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CALIBRATION OF AEROSOL INSTRUMENTS

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21.1 INTRODUCTION

A vast amount of knowledge concerning aerosol properties has been obtained using instruments. These instruments can be categorized as: (1) collection devices such as cascade impactors, virtual impactors, Aitken-type condensation nuclei counters, or filter samplers, which are designed to remove

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[1] The findings and conclusions in this report are those of the authors and do not necessarily represent the views of the National Institute for Occupational Safety and Health (NIOSH), the National Institute of Standards and Technology (NIST), and the Lovelace Respiratory Research Institute (LRRI). Commercial equipment, instruments, and materials, or software are identified in this report to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement of these items by the NIOSH, the NIST, and the LRRI, nor does it imply that they are the best available for the purpose.

Aerosol Measurement: Principles, Techniques, and Applications, Third Edition. Edited by P. Kulkarni, P. A. Baron, and K. Willeke

particles from gas streams to obtain samples for analysis; and (2) real-time, direct-reading instruments such as optical particle counters, condensation particle (or nucleus) counters, aerosol electrometers, or photometers. Ideally, instrument response can be theoretically computed based on equations and procedures described in the previous chapters. Theoretical prediction of instrument response based on ideal conditions may not always be realized. For example, although 50% effective cutoff diameters and collection efficiencies for an impactor stage can be computed, the phenomena of particle bounce, re-entrainment, electrostatic charge effects, and wall losses can modify its performance (Rao and Whitby 1978; Cheng and Yeh 1979). Therefore, experimental calibration is essential.

Instruments are usually calibrated and evaluated by the manufacturer or the inventor before being used by others. For an instrument intended to collect an aerosol for analysis, collection efficiency and wall loss are generally determined in the calibration. For a real-time, direct-reading instrument, calibration establishes the relationship between an instrument's 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 calibrate the instrument to operate it with confidence. In general, a reliable and accurate calibration requires (1) sufficient knowledge of capabilities and limitations of the instrument, (2) adequate information on the environment where the instrument will be used, (3) appropriate test facilities, (4) proper selection of a desired test aerosol, (5) a thorough investigation of relevant parameters, and (6) a quality assurance program that is followed throughout the test.

In the last three decades, developments in aerosol generation and classification, progress in electron microscopy and imaging analysis, and improvement of test facilities have made instrument calibration easier and the results more reproducible. This chapter reviews calibration techniques relevant to aerosol measurement devices, such as sizing instruments, condensation particle counters, and mass monitors. The generation methods for test aerosols and important parameters in instrument calibration are emphasized. Also reviewed are the calibration and use of flow-monitoring devices that play an integral role in aerosol sampling and instrument calibration.

21.2 MEASUREMENT METHODS AND CALIBRATION STANDARDS

Aerosol instruments can be categorized according to the particle properties characterized (inertial, gravitational, optical, diffusional, thermal, or electrical) or the measuring techniques (real time or sample collection, personal or area, passive or active). The measured parameters are usually particle size, number concentration, or mass concentration, and distributions of these parameters. The calibration of an aerosol instrument implies that the instrument response will be related to a particle standard; for example, for particle sizing, a suitable standard may be latex particles that have been independently sized by methods traceable to the usual laboratory standards. For concentration, the sampled volume is needed, necessitating calibration of the instrument's flow rate. This can be accomplished by the use of various flow meters that have been calibrated by traceable standards.

Some aerosol parameters can be measured absolutely in the laboratory, that is, by determining the parameter through combined measurements of length, mass, and time (LMT). For example, the aerodynamic diameter of large particles can be determined by measuring the time required for a particle to fall a given distance (Wall et al. 1985). The particle diameter calculated from the operating parameters of the vibrating-orifice generator can be considered absolute standard since all of the involved quantities can be reduced to LMT (see discussion of the vibrating-orifice generator in Section 21.5.1.2). Geometric particle diameters can be measured in an electron microscope. However, attention must be paid to possible effects of electron beam heating and exposure to vacuum. Liquid particles are sometimes sized by deposition on a plate and optical microscopy. This requires a correction for droplet distortion (Liu et al. 1982b; John and Wall 1983; Cheng et al. 1986). Manual measurements by microscopy have limited accuracy because of the small sample size. This can be overcome by using image analysis under computer control. Monodisperse, submicrometer particles of known size can be obtained by using an electrostatic classifier where the particles are selected by their electrical mobility (Liu and Pui 1974; Mulholland et al. 1999).

For the calibration of filter samplers, the collected aerosol mass concentration can be obtained directly by weighing the filters on a microbalance. Other mass samplers can be calibrated by comparison to a filter sampler. The air volume is calculated from the flow rate and the sampling duration. Flow rates can be calibrated with a variety of flow meters. In the case of a bubble meter, the volume swept out by the bubble is measured for a given time.

Because instrument calibration is time consuming, there is a trend toward using well-calibrated, real-time laboratory instruments with direct readouts to characterize the test aerosol and to measure the aerosol penetrating the instrument under test. It is important that the laboratory instrument be operated according to the manufacturer's specifications and that laboratory personnel performs checks on its functioning.

21.3 GENERAL CONSIDERATIONS

Instrument calibration is essential to a successful measurement of aerosol properties. Certain considerations are relevant before engaging in a rigorous process of instrument calibration. One should decide the reason for the calibration, where the calibrated instrument is to be used, what type of aerosol and associated parameters are to be measured, the extent or level of calibration necessary (related to motivation for calibration), how the calibration is to be performed, and how the data is to be processed. Associated with data analysis and experimental design is the determination of measurement uncertainty. Scientific knowledge and technical experience play important roles in making the right decisions, facilitating the calibration process, and resulting in defendable outcomes.

21.3.1 Rationale for Instrument Calibration

To assure high-quality, reliable data, the sampling instrument must be calibrated. For some applications, the instrument must perform according to performance criteria recommended by different organizations, such as the International Organization for Standardization (ISO), National Institute for Occupational Safety and Health (NIOSH), American Industrial Hygiene Association (AIHA), American Conference of Governmental Industrial Hygienists (ACGIH), American National Standards Institute (ANSI), and the European Committee for Standardization (CEN), or regulatory standards established by government agencies, such as Occupational Safety and Health Administration (OSHA), Mine Safety and Health Administration (MSHA), and the U.S. Environmental Protection Agency (USEPA). In the last few years, calibrations traceable to recommended methods have become important for legal compliance and other reasons. Case in point is the voluntary ISO standard now in draft form, ISO/WD 27891:2008 “Calibration of aerosol particle number concentration measuring instruments—Calibration of condensation particle counters with Faraday cup aerosol electrometers” (2008) that provides a method for calibration of a condensation particle (nuclei) counter (CPC or CNC) with an aerosol electrometer (AE). ISO 27891 is motivated by the need for traceable concentration calibration methods for (1) the European diesel exhaust type certification requirement, (2) semiconductor cleanroom monitoring using CPCs, (3) possible outdoor environmental monitoring, (4) filter efficiency measurements, and (5) for fit testing of respirators. The other ISO document relevant to the aerosol community is ISO 15,900:2009 “Determination of particle size distribution—Differential electrical mobility analysis for aerosol particles” (2009).

Table 21-1 summarizes some existing performance criteria and regulatory standards for aerosol sampling instruments. As an example, according to 40 CFR Part 53-Ambient Air Monitoring Reference and Equivalent Methods (Federal Register, 1987), a PM-10 sampler is to be calibrated in a wind tunnel using 10 different sizes of solid or liquid particles ranging from 3 to 25 μm at wind speeds of 2, 8, and 24 km/h (0.56, 2.2, and 6.7 m/s, respectively).

TABLE 21-1 List of Existing Performance Criteria for Aerosol Sampling Instrumentation

Type of Instrumentation	Performance Criteria/ Guidelines	Regulatory Agency or Organization
PM-10 inlet sampler	Sampling effectiveness, 50% cutpoint, precision, flow-rate stability	EPA
Workplace sampling instrument	Inhalable, thoracic, and respirable fractions; sampling efficiency, 50% cutpoint, sampling precision	ACGIH, ISO, CEN
Portable field instrument	Portability, reliability, calibration, interference, etc.	NIOSH
Respirable dust sampler	Entry, penetration, and sampling efficiencies	ISO, CEN
Personal sampling pump	Interferences	OSHA, ANSI

Kenoyer and Leong (1995).

21.3.2 Environment to be Surveyed

The expected ambient conditions under which aerosol instrumentation is employed may determine the conditions in which the calibration is conducted. For example, aerosol sampling instruments may be expected to perform under various wind conditions and thus may need to be calibrated in a wind tunnel. Depending on the wind speed in the environment, the sampling can be classified as still (or calm) air sampling or sampling in a moving airstream (Vincent 1995, 2007; Hinds 1999). Still air sampling generally refers to a wind speed less than 0.5 m/s and applies to indoor environments, including residential homes, offices, schools, and factories. Settling chambers with uniform, low flow rates are suitable for testing instruments under still air conditions (Kenny et al. 1999); while wind tunnels are more appropriate for testing instruments under moving airstream conditions. Other environmental parameters, such as temperature, pressure, and relative humidity, should be considered prior to calibration, as these conditions may affect instrument performance.

21.3.3 Parameters to be Investigated

The calibration parameters are determined by the measurement or sampling process for which the calibration is performed. These parameters are also dependent on the type of instrument as well as the aerosol properties of interest in the sampling environment. For example, volumetric flow rate, pressure drop, and light source intensity are operating parameters; while particle size and composition and the nature of the suspending gas medium are aerosol parameters. To calibrate an aerodynamic sizing device, the effects of particle density, velocity, and ambient pressure on the instrument response are important, while in an optical particle counter (OPC), the particle refractive index, wavelength of light source, and collection angles of scattered light are the important parameters.

21.3.4 Design of the Calibration Program

The level or extent of the calibration may be dependent upon the importance of the final measurement results. A full-scale calibration examining the instrument response over its full operational range requires elaborate test facilities and extensive effort in terms of time and labor. However, when a calibration curve with the full-range response is available, it may be sufficient to perform a single- or two-point calibration. It is not unusual for different components of the same instrument to be calibrated separately. For example, when one is interested in the performance of the sampling inlet (inlet efficiency), transport line (transport loss), and detection (counting efficiency) or collection section (collection efficiency) of a high volume aerosol sampling system, a series of calibrations can be made, one for each individual component (Chen et al. 1999). Different parameters might be selected for investigation in the different components and, consequently, different test facilities with different test aerosols are often employed during the component calibrations. By contrast, several instruments are sometimes arranged serially and calibrated as an integrated unit. For example, a two-stage virtual impactor and an electrical classifier were combined in series to investigate their integrated performance in fiber classification (Chen et al. 1996).

21.3.5 Selection of Test Aerosols

Proper selection of test aerosols is essential to instrument calibration. Because most instruments have responses strongly dependent on the physical and chemical properties of the aerosol particles, the calibration curve of an instrument is strictly valid only for the test aerosol. For an aerosol whose physical and chemical properties are significantly different from those of the test aerosol, data interpretation based on the calibration could be misleading (Willeke and Baron 1990). For example, one would underestimate the size distribution of a carbon black aerosol by using the calibration curve of an OPC obtained from polystyrene latex (PSL) spheres, because the carbon particles cannot scatter as much light as the (PSL) spheres. Ideally, an aerosol that has similar physical and chemical properties (e.g., size, shape, density, refractive index, dielectric constant, and thermal conductivity) to the aerosol to be measured should be selected as the test aerosol during calibration. For example, Marple and Rubow (1976) used an impactor stage to give an aerodynamic particle size calibration of an OPC and Hering and McMurry (1991) calibrated an OPC using monodisperse fractions of atmospheric aerosols obtained from a differential mobility analyzer (DMA).

Sampler performance can be investigated by use of selected test aerosols. Liquid particles can be used to simulate sticky particles, which suffer wall losses. Solid, bouncy particles can be used to test for particle bounce and/or reentrainment.

21.3.6 Data Analysis

A calibration curve contains the relationship between the instrument response and the values of a certain aerosol property to be measured. As mentioned above, it often represents the properties of the calibration aerosol. In the case of a direct-reading instrument, the calibration provides an adjustment (or a correction factor) to the indicated value. In addition, the resolution (smallest difference between displayed indications that can be meaningfully distinguished) and sensitivity (quotient of the change in an indication of a measuring system and the corresponding change in a value of a quantity being measured) of the instrument should be examined and analyzed. After collecting calibration data, it is desirable to express that data in a generalized mathematical equation, relating the instrument response to a single parameter (Chen et al. 1985; Zhang and Liu 1990). The uncertainties associated with the calibration measurements should be identified.

For data analysis, instrument manufacturers sometimes provide a built-in algorithm whose properties, accuracy, and limitations are often unknown to the user. It may be desirable to base the analysis only on the raw calibration data without manipulation by the built-in algorithm.

21.3.7 Traceability

Sometimes a calibration needs to be made traceable to primary standards or to the International System of Units (SI), the metric system agreed upon throughout the world. The motivation can stem from commercial or internal markets and legal reasons. A widely accepted definition (Ehrlich and Rasberry 1998) for traceability found in the *International Vocabulary of Basic and General Terms in Metrology* (ISO 1993) is “property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties.” A more recent definition is found in the *International Vocabulary of Metrology—Basic and General Concepts and Associated Terms* (ISO 2007) “property of a measurement result whereby the result can be related to a reference through an unbroken chain of calibrations, each contributing to the measurement uncertainty.” Traceability does not guarantee the measurement results are “true” or accurate, but rather there is a higher standard to which the measurement is referenced (Larrabee and Postek 1993). Sometimes this standard may be a consensus standard where the value is accepted or agreed upon by a large number of users in a technical community (and may not necessarily be accurate). Accuracy, a highly desirable measurement goal, is achievable through calibration to an accurate reference standard (defined as the true value) with the understanding that the uncertainties in the standard, in the calibration, and in the measurement process can be determined. Traceability to a widely accepted reference standard permits comparisons of measurements among peer users. It also provides a benchmark for reproducing experiments (or products) and serves to satisfy audits or legal issues or provide a measure of goodness to a product (Ehrlich and Rasberry 1998).

