ENVR 416
Aerosol Technology – Laboratory Session
Fall 2009

INSTRUMENT CALIBRATION USING PSL SPHERES

The objective of this lab is to become familiar with the operation, characteristics, and limitations of a real-time instrument used to measure particle size and concentration.

Background

You have now used the optical microscope to count and size particles. Although optical microscopy has advantages it can be tedious. This week, we will use an optical instrument, the Aerosizer, formerly made by Amherst Process Instruments in South Hadley MA, to count and size particles. This instrument has now been phased out of production and is no longer available; however, many aerosol laboratories, including ours, continue to use them.

We have three Aerosizers in our lab. Because this instrument measures particle diameter indirectly, we must be sure of its calibration. This week, we will check the calibration of the instrument using polystyrene latex spheres of known size.

The manufacturer's manual for the Aerosizer is in the laboratory. Aerosol flows through a jet that accelerates the particles. Because they have higher inertia, larger particles accelerate more slowly than smaller particles; as a result, larger particles attain a lower velocity than smaller particles by the time they leave the jet. After leaving the jet the particles pass through two laser beams in series. Light scattered from the particle as it crosses the first beam enters a photodiode and starts a timer, and when the particle crosses the second beam, the light it scatters enters a second photodiode that stops the timer. The distance between the beams divided by the transit time gives particle velocity. Velocity is related to particle size through the software that runs the instrument. Note that the scattered light is used only to start and stop the timer and is not used directly to size the particle. Thus, unlike some optical instruments, the Aerosizer is not sensitive to differences in particle shape or refractive index.

Another instrument that operates using similar principles is the "Model 3321 Aerodynamic Particle Sizer" (APS), also manufactured by TSI. See

http://www.tsi.com/en-1033/products/13996/aerodynamic_particle_sizer%C2%AE_spectrometer/2204/3321.aspx

Both the Aerosizer and the APS use a nozzle to accelerate particles through two ribbons of laser light, then time the interval between the resultant light pulses. The instruments differ in the way they configure the laser light, in how they process the data, and how they present the results. In addition, the Aerosizer is larger and heavier than the APS. The cost of each instrument is, or was, about $42,000; an aerosol diluter for each instrument costs an additional $8000. We also have an APS and APS diluter in our lab.

Until several years ago the Aerosizer and the APS were manufactured by different companies. Then TSI bought the company that manufactured the Aerosizer, took the Aerosizer out of production, and eliminated the competition. Capitalism plays a role, even in aerosol physics.

Limitations of the Aerosizer

An automatic instrument such as the Aerosizer must accomplish two tasks: (1) it must size particles correctly, and (2) it must count all the particles that pass through it. If these two criteria are met, then measured size distributions will be correct. These two questions are independent so they can be studied and discussed separately. Some general comments on both these questions are given below. In this lab, we will determine whether the Aerosizer measures size accurately. We will not investigate the question of counting efficiency.

Particle Sizing

Dr. Paul Baron, an aerosol researcher at NIOSH, has recently reported[1] an error in measured droplet size that occurs in both the Aerosizer and in the APS. The error occurs because droplets distort when they pass through the air jet that accelerates them. The distortion increases their drag, with the result that they accelerate more quickly than they would if they were solid spheres. Paul developed an equation that relates true droplet diameter to measured droplet diameter for both instruments:

$$d_{true} = d_{measured} + \frac{a\, d_{measured}^2}{\sigma^b\ \eta^c}$$

where:

d_{true}	is true particle diameter in micrometers
$d_{measured}$	is measured particle diameter in micrometers,
σ	is oil surface tension in N/m
η	is oil viscosity in Pa s
a, b, and c	are coefficients as given in the table below

Coefficients for Use in Baron's Equation for Droplet Distortion in Sizing Instruments

	a	b	c
Aerosizer	4.061×10^{-4}	0.9583	0.2516
Aerodynamic Particle Sizer (APS)	1.222×10^{-4}	0.5956	0.6916

Values for the properties of several oils used commonly in aerosol work are below.

Oil	η, Pa s	σ , N/m
Oleic Acid	0.0256	0.032
DOP	0.081	0.0309
DOS	0.027	0.0322

The problem of incorrect size due to droplet distortion occurs only when the aerosol is comprised of liquid particles. It can be important for oil droplets larger than about 4 or 5 μm in true diameter.

In our own work, this problem is important because we frequently use oils as test aerosols. We often use oleic acid in our research, but for this liquid droplet distortion is important for droplets larger than about 5 μm in diameter. We have used a 1:10 solution of glycerin in methanol when we need to make larger droplets. The methanol evaporates, leaving a residual particle of glycerin. The viscosity and surface tension of glycerin are such that droplet distortion is minimal even for droplets up to 10 μm in diameter.

