electron microscope requires more knowledge and skill to use effectively than does the optical microscope,
3. A good scanning electron microscope costs 50 to 100 times as much as a good optical microscope.

If small, non-volatile particles must be seen clearly, or if three-dimensional structure is important to see, scanning electron microscopy is the technique of choice. If all particles of interest are larger than a few micrometers in diameter, and if projected area or length is of more concern than shape, the optical microscope will serve very well.

A schematic drawing of a scanning electron microscope is below. Electrons boil from the surface of a filament, and are focused to a beam by a series of lenses. The beam scans across the surface of the sample to be analyzed in a series of rows.

As the electrons strike the surface of the sample, they emit secondary electrons, which are gathered by a detector. Note that the path the secondary electrons take need not be a straight line. Secondary electrons generated in a "hollow" on the sample will likely strike the edge of the hollow and not reach the detector, whereas secondary electrons generated at a "peak" on the

sample will be less likely to be collected by the sample and more likely to reach the detector. Thus the detector receives more electrons when the incident electron beam strikes a peak on the sample than when the beam strikes deep within a hollow.

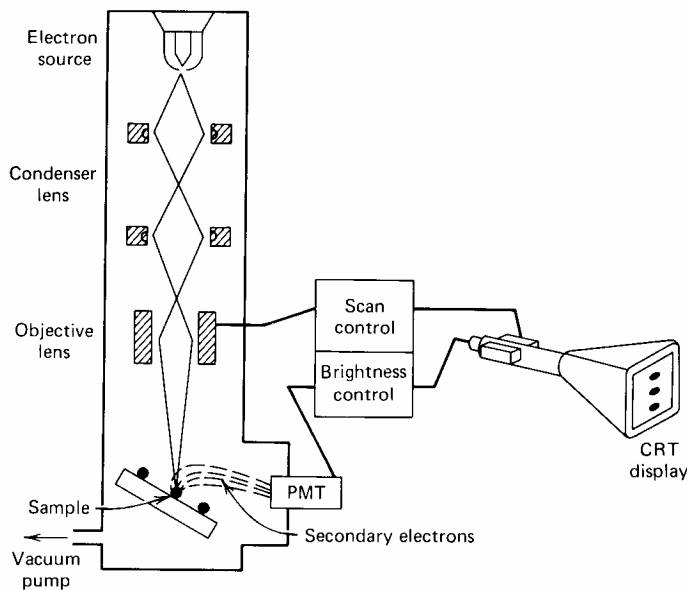


Figure 19.8 Schematic diagram of a scanning electron microscope.

The signal from the detector is amplified, then goes to a CRT display that scans in synchronization with the scan of the electron beam. A high point on the sample results in a bright point on the CRT, whereas a point within a hollow results in a dark point on the CRT. In this way, the scanning electron microscope generates an image with a three-dimensional quality. The ratio of the height and width of the scanned area to the height and width of the CRT display determines the magnification of the SEM image.

For the microscope to work properly, the current from the electron beam must be carried away from the sample; that is, the surface of the sample must be electrically conductive. If the sample surface will not carry away the beam current, the sample will accumulate a surplus of electrons causing a net charge in the area the beam is striking; the excess of electrons on the sample surface will repel the incident beam and an excess of electrons will reach the detector. The image on the CRT will degrade to a white splotch, which does not reveal the detail of the sample examined.

If a sample is electrically conductive, as would occur if one wishes to examine a fracture in a metal part, the sample can be placed directly into the SEM. On the other hand, if the sample is not electrically conductive, it must first be coated with a thin conductive material before it can be examined in the SEM. In aerosol work, samples are often taken on membrane filters that are not electrically conductive. These filter samples must be coated before the particles on them can be examined because current can leave a particle illuminated by an electron beam only by passing over or through the filter on which the particle rests.

Samples can be coated by placing them in a vacuum evaporator equipped with a tungsten filament. A small quantity of gold or gold-palladium alloy is placed in the filament. The samples are placed in the evaporator, a vacuum drawn, and current passed through the filament sufficient to heat it white hot. At this temperature, the gold or gold-palladium vaporizes to coat a thin layer on everything within the evaporator, including the samples. This coating is a few

atoms thick and not sufficient to affect the measurement of particle dimensions larger than 0.01 μm or so.