21.3.8 Safety Precautions

When generating aerosols, it should always be borne in mind that a respiratory health hazard may be created. A primary consideration is containment of the aerosol and minimizing exposure. A ventilated chemical fume hood is a good location for an aerosol generator. Even if the exhaust from the generator is vented, there are usually times when the apparatus is open or there may be leaks. A walk-in hood is especially convenient to accommodate an auxiliary apparatus. If a hood cannot be used, the exhaust should be vented or filtered. Hazardous substances require more stringent containment measures.

Care should be exercised in the choice of aerosol materials. For example, in the past dioctyl phthalate (DOP) was commonly used as a test aerosol, since it has nearly unit density and is an oil with low volatility. However, animal tests have implicated DOP as a possible carcinogen. A good substitute is oleic acid, also a nonvolatile oil, which is available in food grade. An additional benefit is that uranine, which is frequently added as a fluorescent tracer, is soluble in oleic acid whereas it is insoluble in DOP. This means that the uranine is uniformly dispersed in the oleic acid droplets. Uranine is commonly used to trace waterways and is presumably harmless. Of course, even when the aerosol material is believed to be safe, it is prudent to avoid exposure.

Another hazard is associated with the use of radioactive sources to "neutralize" the electrical charges on aerosols resulting from the generation process. ^{85}Kr, a beta (high-energy electron) emitter, is commonly used in source strengths up to 3.7×10^8 Bq [10 mCi]. Unfortunately, ^{85}Kr also emits gamma rays. Whereas the beta rays are absorbed by the walls of the container, the gamma rays penetrate. It is recommended that a qualified health physicist check the radiation level to evaluate the adequacy of the shielding. Alpha particle sources, such as ^{210}Po, represent a hazard when ingested and must be handled with care. For protection, ^{210}Po is sealed between a base of silver and a layer of gold, and is plated in gold by the manufacturer. The user should avoid removing the protective grid or touching the metal foil under the grid.

21.3.9 Uncertainties in the Calibration

Calibration is a form of measurement science, a science that assigns numerical values to properties or objects or events (Eisenhart 1969). Measurements from a calibrated instrument are only as good as the calibration. That is, the data derived from the instrument response should contain the uncertainty associated with the calibration in addition to the uncertainty contributed by the measurement itself. So the uncertainty associated with the calibration can be quite relevant to the overall uncertainty in the result reported from the experiment. Calibration can be accomplished by comparison with another instrument or measurement tool or by measurement of a standard material for which the properties are known to some high level of certainty. Measurements have an associated uncertainty due to a multitude of reasons, such as the standard that one is comparing against, the ambient conditions, natural variability (randomness) associated with making the measurements, and many other factors.

Measurement uncertainty can be deconstructed and defined in many ways. Two concepts often confused are accuracy and precision. Eisenhart (1969), who gives both a philosophical and practical description of uncertainty in measurement, provides a one-sentence definition of accuracy and precision. "In brief, 'accuracy' has to do with closeness to the truth; precision only with closeness together." The difficult issue related to accuracy is obtaining or establishing what is the true value. One definition relies on the true value being the accepted value by experts or national/international certifying agencies. Precision on the other hand provides a description of the imprecision of the measurement values one with another and is often represented in the form of standard deviation.

Taylor and Kuyatt (1994), based on policies of the International Committee for Weights and Measures (CIPM), provided recommendations for methods to express uncertainties. Uncertainties can be classified in terms of random "Type A" uncertainties that can be described by statistical methods (standard deviation or variance). These are related to repeated measurements and are often random in nature. The other class of uncertainties is derived by other than statistical means and is termed "Type B" uncertainties. An example is the stated uncertainty in a property of a standard material used to perform a calibration such as PSL spheres (e.g., the spheres are specified at $5.06\ \mu\text{m} \pm 0.10\ \mu\text{m}$). Sometimes there is an uncertainty based on the manufacturer's specification (e.g., the pressure transducer is specified to be within $\pm 5\%$ of the measurement value). There can also be specified uncertainties in reference data values. Sometimes the Type B uncertainties may have a residual systematic component.

However, as discussed by Taylor and Kuyatt (1994, Sect. 5.2), it is assumed that a correction factor is included to compensate for each recognized systematic effect. Even after making the correction there is still an uncertainty in the correction itself. This uncertainty could be computed either as Type A or Type B.

The direction given by Taylor and Kuyatt (1994) requires the analyst to combine the uncertainties by what is often called the law of propagation of uncertainty (or the square root of the sum-of-the-squares). The expression for the combined uncertainty, u_c, for a measurand, y, determined by a series of quantities $x_1, x_2, \ldots, x_n$ expressed as $y = f(x_1, x_2, \ldots, x_n)$ is

$$u_c(y) = \left[\sum_{i=1}^{n} \left(\frac{\partial f}{\partial x_i} \right)^2 u^2(x_i) + 2 \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} \left(\frac{\partial f}{\partial x_i} \right) \left(\frac{\partial f}{\partial x_j} \right) u(x_i, x_j) \right]^{1/2} \quad \text{(Eq. 21-1)}$$

where $u(x_i)$ is the uncertainty associated with quantity x_i and $u(x_i, x_j)$ is the estimated covariance associated with x_i and x_j. If the quantities are independent, then there is no covariance term (Ku 1966).

Measurements are often made over a range of values and a calibration curve is constructed. Natrella (1963) gives a method to determine the confidence interval around the calibration curve and to obtain an uncertainty associated with predicting a value from the curve. The method is too detailed to describe here, but is useful for deriving uncertainties from calibration curves.

21.4 CALIBRATION APPARATUS AND PROCEDURES

Figure 21-1a is a schematic diagram of a typical calibration apparatus for aerosol instruments. It includes an aerosol generator, aerosol treatment devices (e.g., diffusion dryer, charge neutralizer, aerosol classifier, aerosol concentrator, and dilution air supply), a mixing chamber, pressure and airflow monitoring equipment, the instrument to be calibrated, and a calibration standard. The aerosol produced from the generator can be monodisperse or polydisperse, solid or liquid, wet or dry, charged or uncharged, or spherical or nonspherical (described in 21.5). Generally, this aerosol requires several steps of conditioning before use. For an aerosol containing volatile vapors or water droplets, a diffusion dryer with desiccant (such as drierite) and/or charcoal is commonly used to produce a dry aerosol. In some cases, a heat treatment using a high-temperature furnace is required for the production of test aerosol (Kanapilly et al. 1970; Chen et al. 1990). 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., ^{63}Ni, ^{85}Kr, and ^{241}Am) is often used in the aerosol treatment. This reduces the number of charges on particles and results in an aerosol with charge equilibrium (John 1980). In addition, a size-classifying device is often used in the aerosol treatment to segregate particles of similar size or of desired size fraction (Liu and Pui 1974; Chen et al. 1988; Romay-Novas and Pui 1988). In addition, a concentrator or a dilutor is typically used to adjust the aerosol concentration (Barr et al. 1983; Yeh and Carpenter 1983).

The desired test aerosol can be used to calibrate instruments in several ways. The simplest way, as shown in Figure 21-1a, is to introduce the test aerosol into a small mixing chamber, where the same aerosol (size and concentration) can be delivered to both the instrument to be calibrated and the calibration standard. Pressure in the chamber and flow rate through the instrument are monitored. A sampling device, such as a filter sampler or an electrostatic precipitator is often used to collect reference samples for the calibration standard. To ensure that both the calibration device and the instrument to be calibrated have comparable aerosol samples, an aerosol divider is used as a common sampling port for calibrating a mass monitor (Marple and Rubow 1978). 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 (Fig. 21-1a). This mixing chamber setup is inexpensive, easy to use, and does not require a large working area. It is most widely used for instrument calibrations that require particle sizes less than 5 μm, such as those for obtaining the response curve of an OPC or the collection efficiency of an impactor. For calibrations requiring particles larger than 5 or 10 μm, it is relatively difficult to provide a stable aerosol with sufficiently high concentration. Because the instrument is placed outside the mixing chamber, the setup is not adequate for testing the aspiration efficiency of the instrument inlet.

Another way of calibrating an instrument is to introduce the aerosol particles into an aerosol test chamber (Fig. 21-1b) that houses the subject instrument and the test standard (Marple and Rubow 1983; Chen et al. 1999). This chamber usually has a large test section to provide a quiescent atmosphere in which the entire instrument can be exposed to the aerosol as in the real sampling environment. The test aerosol is introduced at the top of the chamber and uniformly distributed in the section where the instrument is set on a rotating table. Rotation provides a means to reduce any effects due to possible temporal and spatial variations in aerosol concentration. This test chamber setup can provide uniform concentrations of aerosol particles as large as 90 μm for instrument calibration (Maynard and Kenny 1995; Aitken et al. 1999; Kenny et al. 1999). Several instruments can be placed inside the chamber for side-by-side comparison,

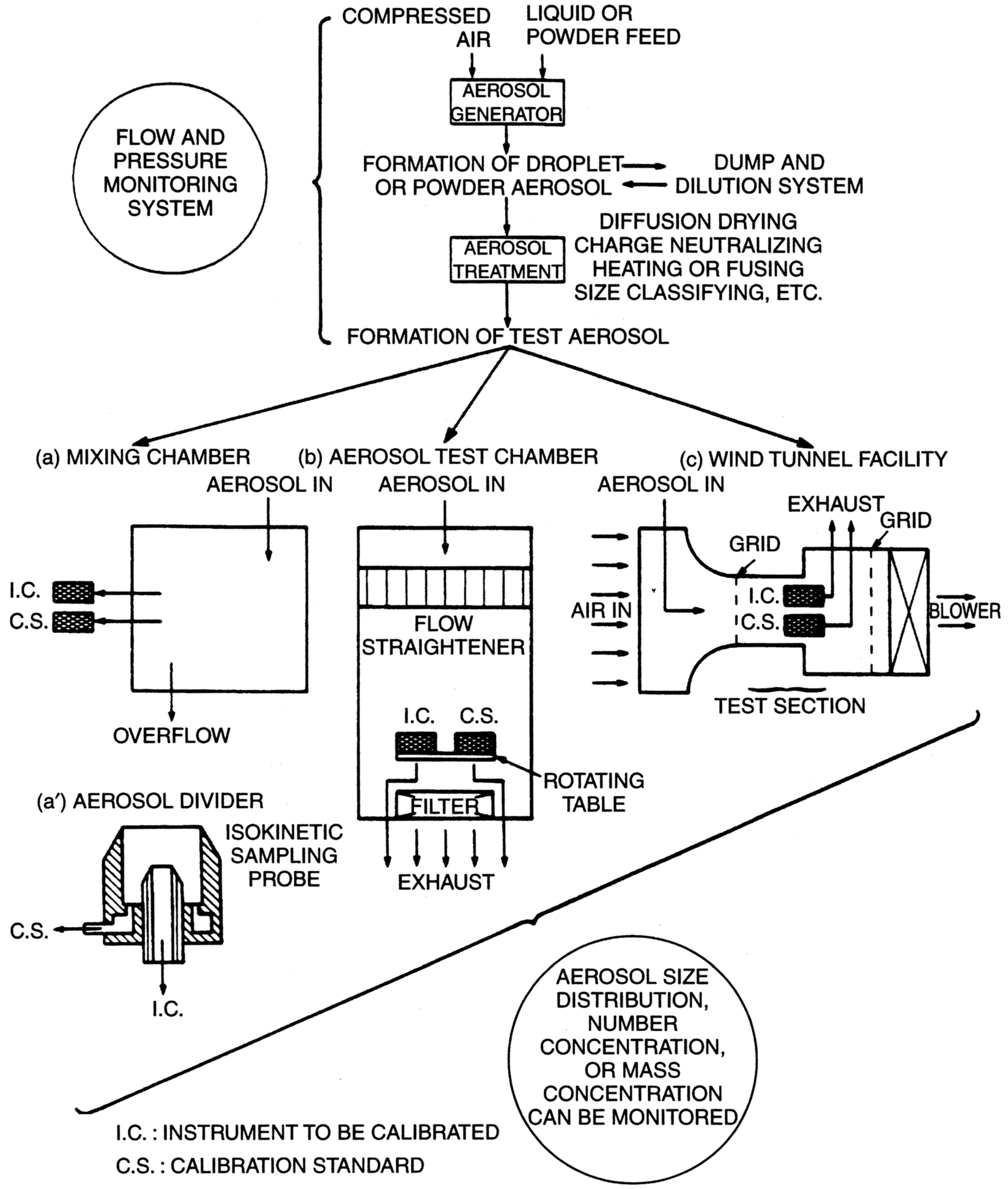

Figure 21-1 Schematic diagrams of a setup for instrument calibration using **(a)** a mixing chamber, **(a′)** an aerosol divider, **(b)** a test aerosol chamber, and **(c)** a wind tunnel facility.

including the sampling inlet. The flow rate and turbulence intensity in the chamber are low, simulating still air sampling conditions. It should be noted that the air is exhausted from the chamber, that is, the air is not completely static. A test chamber with static air is not recommended because it is very difficult to avoid convection currents that can affect the measurements.

