Designation: F 658 – 00a (Reapproved 2006)

Standard Practice for Calibration of a Liquid-Borne Particle Counter Using an Optical System Based Upon Light Extinction[1]

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 (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 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 µm, 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[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[3]. 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:* [4]

D 1193 Specification for Reagent Water

D 3195 Practice for Rotameter Calibration

E 20 Practice for Particle Size Analysis of Particulate Substances in the Range of 0.2 to 75 µm By Optical Microscopy[5]

2.2 *Other Documents:*

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[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.

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[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 Parenteral Science and Technology*, Vol 48, 1994, pp. 255-294.

[4] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[5] Withdrawn.

ANSI/NCSL Z540-1-1994 Laboratories and Measuring and Test Equipment—General Requirements[6]

ISO 11171 Hydraulic Fluid Power—Calibration of Liquid Automatic Particle Counters[6]

ANSI B93.20M-1972 Fluid Sample Containers—Qualifying and Cleaning Methods[6]

ANSI/NFPA T2.9.6 R2-1998 Hydraulic Fluid Power—Calibration of Liquid Automatic Particle Counters. National Fluid Power Association[6]

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 µm; the largest particle size typically reported is approximately 125 µm. When the LPC is calibrated with a polydisperse calibration suspension, the typical lower size sensitivity of the LPC is approximately 2.5 µm and the largest size reported is approximately 50 µm. 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 ± 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 µm to greater than 80 µm.

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 64 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 64 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.

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[6] Available from the American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

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. If the particles to be measured are nearly transparent to light and are nearly spherical in configuration, the monodisperse calibration material described in 8.2.1 should be used. If the particles are irregular in shape and absorb a significant amount of light, then the polydisperse calibration material described in 8.2.2 should be used.

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 calibration 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 suspending 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, the 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 reported increase in the relative standard deviation 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 becomes 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 measured suspension. 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 in sizes or concentration sufficient to interfere with device operation or sufficient to prevent meeting 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 ensure 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^2 with a 1-1 to 2-1 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 as 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, 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. Fig. 1 shows the operation of the optical system of a liquid-borne particle counter using light extinction to count and size particles in liquid. Although a gas laser is shown as the illumination source in Fig. 1, solid-state lasers and incandescent illumination sources are also in common use for some systems.

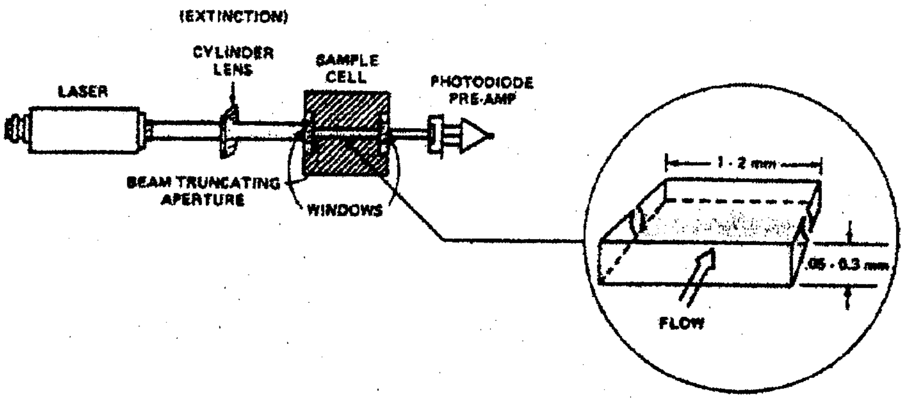

Figure 1. Extinction sensor optical system

FIG. 1 Extinction Sensor Optical System

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-µs 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 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 *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 reagent/liquids shall conform to the specifications of the Committee of Analytical Reagents of the American Chemical Society, where such specifications are available.[7] 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 by 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 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 with diameters ≥2 µm is approximately 10 000 times greater than the number of particles with diameters ≥15 µm. This means that no statistically significant data can be produced for particle sizes larger than 10 to 15 µm unless the suspension concentration is increased to the point where coincidence errors will occur due to the high concentration of the small particles. For this reason, calibration for particles an order of magnitude larger than the smallest particles requires a significant change in the calibration procedure. This change will be discussed in 10.4.

8.2.3 *Size Resolution Determination*—Monodisperse 10-µm 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 µm*—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 monodisperse or polydisperse particles that have been characterized by procedures that are traceable to accepted technology. The NIST offers a medium test dust in hydraulic oil with a certified cumulative size distribution up to 30 µm in diameter. This material is known as SRM 2806. Monodisperse glass and latex particles with traceable mean diameters may also be used.

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

Particle Size, µm	Cumulative Concentration, ISO UFTD Particles ≥ 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

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[7] *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 Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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.[8] 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 µm*—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–µm 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 be filtered through a 0.2-µm pore size filter or filtered at least twice through a 0.45-µm pore size filter.

NOTE 4—**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.

