Chapter 9

Aerosol Sampler Calibration

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Introduction

All sampling instruments require calibration. Some instruments, such as photometers, have no means to theoretically predict response. These instruments rely on empirical calibrations using standard test aerosols. Other instruments, such as the optical counter, impactor, and condensation nucleus counter (CNC), can be described by theories that predict their instrument responses. Yet, the actual performance of these instruments may differ from the theory because not all factors are taken into account in these theoretical models. For example, impactor cutoff diameters can be calculated for ideal sticky aerosols; however, such calculations do not take into account particle bounce, re-entrainment, electrostatic charge effects, and wall losses.(1, 2) Evaluation of the actual performance of the impactor requires experimental calibration. To obtain data that are of high quality, reproducible, and defensible for the intended purposes, the instrument must be calibrated. Furthermore, such calibration must be appropriate to the intended application.

Most instruments are calibrated and evaluated by the manufacturer or the inventor before being used by others. For an instrument intended to collect and analyze an aerosol, collection efficiency and wall losses are generally determined in the calibration. For a real-time, direct-reading instrument, calibration establishes the relationship between the instrument response (e.g., electronic signal or channel number) and the value of the property (e.g., particle size, number concentration, or mass concentration) being measured. However, the operating conditions and the parameters used during the original calibration can vary from those under which the eventual user operates. As a result, the original calibration data may not apply, and the user must recalibrate the instrument to operate it with confidence. In general, a reliable and accurate calibration process requires: 1) a sufficient knowledge of the capabilities and limitations of an instrument; 2) appropriate test facilities; 3) proper selection of a desired test aerosol; 4) a thorough investigation of relevant parameters; 5) sufficient knowledge about the conditions that can be encountered during operation; and 6) a quality assurance program that is followed throughout the test.

In the last two decades, developments in the generation and classification of monodisperse aerosol, along with the improvement of test facilities, have made instrument calibration easier and the results more reproducible. Calibration methods for air sampling instruments have been reviewed.(3, 4) Chapter 7 discusses the calibration and use of flow monitoring devices that play an integral role in aerosol sampling and instrument calibration. This chapter reviews the calibration techniques relevant to aerosol samplers, such as sizing instruments, devices for concentrations, and size-selective samplers. Test facilities, generation of test aerosols, and testing procedures will be emphasized.

General Considerations

Before embarking on a rigorous instrument testing/calibration program, decisions must be made on the frequency of calibration. General descriptions of instrument components and the measured parameter for each aerosol instrument are also discussed here. Finally, the sampling environments, which will influence the selection of appropriate test facility and procedures, must also be considered.

Components of Aerosol Samplers

An aerosol sampler usually consists of a sampling inlet, a detection or collection section, an air mover, and flow controllers. The sampling inlet, which is the entrance to the instrument, is connected to the detection section with a short transport line. The air mover (usually a vacuum pump) draws air into the sampler, and flow controllers control flow rates. Personal samplers and some area samplers, including impactors and filters, have separate pumps and flow meters. A few passive samplers, such as personal photometers, do not have an air mover and flow meter; they rely on air currents in the atmosphere to bring the aerosol into the detector. Most direct-reading aerosol instruments, such as optical counters, CNCs, and photometers, include the sampling inlet, transport line, detector, pump, and flow meter in a single unit. Each unit is indeed a complete system. For other systems such as filter samplers, individual components must be assembled.

It is important to know that there can be significant particle losses in the sampling inlet (aspiration efficiency) and transport lines (transport losses), especially for very large (> 5 μm) and small particles (< 0.01 μm). In addition, every instrument has finite detection limits and detection efficiency. The flow meter controls a constant volumetric flow rate so that the instrument can be operated properly and that accurate sampling volume and, therefore, aerosol concentration can be determined. Calibration of the flow meter is a part of the total instrument calibration. Recalibration of a flow meter is required when it is used at a high altitude or under ambient conditions (temperature, pressure, and gas composition) different from the original factory calibration. Calibration of the flow meter is usually the first step in the process of instrument calibration.

Measured Parameters

Depending on the function of the instrument, the measured parameter can be separated into particle concentration (number or mass) and particle size distribution. For size-selective samplers, mass concentrations for inspirable, thoracic, or respiration fraction will be determined. Often both parameters have to be considered in the instrument calibration. For example, in impactor calibration, the collection efficiency as a function of particle size taking into account the sampling and transport losses has to be determined. We recommend that the investigator measure the detection efficiency of the whole system including the aspiration efficiency of the inlet, losses in the transport lines, and efficiencies of the detector or sensor.

Each instrument has finite detection range and is, therefore, useful in that range. The applicable size range is based on the sampling principle, the detector efficiency, and the inlet design. For example, inertial-type instruments such as impactors usually collect particles between 0.5 and 15 μm; for diffusion batteries, the size ranges between 0.005 and 0.5 μm. For optical instruments, the lower detection limit is about 0.2 μm. Therefore, the instrument influences the selection of the test aerosol. In most calibrations, particles having a size in the applicable range of the instrument are required to establish the calibration curves. Also, most instruments have minimum and maximum detection limits in aerosol concentration. For example, instruments that are based on the detection of scattered light for single particles have very low maximum concentration limits (in the order of 100 particles cm^{-3}). At higher concentrations, increased coincidence errors are due to the presence of two or more particles in the sensing volume of the detector. For aerosol collectors, overloading the substrates causes sampling errors; therefore, appropriate sampling time and mass concentration have to be considered.

Sampling Environments

Depending on the wind speed in an environment, the sampling procedure can be classified as calm air sampling or sampling in the flow stream. Calm air sampling generally refers to a wind speed less than 50 cm/s and applies to indoor environments, including residential homes, offices, and factories. Flow stream sampling refers to environments with higher wind speed, such as ambient atmosphere or inside ventilation ducts and stacks. The airflow pattern in an environment affects particle movement and, therefore, is an important parameter for the inlet aspiration efficiency. Criteria for calm air and flow stream sampling have been discussed by Davies,(5) Hinds,(6) and Brockmann.(7) To simulate various flow conditions in the environment, test facilities with different capabilities should be considered. Instrument chambers with uniform, low air speeds are suitable for testing samplers under calm air conditions, and aerosol wind tunnels are required for testing samplers under flow stream conditions.

Test Programs

Aerosol instruments can be tested on several levels for performance. The decision on the appropriate test program is largely driven by regulatory and/or scientific needs. The three levels of test programs include:

1. Flow calibration and system integrity
2. Single point check
3. Full-scale calibration.

The simplest test procedure checks flow rates and system leaks. Scheduled and frequent checks are recommended for routine use of any instrument. However, passive samplers such as a personal mass monitor or personal DataRAM, do not require flow calibration.

The next level of testing involves instrument response for a single point. For an aerosol sizing instrument, a test aerosol (usually polystyrene latex [PSL] particles) of a defined size is used, and the response is compared to an existing calibration curve. This procedure assumes that a full-response calibration curve is available, and the user performs the test to make sure the instrument is functioning normally. A full-scale calibration requires testing the instrument response over its full operational range. Therefore, the calibration curve in terms of response as a function of particle size or concentration can be established. The efforts, equipment, and test facilities needed to perform these programs increase substantially from the simple flow calibration to the full-scale test. Only a standard flow meter and a pressure gauge are needed for a flow calibration and system check. The effort is minimum, and the benefit to the user in terms of improved quality of data is high. For a single point test, aerosol generation and monitoring systems are needed, but if they are available in the laboratory, the effort required is relatively small. A full-scale calibration requires more elaborate test facilities and extensive efforts in terms of time and labor for the exercise. Essentially, any user can perform a regular flow check, and any laboratory with experience in aerosol instruments can check a single point. However, only aerosol laboratories with appropriate facilities and equipment can perform a full-scale calibration.

When Should an Instrument Be Calibrated?

Full-scale calibration is needed to establish a calibration curve for each new instrument. Therefore, one would assume that the manufacturer or instrument developer should provide such data; however, there are some commercial instruments lacking calibration data. In many cases, independent investigators provide careful evaluation and calibration of such instruments, and their results are usually published in the open literature.

A user needs to obtain aerosol measurement data of high quality in order to meet scientific guidelines or regulatory standards established by government agencies. The user must ensure that the instrument performs according to its specifications. The flow and system integrity should be checked whenever possible, and a single point check should be considered for scientific validation. Decisions on full-scale calibration to a large extent depend on the scientific justifications and regulatory requirements for each study. Aerosols are usually measured for scientific research, regulatory compliance for health protection purposes, and toxicity testing.

For scientific research, calibration data provided by the manufacturer or published in a scientific journal can be utilized, when the instrument is used under normal conditions. If the instrument is used under different ambient pressures or flow rates, then it may need to be re-calibrated under the actual operating conditions. At a minimum, the flow meter must be calibrated and a single point check performed to see whether the instrument response differs from the original calibration.

Aerosols in work environments are measured by industrial hygienists because of mandates by government agencies including the U.S. Occupational Safety and Health Administration (OSHA) or Mine Safety and Health Administration (MSHA) regulations. Similarly, determination of release of aerosols to the ambient atmosphere from fixed-point and mobile sources is required by the U.S. Environmental Protection Agency (EPA) regulations. These regulations often specify standard sampling methods or equivalent methods that follow the same performance specifications. For example, the EPA performance specifications and test procedures applicable to a size-selective instrument, PM_{10} are contained in 40 CFR Part 53-Ambient Air Monitoring Reference and Equivalent Methods.(8) The PM_{10} samplers should be tested in a wind tunnel with liquid particles (10 sizes ranging from 3 to 25 μm in aerodynamic diameter) at wind speeds of 2, 8, and 24 km/h. The 50% cut-off determined for each speed must be 10 ± 0.5 μm. The precision for determination of concentration and flow stability is also specified.(9)

Finally, toxicity tests using inhalation exposures for new products or chemicals must be conducted under Good Laboratory Practice Standards established by the U.S. Food and Drug Administration (FDA)(10) and the Toxic Substances Control Act Test Guidelines promulgated by EPA.(11) Based on these guidelines, specific procedures for the calibration of mass monitors to determine aerosol concentrations in exposure chambers are required. For example, the laboratory must provide data on instrument responses of real-time aerosol monitors for the test material, with filter samples as a reference standard in the concentration range used for the study.

Calibration Standards

Calibration curves are generated by comparing responses from the test instrument to those of a calibration standard. Several standard methods are now available for calibrating aerosol instruments. The primary standard method for particle size and number concentration determination is the microscopic examination, whereas the gravimetric method is the primary standard for mass concentration determination. However, several secondary standard methods have been developed and frequently used because they are often easier to use than the primary method. Table 9-1 lists aerosol instruments and test standards that have been used for their calibration. The following describes each test standard in detail.