Both the mixing chamber and the aerosol test chamber are used when the instrument to be calibrated is operated in a low or zero ambient wind velocity. To evaluate a sampler that will be operated in moving air, a wind tunnel facility (Fig. 21-1c) is needed (Prandtl 1952). The sampler is located inside the tunnel and should not occupy more than 10–15% of the cross-sectional area of the tunnel's test section to avoid blockage effects. Personal samplers are often mounted on the upper torso of a full-size mannequin placed within the test section of the wind tunnel (Vincent and Mark 1982; Kenny et al. 1997). The wind tunnel provides a wide range of wind speeds (0.5–10 m/s) to simulate different atmospheric conditions. The wind velocity, flow uniformity, and turbulence are monitored using flow monitoring devices (described in 21.6). During calibration of the test sampler, an isokinetic sampler is generally used to collect reference samples. Two types of wind tunnels are commonly used: an open-circuit tunnel (Vincent and Mark 1982) and a closed-circuit tunnel (Ranade et al. 1990). The open-circuit tunnel operates by drawing filtered ambient air into the system and exhausting the air into the ambient downstream of the test section; the closed-circuit tunnel circulates the air in a continuous path. Each type of tunnel has advantages and disadvantages. For example, the open tunnel occupies a smaller space with less installation cost, while the closed tunnel is quieter and requires less energy consumption.

Before any calibration, a standard operating protocol should be prepared. First, the manual for the instrument to be calibrated should be read carefully to learn as much as possible about the operating principles of the instrument, the construction, and the recommended operating procedures. However, the manual may not cover all aspects relevant to the application. For example, a laser-operated OPC tends to produce oscillatory responses when the particles are larger than the wavelength of the laser beam; however, the calibration curve provided by the manufacturer seldom shows this phenomenon (Chen et al. 1984).

The condition of the instrument should be checked prior to calibration. The integrity of the flow system can be quantified by a series of pressure measurements on a sealed system that has initially been brought to a pressure slightly above or below ambient pressure (Mokler and White 1983). Any leakage can be discovered by various methods. The simplest one is to pressurize the system slightly and then put soapy water on the surface of the system to detect the leakage. A tracer gas can also be injected into the system to detect the leak location. It may be advisable to check the electronics by observing the signals on an oscilloscope, especially if the instrument outputs a signal to be processed by other instruments.

21.4.1 General Approaches to Instrument Calibration

Calibration methods can be characterized by whether the test aerosol is monodisperse or polydisperse and how the aerosol is measured. Monodisperse aerosol is either produced directly by a generator or classified after generation by an auxiliary apparatus (e.g., atomization followed by electrical mobility classification or two-stage virtual impaction). The use of polydisperse test aerosol implies that particle sizing will be done on the airborne particles or by analysis of collected samples. In one approach to calibration, the particles collected or deposited within the instrument or sampler are analyzed, for example, gravimetrically or by analysis of tracers. An alternative approach is to measure the aerosol entering and leaving the sampler. This may involve testing the sampler without a final filter.

The choice of the calibration method is made in consideration of the type of instrument to be calibrated, the kind of information needed, and available resources. The various methods have distinct advantages and disadvantages. The use of monodisperse particles necessitates many repetitions with different particle sizes. However, it may yield unambiguous information, for example, on whether 20-μm particles penetrate a size selector for PM-10. Similarly, the measurement of deposited particles may involve tedious extractions and quantitations, but may determine where wall losses occur. The use of polydisperse aerosol with a sizing instrument that produces a real-time size distribution makes possible rapid calibration measurements. This can be important if a parameter such as sampler flow rate is to be varied between calibrations, or variations in the sampler's configuration are to be explored (John and Kreisberg 1999).

21.5 TEST AEROSOL GENERATION

Test aerosols contain either monodisperse or polydisperse, spherical or nonspherical, and solid or liquid particles (Mercer 1973; Raabe 1976; Hinds 1999; Cheng and Chen 1995). The characteristics of an ideal generator are a constant and reproducible output of stable aerosol particles whose size and concentration can be easily controlled. For general instrument calibration, the test aerosol often contains monodisperse, spherical particles. Table 21-2 lists the test aerosols frequently used for instrument calibration. Monodisperse aerosols containing spherical particles are frequently 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.

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

21.5.1 Monodisperse Aerosols with Spherical Particles

The methods for producing monodisperse aerosols with spherical particles have been reviewed by Fuchs and Sutugin (1966), Mercer (1973), and Raabe (1976). These methods include the atomization of a suspension of monodisperse particles, the formation of uniform droplets by dispersion of liquid jets with periodic vibration or a spinning disk, and the growth of uniform particles or droplets by controlled condensation.

21.5.1.1 Atomization of Suspensions of Monodisperse Particles A common way of producing monodisperse aerosols is by nebulizing a dilute liquid suspension containing

TABLE 21-2 List of Test Aerosols and Generation Methods Used for Instrument Calibration

Test Aerosol[a]	Particle Morphology	Size Range[b]		Density (kg/m^3)	Refractive Index	Generation Method	Aerosol Output (particles/m^3)
		d_p (μm)	σ_g				
PSL (PVT)	Spherical, solid	0.02–>100	≤1.02[c]	1050 (1027)	1.59	Nebulization	$<10^{10}$
Fluorescent PSL	Spherical, solid	6–>100[d]	1.08–1.17	1050	1.59	Dry powder dispersion	–[e]
Soda lime glass (borosilicate glass)	Spherical, solid	1.1–>100	1.07–1.3	2460 (2500–2550)	1.51 (1.56)	Dry powder dispersion	–[e]
Oleic acid	Spherical, liquid	0.5–40	≤1.1	890	1.46	Vibrating orifice atomization	$<10^{11}$
Ammonium fluorescein	Spherical, solid	0.5–50	≤1.1	1350	–	Vibrating orifice atomization	$<10^{11}$
Fused ferric oxide	Spherical, solid	0.2–10	≤1.1	2300	–	Spinning disk Atomization	$<10^{13}$
Fused aluminosilicate	Spherical, solid	0.2–10	≤1.1	3500	–	Spinning disk Atomization	$<10^{13}$
Fused cerium oxide	Spherical, solid	0.2–10	≤1.1	4330	–	Spinning disk Atomization	$<10^{13}$
Sodium chloride	Irregular, solid	0.002–0.3	≤1.2	2170	1.54	Evaporation/condensation	$<10^{12}$
Silver	Irregular, solid	0.002–0.3	≤1.2	10,500	0.54	Evaporation/condensation	$<10^{12}$
Coal dust	Irregular, solid	~3.3	~3.2	1450	1.54–0.5i	Dry powder dispersion	<30 mg/m^3
Arizona Road Dust	Irregular, solid	~3.8	~3.0	2.61	–	Dry powder dispersion	<30 mg/m^3

[a] Standard particles, such as the PSL, fluorescent PSL, glass spheres, and Arizona Road Dust, are commercially available from companies such as ***BAN***, ***DUK***, ***IDC***, or ***POL***.

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

[c] For d_p less than 0.1 μm, σ_g is between 1.03–1.14.

[d] This size range is for fluorescent particles in dry powder form; particles of submicrometer sizes are available in suspension.

[e] The aerosol output for these dry particles depends on the particle size, bulk concentration, as well as generation parameters. Normally, particles of a bigger size have a smaller concentration.

monodisperse PSL spheres or polyvinyltoluene (PVT) latex spheres. These spheres are commercially available in a size range from 0.02 to over 100 μm (***BAN***, ***DUK***, ***DYN***, ***JSR***, ***MMM***, ***POL***, ***SER***[2]). Polystyrene latex particles of different sizes have also been 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 (Newton et al. 1980; Chen et al. 1999).

Two problems arise in the generation of these latex particles: formation of agglomerates and existence of residual particles. Agglomerates are formed when more than one latex particle is in a nebulized droplet. The percentage of agglomerates 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 lognormal distribution, Raabe (1968) 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:

$$Y = F(d_v)^3 \exp(4.5 \ln^2 \sigma_g) \, [1 - 0.5 \exp(\ln^2 \sigma_g)]/(1 - R)d_p^3 \qquad \text{(Eq. 21-2)}$$

where *F* is the volume fraction of individual particles of diameter d_p in the original latex suspension, and d_v and σ_g are the volume median diameter and the geometric standard deviation of the droplet size distribution, respectively. The values of d_v and σ_g of commonly used air-blast atomizers are listed in Table 21-3. This equation is limited to values of $\sigma_g < 2.1$ and $R > 0.9$.

The second problem arises when non-latex residual particles are present in the aerosol as a result of the surfactant usually present in the liquid suspension to prevent coagulation. Since most of the atomized droplets contain no latex particles, the non-latex particles form a large background of small particles. If this background interferes with the measurements, the surfactant may be removed from the suspension before use by diluting, centrifuging, and discarding the supernate. In recent years, latex suspensions containing no surfactant have become available. These suspensions are stabilized by surface coatings of functional groups (***IDC***).

EXAMPLE 21-1

A bottle of 1-μm PSL suspension containing a 10% solid is being used to produce a test aerosol containing at least 95% singlets. What is the dilution factor required in this suspension if the Retec X-70/N nebulizer is used and operated at 20 psig?

Answer: Using Equation 21-2: $Y = F(d_v)^3 \exp(4.5 \ln^2 \sigma_g)[1 - 0.5 \exp(\ln^2 \sigma_g)]/(1 - R)d_p^3$

$$F = 10\% = 0.1,$$
$$R = 95\% = 0.95, \text{ and}$$
$$d_p = 1 \ \mu\text{m}.$$

Based on size distribution data given in Table 21-3:

$$\begin{aligned} d_v &= 5.7 \ \mu\text{m}, \sigma_g = 1.8, \text{ and} \\ Y &= (0.1)(5.7)^3 \exp[4.5 \ln^2(1.8)] \\ &\quad \{1 - 0.5 \exp[\ln^2(1.8)]\}/(1 - 0.95)(1)^3 \\ &= (0.1)(5.7)^3 \exp(1.555)[1 - 0.5 \exp(0.345)]/(0.05) \\ &= (0.1)(5.7)^3 (4.734)(0.294)/(0.05) \\ &= 514.9 \end{aligned}$$

A dilution factor of at least 515 is needed to produce an aerosol containing 95% of singlet PSL particles.

21.5.1.2 Vibrating-Orifice and Spinning-Disk Aerosol Generators The vibrating-orifice aerosol generator can produce highly monodisperse aerosols in the approximate size range from 0.5 to 50 μm (Fulwyler et al. 1969; Raabe and Newton 1970; Berglund and Liu 1973). The particle diameter can be calculated from the generator's operating conditions so that the aerosol can be considered a primary particle size standard. Also, the aerosol concentration is inherently stable. In the vibrating-orifice generator (Fig. 21-2), a liquid is forced through an orifice. The resulting liquid jet is made to break up into uniform droplets by subjecting the jet to a mechanical disturbance of constant frequency. The droplet diameter, d_d, is then given by

$$d_d = 10^6 (6Q_L/\pi f)^{1/3} \qquad \text{(Eq. 21-3)}$$

where Q_L is the liquid feed rate in m^3/s and *f* is the vibrating (disturbing) frequency in Hz. The droplet diameter is typically some tens of micrometers. In order to generate smaller particles, a nonvolatile solute can be dissolved in a volatile solvent. After evaporation of the solvent, the particle diameter is related to the volumetric concentration of the solute, C_v, by

$$d_p = C_v^{1/3} d_d \qquad \text{(Eq. 21-4)}$$

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[2] Refer to Appendix I for complete addresses of manufacturers indexed to three-letter code.

TABLE 21-3 Operating Parameters of Air-Blast and Ultrasonic Nebulizers

	Operating Conditions					Droplet Size Distribution		
Nebulizer	Orifice Diameter (mm)	Air Pressure (kPa [psig])	Frequency (mHz)	Flow[a] Rate ($\times 10^{-5}$ m^3/s [L/min])	Aerosol Output (μL/L)	d_v (μm)	σ_g	Commercial Source
				Airblast Type				
Collision	0.35	100 [15]		3.3 [2.0]	8.8	2.5–3.0	3.0	***BGI***
		170 [25]		4.5 [2.7]	7.7	1.9–2.0	2.0	
DeVilbiss[b]	0.84	100 [15]		20.7 [12.4]	15.5	4.2	1.8	***DEV***
D-40		200 [30]		34.8 [20.9]	12.1	2.8	1.9	
DeVilbiss	0.76	100 [15]		15.7 [9.4]	23.2	4.0	–	***DEV***
D-45		200 [30]		24.2 [14.5]	22.9	3.4	–	
Lovelace	0.26	140 [20]		2.5 [1.5]	40	5.8	1.8	***INT***
		350 [50]		3.8 [2.3]	27	2.6	2.3	
Retec	0.46	140 [20]		8.3 [5.0]	46	5.7	1.8	***INT***
X-70/N		350 [50]		16.2 [9.7]	47	3.2	2.2	
				Ultrasonic Type				
DeVilbiss (2)[c]			1.35	68.3 [41.0]	54	5.7	1.5	***DEV***
880 (4)[c]			1.35	68.3 [41.0]	150	6.9	1.6	
Sono-Tek			0.025–0.12	10^{-6}–0.73 [10^{-6}–0.44]	–	18–80	–	***SON***

[a] Output per orifice.
[b] Vent closed.
[c] Power settings.

EXAMPLE 21-2

Oleic acid aerosol is produced by a vibrating-orifice atomizer from a solution of oleic acid in isopropyl alcohol, with a volume concentration of 1.48×10^{-2}. The liquid feed rate is 3×10^{-9} m^3/s [0.18 cm^3/min] and the vibrating frequency is 5.5×10^4 Hz. The dilution airflow rate is 3.33×10^{-4} m^3/s [20 L/min]. What are the diameters of the droplets and the oleic acid particles? What is the particle number concentration?

Answer: Using Equations 21-3 and 21-4:

$$d_d = 10^6 (6 Q_L / \pi f)^{1/3},$$
$$d_p = C^{1/3} d_d \text{ and}$$

substituting Q_L, f, and C, we find the droplet diameter $d_d = 10^6 (6(3 \times 10^{-9})/55{,}000\pi)^{1/3} = 47.1$ μm, and the particle diameter $d_p = (1.48 \times 10^{-2})^{1/3}(47.1) = 11.6$ μm.

The rate of particle production is the same as the vibrating frequency, 55,000 s^{-1}. Dividing this by the flow rate of the dilution air, we find the particle number concentration is 1.7×10^8 m^{-3}.