10. Procedures for Calibration

10.1 *Sample Volume and Flow Rate*—Verify that the sample flow rate is within the manufacturer's specifications for the LPC sensor used. For a pressure sample feeder using a burette directly downstream of the LPC sensing element with optical or other noncontacting detectors to indicate passage of the liquid through the burette, verify the accuracy of the measured volume between the "start" and "stop" indicators first.

10.1.1 Add liquid to the burette so that the liquid level is above the location of both detectors. Next, open the vent valve slowly and allow liquid to spill from the burette. When the level reaches the uppermost detector, collect liquid in a tared container. When the level reaches the lower detector, close the vent valve. Measure the liquid collected in the tared container and verify that the detectors are located to indicate the correct volume. Next, with clean liquid in a sample container in the feeder pressure vessel, set pressure to the recommended level and open the valve at the burette base so that liquid flows into the burette. Use a calibrated stopwatch to measure the time for liquid to fill the burette space between the two detectors. Calculate the liquid flow rate from these data. If necessary, adjust pressure to control the flow rate to the correct value. Following flow rate calibration, it must be kept in mind that change to a liquid with physical properties other than those of the liquid used for this calibration will result in flow rate changes at the same pressure. Liquids with high viscosity or high specific gravity will feed slower than those with lower values of viscosity or density.

10.1.2 With a sample feeder which uses aspiration to draw liquid into a burette, the procedure of 10.1.1 can be used. If the sample is aspirated directly to a syringe pump, no calibration is required. It is necessary only to ensure that no leaks in the feed system are present and that any gas in the feeder lines is removed before measurements are made.

10.2 *Zero Count Rate*:

10.2.1 Set the LPC to report data in the cumulative mode. Set the LPC to report particle counts for a time period of at least 20 s. Set the first channel at the LPC manufacturer's level for the lower size sensitivity level for that LPC.

10.2.2 With no liquid flowing through the LPC sensing volume, record accumulated particle count data for at least three replications of the time period selected in 10.2.1. Determine the average value of the accumulated counts, if any counts are reported with no liquid flowing through the LPC.

10.2.3 Using a sample container that has been carefully cleaned as defined in 7.1, place at least 250 mL of clean liquid, as defined in 8.2, in that container and place the container in the LPC sample feeder system. Flush the LPC and sample line to the sensing zone with three 10-mL batches of clean liquid. No data are recorded for this process.

10.2.4 Set the sample feeder to pass samples of 20 mL or more to the LPC for measurement.

10.2.5 Pass at least three samples (as selected in 10.2.3) of the clean liquid through the LPC and record the particle count

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[8] 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.

data collected. Determine the average count per millilitre of liquid and the relative standard deviation of the counts. The average value will represent the zero count or noise count rate for the LPC.

10.3 *Particle Sizing Accuracy Based on Response to Monodisperse Calibration Particles*:

10.3.1 Select the smallest monosize particles of interest and mix with clean liquid to make a suspension containing up to 10 000 particles per millilitre of liquid. Maintain the concentration of particles in the suspension at approximately 25 % of the manufacturer's recommended maximum. To ensure complete dispersion, place the container of suspension in the ultrasonic cleaner of 7.4 for up to 5 min. Depending upon the liquid, some surfactant addition may be necessary. Repeat with sufficient monosize particle batches of sizes to cover the LPC size range.

10.3.2 Filter a known volume aliquot sample of one suspension prepared in 10.3.1 through a membrane filter with pore size no more than half the size of the particles in the suspension. Determine the number concentration in the suspension by the aliquot sample size and the microscope count data in accordance with Practice E 20. Particle size data are not required here. If particles smaller than 5 µm are to be measured, then make the membrane transparent with clean solvent on a microscope slide. Use transmitted light illumination for viewing. For particles larger than 5 µm, observation on the membrane surface by oblique illumination may be used. Make sure that the doublet and other agglomerated particle number is less than 1 % of the total particle number seen on the membrane. If this level is exceeded, return the particle suspension to the ultrasonic cleaner for an additional 5 min. Repeat the filtration process and verify that the suspension is adequately dispersed. If adequate dispersion cannot be produced after 15 min of sonication, then the particle batch can be assumed to be irreversibly agglomerated. Discard that suspension and replace with fresh material. If a recently calibrated LPC is available, then the dispersion suitability and an indication of suspension concentration can be verified by measurements with this LPC.

10.3.3 Perform the manufacturer's recommended warm-up and field start-up procedures on the LPC to be calibrated. These include electronic and hydraulic system operation and control settings.

10.3.4 Using a series of 10 to 25-mL batches of clean filtered liquid, flush the LPC and its feeder system at least three times. (The flushing liquid may be recovered and cleaned for later use if cost, disposal, or hazard dictates minimum quantity consumption of some liquids.) Adjust the sample feeding system flow-rate controls during the flushing operation so that the recommended flow rate for the LPC under test is obtained. During the final flush, record the particle count, if any, and flush volume as a background measurement of cleanliness of the clean liquid, which is to be used for dilution. If the total background count is greater than 2 % of the level chosen in 10.3.1, then repeat the flushing operation until it has dropped to this level.

10.3.5 Pass at least 5 mL of the first suspension prepared in 10.3.1 through the LPC and discard. This liquid flow precharges system surfaces with calibration material for purposes of equilibration.

