



# Standard Practice for Calibration of a Liquid-Borne Particle Counter Using an Optical System Based Upon Light Extinction<sup>1</sup>

This standard is issued under the fixed designation F 658; 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 ( $\epsilon$ ) 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 liquid-borne particle counter (LPC) which uses an optical system based upon light extinction measurement. This practice is directed towards determination of accuracy and resolution of the LPC for characterizing the size and number of particles, which have been passed into the sample inlet of the LPC. Consideration of inlet sampling efficiency is not part of this practice.

1.2 The procedures covered in this practice include those to measure sample volume and flow rate, zero count level, particle sizing and counting accuracy, particle sizing resolution, particle counting efficiency, and particle concentration limit.

1.3 The particle size parameter reported in this practice is the equivalent optical diameter based on projected area of calibration particles with known physical properties dispersed in liquid. The manufacturer normally specifies the minimum diameter that can be reported by an LPC; the dynamic range of the LPC being used determines the maximum diameter that can be reported for a single sample. Typical minimum reported diameters are approximately 2  $\mu\text{m}$  and a typical dynamic range specification will be approximately from 50 to 1.

1.4 The counting rate capability of the LPC is limited by temporal coincidence of particles in the sensing volume of the LPC and by the saturation level or 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 LPC optically defined sensing zone at any time. The coincidence limit is a statistical function of particle concentration in the sample and the sensing zone volume when particle size is insignificant in comparison to the sensing volume dimensions<sup>2</sup>. 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<sup>3</sup>. The

saturation level rate of the electronic counting circuitry shall be specified by the manufacturer and is normally greater than the LPC recommended maximum counting rate for the particle concentrations 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 an LPC unless significant changes have occurred in operation of the LPC or major component repairs or replacements have been made. The LPC shall then be taken to a suitable metrology facility for complete calibration. Normal routine field calibration may determine sample flow rate, zero count level, and particle sizing accuracy. The specific LPC functions to be calibrated shall be determined on the basis of agreement between the purchaser and the user. The maximum time interval between calibrations shall be determined by agreement between the purchaser and the user, but shall not exceed twelve months, unless LPC stability for longer periods is verified by measurements in accordance with this practice.

1.6 *This standard may involve hazardous materials, operation, and equipment. 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 Water<sup>4</sup>
- D 3195 Practice for Rotameter Calibration<sup>5</sup>
- E 20 Practice for Particle Size Analysis of Particulate Substances in the Range of 0.2 to 75 Micrometres by Optical Microscopy<sup>6</sup>

### 2.2 Other Documents:

- ANSI/NCSL Z540-1-1994 Laboratories and Measuring and Test Equipment—General Requirements<sup>7</sup>
- ISO 11171 Hydraulic Fluid Power—Calibration of Liquid Automatic Particle Counters<sup>7</sup>
- ANSI B93.20M-1972 Fluid Sample Containers—Qualifying and Cleaning Methods<sup>7</sup>

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee E-29 on Particle and Spray Characterization and is the direct responsibility of Subcommittee E29.02 on Non-Sieving Methods.

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

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

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 11.03.

<sup>6</sup> Discontinued; see 1994 *Annual Book of ASTM Standards*, Vol

<sup>7</sup> Available from the American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

### 3. Terminology

#### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *calibration*—measurement, reporting, and adjustment if required, of an instrument in comparison with a certified standard material or instrument of known adequate accuracy. Primary calibration is carried out with a standard material with characteristics that are directly traceable to a recognized standards agency. Secondary calibration is carried out using a method recognized by a voluntary standards-producing agency, even if a reference material rather than a standard material is used for the calibration.

