Designation: F 50 – 92 (Reapproved 2001)$^{\epsilon 1}$

Standard Practice for Continuous Sizing and Counting of Airborne Particles in Dust-Controlled Areas and Clean Rooms Using Instruments Capable of Detecting Single Sub-Micrometre and Larger Particles[1]

This standard is issued under the fixed designation F 50; 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.

ϵ^1 NOTE—Keywords were added editorially in July 2001.

1. Scope

1.1 This practice covers the determination of the particle concentration, by number, and the size distribution of airborne particles in dust-controlled areas and clean rooms, for particles in the size range of approximately 0.01 to 5.0 µm. Particle concentrations not exceeding 3.5×10^6 particles/m^3(100 000/ft^3) are covered for all particles equal to and larger than the minimum size measured.

1.2 This practice uses an airborne single particle counting device (SPC) whose operation is based on measuring the signal produced by an individual particle passing through the sensing zone. The signal must be directly or indirectly related to particle size.

NOTE 1—The SPC type is not specified here. The SPC can be a conventional optical particle counter (OPC), an aerodynamic particle sizer, a condensation nucleus counter (CNC) operating in conjunction with a diffusion battery or differential mobility analyzer, or any other device capable of counting and sizing single particles in the size range of concern and of sampling in a cleanroom environment.

1.3 Individuals performing tests in accordance with this practice shall be trained in use of the SPC and shall understand its operation.

1.4 Since the concentration and the particle size distribution of airborne particles are subject to continuous variations, the choice of sampling probe configuration, locations and sampling times will affect sampling results. Further, the differences in the physical measurement, electronic and sample handling systems between the various SPCs and the differences in physical properties of the various particles being measured can contribute to variations in the test results. These differences should be recognized and minimized by using a standard method of primary calibration and by minimizing variability of sample acquisition procedures.

1.5 Sample acquisition procedures and equipment may be selected for specific applications based on varying cleanroom class levels. Firm requirements for these selections are beyond the scope of this practice; however, sampling practices shall be stated that take into account potential spatial and statistical variations of suspended particles in clean rooms.

NOTE 2—General references to cleanroom classifications follow Federal Standard 209, latest revision. Where airborne particles are to be characterized in dust-controlled areas that do not meet these classifications, the latest revision of the pertinent specification for these areas shall be used.

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.* For specific hazards statements, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres[2]

F 328 Practice for Calibration of an Airborne Particle Counter using Monodisperse Spherical Particles[3]

F 649 Practice for Secondary Calibration of Airborne Particle Counter using Comparison Procedures[3]

F 658 Practice for Calibration of a Liquid-Borne Particle Counter using an Optical System Based Upon Light Extinction[3]

2.2 *U.S. Federal Standard:*

Federal Standard No. 209D, Clean Room and Work Station

[1] This practice is under the jurisdiction of ASTM Committee E21 on Space Simulation and Applications of Space Technology and is the direct responsibility of Subcommittee E21.05 on Contamination.

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[2] *Annual Book of ASTM Standards*, Vol 11.03.

[3] *Annual Book of ASTM Standards*, Vol 15.03.

Requirements, Controlled Environment[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *dust-controlled area*—a clean room or clean work space in which airborne and deposited particulate contamination levels, or both, are controlled on the basis of a documented standard such as Federal Standard 209D.

3.1.2 *dynamic range*—the particle size range, expressed as a multiple of the minimum measured size, over which the SPC can measure particles with size resolution of 10 % or less.

3.1.3 *particle concentration*—the number of individual particles per unit volume of ambient temperature and pressure air, particles/m^3 or particles/ft^3.

3.1.4 *particle size*—equivalent diameter of a particle detected by an SPC.

3.1.4.1 *Discussion*—The equivalent diameter is the diameter of a reference sphere of known size and physical characteristics (for example, refractive index when using an OPC; density when using an aerodynamic particle sizer; etc) and generating the same response in the SPC sensing zone as the particle being measured. Spherical particles are used for calibration of the SPCs considered here. The SPC response is related to the size, shape, orientation and physical properties of the particle passing through the SPC sensing zone. If an optical particle counter is used, the geometry of the optical system, as well as the spectral distribution of the illuminating light influences the reported particle size. If a condensation nucleus counter with a size-fractionation device is used, the SPC operating parameters and the particle properties that affect the nucleation efficiency and, for example, the diffusion coefficient, will influence reported data. The SPC instruction manual should make the user aware of the effects of such factors on the indicated particle size data.

