BS ISO 11171:2016

BSI Standards Publication

Hydraulic fluid power — Calibration of automatic particle counters for liquids

National foreword

This British Standard is the UK implementation of ISO 11171:2016. It supersedes BS ISO 11171:2010 which is withdrawn.

Attention is drawn to the fact that the UK committee voted against the approval of this International Standard.

The UK committee has concerns that there are three options for presenting data which will give significant differences in particle counts when analysing the same fluid with automatic particle counters (APCs). The difference in particle counts is because the previous calibration suspensions, SRM 2806 and 2806a, where the calibrated size is labelled "µm(c)", are no longer available. The latest characterization of the particle sizes by electron microscope has shown that there is an increase in size of the same particles. The result is that calibration using SRM 2806b results in higher particle counts compared to calibration using SRM 2806 or SRM 2806a, when testing the same fluid.

Clause 6.8 of this standard describes the process for constructing an additional calibration curve so that an APC calibrated using SRM 2806b can give results equivalent to the earlier standard that used SRM 2806 or SRM 2806a when option 7.1 b) is selected, but use of this is not required when reporting to options 7.1 a) and 7.1 c).

Any standard using APCs calibrated to ISO 11171 will be affected by these changes e.g. ISO 4406. It is beyond the scope of this standard to state how to apply the results derived in clause 7.1 to generate ISO 4406 codes. At the time of publication, ISO 4406 has not been revised to account for these changes and the UK committee suggests, therefore, that the ISO Code is followed by:

- "µm" if option 7.1 a) is chosen;
- "µm(c)" if option 7.1 b) is chosen, or
- "µm(b)" if option 7.1 c) is chosen.

The UK participation in its preparation was entrusted by Technical Committee MCE/18, Fluid power systems and components, to Subcommittee MCE/18/-/6, Contamination control.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

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INTERNATIONAL STANDARD

ISO 11171

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2016-10-01

Hydraulic fluid power — Calibration of automatic particle counters for liquids

Transmissions hydrauliques — Étalonnage des compteurs automatiques de particules en suspension dans les liquides

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Contents

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 131, *Fluid power systems*, Subcommittee SC 6, *Contamination control*.

This third edition cancels and replaces the second edition (ISO 11171:2010), of which it constitutes a minor revision.

This edition includes the following significant changes with respect to the previous edition:

- 6.8: defining µm equation, Table 3 – revised to show µm(b) and µm(c) to be reported;
- 7.1: revised to show how to report µm(b) and µm(c).

Introduction

In hydraulic fluid power systems, power is transmitted and controlled through a liquid under pressure within an enclosed circuit. The fluid is both a lubricant and a power-transmitting medium. Reliable system performance requires control of the contaminants in the fluid. Qualitative and quantitative determination of the particulate contaminants in the fluid medium requires precision in obtaining the sample and in determining the contaminant particle size distribution and concentration. Liquid automatic particle counters (APCs) are an accepted means of determining the concentration and size distribution of the contaminant particles. Individual APC accuracy is established through calibration.

This International Standard establishes a recommended standard calibration procedure for determining particle sizing and counting accuracy. The primary particle-sizing calibration is conducted using NIST SRM 2806 suspensions with particle size distribution certified by the United States' National Institute of Standards and Technology (NIST). A secondary calibration method with traceability to NIST uses suspensions of ISO MTD which are independently analysed using an APC calibrated by the primary method. Concentration limits are determined through the use of serial dilutions of a concentrated suspension. Operation and performance limits are also established using this International Standard.

Hydraulic fluid power — Calibration of automatic particle counters for liquids

1 Scope

This International Standard specifies procedures for the following:

a) primary particle-sizing calibration, sensor resolution and counting performance of automatic particle counters (APCs) for liquids capable of analysing bottle samples;

b) secondary particle-sizing calibration using suspensions verified with a primary calibrated APC;

c) establishing acceptable operation and performance limits;

d) verifying particle sensor performance using a truncated test dust;

e) determining coincidence and flow rate limits.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3722, *Hydraulic fluid power — Fluid sample containers — Qualifying and controlling cleaning methods*

ISO 5598, *Fluid power systems and components — Vocabulary*

ISO 12103-1, *Road vehicles — Test dust for filter evaluation — Part 1: Arizona test dust*

ISO 16889, *Hydraulic fluid power — Filters — Multi-pass method for evaluating filtration performance of a filter element*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 5598 and the following apply.

3.1

automatic particle counter

APC

instrument that automatically counts and sizes individual particles suspended in a fluid, typically relying on optical light scattering or light extinction principles of particle sizing

Note 1 to entry: An APC consists of, at a minimum, a particle sensor, a means for delivering a known volume of sample to the sensor at a controlled rate, a signal processor, an analyser that compiles the sensor output for the sizes of individual particles into particle size distribution and a means for outputting particle size distribution results for the sample.

3.2

threshold noise level

minimum voltage setting of an automatic particle counter at which the observed pulse-counting frequency does not exceed 60 counts/min due to electrical noise in the absence of flow in the sensing volume

3.3
sensing volume
portion of the illuminated region of the sensor through which the fluid stream passes and from which the light is collected by the optical system

3.4
resolution
measure of the ability of an automatic particle counter to distinguish between particles of similar, but different, sizes

3.5
coincidence error limit
highest concentration of NIST RM 8632 that can be counted with an automatic particle counter with an error of less than 5 % resulting from the presence of more than one particle in the sensing volume at one time

3.6
working flow rate
flow rate through the sensor used for sizing calibration and sample analysis

3.7
particle size
projected area equivalent diameter of particles as determined using scanning electron microscopy or as determined using a calibrated liquid optical single particle automatic particle counter

Note 1 to entry: Unless otherwise stated, an APC used for particle size determination is calibrated in accordance with this International Standard.

Note 2 to entry: NIST uses scanning electron microscopy to determine the projected area equivalent diameter of particles in its reference materials.

3.8
particle size distribution
number concentration of particles, expressed as a function of particle size

3.9
primary calibration
sizing calibration conducted using NIST standard reference material 2806x

Note 1 to entry: The procedure is specified in Clause 6.

Note 2 to entry: For details of NIST standard reference material 2806x, see 4.4.

3.10
secondary calibration
sizing calibration conducted using calibration suspensions

Note 1 to entry: The procedure is specified in Clause 6 and the calibration suspensions are prepared in accordance with Annex F.

4 Materials and equipment

4.1 Polystyrene latex spheres, nearly monodispersed in aqueous suspension. Polystyrene latex spheres with a nominal diameter of 10 µm are required in Annex D for resolution determination and polystyrene latex spheres with other nominal diameters larger than 50 µm are required in Clause 6, if size calibration for particle sizes of 50 µm and larger is performed. In certain situations, it may also be useful to use additional sphere sizes. Regardless, the coefficient of variation of each polystyrene latex sphere size shall be less than 5 %. The supplier of the polystyrene latex spheres shall provide a certificate of analysis with each batch, which indicates that the sphere particle size has been determined using techniques with traceability to National or International Standards.

Once opened, suspensions of polystyrene latex spheres shall be used within three months unless the size distribution and cleanliness of the suspension have been verified.

NOTE 1 The size distribution and cleanliness of polystyrene latex spheres can be verified using the method described in D.13.

NOTE 2 Polystyrene latex spheres in aqueous suspension have a limited shelf-life. Shelf-life is a function of a variety of factors including temperature and microbial contamination of the suspension.

4.2 Clean dilution fluid, consisting of the test fluid used in ISO 16889 and an antistatic additive that gives a conductivity of 2 500 pS/m ± 1 000 pS/m at room temperature. The fluid shall contain less than 0,5 % of the number of particles equal to or larger than the smallest particle size of interest expected to be observed in the samples.

4.3 Clean aerosol OT dilution fluid, to determine sensor resolution in Annex D (the clean dilution fluid specified in 4.2 is used for all other operations in this International Standard). It is prepared from a concentrate made by adding 120 g of aerosol OT to each litre of clean dilution fluid (4.2). Heat the concentrate to about 60 °C and stir until the aerosol OT has completely dissolved. Prepare the aerosol OT dilution fluid by diluting the concentrate with clean dilution fluid (4.2) to a final concentration of 12 g of aerosol OT per litre. The clean aerosol OT dilution fluid shall meet the same cleanliness levels as the dilution fluid specified in 4.2.

