



Standard Practice for Calibration of an Airborne Particle Counter Using Monodisperse Spherical Particles¹

This standard is issued under the fixed designation F 328; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers procedures for calibrating and determining performance of an optical discrete airborne particle counter (DAPC) when presented with a challenge aerosol of near-monodisperse spherical particles. The practice is directed towards determination of accuracy and resolution of the DAPC for particles which have entered the sampling inlet of the DAPC. Consideration of inlet sampling efficiency is not part of this practice.

1.2 The procedures covered here include inlet sample flow rate, zero count level, particle sizing accuracy, particle sizing resolution, particle counting efficiency, and particle concentration limit.

1.3 The particle size parameter that is reported is the equivalent optical diameter based on projected area of a particle of known refractive index which is suspended in air. The minimum diameter that can be reported by a DAPC is normally specified by the manufacturer and the maximum diameter that can be reported for a single sample is determined by the dynamic range of the DAPC being used. Typical minimum diameters are in the range from approximately 0.05 μm to 0.5 μm and a typical dynamic range specification will be between 10 to 1 and 50 to 1.

1.4 The counting rate capability of the DAPC is limited by temporal coincidence for the specific instrument and by the maximum counting rate capability of the electronic sizing and counting circuitry. Coincidence is defined as the simultaneous presence of more than one particle within the DAPC optically defined sensing zone at any time. The coincidence limit is a statistical function of the airborne particle concentration and the sensing zone volume (1).² This limitation may be modified by the presence of particles with dimension so large as to be a significant fraction of the sensing zone dimension (2).³ The

saturation level or maximum counting rate of the electronic counting circuitry shall be specified by the manufacturer and is always greater than the DAPC counting rate for the challenge aerosol used for any portion of this practice.

1.5 Calibration in accordance with all parts of this practice may not be required for routine field calibration of a DAPC unless significant changes have been noted in operation of the DAPC or major DAPC component repairs or replacements have been made. In that situation, the DAPC should be taken to a suitable metrology facility for complete calibration, following necessary repairs or modifications. Normally, the routine field calibration may consist of determination of inlet flow rate, zero count level, and particle sizing accuracy. The DAPC functions to be calibrated shall be field or metrology facility calibrations shall be determined by agreement between purchaser and user, but shall not exceed 12 months, unless DAPC stability for longer periods is verified by measurements in accordance with this practice.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water⁴

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres⁵

E 20 Practice for Particle Size Analysis of Particulate Substances in the range of 0.2 to 75 μm by Optical Microscopy⁶

D 3195 Practice for Rotameter Calibration⁵

2.2 U.S. Federal Standard:

U. S. Federal Standard 209E, Airborne Particulate Cleanliness Classes in Cleanrooms and Clean Zones⁷

2.3 Japanese

¹ This practice is under the jurisdiction of ASTM Committee E29 on Particle and Spray Characterization and is the direct responsibility of Subcommittee E29.02 on Non-Sieving Methods.

Current edition approved June 10, 1998. Published December 1998. Last previous edition F 328-80 (1998).

² Jaenicke, R., "The Optical Particle Counter: Cross Sensitivity and Coincidence: Journal of Aerosol Science Vol. 3, 1972, pp. 95-111.

³ Knapp, J.Z. and Abramson, L.R., "A New Coincidence Model for Single Particle Counters. I. Theory and Experimental Verification" *Journal of Pharmaceutical Science and Technology*, Vol. 48, 1994, pp. 255-294.

⁴ *Annual Book of ASTM Standards*, Vol 11.01.

⁵ *Annual Book of ASTM Standards*, Vol 11.03.

⁶ *Annual Book of ASTM Standards*, Vol 14.02.

⁷ Available from U.S. General Services Administration, Federal Supply Service Standardization Division, Washington, DC 20406.

3. Terminology

3.1 For definitions of general terms used in this practice refer to Terminology D 1356⁵.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *calibration, n*—measurement, report, and adjustment as necessary, of test instrument operation in comparison with a standard material or an instrument of known adequate accuracy.

3.2.2 *calibration particles, n*—monodisperse, isotropic particles of known dimension and physical properties. Polystyrene latex spheres are typically used for calibrating a DAPC. These are available in diameters covering the operating range of most DAPCs.

3.2.3 *coincidence, n*—the simultaneous presence of more than one particle within the sensing volume of the instrument, causing the instrument to report the combined signal from the several particles as arising from a single larger particle.

3.2.4 *concentration, n*—number of particles within a specific particle size range or equal to and larger than a specific particle size per unit volume of air at ambient temperature and pressure.

3.2.5 *concentration limit, n*—the concentration specified by the DAPC manufacturer where the coincidence error is below 10 %. An error less than 10 % may be chosen, as required.

3.2.6 *counting efficiency, n*—the ratio, expressed as a percentage, of the reported particle concentration in a given size range to the actual concentration in the measured aerosol suspension.

3.2.7 *dynamic range, n*—the particle size range over which the DAPC produces particle size data with both a lower and an upper size boundary. The range may also be expressed as a particle size ratio, when the lower size is known.

3.2.8 *inlet flow, n*—the gas flow which enters the DAPC through the gas inlet. Flow rate is expressed as volume per unit time, at ambient temperature and pressure.

3.2.9 *lower sizing limit, n*—the smallest particle size at which the DAPC is capable of measuring with counting efficiency of 50 % ± 10 %.

3.2.10 *monodisperse, n*—a particle size distribution with relative standard deviation less than 5 %. Polystyrene latex (PSL) particles are commercially available with this property in particle sizes ranging from less than 0.1 µm to greater than 40 µm.

3.2.11 *particle size, n*—for calibration purposes, particle size is the modal diameter of the calibration particle batch used for each size threshold definition. For application purposes, particle size is the diameter of a reference sphere with known properties which produces the same response from the DAPC as the particle being measured.

3.2.12 *pulse height analyzer (PHA), n*—an electronic device for accumulating and sorting electronic pulses by voltage level. The output is generally a histogram with 64 to 4096 levels (referred to as “channels”). A PHA may be built into a DAPC or may be connected to a DAPC output test point. The PHA should have at least 64 channels and be capable of defining 1 % of the maximum voltage pulse level with 95 % accuracy.

3.2.13 *relative Standard Deviation, n*—a measure of the width of distribution data. It is quantified in terms of the ratio of the standard deviation of the distribution to the mean of the distribution. It is normally expressed as a percentage.

3.2.14 *resolution, n*—a measure of the ability of a DAPC to differentiate between particles of nearly the same size; also, the range of sizes which a DAPC would report for a particular particle if its size was determined repeatedly.