Direct Measurement with Microscopy

The primary standard method of instrument calibration is direct measurement of collected particles under an optical or electron microscope. This technique provides information on particle size distribution and particle number concentration. For example, a number concentration determined from an aerosol collected in a liquid impinger has long been used to determine dust levels in occupational environments. Also the response of a CNC was calibrated against a photographic CNC(12) in which the particles are photographed and the total number of the particles counted to yield the number concentration. However, this technique is labor-intensive, and the observations varied substantially among different operators. With advanced imaging techniques, the procedure can be automated, and the accuracy of the measurements in both size and number concentration determinations is greatly improved. The microscopic technique is still used routinely in determination of particle size and number concentration of fiber aerosols. It is also used to determine the number concentrations of bioaerosols. It is a good quality assurance practice to check the size of standard test aerosols with the primary method during the test.

One problem in using the microscopic method to determine the diameter of a liquid droplet is deformation of the droplet on a collection surface. Because of surface tension, a droplet loses its spherical shape and forms a shape similar to a segment of sphere with a height of h and diameter of D as shown in Figure 9-1. This diameter, D, is larger than the actual diameter, d, and the ratio of the projected and the actual diameter, $B = d/D$, is called the spread factor. This factor is a function of the surface tension of the droplet and the adhesion force between the particle and the surface; the spread factor is needed for accurate size measurement of droplets using the microscopic technique. For routine collection of known test aerosols such as dioctyl phthalate (DOP), oleic acid, and dibutyl phthalate (DBP) on glass slides

TABLE 9-1. Calibration Standards of Aerosol Instruments

Instrument	Measured Parameter	Particle Size Range (μm)	Calibration Standard
Size Measurement			
Cascade impactor	Flow rate, gas medium, physical dimension in and around the nozzle	0.05–30	Monodisperse spherical particles with a known size and density
Aerodynamic particle sizing instrument	Flow rate, pressure, gas medium	0.5–20	Monodisperse spherical particles with a known size, shape, and density
Optical particle counter	Wavelength of the light source, range of scattering angles, sensitivity of detector	0.3–15	Monodisperse, spherical particles with a known size and refractive index
Electrical mobility analyzer	Flow rate, charging mechanism, electric field strength	0.001–0.1	Monodisperse, spherical particles with a known size
Diffusion battery	Flow rate, temperature, deposition surface	0.001–0.1	Monodisperse, spherical particles with a known size
Number Concentration Measurement			
Condensation nuclei counter	Flow rate, saturation ratio, temperature gradient	0.001–0.5	Electrical classifer with electrometer
Mass Concentration Measurement			
Photometer	Wavelength of the light source, range of scattering angles, sensitivity of detector	0.3–1.5	Gravimetric measurement of filter samples
β-attenuation monitor	Uniformity of particle deposit	1–15	Gravimetric measurement of filter samples
Quartz crystal mass balance	Sensitivity of the sensor	0.02–10	Gravimetric measurement of filter samples

coated with a special agent, spread factors can be developed.(13–15) Using a test aerosol generated from a vibrating orifice monodisperse aerosol generator (VOMAG) as described in the section on "Test Aerosol Generation," the spread factor can be determined by comparing the measured diameter, D, and the theoretically calculated aerosol diameter, d. A more general but direct method has been described by Cheng *et al.*(16) This technique is used to determine the volume of a deformed droplet by examining the droplet under a microscope with an angle other than 90° (Figure 9-1). From the measured axes of the projected image, the height of the spherical segment is calculated, and therefore the volume equivalent diameter d is also calculated.

Particle Size

Monodisperse, spherical solid particles of PSL have been used widely as a secondary test standard to determine the responses of aerosol sizing instruments including optical counters, impactors, and other real-time monitors. These particles are uniform, spherical, and smooth with the aerosols having very small standard deviations. The particle sizes have been determined by the manufacturer using microscopic techniques. They have well-defined physicochemical properties, including the refractive index, density, and chemical composition. They are commercially available in the size range of 0.03 to 200 μm and are relatively easy to use.

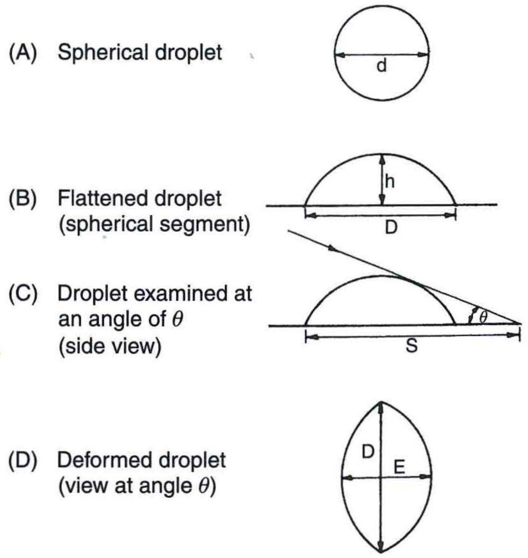

FIGURE 9-1. Schematic of drop particles collected on a surface.

A VOMAG or an electrostatic classifier (EC) is also frequently used to produce monodisperse spherical test aerosols with well-defined characteristics. The VOMAG produces particles in the size range of 1.5 to 30 μm and the EC in the size range of 0.003 to 1 μm. The sizes of these test particles can be accurately predicted based on the operational principles, when the devices are operated appropriately. Theoretical sizes have been verified with the primary standard using microscopic techniques.(17, 18)

Number Concentration

The primary standard for calibration of number concentration is to collect aerosol particles using a high efficiency membrane filter and then to count the particles using a microscope. From the number of particles counted in a unit area, the total collection area, and the volume of air sample, the number concentrations (particles cm^{-3}) can be estimated. A secondary standard and more commonly used method is the electrometer technique, which measures the electrical current of mobility-classified aerosols produced by the EC.(19) A monodisperse test aerosol generated by an EC carries mostly a single positive charge. By measuring the electrical current with an electrometer, the aerosol concentration can be determined by dividing the measured current with the volumetric flow rate and the elementary electric charge.(18) For particles greater than 0.05 μm, a significant portion of the charged particles (depending on the aerosol size distribution) would include doubly-charged particles; therefore, corrections for the doubly-charged particles are needed to obtain an accurate number concentration from this method.(18) The electrometer technique is very useful for particles smaller than 1 μm.

For micrometer-size particles, a secondary method is to determine the mass concentration by the gravimetric or colorimetric method of monodisperse, spherical test particles, and then to convert the mass concentration (C_m, mass/volume) to the number concentration (C_n, particles/volume):

$$C_n = \frac{C_m}{\frac{\pi}{6}\rho_p d_{avg}^3} \qquad (1)$$

where ρ_p is the particle density, and d_{avg} is the particle diameter of the average volume.

Mass Concentration

The primary standard for aerosol mass concentration is to pass a known volume of an aerosol through a high-efficiency filter and determine the increase in mass of the substrate due to the collected aerosol particles. The mass can be determined using either an analytical balance (10 μg precision) or an electronic balance (1 μg precision). Mass concentration is obtained by dividing the increased mass by the gas volume sampled. Colorimetric or fluorescent techniques can be used to determine the mass concentration of test aerosols tagged with dye. Generation of tagged particles will be discussed in the section on "Test Aerosol Generation." Criteria for representative samples in either the flow stream or calm air sampling conditions should be followed to minimize sampling biases.

Calibration Systems and Test Facilities

Components of Calibration Systems

Figure 9-2 is a schematic diagram of a typical calibration apparatus for aerosol instruments. It includes an aerosol generator, conditioning devices, a flow mixer, a

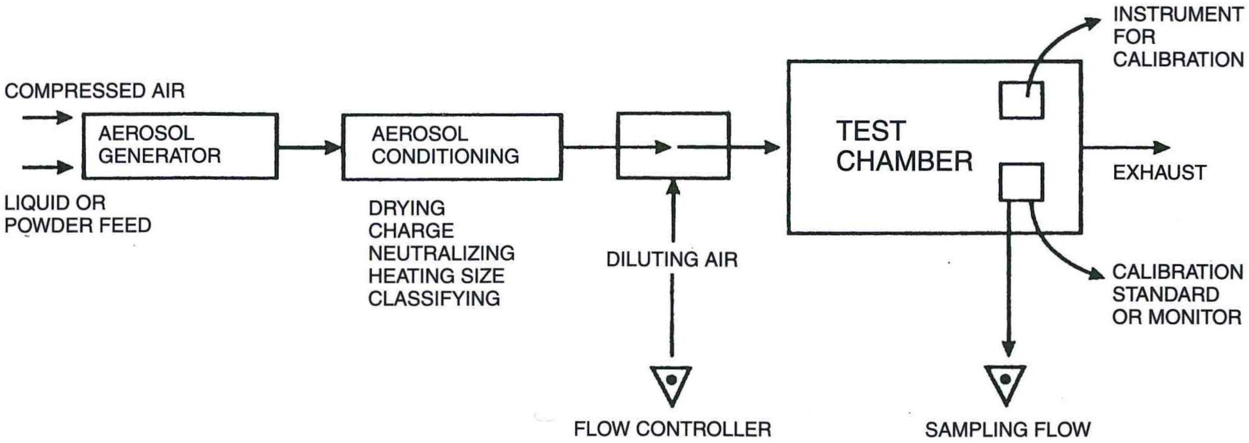

FIGURE 9-2. Schematic of a typical calibration system for aerosol samplers.

test chamber, pressure and air flow monitoring equipment, the instrument to be calibrated, and the calibration standard. The aerosol from the generator can be monodisperse or polydisperse, solid or liquid, wet or dry, charged or uncharged, or spherical or nonspherical (described later). Generally, this aerosol requires several steps of conditioning before use. For an aerosol containing volatile vapors or water droplets, a diffusion dryer with desiccant and/or charcoal is commonly used to remove the solvent. In some cases, a heat treatment using a high-temperature furnace is required for the production of a test aerosol.(20, 21) The heat treatment involves either sintering or fusing the particles to reach the desired particle morphology and chemical form, or initiating particle evaporation and subsequent condensation to produce monodisperse particles. Because aerosol particles are usually charged by static electrification during formation, a neutralizer containing a bipolar ion source (e.g., ^{85}Kr, ^{241}Am, and ^{63}Ni) or corona discharge in an air jet are often used in the aerosol treatment.(22, 23) This reduces the number of charges on the particles and results in an aerosol with charge equilibrium. In addition, a size-classifying device is often used in the aerosol treatment to segregate particles of a similar size or of a desired size fraction.(24–26) Furthermore, a concentrator or a dilutor is often used to adjust the aerosol concentration.(27, 28)

Test Facilities

The desired test aerosol is then delivered to the test chamber, where the instrument under testing and the test standard are located. Test facilities may include mixing chambers, instrument chambers for calm air sampling, and aerosol wind tunnels. All test chambers require uniform aerosol concentrations in the test section where the aerosol instrument is located. However, the aerosol wind tunnel can provide a test section with various wind speeds for testing a size-selective aerosol sampler under simulated ambient flow conditions. Vertical instrument chambers with low velocity are used for calm air sampling. Mixing chambers with small, simple plenums are used to provide steady aerosol streams for limited calibration of the detection or collection efficiency of the instrument detector. These chambers usually do not provide performance data for calm air or flow stream sampling conditions. The space, facility requirements, and cost associated with these three types of test chambers are quite different. Mixing chambers are usually placed on top of a laboratory bench, whereas the instrument chamber requires a much larger space, and the wind tunnel often occupies a whole room. A vacuum cleaner or pump is sufficient to move air within the mixing chamber. For vertical chambers, air movers with capacities between 200 and 1000 L/min are required, whereas larger fans are part of the wind tunnel systems.