CAUTION — Follow the precautions for safe handling and usage described in the materials safety data sheet (available from the supplier of the aerosol OT).

Aerosol OT (dioctyl sulfosuccinate, sodium salt) is a waxy, hygroscopic solid. If it appears to be damp or has absorbed water prior to use, dry it first for at least 18 h at about 150 °C.

4.4 NIST standard reference material 2806*x* (SRM 2806*x*) primary calibration suspension, where *x* is the letter used by NIST to designate the batch number of the certified primary calibration suspension, available from NIST. Primary calibrations shall use SRM 2806.

NOTE ISO/TR 16144 describes the procedures used to certify the standard reference material SRM 2806.

4.5 NIST reference material 8631 (RM 8631) dust, prepared by drying the dust for at least 18 h at a temperature between 110 °C and 150 °C, required if secondary calibration is to be performed (see 6.1).

4.6 ISO medium test dust (MTD) in accordance with ISO 12103-1, dried for at least 18 h at a temperature between 110 °C and 150 °C before use.

4.7 NIST reference material 8632 (RM 8632) dust, prepared by drying the dust for at least 18 h a temperature between 110 °C and 150 °C before use, if required for determination of coincidence error limit or in Annexes B, C and E.

NOTE The reference materials specified in 4.4, 4.5, 4.6 and 4.7 are created using "living" documents that may change as new batches are produced. Users of this International Standard are advised to ensure that they are using the latest batch available.

4.8 Automatic particle counter (APC) for liquids, with bottle sampler.

4.9 Clean sample containers, with closures (appropriate bottle caps, for example), and **volumetric glassware** of at least class B. The cleanliness levels of the sample containers, closures and glassware shall be less than 0,5 % of the number of particles (larger than the smallest particle size of interest) expected to be observed in the samples. The cleanliness levels shall be confirmed by ISO 3722.

4.10 Mechanical shaker, such as a paint or laboratory shaker, suitable for dispersing suspensions.

4.11 Ultrasonic cleaner, with a power density of 3 000 W/m^2 to 10 000 W/m^2 of bottom area.

4.12 Linear-linear graph paper or computer software for generating graphics.

4.13 Log-log graph paper or computer software for generating graphics.

4.14 Analytical or electronic balance with the following minimum specifications:

a) readability: 0,05 mg;

b) accuracy (agreement with true mass): ±0,05 mg;

c) precision (repeatability): ±0,05 mg;

d) front or side doors and a covered top to eliminate the effect of air currents.

5 Sequence of APC calibration procedures

5.1 See Figure 1 for a recommended sequence of steps to be followed when performing a full calibration on a new APC. Conduct the procedures of this clause when a new APC is received or following the repair or readjustment of an APC or sensor (see Table 1). Proceed to Clause 6 if neither the APC nor the sensor has been repaired or readjusted, if no detectable change in the operating characteristics has occurred since the last sizing calibration was performed, or if the APC has been subjected to the procedures in Annexes A, B, C, D, and E and the results have been documented. The specific order of Annexes and Clauses specified in Figure 1 and Table 1 are recommendations. The operator may follow a different order, as long as all required parts are performed.

NOTE 1 Annexes A, B, C, and D can be performed by an individual laboratory or by the manufacturer of the APC prior to delivery.

A change in the operating characteristics of the APC can be detected by several different methods, including but not limited to the following:

a) using particle data from control samples collected over time and a statistical process control chart, such as an individuals moving range (IMR) chart, to detect significant changes in calibration;

b) comparing calibration curves over time to detect a significant change in calibration;

c) returning the APC to its manufacturer for evaluation and assessment of the change in calibration;

d) analysing a primary or secondary calibration suspension in accordance with 6.2 and 6.3, then comparing the resulting particle concentration data to the corresponding particle size distribution for the sample. If the results agree within the limits for the maximum allowable D_Q given in Table C.2, the ability of the APC to size and count particles has not been significantly affected. If the results do not agree, a significant change has occurred and the operator is instructed to proceed as indicated in Table 1;

e) analysing a primary or secondary calibration suspension and resulting data as described in item d), then analysing an ISO UFTD sample prepared in accordance with Annex A, then comparing the resulting particle concentration data with the limits given in Table A.1. If the results agree within the limits given in Table A.1, the ability of the APC to size and count particles has not been significantly affected. If the results do not agree with the limits of Table A.1, the APC has experienced a significant change and the operator is instructed to proceed as indicated in Table 1.

NOTE 2 For the purposes of this subclause, repair or readjustment of an APC refers to service or repair procedures that affect the ability of the APC to accurately size and count particles.

If the light source or any part of the optics is adjusted, repaired or replaced, the procedures of Clause 6 and Annexes A, B, D, and E shall be repeated.

If the sensor or counting electronics is adjusted, repaired or replaced, the procedures of Clause 6 and Annexes A, B, C, D, and E shall be repeated.

If the volume measurement system is repaired, replaced or readjusted, the procedures of Annex A shall be repeated.

It is not necessary to repeat these procedures following normal cleaning procedures, the attachment of cables or peripheral equipment, the replacement of plumbing lines or connections, or following other operations that do not involve disassembly of the APC, sensor or volume measurement system.

5.2 Perform the preliminary APC check, which includes volume accuracy, in accordance with Annex A.

5.3 Determine the coincidence error limits of the APC in accordance with Annex B.

5.4 Perform the sizing calibration procedure in accordance with Clause 6.

5.5 Determine the flow rate limits of the APC in accordance with Annex C.

5.6 Determine the APC resolution in accordance with Annex D.

5.7 Verify the particle-counting accuracy in accordance with Annex E.

5.8 In order to conform to the requirements of this International Standard, the APC shall include the following:

a) be calibrated in accordance with 5.4;

b) meet the volume accuracy, resolution and sensor performance specifications determined in 5.2, 5.6 and 5.7;

c) be operated using the calibration curve determined in 5.4 within the coincidence error and flow rate limits determined in 5.3 and 5.5.