10.3.6 Pass 5 to 25-mL aliquot samples of one of the suspensions prepared in 10.3.1 through the LPC and record the particle count for a fixed sample volume as a function of pulse voltage threshold setting(s). A series of particle size and count versus threshold setting data will be produced. These data will be used to calculate the LPC size calibration and may also be used to determine particle size resolution if a suitably programmed microprocessor is built into the LPC. Each suspension shall be well-mixed, and any air bubbles shall be removed before data are generated. Expose the suspension to ultrasonic energy to 30 s to dislodge mechanically interlocked particles. The suspension shall then be vigorously hand shaken while rotating the bottle for a period of 5 min or shall be shaken for 5 min on a laboratory wrist action shaker. (If a commercial paint shaker is used for mixing, care is required to ensure that the sample container is securely retained on the shaker.) Exposure to low-intensity ultrasonic energy for 30 s before exposing the sample to reduced pressure will aid in removing gas bubbles. Next, expose the liquid sample to reduced pressure for a period sufficient to remove bubbles (at least 1 min) just before data are taken. The pressure shall be reduced slowly to reduce the possibility of boil-over if the liquid is exposed to the final pressure too rapidly. The pressure for bubble removal shall be at the level of the saturation vapor pressure of the liquid being used plus 10 % in absolute pressure units.

10.3.7 If a multichannel analyzer (MCA) is available, then the signal voltage that describes the modal pulse amplitude for a mono-size particle batch mean size can be obtained directly from the MCA readout. If no MCA is available, then the signal voltage values for the mean pulse amplitudes for each monosize particle batch can be obtained by the following manual procedure. This procedure is known as the "moving window differential half count" calibration procedure. This procedure is based on the fact that the LPC response varies with the particle diameter squared over a major portion of the particle size range of interest and that the portion of the size distribution curve being examined can be restricted sufficiently to omit signals from both small particle debris and from agglomerated particles.

10.3.8 Choose a relative standard deviation limit that is sufficient to include data that allows definition of the breadth of the particle size distribution of the calibration particle batch(es) being measured as well as the LPC resolution effects that will broaden the reported particle size distribution. As an example, 20 % may be used. This value is based on the combination of typical LPC resolution of 10 % or better and of worst case standard deviation of 5 % for calibration particles.

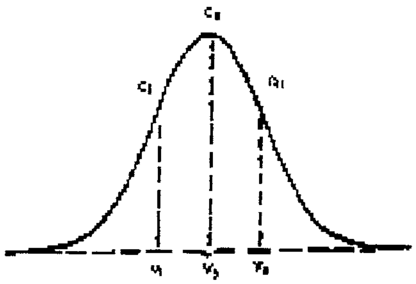
10.3.9 Select a value for the modal signal level expected for the first batch of the suspensions prepared in 10.3.1. This value can be based upon signal levels recorded from a previous calibration. Alternately, calibration data from an LPC with similar design can be used for initial selection levels.

10.3.10 Calculate the upper and lower limit voltages for the upper and lower windows. This calculation is based on the relative standard deviation limit chosen in 10.2.8. The upper window voltage limit is defined by multiplying the selected modal voltage by $(1 + 0.2)^2$. The lower window voltage limit is defined by multiplying the selected modal voltage by $(1 - 0.2)^2$.

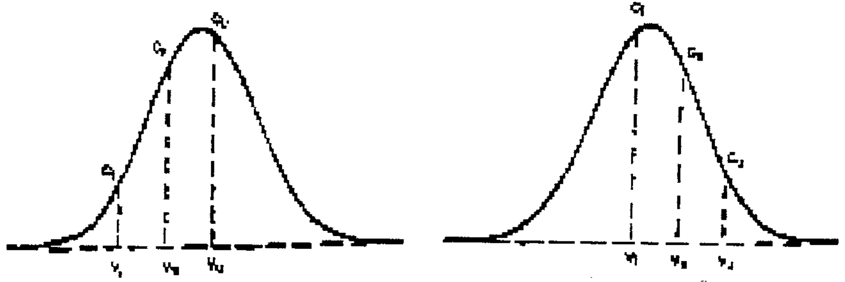
10.3.11 Adjust the LPC to report data in the differential mode and set the calculated lower window voltage for Channel 1 (the lower voltage limit for Channel 1). Set the selected voltage chosen in 10.3.9 for Channel 2 (the upper voltage limit for Channel 1 and the lower voltage limit for Channel 2). Set the calculated upper window voltage limit for Channel 3 (this is the upper voltage limit for Channel 2 and the lower voltage limit for channel 3). The nonactive channels normally used for reporting particles larger than those to be reported for Channel 3 shall be set at the highest voltage levels possible for the LPC being tested. Disregard data in those nonactive channels.