Liquid particles can be produced, for example, from a solution of oleic acid in isopropyl alcohol, or solid particles of sodium chloride can be produced from an aqueous solution. The minimum particle size attainable, in practice, depends on the purity of the solvent. The maximum practicable particle size is not well defined. Particles larger than about 20-μm diameter become more difficult to generate. As the diameter is increased, it also becomes more difficult to avoid particle losses in transport. Therefore, the generation of particles larger than about 50 μm requires special effort.

Referring to Figure 21-2, the solution is forced through the orifice by a syringe pump. An alternating voltage from a signal generator is applied to a piezoelectric crystal, which then vibrates the assembly holding the orifice plate. A turbulent jet of air issuing from the hole in the cover above the orifice disperses the droplets before they can coagulate. Filtered, dry dilution air is introduced to dry the droplets and transport the aerosol from the generator.

There is a range of about a factor of two in the frequency producing uniform droplets. Within this range, certain frequencies may produce satellite droplets, that is, droplets much smaller than the main drops being produced. They can be eliminated by adjustments of the vibrating frequency. Another undesirable characteristic is the production of multiplets because coagulation of the droplets cannot be completely suppressed. In practice, the operating conditions are adjusted to minimize the multiplets, and corrections are applied to the data taken with the aerosol, if necessary.

Particles from the vibrating orifice typically carry several thousand elementary charges. Since the presence of a high electrical charge may affect subsequent processes involving

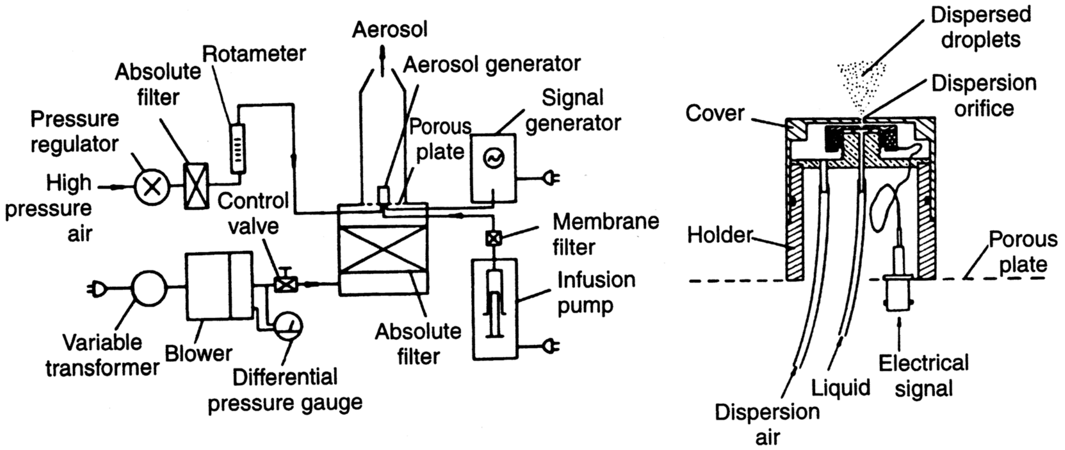

Figure 21-2 Diagram of a vibrating-orifice aerosol generator of the Berglund-Liu design. Reprinted from Liu (1974) with the permission.

the aerosol, it is common practice to "neutralize" the charge by exposure of the aerosol to a radioactive source. The source creates charged ions in the gas, which are attracted to charges of the opposite sign on the particles, reducing the particle charge distribution to a Boltzmann equilibrium. The radioactive sources commonly used include beta emitters such as ^{85}Kr or tritium and alpha emitters such as ^{210}Po or ^{241}Am.

In general, solid particles dry with the formation of voids, resulting in a density less than that of the bulk material. To some extent, the drying process can be controlled by varying the volatility of the solvent (e.g., by varying the proportions of water and alcohol) and by controlling the amount of dilution air. If the drying is too rapid, the particles tend to have more voids. The average density of the particles, including voids, can be determined by the following equation:

$$\rho_{p,av} = (d_d/d_g)^3 C_m \qquad \text{(Eq. 21-5)}$$

where $\rho_{p,av}$ is the average density of the particles (in kg/m^3), d_g is the particle geometric diameter (in μm) determined with a microscope, and C_m is the mass concentration (in g/L) of the solute (John and Wall 1983).

One example of a solid particle aerosol, ammonium fluorescein, deserves special mention because of its useful particle properties and the requirement for special generation procedures. Ammonium fluorescein particles are very smooth and have the bulk material density 1350 kg/m^3 [1.35 g/cm^3]. The material has low hygroscopicity and the fluorescence can be used for detection with high sensitivity. The particles are useful for sampler calibration and for checking for particle bounce. The solution is prepared by dissolving fluorescein in ammonium hydroxide. A reaction takes place with an ammonium group replacing a hydrogen atom on the fluorescein molecule. As a result, the relative molecular weight increases by 5%; thus, 12.8 g fluorescein per liter of solution is a 1% volume concentration. When particles of ammonium fluorescein larger than about 10 μm are generated, the droplets tend to dry too fast. To produce smooth particles, it is necessary to humidify the dilution air to slow down the drying. By this method, particles as large as 70 μm have been generated (Vanderpool and Rubow 1988).

The vibrating-orifice aerosol generator can be used to produce uniformly charged, monodisperse aerosols (Reischl et al. 1977). The technique involves insulating the cap over the orifice assembly and applying a DC voltage to the cap. Charges are induced onto the top of the jet and are trapped on the droplets when they separate from the jet. The monodispersity of the droplets leads to uniform charging. The application of modest voltages, that is, up to ±10 V produces particles with $\pm 10^4$ elementary charges. Reischl et al. (1977) present the theory and data demonstrating the method, which is useful for experimentation with charged aerosols.

Another method of producing monodisperse droplets is by the spinning disk, in which a liquid jet is fed at a constant rate onto the center of a rotating disk. The liquid spreads over the disk's surface in a thin film, accumulating at the rim until the centrifugal force exceeds the capillary force acting to hold it together, and a droplet is thrown off. Droplet size d_d depends on disk diameter, d_s (μm), and rotating speed, ω_s (rpm) as follows:

$$d_d = (W\gamma_L/\rho_L\omega_s^2 d_s)^{1/2} \qquad \text{(Eq. 21-6)}$$

where γ_L and ρ_L are the surface tension and the density of the liquid, and W is a constant. Spinning disks have been investigated by Walton and Prewett (1949) and May (1949) using an air-driven spinning top, and by Whitby et al. (1965) and Lippmann and Albert (1967) using a motor-driven spinning disk. Unlike a vibrating-orifice atomizer, aqueous suspensions as well as solutions can be used. The spinning disk produces an order of magnitude higher aerosol concentrations compared to the vibrating orifice. However, the monodispersity is not as high; the σ_g values are approximately 1.02 and 1.1 for the vibrating orifices and spinning disks, respectively.

A disadvantage of the spinning disk is that undesired satellite droplets are formed and must be removed from the useful aerosol. In addition, the constant W (Eq. 21-6) varies with the instrument and the feed material used, so that the particle size cannot be as easily calculated as for the vibrating-orifice atomizer.

21.5.1.3 Controlled Condensation Techniques Condensation is also a method that produces monodisperse aerosols for calibration purposes. In this method, the heated vapor of a substance is mixed with nuclei on which it subsequently 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

$$d_t^2 = d_o^2 + bt \qquad \text{(Eq. 21-7)}$$

where b is a constant, related to the concentration, the diffusivity of the vapor, and to the temperature. If bt is the same for all particles and much larger than d_o^2, an aerosol containing monodisperse particles is produced. In practice, a uniform temperature profile, sufficient vapor concentration, and sufficient residence time in the condensation region are the key controls, and a constant nuclei concentration provides a stable aerosol concentration (Sinclair and LaMer 1949; Rapaport and Weinstock 1955; Prodi 1972; Liu and Lee 1975; Tu 1982). Particle sizes from 0.03 to greater than 2 μm with σ_g of 1.2 to 1.3 can be produced this way. The number concentration can be as high as 10^{13} particles/m^3 [10^7 particles/cm^3]. An example of a condensation aerosol generator is shown in Figure 21-3.

21.5.1.4 Electrospray Technique Another method for producing monodisperse aerosols is to use an electrostatic atomizer or electrospray device (see Chapter 20; Hayati et al. 1987a,b; Fernandez de la Mora et al. 1990; Meesters et al. 1992; Grace and Marijnissen 1994). Electrospraying refers to the generation of liquid droplets by feeding semiconducting fluid through a capillary tube. A high potential difference is established between the tube and a coaxial plate downstream to create an electric field near the tip of the tube. There are several modes by which the liquid can break up into droplets, depending on the field strength, hydrodynamic parameters, and liquid properties. For certain conditions, when the field is strong enough, the liquid meniscus at the capillary outlet forms a cone from whose tip a very thin liquid jet emerges in the cone-jet mode (Cloupeau and Prunet-Foch 1989, 1994). The microjet breaks by varicose wave instabilities into a stream of charged droplets, having diameters roughly twice as large as the jet diameter but much smaller than the capillary diameter (Rosell-Llompart and Fernandez de la Mora 1994; Tang and Gomez 1994). A system using this technique can generate very small droplets without the clogging problems associated with a very small orifice. The mean droplet size is usually in the range of 0.3 to 50 μm, but can be as small as 10 nm. The size is a function of the nozzle diameter, liquid feed rate, field strength, current, and properties of the liquid, including surface tension, electrical conductivity, and viscosity (Smith 1986). The droplets are charged up to an appreciable fraction of the Rayleigh limit and initially repel each other, until they are neutralized.

Tang and Gomez (1994) have demonstrated a generation system producing monodisperse droplets in the size range from 2 to 12 μm with a 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 a σ_g of 1.1 (Rosell-Llompart and Fernandez de la Mora 1994). By using volatile solvents, particles down to nanometer diameters can be produced. More recently, a practical system has been developed using the electrospray technique to produce monodisperse particles with a mean diameter from 4 nm to 1.8 μm and a σ_g of 1.1. The operating ranges of the important parameters such as liquid feed rate, electrical conductivity, and concentration of the solution were determined (Chen et al. 1995). Unfortunately, the main group of primary particles is accompanied by a second group of satellite particles having diameters approximately eight times smaller. For some applications it will be necessary to remove the second group by using a size-selective device.

The electrospray technique produces supermicrometer aerosol less monodisperse and with lower concentration than that from a vibrating-orifice generator. However, for submicrometer particles, and especially for nanoparticles, electrosprays offer unique advantages.

21.5.2 Monodisperse Aerosols with Nonspherical Particles

The effects of particle shape on instrument response are important, especially for sizing instruments. The effects of shape on instrument response can be investigated by using monodisperse aerosols of nonspherical particles during calibration. One way of generating these aerosols is to nebulize a liquid suspension containing monodisperse nonspherical particles. Various techniques have been used to produce monodisperse particles of highly uniform particle size and shape. Matjevic (1985) 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 (Esmen et al. 1980; Loo et al. 1982; Hoover et al. 1990; Vaughan 1990; Baron et al. 1994; Chen et al. 1996; Deye et al. 1999). The vibrating-orifice and spinning-disk aerosol generators described above can also be used to generate nonspherical particles, such as crystalline sodium chloride particles. Although the generators produce spherical droplets, the

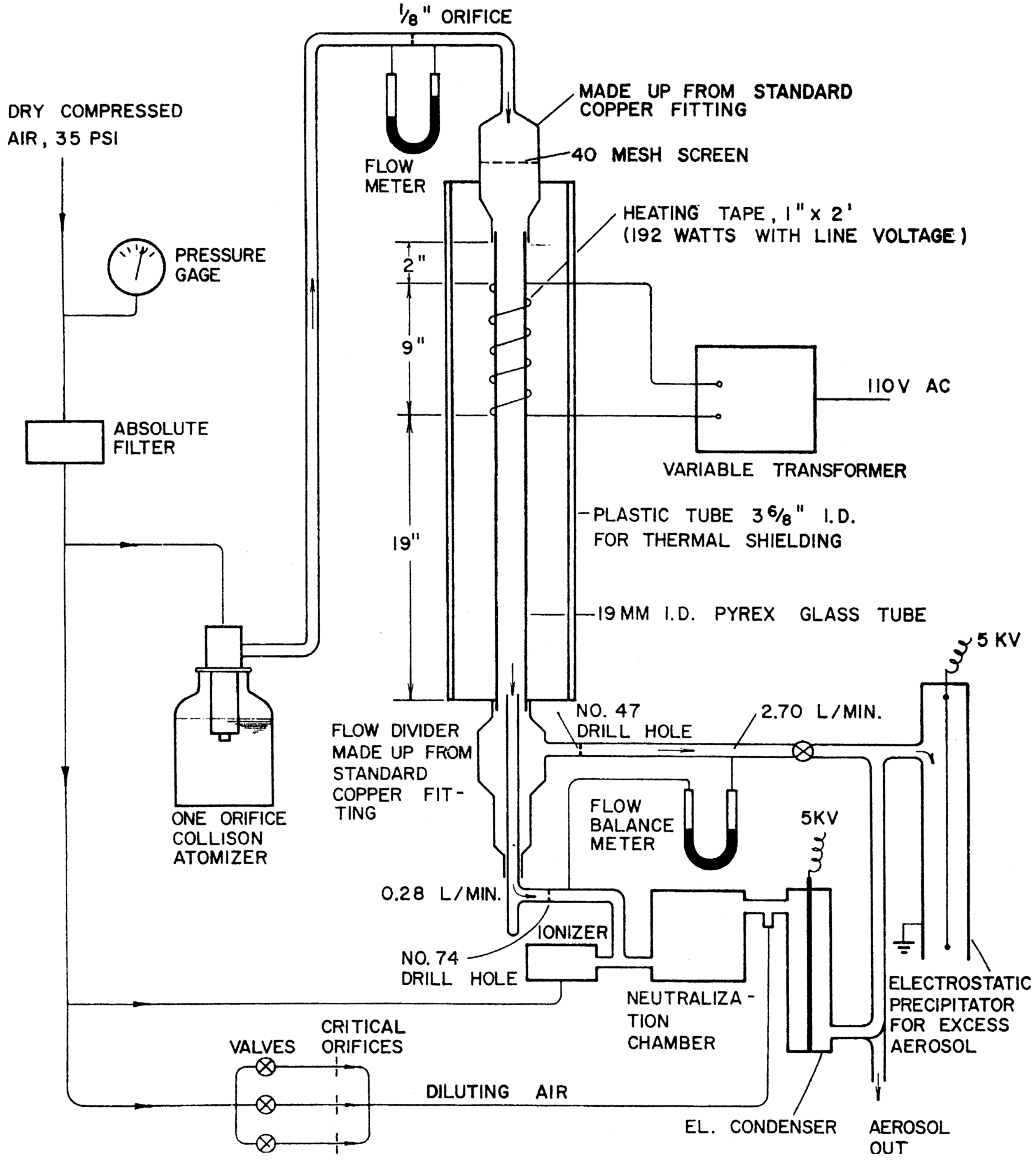

Figure 21-3 Condensation-type monodisperse aerosol generator. Reprinted from Tomaides et al. (1971) with permission.

crystal form of the solid particles determines the shape of the final aerosol after drying the liquid vapor.