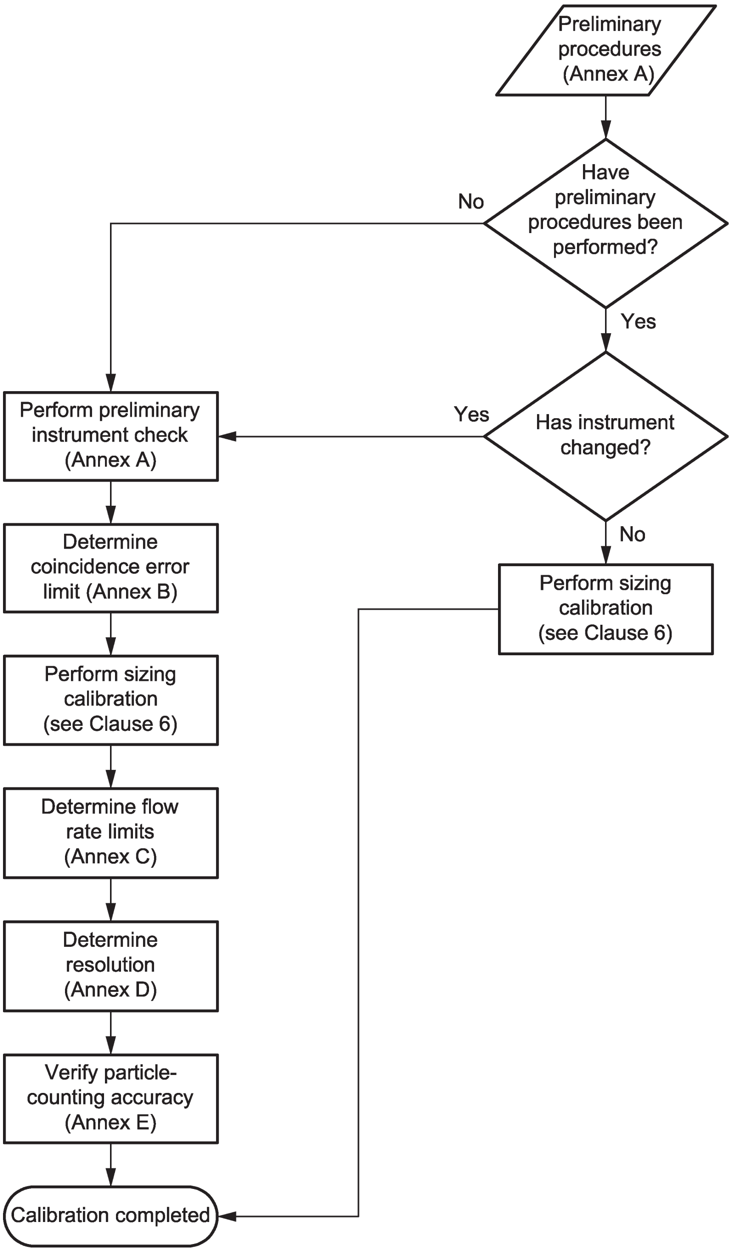


Figure 1 — Sequence of APC calibration procedures

Table 1 — Schedule of APC calibration procedures

APC status[a]	Relevant Clause and Annexes of this International Standard					
	Clause 6	Annex A	Annex B	Annex C	Annex D	Annex E
	Sizing calibration procedure	Preliminary APC check	Coincidence error limits	Flow rate limits	Resolution	Accuracy
New APC or existing APC not calibrated to this International Standard	x	x	x	x	x	x
Last calibration was more than 6 m to 12 m ago	x	—	—	—	—	—
Suspicion that calibration has changed significantly	x	—	—	—	—	—
Optics (including light source) repaired or readjusted	x	x	x	—	x	x
Sensor or counting electronics repaired or readjusted	x	x	x	x	x	x
Volume measurement components (e.g. flowmeter, burette, level detectors) repaired or readjusted	—	x	—	—	—	—
Sensor cleaned	No action necessary					
Cables or peripheral equipment attached	No action necessary					
Plumbing lines and connections replaced	No action necessary					
Operation performed that does not involve disassembly of APC, sensor or volume measurement system	No action necessary					

[a] Repair or readjustment refers only to service or repair procedures that affect the ability of the APC to accurately size and count particles. In order to verify the ability of an APC to accurately size and count particles, analyse a primary or secondary calibration suspension in accordance with 6.2 and 6.3, then compare the resulting particle concentration data to the corresponding particle size distribution for the sample. If the results agree within the limits given for the maximum allowable D_Q in Table C.2, the ability of the APC to size and count particles has not been significantly affected. If the results do not agree, proceed as indicated in this table.

6 Sizing calibration procedure

6.1 Refer to Figure 2 for a flow chart describing the sizing calibration procedure. Conduct the sizing calibration every three to six months, when a new APC is received, or after the repair or readjustment of an APC or sensor. For primary calibrations, use NIST calibration suspensions (see 4.4). For secondary calibrations, use calibration suspensions prepared in accordance with Annex F.

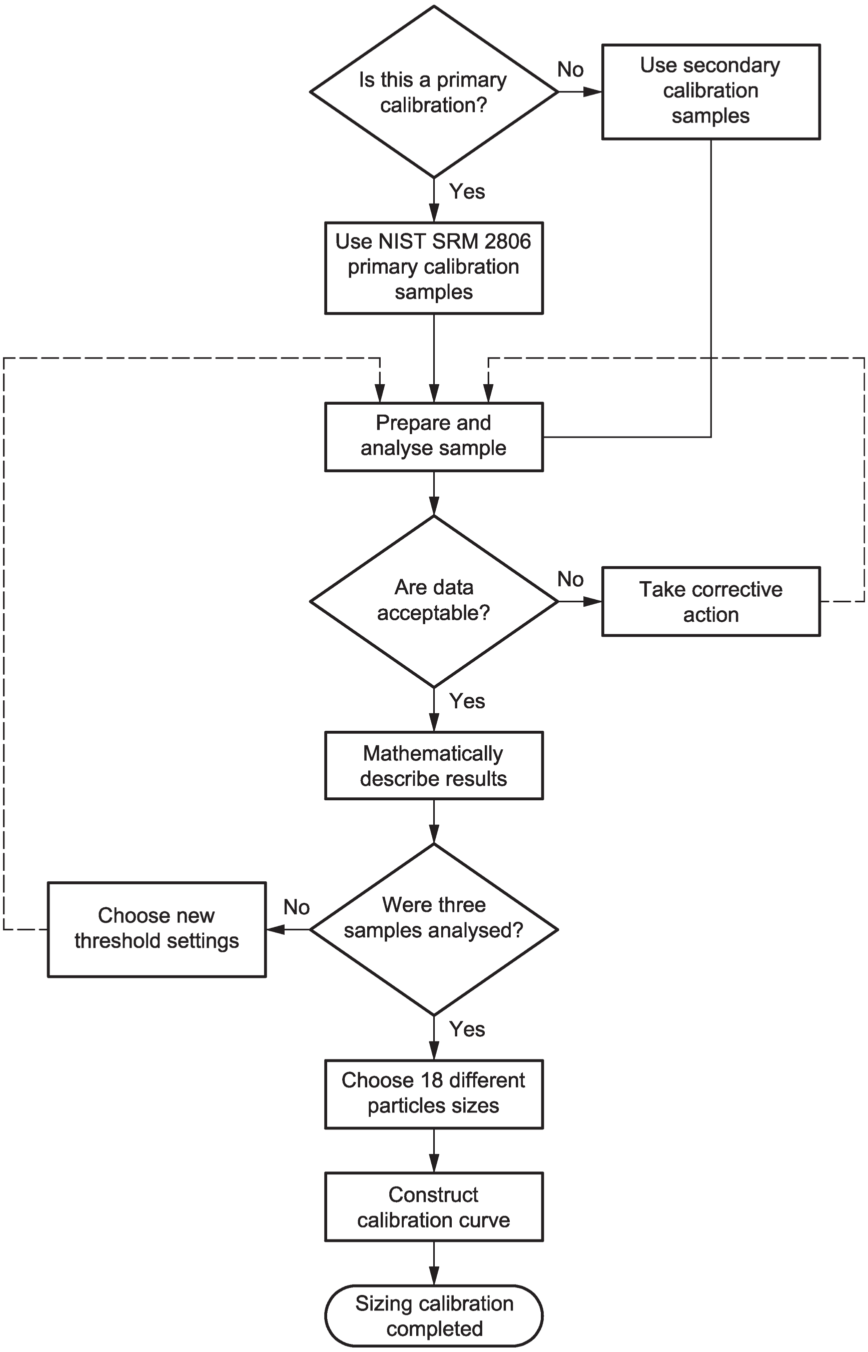


Figure 2 — Sizing calibration procedure

After a suitable calibration history for an APC and sensor has been developed, the frequency of calibration can gradually decrease, but the time interval between successive calibrations shall not exceed one year.

All phases of the calibration shall be conducted at the same flow rate. The flow rate limits of the APC are determined in Annex C. Any data obtained at flow rates outside these limits shall be discarded and the corresponding part of the procedure repeated using the proper flow rate.

Conduct the sizing calibration using the same sample volume used in 5.2. If a different volume is used, the procedure in 5.2 shall be repeated using the new sample volume to avoid volume measurement errors.

It is recommended that the threshold noise level of the APC be determined using the method in A.2 before proceeding to 6.2. If the threshold noise level has changed by more than 30 % since the last time it was determined, this can be an indication that the calibration of the APC has changed and the APC is in need of repair. Failure to check the threshold noise level before proceeding to 6.2 can result in lost time spent trying to calibrate a defective APC and invalidation of particle count data.

6.2 Set the APC to the cumulative mode and, using at least six different channels, set the threshold voltage as follows:

a) the lowest threshold setting shall be at least 1,5 times the threshold noise level of the APC, this determines the minimum detectable particle size;

b) the highest threshold setting is limited by the working-voltage range of the APC (consult the APC manufacturer to determine this), the particle size distribution and the volume of the calibration sample;

c) intermediate threshold settings shall be chosen to cover the size range of interest.

Prepare a calibration suspension sample for analysis. Shake the sample vigorously by hand. Agitate the sample ultrasonically for at least 30 s and then shake it on a mechanical shaker for at least 1 min to disperse the dust in the liquid. Continue shaking the sample until it is to be analysed.

The procedure described in 6.2 to 6.8 assumes manual calibration of an APC with a small number of threshold settings. Alternatively, calibration can be performed using a multichannel analyser (MCA) or software that follows the same procedure. If an MCA is used, it is essential that the relationship between the measured voltage of the MCA and the APC threshold setting be first established. In general, software and MCA methods tend to be faster and more accurate than manual methods.