3.2.15 *sampled flow, n*—the air which passes through the sensing volume of a DAPC. The sampled flow may be either a portion of or the entire inlet flow. Sampled flow is expressed as volume per unit time, at ambient temperature and pressure.

3.2.16 *saturation level, n*—the maximum counting rate of the electronic circuitry at which accurate pulse amplitude sizing data are produced. The counting rate depends upon both the particle concentration and the sampled flow rate.

3.2.17 *sensing volume, n*—the portion of the illuminated volume in the DAPC through which the sample passes and from which scattered light is collected by the DAPC photodetector.

3.2.18 *zero count rate, n*—the maximum count indicated by a DAPC in a specified time period when the DAPC is sampling air free of particles larger than the lower sizing limit of that DAPC. This is also referred to as False Count Rate or Background Noise Level.

4. Summary of Practice

4.1 *Inlet Sample Flow Rate*—To report sampled particle concentration accurately, it is necessary to define the sample flow rate accurately. That flow rate may change if flow components in the DAPC are affected by long term operation or plugging by deposition of particulate material. In addition, DAPC flow is normally defined at ambient pressure and temperature and is not normally corrected to standard conditions. For these reasons, it is necessary to verify the inlet sample flow rate. A calibrated rotameter⁵ or other volumetric flow measurement device is required which operates with a pressure drop small enough so that the DAPC sampling pump is not loaded to the point where flow is degraded. The flow measurement device should report volumetric flow rate at ambient temperature and pressure. If a mass flowmeter is used, then correction to ambient conditions will be required. The flow measurement device is coupled to the DAPC inlet and the DAPC sampling pump is operated. The flow indication on the built-in DAPC flowmeter (if available) is recorded and compared with the flow indication on the calibrated flow measurement device. If the flow or the flow indication do not meet the required level, as indicated by the volumetric flow measurement device, the incorrect flow or flow indication shall be corrected and the remedial measures recorded.

4.2 *Particle Sizing Accuracy*—Although the DAPC may be used to characterize particulate suspensions containing materials that vary in shape and composition, consistent response to standard materials is required. An air suspension of calibration particles is generated by atomizing a liquid suspension of those particles, evaporating the liquid droplets to leave an air suspension of individual particles. The DAPC samples a



portion of this suspension⁸. The suspension may be diluted with clean gas, as required, to keep the particle concentration sufficiently low so that the coincidence error is below 3 %. The particle size and relative standard deviation of the calibration particles is either measured before the sizing accuracy determination or the particle vendor reports data traceable to the National Institute of Standards and Technology (NIST) or equivalent agency on the modal size and relative standard deviation of the calibration particles. The DAPC modal pulse amplitude response to the calibration particle suspension is recorded along with the standard deviation of the DAPC pulse data. This procedure is carried out with batches of monodisperse calibration particles whose sizes are within the DAPC dynamic range.

4.3 Particle Sizing Resolution—Sizing resolution of the DAPC defines its capability to differentiate between particles of nearly the same size. This DAPC parameter is determined after the particle sizing accuracy measurements are completed. Data obtained during determination of particle sizing accuracy can be used to determine particle sizing resolution. An air suspension of monodisperse calibration particles is generated as stated in 4.3. Particles shall be larger than the DAPC lower sizing limit by a factor of at least 2. The modal pulse amplitude and relative standard deviation is determined for the batch (or batches) of particles used; these data are converted to particle size mode and standard deviation. The increased relative particle size standard deviation as compared to the NIST-traceable data is used to calculate the DAPC particle sizing resolution. Normal practice is to report the particle sizing resolution as the increased relative standard deviation as a percentage of the size for the particular batch of calibration particles.

4.4 Zero Count Rate—When a DAPC is used for measurement of particle concentration in very clean gases, the number of particles counted per unit time may be very low. If the DAPC electronic or optical system is generating any noise pulses with amplitudes similar to those reported for particles at the lower sizing limit, some noise pulses may be reported as particles. The noise count rate is determined by operating the DAPC with air known to be free of particles larger than the lower sizing limit of that DAPC. Filtering the sampled air to the DAPC inlet with a filter which recording the DAPC count data over a specified time period will provide valid zero count rate data.

4.5 Particle Counting Efficiency—A suspension of airborne particles is generated into a chamber where a well-mixed suspension can be produced. Samples are withdrawn from a single location within the chamber by the DAPC and by a reference particle counting device (RAPC). The sample handling systems for both units should be designed so particle losses during transit from the chamber to either unit are either identical or negligible. The reference device should be one known to have 100 % counting efficiency for the smallest particles in the suspension. Counting efficiency is defined as the ratio of the DAPC particle count to the reference device

count for the particle size ranges of concern. Primary counting efficiency data are procured using monodisperse spherical calibration particles. Counting efficiency data for specific materials can also be procured with a suspension of polydisperse particles that may contain particles of varied materials and shapes. The counting efficiency determined for such materials may vary from the primary counting efficiency.

4.6 Particle concentration limit—If the particle concentration in the DAPC becomes excessive, then the probability of more than one particle being present in the sensing volume at any time may become significant. In that situation, several small particles simultaneously present in that volume will be reported as a single larger particle, resulting in a report of larger and fewer particles than those actually present in the suspension being measured. The particle concentration limit is determined by producing a series of aerosol suspensions carefully diluted and sampled by the DAPC. Each of the suspensions is diluted so that the concentration of each succeeding suspension is reduced by a constant factor from that of the previous suspension. The reported concentration of each suspension is recorded. At excessive concentrations, the ratio of succeeding reported concentrations will be less than the dilution ratio. This indicates that coincident particles in the sensing volume are being reported as single particles. When the concentration is low enough so that only individual particles are being counted, then the reported concentration ratio between two succeeding measurements will be nearly the same as the dilution ratio. The upper concentration where this phenomenon occurs is then accepted as the DAPC particle concentration limit.

5. Significance and Use

5.1 Much present-day technology depends upon operation of components whose performance may be degraded if particulate materials are present within their working elements. Production of these components in an environment contaminated by airborne particles may cause failure or poor performance. For pharmaceutical and medical products, regulatory agency documents specify the maximum number and size of airborne particles that may be present in the production area. An accurate measurement of the number and size of particles is required to determine the effectiveness of control methods to maintain cleanliness in that environment. A DAPC is frequently used to characterize air cleanliness. The usefulness of data from that DAPC requires reliability of the DAPC for accurate sizing and counting of the particulate materials in the air which it is sampling.