10.3.12 Pass a sample of 5 mL or larger of the first suspension prepared in 10.3.1 through the LPC and note particle count data in Channels 1 and 2. If the correct modal voltage was selected in 10.3.9, then the number of particles in the "windows" above and below that voltage would be equal. As shown in Fig. 2(a), the "windows" include the major part of the pulse height distribution. No small debris pulses and LPC noise or large pulses from agglomerated particles are included within the windows, when the correct modal voltage has been selected for the calibration particles being measured. For the correct voltage settings, the area under the particle size distribution curve between the lower-level voltage setting (mean – 20 % relative standard deviation) and the mean size voltage is equal to the area under the curve between the upper level voltage setting (mean + 20 % relative standard deviation) and the mean size setting. The number of particle counts in these windows will also be equal. When the correct mean size voltage has been selected, the number of counts in the two channels on both sides of that setting will be equal. If too low a mean voltage has been chosen, then the window boundary positions will be as shown in Fig. 2(b). The particle counts in the lower channel will then be less than those in the upper channel. If too high a modal size voltage has been selected, then the window boundary positions will be as shown in Fig. 2(c). The particle counts in the lower channel will be greater than those in the upper channel.

10.3.13 If either situation shown in Fig. 2(b) or Fig. 2(c) occurs, due to unequal particle numbers in Channels 1 and 2, the mean size voltage selected in 10.3.9 was incorrect. A new voltage must be selected. If counts in Channel 1 are smaller than those in Channel 2, then the selected voltage must be increased. If counts in Channel 1 are larger than those in Channel 2, then the selected mean size voltage must be decreased. Select a new voltage and repeat 10.3.10-10.3.12. When the correct voltage has been selected and the corresponding lower and upper window limits have been calculated and used, then the particle counts in Channels 1 and 2 will agree to within 3 %.



(a) v_s Correctly estimated



(b) v_s Under- estimated

(c) v_s Over- estimated

C_s: median diameter: sensor output is v_s

C_l : deviates from median diameter by –20%

C_u : deviates from median diameter by +20%

FIG. 2 Manual Moving Window Calibration

10.3.14 Repeat 10.3.9-10.3.13 for each of the batches prepared in 10.3.1.

10.3.15 Selection and adjustment of threshold voltage settings in 10.3.9-10.3.13 can be made manually or by using appropriate software for microprocessor-controlled LPCs. If a MCA is available, the modal pulse voltage can be seen directly on the MCA display as the peak of the display of the nearly Gaussian pulse amplitude distribution on the MCA screen.

10.3.16 If the data of 10.3.14 are collected manually, make a size calibration curve for the LPC by plotting threshold voltage settings on the ordinate and particle size on the abscissa. If a multichannel analyzer (MCA) was used to determine voltage settings for the maximum count for each particle size, those data can be obtained directly from the MCA readout. If an LPC operating with a built-in microprocessor control system is calibrated, verify that the installed microprocessor software contains a calibration program and use that program to calibrate the LPC. In any case, extrapolation of the calibration voltage threshold settings beyond the actual size of particles used in 10.3.14 shall be avoided, unless accurate noise level determinations are available and the extinction counter square law response is maintained throughout the extrapolated size ranges.

10.4 *Particle Sizing Accuracy Based on Polydisperse Particle Calibration Suspension*:

10.4.1 Set the LPC to report particle data in the cumulative mode. Using at least six size range channels, set threshold voltages as follows:

10.4.1.1 The lowest size threshold setting shall be 1.5 times the threshold noise level of the LPC as determined in 10.2. This noise level determines the lower size sensitivity level of the LPC.

10.4.1.2 The highest threshold setting shall be selected within the limitations of the LPC's voltage measurement range, the particle size distribution, and volume of the calibration suspension.

10.4.1.3 Intermediate threshold settings can be chosen to cover the size range of concern.

10.4.2 Prepare a calibration suspension for analysis. If the suspension is to be prepared by mixing dry dust with clean dilution liquid, the dry dust should be dried at least 18 h at 110 to 150°C before a sample is removed and weighed for addition to dilution liquid. Place a dry dust sample in the weight range from 100 to 500 mg in a clean sample container capable of containing 1 L of liquid. Add sufficient dilution liquid to fill that container to approximately 10 % of its capacity. Vigorously shake the sample by hand or with a mechanical shaker for at least 1 min. Ultrasonically disperse the dust for at least 30 s and add sufficient additional dilution liquid to produce a suspension with a dust concentration of 1 to 10 mg/L. If the calibration suspension to be used has been stored for more than 1 h, carry out the dispersion and shaking procedures before using it for calibration.

NOTE 5—Scoop-sampling directly from a container of polydisperse reference particles is prohibited because it can result in removal of a nonrepresentative (size-fractionated) sample that will permanently alter the size distribution in the remainder of the reference material bottle. If the entire container of reference material cannot be used, special care must be exercised in taking subsamples from the container. To subsample correctly, follow an accepted procedure including spin riffling, cone and quarter sampling, or other typical powder subdivision procedures.

10.4.3 Degas the sample ultrasonically or by using vacuum. Place the sample container in the pressure feeder sample holder; pass one 10-mL sample through the LPC to flush any previous residue from the feeder line and sensor surfaces. No data record is kept for this operation.