3.1.2 *calibration particles*—two types of calibration particles are used to calibrate the LPC. The size or size distribution of the calibration particles shall be determined by procedures traceable to a recognized standards laboratory. Monodisperse, isotropic particles of known dimension and physical properties are used directly for size calibration. These are available in diameters covering the operating range of most LPCs. When size calibration is carried out with polydisperse particles, the particle size distribution is specified over the size range of concern, the mass concentration of the polydisperse particles in the calibration suspension is known, and the reported particle population data are used to establish a calibration. The calibration particles are described further in 4.2, 10.3, and 10.4

3.1.3 *calibration suspension*—a suspension of calibration particles with a known particle size distribution dispersed in a clean liquid. The mass concentration of the particles may be specified and the particle concentration in specific size ranges can be determined from these data.

3.1.4 *coincidence*—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.

3.1.5 *concentration*—number or mass of particles within a specific size range or equal to and larger than a specific particle size per unit volume of liquid at ambient temperature and pressure.

3.1.6 *concentration limit*—the upper concentration by number per unit volume of liquid specified by the LPC manufacturer where the coincidence error is below 10 %. A maximum concentration limit producing an error less than 10 % may be chosen, as required.

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

3.1.8 *dilution ratio*—when preparing particle suspensions to define the particle concentration limit (see 4.6 and 10.7), the dilution ratio is the ratio of the volume of the undiluted suspension plus particle-free diluent to the volume of the undiluted suspension.

3.1.9 *dynamic range*—the particle size range in which the LPC produces particle size data with both a lower and a upper size boundary. The range may be expressed as a particle size ratio, when the lower size is known. When the LPC is calibrated with monodisperse calibration particles, the typical lower size sensitivity of an LPC is 2  $\mu\text{m}$ ; the largest particle size typically reported is approximately 125  $\mu\text{m}$ . When the LPC is calibrated with a polydisperse calibration suspension, the

typical lower size sensitivity of the LPC is approximately 2.5  $\mu\text{m}$  and the largest size reported is approximately 50  $\mu\text{m}$ . The difference in size ranges for monodisperse and polydisperse particle calibration results from the differences in physical properties for the two particle types. The effect on the dynamic range limitation of the limited large particle concentration for the polydisperse material is discussed in 8.2.

3.1.10 *inlet flow*—the sample flow that enters the LPC through the flow inlet. Flow rate is expressed as volume per unit time, at ambient temperature and pressure.

3.1.11 *lower sizing limit*—the smallest particle size at which the LPC is capable of measuring with counting efficiency of 50  $\pm$  10 %.

3.1.12 *monodisperse*—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 2  $\mu\text{m}$  to greater than 80  $\mu\text{m}$ .

3.1.13 *particle size*—for calibration, particle size is either the modal diameter of the monodisperse calibration particle suspension used for each size threshold definition or it is the size associated with a specified cumulative particle population when a polydisperse particle suspension is used. For application purposes, particle size is the diameter of a reference particle with known properties, which produces the same response from the LPC as the particle being measured.

3.1.14 *pulse height analyzer (PHA)*—an electronic device for collecting and sorting electronic pulses by voltage level. The output is a histogram with 16 to 4096 levels, (referred to as “channels”). A PHA may be built into an LPC or may be connected to an LPC output test point. The PHA shall have at least 16 channels and shall be capable of defining the voltage pulse level in any channel with 95 % accuracy.

3.1.15 *relative standard deviation*—a measure of the width of a particle size distribution data histogram. 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.

3.1.16 *resolution*—a measure of the ability of an LPC to differentiate between particles of nearly the same size; also, the range of sizes, which an LPC would report for a particular particle if its size was determined repeatedly. It can be quantified as the ratio of the difference between the reported and true relative standard deviations for a measured series of monodisperse particles.

3.1.17 *sampled flow*—the fluid, which passes through the sensing volume of an LPC. 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.

3.1.18 *saturation level*—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.

3.1.19 *sensing volume*—the portion of the illuminated volume in the LPC through which the sample passes and from which absorbed light signals are collected by the LPC photodetector.

3.1.20 *zero count rate*—the maximum count indicated by an

LPC in a specified time period when the LPC is sampling liquid free of particles larger than the lower sizing limit of that LPC. This is also referred to as “false count rate” or “background noise level.”