6.3 Degas the sample under vacuum or ultrasonically until the bubbles rise to the surface and gently turn the sample bottle over at least five times, taking care not to introduce air bubbles into the liquid. Obtain at least five consecutive particle counts, each consisting of at least 10 mL and 10 000 particles at the smallest threshold setting.

Calculate the total number, N, of particles counted for each channel using Formula (1):

$$N = 5\overline{X}V \tag{1}$$

where

X is the mean particle concentration, in particles per millilitre, for the five counts for a particular channel;

V is the sample volume, in millilitres, for a single count.

The value of N shall be greater than or equal to 1 000 in order to ensure statistically significant results for that particular channel.

Calculate D_Q, which is the difference expressed as a percentage between the minimum, X_{min}, and maximum, X_{max}, observed particle count for each channel, using Formula (2):

$$D_Q = \frac{X_{max} - X_{min}}{\overline{X}} \times 100 \tag{2}$$

Record in Table 2, the threshold voltage setting, particle concentration data, $\overline{X}$, and D_Q for each channel.

Using Table C.2, find the maximum allowable difference expressed as a percentage corresponding to the value of $\overline{X}$ for each channel. If the value of D_Q is less than the maximum, then the value of $\overline{X}$ for that channel is acceptable for use. If there are at least six channels with acceptable data, proceed to 6.4. If not, examine the results of any unacceptable channels as specified in the following.

Calculate D_0 using Formula (3):

$$D_0 = \frac{X_{\max} - X_{\min}}{\left| X_0 - X_N \right|} \tag{3}$$

where

X_0 is the observed particle count of the suspected outlier (either $X_{\max}$ or $X_{\min}$);

X_N is the observed particle count closest in value to X_0.

If D_0 for a particular channel is less than 1,44, discard the related outlier data point, X_0, recalculate $\overline{X}$ using the remaining four data points, and use the recalculated value of $\overline{X}$ for calibration purposes. If D_0 for a particular channel is greater than 1,44, data from this channel are not acceptable and shall be discarded. If there are at least six channels of acceptable data (using the D_Q and D_0 criteria), proceed to 6.4. If not, take appropriate corrective action and repeat 6.1 to 6.3.

If N is less than 1 000 for any channel, the data for that channel shall not be used. If sufficient numbers of particles counted is the only quality criterion that is not met, change the threshold settings to correspond to particle sizes that yield sufficient counts, or repeat 6.1 to 6.3 using a larger sample volume.

Primary and secondary calibration samples shall not be collected and reused.

NOTE Other failures to meet the quality criteria can arise from a number of sources, including contaminated dilution fluid or glassware, volumetric errors, calculation errors, operating too close to the threshold noise level of the APC, or bubbles in the samples. Flow rate variability due to counting while the sample chamber is being pressurized or due to other sources also leads to problems. Particle settling can occur. If excessively high stirring rates are used, particles can be centrifuged out or bubbles can be introduced.

Table 2 — APC particle sizing calibration worksheet (see 6.3, 6.8 and A.9)

APC		Model		Date	
		Serial number		Operator	
Sensor type		Model		Calibration sample	
		Serial number		Lot number	
Noise level		Flow rate	mL/min	Concentration	

First calibration suspension **Calibration suspension identification number**

Threshold setting						
Count 1						
Count 2						
Count 3						
Count 4						
Count 5						
$\overline{X}$ (particles/mL)						
D_Q						

Second calibration suspension **Calibration suspension identification number**

Threshold setting						
Count 1						
Count 2						
Count 3						
Count 4						
Count 5						
$\overline{X}$ (particles/mL)						
D_Q						

Third calibration suspension **Calibration suspension identification number**

Threshold setting						
Count 1						
Count 2						
Count 3						
Count 4						
Count 5						
$\overline{X}$ (particles/mL)						
D_Q						

6.4 Plot the particle concentrations (in particles larger than the indicated size per millilitre) versus to the corresponding threshold settings, in millivolts, on a $\log_{10}$-$\log_{10}$ graph using only the acceptable data points (as determined in 6.3). Use appropriate statistical regression techniques to define the relationship between concentration and threshold setting.

6.5 Determine the expected particle concentrations for at least six different particle sizes using the appropriate particle size distribution data for the calibration samples. Using the mathematical relationship determined in 6.4, determine the threshold setting expected to yield these concentrations. Do not extrapolate to sizes outside the range given in the particle size distribution data. If any of the threshold settings are less than 1,5 times the threshold noise level of the APC, choose particle concentration data for a larger size that yields an acceptable threshold setting. Set the threshold settings of the APC to these values.

NOTE Throughout this International Standard, reference to size distribution data refers either to particle size, concentration and standard deviation tables available for NIST calibration suspensions or to size, concentration and standard deviation data obtained in Annex F for secondary calibration suspensions.

6.6 Repeat 6.1 to 6.5 using at least six different threshold voltage settings, but use all acceptable data (as determined in 6.3) from both samples to determine the relationship between particle concentration and threshold setting in 6.4 and 6.5.

6.7 Repeat 6.1 to 6.5 once more using at least six different threshold voltage settings, but use all acceptable data (as determined in 6.3) from all three samples to determine the final relationship between particle concentration and threshold setting.

6.8 Construct a calibration curve using the relationship between particle concentration and the threshold setting determined in 6.7. Choose at least 18 different particle sizes from the appropriate particle size distribution data. Choose only particle sizes that fall within the size range actually observed in 6.3 to 6.7. Record in Table 3 these 18 sizes in units of µm(c) and µm(b) and the corresponding concentrations and threshold settings (determined using the concentration versus threshold setting plot constructed in 6.7), where µm(c) refers to particle sizes obtained using primary or secondary calibration samples traceable to NIST SRM 2806 or NIST SRM 2806a, and µm(b) refers to particle sizes obtained using primary or secondary calibration samples traceable to NIST SRM 2806b. Particle sizes in units of µm(c) and µm(b) are mathematically related by Formula (4):

$$d_c = 0{,}898\, d_b \tag{4}$$

where d_c is the particle size given in units of µm(c) and d_b is the particle size given in units of µm(b).

Use this formula to convert µm(b) sizes to µm(c) sizes for sizes smaller than or equal to 38 µm(b) when NIST SRM 2806b traceable calibration samples are used. For sizes greater than 38 µm(b), the particle sizes given in µm(b) and µm(c) are numerically equivalent. Plot the corresponding threshold settings versus particle size. Use the statistical regression technique to define the calibration curve and for interpolation. Do not extrapolate to sizes outside the size range used for calibration.

Some applications may require calibration at larger particle sizes than are reported in SRM 2806. To calibrate APCs for counting particles larger than 50 µm(c), ISO 21501-3 should be used. In any case, the user is cautioned that counting larger-sized particles is subject to many sources of error. Among the most likely sources of error are: a) the settling of large particles during all phases of sample collection, handling and analysis, and b) the inherently poor particle-counting statistics resulting from the typically low concentrations of large particles in hydraulic fluid samples.

ISO 21501-3 is a particle size calibration method that uses monodispersed polystyrene latex spheres. In contrast, the calibration method described in this International Standard is a count calibration method using a polydispersed test dust. Both methods determine the relationship between APC threshold voltage and particle size. A particle size calibration method, such as ISO 21501-3 can be used for particles larger than 50 µm(c) because the NIST particle size distribution used in this International Standard is also based on the projected area diameter of the particles. The signal detected by APCs for particles larger than 50 µm(c) is not strongly dependent on the refractive index of either the particle or the liquid.

If a calibration method based on a polystyrene latex sphere suspension is used, the polystyrene latex spheres shall have a size traceable to national or international standards and have a coefficient of variation of less than 5 %. The polystyrene latex spheres shall be suspended in MIL-PRF-5606[11] hydraulic fluid using the procedure described in Annex D (if the particles are supplied in aqueous suspension), or mixed directly into MIL-PRF-5606[11] using ultrasound to disperse the particles (if the particles are supplied dry).