10.4.4 Obtain five consecutive particle counts each consisting of at least 10-mL liquid volume measured and 10 000 particles counted at the smallest threshold setting. The mean particle concentration for the five counts, X_{av}, for each channel must be greater than or equal to 100 for statistically significant results. Calculate the percent difference, D_q, between the minimum, X_{min}, and maximum, X_{max}, observed particle count for each channel using Eq 1 as follows:

$$D_q = 100\,\frac{(X_{max} - X_{min})}{X_{av}} \tag{1}$$

10.4.5 Record the threshold voltage setting, particle concentration data, X_{av}, and D_q data for each channel. Using Table 2, find the maximum allowable percent difference corresponding to the X_{av} value for each channel. If the value of D_q is less than the maximum, then the value of X_{av} for that channel is acceptable. If there are at least six channels with acceptable data, plot the particle concentrations versus the corresponding channel threshold settings on logarithmic graph paper for a calibration curve for the LPC. If the LPC is fitted with a built-in microprocessor control system, then that system can be programmed for automated data reduction and processing with the calibration information stored in channels. When acceptable data is less than six, proceed to 10.4.6.

10.4.6 Record the minimum, X_{min}, and maximum, X_{max}, observed particle counts for the suspect channel, along with the observed particle count, X_N, closest in value to either X_{min} or X_{max}. Calculate a difference value, D_o for that channel, using Eq 2 as follows:

$$D_o = \frac{(X_{max} - X_{min})}{|X_o - X_N|} \tag{2}$$

where X_o is the observed particle count of the suspected outlier (either X_{max} or X_{min}). If D_o for a channel is less than 1.44, discard the outlier data point (X_o), recalculate X_{av} using the remaining four data points, and use the recalculated value of X_{av} for calibration purposes. If D_o is greater than 1.44 for a channel, data from this channel are not acceptable and should be discarded.

10.4.7 If there are less than six channels of acceptable data (using the D_q and D_o criteria), repeat 10.4.1-10.4.6 after appropriate remedial action for the LPC.

TABLE 2 Maximum Allowable Percentage Differences in Particle Counts Between Runs

If X_{av} is Greater Than or Equal to:	If X_{av} is Less Than:	Maximum Allowable % Value for D_q is:
10 000	20 000	11.0
5 000	10 000	11.3
2 000	5 000	11.9
1 000	2 000	13.4
500	1 000	15.6
200	500	19.3
100	200	27.5

NOTE 6—If the only criterion not met in this calibration process is the requirement for adequate count data, reduce the LPC voltage threshold settings to report smaller particle sizes or repeat 10.4.1-10.4.6 using larger aliquot samples. Other failures to meet this criterion may arise from other sources such as volume measurement errors, calculation errors, operating with threshold voltages too close to the LPC noise level, bubbles in the samples, and so forth. Flow rate variability due to operation of the sample feeder chamber before the required constant pressure level is reached also leads to problems. Particle losses to the sample container walls or bubble generation due to excessive stirring rates can also occur. If the procedures to produce sufficient count data in the larger size ranges to generate statistically valid data are not feasible with the polydisperse calibration materials, then monodisperse particles larger than approximately 20 µm can be used to provide threshold voltage level data that is acceptable for defining other materials. This situation occurs since the extinction efficiency stabilizes at a value of 2 for almost any particles larger than 20 µm, as shown in Fig. 3.

10.4.8 Following acquisition of sufficient data to ensure six or more channels producing acceptable data, make a plot of particle concentration versus the corresponding LPC threshold settings for data from those channels. Depending upon availability of an MCA or built-in microprocessor control system, automated preparation of a calibration curve may be carried out, as discussed in 10.3.16.

10.5 *Particle Sizing Resolution*:

10.5.1 Particle sizing resolution shall be determined using monodisperse calibration particles at least two times larger than the lower sizing limit of the LPC. The diameter and relative standard deviation in diameter for those particles shall be recorded before testing for this parameter begins.

10.5.2 Prepare a suspension of the selected particles using the procedures of 10.3.1.

10.5.3 Carry out the procedures of 10.3.4 to ensure that no particles are in the LPC flow system.

10.5.4 Adjust the LPC counter to report data in the cumulative mode. Refer to the calibration data derived in 10.3 and determine the threshold voltages for the diameter of the particles selected in 10.5.1 and for sizes 10 % smaller than and 10 % larger than the selected particles.

10.5.5 Adjust the first three size channels of the LPC as follows: Channel 1 is set for a size 10 % smaller than the average size of the selected particles; Channel 2 is set for the average size of the selected particles; and Channel 3 is set for a size 10 % larger than the selected particle size.

10.5.6 Pass a batch of the particle suspension selected in 10.5.1 through the LPC, observing the particle count accumulation in Channel 2. When the count in that channel has reached 1000, stop the counting process and observe the count data in Channels 1 and 3. Check that the count in Channel 1 is 1680 (±3 %) and the count in Channel 3 is 320 (±3 %).