#### 4. Summary of Practice

**4.1 Inlet Sample Volume and Flow Rate**—To report sampled particle concentration accurately, it is necessary to define the sample volume and to control the flow rate accurately. That flow rate may change if flow components in the LPC or in the liquid feeding system are affected by long-term operation or become plugged by deposition of particulate material. The LPC flow is normally defined at a specific pressure and should not be changed during measurements. A calibrated volumetric flow measurement device is required which operates with a pressure drop small enough so that the LPC flow control system is not loaded to the point where flow is degraded. If a mass flowmeter is used, correction to volumetric flow may be required when liquid of different density than that used for calibration is being measured. The flow measurement device is coupled to the LPC inlet and the LPC feeder sampling pump is operated. The flow indication on the calibration flowmeter is recorded and compared with the collected sample volume over the sample measurement time or with the LPC sample feeder flow indication. If the flow or the flow indication does 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. Refer to 10.1 for a complete description of this procedure.

**4.2 Particle Sizing Accuracy**—Although the LPC may be used to characterize particulate suspensions containing materials that vary in shape and composition, consistent response to standard materials is required. The basic calibration procedure for verifying particle-sizing accuracy of the LPC is based on determining the size channel in which calibration particles observed by the LPC are reported. There are two types of calibration materials that may be used to carry out this part of the calibration procedure. Selection of the optimum material is based on knowledge of the physical characteristics of the particle suspension to be measured so that the calibration material with the most similar characteristics is used for that application.

**4.2.1 Particle Sizing Accuracy Based on Response to Monodisperse Calibration Particles**—A suspension of monodisperse calibration particles is prepared by dispersing these particles in clean liquid. The LPC samples a portion of this suspension. Measurement is made of the modal voltage of the Gaussian pulse height distribution generated by the LPC for those particles. This process is repeated for monodisperse particle suspensions in sizes that allow definition of the modal voltages for several particle sizes within the LPC dynamic range. The suspensions are diluted with clean liquid, as required, to keep particle concentration sufficiently low so that the coincidence error is below 3%. The particle size and relative standard deviation of the calibration particles are measured before the sizing accuracy determination or the particle vendor reports diameter data produced by measurement methods traceable to a recognized national or international standards development agency on size and relative standard deviation of the calibra-

tion particles. The LPC modal pulse amplitude response to the calibration particle suspensions is recorded along with the standard deviation of the LPC pulse data. Refer to 10.3 for a complete description of this procedure.

**4.2.2 Particle Sizing Accuracy Based on Response to Polydisperse Particles With Known Particle Size Distribution**—A suspension of polydisperse calibration particles is prepared by dispersing a known weight of these particles in a known volume of clean liquid. Extreme care is required when removing a sample of polydisperse particles from the container to ensure that a truly representative sample is procured in such a way that its removal does not change the size distribution of the remaining material in the container. The liquid shall be chosen with viscosity and specific gravity sufficient to keep the largest particle of concern suspended in the liquid for a time sufficient for measurement. The particle size distribution data of these calibration particles shall be characterized by a measurement method traceable to a national or international standards development agency. A selected volume of suspension liquid is passed through the sensor LPC. That volume is selected so that there will be a sufficient number of the largest particle for which calibration data are required present in that volume of suspension. The number of particles per selected volume of suspension with sizes equal to and larger than the particle sizes chosen to define the response of the LPC is determined from the particle size distribution for the calibration particles and the weight of the dispersed calibration particles in the calibration suspension. The voltage pulse amplitudes for each of these calibration particle numbers are used to specify the LPC response as a function of particle size. Refer to 10.4 for a complete description of this procedure.

**NOTE 1**—When the LPC is calibrated by use of polydisperse particle suspensions, its response will differ from that obtained after calibration with monodisperse particles. This is due to the differences in the physical properties of the two materials. In addition, use of polydisperse calibration particles is recommended primarily for measurements which involve particle suspensions in oil systems.