In addition, naturally occurring materials, such as fungal spores, pollens, and bacteria or multiplets of spheres, have been used as test aerosols of nonspherical particles (Corn and Esmen 1976; Adams et al. 1985). The aerosols of fungal spores, bacteria, and pollens are commonly generated using either the wet dispersion or the dry powder dispersion technique (described in 21.5.4). Depending on the needs in using these test particles of biological origin, the concerns of viability and culturability might need to be considered (Henningson and Ahlberg 1994; Griffiths et al. 1996; Reponen et al. 1996; Ulevicius et al. 1997). The details of instrument calibration such as the generation, collection, and assay methods for microorganisms and other bioaerosols are described in Chapter 24.

21.5.3 Size Classification of Polydisperse Aerosols

An aerosol with a narrow size range can be produced from a polydisperse aerosol by passing the aerosol through a size classifier. For particles smaller than 0.1 μm, Liu and Pui (1974) developed a differential electrical mobility analyzer to classify aerosol particles of the same electrical mobility by using the design of Knutson and Whitby (1975). Because most of the classified particles are singly charged, most of the aerosol produced is monodisperse, but there is a smaller amount of doubly-charged particles with the same electrical mobility but different particle size. This classification technique has been used to produce a submicrometer aerosol for calibrating CNCs (or CPCs) and diffusion batteries, and for determining particle deposition in human nasal and oral casts (Liu et al. 1975; Scheibel and Porstendörfer 1984; Cheng et al. 1990). For particles greater than 1 μm, a size-classifying technique based on particle inertia is generally used. Two virtual impactors can be placed in a series to segregate the desired fraction of the input aerosol for use in instrument calibration (Chen et al. 1988; Pilacinski et al. 1990). To classify aerosols in the 0.1- to 1.0-μm range, a technique involving both the mobility analyzer and a single-stage micro-orifice impactor has been used (Romay-Novas and Pui 1988). The above techniques are also used for reducing undesired particles, such as PSL agglomerates from an air nebulizer or satellite particles from a spinning disk generator.

All the devices and techniques described above classify aerosol particles while the particles are airborne. Other instruments, such as elutriators, spectrometers, cascade impactors, and cascade cyclones, can be used to classify particles by collecting size-classified particles on a substrate that can then be resuspended. For example, a spiral centrifuge can collect 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 (Kotrappa and Moss 1971). The disadvantage of most size-classifying techniques is that only a small quantity of particles is produced.

21.5.4 Polydisperse Aerosols

Polydisperse aerosols can be used as test aerosols to calibrate instruments and samplers when used with an auxiliary sizing instrument. Because the entire size distribution typically can be obtained in a minute, this method has significant advantages. Some polydisperse aerosols, such as aluminum oxide, coal dust, and Arizona Road Dust, are used in calibrating dust monitors, including samplers for respirable dust. There are two ways to generate polydisperse aerosols, wet droplet dispersion and dry powder dispersion.

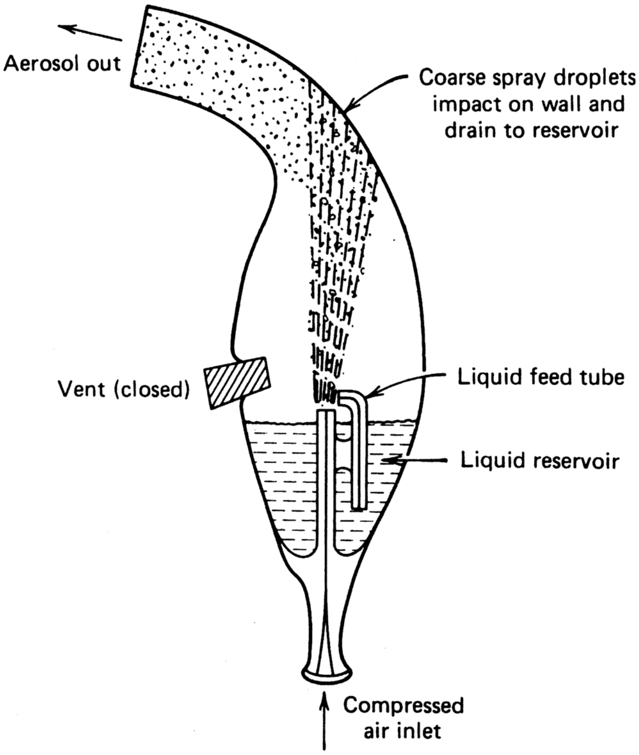

Figure 21-4 Drawing of a DeVilbiss Model 40 Glass Nebulizer. Reprinted from Hinds (1999) with permission.

21.5.4.1 Wet Dispersion The simplest way to disperse a droplet aerosol is by nebulization. Two types of nebulizers are commonly used to produce aerosols. Air-blast nebulizers (Mercer et al. 1968) use compressed air (1.03 to 3.44×10^5 Pa [15 to 50 psig]) to draw bulk liquid from a reservoir as a result of the Bernoulli effect (Fig. 21-4). The high-velocity air breaks up the liquid into droplets and then suspends the droplets to form an aerosol. Droplets produced from this method have a d_v of 1 to 10 μm and σ_g of 1.4 to 2.5 (Table 21-3). The particle size distribution can be modified by varying the pressure of the compressed air or the dilution ratio in the solution. A problem arises when the bulk liquid contains a volatile solvent that evaporates rapidly after droplet formation. The continuous loss of solvent increases the solute concentration in the reservoir and causes particle size to increase gradually with time. This problem can be circumvented by circulating the solution through a large reservoir (DeFord et al. 1981), delivering the solution at constant rate

(Liu and Lee 1975), and by presaturating the supply air, and cooling the nebulizer.

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. The vibrations are transmitted through a coupling fluid to a cup containing the solution to be aerosolized. At a certain frequency (1.3 to 2.5 MHz), a heavy mist appears above the liquid surface of the cup. The diameter, d_v, of the droplets making up the mist is related to the wavelength of the capillary waves, which decreases with increasing frequency, f, of the ultrasonic vibrations and is related to the surface tension, σ_L, and the density, ρ_L, of the liquid, as follows:

$$d_v = k_p(\sigma_L/\rho_L f^2)^{1/3} \qquad \text{(Eq. 21-8)}$$

where k_p is the proportionality in the equation and has been found to hold for f between 12 kHz and 3 MHz (Mercer 1973; Hinds 1999). Normally the d_v is 5 to 10 μm, with a σ_g of 1.4 to 2.0 (Table 21-3).

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. (1970) and Newton et al. (1980).

21.5.4.2 Dry Dispersion The dry dispersion of powders can produce aerosols that have physical and chemical characteristics that are the same or similar as those which will be sampled by the instrument under calibration. Numerous techniques have been described by Hinds (1980) for dispersing dust or fiber particles (Table 21-4). Basically, the techniques consist of two steps: (1) a means of delivering powder into the dispenser at a constant rate, and (2) a means of dispersing the powder to form an aerosol. However, the dispersibility of a powder depends on the powder material, particle size, particle shape, and moisture content. Two common methods for generating aerosols from powders are the Wright Dust Feed (Fig. 21-5) and the fluidized bed (Fig. 21-6).

Aerosol generators using fluidized beds as the dispersing mechanism have the ability to thoroughly deagglomerate powdered samples. When equipped with a suitable dust feed mechanism, fluidized beds can operate stably over long periods of time. Fluidized beds can be scaled over a wide range of sizes, from very small to extremely large, producing aerosol concentrations from milligrams to tens of grams per cubic meter.

A fluidized bed consists of relatively large bed particles, typically of the order of 100-μm diameter, in a cylindrical container. The floor of the bed is of a porous material, such as a fine screen or a filter, which can retain the bed particles but allow an upward flow of air. In operation, a fluidized bed resembles a boiling liquid, but the best indicator of fluidization is the pressure drop across the bed. As the upward airflow is increased from zero, the pressure drop initially rises linearly with flow rate. Eventually, a condition is reached where the air drag on the particles is equal to the weight of the bed. The pressure drop curve then levels off. The flow velocity at the break in the curve is called the minimum fluidization velocity (MFV; Carpenter and Yerkes 1980). The MFV is correlated with the bed particle Reynolds number. For a typical fluidized-bed aerosol generator, the MFV is of the order of 10 cm/s.

In the two-component fluidized bed initially described by Guichard (1976), a relatively fine powder of the material to be aerosolized is added to a fluidized bed containing larger bed particles. An airflow velocity sufficient to fluidize the bed will exceed the elutriation velocity of the powder particles. The details of the aerosolization process are not known, but a plausible scenario is that in the collisions between bed particles, adhering powder or dust particles are knocked off the bed particles. The constant action of the bed promotes uniform coating of the bed particles with a layer of dust particles, accounting for the deagglomeration that is observed. Fresh surfaces are constantly generated by the grinding action in the bed. Therefore, the use of dry air is recommended because moisture promotes oxidation of particle surfaces. Strong electrical charging is usually produced from contact and triboelectrification. This indicates that the aerosol should be neutralized before use. Some fluidized-bed generators incorporate strong sources of sound or vibration. Vibration can improve the performance in several ways. There is a tendency for channeling to occur in the bed, whereby the air flows at a higher rate in localized areas. Vibration can suppress the channeling, promoting a more uniform flow. Dust may collect on the walls above the bed. Periodically, the accumulation breaks loose, producing a burst in concentration. Vibration inhibits such an accumulation of dust. Vibration also improves the feeding of dust into the bed from a screw or chain.

Although dry air has often been recommended for dry dust generation for the reason noted above, it also has been suggested that more efficient generation of dust may occur by increasing the relative humidity to a higher level (e.g., 10–30%). This higher humidity level was shown to decrease the charge level on a generated asbestos aerosol, thus reducing losses in the generation system (Baron and Deye 1990). Anecdotal evidence suggests that very dry air can often charge other aerosol particles (especially nonconducting particles such as latex) to such high levels that aerosol is prevented from leaving the generator (P.A. Baron, personal communication, 2008).

An example of a two-component fluidized bed aerosol generator (Marple et al. 1978) is shown in Figure 21-6. The fluidized bed has a 1.4-cm-thick layer of 100-μm brass beads (stainless steel is also frequently used) in a 5.1-cm-diameter chamber. This type of generator is commercially

TABLE 21-4 Operating Parameters of Dry Powder Dispersers

	Wright Dust Feed	Fluidized Bed	NBS II Dust Generator	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 metering gear to deliver the powder and air dispersing 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, ($\times 10^{-5} \mathrm{m}^3/\mathrm{s}$ [L/min])	14–67 [8.5–40]	8–33 [5–20]	80–140 [50–85]	20–35 [12–21]	23–188 [14–113]
Feed flow rate, $\mathrm{mm}^3/\mathrm{min}$	0.24–210	1.2–36	1200–50,000	0.9–2.5	2000–30,000
Output mass concentration, $\mathrm{g/m}^3$ ($\rho = 1000\ \mathrm{kg/m}^3$)	0.012–11.5	0.13–4.0	15–100	0.0003–0.04	10–1500
Recommended size range, μm	0.2–100	0.5–100	1–100	1–50	0.2–30
Source	***BGI***	***TSI***	***BGI***	***TSI***	***FLU***

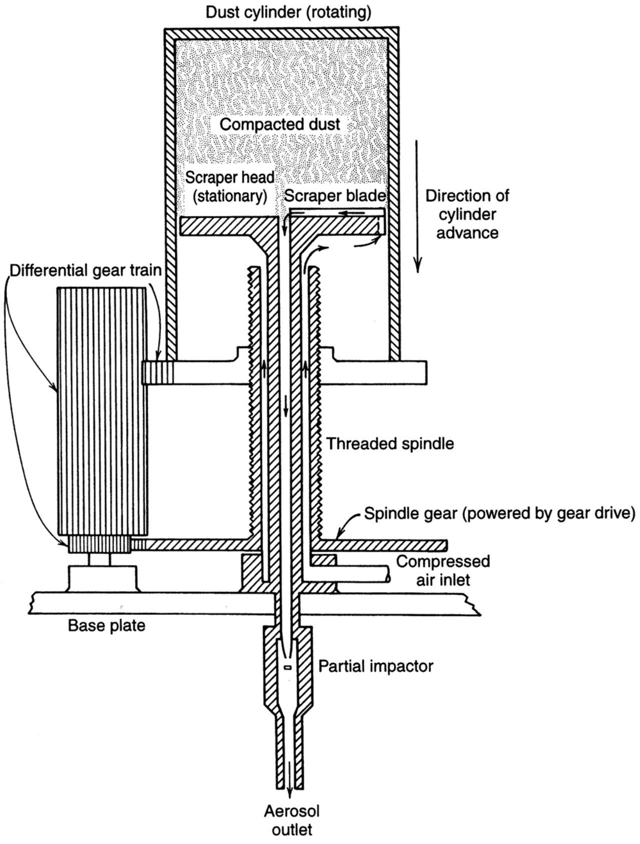

Figure 21-5 Wright Dust Feed. Reprinted from Hinds (1999) with permission.

available (Model 3400, ***TSI***). While such a generator is useful for many purposes, there are some non-ideal aspects that require attention. When the generator is turned on with fresh metal bed particles, an aerosol is produced from the bed particles themselves. Initially, the source of particles is the fraction of small particles present in the bed particle sample. These small particles can be rapidly cleared from the bed by operating first at a higher than normal flow velocity. Even after the small particles are removed, a fine aerosol persists for a long period of time due to the grinding action that removes asperities from the bed particles. This type of background is not observed with glass beads, which are smoother and which have less violent collisions because of their lower mass. After operation with a dust sample, a fluidized bed containing the beads with some remaining dust cannot be cleaned effectively. If the same type of aerosol is needed again, the bed can be emptied and the bed material stored for future use.