Mixing Chambers

A mixing chamber is usually a small cylindrical chamber made of glass, plastic, or metal, where the aerosol can be uniformly distributed (Figure 9-3). The aerosol instrument is placed outside the chamber. Samples are taken via sampling probes inside the chamber and delivered to both the instrument to be calibrated and the calibration standard or monitor. Pressure in the chamber and flow rates through instruments are monitored. Multiple samples can be taken simultaneously to characterize the test aerosol, including aerosol concentration and size distribution. The key to successful calibration is to ensure that the same aerosol (size and concentration) is delivered to both the test standard/monitor and the instrument to be calibrated. The same sampling probes and transport line between the probe and the instrument inlet should be used. Also, the same flow rate through the test standard/monitor and the instrument is preferred. If the same flow rate can be used for the tested instrument and the standard, we recommend sampling a single aerosol flow from the chamber, then splitting the flow in two with a three-way valve. Alternate sampling by switching the valve between the tested instrument and the calibration standard minimizes experimental errors. In addition, an aerosol divider is a common sampling port for calibrating a mass monitor.(29) In the aerosol divider, the flow is split isokinetically into two streams: one passes directly into the instrument to be calibrated; the other flows through the calibration standard (Figure 9-3).

The mixing chamber is an inexpensive piece of equipment that is easy to setup and does not require a large working area. Therefore, many instrument user and factory calibrations have been performed in this fashion. The mixing chamber can be used to determine detector efficiency, such as the counting efficiency of a CNC; collection efficiency, as well as internal losses of impactors; and response of real-time monitors. It is especially useful for smaller particles; for particles larger than 3 to 5 μm, it is increasingly difficult to provide a stable aerosol stream in sufficiently high concentrations. Because the instrument is placed outside the chamber, it is not possible to test the aspiration efficiency of the instrument inlet under different wind conditions.

Test Chambers for Calm Air Conditions

Another way of calibrating an instrument is to introduce an aerosol into a test chamber that contains the

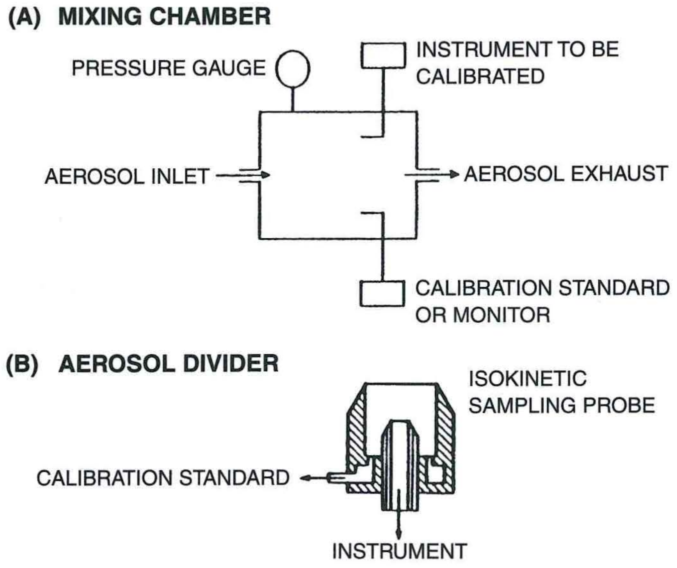

FIGURE 9-3. Schematic of a setup for instrument calibration using a mixing chamber (A) and an aerosol divider (B).

subject instrument and the test standard/monitor. This type of chamber usually has a large cross-sectional area in the test section, and therefore a low air velocity (<50 cm sec^{-1}), simulating quiescent atmosphere. For example, large exposure chambers for inhalation experiments(30) have been used for calibration of real-time aerosol monitors.(31) Also, dedicated instrument chambers have been designed for calibration of several instruments simultaneously.(32–34) A vertical flow instrument chamber with large test volume (1.8 m^3) similar to the design of Marple and Rubow(34) is shown in Figure 9-4. The test aerosol enters vertically from the top of the chamber. The opposing air jets create turbulence to mix the aerosol. To improve the uniformity of aerosol distribution, a 25-cm box fan is placed underneath the chamber entrance for mixing. A 10-cm-thick honeycomb structure is inserted above the test section of the chamber to reduce eddies created by flow turbulence and present the sampling area with a well-defined downward air flow. To ensure that spatial variations of concentration and size distribution are minimized, samplers are placed on a 75-cm platform rotating at 0.5 or 1 rpm. Variations of aerosol concentrations in the sampling platform based on filter samples are less than 5.5% for particles of 12 μm.

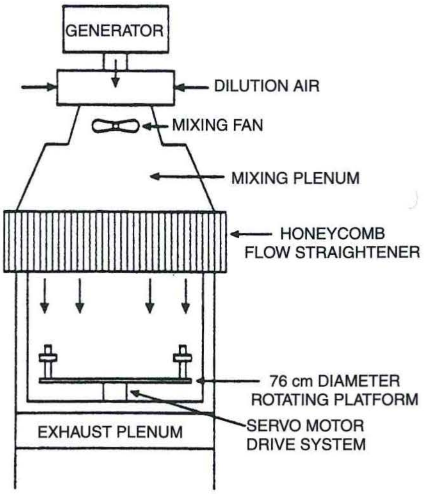

FIGURE 9-4. Schematic of a vertical aerosol instrument chamber.

This type of instrument chamber provides uniform concentrations of aerosols in the test section for large particles. Several instruments could be placed inside the chamber for simultaneous calibration and comparison. The flow rate and turbulence intensity in the chamber are low, simulating calm air sampling conditions.

Aerosol Wind Tunnels

Aerosol wind tunnels have been used to test many aerosol samplers for wind speeds between 0.5 and 10 m sec^{-1}.(9, 35–37) Two types of wind tunnels are commonly used: an open circuit tunnel and a closed circuit tunnel. Figure 9-5 shows a diagram of an open circuit tunnel. Room air is taken into the system with the test aerosol and then it passes through honeycomb screens to reduce the turbulence and to provide uniform velocity. A section of contraction cone connects the test section and the screens. The test section usually has a rectangular shape and is large enough to accommodate the instrument and filter samplers with isokinetic inlets. The cross-sectional area of the test instrument should not occupy more than 10–15% of the test section so that the uniform velocity and aerosol profiles will not be affected. The aerosol then is filtered through a HEPA filter bank and clean air is exhausted to the room. A fan with sufficient capacity is used to move the air through the tunnel. Large open circuit wind tunnels that can accommodate a manikin are also used extensively to study the inspirability of large particles.(35, 38, 39)

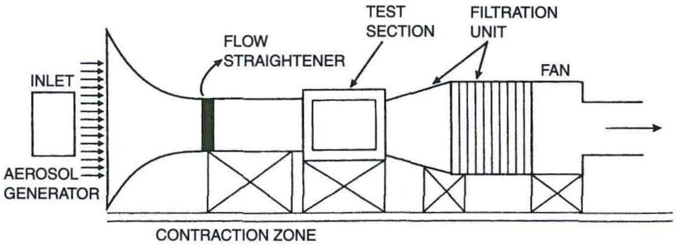

FIGURE 9-5. Schematic of an open circuit aerosol wind tunnel.

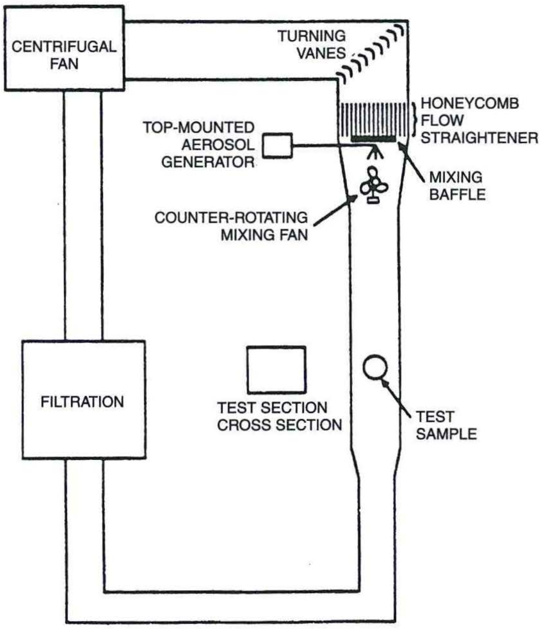

FIGURE 9-6. Schematic of a closed circuit aerosol wind tunnel.

Figure 9-6 shows a schematic of a closed-circuit wind tunnel which has a continuous path for the air. The HEPA filter removes aerosols after the test section, and the air is recirculated. Consistent flow profiles are easier to maintain in the recirculating tunnel, and less energy consumption is required for a given test section size and velocity as compared to an open tunnel. It is also less noisy than the open tunnel. However, it occupies at least twice the space and has a higher initial installation cost. To dissipate the heat produced, the closed tunnel may need to be cooled for continuous operation during the summer months.