Table 3 — APC calibration summary

APC ____________ Model ____________ Date ____________
Serial number ____________ Operator ____________
Sensor ____________ Model ____________
Serial number ____________
Noise level ____________ Sample volume ____________ mL Flow rate ____________ mL/min
$C_{V,vol}$ ____________ % Flow rate limits ____________ mL/min
Coincidence error limit ____________ particles/mL
s_R ____________ µm R_R ____________ %
s_L ____________ µm R_L ____________ %
d ____________ µm R ____________ %

Sizing calibration

Calibration sample ____________ Lot number ____________

Size		Threshold setting	Observed particle concentration
µm(c)	µm(b)	mV	Particles/mL

Verification of particle-counting accuracy

Size		Expected particle concentration	Observed particle concentration
µm(c)	µm(b)	Particles/mL	Particles/mL
5	5,6		
10	11,1		

7 Data presentation

7.1 Report all particle sizes obtained using an APC calibrated in accordance with this International Standard, in one of the following ways:

a) as “µm” or “micrometres”, with the following statement: “The sizes quoted in this document were obtained using an APC calibrated in accordance with ISO 11171 and calibration samples traceable to NIST SRM 2806*x*” where “*x*” is the NIST SRM 2806 batch identification letter of the primary calibration samples used to establish traceability for the APC calibration;

b) as “µm(c)”, where the sizes were obtained through the use of an APC calibrated in accordance with ISO 11171 using calibration samples traceable to NIST SRM 2806 or SRM 2806a, or were obtained through the use of an APC calibrated with ISO 11171 using calibration samples traceable to NIST SRM 2806b and the resultant µm(b) sizes mathematically converted to µm(c) sizes using Formula 4;

c) as “µm(b)”, where the sizes were obtained through the use of an APC calibrated in accordance with ISO 11171 using calibration samples traceable to NIST SRM 2806b.

7.2 Retain completed Tables 2, 3, B.1, C.1, and F.1 on file for inspection.

8 Identification statement

Use the following statement in test reports, catalogues and sales literature when electing to comply with this International Standard:

“Calibration of liquid automatic particle counters conforms to ISO 11171, *Hydraulic fluid power — Calibration of automatic particle counters for liquids*.”

Annex A
(normative)

Preliminary APC check

A.1 Figure A.1 is a flow chart of the preliminary APC check procedure. Conduct the preliminary APC check when a new APC is received, or following the repair or readjustment of an APC or sensor.

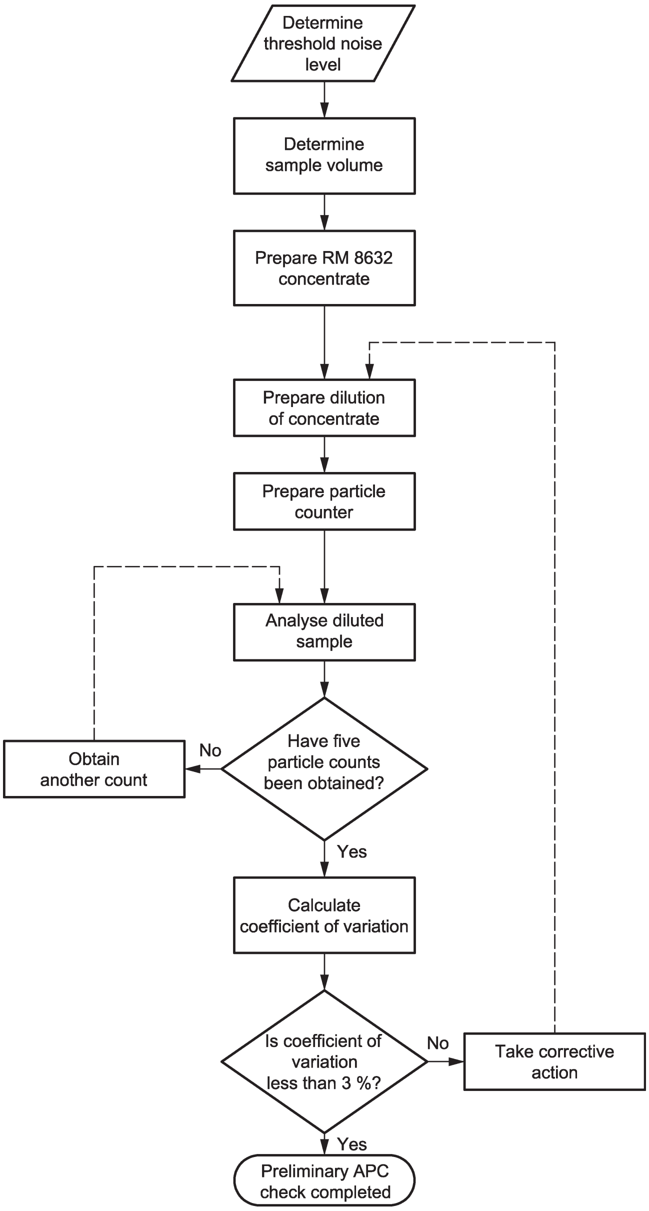


Figure A.1 — Preliminary APC check procedure

A.2 Determine the threshold noise level of the APC under no-flow conditions with clean dilution fluid (4.2) in the sensor. Ensure that the noise levels do not differ significantly for all the channels of the APC. If significant differences occur, readjust the APC. Record the APC and sensor model and serial number, the date and the threshold noise level of the first channel in Tables 2, 3, B.1, C.1 and F.1.

For APCs that use pulse height analysers (as opposed to comparator circuits), determine only the first-channel threshold noise level. Contact the APC manufacturer in order to determine the type of APC being used (pulse height analyser or comparator circuit type).

NOTE The APC manufacturer can provide guidance on how to determine the threshold noise level described in A.2.

A.3 Determine the sample volume actually counted during a particle-counting run using a method with traceability to a national or international standard. Record this value in Table 3 and use it to calculate particle concentrations in all subsequent work.

NOTE Contact the APC manufacturer in order to determine an appropriate method of determining the sample volume.

A.4 Prepare an RM 8632 concentrate of about 100 mg/l as follows:

a) accurately weigh out the required amount of dry RM 8632 (±0,1 mg) and transfer it to a clean sample bottle;

b) fill the bottle about three-quarters full with an accurately measured amount (±1 mL) of clean dilution fluid.

Calculate the mass concentration of dust, γ_A, in milligrams per litre, in the concentrate using Formula (A.1):

$$\gamma_A = \frac{1\,000\,m}{V_0} \tag{A.1}$$

where

m is the mass, in milligrams, of RM 8632;

V_0 is the volume, in millilitres, of clean dilution fluid.

The RM 8632 concentrate prepared in this annex is also used to determine the coincidence error limits (Annex B) and flow rate limits (Annex C) of the APC, as well as to verify particle-counting accuracy (Annex E). For this reason, special care shall be taken in determining the dust concentration of the concentrate and to ensure that the concentrate is not contaminated. Failure to do so can cause an otherwise suitable APC to be deemed unacceptable for use.

A.5 Cover the bottle with a clean closure and vigorously shake the concentrate by hand. Disperse the RM 8632 concentrate ultrasonically for at least 30 s and then shake it on a mechanical shaker for at least 60 s to disperse the dust.

A.6 Calculate the amount of concentrate required to prepare a dilution that is about 25 % of the concentration limit for the sensor recommended by the manufacturer. The number concentration corresponding to a particular mass concentration can be estimated from Table A.1. Add accurately the required amount of concentrate and clean dilution fluid to a clean sample container in order to obtain the correct total volume of diluted RM 8632 suspension. Put a particle-free closure on the sample container.

Table A.1 — Particle size distribution for sensor performance verification (see A.6 and B.4)

Particle size µm(c)	Particle concentration (particles/mL greater than indicated size for a 1 mg/l sample of RM 8632) shall be	
	greater than or equal to	less than or equal to
5	3 300	4 500
6	1 500	2 500
7	660	1 400
8	280	760
9	120	410
10	58	220
11	28	120
12	14	63
13	7,4	34
14	4,1	19
15	2,3	11

A.7 Set the APC to the cumulative mode. Set the lowest threshold setting of the APC to 1,5 times the threshold noise level of the APC. Using clean dilution fluid, adjust the flow rate to the working flow rate. Record the flow rate in Tables 2, 3, B.1, C.1, and F.1.

All of the procedures shall be conducted at the same flow rate. The flow rate limits of the APC are determined in Annex C. Any data obtained at flow rates outside these limits shall be discarded and the corresponding part of the procedure repeated using the proper flow rate.