3.1.5 *primary calibration*—calibration with standard reference particles for particle size and (optionally) concentration. Initially carried out by the SPC manufacturer.

3.1.6 *resolution*—the capability of the SPC to differentiate between particles with small difference in size.

3.1.6.1 *Discussion*—It can be quantified as the ratio of the square root of the difference between the measured and actual variances of a monosized particle size distribution to the mean diameter of those monosize particles, using procedures as shown in Practice F 658.

3.1.7 *standardization*—secondary calibration of electronic system voltage and signal response threshold levels using the reference system built into the SPC.

3.1.7.1 *Discussion*—The SPC should be capable of carrying out this procedure with a simple, rapid manual operation or by internal timed or microprocessor controlled components.

3.2 For definitions of other terms used in this practice, see Terminology D 1356 and Federal Standard 209D.

4. Summary of Practice

4.1 Satisfactory primary calibration within the manufacturer's recommended time period and routine standardization should be verified as a first step.

4.2 A sample acquisition program is established on the basis of the cleanliness level that is to be verified or monitored. This program will include sample point identification, sample size definitions and sampling frequency, specification of the sampler inlet and sample transport system, definition of the particle size ranges to be measured, and any other parameters of concern in the dust-controlled area or clean room.

4.3 Air samples are passed through the SPC and the particle content of each sample is defined by the SPC. Particles contained in the sampled air pass through the sensing zone of the SPC. Each particle produces a signal that can be related to particle size. An electronic system sorts and counts the pulses, registering the number of particles of various sizes that have passed through the sensing zone during passage of a known gas volume. The concentration and particle size data can be displayed, printed or otherwise processed, locally or remotely.

5. Significance and Use

5.1 The primary purpose of this practice is to describe a procedure for collecting near real-time data on airborne particle concentration and size distribution in clean areas as indicated by single particle counting techniques. Implementation of some government and industry specifications requires acquisition of particle size and concentration data using an SPC.

5.2 The processing requirements of many products manufactured in a clean room involves environmental cleanliness levels so low that a single particle counter with capability for detecting very small particles is required to characterize clean room air. Real-time information on concentration of airborne particles in size ranges from less than 0.1 µm to 5 µm and greater can be obtained only with an SPC. Definition of particles larger than approximately 0.05 µm may be carried out with direct measurement of light scattering from individual particles; other techniques may be required for smaller particles, such as preliminary growth by condensation before particle measurement.

5.3 Particle size data are referenced to the particle system used to calibrate the SPC. Differences in detection, electronic and sample handling systems among the various SPCs may contribute to differences in particle characterization. Care must be exercised in attempting to compare data from particles that vary significantly in composition or shape from the calibration base material. Variations may also occur between instruments using similar particle sensing systems with different operating parameters. These effects should be recognized and minimized by using standard methods for SPC calibration and operation.

5.4 In applying this practice, the fundamental assumption is made that the particles in the sample passing through the SPC are representative of the particles in the entire dust-controlled area being analyzed. Care is required that good sampling procedures are used and that no artifacts are produced at any point in the sample handling and analysis process; these precautions are necessary both in verification and in operation of the SPC.

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

6. Interferences

6.1 Since the SPC is typically a high sensitivity device, it's response may be affected by internally or externally generated noise. The SPC should not be operated at a sensitivity level so high that internal noise produces more than 5 % of the data signals.

6.2 Precautions should also be taken to ensure that the test area environment does not exceed the radio frequency or electromagnetic interference capabilities of the SPC.

6.3 Operation at acceptably low levels of internal noise can be verified by drawing a sample into the SPC through a filter or other gas cleaning device that will positively remove at least 99.97 % of all particles of size equal to and greater than that which the SPC will measure. After a short stabilization period, any signals reported by the SPC can be assumed to arise from internal or external noise sources.

7. Apparatus

7.1 *SPC*—The apparatus shall consist of a SPC, selected on the basis of its ability to count and size single particles in the required size range. The SPC shall include a sample air flow system, a particle characterization system, and a data processing system. The minimum measurable particle size shall be selected from the clean area definition stated in Table I of Federal Standard 209D, or from a different specification of clean-area airborne particle concentration at a stated minimum particle size. For classification levels based on measurement of particles larger than 0.05 µm, an optical particle counter (OPC), an aerodynamic particle sizer or an equivalent SPC can be used. For classification levels based on particles less than 0.05 µm, a CNC in combination with a diffusion battery, a differential mobility analyzer or an equivalent SPC can be used.