6. Interferences

6.1 Uncontrolled intake of environmental airborne particles during testing will cause errors. Since the particles used during the DAPC testing are in the size range and concentration that may be similar to those of normal airborne particles in an urban environment, it is necessary to ensure that neither normal airborne particles nor dust particles entrained from surfaces in the area are allowed to enter the test system or the DAPC under test. Operation in environments where clean air is present is required. If a Federal Standard 209E Class M4.5 (1000) or

⁸ Rimberg, D. and Thomas, J.W., "Response of an Optical Counter to Monodisperse Aerosol", *Atmospheric Environment*, Vol. 4, 1970, 680-688.

better cleanroom is not available for testing, a vertical or horizontal flow clean bench should be used.

6.2 Since the DAPC is a high sensitivity device, it may be affected by radio-frequency or electromagnetic interference. Precautions should be taken to ensure that electrical noise in the test area environment does not exceed the capabilities of the DAPC. Electronic or operational verification, such as indication of acceptable background levels, can be made to verify the noise level.

7. Apparatus

7.1 *Aerosol Neutralizer*—A bipolar ion source may be used to reduce electrostatic charge on aerosol particles in order to reduce agglomeration of airborne particles and losses by deposition on transit tubing walls. Either a radioactive source or a bipolar electric field generator can be used to generate sufficient bipolar ions to produce a nearly neutrally charged particle suspension.

7.2 *Barometer*—A calibrated barometer with accuracy of 133 Pa (1 mm Hg).

7.3 *Filter*—A cartridge filter or filter assembly unit with at least 99.97 % removal efficiency for particles equal to and larger than the DAPC lower sizing limit. Pressure drop across the filter unit should be small enough so that the air flow rate to the DAPC can be maintained as its rated level with this filter attached to the DAPC inlet.

7.4 *Flowmeter*—A gas flow measuring device with flow rate error of less than 3 % of full scale flow. Full scale flow should be no more than 150 % of the specified DAPC flow rate. The flowmeter should have a pressure drop small enough so that it does not cause an error greater than 2 % in the sampled flow rate due to that pressure drop. The flowmeter data output should report the volumetric gas flow rate at ambient pressure and temperature. If a mass flowmeter is used, then the mass flow rate should be corrected to volumetric flow rate at ambient pressure and temperature.

7.5 *Mixing Chamber*—A vessel with fittings to allow introduction of a monodisperse aerosol stream along with a dilution air stream, if required. Both inlet fittings are located in such a way that the two streams will mix thoroughly. The total volumetric flow rate of the two streams should be in the range from 150 to 200 % of the combined volumetric flow rates of the DAPC and an RAPC. Additional tubing with fittings lead to a single location within the mixing chamber to the DAPC and the RAPC are included and laid out so that the flow path configurations to the DAPC and the RAPC are similar. A filtered vent line is included with a filter of sufficient capacity so that the pressure in the mixing chamber remains in the range of ambient pressure ± 5 % when aerosol and dilution air flow into, and sample flows and vent flow out of the chamber take place.

7.6 *Monodisperse Aerosol Generator*

7.6.1 *Suspension Droplet Atomizer*—This device is used to spray a dilute water suspension of monodisperse particles. Clean compressed gas is used to generate the spray and to carry particles to the DAPC. Clean dry air is added to the spray to evaporate the water droplets, leaving the test particles. The amount of added air may also be used to modify the dry

particle concentration by dilution. Aerosol generators of this type are typically available from DAPC manufacturers.

7.6.2 *Vibrating Orifice Aerosol Generator*⁹: A vibrating orifice aerosol generator¹⁰ can produce uniform droplets with diameter between 20 μ m and 400 μ m at a controlled rate. By spraying a liquid with or without dissolved materials present in that liquid, the generator can create solid or liquid particles with good uniformity of size, shape, density, and surface characteristics.

7.7 *Oscilloscope*—An oscilloscope is used to view the voltage pulses generated by the DAPC. Indication of pulse amplitude levels can be seen and observation of the pulse pattern aids in maintaining the particle inlet rate at a level where coincidence in the sensing volume does not occur.

7.8 *Pulse Height Analyzer*—A pulse height analyzer (PHA) collects and analyzes the distribution of voltage pulses from monodisperse particles. The required pulse voltage range, rise time, duration, and time between pulses should be procured from the DAPC vendor. The PHA should display data in at least 64 channels with resolution of at least 1 % of the average voltage measured. If the DAPC electronic system includes a PHA which meets these criteria, then it can be used.

7.9 *Reference Particle Counter*—A reference particle counter (RAPC) is a DAPC whose counting efficiency has been verified to be 100 ± 5 % at the lower sizing limit of the DAPC under test. The RAPC sizing resolution should be no greater than 5 % at the lower sizing limit of the DAPC under test. A condensation nucleus counter (CNC) can be used as an RAPC with an appropriate means of removing all possible liquid suspension residue particles significantly smaller than the monodisperse particles used for testing, but sufficiently large to be counted by the CNC. If a DAPC with lower sizing limit below that of the DAPC under test is used as an RAPC, then it should have been calibrated for counting efficiency with a validated RAPC.

7.10 *Temperature Measuring Device*—A calibrated thermometer or thermocouple with accuracy of 0.2° Celsius.

7.11 *Tubing*—Flexible tubing is required to connect the DAPC inlet to the test aerosol source, to a filtered air supply, or to flow measurement systems. The inside diameter of the tubing should be essentially the same as that of the DAPC inlet fitting and large enough so that flow is not restricted. The tubing material should have sufficient conductivity so that it does not build up or retain electrostatic charge such that particles will be retained upon the tubing surfaces. Materials such as polyurethane, plasticized polyvinyl chloride or other conductive polymers are suitable.

7.12 *Tubing Fittings and Connections*—Fittings, as necessary, to make leak-free connections between the inlet to the DAPC and any apparatus used for testing. If changes in tubing direction are to be used, avoid elbow fittings with small radius of curvature; these can remove some particles due to deposition as a result of centrifugal force.

⁹ Berglund, R.N., and Liu, B.Y.H., "Generation of Monodisperse Aerosol Standards", *Environmental Science and Technology*, Vol. 7, 1973, pp. 147-153.

¹⁰ A suitable source for this generator is TSI, Inc., P.O. Box 64394, St. Paul, MN 55164.

7.13 *Ultrasonic Cleaner*—An ultrasonic cleaner rated at 30 to 60 W with a one to two liter tank is recommended for dispersion and de-agglomeration of monodisperse particles. For most particle suspensions, sonication for time periods from 10 s to 5 min is adequate.

8. Reagents and Materials

8.1 Deionized or distilled water is required to disperse and dilute the monodisperse latex suspensions before aerosol generation. References to water shall be understood to mean reagent grade or cleaner level water as defined in Specification D 1193.