Aerosol wind tunnels are similar to those used in aerodynamic tests, but they require provisions to introduce aerosols in the test section and remove test aerosols after the test section. In addition to a uniform velocity profile in the section, a uniform aerosol concentration profile is required. For example, EPA testing requirements specify that air velocity and aerosol concentration through the test section must be within ± 10% of the mean values in the test section.(9)

Test Aerosol Generation

Test aerosols contain either monodisperse or polydisperse, spherical or nonspherical, solid, or liquid particles.(3, 4, 6, 40, 41) The characteristics of an ideal generator are a constant and reproducible production of monodisperse and stable aerosol particles whose size and concentration can be easily controlled. For general instrument calibration, the test aerosol often contains monodisperse, spherical particles. To calibrate an instrument for a specific environment, the test aerosol should have similar physical and chemical properties to those of the aerosol of interest. For example, for sampling atmospheric particles, which can be either solid or liquid, impactor calibration should include tests with both solid and liquid particles. Solid particles are needed to assess the extent of particle bounce. Liquid particles are needed to assess wall losses. Another example is optical counters, which are most commonly calibrated with polystyrene latex particles. However, for measuring atmospheric aerosol size distributions, where the average refractive index is much lower, there can be significant errors in the particle size measurement. Calibration with atmospheric aerosols has been done.(42, 43)

In addition, the environment in which the instrument is to be operated must be considered when selecting the test aerosol. For example, if the instrument is to be operated in a high-temperature environment, the desired test aerosol could be a refractory metal oxide, such as cerium oxide, because of its thermal stability and chemical inertness. Generally, as long as the desired aerosol is determined, the appropriate method of generation can be identified. Table 9-2 lists the test aerosols frequently used for instrument calibration. Monodisperse aerosols containing spherical particles are the most widely used. Particles with nonspherical shapes are sometimes used in calibration to study the possible effect of shape on the instrument response. Polydisperse dust particles have also been used in calibrating dust monitors. This is important because most real aerosols contain nonspherical particles of different sizes and densities.

Size distribution and concentration of a test aerosol depend on the characteristics of the generator and the feed material. The information given in this section is intended to assist in the selection of appropriate generation techniques. The actual size distribution in each application should always be measured directly with the appropriate instruments.

Polydisperse Aerosols

Polydisperse aerosols are seldom used as test aerosols to calibrate sizing instruments; however, some polydisperse aerosols, such as coal dust and Arizona road dust, are frequently used in calibrating dust monitors, which provide information on the mass concentration of total and/or respirable dust. There are two ways to generate polydisperse aerosols: wet droplet dispersion and dry powder dispersion.

Wet Dispersion

The simplest way to disperse a droplet aerosol is by wet nebulization. Two types of nebulizers are often used

TABLE 9-2. List of Test Aerosols and Generation Methods Used for Instrument Calibration

Test Aerosol	Particle Morphology	Size Range[A,B] VMD (μm)	Size Range[A,B] σ_g	Density (g/cm³)	Refractive Index[C]	Generation Method
PSL (PVT)	Spherical, solid	0.01–220	≤ 1.02	1.05 (1.027)	1.58	Nebulization
Glass	Spherical, solid	1.1–150	1.07-1.3	2.46	1.51	Dry powder dispersion
Fluorescent uranine	Irregular, solid	< 8	1.4-3	1.53	—	Nebulization
Dioctyl phthalate	Spherical, liquid	0.5–40	≤ 1. 1	0.99	1.49	Vibrating atomization
Oleic acid	Spherical, liquid	0.5–40	≤ 1. 1	0.89	1.46	Vibrating atomization
Ammonium fluorescein	Spherical, solid	0.5–50	≤ 1.1	1.35	—	Vibrating atomization
Fused ferric oxide	Spherical, solid	0.2–10	≤ 1.1	2.3	—	Spinning disc (top) atomization
Fused alummosilicate	Spherical, solid	0.2–10	≤ 1. 1	3.5	—	Spinning disc (top) atomization
Fused cerium oxide	Spherical, solid	0.2–10	≤ 1. 1	4.33	—	Spinning disc (top) atomization
Sodium chloride	Irregular, solid	0.002–0.3	≤ 1.2	2.17	1.54	Evaporation/ condensation
Silver	Irregular, solid	0.002–0.3	≤ 1.2	10.5	0.54	Evaporation/ condensation
Coal dust	Irregular, solid	~3.3	~3.2	1.45	1.54-0.5i	Dry powder dispersion
Arizona road dust	Irregular, solid	~3.8	~3.0	2.61	—	Dry powder dispersion

[A] Aerosol treatment of drying, charge neutralization, and size classification is generally used.

[B] VMD = volume median diameter; σ_g = geometric standard deviation

[C] — indicates refractive index (RI) unknown; i indicates imaginary RI for absorption coefficient

to produce droplet aerosols. Air-blast nebulizers(44) use compressed air (15–50 psig, 1 psig = 6.87 × 10^4 dyne/cm²) to draw bulk liquid from a reservoir as a result of the Bernoulli effect. The high-velocity air breaks up the liquid into droplets, then suspends the droplets as part of the aerosol. Droplets produced from this method have a VMD of 1–10 μm and σ_g of 1.4–2.5 (Table 9-3). The aerosol size distribution can be modified by varying the pressure in the compressed air or the dilution ratio in the solution. One problem arises when the bulk liquid contains a volatile solvent that evaporates rapidly after formation of a droplet. The continuous loss of solvent increases the solute concentration in the reservoir and causes the particle size to increase gradually with time. This problem can be circumvented by circulating the solution through a large reservoir,(45) by delivering the solution at constant rate(46) (Figure 9-7), and by cooling the nebulizer. Figure 9-7 shows a modified reservoir for the Retec nebulizers that maintain stable aerosol generation.

In the ultrasonic nebulizer, the mechanical energy necessary to atomize a liquid comes from a piezoelectric crystal vibrating under the influence of an alternating electric field produced by an electronic high-frequency oscillator. The vibrations are transmitted through a coupling fluid to a nebulizer cup containing the solution to be aerosolized. At a certain frequency (1.3–1.7 MHz), a heavy mist appears above the liquid surface of the cup. The diameter of the

TABLE 9-3. Operating Parameters of Air-Blast and Ultrasonic Nebulizers

	Operating Conditions					Droplet Size Distribution		
Nebulizer	**Orifice Diameter (mm)**	**Air Pressure (psig)**	**Frequency (mHz)**	**Flow[A] Rate (L/min)**	**Aerosol Output (µL/L)**	**VMD (µm)**	**σ_g**	**Commercial Source**
Airblast type								
Collison	0.35	15		2.0	8.8	2.5–3	—	BGI
		25		2.7	7.7	1.9–2	—	
DeVilbiss[B] D–40	0.84	15		12.4	15.5	4.2	1.8	DEV
		30		20.9	12.1	2.8	1.9	
DeVilbiss D–45	0.76	15		9.4	23.2	4.0	—	DEV
		30		14.5	22.9	3.4	—	
Lovelace	0.26	20		1.5	40	5.8	1.8	INT
		50		2.3	27	2.6	2.3	
Retec X-70/N	0.46	20		5.0	46	5.7	1.8	INT
		50		9.7	47	3.2	2.2	
Ultrasonic Type								
DeVilbiss 880 (2)[C]			1.35	41.0	54	5.7	1.5	DEV
(4)[C]			1.35	41.0	150	6.9	1.6	
Sono-Tek			0.025–0.12	10^{-6}–0.44	—	18–80	—	SON

[A] Output per orifice.
[B] Vent closed.
[C] Power settings.

droplets making up the mist is related to the wavelength of the capillary waves, which decreases with increasing frequency of the ultrasonic vibrations. Normally the VMD is 5–10 µm, with a σ_g of 1.4–2.0 (Table 9-2).

Aerosol particles with chemical properties different from those of the liquid feed material can be produced through wet dispersion by using suitable gas phase reactions, such as polymerization or oxidation. Production of spherical particles of insoluble oxides and aluminosilicate particles with entrapped radionuclides has been described by Kanapilly *et al.*(20) and Newton *et al.*(47)

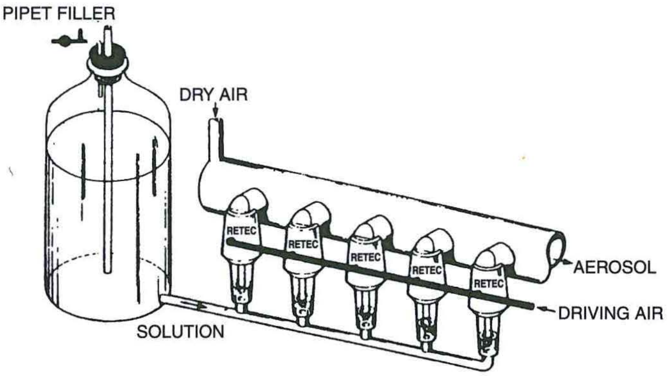

FIGURE 9-7. Schematic of a modified Retec Nebulizer for constant output.

Dry Dispersion

Aerosolization of dry powders and fibers requires different techniques than aerosolization of droplets or suspensions. The dry powder generation methods usually include a two-step operation: 1) feeding or delivery of dry powder at a constant rate to the disperser; and 2) dispersing the powder pneumatically in the aerosol form. The ease of dispersing a powder depends on the powder material, particle size, particle shape, electrostatic charge, and moisture content. Dry powder usually forms clumps or aggregates. A sticky powder is difficult to generate because the powder usually clogs the feeding system and requires high energy from the disperser to break apart the agglomerates into individual aerosol particles. Aerosols generated from dry powder dispersers are also highly charged and, therefore, require discharging to reduce the electrostatic charge.

Powder delivery systems(6, 48) can use hoppers, screw feeders, rotating disks, conveyor belts composed of chains, tubing, brushes, troughs, or compressed cylindrical packs, in which powder is delivered by scraping off the top layer (Figure 9-8). Gravity delivery systems are composed of simple hoppers designed to drop their contents into grooves cut in a plate or directly into a fluidized bed. The TSI fluidized-bed system uses a chain to deliver a

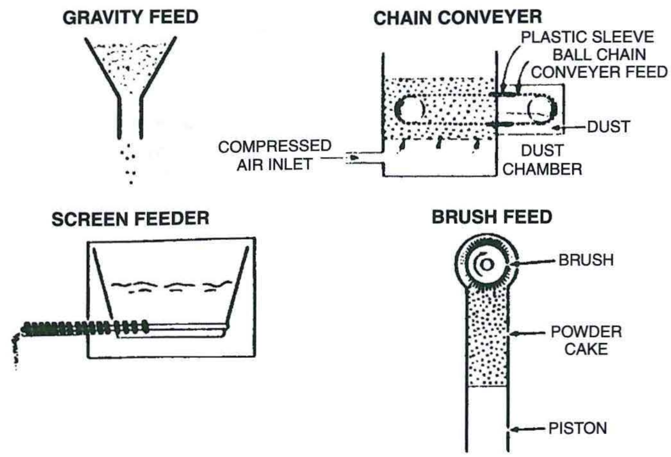

FIGURE 9-8. Schematic of dry powder delivery mechanisms.

powder from a hopper to the multi-component bed. Commercial screw feeders(49) and flexible-walled, brush-bristle augers(50, 51) have been used to successfully deliver powders to an airstream. In the instrument calibration and aerosol generation for inhalation exposures, the dry powder aerosol generator most often used is the Wright dust feed mechanism. Figure 9-9 is a view of the Wright Dust Feed scraper mechanism that consists of a cup in which the material is packed and a scraper blade moves through the packed material. As it moves, the blade removes material from the pack to the central tube. This generator is very dependable if the material can be uniformly compressed into the cup. If the cup is not well packed, air blowing across the blade of the scraper may cause large amounts of material to slough off and become airborne.