**4.3 Particle Sizing Resolution**—Sizing resolution of the LPC defines its capability to differentiate between particles of nearly the same size. This parameter is determined after particle sizing accuracy measurements are made using monodisperse particles, as summarized in 4.2.1. Data obtained during determination of particle sizing accuracy can be used to determine particle-sizing resolution. A suspension of monodisperse calibration particles is generated as summarized in 4.2.1. The modal size and relative standard deviation for those particles, as specified by the supplier of the particles, shall be recorded. Particles shall be larger than the LPC lower sizing limit by a factor of at least 2. The modal pulse amplitude and relative standard deviation reported by the LPC under test are determined for the batch (or batches) of particles used; these data are converted to particle size and mode and standard deviation. The increase in the relative standard deviation as compared to the reported data is used to calculate the LPC 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. Refer to 10.5 for a complete description of this procedure.

**4.4 Zero Count Rate**—When an LPC is used for measurement of particle concentration in very clean liquids, the number of particles counted per unit time may be very low. If the LPC electronic or optical system is producing 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 LPC with liquid known to be free of particles larger than the lower sizing limit of that LPC. Filtering the liquid fed to the LPC inlet with a filter which removes at least 99.9 % of particles of that size range will provide a suitable test condition and recording the LPC count data over a specified time period will provide valid zero count rate data. Refer to 10.2 for a complete description of this procedure.

**4.5 Particle Counting Efficiency**—A suspension of either monodisperse or polydisperse particles is placed into a container where a well-mixed suspension can be maintained. Samples are withdrawn from a single location within the chamber by the LPC and by a reference particle counter (RLPC). The sample handling systems for both units should be designed so particle losses during transit from the container to both LPC and RLPC are either identical or negligible. An alternate sample feeding arrangement can be used where the LPC and the RLPC are connected in series with a short, straight tube and the same sample is fed through the LPC and the RLPC. Care is required so there are no particle losses in the connector element(s) nor is there debris in the connector tube. The RLPC should be known to have 100 % counting efficiency for the smallest particles that the LPC can measure. Feed a nearly identical quantity of suspension to the LPC and the RLPC and report the particle count data from both instruments. Counting efficiency is expressed as the ratio of the LPC count to the RLPC 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 varied materials and shapes. The counting efficiency determined for such materials may vary from the primary counting efficiency. Refer to 10.6 for a complete description of this procedure.

**4.6 Particle Concentration Limit**—If the particle concentration in the LPC becomes excessive, then the probability of more than one particle being present in the LPC sensing volume at any time may become significant. In that situation, several 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 particle suspensions carefully diluted and sampled by the LPC. 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 difference in the ratios 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 agreement is found is then the LPC maximum recommended particle concentration limit. Refer to 10.7 for a complete description of this procedure.

## 5. Significance and Use

5.1 Reliable and accurate operation of many devices requires that they be lubricated or cooled, or both, or power is transmitted by liquids. Clean liquids are used as processing materials or are produced as end product. If the liquid contains particulate materials whose size is so large or are present in concentrations sufficient to interfere with device operation or not to meet specifications for liquid cleanliness, then device performance is degraded. Accurate measurement of the number and sizes of particles in the liquid must be carried out. The same needs exist for measurement of powders and particles as well as for specifying cleanliness of various liquids. The interpretation of data from an LPC includes reliance on the integrity of the instrument for counting and sizing the particles in the liquid that is being examined.

5.2 In applying this practice, the fundamental assumption is made that the particles in the liquid sample passing through the LPC are representative of the particles in the liquid from which the sample was taken. Care is therefore required to make sure that good sampling is accomplished from a well-mixed volume of liquid and that no artifacts are introduced in the sample acquisition, storage, or handling operations. Those precautions are necessary both in testing and operation of the LPC.