NOTE 7—The particle population values of 10.5.6 in the channels contiguous to Channel 2 are based on the relationship between population and parametric values in random distribution curves. Given a normal distribution curve, N_y, where y is a set of measurements of a parameter and N is the number of times a given value of y is observed. The equation for such a curve is as follows:

$$N_y = K \exp - (y - m)^2/2s^2 \tag{3}$$

where:
K = constant,
m = mean value for y, and
s = standard deviation.

At the mean value of the parameter measured, y is equal to m and N_y, the population value, is at a maximum. Solving for N_y when $(y - m)$ is equal to s, then N_y is equal to 0.607 times the maximum value. When the probability curve is presented in the cumulative mode (see Fig. 3), then the total population equal to and greater than the value for the mean less one standard deviation is 168 % of that at the mean, while the total population equal to and greater than the value for the mean plus one standard deviation is 32 % of that at the mean.

10.5.7 If the count data in Channels 1 and 3 are not at the values shown in 10.5.6, readjust the Channels 1 and 3 thresholds to either decrease or increase the particle counts in

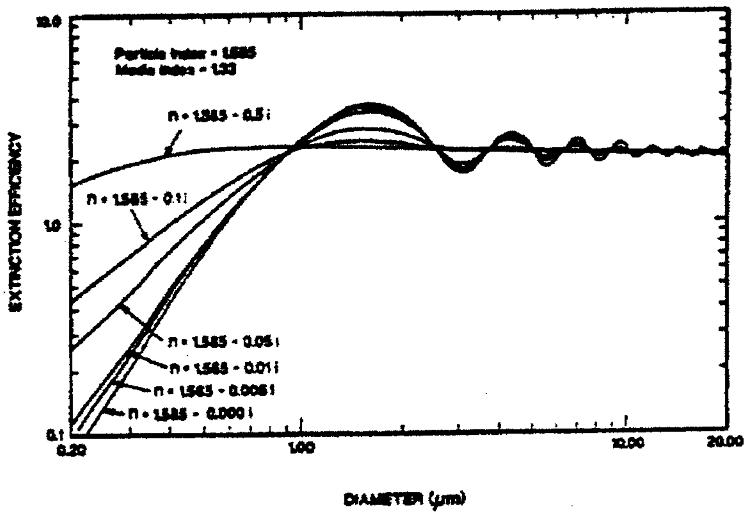


Fig. 3 Particle Size Effect on Extinction Efficiency Variation

FIG. 3 Particle Size Effect on Extinction Efficiency Variation

those channels to the criteria of 10.5.6. Thresholds may be made manually. If an LPC with microprocessor control is used, then the adjustments may be made automatically by use of appropriate software.

10.5.8 When the criteria of 10.5.6 are satisfied, repeat 10.3.6 until the particle count of Channel 2 has reached 10 000 and verify that the count in Channel 1 is 16 800 (±3 %) and that the count in Channel 3 is 3 200 (±3 %). Determine and record the particle size for the threshold settings just determined for Channels 1 and 3, as determined in 10.3.

10.5.9 If an MCA is available, then following 10.5.3, the sample selected can be run through the LPC with its output, set in the differential mode, connected directly to the MCA. Note the pulse height distribution display of the MCA and allow data to accumulate until 10 000 counts have accumulated in the MCA channel with the maximum count. Stop data accumulation and note the MCA channels above and below that maximum count channel where 60.7 % of that maximum value was accumulated. Based on the pulse height associated with the three channels so noted, report the particle sizes associated with the maximum count channel (the modal size), with the channel above the maximum count channel (the size one standard deviation greater than the modal size), and with the channel below the maximum count channel (the size one standard deviation smaller than the modal size).

10.5.10 Using the size data developed in 10.5.8, subtract the size for Channel 3 from the size for Channel 2; this difference will be the standard deviation for the particle size distribution derived from the positive side of the LPC reported pulse height distribution for the monodisperse test particles. Subtract the particle size for Channel 1 from the size for Channel 2; this difference will be the corresponding standard deviation on the negative side of the LPC reported pulse height distribution. The two standard deviations should agree within 5 % for non-skewed particle size distributions. Note the standard deviation and size data reported by the supplier for the particles used here.

10.5.11 Convert the standard deviations to variances by squaring the values obtained in 10.5.9. Subtract the variance defined by the supplier for the reported particle size distribution from the variance obtained from the 10.5.9 data and take the square root of that difference. This value will then be the standard deviation of the particle size data added to any measurement due to the resolution limit of the LPC under test. The resolution of the LPC is defined as in 3.1.16 and is calculated by dividing that added standard deviation value by the reported mean diameter of the particles used in 10.5. If the LPC is fitted with a suitably programmed microprocessor control system, data collected in 10.3.6 may be used directly. The resolution can be expressed as "percent resolution" by multiplying the resolution by 100. If the percent resolution is greater than 10 %, remedial measures (for example, illumination intensity uniformity improvement, sample flow control, or electronic drift elimination) may be required to improve LPC performance to an acceptable level.

10.6 *Counting Efficiency*:

10.6.1 Select a batch of monosize particles with the diameter in the size range from 10 to 25 µm. These limits are chosen to permit ease of viewing at the small size end of the range and to minimize problems of loss due to deposition, settling, or inadequate dispersion at the large size end of the range. Use procedures 10.3.1 and 10.3.2 for preparation and measurement of suspensions to be used for this determination. Record the total particle concentration in that suspension.