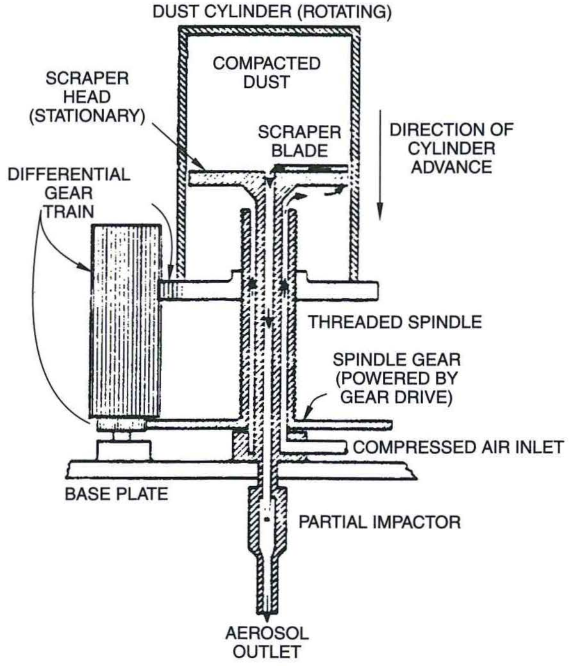

FIGURE 9-9. Schematic of the Wright Dust Feed.

The TSI small-scale powder generator uses a rotational table in which the powder is loaded. Aerosol concentration from any powder generator is to a large extent controlled and adjusted by the feeding rate. A device for dispersing fibers, similar to the Wright Dust Feed, has been designed by Timbrell(52) and is commercially available. The fibers are compressed into a cylindrical plug which is advanced by a threaded piston into a path of rotating blades. The motion of the blades disperses the fibers throughout the small chamber; air flowing through the chamber carries the dispersed fibers out of the device.

The powder is delivered to the disperser for aerosolization, dilution, and deagglomeration. Airstreams, Venturi tubes, air jet mills, and fluidized beds have been used to disperse powders as fine aerosols (Figure 9-10). The most common methods are to feed the dust into a high-velocity airstream or to blow air over the powder. The shear forces in the turbulent airstream disperse powder and break up agglomerates. In the Venturi design, a high-velocity air jet blows across a nozzle or restriction in the pipe to produce suction, which draws clumps of powders into the shear flow of air (Figure 9-11). Both the Venturi dry powder generator(53) and TSI small-scale powder generator use this principle. Fluid energy mills used for dispersion include the Trost jet mill,(51) Jet-O-Mizer,(49) and the microjet mill.(54) The fluid energy is delivered in high-velocity streams, which circulate around a grinding and classifying chamber, where turbulence and centrifugal forces deagglomerate particles. Fine particles carried by the fluid exit at the center of the chamber; coarse particles are recirculated for further size reduction. The Trost jet mill uses two opposing jets to grind and reduce particles with higher efficiency. In a one- or two-component fluidized bed, the minimal air flow required to cause the bed to fluidize is used so that only the smallest particles are released.

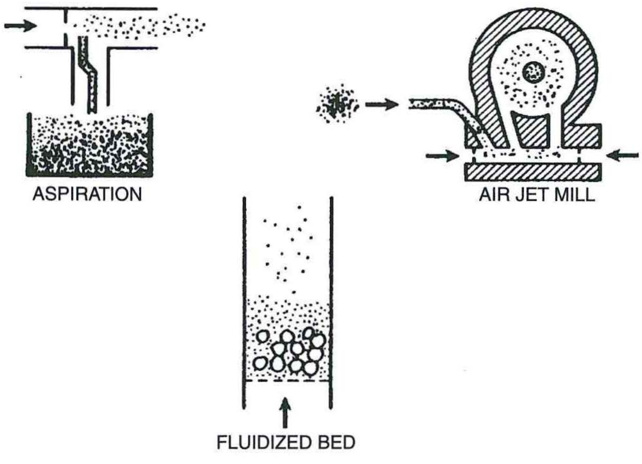

FIGURE 9-10. Schematic of dry powder dispersing mechanisms.

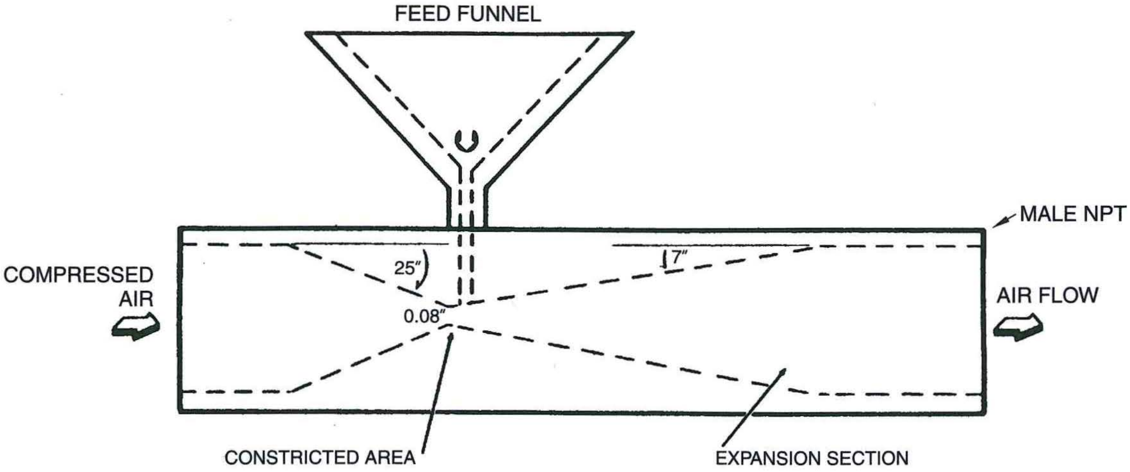

FIGURE 9-11. A Venturi powder disperser.

Commercial and laboratory powder generators listed in Table 9-4 consist of a combination of these delivery and dispersion mechanisms. Figure 9-12, on page 190, shows an example of a fluidized bed generator with gravity feed system. Other delivery systems, including a screw feed(55) and a chain belt,(56) have been used with the fluidized bed generator. The Jet-O-Mizer and Venturi with the screw feed are used to disperse sticky organic powders.(49, 53) Most dry powder generators are material-specific because delivery strongly depends on the bulk powder properties, including size, shape, compactness, and stickiness. The bulk material must be compact for delivery by a Wright Dust Feed, where the powder is packed under pressure to form a solid cylinder. Loose or uneven packing causes the packed powder to break up during the generating process.

Most dry dust generators work best for nonsticky, dry powders. When sticky materials are dispersed as powder, they tend to form clumps that require more energy for dispersion, or they cannot be broken up, thus clogging the generator. The kinetic energy of an air dispersion system is proportional to the square of the air velocity. The fluidized bed has the least kinetic energy and therefore cannot be used to generate sticky powders. The Venturi and fluid energy mills have the highest velocity; therefore, they are more suitable for sticky powders.

One advantage of this dry dispersion method is that the aerosol generated has a similar size distribution as single powder particles when they are suspended in the air. In addition, because no solvent or heat treatment is required, the physical and chemical forms of the material are preserved. A problem common to dry-dispersion aerosols is the buildup of charge on particles as they touch and separate from the surface in the generator. This process reduces the output concentration due to

TABLE 9-4. Operating Parameters of Commercial Dry Powder Dispersers

	Wright Dust Feed	Fluidized Bed	Small Scale Powder Disperser	Jet-O Mizer Model 00
Type of Operation	Scraping the packed plug and dispersing it with air	Feeding the powder to the bed on a conveyor and air fluidizing it	Using rotating plate to deliver the powder and dispersing it with venturi suction	Using venturi suction to feed the powder into a fluid energy mill in which centrifugal force and air velocity are used to break up the agglomerate and to disperse the powder
Air flow rate, L/min	8.5–40	5–20	12–21	14–113
Feed flow rate, mm³/min	0.24–210	1.2–36	0.9–2.5	2000–30,000
Output mass concentration, g/m³ (ρ=1 g/cm³)	0.012–11.5	0.13–4.0	0.0003–0.04	10–1500
Source	BGI	TSI	TSI	FLU

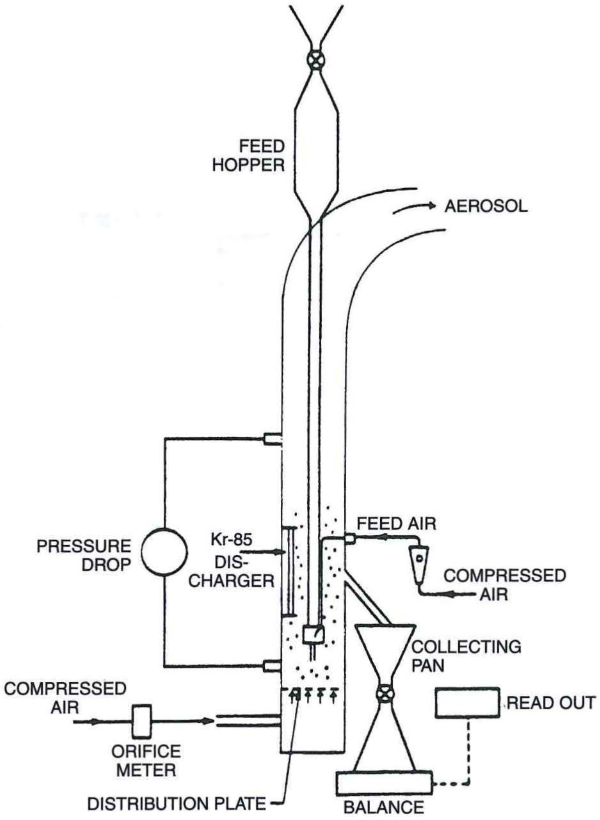

FIGURE 9-12. A fluidized bed powder generator with a gravity feed.

particle losses to the wall of the system. The problem can be solved by passing the aerosol through a chamber containing a bipolar ion source as previously described.