## 6. Interferences

6.1 Miscellaneous debris ingested into the liquids during the test or in normal operation will cause errors. It is necessary to ensure that ingestion of miscellaneous airborne particles or particulate material generated by personnel or by equipment operation does not occur. Operation in clean environments is required. Either a vertical or a horizontal unidirectional flow clean bench should be used. The liquid sample containers and closures used in storing, mixing, diluting, transporting, and so forth, the liquid samples should be clean. Adherence to the procedures of ANSI B93.20M is recommended for ensuring clean, uncontaminated samples.

6.2 After a container of suspended particles has been prepared, it is necessary to mix the sample well to ensure that particles are well-distributed throughout the container before aliquot samples are taken for measurement. If a surfactant or other dispersant has been added to the liquid, then mixing may generate persistent gas bubbles, especially in viscous liquids. The bubbles may be approximately the same size as the particles being measured and will interfere with accurate particle size and count data. Bubbles must be removed before measuring the particles in the suspension. A combination of sonication and application of reduced pressure to the sample can be used for bubble removal.

6.3 Since the LPC is a high-sensitivity instrument, radio frequency or electromagnetic interference (RFI-EMI) may

affect it. Precautions should be taken to ensure that the test area environment does not exceed the RFI-EMI exposure capabilities of the LPC. Electronic or operational verification of this condition can be made, such as indication of an acceptable zero count level.

## 7. Apparatus

**7.1 Cleaned Sample Containers**—A supply of glass bottles, 50 to 1000-mL volume as required, with closures gasketed with polymer films or liners compatible with the liquid(s) to be used, and clean in accordance with definitions of ANSI B93.20M.

**7.2 Graduated Cylinder**—100 mL, glass, Class A, clean in accordance with definitions of ANSI B93.20M.

**7.3 Cleaned Wash Bottle**—A wash bottle of at least 500-mL capacity, filled with clean liquid. Either the liquid in which the particles are to be suspended or a miscible solvent with lower viscosity can be used. A pressurized container with a filter at its outlet is the preferable choice for the wash bottle.

**7.4 Dispersing System**—An ultrasonic cleaner rated at 3000 to 10 000 W/cm<sup>2</sup> of area with a 1 to 2-L tank is recommended for dispersion and ensuring that particles are not agglomerated.

**7.5 Flow Measuring Device**—A liquid volumetric flow measuring device with flow rate error below 5 % of full-scale flow. Full-scale flow measurement capability shall be no more than 150 % of the specified LPC flow rate. The flow-measuring device shall have a pressure drop small enough so that it does not restrict the liquid flow by more than 10 % at the specified flow rate of the LPC under test.

**7.6 Tubing**—Flexible and rigid tubing is used to connect the LPC inlet to the calibration material source or to flow measurement systems. The inside diameter of tubing should be essentially the same as that of the LPC inlet fitting and large enough so that flow is not restricted. The tubing material must be compatible with liquids to be used and should have sufficient electrical conductivity so that it does not build up or retain electrostatic charge resulting in particles being retained upon the tubing surfaces. Materials such as polyurethane, plasticized polyvinyl chloride, or conductive polymers are suitable for flexible tubing; metals such as stainless steel are suitable for rigid tubing.

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

**7.8 Reference Liquid-Borne Particle Counter (RLPC)**—A particle counter, which has been calibrated in accordance with an accepted standard procedure, using certified calibration materials. The counting efficiency of the RLPC shall have been verified to be 100 ± 5 % at the lower sizing limit of the LPC under test.

**7.9 Multichannel Analyzer (MCA)**—A voltage pulse analyzer capable of reporting or displaying the pulse height distribution produced by the LPC when particles are sized and counted. The MCA used should be capable of accurate measurement of pulses with durations in the 10 to 30- $\mu$ s time range, of reporting data in up to at least 512 amplitude levels, of

handling pulses with voltage levels from approximately 10 mV to 10 V, and with a capability of reporting data from either positive or negative voltage pulses.