Counting Efficiency

Aerosizer - We have found that the counting efficiency of both the Aerosizer and the APS, defined as particles detected divided by particles passing through, is not constant. Ideally, the counting efficiency should be 1.0 for particles of all sizes. Jon Thornburg, a doctoral student in our group who graduated several years ago, measured Aerosizer counting efficiency for mist droplets up to about 5 μm in diameter[2]. Jon found that counting efficiency varies from zero to over 1.0, and depends on particle size, on particle concentration, on the Aerosizer photomultiplier tube voltage, and varies from instrument to instrument. Thus, size distributions or concentration results from this instrument are not always accurate. To relate instrument output to true concentration for particles of any size, one must divide output by counting efficiency for particles of any given size. Well, what do you want for $50,000?

The Aerosizer DMP that you will use this week uses newer signal processing that should give more consistent counting efficiencies. We conducted a preliminary check of this instrument and found that it seems to operate better than the previous model.

APS - Further experiments by Al Armendariz, another doctoral student in our lab who recently graduated and is now on the faculty of Southern Methodist University, determined that the APS also varies in counting efficiency with particle size[3]. Al also found some other, important limitations of this instrument which have now been addressed in an instrument upgrade available from TSI at a cost of $4900. Tom Peters, a doctoral student in our lab who graduated in 2004 and is now on the faculty of the University of Iowa, checked the performance of the APS after the $4900 upgrade. A paper based on this work has been published in the Journal of Aerosol Science[4]. Some, but not all, of the limitations for this instrument are addressed in the upgrade. Tom and John Volckens, another lab graduate now on the faculty at Colorado State, measured APS counting efficiency for both solid and liquid particles. They found that counting efficiency is high for bouncy, solid particles when the APS inlet nozzle is clean, but lower for liquid particles or if the inlet nozzle is dirty[5].

Instrument Limitations – Nonuniform counting efficiencies are a problem only if one wishes to determine the size distribution of an aerosol. Measured distributions will be inaccurate to the extent that counting efficiencies vary with particle size. In some applications, though, nonuniform counting efficiencies are not a problem. For example, if one wishes to determine the effectiveness of a filter as a function of particle size, one can use an instrument to measure concentration as a function of size both upstream and downstream of the filter. Filter penetration is the ratio of downstream to upstream concentration. Even if counting efficiency is unknown, the measurement of filter penetration will be accurate.

In summary, both the Aerosizer and the APS have the ability to measure particle size correctly. A correction is necessary when measuring the size of liquid droplets. Unfortunately, neither instrument appears to measure concentration or size distribution correctly all the time. The APS works best when it measures the size distribution of bouncy solid particles with a clean nozzle.

Aerosizer Operation

The Aerosizer is controlled by a computer that controls the duration of each sampling run and disposition of the data gathered. Data can be stored on the computer for analysis later, printed, or discarded. You will likely make some mistakes with this program at first; however, no damage to the instrument will result. The worst that can happen is that you will lose data. If you have questions about the Aerosizer or how to operate it, please ask Maryanne Boundy or me. We both have experience with the instrument.

Regardless of how fancy or expensive an instrument is, its performance should be checked periodically. This can be accomplished by passing an aerosol of "monodisperse" solid particles through the instrument to see whether it sizes them correctly. This process is repeated for particles of different sizes to assure that the instrument works properly for particles of all sizes of interest.

Procedure

The calibration particles are microspheres supplied by Duke Scientific Co., which has recently been acquired by Thermo Scientific. See:

http://www.thermo.com/com/cda/product/detail/1,,10136505,00.html

Note that each small vial of particles we use costs more than $100. The supplier gives the size of the particles in each vial. For careful work, this size should be checked by examining the particles using an optical or scanning electron microscope. For this lab, we will assume the sizes given on the vials are correct; however, articles in the literature suggest the sphere diameters given by the manufacturer are sometimes in error by as much as 30%.

The spheres we will use are made from polystyrene latex (psl). They come in a water suspension with a surfactant that helps keep the particles from agglomerating while in the bottle. Before using these spheres, place the vial in a beaker, and the beaker in the water of an ultrasonic bath. Run this bath for one minute or so before using the spheres to break up any agglomerates that may have formed in the bottle.

The screw caps on the bottles of these spheres are not completely air tight. As a result, the water in the bottle will sometimes evaporate over time while the bottles are stored and not used. When you go to the drawer to use the suspension of psl spheres, you find a dried-out lump of white plastic at the bottom! Maryanne Boundy has found that the psl spheres can be effectively resuspended by adding distilled water to the dried out lump, then placing the bottle in an ultrasonic bath for several minutes. This way, you can save the cost of a new bottle of spheres, if the old one dries out.

In this lab, psl particles will be aerosolized from solution using a nebulizer. Nebulization is a good way to generate concentrations of psl particles smaller than about 3 or 4 μm in diameter. Larger particles cannot be generated by a nebulizer because the droplet sizes produced are mostly smaller than about 5 μm, and it is not possible to get a large particle into a small drop.

The nebulizer produces droplets, many of which contain one or more polystyrene latex spheres. The droplets evaporate, leaving the spheres in suspension in the air. To help with evaporation, we will bleed some lab compressed air into the output from the nebulizer, then pass the aerosol through a drying column so the droplets evaporate entirely. The aerosol that comes out the top of the drying column should contain only the polystyrene latex spheres.