A.8 Disperse the particles as described in A.5. Degas the diluted sample under vacuum or ultrasonically until the bubbles rise to the surface. Obtain five consecutive particle counts of at least 10 000 particles in the first channel for each measured sample volume. Calculate the coefficient of variation for volume measurement, $C_{\mathrm{V,vol}}$, using Formula (A.2):

$$C_{\mathrm{V,vol}} = \frac{100}{\overline{X}} \sqrt{\frac{N_{\mathrm{C}} \sum_{i=1}^{N_{\mathrm{C}}} X_{\mathrm{i}}^{2} - \left(\sum_{i=1}^{N_{\mathrm{C}}} X_{\mathrm{i}} \right)^{2}}{N_{\mathrm{C}} \left(N_{\mathrm{C}} - 1 \right)}} \tag{A.2}$$

where

N_{C}	is the number of consecutive particle counts performed (i.e. five);
$\overline{X}$	is the mean particle concentration, in particles per millilitre, for the five counts;
X_{i}	is the mean particle concentration, in particles per millilitre, observed for count *i*.

A.9 The $C_{\mathrm{V,vol}}$ shall be 3 % or less in order for the APC to be acceptable in accordance with this International Standard. Record the $C_{\mathrm{V,vol}}$ in Table 3.

Although the $C_{\mathrm{V,vol}}$ is considered to be a measure of the ability of the bottle sampler to reproducibly deliver the indicated volume of fluid, other sources of variability, including sample handling and the counting accuracy of the APC, are also included. Regardless of the source of variability, the $C_{\mathrm{V,vol}}$ shall be 3 % or less before proceeding with the calibration.

Annex B
(normative)

Coincidence error procedure

B.1 Figure B.1 is a flow chart of the coincidence error procedure. The coincidence error limit shall be determined when a new APC is received or after the repair or readjustment of an APC or sensor.

The magnitude of the coincidence error at a given particle number concentration is governed by the physical dimensions of the sensing volume, as well as the particle size distribution of the sample, including those particles too small to be counted. The coincidence error limit determined in this Annex shall be regarded as a benchmark indicator that is useful for routine analyses. For other work, multiple dilutions of a sample shall be analysed to establish that the particle size distribution of the sample does not yield a particle number concentration significantly different from that determined using this Annex.

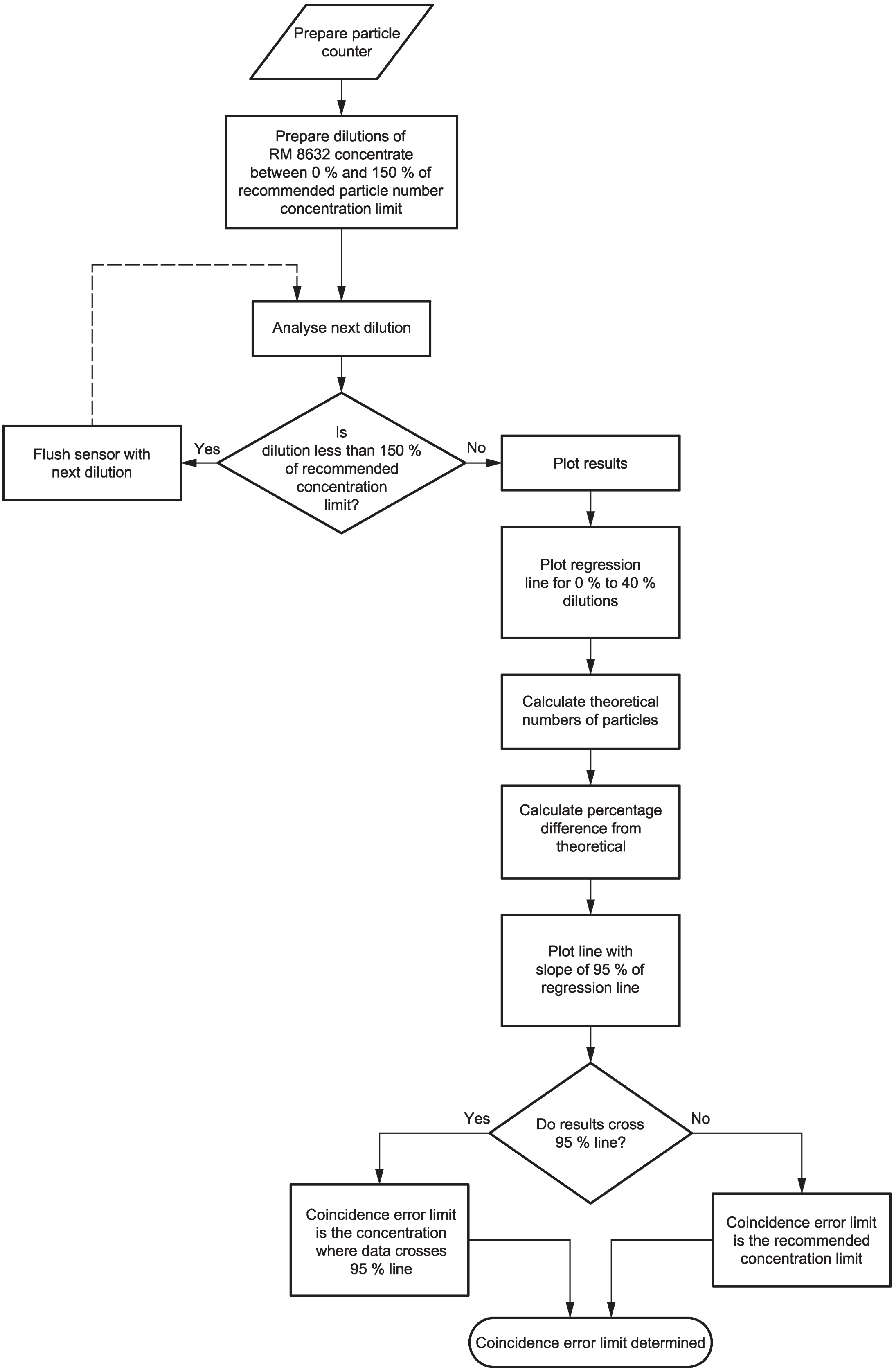


Figure B.1 — Coincidence error determination procedure

B.2 Set the APC to the cumulative mode. Choose a threshold setting corresponding to 1,5 times the threshold noise level of the APC or corresponding to the smallest particle size of interest if it is greater than 1,5 times the threshold noise level. Set the remaining channels to higher settings arranged in ascending order.

B.3 Adjust the flow through the sensor to the working flow rate (see A.7) using clean dilution fluid. Flush the sensor with at least two 150 mL batches of clean dilution fluid.

B.4 Prepare dilutions of the concentrate prepared as in A.4 which are 10 %, 20 %, 30 % ... to 150 % of the particle number concentration limit recommended by the manufacturer for the sensor. Table A.1 can be used to estimate the mass of RM 8632 necessary to obtain the required particle number concentration. Calculate the required amount of concentrate, V_1, in millilitres, needed for each dilution using Formula (B.1):

$$V_1 = \frac{X_M V_S L}{100 X_A} \tag{B.1}$$

where

X_M is the particle number concentration limit, in particles per millilitre, recommended by the manufacturer;

V_S is the final volume, in millilitres, of the diluted sample;

L is the percentage of the limit recommended by the manufacturer;

X_A is the particle number concentration, in particles per millilitre, of the concentrate.

Disperse the RM 8632 concentrate as specified in A.5. Add accurately the required amounts of concentrate, V_1, and clean dilution fluid (4.2) containing antistatic additive to the sample container in order to attain the correct total volume. Put particle-free closures on the sample containers.

B.5 Samples shall be analysed in order of increasing particle number concentration and particles dispersed as specified in A.5. Prior to counting, degas the diluted sample under vacuum or ultrasonically until the bubbles rise to the surface, then remove the bottle from the equipment used to degas the sample and gently turn the sample bottle over at least five times, taking care not to introduce air bubbles into the liquid. Obtain five consecutive particle counts of at least 10 mL for each measured volume.

Evaluate the particle count data using the criteria described in 6.3. If D_Q is acceptable, record in Table B.1 the mass concentration, in milligrams per litre, of the diluted sample, corresponding observed particle number concentrations and mean particle number concentration, $\overline{X}$, before proceeding to B.6. If D_Q is too large, discard the data and suspect an error in the analytical technique. In this case, repeat B.4 and B.5 after taking appropriate corrective action (see Note in 6.3).