## 8. Reagents and Materials

**8.1 Liquids for Particle Suspensions**—Clean dilution liquid may be selected from the following materials:

**8.1.1 Aqueous Liquids**—Water, deionized, distilled, or as selected in accordance with Specification D 1193.

**8.1.2 Nonaqueous Liquids**—Methyl alcohol, isopropyl alcohol, mineral spirits, mineral oil, or phosphate ester-based hydraulic fluid. (Use inert, nonreactive gaskets, seals, O-rings, and so forth, compatible with the specific liquid being used.)

**8.1.3 Purity of Reagents**—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee of Analytical Reagents of the American Chemical Society where such specifications are available<sup>8</sup>. Other grades may be used, provided that it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

**8.2 Aqueous Particle Suspensions:**

**8.2.1 Primary Size Calibration**—Primary calibration shall be performed with monodisperse isotropic particles with mean diameters traceable to NIST or a similar agency. Monodisperse polymer latex spheres with nominal sizes within the dynamic range of the LPC to be calibrated can be used as indicated in 4.2.1. The calibration particle supplier shall provide a certificate of analysis for each batch of material that reports that the particle size was determined using techniques with physical traceability to a national or international standards laboratory.

**NOTE 2**—Latex spheres are supplied in aqueous suspensions, which may have a limited shelf life. Shelf life depends upon several factors including temperature and possible microbial contamination of the suspension. The suspension can be used up to the expiration date stated from the manufacturer. Suspensions supplied without an expiration date shall not be used if they are more than one year old unless the size distribution and cleanliness of the suspension has been verified by microscopic examination. After long storage, agglomerates in suspension may be redispersed by sonication for a time period up to two minutes, depending on the power of the sonic source that is used. The addition of a suitable surfactant may also aid in minimizing agglomeration.

**8.2.2 Secondary Size Calibration and Precision Verification**—Secondary calibration can be carried out using primary calibration standard materials or materials characterized with an LPC calibrated with a primary calibration material. Monodisperse or polydisperse reference materials with a known size distribution are available in several size ranges. An example of the size distribution of one such polydisperse material is shown in Table 1 for NIST RM 8632, also known as ISO UFTD (ultrafine test dust). Variations in size distribution may exist among prepared batches, as shown in the allowable ranges of concentration in Table 1. However, the size distribution for each batch must be verified before shipment by the

<sup>8</sup> *Reagent Chemicals, American Chemical Society Specifications*. American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopoeia and National Formulary*, U.S. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.

**TABLE 1 ISO UFTD Particle Size Distribution, Number of Particles per Microgram**

Particle Size, $\mu\text{m}$	Cumulative Concentration, ISO UFTD Particles $\geq$ Stated Size Must Be Between:
1	70 000 and 100 000
2	19 000 and 29 000
3	8 600 and 13 000
4	5 400 and 8 100
5	3 300 and 4 500
6	1 500 and 2 500
7	660 and 1 400
8	280 and 760
9	120 and 410
10	58 and 220
11	28 and 120
12	14 and 63
13	7.4 and 34
14	4.1 and 19
15	2.3 and 11

producer. When using polydisperse calibration verification particles of this type, as seen in Table 1, the “steep” size distribution function of this material results in a relatively narrow useful size range. For a suspension of one microgram of powder per millilitre of liquid, the cumulative number of particles  $\geq 2 \mu\text{m}$  is approximately 10 000 times greater than the number of particles  $\geq 15 \mu\text{m}$ . This means that no statistically significant data can be produced for particle sizes larger than 10 to 15  $\mu\text{m}$  unless the suspension concentration is increased to the point where coincidence errors will occur due to the high concentration of the smallest particles in the suspension. For this reason, calibration for particles larger than the smallest particles by one order of magnitude requires a significant change in the calibration procedure. This change will be discussed in 10.4.