The nebulizer we will use was formerly supplied by the maker of the Aerosizer, along with a dry powder disperser for an additional cost of about $4000. Several manufacturers can supply nebulizers; many of them were developed to generate aerosols of drugs used for inhalation therapy. We routinely use a single-jet and a six-jet Collison nebulizer, but also use a HEART nebulizer. The Collison nebulizers are available from BGI Inc., Waltham, MA for about $600 each. BGI is a good source for all sorts of aerosol supplies; see

http://www.bgiusa.com/

The HEART nebulizer was previously supplied by Vortran Technologies in California, and cost about $25. This company has also been bought out, and these nebulizers are no longer available.

Aerosizer Operation

To operate the Aerosizer, first turn on the computer that controls it. The program should load automatically. If not, at the >TSI prompt, type “aero3220” to execute the Aerosizer control program. A menu for instrument operation appears on the left of the display. To begin a sampling run, press the F7 key. The instrument is set up to sample for one minute, although this option can be set to a different value by the software.

Each second, the instrument will count and size particles and show the display on the screen. At the end of the sampling period, the computer will compile the data and show a composite size distribution. Press the F9 key to obtain tabulated data for the run just made. The data will also be stored automatically in a file on the hard disk of the computer. After looking at the tabulated data on the screen, press F1 to return to the data plot, and F1 again to return to the menu. To take a second sample, press F7 again.

Experiment

Part I: Aerosizer Calibration

Place about two drops of the liquid from a vial of psl into some distilled water within the sump of the nebulizer, then close the nebulizer. Be careful that the O-ring at the top of the nebulizer sump does not pinch on the nebulizer cap as you close the nebulizer. The O-ring must seal well for the nebulizer to work properly.

Press the F7 key to start the Aerosizer. From the display at the end of the run, decide what size best describes the aerosol you have measured. Look for the presence of peaks in the frequency distribution plot associated with doublets and triplets. These form when a nebulizer droplet contains more than one particle.

Measure the sizes of several psl spheres using the Aerosizer. Between each measurement, rinse out the nebulizer sump with distilled water. Also, try nebulizing some distilled water without particles, for comparison. Compare the measured sizes with those on the bottle, and decide whether the instrument is in calibration for the sizes you have measured.

Part II: Room Air Characterization

Next use the Aerosizer to measure the concentration and size distribution of laboratory air. Disconnect the inlet of the Aerosizer from the nebulizers so that the instrument draws in air from the room. Press the F7 key to start the Aerosizer and save the data as before. In this way take at least three one-minute samples of the particles in room air.

Analysis and Reporting

Part I: Aerosizer Calibration

1. Develop a frequency distribution for the psl particles you use, and for each identify the mode of its distribution. Plot data for all the particle sizes you test using the same figure.

2. Plot a cumulative distribution by count using log-probability paper for the same data, and for each particle size determine the count median diameter. Use the same log-probability figure for all the particle sizes you test.

Think about what size best characterizes each of the psl particles you have measured - the mode, the median, or some other characteristic diameter. Do the measured sizes correspond to the size on the vial? If not, how do they differ?

Part II: Room Air Characterization

3. On the same figure, plot frequency distributions for each of the one-minute samples of lab air that you took

Think about both the accuracy and the precision of your measurement. Precision is related to whether the multiple measurements you made of the same aerosol agree with each other. Do they? Accuracy is more difficult to establish, because it requires knowledge of the "true" aerosol against which the "measured" aerosol can be compared. How can we know the true aerosol? What might limit the accuracy of this instrument, particularly for particles at the larger and at the smaller ends of the frequency distribution?

As with the report you prepared for the first experiment in this course, you and your lab partner should prepare a single report. Be sure to put both your names on the first page of the report. The report should describe very briefly what you did, then present your results and any conclusions you reached in doing this exercise. The total length of your report should not exceed three pages including all figures and tables. The report should be prepared neatly. Your grade for the report will count the same as a quiz. Both you and your lab partner will receive the same grade.

References

1. Baron, Paul, “Size Shifts in Measurements of Droplets with the Aerodynamic Paeticle Sizer and the Aerosizer”, Aerosol Science and Technology, 42: 201-209 (2008).

2. Thornburg, Jonathan, Steven J. Cooper and David Leith, “Counting Efficiency of the API Aerosizer”, *Journal of Aerosol Science*, **30** (4): 479-488 (1999).

3. Armendariz, Alfredo and David Leith, “Concentration Measurement and Counting Efficiency for the Aerodynamic Particle Sizer 3320” *Journal of Aerosol Science*, **33**: 133-148 (2002).

4. Peters, Thomas M. and David Leith, “Concentration Measurement and Counting Efficiency of the Aerodynamic Particle Sizer 3321” *Journal of Aerosol Science*, **34** 627 – 634 (2003).

5. Volckens, John and Thomas M. Peters, “ Counting and Particle Transmission Efficiency of the Aerodynamic Particle Sizer,” *Journal of Aerosol Science* **36** 1400-1408 (2005).

Schematic for the operation of the Aerosizer

The instrument was formerly sold by Amherst Process Instruments, South Hadley, MA, and is now available from TSI, Minneapolis, MN