8.2 *Monodisperse Spherical Particles*—Suspensions of monodisperse polystyrene latex particles (PSL) are available in size ranges with median diameters from less than 0.1 μm to greater than 10 μm and with relative standard deviations less than 5 %. These particles are suspended in water with sufficient wetting agent to permit good dispersion, even after long storage at temperatures above freezing, along with sufficient bactericide to prevent growth of bacteria that may be deposited into the container from the atmosphere when it is opened for use.

8.3 *Compressed Air Supply*—Clean, compressed air will be used to prepare, transport, and dilute particle suspensions used to test the DAPC. The compressed air shall be at a line pressure of at least 170 kPa (25 lbs/in.²) above atmospheric pressure at a flow rate of 0.00236 m³/s (5 ft³/min) when reduced to atmospheric pressure. The compressed air should be final-filtered using a filter with pore size no greater than 20 % of the lower size limit of the DAPC being tested.

9. Calibration and Standardization

9.1 The DAPC under test should be clean and in good operating order. Reference the DAPC vendor’s most recent primary calibration data and standardize the operating levels of the DAPC in accordance with the vendor’s applicable field or metrology facility standardization procedure.

9.2 Where monodisperse particles are to be used for testing, select particle batches with data on median diameters and standard deviations provided by the supplier. These data should be procured using procedures and instruments whose performance has been verified as listed in 2.1.

9.3 Control the particle concentration in the aerosol dispersion to a level where coincidence errors are less than 3 % and maintain the DAPC inlet flow rate to the manufacturer’s specified level. The aerosol particle concentration may be controlled by adjusting the particle concentration in the dispersed particle suspension fed to the monodisperse aerosol generator and by adding particle-free dilution air to the aerosol as required. Refer to the DAPC manufacturer’s specifications for coincidence error, concentration limits and flow rates for the DAPC under test.

10. Procedure

10.1 *Inlet Sample Flow Rate Determination:*

10.1.1 Select a calibration flowmeter as described in 7.4, along with suitable fittings and tubing to connect the flowmeter to the DAPC inlet. The flowmeter should be connected

upstream of the DAPC inlet using as short a tubing length as possible so that the flowmeter is not affected by any pressure drop in tubing to the flow meter inlet and the DAPC pump and flow measuring system are not affected by pressure drop in tubing and fittings to the external flowmeter. Fig. 1 illustrates the recommended setup for inlet sample flow rate determination.

10.1.2 Place the DAPC under test with the calibration flowmeter connected to the DAPC inlet in an environment where the particle concentration is no greater than that for a cleanroom classified at Federal Standard 209E Class M4.5 (Class 1000). This environment will ensure that no excessive particulate contamination during this test will affect the flowmeter or the DAPC operation.

10.1.3 Record the local air temperature and barometric pressure.

10.1.4 Turn on the DAPC and allow sufficient warm-up time to obtain uniform, stable air flow through the calibration flowmeter and into the DAPC inlet.

10.1.5 Record the flow rates reported by the calibration flowmeter and by the DAPC meter. If the DAPC reports only qualitative flow (for example, acceptable or not), record that information.

10.1.6 If the calibration flowmeter indication is not within 10 % of the specified flow rate, or indicates only qualitatively that the flow rate is not correct, then adjust the flow to meet the specified flow rate or define a flow rate correction factor for the DAPC. If the DAPC flow rate cannot be changed, inspect the DAPC flow system for leaks or flow component blockage and make repairs where required. Record any modifications to DAPC components.

10.2 *Particle Sizing Accuracy Determination:*

10.2.1 *Apparatus and Materials:*

10.2.1.1 Monodisperse calibration particles as defined in 8.2. Particle sizes of selected batches shall range from the DAPC lower sizing limit to at least 50 % of the maximum size capability.

10.2.1.2 Clean diluent water as defined in 8.1. It may be necessary to clean the diluent water beyond the reagent grade cleanliness level for work with DAPCs for measurement of submicrometer sized particles. If necessary, clean the water by filtration into well cleaned containers. Use a filter with pore size no more than 20 % of the size of the smallest particles to be suspended in that water.

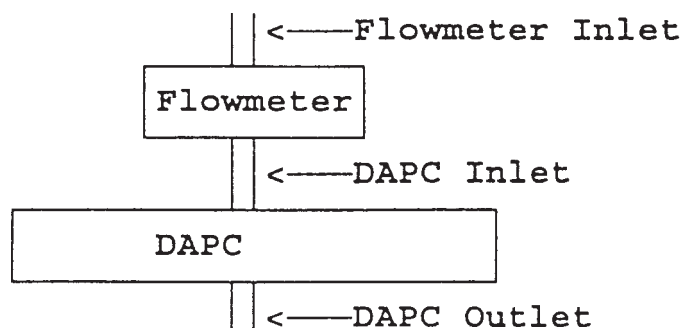


FIG. 1 Inlet Sample Flow Rate Test Setup

10.2.1.3 Aerosol generator as defined in 7.6. Either the latex particle suspension atomizing generator of 7.6.1 or the vibrating orifice aerosol generator of 7.6.2 will require compressed air as defined in 8.3 for generation and transport of test particle suspension to the DAPC under test. If the atomizer generator of 7.6.1 is used, then clean compressed air at a pressure ranging from 68 kPa to 170 kPa above atmospheric pressure (10 to 25 psig) will be required for droplet generation; clean compressed air at a pressure of approximately 250 Pa will be used for dilution and transport of the generated aerosol for both generators. If the vibrating orifice generator is used, only low-pressure clean air will be required for transport and dilution of the aerosol. An aerosol neutralizer, as defined in 7.1, may be used to reduce particle loss from the generator due to agglomeration and deposition. Use of the aerosol neutralizer is optional for determining particle sizing accuracy.

10.2.1.4 Tubing as defined in 7.10. The inside diameter of the tubing should be selected so that flow is not restricted.

10.2.1.5 Tubing fittings as defined in 7.11.

10.2.1.6 Oscilloscope as defined in 7.7.

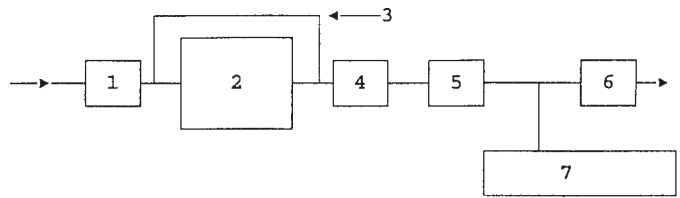
10.2.1.7 Pulse height analyzer as defined in 7.8.

NOTE 1—Response of some DAPCs may be polytonic over portions of the response range. In these portions, scattered light flux levels may increase and decrease as particle size increases. See Fig. 2. Definite particle size data cannot be defined in the voltage outputs and size ranges indicated between the dashed lines on this figure. Calibration particle sizes should be chosen to permit good definition of any such reversals.