Procedure

Dr. C. R. Bagnell, director of the electron microscopy laboratory in the Pathology Department of the UNC Medical School, has kindly agreed to let us visit his laboratory to see and use the instruments there. He will show us the proper techniques for using the vacuum evaporator and scanning electron microscope in this lab. The instruments in Dr. Bagnell's lab are state of the art; we are fortunate that he has agreed to spend the afternoon with us.

We use the SEM to determine the number and sizes of particles that are collected using a passive aerosol sampler under development in our lab. Some articles on this topic are on the course website. Bring the substrate from a passive sampler with you to Dr. Bagnell's lab. Examine it using his SEM, and take digital photographs of the particles at several magnifications.

Determine the size distribution of the particles on the SEM substrate. You can do this using the same stratified counting technique used with the optical microscope. Alternatively, try to convince Maryanne to show you how this analysis can be done using ImageJ, an image analysis program that is available free over the internet from a website at NIH.

<http://rsb.info.nih.gov/ij/>

You can download and use ImageJ by yourself, but you will need some time to become familiar with the program. We already have it running on a computer in our lab.

Analysis

Provide an Excel or text file that lists in one column the particle size, and in the next column the number of particles. If you use ImageJ, this information is provided automatically. We will then process these data through our analysis spreadsheet and determine the average value of PM10 and PM2.5 to which the sampler was exposed.

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ISOKINETIC SAMPLING

Background

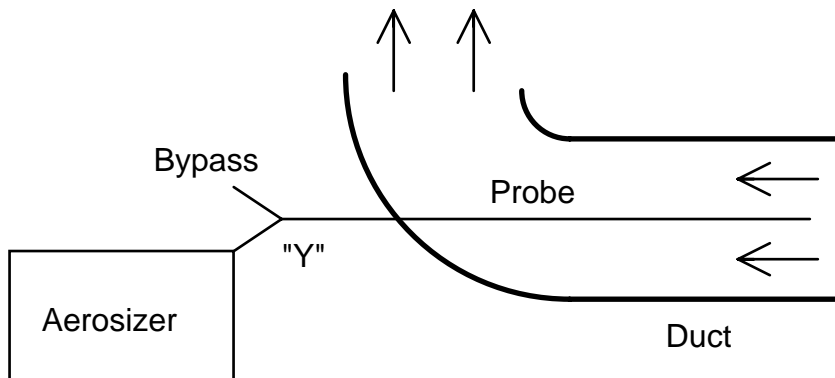
Representative sampling is important. In preparation for this lab, you should review the material in the Hinds textbook on this topic. Much of the definitive work on this topic has been done by Dr. Klaus Willeke and colleagues at the University of Cincinnati. Russ Wiener, whose lab we may visit at EPA, made major contributions to this field through his PhD thesis, which he did with Willeke.

In this lab, we will investigate sampling from a duct that carries aerosol particles. We will determine the effect of sampling rate on the measured concentration and size distribution of the aerosol particles.

The equipment we will use is already in use in our lab. The objectives of this project are: (a) to determine the effectiveness of alternative methods for removing mist particles from air, and (b) to develop models that describe and predict collector efficiency. Mists of coolant droplets are generated in the workplace from liquid coolants that flow over metal parts that are being machined. The mechanical action of the cutting or grinding operation generates mist. Worker exposure to coolant mists is an important problem in plants where automobile engines or transmission parts are made.

Approach

Lab air flows through an absolute filter, an orifice meter, and an inlet duct to a mist eliminator, then through an outlet duct, past a sampling port, and to a damper and fan. Gas flow is set to 1000 cfm (cubic feet per minute) by adjusting pressure drop across the orifice meter to 2.5" w.g. The inside diameter of the duct is 8 inches, so that the average gas velocity through the duct is 2865 fpm. Coolant mist is generated using a pneumatic aspirator, then fed to the duct upstream of the collector. Aerosol that passes through the collector is sampled downstream through a probe mounted at the centerline of the gas outlet duct. The probe has an inlet diameter of 0.38 cm, and is 90 cm long. From the probe, the aerosol flows to a "Y", where part of the flow is diverted. The flow divider is manufactured by TSI Inc, Minneapolis, MN, and costs about \$750. The balance of the flow goes into an API Aerosizer (Amherst Process Instruments, Hadley, MA). The Aerosizer measures aerosol concentration and size distribution. It costs about \$45,000.