10.6.2 In order to select the threshold setting for total count of a monodisperse sample, refer to the data obtained for the sample in 10.3.2. Select a threshold setting that is two to three times the level where counting due to electrical noise or small particle debris in the clean liquid is noted and is no more than one half the threshold level calculated for the particles chosen in 10.6.1. The noise threshold level is determined from the data of 10.2.

10.6.3 Using the threshold voltage setting selected in 10.6.2, pass a known volume of suspension through the LPC and record the number concentration per unit volume of suspension reported by the LPC with that setting. Repeat this procedure so that concentration data from a total of three aliquot samples are collected. Determine an average concentration and a relative standard deviation (RSD). If the RSD is greater than 5 %, repeat this step after mixing the sample more thoroughly.

10.6.4 Note the data obtained in 10.6.2 and compare the concentration information obtained from the aliquot samples passed through the LPC and those samples measured by procedures 10.3.1 and 10.3.2. Define the counting accuracy as the ratio of the LPC concentration to the microscope measurement concentration. The concentration data must be used, rather than the direct particle count data, since the volumes used for the two measurements may be different.

NOTE 8—If an LPC is available that has been calibrated in accordance with 10.6 and found to have a counting accuracy of 100 ± 5 %, it can be used to obtained the reference concentration data for the suspension(s) measured in 10.6.3. These reference data can be obtained by passing separate aliquot samples through the calibrated OPC or by passing the same samples in series through the calibrated LPC and the LPC under test. If the latter procedure is used, care is required to minimize particle losses in the sample transport lines between the two LPCs.

10.7 *Particle Concentration Limit*:

NOTE 9—The maximum concentration, that can be accurately measured, is limited by the accepted coincidence error. This error is due to the probability of more than one particle presence within the LPC viewing volume at any time. The error level is a function of the physical dimensions of the viewing volume, the particle size distribution of the particles in the sample being counted, and the actual particle concentration within that sample. For samples where all particles are insignificant in dimension to that of the viewing volume, the probability of more than one particle being present within a defined volume can be calculated directly from statistical theory[2]. However, the electronic counting circuitry cannot respond to the presence of aparticle entering the viewing volume until the signal from a previous particle has decreased to the system reset level. Where particles may be large enough so that their size is significant in comparison with a viewing volume dimension, the presence of such a particle results in an effective increase in the dimensions of the viewing volume, as shown in Fig. 4. The net result is that the presence of large particles in the sample decreases the particle concentration limit more than expected from the calculation of probability of coincident particles in the viewing volume. For this reason, the recommended procedure for determining the maximum concentration limit uses a polydisperse particle suspension. Determining the maximum concentration limit with small monodisperse particle suspension should be used only if the materials to

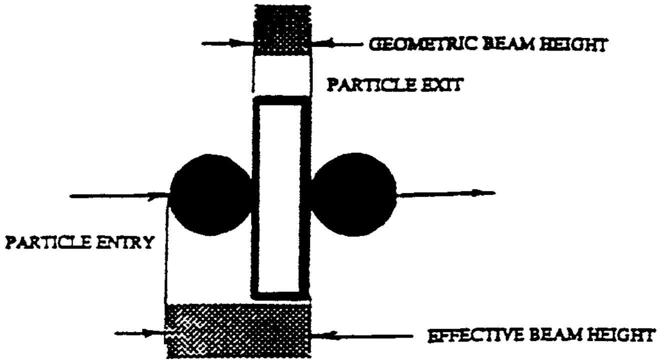


FIG. 4 Large Particle Effect on Viewing Volume Effective Dimension

be counted will never include large particles.

10.7.1 Set the LPC to report data in the cumulative mode and select a particle size voltage level for the first channel corresponding to the voltage level for the smallest particle size of interest, as determined in 10.3 or 10.4. Set voltage levels for the remaining channels for larger particle size ranges in ascending order, up to the largest particle size of interest.

10.7.2 Adjust flow through the LPC to the working flow rate using clean liquid. Flush the LPC sensor and flow system with at least two 50-mL batches of clean liquid.

10.7.3 Prepare diluted 100 to 150-mL suspensions of the concentrated material prepared as in 10.4.2 and 10.4.3 which are 0, 10, 20, 40, 60, 80, 100, 120, and 150 % of the LPC manufacturer's recommended maximum concentration limit for that LPC. Label and place a clean closure on each container of diluted suspension.

10.7.4 Following agitation to disperse particles in the sample container with the lowest concentration prepared in 10.7.3, degas the diluted sample under vacuum or ultrasonically.

10.7.5 Pass five consecutive 10-mL or greater samples through the LPC. Discard data from the first two samples and determine the average for the last three samples. The concentration data from each measurement should not differ from the average by more than 15 %. Record the reported average concentration data.