Monodisperse Aerosols with Spherical Particles

The methods of producing monodisperse aerosols with spherical particles have been reviewed by Fuchs and Sutugin,(57) Mercer,(40) and Raabe.(41) These methods include the atomization of a suspension of monodisperse particles, the formation of uniform droplets by dispersion of liquid jets with periodic vibration, electrical fields, or a spinning disc (top), and the growth of uniform particles or droplets by controlled condensation.

Atomization of Suspensions of Monodisperse Particles

The simplest way of producing monodisperse aerosols is by air-blast nebulizing a dilute liquid suspension containing monodisperse PSL or polyvinyltoluene (PVT) latex spheres. These spheres are commercially available in a size range from 0.01 to 30 μm (Duke Scientific, Palo Alto, CA; Dyno Industrier A. S., Lillestrom, Norway; Japan Synthetic Rubber, Tokyo, Japan; Polysciences, Warrington, PA; Seragen Diagnostics, Indianapolis, IN; 3M, Minneapolis, MN). PSL particles of different sizes can also be concurrently produced in an aerosol to obtain more than one data point per experimental run. Monodisperse latex particles containing fluorescent dye or radiolabeled isotopes are also used in calibrations when quantitative measurements by fluorometric or radiometric techniques are needed.(47, 58)

Three problems arise in the generation of these latex particles: 1) measurement of particle size; 2) formation of aggregates; and 3) existence of a secondary aerosol from the non-latex components in the spray droplets. The diameters of latex particles reported by the manufacturer can be different from those measured by an electron microscope because the particles tend to evaporate and shrink due to electron irradiation, or to increase in size due to absorption of a contaminant under irradiation. Porstendörfer and Heyder(59) and Yamada *et al.*(60) recommend measurement of these particles by an electron microscope, but the particle size should be determined without particle shadowing and with minimal electron beam intensity and short exposure time.

The second problem in generating latex particles is the formation of aggregate latex particles in the aerosol. The percentage of aggregates can be reduced by diluting the suspension. Assuming that the probability of the number of particles in an atomized droplet can be described by Poisson statistics, and that the droplet-size distribution can be approximated by a log normal distribution, Raabe(61) derived the following equation to calculate the latex dilution factor, *Y*, necessary to give a desired singlet ratio, *R*, which is the number of droplets containing single particles relative to the total number of droplets containing particles:

$$Y = \frac{F(VMD)^3\ exp(4.5ln^2\sigma_g)\left[1\ -\ 0.5\ exp(ln^2\sigma_g)\right]}{(1\ -\ R)d_p^3} \qquad \textbf{(2)}$$

where F is the volumetric fraction of individual particles of diameter d_p in the original latex suspension, and VMD and σ_g are the volume median diameter and the geometric standard deviation of the droplet size distribution, respectively. The values of VMD and σ_g of commonly used air-blast atomizers are listed in Table 9-3. This equation is limited to values of $\sigma_g < 2.1$ and $R > 0.9$.

The third problem arises when nonlatex residual particles are present in the aerosol as a result of the impurities and surfactant in the liquid suspension. The nonlatex particles could either distort the size of an individual particle, if they attach to it, or become individual particles with a size different from that of the latex particles in the aerosol. To reduce the impurity in the diluting water, a system that provides deionized and double distilled water is normally used. To remove the surfactant from the suspension, a procedure of diluting, centrifuging, and discarding the supernate is generally followed.

Vibrating-Orifice and Spinning Disc (Top) Atomizers

The generators that produce monodisperse droplets can also produce monodisperse aerosols. Two methods are available. The VOMAG is an atomizer that creates a jet of liquid through an orifice and breaks the jet into droplets of a uniform diameter by applying certain disturbing frequencies(17, 62, 63) as shown in Figure 9-13. The stability, monodispersity, and diameter of the droplets depend on the diameter of the orifice, the density and surface tension of the liquid, the velocity of the jet, and the disturbing frequency. The advantage of this method is that the diameter of the droplet, d_d (µm), and the aerosol particle, d_p (µm), can be determined by the following equations:(17)

$$d_d = 10^4\left(\frac{Q_l}{10\pi f}\right) \qquad \textbf{(3)}$$

$$d_p = C^{1/3} d_d \qquad \textbf{(4)}$$

where Q_l is the liquid feed rate in cm³/min, f is the vibrating frequency in Hz, and C is the volumetric concentration of the solute in the solution. In the frequency range for stable generation of droplets, the droplet size is about twice the orifice diameter. Therefore, with orifices of 5 to 30 µm, the droplet sizes range from 10 to 60 µm. The minimum aerosol particle size after evaporation of the solvent is about 1.5 µm, because the lowest concentration of aerosol solution, C, is limited by impurity in the solution, which is on the order of 0.001 volume fraction. The output aerosol concentrations are low, about 30 to 400 cm^{-3}, depending on the primary droplet size. In addition, liquid suspensions can clog the orifice and should not be used to feed the orifice. In the case of producing monodisperse solid aerosols (e.g., ammonium fluorescein), the drying process for suspended droplets is crucial to the surface smoothness of the final particles. Inclusion of small amounts of oil can improve sphericity by preventing crystallization, but density may be affected if the particle dries too fast. The aerosol generated has a narrow size distribution; however, the instrument is difficult to operate.

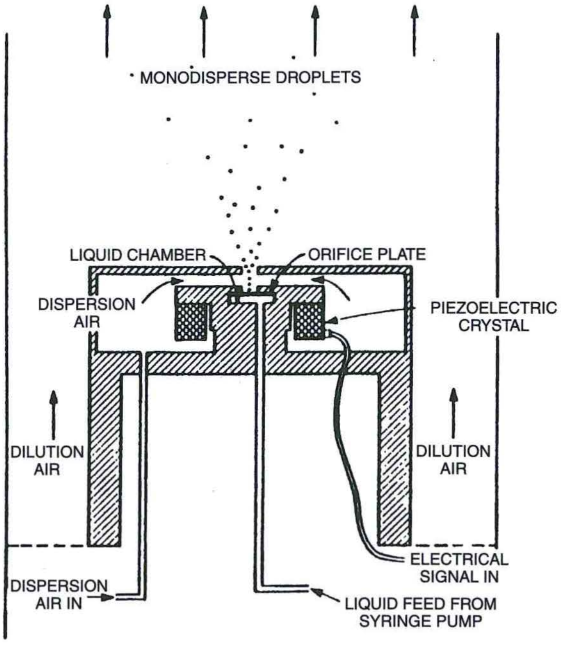

FIGURE 9-13. Schematic of a vibrating orifice aerosol generator.

The second method of producing monodisperse droplets is by the spinning-disc (top) atomizer, in which a liquid jet is fed at a constant rate onto the center of a rotating disc (top). The liquid spreads over the disc (top) surface in a thin film, accumulating at the rim until the centrifugal force discharges it, and a droplet is thrown off. Droplet size d_d depends on disc (top) diameter, d_s (in µm), and rotating speed, ω_s (in rpm) as follows:

$$d_p = (W\gamma / \rho_l \varpi_s^2 d_s)^{1/2} \qquad \textbf{(5)}$$

where γ is the surface tension, ρ_l is the density of the liquid, and W is a constant. The application of this process has been investigated by Walton and Prewett(64) and May(65) using an air-driven, spinning top, and by Whitby *et al.*(66) and Lippmann and Albert(67) using a motor-driven, spinning disc. Unlike a vibrating-orifice atomizer, aqueous suspensions, as well as solutions, can be used. A disadvantage of this method is that undesired satellite droplets are frequently formed and must be removed from the useful aerosol produced by the primary droplets. In addition, the constant W (Equation 5) varies with the instrument and the feed material used, and the droplet size and the final particle size cannot be as easily calculated as with the vibrating-orifice atomizer.

Controlled Condensation Techniques

Condensation is also a method that produces monodisperse aerosols for calibration purposes. In this method, the heated vapor of a substance that is normally liquid or solid at room temperature is mixed with the nuclei on which it condenses when it passes in laminar flow through a cooling zone. If the condensation process is diffusion controlled, the surface area of the growing droplet will increase at a constant rate, producing a particle having a diameter, d_t, at time t related to the initial diameter, d_o, of the nucleus, by

$$d_t^2 = d_o^2 + bt \qquad \textbf{(6)}$$

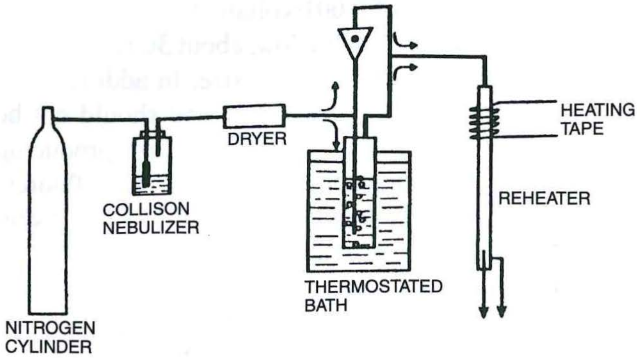

FIGURE 9-14. Schematic of a MAGE generator.

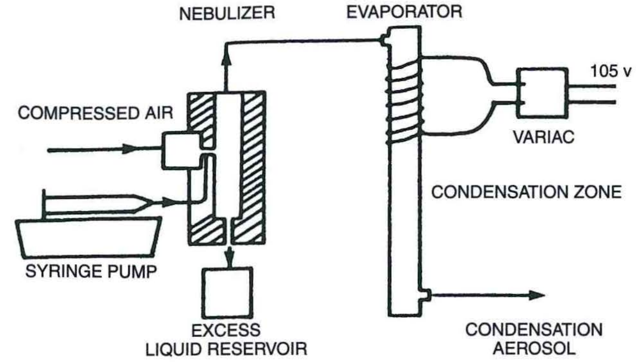

FIGURE 9-15. Schematic of a constant output controlled condensation aerosol generator.

where b is a constant, related to the concentration and diffusivity of the vapor and to the temperature. If bt is the same for all particles and much larger than d_o, the diameter of the nucleus has little effect on the final diameter of the particle, so that an aerosol containing monodisperse particles is produced. In practice, uniform temperature profile, sufficient vapor concentration, and sufficient residence time in the condensation region are the essential controls, and a constant nuclei concentration provides a stable aerosol concentration. Condensation generators are usually for high-boiling-point, low-vapor-pressure liquid droplets, e.g., DOP, triphenylphosphate, and di-octyl sebecate. Solid test aerosols, such as a carnauba paraffin aerosol, can also be generated. Aerosol concentrations are generally in the range of 10^5 to 10^7 particles/cm^3 with narrow size distribution (geometric standard deviation < 1.2).