10.2.2 Procedure:

10.2.2.1 Connect the output line of the aerosol generator to the DAPC inlet with a length of tubing no longer than 1 meter. The connection from the aerosol generator shall be oriented so that the aerosol flow to the DAPC is vertically downward to minimize preferential loss of large particle due to gravitational effects. Fig. 3 illustrates the recommended setup for determining particle sizing accuracy. Use of then aerosol neutralizer shown here is optional for this test.

10.2.2.2 Connect the oscilloscope to the DAPC output to observe the output pulses to the data processing system. If



NOTE 1—

Legend

- 1 = Compressed air cleaner and drier
- 2 = Aerosol generator
- 3 = Dilution air line (with flow control)
- 4 = Diffusion drier
- 5 = Aerosol neutralizer (optional)
- 6 = Flow balancing vent filter
- 7 = DAPC

FIG. 3 Arrangement for Particle Size Calibration Test

there is no external output pulse connector, the DAPC manufacturer can provide information as to availability and location of an internal connection for this connection.

10.2.2.3 Connect the pulse height analyzer to the DAPC electronic system directly after the last amplifier before the pulse sorting portion of the DAPC electronic system. The correct location for this connection can be obtained from the DAPC operating manual or from the DAPC manufacturer. If the DAPC has a built-in pulse height analyzer, then it can be used for this procedure.

10.2.2.4 Select the particle sizes within the DAPC dynamic range which will be used for determining particle sizing accuracy and prepare diluted liquid suspensions from the monodisperse particle suspensions as originally procured. Based on typical delivered concentrations from vendors for the monodisperse latex particle suspensions and the requirement to generate droplets with essentially all containing no more than one particle, recommended concentrations¹¹ of the diluted suspensions for spraying are shown in Table 1.

10.2.2.5 Turn on the DAPC and ensure that it is set up for standard operation. Set the DAPC data reporting system to the cumulative mode and verify that the first channel of the DAPC is set to the lower sizing limit of the DAPC.

10.2.3 Operate the aerosol generator with only dilution air flow to ensure that the flow from the aerosol generator to the DAPC is sufficient to prevent any ambient air entering the flow to the DAPC. This condition will be evident when the DAPC

¹¹ JIS B-9921 Japanese Industrial Standard-Light Scattering Automatic Particle Counter

TABLE 1 Concentration of Diluted Calibration Particle Liquids

Calibration Particle Size	Volume Concentration (approximate %)
0.1 μm	0.00001
0.2 μm	0.0001
0.3 μm	0.0003
0.5 μm	0.001
1.0 μm	0.005
2.0 μm	0.01
5.0 μm	0.1

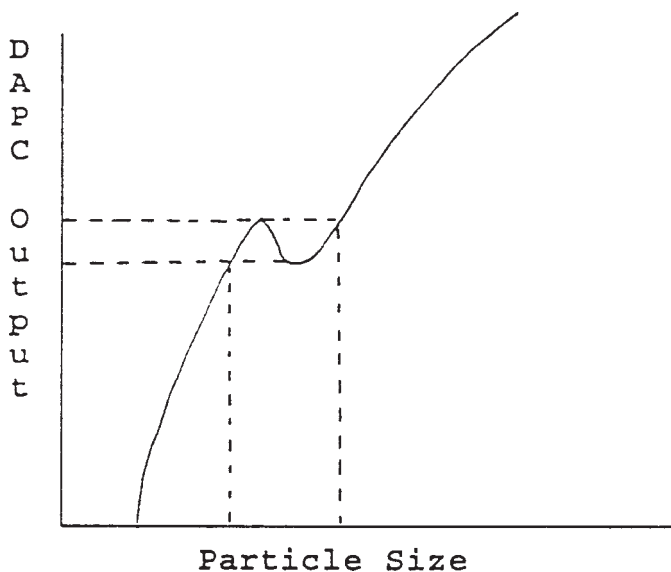


FIG. 2 Polytonic region of DAPC Response Curve

shows zero particle count data. When this condition has been verified, turn on the air flow to the droplet producing portion of the aerosol generator. Adjust that air flow to the recommended pressure for the aerosol generator being used. Adjust the dilution air flow while observing the oscilloscope screen so that individual pulses from the DAPC are being generated with sufficient spacing between pulses so that individual particle count data will be accumulated by the DAPC. The pulse counting rate shall be no greater than that which will indicate a particle concentration of 25 % of the DAPC recommended concentration limit.

10.2.4 Collect data so that at least 5000 pulses are accumulated. Determine the average value of the DAPC output pulse voltage from the pulse height analyzer for the particle size being used for this portion of the test. Either the mode or the median of the voltage pulse height distribution can be used to define the average output voltage. The mode of the pulse height distribution is commonly used to define the average voltage; use of the median voltage is an acceptable alternative that may result in a minor difference in reported average voltage. The calibration report shall identify how the average voltage is defined.

10.2.5 The mode of the voltage pulse amplitude distribution is determined by finding the voltage level at which the greatest number of pulses are produced from a batch of monodisperse particles. The median of the voltage-pulse amplitude distribution is determined by finding the voltage level at which half of the pulses are larger than that level and half are smaller than that level. An example of an idealized pulse height distribution from a batch of monosized particles near the lower sizing limit of a DAPC is shown in Fig. 4. This figure also shows noise pulses from the DAPC. Since the smallest pulses to be reported are at either the mode or the median pulse amplitude level from the test particles, the noise pulses are rarely reported, as long as the measured zero count does not exceed the specified level.

NOTE 2—When determining the reported average size for monodisperse particles larger than approximately one micrometer in diameter, a pulse height distribution will be produced that is nearly Gaussian in configuration. As particle size is decreased to approximately 0.3 μm in