10.7.6 Repeat 10.7.4 and 10.7.5 for the remaining diluted suspensions.

10.7.7 Compare the reported concentration data ratio to the dilution ratio for each succeeding pair of diluted suspensions. If there is no coincidence error, the two ratios will be identical, for example, the concentration reported for the 20 % suspension should be twice that for the 10 % suspension. As the actual concentrations increase, reported concentrations will increase at a rate less than that for the actual concentration, as shown in Fig. 5.

10.7.8 When the reported concentration becomes less than 90 % of the actual concentration, report the true concentration as the recommended maximum concentration limit for that LPC.

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 design variations for different instruments will result in differing results when measuring normal particle suspensions. Thus, testing with particles differing in physical properties from the calibration material can lead to different results when using LPCs that differ in optical and physical design. Where calibration is carried out carefully with accurately characterized calibration materials and by a tested and proven procedure, reproducibility and correlation between different instruments is satisfactory. A brief description of this situation is presented in Appendix X1. This description is a discussion of a detailed round-robin test procedure carried out as part of ISO 11171.

12. Keywords

12.1 calibration; light extinction; liquid-borne particles; particle counting; particle size resolution; particle sizing accuracy; sample flow rate

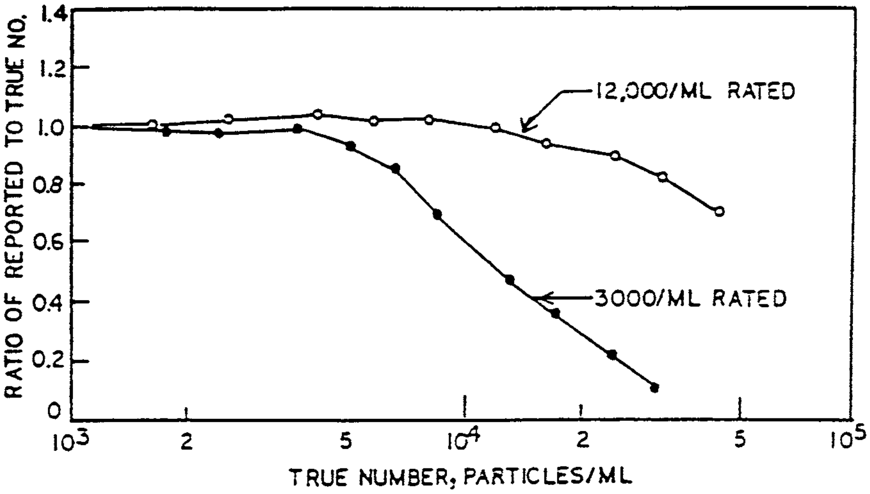


FIG. 5 Coincidence Effect on Reported Concentration Accuracy

APPENDIX

(Nonmandatory Information)

X1. DISCUSSION OF ISO 11171 ROUND-ROBIN TEST RESULTS

X1.1 Since 10.4 of this practice describes a particle size calibration procedure that is new to Practice F 658, a round robin for a new procedure normally is recommended. However, ISO TC 131 on hydraulic fluid properties and measurements recently has completed a round robin on the procedure that is essentially identical to that described in 10.4 of this practice. There is no requirement to duplicate work that has been described elsewhere. This appendix briefly discusses the ISO TC 131 round-robin work. As part of the evaluation of ISO 11171 on calibration of light extinction particle counters used for evaluation of hydraulic fluid cleanliness, a round-robin calibration and test procedure was performed. More than 24 laboratories in different parts of the world participated in this activity. All laboratories were provided with calibration samples of ISO Medium Test Dust (MTD) in clean fluid, with verification samples of ISO Ultrafine Test Dust (UFTD) and with two test samples. One test sample contained a 1.00-mg/L suspension of ISO UFTD and the other was a batch of 0.03-µm particles in clean dilution fluid. These samples represent the types of materials that are frequently found in hydraulic fluids, lubricants, and fuel samples.

X1.2 Following careful calibration with the MTD calibration samples, the lower sizing limit and dynamic range capabilities of the LPCs in the testing laboratories were specified, along with their resolution capabilities. The verification samples were used for this purpose. Sizing variability between LPCs of different design was determined in order to minimize effects of significant sensor design differences. The importance of sensor resolution was noted as a possible source of sizing variability. Maximum concentration capability before coincidence errors became excessive were also noted for each instrument type tested.

X1.3 Following careful calibration in accordance with the calibration material and calibration procedure requirements of ISO 11171, evaluation of the data for the two test samples was carried out. The sample containing the ISO UFTD and that containing the 0 to 30-µm particles were characterized for particle size distribution and concentration. The data from those laboratories that reported good sizing and resolution capabilities showed that the concentrations for all sizes within that material could be reported as being within 15 % of the stated size with a 95 % confidence limit as long as the concentrations were at least 100 particles/mL of fluid. The UFTD sample confidence limit range increased for particles larger than approximately 10 µm, but this increase is due to the rapid decrease in concentration for those larger particles that results in poor statistical data. The data for the 0 to 30-µm sample showed that those laboratories reported the same size distribution with all particles within 10 % of the modal sizes with a 95 % confidence limit. These “better” data are due to the higher concentration of particles in the entire size range of that sample.

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