**8.2.3 Size Resolution Determination**—Monodisperse 10- $\mu\text{m}$  latex particle batches with diameters traceable to NIST or another similar agency shall be used to determine the resolution of the LPC. The relative standard deviation of these particle batches shall be less than 5 %.

### 8.3 Particle Suspensions in Nonaqueous Liquids:

#### 8.3.1 Primary Calibration:

**8.3.1.1 Calibration from 1 to 30  $\mu\text{m}$** —Calibration suspensions are available of other test dusts in clean low-viscosity hydraulic oil with specific concentration levels, as well as in the form of dry dusts. Primary calibration of an LPC using a nonaqueous suspension can be carried out using traceable monodisperse particles or traceable polydisperse particles. The NIST offers a medium test dust in hydraulic oil with a certified cumulative size distribution up to 30  $\mu\text{m}$  in diameter. This material is known as SRM 2806. Monodisperse glass and latex particles with traceable mean diameters may also be used.

**NOTE 3**—For calibration of LPCs used with oils, calibration particles shall be suspended in oil. Many nonpolar liquids attack latex particles. A method has been developed to suspend the latex spheres in nonpolar liquid containing ionic surfactant to form a thin protective coating around each particle. This prevents attack by oil, but will not produce significant

change in the particle size<sup>9</sup>. The dilution liquid in which the spheres are suspended is prepared from a concentrate of 120 g of Aerosol OT (dioctyl sulfosuccinate, sodium salt) per litre of clean oil. The concentrate is heated to about 60°C (140°F) and stirred until the Aerosol OT has dissolved. The concentrate is diluted with clean oil to a concentration of 12 g Aerosol OT per litre of oil. Some alcohols and nonaqueous fluids will dissolve latex particles. In those cases, latex particles can not be used.

**8.3.1.2 Calibration from 30-100  $\mu\text{m}$** —Primary calibration of an LPC using a nonaqueous suspension can be carried out using traceable monodisperse glass or polymer particles. Polymer latex particles in this size range do not require the procedure in Note 3.

**8.3.2 Secondary Calibration and Precision**—Reference materials such as those mentioned in 8.2.2 are suitable for dispersion in nonaqueous media for the purposes of calibration verification.

**8.3.3 Size Resolution Determination**—Monodisperse, traceable 10- $\mu\text{m}$  glass or polystyrene particles with a relative standard deviation less than 5 % can be used to determine the resolution of the LPC. (See Note 3 with regard to working with latex particles in oil.)

**8.4 Clean Liquid**—Approximately 1 L of liquid is required. The liquid must have been filtered through a 0.2- $\mu\text{m}$  pore size filter or filtered at least twice through a 0.45- $\mu\text{m}$  pore size filter. **Warning:** Appropriate precautions must be taken when handling hazardous liquids. Disposal shall be carried out in accordance with national, state, and municipal requirements.

## 9. Calibration and Standardization

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

**9.2** Where monodisperse particles are used for testing, select particle batches with data on diameters and standard deviations provided by the supplier. These data should be procured using procedures and instruments whose performance has been verified in accordance with documents as listed in Section 2.

**9.3** Control the particle concentration in the calibration suspension to a level where coincidence errors are less than 3 % and maintain the LPC sample flow rate at the manufacturer’s specified level. Adjusting the particle concentration in the suspension fed to the LPC by adding particle-free dilution liquid as required may be used to control the particle concentration. Refer to the LPC manufacturer’s specifications for coincidence error as a function of particle concentration and recommended liquid flow rates for the LPC under test.

**9.4** For all calibration procedures, three or more replicate measurements are recommended for each LPC operation being calibrated. If any calibration procedure produces data indicating that the LPC requires repair or modification, report the data collected, any remedial actions taken to modify the LPC, and the LPC performance following those actions.

<sup>9</sup> Verdegan, B. M., Thibodeau, L., and Stinson, J. A., “Using Monodisperse Latex Spheres Dispersed in Nonpolar Liquids,” *Microcontamination*, Vol 8, No. 2, 1989, pp. 35–39.