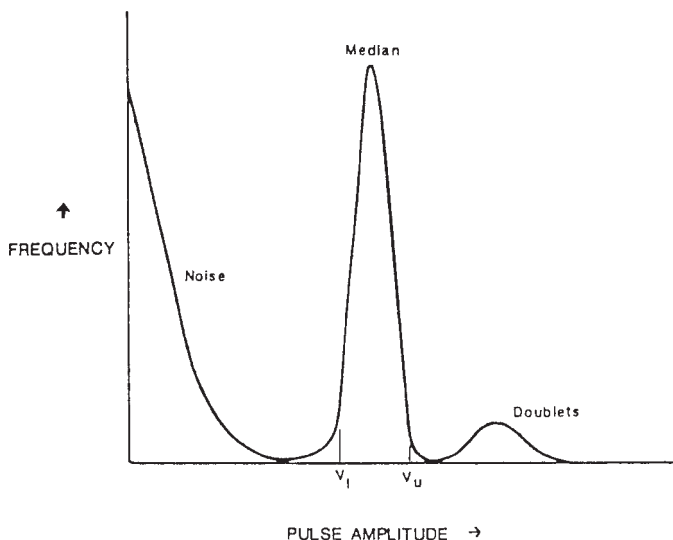


FIG. 4 Pulse Height Distribution near DAPC Lower Sizing Limit

diameter, the light scattering as a function of particle size will increase from a second power function of diameter to a sixth power function of diameter; this results in a skewed distribution. As the test particle size decreases to the lower size limit for the DAPC being tested, the amplitude of pulses generated by particles will decrease to a level where electronic and optical system noise may increase the particle pulse level by a significant amount unless the DAPC noise level is no more than an insignificant fraction of the particle signal level. For older DAPCs using comparator systems to define particle data, the voltage level setting for the DAPC lower size limit shall be at least 1.15 times the noise level voltage where one noise pulse occurs in a time period required to accumulate a defined minimum number of particle counts. Otherwise the validity of data for the DAPC lower size limit may be questionable. For DAPCs using microprocessor interpretation of pulse-height analyzer data to count and size particles, a similar limitation on the zero count rate is required. For these instruments, a batch of monosize particles at or near the lower size limit specified for that instrument is passed through the DAPC sensing volume and the PHA channel at which the peak of the pulse height distribution occurs is noted. The measured noise count rate which occurs at that PHA channel level shall not exceed the specified maximum zero count rate (see 10.3) for that instrument.

10.2.6 Repeat 10.2.3-10.2.5 for each batch of monodisperse particles selected to determine particle sizing accuracy for the DAPC. If data are collected from an external PHA, then record a tabulation of average voltages for each selected test particle size. This tabulation can be used to prepare a log-log plot of DAPC output voltage as a function of particle size. If any polytonic response, as shown in Fig. 2, is noted during the calibration, the size range(s) over which this response occurs shall be reported.

10.2.7 Compare the average pulse voltage versus particle size calibration data to the data from the most recent calibration. If the difference between the two sets of measurements is more than 15 % in voltage for the same particle size levels, then remedial measures should be taken to ensure that components performance will not continue to vary.

10.3 Zero Count Level Determination:

10.3.1 Apparatus and Materials:

10.3.1.1 Filter as defined by Item 7.3.

10.3.1.2 Tubing as defined by Item 7.10 to connect the filter outlet to the DAPC inlet. Use a short section of tubing as possible.

10.3.1.3 Tubing fittings as defined by Item 7.11.

10.3.2 Procedure:

10.3.2.1 Securely connect the filter outlet to the DAPC inlet as shown in Fig. 5.

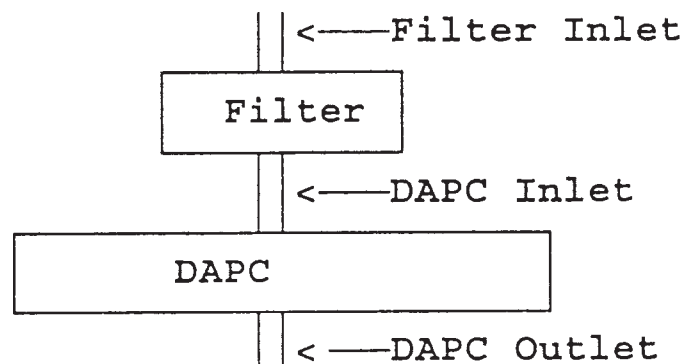


FIG. 5 Zero Count Level Test Setup

10.3.2.2 Adjust the DAPC to report particle count data in the cumulative mode.

10.3.2.3 Operate the DAPC for a maximum period of 60 min in order to remove any residual particles from the interior of the DAPC optical system. Although data are not collected during the purge time, observe the reported data accumulation rate for particles \geq the lower sizing limit to ensure that release of residual particles is no longer occurring at a significant rate.

10.3.2.4 Reset the DAPC data display to zero, measure and record total count of particles \geq the lower sizing limit for at least two ten minute periods.

10.3.2.5 Compare the data collected in 10.3.2.4 for reported number of particles \geq the lower sizing limit with the DAPC manufacturer's specified zero count rate. If the reported zero count rate exceeds the specified zero count rate by more than 50 %, remedial measures will be required.

10.4 Particle Sizing Resolution Determination:

NOTE 3—DAPC sizing resolution shall be determined at a particle size within a monotonic region of the DAPC output. The DAPC output versus particle size and particle sizing accuracy shall be defined before determining sizing resolution. Particle size(s) chosen for defining resolution shall be two to five times larger than the lower sizing limit of the DAPC so that all pulses produced by those particles have amplitude greater than the pulse amplitude for the lower sizing limit of the DAPC.

10.4.1 Apparatus and Materials:

10.4.1.1 Use the apparatus and materials described under 10.2.1.

10.4.2 Procedure:

10.4.2.1 Set up the aerosol generator and connect its output to the DAPC as shown in section 10.2. The oscilloscope and PHA should be connected to the DAPC as specified in 10.2. Select a particle size approximately twice that of the lower sizing limit of the DAPC.

10.4.2.2 Adjust the DAPC to report data in the differential mode. Based on the calibration data derived in 10.2, define the pulse amplitude mode for the particle size selected in 10.4.2.1 and for particle sizes approximately 25 % larger and smaller than that size. Set the PHA to report pulses within this range.

10.4.2.3 Generate an aerosol using the method described in 10.2.2. Adjust the dilution air flow to keep the concentration to a level approximately 25 % of the recommended maximum concentration for the DAPC under test. Observation of the oscilloscope display should then indicate discrete pulses with amplitudes nearly identical.

10.4.2.4 Set the PHA cursor at the pulse amplitude mode (the calibration voltage as defined by the particle sizing accuracy measurement of 10.2) and collect data until 1000 pulses are collected at that level. Move the cursor to the points on both sides of the calibration voltage point where the numbers of pulses have decreased to 61 % of the number at the calibration voltage. Where no PHA channel has exactly 610 counts, define a voltage for that count number by interpolating between channels spanning that value. The particle sizes for those points will be the particle sizes for particle populations one standard deviation greater than and one standard deviation smaller than the calibration particle mean size. Fig. 6, a plot of PHA data from small particles, illustrates the points used for this definition. The pulse amplitude is a function of the particle

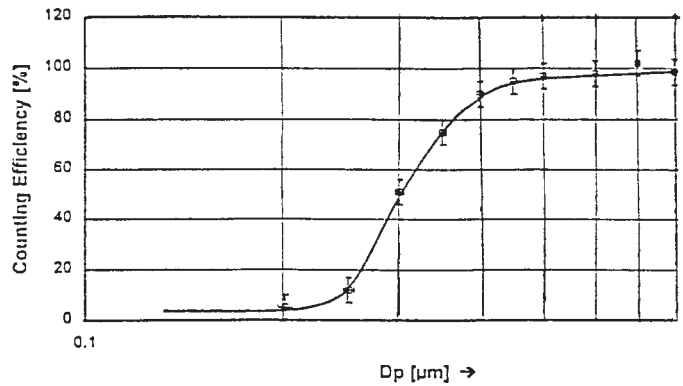


FIG. 6 Voltage Pulse Distribution for Sizing Resolution Determination

size to a power that may vary for submicrometer particles. Thus, the distribution of voltage pulse amplitude will not be a Gaussian distribution, as expected for the test particle size distribution. Fig. 6 shows how some DAPC larger noise pulses and smaller particle pulses may overlap. In addition, pulses from small suspensions water background debris and surfactant particle residue may distort the calibration particle pulse height distribution. As seen from this figure, these artifact data are not considered when determining the \pm one sigma deviation values.