Pete Raynor, a former student in our lab who is now on the faculty at University of Minnesota, has established that the velocity profile in the outlet duct at the sampling point is flat; that is, the actual velocity at the sampling point is the same as the average velocity across the duct at that point. He established this by measuring the air velocity at multiple points across the duct in the horizontal and vertical directions using a pitot tube. Pete has also established that the concentration and size distribution of aerosol particles at the sampling point is the same as the average across the duct. He measured the aerosol concentration and size distribution at multiple traverse points across the duct in the horizontal and vertical directions using our PMS laser spectrometer. Thus, through Pete's prior work, we know that samples we take at the duct centerline are representative of the aerosol in the duct as a whole.

The air flow to the Aerosizer will be constant, and set to 1.0 Lpm for this experiment. We can vary the total flow extracted through the probe to values higher and lower than the isokinetic rate by adjusting the air flow through the bypass line attached to the "Y" upstream of the Aerosizer. We will assume that the aerosol analyzed by the Aerosizer is representative of the aerosol sampled through the probe; that is, we will assume that adjustments to the flow through the bypass line do not appreciably affect the results obtained using the Aerosizer.

Before coming to class, calculate the isokinetic sampling rate for this set-up. Also calculate the "best" sampling rate; that is, one that will provide the most accurate representation of the duct aerosol based on equations taken from the Hinds book.

Try to sample at 0.25, 0.5, 1, 2, and 4 times the "best" sampling rate. For each sample, determine the number concentration and the size distribution using the Aerosizer.

Analysis

Assume the sample at the "best" sampling rate accurately represents the aerosol in the duct. For each sampling rate you investigate, determine the sampling efficiency for the range of particle sizes measured using the Aerosizer. Be ready to discuss the following points:

- (a) In theory, how important is aspiration efficiency compared to transport efficiency for this set-up;

Isokinetic Sampling

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- (b) At each sampling rate (R value), compare sampling efficiency predicted by the model with sampling efficiency that you measured in this lab. Do this for a range of particle sizes.
- (c) Discuss reasons for any differences between the model predictions and the results from your measurements.

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THE CASCADE IMPACTOR

Background

Cascade impactors are widely used because they provide aerodynamic size distributions, are relatively easy to use, and are relatively cheap. Unfortunately, they also have drawbacks: they are subject to interstage losses, to overloading or underloading errors, and require data processing before results are known.

Several years ago, we conducted tests to evaluate the performance of a commercial mist collector at Ford Motor Company's transmission plant in Sharonville, Ohio. We needed instruments that we could use in our lab and that we could transport to Ohio for the tests there. We had a low budget. We chose cascade impactors. Shortly after making this decision, some colleagues at NIOSH volunteered to help us with the study, and loaned us two Aerosizers to help with the work. Thus, we have not had to use the impactors in the field.

In this lab you will use an Andersen cascade impactor to measure mist concentrations. When you have completed this lab, you should have an idea of how impactors work, and how to analyze data from impactor measurements.

Andersen Impactor

The Andersen non-viable impactor is widely used, and has become the industry standard even though it has many shortcomings. A complete impactor, with pre-separator, costs about \$8000. We have had trouble with the Andersen company (now Thermo) in that stages bought at different times for the same impactor are not always interchangeable. The design air flow for this impactor is 1.0 cfm (28.3 L/min). Cut points for the eight impactor stages and pre-selector are given below. Ideally, each impactor stage collects all particles larger than its cut point and smaller than the cutpoint of the stage preceding.

Cut points for Andersen Non-Viable Cascade Impactor (from manufacturer)

Stage	pre-sel	0	1	2	3	4	5	6	7	filter
Cut, μm	10.0	9.0	5.8	4.7	3.3	2.1	1.1	0.65	0.43	0.0

Collection occurs directly on the stage. In some cases, paper substrates can be used on the surface of each stage. To draw sample through the impactor, connect it to a rotameter, flow

control valve, and sampling pump or vacuum source. Because the air downstream from the impactor is in a vacuum, the downstream rotameter must be calibrated under operating conditions to establish the proper reading to obtain 1 cfm at the impactor inlet.