Sinclair and LaMer(68) described the first condensation liquid aerosol generator based on this principle. Simplifications and modifications of the Sinclair and LaMer generator have been used in the laboratory(69, 70) Figure 9-14 shows a photograph of a modified Sinclair-LaMer generator (MAGE) based on Prodi's design.(70) The size range of particles generated is 0.2 to 8 μm for liquid droplets, and 0.2 to 2 μm for solid particles (waxes and paraffins). A somewhat different version of the condensation generator as described by Liu and Lee(46) is illustrated in Figure 9-15.

A Collison-type nebulizer is used to atomize liquid into polydisperse droplets. An aerosol solution is delivered to the nebulizer by a syringe pump to control the feed rate and to prevent the evaporation of solvent and subsequently the increased concentration of the aerosol solution. The droplets are heated to vaporize in the heat section, and residue nuclei consisting of impurities in the solution are formed. The nuclei number concentration and the vapor pressure are constant, providing a stable environment for condensation in the down flow section of the glass tube. Both the Liu-Lee generator and MAGE are commercially available (Table 9-5).

Condensation generators for ultrafine solid test particles have been designed and used in instrument calibration(71–74) Figure 9-16 illustrates a basic solid aerosol generator using a tube furnace.(71, 74) The solid material is placed in a quartz boat inside the furnace, a stream of inert gas (e.g., nitrogen) carries the vapor out of the boat, and the vapor condenses to form particles in the condensation chamber. Test aerosols whose particle is carnauba wax, sodium chloride, or silver are commonly used. The aerosol has a narrow size distribution with a geometric

TABLE 9-5. Operating Parameters of Monodisperse Aerosol Generators

Generator	Principles	Aerosol Flow Rate (L/min)	Particle Size Range (μm)	Source
Electrostatic classifier (Model 3071)	Electrical mobility	2–4	0.005–1	TSI
Constant output generator (Model 3076)	Controlled condensation	2–4	0.04–1.3	TSI
CMAGE	Controlled condensation	1–3	0.9–2.5	TSI
Spinning-top aerosol generator	Atomization	120	1–100	BGI
Vibrating orifice aerosol generator (Model 3050)	Atomization	100	1–40	TSI

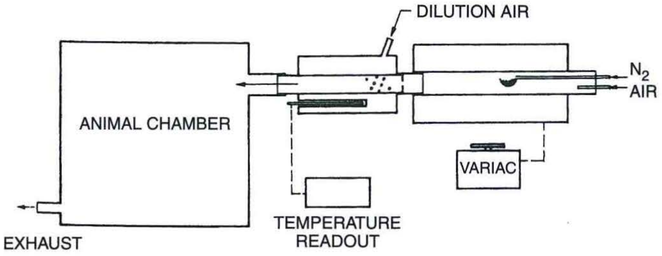

FIGURE 9-16. Schematic of a condensation aerosol generator for solid particles.

standard deviation between 1.1 to 1.3. More narrow size distributions (with geometric standard deviations smaller than 1.1) can be obtained by classifying the aerosol using an EC as described later in this section. The temperature control in the furnace is essential for the stable generation of aerosols. For generation of metal aerosols of lead, zinc cadmium, and antimony, a high-frequency induction furnace has been used.(75)

Electrospray Techniques

A method for producing monodisperse droplets using electrostatic spray of a semi-conductive fluid has recently been described.(76–79) Electrosprays refer to generation of liquid droplets through a capillary tube by feeding liquid with a finite electric conductivity and applying an electrical field. When the potential is sufficiently high, the liquid meniscus at the capillary outlet takes the shape of a cone from whose tip a very thin liquid jet emerges in the cone-jet mode.(80, 81) The microjet breaks by varicose wave instabilities into a stream of charged droplets, having diameters roughly twice as large as the jet diameter.(82, 83) The droplets have diameters an order of magnitude smaller than the capillary diameter. A stable spray can be generated if the applied electrical field exceeds a critical value. The droplet size is smaller than those produced in a vibrating orifice. The droplet size is a function of nozzle diameter, liquid feed rate, and surface tension, conductivity, and viscosity of the liquid.(84) Thus it can generate very small droplets without clogging problems in the liquid line such as are frequently encountered in the vibrating orifice aerosol generator. The mean droplet size is usually in the range of 0.3 to 50 μm. Tang and Gomez(83) have demonstrated a generation system producing monodisperse droplets in the size range from 2 to 12 μm with σ_g of 1.15 for small droplets and 1.05 for large droplets. Monodisperse droplets of 0.3 to 4 μm have been produced with σ_g of 1.1(82) More recently, monodisperse droplet sizes of 40 nm to 1.8 μm have been generated (σ_g of 1.1) by varying the liquid feed rate and electrical conductivity.(85) With evaporation of droplets, this technique can generate monodisperse particle of nanometer size (4 nm).

Monodisperse Aerosols with Nonspherical Particles

The effects of particle shape on instrument response are important, especially for the sizing instruments in which the measured properties are generally dependent on particle shape. Information concerning the effects of shape on instrument response can be obtained by using monodisperse aerosols of nonspherical particles during calibration. One way of generating these aerosols is to nebulize the liquid suspension containing the monodisperse, nonspherical particles. Various techniques have been used to produce monodisperse particles of highly uniform particle size and shape. Matijevic(86) produced inorganic and polymer colloid particles of cubic, spindle, and rhombohedral shapes by chemical reactions. Fiber-like particles of a narrow size range were also produced using different methods.(87–92) The vibrating-orifice and spinning-disc (top) aerosol generators described above can also be used to generate irregularly shaped particles, such as crystalline sodium chloride particles. Although the generators produce spherical droplets, the crystal form of the solid particles becomes the shape of the final aerosol after drying the liquid vapor.

In addition, naturally occurring materials, such as fungal spores, pollens, and bacteria or the fortuitous occurrence of multiplets of spheres, are also frequently used as test aerosols of nonspherical particles.(93, 94) The aerosols of fungal spores and pollens are commonly generated by using the dry powder dispersion technique described in the previous section.

Size Classification of Polydisperse Aerosols

Although polydisperse aerosols may be used for instrument calibration or to simulate the actual use of equipment under controlled laboratory conditions, they can also be classified according to size in order to provide an aerosol with narrow size range for instrument calibration. For particles smaller than 0.2 μm, Liu and Pui(24) developed a differential electrical mobility analyzer to classify aerosol particles of the same electrical mobility. Figure 9-17 shows that the schematic of the EC consists of a bipolar discharger to maintain the aerosol charge distribution and a concentric mobility analyzer. The polydisperse aerosol is charged and separated in the mobility analyzer, and only particles having the same mobility can exit at the same time. Because most classified particles of submicrometer size are singly charged,

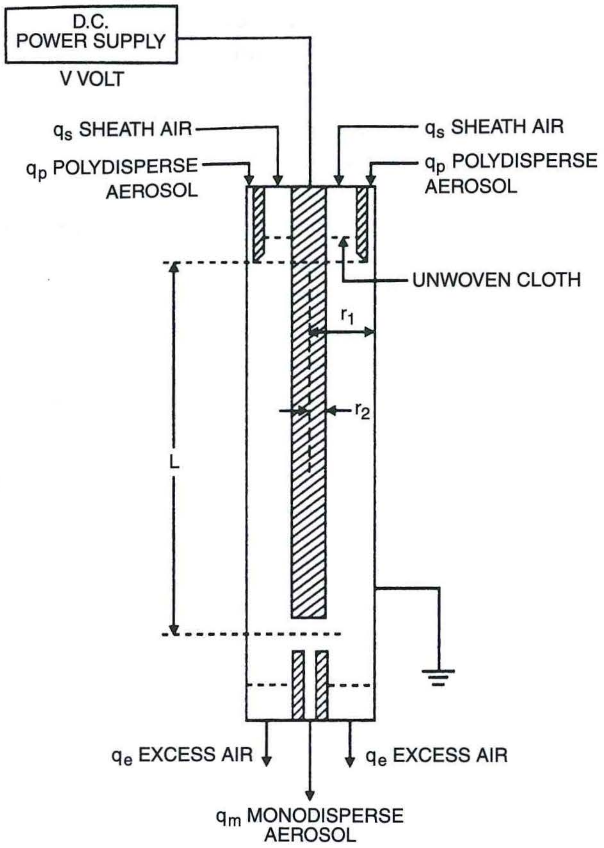

FIGURE 9-17. Schematic of an electrostatic classifier.

the aerosol produced is monodisperse. The particle size of the classified aerosol has a triangular size distribution with the median diameter d calculated from the following equation:

$$d = \frac{4ne\Lambda V\, C(d) 1 \ x\ 10^7}{3\mu(q_s + q_e)} \tag{7}$$

where n is the number of charges, e is the elementary charge (1.6 x 10^{-19} Coulomb), C is the Cunningham slip correction, μ is the gas viscosity (g/cm^{-1} sec^{-1}), q_s is the sheath flow rate (cm^3/sec^{-1}), q_e is the excess flow rate (cm^3/sec^{-1}), V is the applied voltage, $\Lambda = L/\ln(r_2/r_1)$, r_2 and r_1 are the outer and inner radii of the mobility analyzer, and L is the length of the analyzer. The geometric standard deviation of the classified aerosol is related to the flow rates:(18)

$$\sigma_g = exp\left[\left(\frac{1}{6}\right)^{1/2} \frac{(q_p + q_m)}{(q_p + q_m + q_s + q_e)}\right] \tag{8}$$

where q_m and q_p are the monodisperse and polydisperse flow rates, respectively. This classification technique has been used to produce a submicrometer aerosol standard in calibrating CNCs and diffusion batteries, and in determining particle deposition in human nasal, oral and tracheobronchial casts.(46, 95–97)

For particles greater than 1 μm, the size classifying technique based on aerodynamic properties is generally used. Two virtual impactors can be placed in a series to segregate the desired fraction of an input aerosol for use in instrument calibration.(25, 98) To classify aerosols in the 0.1–1.0 μm range, a technique that involves both the mobility analyzer and a single-stage micro-orifice impactor has been used.(26) One can also nebulize latex test spheres and use a mobility classifier to remove aggregates and other impurity. In fact, this method is recommended to generate PSL test particles in the size range from 0.03 to 0.1 μm. The above techniques are also used for removing undesired particles, such as PSL aggregates from an air nebulizer or satellite particles by the spinning-disc generator.