7.1.1 *Sample Air Flow System*, consists of an intake tube, the particle sensing/measurement chamber, an air flow metering or control system, and an exhaust system. No abrupt transitions in dimension should occur within the air flow system. The inlet tube should consist of a sharp-edged inlet nozzle connected to a tube that will transport the sample air to the particle characterization system. The sample inlet nozzle should have a cross-sectional area equivalent to that of a circle of diameter at least 2 mm. The nozzle can be attached to a transit tube with dimensions so that residence time in the tube will not exceed 10 s. Sample tubes should be configured so that the flow Reynolds number is maintained in the range 5 000 to 25 000. For particles in the size range 0.1 µm to ≈ 2 µm in diameter and a SPC flow rate of 0.028 m^3/min (1 ft^3/min), a transit tube up to 30 m long can be used. For particles in the size range ≈ 2 µm to 10 µm, a maximum transit tube length of 3 m can be used. If a flexible transit tube is to be used, then no radius of curvature below 15 cm shall be used.

7.1.2 *Particle Sensing/Measurement Chamber*—Defined by the nature of the SPC that is used. It should be verified that minimum recirculation and recounting of particles occurs in that chamber. If the particle characterization system includes any particle manipulation (for example, diffusion battery or nucleation chamber, etc) before particle sensing occurs, then the SPC element that manipulates the particles shall not result in significant particle number change during that process.

7.1.3 *Air Flow Metering of Control System*, shall be located after the particle sensing/measurement chamber so as to minimize particle losses or artifact generation before measurement occurs.

7.1.4 *Exhaust System*, may consist of either a built-in vacuum source or an external vacuum supply. If the built-in vacuum source is used, then the exhaust stream from that source shall be suitably filtered so that particles sampled by and internally generated by the SPC, or both, are not returned to the dust-controlled area.

7.2 *Particle Characterization System*, shall be capable of both detecting and sizing the particles that are sampled by the SPC. The characterization system particle sizing resolution, expressed as a percentage, shall not exceed 10 % over the operating dynamic range. The SPC specifications shall include information as to the maximum particle concentration that can be measured before coincidence error > 10 % of the indicated particle count, occurs in the detection process. The specifications shall also define the pulse rate where the data processing system becomes saturated and can no longer produce accurate pulse size and frequency information.

NOTE 3—Dynamic range for SPCs will frequently vary with particle size sensitivity. For an SPC operating solely in the size range < 1 µm, a dynamic range of 20 to 1 is typical. For an SPC used for particle measurement > 1 µm, a dynamic range of 20 to 40 is typical. The dynamic range limitations occur as a consequence of both typical particle size distributions in clean areas and of data processing system gain limitations.

7.3 *Particle Data Processing System*, shall include components for counting and sizing data signals from particles observed by the SPC, a means of converting data signal level to particle size information, sufficient data processing capability to relate particle count and air flow data to particle concentration information, and internal monitoring capability to verify that critical SPC components are operating correctly. Data shall be available as front-panel display, convenient on-board hard-copy format or as signals that can be transmitted to a remote data reception device in a format that will allow either direct storage or further processing. The particle data processing system shall also include the necessary components to carry out standardization of the SPC. The standardization may be done either manually or by internal SPC control.

8. Hazards

8.1 Some SPCs use laser illumination devices. These are normally contained within a safety-interlocked SPC cabinet. If the SPC cabinet safety interlock has been bypassed for any reason, the operator should make sure that no personnel are exposed to the direct laser beam.

8.2 Some SPC components may operate at hazardous electrical voltage levels. Care is required to make sure that personnel are protected from such potential hazards.

8.3 If the SPC is used to measure particle content of toxic, flammable or otherwise hazardous gases, the SPC exhaust must be vented safely, as specified by the Material Safety Data Sheet document for the gas.

9. Sampling

9.1 Sample collection considerations are based on acquisition of sufficient sample so that adequate data are procured. If the objective of sampling is to permit definition of dust-controlled area characteristics in terms of anticipated cleanliness level, then sufficient data should be procured for a sample so that statistical requirements for defining cleanliness can be satisfied. Further details are provided in Section 12 for these requirements. For routine monitoring, statistically valid data may not be required. However, sufficient data should be collected so that the operator may be reasonably confident that a predetermined maximum particle concentration at the SPC minimum size level is never exceeded.