Andersen High-Volume Cascade Impactor

We will not use the High-Volume Cascade Impactor in this lab, but you should know about it anyway. Where the concentration of particles is low, collection of sufficient sample to weigh can be a problem. In this case, the high-volume impactor can be used. Flow through this device is 20 cfm, so that sampling time is reduced by a factor of 20 for equivalent mass collection, compared to the standard Andersen impactor. The high-volume impactor runs from a high-vol sampling pump. The pump costs about \$500, and each impactor stage costs about \$600. You can buy as many or as few stages as you wish, to put together your impactor. Cut points for the Andersen high-volume impactor are given below:

Cut Points for Andersen High Volume Impactor

Stage	1	2	3	4	5	6	filter
Cut Point	10.2	4.2	2.1	1.4	0.73	0.41	0.0

Collection with the high-volume impactor is on aluminum foil substrates placed on top of each impactor stage. Because the stages are relatively large and massive, it is not possible to weigh them directly. The substrates, however, are light and can readily be weighed on an analytical balance equipped with a sub-stage, balance pan for weighing large objects. Because the aluminum foil substrates cost \$4 each, we clean and re-use them in our lab to keep costs down.

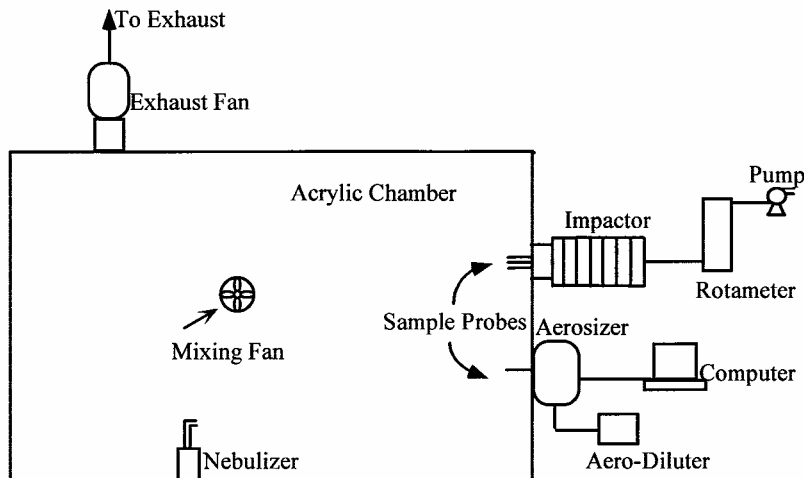
Notice that the stage number is stamped on the edge of each impactor stage. Notice also that the slots on each stage are offset, so that the jet from the stage above will focus on the flat place between the jets on the stage below. If you install a stage backwards, the impactor will not work properly and you will have to redo the experiment, a situation we have experienced more times than we like to remember. We have marked the edges of the stages with black magic marker to facilitate proper stage assembly.

Procedure

Apparatus

We will generate mist in a plexiglas chamber using a Collison nebulizer. We will then measure the concentration and size distribution of the mist using an Andersen impactor that samples from the chamber. In addition, we will measure the mist concentration and size distribution using an Aerodynamic Particle Sizer (APS) or an Aerosizer, depending on which instrument is available. Comparison of the data from the impactor and Aerosizer or APS will

provide interesting insights. A figure that shows how we will try to set up the equipment is on the next page.



Method

Weigh impactor plates and the final filter to the nearest 0.1 mg using the Mettler balance. Assemble the impactor. Arrange the impactor and the APS or Aerosizer to sample from the chamber.

Generate an aerosol of oleic acid mist within the test chamber. Use a real-time sampling instrument such as the DataRAM or Dust Trak II to measure the mass concentration of aerosol within the chamber. Aim for a mass concentration of about 30 mg/m^3 . Estimate how long you will have to sample to collect a reasonably weighable amount of mist on the impactor stages, and sample for that period. During the same time, sample using the APS or Aerosizer.

Analysis

Convert the size distribution by count measured using the Aerosizer into a size distribution by mass. Do not assume log-normality of the size distribution. Also calculate, from the count data, the overall mass concentration of the aerosol from the Aerosizer data. Compare the overall mass concentrations determined by the real-time sampling instrument, the Aerosizer, and the cascade impactor. Compare the size distributions measured using the Aerosizer and cascade impactor. Which measurements are correct? Discuss reasons for any disagreements.

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CLASS PROJECT

Background

You have now used many of the instruments in our laboratories that measure aerosol properties. The instruments you have used include the optical microscope, the cascade impactor, the scanning electron microscope and the Aerosizer instrument. We have used these instruments to explore the characteristics of aerosols.

Procedure

We will define a project, then carry it out in our lab and prepare a report based on our findings.