Table B.1 — Coincidence error data sheet

t ____ Model ____ Date ____ Serial No. ____ Operator ____ Sensor ____ Model ____ Serial No. ____

Noise level ____ Flow rate ____ Channel setting ____ Flow rate limits ____

Concentration (mg/l)																					
Count 1 (particles/mL)																					
Count 2 (particles/mL)																					
Count 3 (particles/mL)																					
Count 4 (particles/mL)																					
Count 5 (particles/mL)																					
$\overline{X}$ (particles/mL)																					
X_t (particles/l)																					
E																					

Coincidence error limit (particle counts/mL) = ____

$X_t = a\,\gamma$ See A.2, A.7, B.5, B.7, B.8, B.9, B.10 and C.13

a = ____

r = ____

B.6 Flush the sensor with the next sample to be counted using at least three times the sample volume determined in A.3. Repeat B.5 and B.6 for all required dilutions.

B.7 Plot the mean particle number concentration, $\overline{X}$, as a function of the mass concentration of RM 8632, γ, on a linear-linear graph. Determine the linear regression line for the results from the 10 % to 40 % particle number concentration samples using the least-squares method, and set the intercept to 0. The regression equation shall pass through the origin and be of the form given in Formula (B.2):

$$\overline{X} = a\,\gamma \tag{B.2}$$

where a is the slope of the regression line.

Plot the regression line on the graph for particle number concentrations ranging from 0 % to 150 % of the limit recommended by the manufacturer. This line defines the theoretical relationship between the number of particles and the mass concentration. Record in Table B.1 the slope, a, and correlation coefficient, r, obtained by regression analysis.

B.8 Use the regression formula obtained in B.7 to calculate the theoretical number of particles for each particle number concentration, X_t. Record the value of X_t for each particle number concentration in the appropriate cell in Table B.1.

B.9 Calculate the difference, E, expressed as a percentage, between the theoretical, X_t, and mean, $\overline{X}$, particle number concentration for each dilution using Formula (B.3):

$$E = \frac{\overline{X} - X_t}{X_t} \times 100 \tag{B.3}$$

Record in Table B.1 the value of E for each particle number concentration.

B.10 Determine the coincidence error limit of the sensor by plotting a line with a slope of 95 % of the theoretical relationship (see B.7) and the same intercept, b, as the theoretical relationship on the graph constructed in B.7. The lowest particle number concentration, in particles per millilitre, at which the data line crosses the 95 % line is the coincidence error limit of the sensor. If the data line does not cross the 95 % line, report the limit recommended by the manufacturer as the coincidence error limit of the sensor and use this value for all subsequent work. Record the coincidence error limit, in particles per millilitre, of the sensor in Table 3 and Table B.1.

Annex C
(normative)

Flow rate limit determination

C.1 Figure C.1 is a flow chart of the flow rate limit determination procedure. Perform the flow rate limit determination when a new APC is received, or following the repair or readjustment of an APC or sensor.

IMPORTANT — It is essential that the flow rate limits determined in this Annex be followed in all APC calibration and sample analysis work. APC calibration is sensitive to flow rate. At high flow rates, contaminant particles may not be present in the sensing volume long enough for the electrical signal to develop fully. Also, the time interval separating successive particles may be so small that the electronics are unable to distinguish them as individual particles.

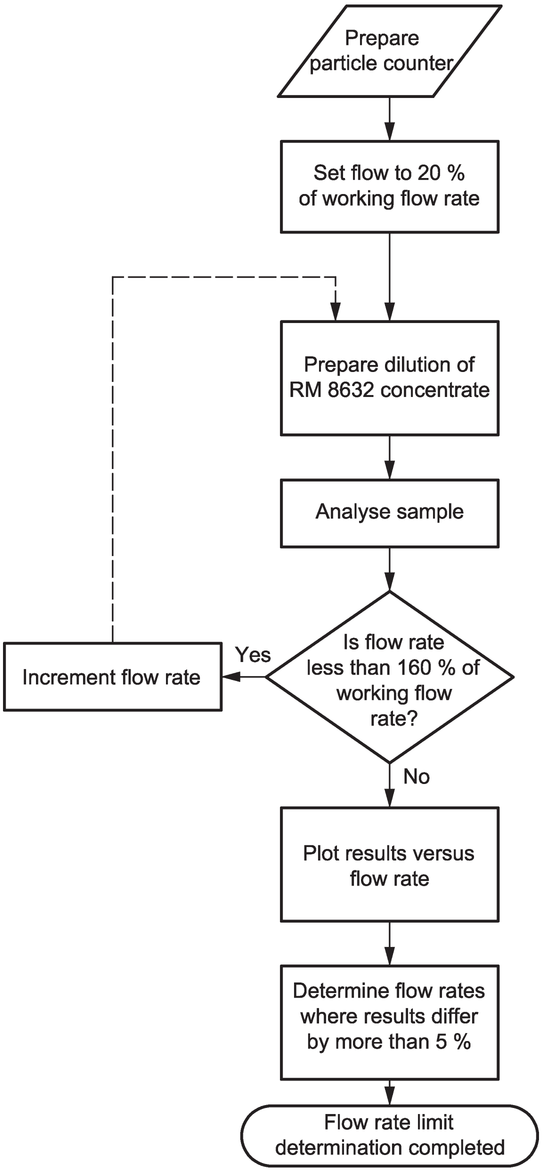


Figure C.1 — Flow rate limit determination procedure

C.2 Record the date, operator, APC and sensor model and serial numbers in Table C.1. Determine whether the flow rate of the APC bottle sampler can be adjusted by the operator (adjustable flow rate type) or is designed to deliver the sample at a fixed flow rate (fixed flow rate type)

Table C.1 — Flow rate limit worksheet

APC ____________ Mode ____________ Date ____________
Serial no. ____________ Operator ____________
Sensor ____________ Model ____________
Serial no. ____________

Noise level ____________
Working flow rate ____________
Flow rate limits ____________
Coincidence error limit ____________

	Particle counts							
n_{fr}	1	2	3	4	5	6	7	8
Flow rate								
Count 1								
Count 2								
Count 3								
Count 4								
Count 5								
$\overline{X}$								

C.3 If the APC is of the adjustable flow rate type, proceed to C.4. If it is of the fixed flow rate type, proceed to C.14.

C.4 Set the APC to the cumulative mode. Use the calibration curve determined in 6.8 to set the first channel to 5 µm(c) or the smallest particle size of interest. The data from other channels are ignored during the flow rate limit determination. Let the flow rate number, $n_{fr} = 1$.

NOTE All channels are typically arranged in order of increasing threshold setting, unless otherwise recommended by the APC manufacturer.

C.5 Calculate the flow rate, q, to be used using Formula (C.1):

$$q = \frac{n_{fr}\ q_m}{5} \qquad \text{(C.1)}$$

where

n_{fr} is the flow rate number;

q_m is the working flow rate, in millilitres per minute.

Using clean dilution fluid, establish a flow rate approximately equal to q.

C.6 Prepare a sample of RM 8632 in clean dilution fluid as specified in A.4 to A.6.

C.7 Shake the sample vigorously by hand. Disperse the sample ultrasonically for at least 30 s and then shake it on a mechanical shaker for at least 1 min to disperse the dust.

C.8 Degas the sample under vacuum or ultrasonically until the bubbles rise to the surface and gently turn the sample bottle over at least five times, taking care not to introduce air bubbles into the liquid. Obtain five consecutive particle counts for volumes of at least 10 mL each for the dust sample prepared in C.5. Record the flow rate and the number of particles observed at 5 µm(c) or the smallest particle size of interest for each value of N shown in Table C.1.

C.9 Evaluate the particle count data using the criteria described in 6.3. If D_Q is less than or equal to the appropriate value shown in Table C.2, proceed to C.9. If D_Q is greater than the appropriate value shown in Table C.2, discard the data and suspect an error in the analytical technique. Repeat C.6 to C.8 after taking appropriate corrective action (see Note in 6.3).