9.2 *Cleanroom Class Level Verification*—The locations and minimum number of sampling points shall be established in accordance with the requirements of Federal Standard 209D, Section 5.1.3. A sample point grid will be established within the dust-controlled area. The minimum number of sample locations stated in Federal Standard 209D, Sections 5.1.3.1 and 5.1.3.2 will be measured. A greater number of locations can also be used. Air sample volumes shall be at least those stated in Table II, Federal Standard 209D. These volumes are defined on the bases of both the clean room class and the particle size(s) being measured for that determination. When the dust-controlled area airborne-particle cleanliness-verification process is carried out, the SPC shall be located in that area, preferably at the sample point location. If process tools or equipment are located so that the SPC cannot be placed at the desired sample point location, a sample transport line may be used. Two or more samples shall be taken at each location and airborne particle concentration data from at least two locations shall be used to verify the class level for each dust-controlled area. If exception to these values can be justified, then a written report to that effect shall be included with the report of the clean room class-level verification.

9.3 *Clean Room Monitoring*—The locations and the number of sampling points shall be established on the basis of the activity that will be carried out in the dust-controlled area. Sampling can be carried out at locations expected to be representative of conditions within the overall dust-controlled area. Alternately, sampling can be carried out at locations that may be critical to production in terms of potential particle source generation or reception. Continuous sampling or a timed sample collection routine can be used for clean area monitoring. The sample inlet nozzle should be isokinetic, not necessarily for optimum particle collection efficiency, but so that point sources of airborne contamination can be identified more easily. A number of preselected sample point locations can be monitored by using either multiplexed SPC sensors to a single data center or by multiplexing air flow lines to a single SPC. The former permits monitoring several locations simultaneously, while the latter allows use of a single SPC for monitoring several locations sequentially. In the latter system air sample transport considerations may limit SPC operation to measurement of particles in the size range below 1 µm.

10. Calibration and Standardization

10.1 There shall be a record of primary calibration showing that the SPC has been calibrated using a standard procedure, such as Practices F 328 or F 649, and particles of known size. Make sure that the time since the last calibration is within the manufacturer's recommended time interval. The SPC shall be calibrated for particle size response and for sample volume flow rate. Particle size response can be recorded as hard copy or can be stored within suitable SPC electronic circuitry.

10.2 The SPC background-noise-count rate shall be recorded at the time of primary calibration and shall be verified if any anomalous data are recorded or other problems occur.

11. Procedure

11.1 Verify that the calibration and background noise records are current. If not, then calibrate the SPC and determine background-noise-count rates. Calibration may require that trained metrology personnel carry out the necessary work. Background noise measurements can be carried out in the dust-controlled areas to be characterized. Place a filter on the SPC inlet that will remove at least 99.97 % of particles equal to and larger than the minimum size detected by the SPC. Turn on the sample inlet flow and adjust (if required) for correct flow rate. Record the particle count rate.

11.2 Establish and record the sample point locations for the dust-controlled area in accordance with 9.2 and 9.3. If sample transport in tubing is required, place the tubing so that it will not be subject to vibration or motion during any sample acquisition procedures.

11.3 Define the sampling schedule for the dust-controlled area in terms of number and location of sample points, sample volume, sample frequency, and sample size. The minimum number of sample points and replicate measurements will be determined for area verification in accordance with Federal Standard 209D, Sections 5.1.3.1 and 5.1.3.2. The number and location of sample points for monitoring will be determined in accordance with 9.3. For area verification, the sample frequency and sample size will be scheduled so as to ensure acquisition of statistically acceptable data. The primary requirement here is that sufficient data be procured so that the dust-controlled area air cleanliness can be stated with assurance. For very clean areas, the concentration of airborne particles will be very low, so that large samples may be required. In addition, the spatial distribution of particles in the air is random in nature, so that sample-to-sample variability must be considered in defining the sample size so as to minimize effects of such variability. The SPC sample flow rate must also be considered in defining sample volume; if sample flow rates are very low, then the time required for an acceptable sample may be so long that clean room conditions may change appreciably during and between samples.

11.4 Operate the SPC in accordance with the sampling schedule defined in 11.3. Particle data shall be recorded for each sample point measurement. The data shall include particle count information for each size range within the SPC dynamic range and data processing system capability.