All devices and techniques described above classify aerosol particles when they are still airborne. Other instruments, such as elutriators, spectrometers, cascade impactors, and cascade cyclones, classify particles by means of collecting size-classified particles on a substrate, then resuspending the size-classified particles. For example, a spiral centrifuge collects aerodynamically classified particles on aluminum foil; resuspension of the particles caught on a narrow segment of the foil can be used to produce monodisperse aerosols.(99) The disadvantage of all size-classifying techniques is that only a small quantity of particles is produced.

Test Aerosols with Tagging Materials

For some applications, particle detection is often facilitated by incorporating dyes or radioisotope tags in the particles during their production. For example, test aerosols composed of fluorescent dyes can be analyzed in solutions containing as little as 10^{-10} g/m^{-3}. The radioactivity of the aerosols can be determined by counting the samples with much lower detection limits than can be obtained with electrobalances. Assuming tagged materials are evenly distributed in the aerosol material and therefore are proportional to the mass, the tagged material can be measured by colorimetric, fluorometric, or radioactive counting techniques to determine the particle mass with low detection limits. These techniques are especially useful for calibration of instruments having low flow rates smaller than 1 L min^{-1} such as Mercer impactors or low pressure impactors. Tagged particles deposited in the inlet section, transport lines, and internal wall of the detector can be washed and measured.(2, 100)

Commonly used dye materials include eosin,(2) methylene blue,(1) and ammonium fluorescein. Minute amounts of these chemicals can be dissolved in aqueous or alcohol solutions with oleic acid and DOP. Monodisperse-tagged liquid droplets can be generated by techniques described in the previous sections, including a VOMAG, a spinning-top generator, and condensation techniques. Commercial PSL particles that incorporate fluorescent dyes are also available as listed in Table 9-6. Radiolabeling techniques have been used in many forms and can usually be detected at extremely low concentrations.(47, 101) Radiolabels including ^{99}Tc, ^{51}Cr, and ^{137}Cs have been incorporated into PSL to produce monodisperse test particles. Similarly, several radiolabels have been incorporated into aluminosilicate clay by ion exchange; the clay solution is then nebulized and heat treated at 1150°C to form insoluble spherical particles.(47, 102) Ultrafine particles (<0.2 μm) of metal oxides (^{67}Ga$_2$O$_3$, ^{144}CeO$_2$, and ^{57}Co$_3$O$_4$) have been produced by evaporation, subsequent thermal degradation, and condensation of organic ketone compounds.(71)

Calibration Procedures

This section describes the steps used in a typical calibration practice. It is always useful to describe detailed procedures in a test plan or protocol, and then follow through during the test.

Set up Test Facility

The criteria for selecting a proper test facility for a calibration program have been described in detail in previous sections. The selected test facility may already exist in the laboratory. In this case, one only needs to ensure the performance system for the specified procedure. If a new test facility or experimental apparatus is needed, then the system should be designed accordingly, and individual components of the test facility purchased and installed.

Select Test Aerosols

Proper selection of test aerosols is essential to instrument calibration. A variety of test aerosols and their generation methods have been described. For general purposes, standard test aerosols as listed in Table 9-2 provide sufficient choices. The selection is further narrowed by considering the particle size, monodisperse versus polydisperse aerosol, and liquid versus solid particles. Sometimes the choice is dictated by the standards specified in a regulation. In other cases, special test aerosols are required for certain classes of instruments. For example, fiber test aerosols should be used to calibrate the response of real-time fiber monitors. Once the test aerosol is selected, an appropriate aerosol generator and accessory parts are assembled and become part of the test system.

Test Calibration Systems

After the test system is assembled, the system should be checked for leaks. All meters used to regulate flows in the generator, dilution air, chamber supply, exhaust, and sampler should be calibrated and documented. The system should be tested without aerosol generation to determine the stability of the flow, the uniformity of flow in the test chamber, and the turbulence intensity in case a wind tunnel is used. Test runs with aerosols should be made to determine the stability of aerosol concentration at target levels, the particle size distribution, and distribution of aerosol concentration in the test section. The operating conditions can be adjusted and the test system modified during the test run to obtain the desired test conditions.

On-line detection of aerosol size and/or concentration in the calibration system is also recommended to assure the stable generation of aerosol. For example, an optical counter or an aerodynamic particle sizer can be used to monitor the satellite's generation of a VOMAG or spinning-disc generator.

TABLE 9-6. Commercial Sources of Monodisperse Latex Test Particles with Fluorescent or Radioactive Tags

Materials	Tags	Size Range (μm)	Source
Polystryene latex	Blue, green, red, yellow	0.44–7	BAN
Polystryene latex	Green, red, blue	0.025–3	DUK
Polystyrene latex with surface modification	Blue, orange, yellow-green, red, dark red	0.02–4	IDC
Polystyrene latex	Yellow-green	0.05–10	POL

Data Collection, Analysis, and Documentation

A calibration curve that contains the relationship between the instrument's responses and the values of a certain aerosol property is established after calibration. In the case of an instrument that directly indicates a value of the measured parameter, calibration provides an adjustment (or a correction factor) to the indicated value. In addition, resolution and sensitivity of the instrument should be examined and analyzed. The rule of thumb is to conduct the calibration based on all important parameters, to assemble all the data, and express those data in a generalized mathematical equation, relating the instrument response to a single parameter.

For data analysis, instrument manufacturers sometimes provide a built-in algorithm whose properties, accuracy, and limitations are often unknown to the user. Unless a user understands the algorithm, the analysis should be developed only based on the raw calibration data without any manipulation by the built-in algorithm.

All pertinent data including experimental conditions, aerosol data, and analysis should be documented carefully. This is especially important if the study is intended for regulatory or compliancy purposes. It is likely the information will be examined and scrutinized carefully. Examination of the documents is needed to remove any mistakes before submitting to the agency for approval.

Quality Assurance

To obtain high quality test data and to minimize experimental errors, steps should be taken to ensure that the test facility, instruments, and aerosol generator are functioning properly. Integrity of the test system and the instrument itself should be maintained by avoiding possible leakage or blockage of flow. Techniques and criteria of isokinetic sampling and sampling from still air should be followed, especially for aerosol particles greater than 5 μm in aerodynamic diameter. In order to generate reliable data, pre-calibration tests should be carried out to ensure that a stable aerosol can be maintained for a long period of time, that the aerosol concentration and size are constant, and that the aerosol is uniformly distributed in the test chamber. For each data point, at least three calibrations are required to provide statistically valid information. Also, it is a good practice to calibrate the test standard before the test is undertaken. For example, as noted above, PSL test particles may need to be examined in a microscope to assure the accuracy of particle size and the absence of impurities.

TABLE 9-7. Commercial Sources

Symbol	Source	Symbol	Source
BAN	Bangs Laboratories Inc. 979 Keystone Way Carmel, IN 46032 (317)844-7176 FAX (317)575-8801	IDC	Interfacial Dynamics Corp. 17300 SW Upper Boones Ferry Rd. Suite 120 Portland, OR 97224 (800)323-4810
BGI	BGI Inc. 58 Guinan St. Waltham, MA 02154 (781)891-9380 FAX (781)891-8151	INT	In-Tox Products 115 Quincy, NE Albuquerque, NM 87108 (505)286-2233
DEV	The DeVilbiss Co. P.O. Box 635 Somerset, PA 15501 (814)443-4881	POL	Polyscience Inc. 400 Valley Rd. Warrington, PA 18976 (800)523-2575
DUK	Duke Scientific Co. 2463 Faber Pl. Palo Alto, CA 94303 (800)334-3883	SON	Sono-Tek 313-A Main Mall Poughkeepsie, NY 12601 (914)471-6090
FLU	Fluid Energy Aljet 5136 Applebutter Rd. Plumsteadville, PA 18949 (215)766-0300.	TSI	TSI Inc. 500 Cardigan Rd. St Paul, MN 55164 (800)677-2708 FAX (651)490-3860

A quality assurance program should also include written documentation of the protocol, standard operating procedures for each procedure used in the protocol, and careful documentation of the results. After the procedures are finalized, the operator should adhere to the established procedures. If problems occur during the test, the cause should be identified, corrected, and documented before the test is continued. A quality assurance program is mandatory, if a study is to be conducted under the Good Laboratory Practices Guidelines or other compliance regulation.

Conclusions

Because the accuracy of measuring aerosols depends on the precision of the aerosol instrument, the instrument must be calibrated very carefully. The following comments summarize this chapter and the philosophy of aerosol instrument calibration:

1. The developer or manufacturer of an instrument has the responsibility for providing instrument performance data that include full-scale calibration data covering the whole applicable range (i.e., the particle size for sizing the instrument and the concentration for mass or number concentration monitors), the test aerosol used, and the conditions under which the instrument is tested (flow rates, ambient temperature, and pressure). This calibration should be performed for new instruments and after extensive repair.
2. The user should understand the principles of the instrument operation and be familiar with the calibration data. The decision on when to recalibrate the instrument is based on scientific and regulatory requirements. Frequent checks of the instrument and its flow meter are minimum requirements, and a single point check is recommended for routine use of any sampling instrument. Full-scale calibration requires substantial effort, appropriate equipment, and facilities that may not be available for every user.
3. Quality assurance is an important aspect of a successful test program. The calibration procedure should be carefully planned and documented. Test runs should be made to ensure that the system is functioning, that the correct procedures are used, and that the aerosol and flow stability are achieved and maintained. Standard operating procedures should be followed during the calibration, and any problems encountered should be resolved and documented. All records including test runs, data, problems with appropriate comments, instrument identification, barometric pressure, temperature, flow rate, properties of the test aerosol, and the name of the operator should be documented.
4. Proper test facilities should be used for an aerosol sampler intended for different environments. Instrument chambers with low air flow should be used for calm air sampling conditions, whereas aerosol wind tunnels should be used to simulate sampling in the flow streams or ambient environments.
5. Proper test aerosols should be used. Standard spherical test aerosols such as PSL and others provide data to compare the instrument response with other instruments using the same kind of aerosol. Whenever possible, test aerosols with physicochemical properties similar to the measured aerosol should be used.

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