The properties of some dusts may be altered in a generator having large metal bed particles. For example, aluminum particles can be flattened and aluminum oxide particles can be broken up in the bed. Such alterations of particle properties could be significant for the subsequent use of the aerosol. The problem can be alleviated by using smaller bed particles or bed particles of lower density.

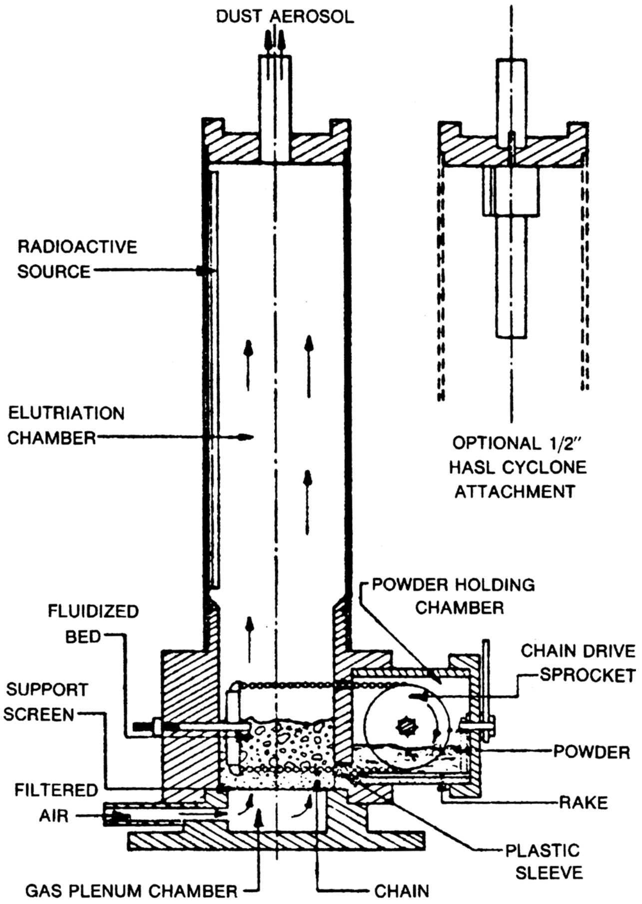

Figure 21-6 Two-component fluidized-bed aerosol generator. Reprinted from Marple, Liu, and Rubow (1978) with permission.

John and Wall (1983) developed a sonic fluidized bed, which avoids some of the problems of the large beds and which is useful for some applications. The bed's main feature is its small size, 25-mm diameter at the base, requiring less than 1 g of bed particles. The bed is funnel shaped so that the fluidizing velocity is higher than the exit velocity, favoring control of the elutriation velocity. For bed particles, 200-μm glass beads of the type used in gas chromatographic columns can be used. Such beads are highly uniform and clean. Because of the small amount of glass beads required, the beads can be discarded when changing dust samples. A refinement is the addition of sonic energy to the bed. The sonic fluidized bed (Fig. 21-7) is vibrated by inexpensive piezoelectric crystals driven by an electronic oscillator at approximately 9 kHz. Because the bed lacks a feed system, it can only be used in a batch mode. It has been used successfully to generate aerosols of glass beads, A/C test dust, and soil for the testing of aerosol samplers. The soil was simply passed through a coarse screen and placed in the bed without bed particles, the coarsest soil particles functioning as bed particles.

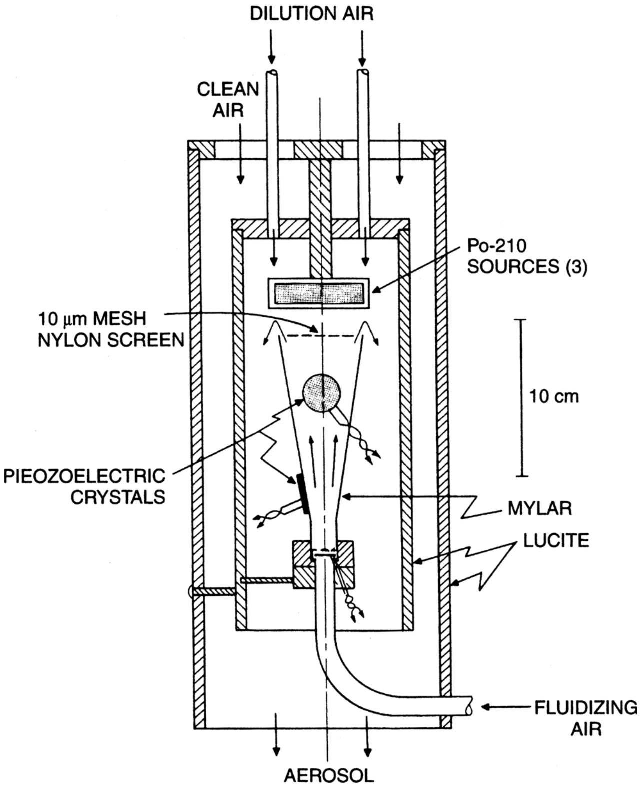

Figure 21-7 Sonic fluidized-bed aerosol generator. Reprinted from John and Wall (1983) with permission.

A feed system for a fluidized bed generator was designed by Sussman et al. (1985) that allowed more constant and controllable output over time. The powder and bed beads were mixed, placed in a hopper, and pneumatically fed in small amounts at selected time increments into the fluidized bed. The overflow from the fluidized bed was allowed to fall into an overflow chamber to keep the bed height constant.

Spurny et al. (1975) developed a fluidized bed for the generation of aerosols of asbestos fibers. A special feature of this generator is a mechanical vibrator with adjustable amplitude and frequency. The effect of these vibration parameters on the aerosol concentration, fiber diameter, and fiber length was explored for several varieties of asbestos. It was found that the aerosol characteristics can be controlled to some extent by adjusting the vibration parameters. The generator was found capable of producing useful asbestos aerosols. Using a similar approach, Weyel et al. (1984) used a low frequency sonically fluidized bed to generate cotton fibers.

Besides the Wright Dust Feed and the fluidized bed, several dry powder generators are commercially available (Table 21-4). The ***TSI*** small-scale powder disperser is used to produce a small quantity of powder aerosols primarily for laboratory testing (Chen et al. 1995) and the Jet-O-Mizer$^{\mathrm{TM}}$ is able to produce a large quantity of powder aerosols for inhalation studies (Cheng et al. 1985).

A simple technique for generating brief bursts of latex particles is to place a small quantity of the suspension on a glass slide or other clean surface, allow the suspension to dry, and gently brush the deposit off the surface toward the inlet of the instrument to be calibrated. In the approximate range from 2 μm to 20 μm, this approach is useful for size calibration of high resolution instruments. When generated in this fashion, particles are more likely to be agglomerated, especially at the low end of the indicated size range.

Some generation techniques for fibrous aerosols are described in Chapter 23.

21.5.5 Test Aerosols with Tagging Materials

For some applications, particle detection is facilitated by incorporating fluorescent dye or radioisotope tags in the particles during their production. A fluorescent tagging material such as fluorescein can be analyzed in solution with nanogram sensitivity. Colored substances such as methylene blue can be analyzed with microgram sensitivity. The tagged aerosol may be extracted from a filter or a surface, enabling the quantitation of collection efficiency and wall losses within a sampler. Radiolabeling techniques have been used with the capability of detection of extremely low concentrations (Newton et al. 1980).

21.6 CALIBRATION OF FLOW, PRESSURE, AND VELOCITY

Accurate measurement of gas flow rate, pressure, and velocity is an integral part of instrument calibration (Mercer 1973; Lippmann 1995; Hinds 1999). Various instruments (Table 21-5) and techniques involved in the measurement of these parameters are discussed in this section.

21.6.1 Flow Rate Measurement

Depending on the flow rate and other practical considerations, various types of flow meters can be used in an aerosol system to measure flow rate: variable (pressure) head meters such as orifice or venturi meters, variable area meters such as rotameters, bubble meters, and others (Table 21-5). Normally, the meters have a flow restriction that causes an increase in gas velocity and kinetic energy with a corresponding decrease in potential energy, that is, static pressure. The flow rate can be calculated by knowing the pressure drop, the cross-sectional area upstream and at the constriction, the density of the gas, and the discharge coefficient. Taking into account the flow constriction and frictional effects, the discharge coefficient is the ratio of actual flow rate to ideal flow rate and is dependent on the design of the flow restriction.

The variable head meter determines the average flow rate by measuring the pressure differential across a calibrated

TABLE 21-5 Instruments that Measure Flow Rate, Volume, Pressure, and Velocity of Gases[a]

Quantity Measured	Instrument	Range	Standard
Volume	Spirometer	1 L to 1 m^3	Primary standard for flow rate calibration
	Bubble flow meter[b]	1 cm^3 to 10 L	
	Piston-displacement meter[c]	1 cm^3 to 12 L	
	Dry gas meter	Unlimited[d]	Secondary standard for flow rate calibration
	Wet gas meter	Unlimited[e]	
Volumetric flow rate	Venturi meter[f]	0.001 m^3/s to 100 m^3/s	
	Orifice meter[f]	10^{-6} m^3/s to 100 m^3/s	
	Rotameter[f]	10^{-8} m^3/s to 0.05 m^3/s	
Pressure differential	Manometer	0 to 2.0×10^5 Pa	Calibration standard
	Pressure gauge[f]	0 to 2.0×10^6 Pa	
	Pressure transducer[f]	0 to 2.2×10^7 Pa	
Velocity	Pitot tube	>5 m/s	Calibration standard
	Hot wire anemometer[f]	5 cm/s–40 m/s	

[a] Commercial sources of these instruments can be found in Lippmann (1995).

[b] Primary only if volume is determined by geometric measurement.

[c] Mercury-sealed. No longer in production due to mercury hazard.

[d] Range for flow rate calibration is 5–150 L/min.

[e] Range for flow rate calibration is 0.5–230 L/min.

[f] Frequent calibration against a standard is needed.

resistance in the flow stream. The venturi meter has a streamlined constriction throat in the flow stream to minimize energy loss and the discharge coefficient for this meter is slightly less than the ideal value of unity. A simpler form of a variable head meter is the orifice meter, in which a thin plate with a sharp-edged circular orifice is inserted at the center of the flow. Although a large energy loss takes place in the orifice meter, the meter is widely used because of its ease of installation and low cost. The discharge coefficient for an orifice meter depends on the orifice design and is generally much less than unity (~0.61, see Mercer 1973). For constant flow control of filter sampling, a type of orifice meter called a critical orifice meter can be used downstream of the filter. The orifice is small enough to provide a downstream pressure less than 0.53 of the upstream pressure, under which conditions the velocity in the constriction reaches the speed of sound; a further reduction in the downstream pressure does not increase the velocity through the system, resulting in a "choked flow." For a critical orifice, the flow rate Q (m^3/s [cm^3/s]) is proportional to the air pressure P_1 (Pa [$dyne/cm^2$]) and air density ρ_1 (kg/m^3 [g/cm^3]) at the upstream and is expressed as

$$Q = 0.58\, k\, \gamma^{1/2}\, (A_o/\rho_a)\, (\rho_1 P_1)^{1/2} \qquad \text{(Eq. 21-9)}$$

where k is the discharge coefficient, γ is the ratio of specific heats (=1.4 for air), A_o (m^2 [cm^2]) is the area of the orifice, and ρ_a (kg/m^3 [g/cm^3]) is the ambient air density. It is therefore essential during sampling practice not to overload the filter to cause significant reduction in pressure P_1 unless the pressure is monitored and appropriate correction of the flow rate can be made afterward. Kotrappa et al. (1977) reported a mean discharge coefficient of 0.70 when using 2-mm-long hypodermic needles as critical orifices. No systematic variation was found over the range of diameters between 0.25 mm (needle no. 26) and 1.23 mm (needle no. 16).

EXAMPLE 21-3

A critical orifice with 4×10^{-3} m [0.4 mm] diameter is fabricated for air sampling purposes and used downstream of a filter. The flow rate is measured to be 1.67×10^{-5} m^3/s [1 L/min] when the upstream pressure is close to the ambient (1.01×10^5 Pa [760 mm Hg], 20EC).

1. What size of orifice must be fabricated if the sampling flow rate is 3.33×10^{-5} m^3/s [2 L/min] (assuming that the downstream pressure is still less than 0.53 of the upstream pressure)?
2. What is the sampling flow rate when this orifice is used in Albuquerque, New Mexico (8.33×10^4 Pa [625 mm Hg])?
3. When the filter is loaded and the pressure gauge at the upstream of the orifice reads −980 Pa [−10 cm H_2O], what is the sampling flow rate?