12. Calculations

12.1 For verification of dust-controlled area airborne-particle concentration, air samples are taken at numerous locations throughout the area. As shown in Federal Standard 209D, one or more samples are taken at each of the locations (*L*). At each location a sample average *A* is calculated. If only one sample is taken, the reading for that one sample is used. The mean of the sample averages for all the locations is determined and used as the best estimate of the true mean concentration in the area. A standard deviation, SD, of these averages is then calculated and then a standard error, SE, is calculated. The standard error is equal to the standard deviation divided by the square root of the number of locations, $SE = SD/L^{1/2}$. Upper confidence limits on the true mean concentrations, are determined as the mean of the sample averages plus the product of *k* and *SE*, where *k* is obtained from tables of the Student's *t* distribution. The 95th percentile upper confidence limit would be obtained by adding the product of specific values of *k* and the standard error to the mean concentration, with the value of *k* determined by the number of sample locations as shown in Table 1. For many more than 15 sample locations, *k* becomes 1.65. If the number of particle count data is less than 20, then confidence intervals may be used from Table 2, as shown in 12.2.

12.2 For monitoring dust-controlled area air cleanliness, the measurement data may be used to calculate anticipated mean levels with an appropriate confidence level, much as in 12.1 when more than one reading is used. Data may also be observed simply to indicate trends in particle concentration without defining any quantitative concentration levels. If single readings are used and if the count level is in the range of 50 or more, then the upper 95th percentile confidence limit on the true count can be estimated as the count plus 1.65 times the square root of the count (an estimate of the count standard deviation). If single readings with 50 or fewer counts are used, then the upper 95th percentile confidence limit on the true count can be obtained from Table 2.[5] It is assumed that the true mean is constant during the measurement. The values are obtained by finding the Poisson distribution true mean for which the indicated count is at the 95th percentile value. If multiple readings are used and they have only a few counts, then either Table 2 or the Student's *t* analytical procedure shown in 12.1 can be used to approximate the 95th percentile upper confidence level.

TABLE 1 95th Percentile Upper Confidence Limit Factor, *k*

Number of Locations	UCL Factor *k*
2	6.31
3	2.92
4	2.35
5	2.13
6	2.02
7	1.94
8	1.90
9	1.86
10	1.83
11	1.81
12	1.80
13	1.78
14	1.77
15	1.76
16	1.75

TABLE 2 Upper 95th Percentile Limit for True Mean of Poisson Distribution

Observed Number	Upper Limit	Observed Number	Upper Limit
0	3.00	14	21.89
1	4.74	15	23.10
2	6.30	16	24.30
3	7.75	17	25.50
4	9.15	18	26.69
5	10.54	19	27.88
6	11.84	20	29.06
7	13.15	25	34.92
8	14.43	30	40.69
9	15.71	35	46.40
10	16.96	40	52.07
11	18.21	45	57.69
12	19.44	50	63.29
13	20.67		

13. Report

13.1 Report the following information, as specified:

13.1.1 Clean room or dustfree area identification and location,

13.1.2 Dust-free area airflow type, for example, "unidirectional," "nonunidirectional," "mixed",

13.1.3 SPC identification and calibration status,

13.1.4 SPC background noise count data,

13.1.5 Date and time when SPC use for verification begins and ends,

13.1.6 Clean room or dustfree area status as "as-built," "at-rest," "operational" or "other",

13.1.7 Dust-free-area air-cleanliness-classification target level,

13.1.8 Area classification verification or area monitoring,

13.1.9 SPC particle size range(s) being measured,

13.1.10 SPC inlet sampling flow and sensor measured sample flow,

13.1.11 Location of sampling points,

13.1.12 Sampling schedule for verification or monitoring protocol,

13.1.13 Raw data for each sample point, and

13.1.14 Particle distribution used in data reduction.

14. Precision and Bias

14.1 The precision of data from this practice will vary with the conditions under which the data are acquired. If the practice is used in dustfree areas where fewer than 10 data points are obtained in any single measurement, then the operator may either define a mean value for the cleanliness of the dustfree

[5] Garwood, F., "Fiducial Limits for the Poisson Distribution," Biometrika, United Kingdom, Vol 28, 1936, pp. 437–442.

area with a preselected confidence limit or the operator may state the maximum particle concentration in a specific number of measurements of defined sample volume.

14.2 The precision and bias of particle data obtained when using this practice is equivalent to those obtained with Practices F 649 or F 328.

15. Keywords

15.1 air sampling; airborne particles; particle counting; particle size distribution; particles