10.4.2.5 Using the particle size data developed in 10.4.2.4, determine the differences between the mean size and the sizes for populations plus and minus 61 % of that at the mean size. Those differences represent one standard deviation above and below the mode for a Gaussian size distribution. The differences above and below the mean size shall agree within 5 %. Note the standard deviation reported by the standard particle supplier for the batch of monosized particles used for this test.

10.4.2.6 Determine the average of the standard deviations and square that average to convert it to a variance. If the value of either of the standard deviations differs from the average by more than 20 %, realignment is required for the DAPC optical system. Subtract the variance defined by the particle supplier's data from the variance developed by the measurements just carried out and take the square root of the difference. This will be the standard deviation of the size distribution added by the resolution limit of the DAPC. The DAPC resolution is calculated by dividing the square root value just obtained by the mean diameter of the test particles. The resolution can be expressed as *percent resolution* by multiplying the resolution by 100. If the percent resolution is greater than 10 %, remedial measures may be required to realign optics or electronic components.

10.5 Particle Counting Efficiency Determination:

NOTE 4—Counting efficiency determination can be determined by one of two procedures. A primary method¹² requires the use of a condensation nucleus counter (CNC) in combination with an aerosol size separator (for example, a differential mobility analyzer [DMA]) as a reference particle counter to ensure that the concentration reported for the batches of

¹² Peters, C., Gebhart, J., and Sehart, S. 1991 "Test of High Sensitivity Laser Particle Counters with PSL-1 Aerosols and a CNC Reference". *Journal of Aerosol Science* 22(S1):S363-S366.

monodisperse aerosol challenge does not include data from residual particles smaller than the selected size. A secondary method¹³ uses a secondary standard DAPC which has been calibrated, shown to have good sizing resolution, and is verified to have 100 % ± 5 % counting efficiency at the lower size limit of the DAPC being tested. The primary method is normally used by DAPC manufacturers and by DAPC users with access to a capable metrology laboratory with the required CNC/DMA system and trained personnel. The secondary method can be used for field measurement and for metrology laboratory applications.

10.5.1 Apparatus and Materials:

10.5.1.1 Use the apparatus and materials described in section 10.2.1.

10.5.1.2 Mixing chamber, as defined by 7.5.

10.5.1.3 Reference particle counter, as defined by 7.8.

10.5.1.4 Arrange these components as shown in Fig. 7.

10.5.2 Procedure:

10.5.2.1 Set up the apparatus as shown in Fig. 7.

10.5.2.2 Set the DAPC being tested to report particle counts in the cumulative mode and set the smallest size range channel of that DAPC to its lower sizing limit.

NOTE 5—The lines from the mixing chamber to the DAPC and the RAPC shall acquire samples from a well-mixed volume in the mixing chamber and shall be configured to minimize differential losses in transit to the DAPC and the RAPC. If the DAPC and the RAPC do not sample at the same flow rate, use an inlet fitting to each which will ensure isokinetic sampling.

10.5.2.3 Select the largest monodisperse particle size to be used for this test. These particles should be in the size range of 10 to 20 times larger than the lower sizing limit. If polystyrene latex sphere (PSL) suspensions are to be used, PSL particles no

larger than 5 µm are suggested here. Place a diluted suspension (see Table 1) of these particles in the aerosol generator.

10.5.2.4 Operate the aerosol generator and add dilution air so that the particle concentration present in the mixing chamber is no more than 25 % of the maximum recommended concentration limit of the DAPC or the RAPC, whichever is lower.

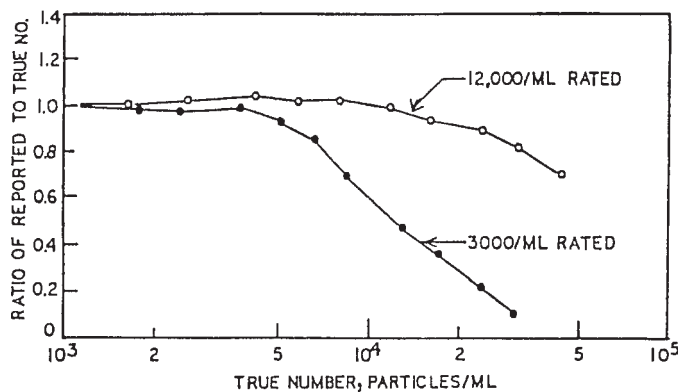
10.5.2.5 Allow five minutes for systems stabilization and record the number of particles counted by the DAPC and by the RAPC. Repeat this procedure by switching the DAPC and RAPC inlet tubes and verify that the calculated concentration data from both the DAPC and the RAPC do not differ by more than 5 %. If greater differences are seen, move the sample line inlet locations in the mixing chamber until a location is found where acceptable data are produced.

10.5.2.6 When similar data are generated by both the DAPC and the RAPC, collect samples until a total count of at least 1000 is reported by the unit with the smallest sample inlet flow rate.

10.5.2.7 Calculate the counting efficiency for the DAPC by using the definition that the counting efficiency is equal to the 100 times the ratio of the calculated concentration reported by the DAPC to that reported by the RAPC.

10.5.2.8 Repeat 10.5.2.3-10.5.2.7 with selected monodisperse PSL suspensions with sizes decreasing until a counting efficiency value of approximately 20 % is reached. For adequate definition of the counting efficiency, at least three measurements should be taken for counting efficiencies between 20 and 90 % with one point at the particle size at the DAPC lower sizing limit. At the lower sizing limit, the counting efficiency value should be 50 ± 10 %. (It will not be necessary to repeat the inlet tube switching operation of 10.5.2.4).

¹³ Wen, H.Y.1 and Kasper, G.J., 1986. "Counting Efficiencies of Six Commercial Particle Counters", Journal of Aerosol Science 17(6):947-961.