Answer: Using Equation 21-9: $Q \,\alpha\, A_o(\rho_1 P_1)^{1/2}/\rho_a$:

1. $Q \propto A_o$. If the sampling flow rate is doubled, then the orifice diameter needs to be $(2)^{1/2}$ times of the original size, that is, $(4 \times 10^{-3}) \times (2)^{1/2} = 5.66 \times 10^{-3}$ m [0.57 mm].
2. $Q \propto (\rho_1 P_1)^{1/2}/\rho_a$. Because $\rho_1 = \rho_a$ and $P_1 \propto \rho_1$, the sampling flow rate remains unchanged (1.67×10^{-5} m^3/s [1 L/min]).
3. $Q \propto (\rho_1 P_1)^{1/2} \propto P_1$ (because $\rho_1 \propto P_1$). The sampling flow rate is $1.67 \times 10^{-5}\,(1.01 \times 10^5 - 980)/1.01 \times 10^5 = 1.65 \times 10^{-5}$ m^3/s [0.99 L/min].

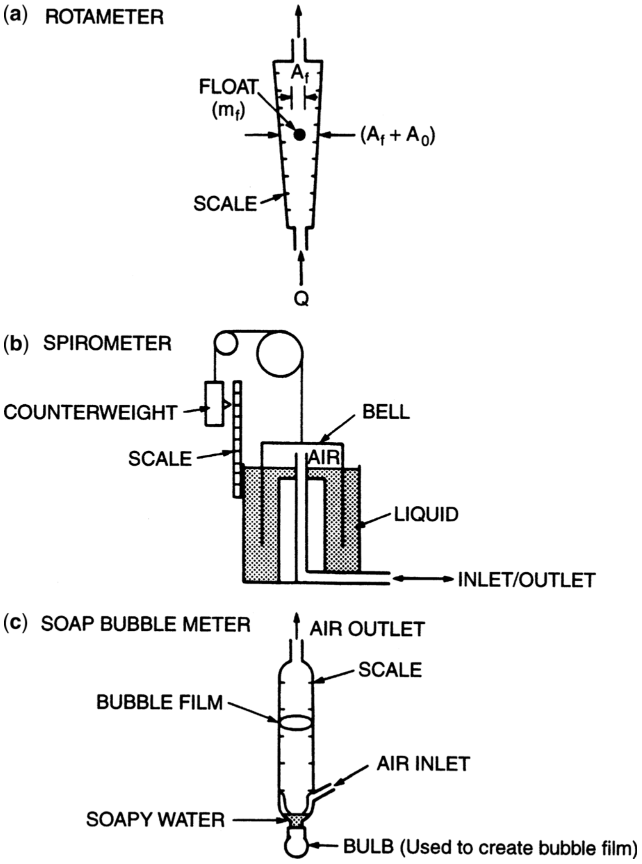

Figure 21-8 Schematic diagrams of flow-measuring instruments: **(a)** a rotameter, **(b)** a spirometer, and **(c)** a soap bubble meter.

Different from the variable head meter, the variable area meter changes the orifice area with flow to maintain a constant pressure drop. The most common type of the variable area meter is the rotameter (Fig. 21-8a). It consists of a "float" that moves up and down within a vertical tapered tube which is larger at the top than the bottom. The gas flows upward, causing the float to rise until the pressure drop across the annular area between the float and the tube wall is just sufficient to support the float. The height of the float indicates the flow rate. For a rotameter calibrated at an ambient pressure, $P_{a,c}$, and used at a different ambient pressure, $P_{a,i}$, the true volumetric flow rate Q_i at a fixed float position is given by

$$Q_i = Q_c(P_{a,c}/P_{a,i})^{1/2} \qquad \text{(Eq. 21-10)}$$

where Q_c is the flow rate indicated by the rotameter during calibration (Mercer 1973). For a rotameter calibrated and used at the same ambient pressure, the measured flow rate depends on the gas density ρ_g (pressure, P_g) in the tube. For example, if the rotameter is operated at a different gas density in the tube than that used during calibration (e.g., when the rotameter is located downstream of a filter or an impactor during aerosol sampling), then the actual volumetric flow rate at a fixed float position is given by

$$Q_i = Q_c(\rho_{g,i}/\rho_{g,c})^{1/2} = Q_c(P_{g,i}/P_{g,c})^{1/2} \qquad \text{(Eq. 21-11)}$$

where the subscripts i and c refer to the actual condition in the rotameter and the condition during calibration, respectively. Normally, $P_{g,c}$ is the ambient pressure, P_a, and $P_{g,i}$ is $(P_a - \Delta P)$ where ΔP is the gauge pressure downstream of the sampler.

EXAMPLE 21-4

A rotameter is calibrated by the manufacturer at sea level (1.01×10^5 Pa [760 mm Hg]) and used in Albuquerque, New Mexico (8.33×10^4 Pa [625 mm Hg]). What percentage of error in flow rate will result if the rotameter is not recalibrated? Assuming that this rotameter is recalibrated and then used downstream of a filter to measure the sampling flow rate, what will be the true flow rate if the flow rate indicated by the recalibrated rotameter is 8.33×10^{-5} m^3/s [5 L/min] and the pressure drop is 2.49×10^3 Pa [10 in H$_2$O]?

Answer: Using Equation 21-10:

$$Q_i = Q_c(P_{a,c}/P_{a,i})^{1/2},$$
$$P_{a,c} = 1.01 \times 10^5 \text{ Pa},$$
$$P_{a,i} = 8.33 \times 10^4 \text{ Pa},$$
$$Q_c/Q_i = (8.33/10.1)^{1/2} = 0.907, \text{ and}$$
$$\%\text{error} = [(Q_i - Q_c)/Q_i]100 = (1 - Q_c/Q_i)100 = 9.3\%.$$

Using Equation 21-11:

$$Q_i = Q_c(P_{g,i}/P_{g,c})^{1/2},$$
$$P_{g,i} = P_a - \Delta P = 8.33 \times 10^4 - 2.49 \times 10^3$$
$$= 8.08 \times 10^4 \text{ Pa [606 mm Hg]},$$
$$P_{g,c} = P_a = 8.33 \times 10^4 \text{ Pa [625 mm Hg]},$$
$$Q_c = 8.33 \times 10^{-5} \text{ m}^3/\text{s, and the true flow rate } Q_i = 8.33 \times 10^{-5}(8.08/8.33)^{1/2}$$
$$= 8.20 \times 10^{-5} \text{ m}^3/\text{s [4.92 L/min]}.$$

A rotameter is generally calibrated using a calibrated dry gas meter, bubble flow meter, or bell prover. A bell prover is frequently referred to as a spirometer (Fig. 21-8b) because it was derived from miniature bell provers developed for measuring respiratory function. It measures the volume of oil displaced in a container when the air under measurement is introduced; the volume can be as high as 1 m^3. For a smaller volume, a soap bubble meter (Fig. 21-8c) is a widely used primary standard. A bubble film is created in a tube from a reservoir of soapy water and acts as a nearly frictionless piston as the air passes through the tube. The distances of the bubble displaced along the tube and the cross-sectional area of the tube are used to determine the volume of the air entering the bubble meter. Several automated bubble meters that incorporate bubble detecting sensors, automatic timing, and readout of flow rate are commercially available (***BUC***, ***GIL***) and widely used as calibration standards for flow rates up to 6.7×10^{-4} m^3/s [40 L/min].

In addition, a flow calibrator using a solid graphite piston (***BII***), instead of the manually created soap films, has become popular as a flow calibration device because it eliminates some problems associated with consistent bubble generation. However, one should be aware that air leakage or unnecessary friction could occur between the solid piston and the internal wall surface and regular checkup is strongly recommended. Because of its valving system, this type of calibrator can also cause errors of a few percent when measuring flow from mass flow controllers.

A dry gas meter is also often used to calibrate rotameters and orifice meters, although it must be calibrated against a primary standard, such as a spirometer. The dry gas meter contains two bellows that are alternately filled and emptied by the metered gas. Movement of the bellows controls the action of mechanical valves that direct flow and operates a cycle-counting device that registers the total volume of gas passing through the meter. In operation, one inlet of the meter is always open to ambient pressure, because the housing of the device cannot support a very large pressure drop. In addition, at least 10 revolutions are recommended for each measurement to avoid the effect of nonlinear strokes. In a wet test meter, gas flows into a rotating system of chambers that connects to a revolution counter. The water level seals the chambers and acts as a valve to direct the flow to the proper chamber. The measured volume from this meter must be corrected for water vapor content. There is, however, no limit on the total gas volume measured.

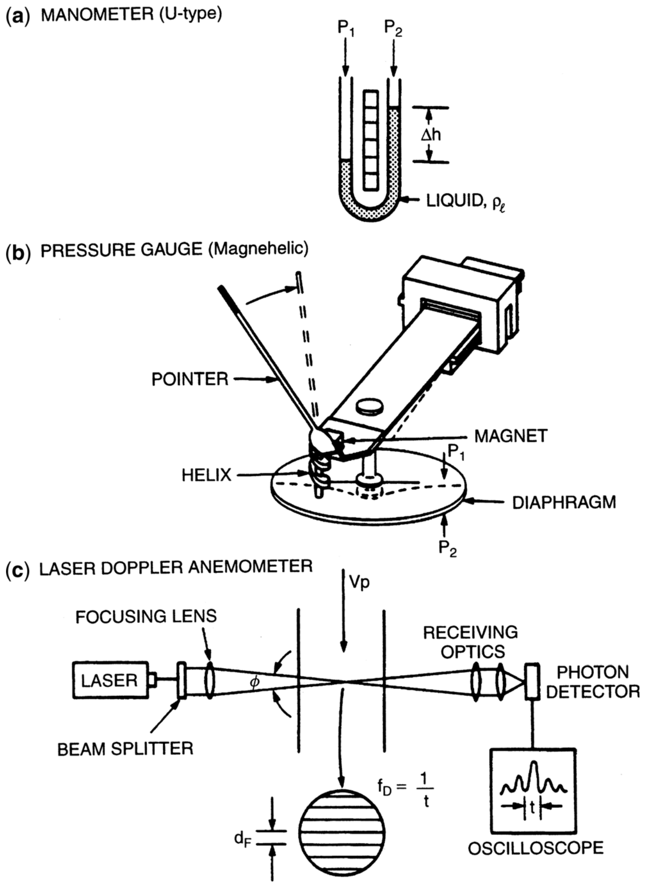

Figure 21-9 Schematic diagrams of instruments: **(a)** a manometer, **(b)** a pressure gauge (for gas pressure measurement), and **(c)** a laser Doppler anemometer (for particle velocity measurement).

21.6.2 Pressure Measurement

Three types of pressure sensing devices are commonly used to determine the pressure differential between two points in a system or the gauge pressure: manometers, mechanical gauges, and pressure transducers (Table 21-5). The liquid-filled manometer (Fig. 21-9a) consists of a glass or plastic tube sized to allow the height of the fluid level to balance against the incoming pressure. The manometer expresses the pressure differential by the difference in liquid column height. There are three types of manometers: U-tube, well-type, and inclined-type. Normally, a manometer does not require any calibration and can be used as a pressure standard provided that the specific gravity of the liquid is known.

The mechanical pressure gauge (Fig. 21-9b) is widely used as a pressure sensor of an aerosol system both in the laboratory and in the field. It normally consists of a metal or plastic housing that contains a diaphragm assembly. The diaphragm movement due to the pressure differential is transferred to the dial indicator mechanism. The pressure gauge provides an accurate reading as a percentage of the full-scale range of the device. The most common pressure gauge is the Magnehelic® (***DWY***), with a range from 2.49 Pa [0.01 inch of water] to 6.87×10^5 Pa [100 psig]. In this device, the diaphragm transmits the effect of pressure to an indicator by means of magnetic linkage without direct physical contact to ensure the accuracy and sensitivity of the instrument.

Pressure transducers are available to convert pressure to an electrical voltage. A transducer can be used to determine the pressure differential across an orifice meter and provide a real-time digital reading of pressure.

21.6.3 Velocity Measurement

Measurement of the local gas velocity in a duct is needed for proper isokinetic sampling and for calibrating flow-measuring devices. The most widely used device for measuring velocity is the pitot tube, which directly measures the velocity pressure in a moving gas flow. The pitot tube is normally considered as the calibration standard for gas velocity measurement.

A hot wire anemometer measures gas velocity by sensing the convective heat loss on a hot wire when the gas flows across it. The wire is heated electrically; heat loss to the air changes the wire's temperature, and the resulting change in resistance is sensed electronically and converted to velocity.

Note that this device's output is proportional to the convective heat transfer. Temperature and pressure are needed to obtain the velocity, and periodic calibration is needed to provide laboratory-quality accuracy (Chen 1993).

The velocity of particles in the air can be measured using a laser Doppler anemometer (or velocimeter, LDV; Fig. 21-9c). This allows either measurement of air velocity using small particles (on the order of 1 μm) or the velocity of larger particles that move at different velocities due to inertial or gravitational effects. This device does not require insertion of a sensing probe. It uses two laser beams to form an interference pattern with fringes. As a particle travels through the fringe pattern, its scattered light intensity provides a shift in the detected frequency, a phenomenon known as the Doppler effect. Another device, the particle imaging velocimeter (PIV), uses two laser-illuminated images of particles separated by a specified time interval to measure particle displacement. Particle velocity is computed from the measured displacement over the known time interval. The velocity can be measured in near real time over an area defined by the intersection of the laser illumination plane and the detector viewing area. The LDV measures velocity at a point, while the PIV measures velocity over a selected area. Several companies produce these instruments (***DAN***, ***OXF***, ***TSI***).

21.7 INSTRUMENT CALIBRATION

In this section, several aerosol instruments that determine particle size, number concentration, and mass concentration are briefly described. Their calibration standards and important parameters are summarized in Table 21-6.

21.7.1 Particle Sizing Instruments

Most instruments used in particle size analysis actually measure some physical property of the particle rather than a simple linear dimension. Particle size is then related to the diameter of a spherical reference particle that possesses the same physical property in the same amount. This can be an aerodynamic, optical, electrical, or diffusional property of the particle (Chen et al. 1989).

Aerodynamic sizing instruments, including collection-and-analysis devices (cascade impactors, elutriators, aerosol centrifuges, and cascade cyclones) and real-time analyzers are generally designed to measure particle sizes between 0.2 and 25 μm. Modern impactors, such as the micro-orifice uniform deposit impactor and electrical low pressure impactor, can classify particles down to nanometer sizes. These collection-and-analysis devices measure masses of particles that have been separated according to their aerodynamic properties in different force fields (Chapter 8). Real-time analyzers measure particle velocity as they pass through a sensing zone that can be a Doppler interference fringe pattern or a two-laser beam arrangement (Chapter 14). Parameters such as particle size, flow rate (velocity), density, and intrinsic gas properties can affect collection efficiency or instrument response (Marple and Willeke 1976; Stöber 1976; Chen et al. 1985; Baron 1986; Hering 1995; Marple et al. 2003). In addition, loading capacities and wall losses in each instrument should be fully examined to avoid incorrect data interpretation.

The optical particle counter (OPC) is a real-time instrument that uses single particle light-scattering technique to measure aerosol size distribution (0.1 – 20 μm) and number concentration (Chapter 13). Important parameters affecting the response include the size, shape, orientation, and refractive index of the aerosol particles, as well as the wavelength of the light source, the range of scattering angles, and the sensitivity of the photodetector. Particle size and refractive index are the two most important variables in OPC calibrations. Mie scattering equations are used to predict theoretically the response of an OPC, however, an OPC is usually calibrated with test aerosol (Hodkinson 1966; Gebhart et al. 1976; Willeke and Liu 1976; Chen et al. 1989). The particle concentration in an OPC must be limited to minimize the error due to multiple particles in the sensing zone; the coincidence error can be calculated from Poisson statistics.

Electrical mobility analyzers (Chapter 15) and diffusion batteries (Chapter 16) are sizing instruments based on the electrical and diffusional properties of submicrometer particles (<0.5 μm). Important parameters for the electrical mobility analyzer are the physical size and dielectric constant of the particle, and the flow rate and charging mechanism in the instrument (Liu and Pui 1974; Pui and Liu 1979; Yeh et al. 1983). The flow rate, temperature, particle size, as well as the geometric dimensions of the diffusion surfaces (e.g., screen wire diameter and tube length) are important for diffusion batteries (Cheng 1995).

21.7.2 Condensation Particle Counters

Particle number concentration can be determined by sampling particles through a high-efficiency membrane filter counting the particles with an optical or electron microscope. This is, however, time consuming. Liu and Pui (1974) reported a method utilizing a charged aerosol, a DMA, and an aerosol electrometer (AE) to calibrate a CPC (CNC). Aerosol electrometer calibration has become a widely used approach to calibrate CPCs (CNCs) (Liu et al. 1975; Liu et al. 1982a; ***TSI*** 3022 manual; Chapter 17).

Recent work from two sources has built on the work of Liu and Pui (1974). The National Institute of Advanced Industrial Science and Technology (AIST) in Japan (H. K. Sakurai, K. Saito, and K. Ehara, private communication, 2008) has developed a CPC calibration standard test facility

TABLE 21-6 Summary of Direct Measurements and Primary Standards of Aerosol Instruments and Important Parameters to be Considered in Instrument Calibration

Instrument	Operating Principle	Measured Quantity	Important Aerosol Parameters	Important Instrument Parameters	Particle Size Range (μm)	Direct Calibration Standard	Primary Calibration Standard	Main Advantage	Main Disadvantage
Size Measurement									
Cascade impactor	Particle inertial impaction	Mass	Size, shape, density	Flow rate, gas medium, physical dimensions	0.05–30	Monodisperse spherical particles with a known size and density	–	Aerodynamic size distribution based on mass concentration	Internal loss, particle bounce and re-entrainment
Aerodynamic particle sizing instrument	Time of flight during deceleration	Velocity	Size, shape, density, rigidity	Flow rate, pressure, gas medium	0.5–20	Monodisperse spherical particles with a known size, shape, and density	–	Real-time instrument with good sensitivity and resolution	Coincidence, density, and shape effects
Optical particle counter	Interaction between particle and light	Scattered light intensity from Single particles	Size, shape, refractive index	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	–	Noninvasive, real-time, *in situ* measurement; also good for number concentration measurement	Calibration changes with the material
Electrical mobility analyzer	Size classification based on electric mobility	Electric charge or particle count	Size, shape, dielectric constant, humidity	Flow rate, charging mechanism, electric field strength	0.001–0.1	Monodisperse,[a] spherical particles with a known size and dielectric constant	–	Suitable for particles smaller than 0.1 μm	Multiple charges on the particle
Diffusion battery	Particle diffusional deposition	Particle count or mass	Size, shape, number concentration	Flow rate, temperature, deposition surface	0.001–0.1	Monodisperse,[a] spherical particles with a known size	–	Suitable for particles smaller than 0.1 μm	Unsuitable for large particles or particles with a large aspect ratio
Number Concentration Measurement									
Condensation particle (nuclei) counter	Vapor condenses on particles and makes them detectable	Particle count	Size, number concentration, solubility, surface wettability	Flow rate, saturation ratio, temperature gradient, working fluid	0.001–0.5	Aitken counter with a microscopic measurement	Pollak counter, photographic counter, or electrically classified monodisperse aerosol	Suitable for concentration measurement of submicrometer particles	Size dependent counting efficiency
Mass Concentration Measurement									
Photometer	Interaction between particle and light	Total light scattering from all particles in sensing volume	Size, shape, refractive index, density	Wavelength of the light source, range of scattering angles, sensitivity of detector	0.3 – 1.5	Gravimetric measurement of filter samples	–	Real-time, continuous readout	Calibration changes with material type
Beta attenuation Monitor	Mass dependent absorption of β-radiation	Mass	Size, elemental composition	Uniformity of particle deposit	1 – 15	Gravimetric (equivalent) measurement of filter samples	–	Real-time measurement	Low sensitivity
Quartz crystal mass balance	Mass dependent resonant frequency of the crystal	Mass	Size	Sensitivity of the sensor, mass loading	0.02–10	Gravimetric measurement of filter samples	–	Real-time measurement	Frequent sensor cleaning

[a] For particle size smaller than 0.01 μm, electrically classified monodisperse aerosols are used as a calibration standard.

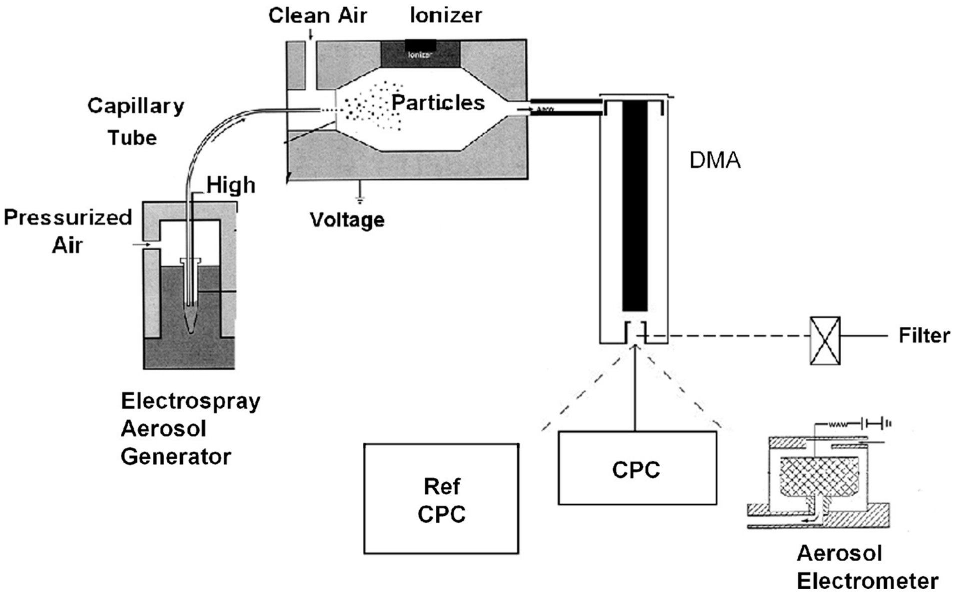

Figure 21-10 Schematic of test stand used to compare the test CPC and AE. A low sample volume reference CPC was operated during the experiments and filters were collected for certain aerosol concentrations. Electrospray sketch adapted from ***TSI*** manual.

where a CPC or an AE can receive a certified calibration. This approach employs a new Faraday cup design aerosol electrometer, an OPC, and precise airflow controllers. An independent approach has been taken at the National Institute of Standards and Technology (NIST) to demonstrate a calibration method traceable to NIST (Fletcher et al. 2009). This work compares the measurement results of three independent techniques for measuring aerosol concentration: continuous flow CPC, aerosol electrometer, and the aerosol concentration derived from microscopic particle counting (see Fig. 21-10).

An overview of the NIST approach is described below. A DMA was used to produce a nearly monodisperse, singly-charged, 80-nm, PSL test aerosol to challenge the CPC and the AE, and for filter collection/electron microscopy analysis. The test stand design incorporates a continuous, small sampling volume CPC aerosol concentration monitor to verify the aerosol stability, because the aerosol measurements are performed sequentially due to the relatively low flow rate ($\approx 2\,\text{L/min}$) of the test aerosol leaving the DMA. That is, the AE measurement is made, then the output tubing from the DMA is switched to the CPC, and in a few cases, the output tube is connected to the filter collection equipment for collection over a period up to 1 h.

The CPC determines particle concentration by single particle counting at a constant sample flow rate. The AE has been calibrated to a NIST-traceable electrical current standard. The subsequent aerosol concentration measurement is obtained by determining the electrical current produced by a charged aerosol transported to the detector by a controlled aerosol flow rate. The NIST traceability was reported for flow rates for all methods and a methodology to calibrate the AE to NIST-traceable electrical standards. The latter provides a calibration and a determination of the uncertainty in the aerosol-derived current measurement. The electrospray generator was able to produce aerosol concentrations over the range of $\approx$100 particles/cm^3 to $\approx$15,000 particles/cm^3 with a low number of dimer particles ($\approx$1%). An independent measurement of aerosol concentration is obtained by quantitatively collecting samples of the airborne PSL spheres on a small pore filter material and using electron microscopy to determine the number of particles collected.

21.7.3 Mass Concentration Monitors

The most common way to determine aerosol mass concentration is by determining the mass collected on a filter and the gas volume sampled (Chapter 7). This direct gravimetric approach is best achieved with a filter of high collection efficiency, for example, a glass-fiber filter or a membrane filter (Liu et al. 1983). Several real-time monitors have been developed to determine aerosol mass concentration: beta attenuation mass monitors, the tapered element oscillating microbalance (TEOM; described in Chapter 12), and photometers (Chapter 25). These monitors can provide the total mass concentration or only a desired mass fraction such as the respirable, thoracic, PM-2.5, or PM-10 mass concentrations by preceding them with a suitable particle size-selective device.

The beta attenuation monitor determines aerosol concentration on a filter or other substrate by measuring the attenuation of beta particles from a radioactive source. In the TEOM, particles are collected on a filter supported by an oscillating element. Increase in mass causes a shift in frequency. Besides these three collection-and-analysis instruments, real-time photometers have been used for determining mass concentration of aerosols. In a photometer, integral light scattering (or extinction) signals due to many particles in a sensing volume are related to mass concentrations and instrument calibration is always required. Several calibrations and comparisons among the above instruments have been done by Kuusisto (1983), Marple and Rubow (1984), Smith et al. (1987), and Baron (1988). Filter samples taken in parallel to real-time mass monitors can be used to calibrate the integrated mass response; however, it is difficult to calibrate real-time monitors over the short time intervals for which they are designed.

21.8 SUMMARY OF CALIBRATION PROCEDURES

Because the accuracy of aerosol data depends directly on the measurements, care must be taken to properly calibrate aerosol instruments and samplers. The following comments summarize calibration procedures and recommendations:

1. Aerosol instruments and samplers should be checked periodically to ensure that they are in proper operating condition. Before use, the calibration should be checked.
2. A device should be calibrated after it has been changed or repaired by the manufacturer, mishandled or damaged, or at any time when there is a question as to its accuracy.
3. A newly acquired instrument should be calibrated. Data supplied by the manufacturer may not be directly applicable to the user if the ambient pressure (altitude), temperature, and wind velocity in the sampling environment are different from those used during the manufacturer's calibration.
4. Prior to calibration of a new instrument, the operating principles and the construction should be studied. The instrument's manual should be read and the manufacturer's recommended operating procedures should be noted. It should be verified that the instrument is in proper operating condition.
5. Test aerosols that have physical and chemical properties similar to the aerosol to be measured should be selected for instrument calibration. The test aerosol should be monitored during the calibration to ensure its consistency in particle size and concentration.
6. Care must be taken in designing the setup to ensure that both the calibration device and the instrument to be calibrated will receive comparable aerosol samples. If one of the instruments needs dilution or augmentation of its flow, for example, by addition of particle-free air, this must be accomplished without alteration of the particle size distribution. This point concerns one of the most difficult aspects of instrument calibration. In addition, it is necessary to ensure sufficient particle mixing so that the particle concentration is uniform before delivered to the instruments.
7. Sufficient time should be allowed for the instruments to warm up, flow equilibrium to be established, and conditions to stabilize.
8. Flow rates should be checked before and after calibration runs.
9. Enough data should be obtained to give confidence in the calibration curve for a given parameter. Each calibration point should be made up of a sufficient number of readings to ensure statistical confidence in the measurement. The entire calibration should be repeated in separate runs.
10. A permanent record of all procedures, data, and results should be maintained.
11. Calibration curves and factors should be properly identified, including the date and conditions of calibration, the instrument involved, and who performed the procedure. It is useful to attach a tag to the instrument indicating where the original data are filed.

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