NOTE 1—

Legend

- 1 = Compressed air cleaner and drier
- 2 = Aerosol generator
- 3 = Dilution air line with flow control
- 4 = Diffusion drier
- 5 = Aerosol neutralizer
- 6 = Flow balancing vent filter
- 7 = Mixing chamber
- 8 = DAPC
- 9 = RAPC

FIG. 7 Arrangement for Determining Counting Efficiency

10.5.2.9 Plot the counting efficiency percentage as a function of particle size for the DAPC. An example of such a plot is shown in Fig. 8 with error bars assuming that the only errors are due to counting statistics.

10.6 Particle Concentration Limit Determination:

NOTE 6—The particle concentration limit for a DAPC is affected by both particle coincidence in the sensing volume and the counting/sizing data processing rate limitations of the electronic system. Coincidence probability increases directly with the sensing volume size. The particle size is assumed insignificant in comparison with the sensing volume size. Large particles passing through the sensing volume can immobilize counting circuitry until the particle is completely out of the volume. Counting rate limitations are inherent in the electronic pulse processing circuitry, although the pulse processing rate limit is frequently less restrictive than the physical coincidence limit. The rate at which particles pass through the sensing volume can be estimated by assuming a uniform particle spatial distribution, determining the number of particles per unit sensing volume as a function of concentration and stating the DAPC sampling flow rate to calculate the number of pulses per unit time. Since the particle spatial distribution is not uniform, but is random, short time spatial concentrations an order of magnitude greater than the long term concentration may exist. Therefore, the electronic system must be capable of counting a rate one order of magnitude greater than that for the average pulse rate.

10.6.1 Apparatus and Materials:

10.6.1.1 Use the apparatus and materials described in section 10.5.1. The RAPC shall be one that is known to have a particle concentration limit at least one order of magnitude greater than that specified for the DAPC being tested. The apparatus should be arranged as shown in Fig. 7.

10.6.2 Procedure:

10.6.2.1 Set the RAPC and the DAPC to report particle counts in the cumulative mode with both DAPC and RAPC reporting counts for particles \geq the lower sizing limit of the DAPC.

10.6.2.2 Select a batch of monodisperse particles that are in the size range of two to five times the lower sizing limit of the DAPC being tested and place a diluted suspension (see Table 1) of these particles in the aerosol generator.

10.6.2.3 Operate the RAPC with dilution air only entering the mixing chamber at this time.

10.6.2.4 Operate the aerosol generator, adjusting the dilution air until the RAPC indicates a concentration approximately twice the recommended maximum concentration for the DAPC.

10.6.2.5 Turn on the DAPC and record the reported particle count per unit time and sample flow rate for the reported particle concentration for both the DAPC and the RAPC.

10.6.2.6 Repeat 10.6.2.5-10.6.2.8 with the dilution air flow changed for each repetition so that the RAPC indicates concentrations that are 150, 100, 90, 80, 65, and 50 % of the recommended maximum concentration for the DAPC being tested. Record the DAPC reported concentrations at each of the RAPC concentrations. These data can be used to prepare a true versus indicated concentration plot for the DAPC, as shown in Fig. 9, where the RAPC data are taken as true concentrations. The indicated concentration should be $100 \pm 5\%$ of the true concentration when the true concentration is at or less the specified maximum recommended concentration. If this does not occur, examine the DAPC to ensure that sample flow rate and size range settings are correct. If these are correct, then it may be necessary to realign the optical system to ensure that the sensing volume and sample flow streamlines are not misaligned.

10.6.2.7 An alternate method of defining the particle concentration limit can be used if a suitable RAPC is not available. This method involves a series of accurately defined sequential dilutions of the test aerosol. Repeat 10.6.2.2 and 10.6.2.4 with the aerosol generator operating so as to produce an aerosol concentration that is significantly greater than the DAPC specified maximum concentration limit. Note the concentration reported by the DAPC.

10.6.2.8 Adjust the aerosol generator until the concentration is diluted by a factor of two. Note the concentration reported by the DAPC.

10.6.2.9 If the concentration is reduced by a factor of two, then that concentration is less than the DAPC particle concentration limit. If it is decreased by a factor of less than two, then that concentration is above the DAPC concentration limit. If the latter occurs, then repeat 10.6.2.8 until the reported concentration decreases by the same factor as the dilution ratio.

NOTE 7—If adjusting the aerosol generator concentration is not feasible, then use a commercially available aerosol dilution system to control the reduction of particle concentration to the DAPC being tested.

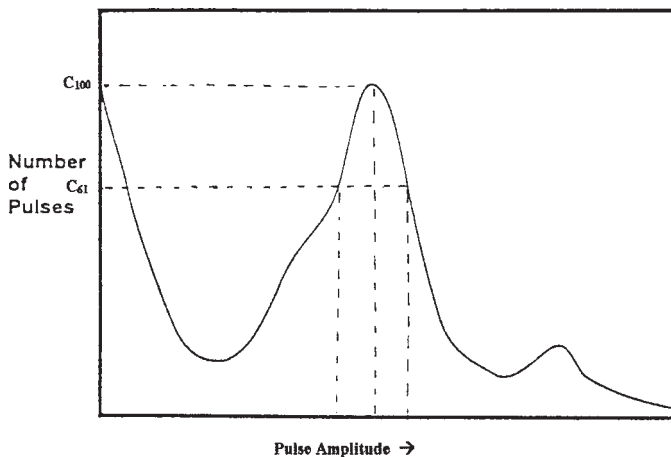


FIG. 8 DAPC Particle Counting Efficiency

11. Precision and Bias

11.1 No statements are made about the precision or bias of this practice since results state whether there is conformance for success, as specified in the procedure. Operational and

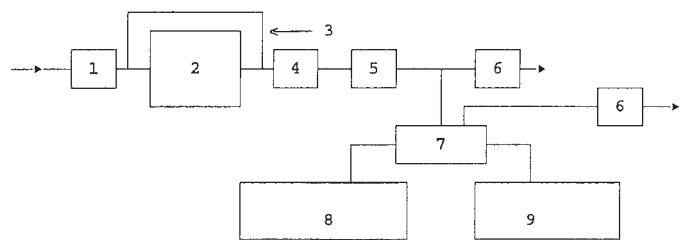


FIG. 9 Indicated Concentration versus True Concentration

design variations for different instruments will result in differing results when measuring normal aerosols. Thus, testing with contaminant aerosols different in physical properties from the calibration material can lead to different results when using DAPCs that are not identical in optical design.

12. Keywords

12.1 calibration; particle counting; particle size resolution; particle sizing accuracy; sample flow rate; zero count rate

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