Table C.2 — Maximum allowable differences, expressed as percentages, in particle counts between runs (see 6.3 and D.4)

If $\overline{X}$ (average number of counts) is		**Step 1: use these values for the maximum allowable D_Q for individual samples**	**Step 2: use these values for the maximum allowable C_V when the number of samples analysed is (see F.5 and E.9)**				
greater than or equal to	but less than	(see 6.3, B.5, C.9, D.4, D.8, and F.5)	$N = 3$	$N = 6$	$N = 10$	$N = 20$	$N = 40$
10 000		11,0	7,8	5,3	4,0	2,7	1,8
5 000	10 000	11,3	7,8	5,3	4,0	2,7	1,9
2 000	5 000	11,9	7,9	5,4	4,1	2,9	2,1
1 000	2 000	13,4	8,0	5,6	4,3	3,1	2,4
500	1 000	15,6	8,2	5,9	4,7	3,6	2,9
200	500	19,3	8,9	6,8	5,7	4,7	4,1
100	200	27,5	10,0	8,0	7,0	6,1	5,5

C.10 Calculate the mean observed particle concentration, $\overline{X}$, in particles per millilitre, and record this result in Table C.1.

C.11 If $n_{fr} \geq 8$, proceed to C.12. If not, increase n_{fr} by 1 and repeat C.4 to C.10.

C.12 Plot the number of counts as a function of the flow rate on a linear-linear graph and connect the data points by a smooth curve.

C.13 Determine the flow rates above and below the working flow rate that yield counts that differ by more than 5 % from those observed at the working flow rate. These are the flow rate limits of the APC. Record the flow rate limits in Tables 3, B.1, and C.1.

C.14 Establish a flow rate approximately equal to the working flow rate. Prepare and analyse five samples as specified in C.6 to C.9. Record the number of counts observed at 5 µm(c) or the smallest particle size of interest for each sample in Table C.1.

C.15 Calculate the mean, standard deviation and coefficient of variation for the five samples analysed in C.14. If the coefficient of variation of the five samples is less than or equal to 3 %, then the APC meets the flow rate specifications of this International Standard. If the coefficient of variation of the five samples is greater than 3 %, the APC is unacceptable and needs to be serviced or replaced.

Ideally, the APC is always used at the working flow rate. APCs shall be calibrated at each flow rate for which they are used. The APC shall always be used within the flow rate limits determined in this Annex.

If any of the data used for size calibration were obtained using flow rates outside these limits, the size calibration shall be repeated within these flow rate limits.

Annex D
(normative)

Resolution determination

D.1 Figure D.1 is a flow chart of the resolution determination procedure. Perform the resolution determination when a new APC is received, or following the repair or readjustment of an APC or sensor.

Resolution shall be determined using a polystyrene latex sphere suspension with a nominal particle size of 10 µm (4.1). Because resolution varies as a function of particle size, operators may also wish to determine the resolution at smaller sizes when a high degree of particle-counting accuracy is required. The smallest size at which resolution should be measured is 2,1 times the threshold noise level of the APC (see Annex A). Use the calibration curve determined in Clause 6 to determine the corresponding size as discussed in D.3. Operators should be aware that optical artefacts may occur that yield apparently poor resolution results. If poor resolution is obtained, contact the APC manufacturer to determine the reason and discuss the appropriate action to be taken.

NOTE The procedures can be carried out manually or by the use of appropriate software for those APCs with microprocessor controls. Alternatively, resolution can be determined through the use of an MCA. Such practice is acceptable, provided the relationship between the measured voltage of the MCA and the APC threshold setting has been established. In general, an MCA provides faster, more accurate results than the differential half-count method.

D.2 Prepare a sample of polystyrene latex sphere suspension as follows:

a) calculate the volume of polystyrene latex sphere concentrate (4.1) that needs to be added to clean aerosol OT dilution fluid (4.3) to achieve a particle concentration that is approximately 25 % of the concentration limit for the sensor;

b) estimate the volume of concentrate, V_0, in millilitres, needed to make the polystyrene latex sphere suspension using Formula (D.1):

$$V_0 = \frac{V_S\ X_L\ \pi\ d_L^3}{6 \times 10^{10} \times w_s} \tag{D.1}$$

where

V_S is the final volume, in millilitres, of the polystyrene latex sphere suspension;

X_L is the desired particle concentration, in particles per millilitre;

d_L is the mean diameter, in micrometres, of the polystyrene latex spheres indicated on the certificate of analysis (see 4.1);

w_S is the solids content, expressed as a percentage mass fraction, in the polystyrene latex sphere concentrate (this value is typically supplied by the manufacturer of the polystyrene latex spheres);

c) shake the concentrate vigorously by hand, then disperse it ultrasonically for 30 s and finally shake it mechanically for at least 1 min to suspend the polystyrene latex spheres;

d) prepare a suspension of polystyrene latex spheres by adding the required amount of polystyrene latex sphere concentrate, V_0, to a sample container approximately 75 % full of clean aerosol OT dilution fluid (4.3) and dispersing the sample by mechanical shaking for at least 1 min.

The final dilution of the polystyrene latex sphere suspension which is actually counted shall be clear in appearance. Cloudiness in the samples is evidence of insufficient dilution and shall be corrected before proceeding.

D.3 Set the APC to the differential mode. Use the calibration curve (see Clause 6) to estimate the threshold setting that corresponds to the polystyrene latex sphere size, as follows:

a) set the first channel to 1,5 times the threshold noise level of the APC;

b) set the third channel to the threshold voltage setting corresponding to the polystyrene latex sphere size;

c) set the second channel to the threshold voltage setting corresponding to 0,72 times the threshold voltage setting of the third channel;

d) set the fourth channel to the threshold voltage setting corresponding to 1,32 times the threshold voltage setting of the third channel.

Only the differential particle counts of the second and third channels are used.

NOTE 1 D.3, D.4 and D.5 specify the moving-window differential half-count method for determining the threshold setting that corresponds to the median polystyrene latex sphere size. These data are critical to determining sensor resolution. Inaccurate determination of the threshold setting corresponding to the median polystyrene latex sphere size can result in apparently poor sensor resolution and failure of the APC to pass the sensor performance specification.

NOTE 2 Due to differences in the optical properties of the polystyrene latex spheres and the calibration dust (4.4), the apparent particle size obtained using an APC calibrated in accordance with this International Standard differs from the size stated by the supplier of the polystyrene latex spheres. As a first approximation, choose a particle size that is 10 % to 50 % larger than the particle size stated by the supplier of the polystyrene latex spheres.

NOTE 3 To determine the half-count setting more rapidly, perform D.3 to D.5, but obtain only one count (instead of five as indicated in D.4) of at least 500 particles in the second channel. Readjust the channels as necessary and repeat the process until the half-count condition described in D.6 is achieved. Following this, repeat D.3 to D.6 using five counts as described in D.4.

D.4 Disperse the diluted polystyrene latex sphere sample by mechanical shaking for at least 1 min. Degas the sample under vacuum or ultrasonically until the bubbles rise to the surface. Obtain five consecutive particle counts, each consisting of at least 10 mL and 2 500 particles in the second channel. Evaluate the particle count data using the criteria described in 6.3. If D_Q is less than the appropriate value shown in Table C.2, proceed to D.5. If not, discard the data and suspect an error in the analytical technique. Repeat D.2 to D.4 after taking appropriate corrective action (see Note in 6.3).

D.5 Calculate the difference, D, expressed as a percentage, between the second and third channels using Formula (D.2):

$$D = \left(1 - \frac{N_2}{N_3}\right) \times 100 \qquad \text{(D.2)}$$

where

N_2 is the mean number of particles counted for the second channel;

N_3 is the mean number of particles counted for the third channel.

If the absolute value of D is less than or equal to 3 %, the threshold setting of the third channel corresponds to the size of the polystyrene latex sphere. In this case, proceed to D.6. If not, and if the value of D is negative, the threshold settings in D.3 are too high. If D is positive, the threshold settings in D.3 are too low. Repeat D.3 to D.5 using threshold settings for the second, third and fourth channel which have been readjusted. If no threshold setting yields an absolute value for D less than 3 %, the threshold setting of the third channel yielding the minimum absolute value of D corresponds to the size of the polystyrene latex sphere.

D.6 Using the calibration curve obtained in Clause 6, determine the particle size that corresponds to the threshold setting of the third channel. Determine the sizes and threshold settings that correspond to 0,9 and 1,1 times this size.

D.7 Set the first five channels of the APC as follows:

a) set channel A, the first channel, to the threshold voltage corresponding to 0,72 times the threshold voltage of channel C;

b) set channel B, the second channel, to correspond to a size 0,9 times that of channel C;

c) keep channel C, the third channel, at the threshold setting corresponding to the size of the polystyrene latex sphere determined in D.5;

d) set channel D, the fourth channel, to correspond to a size 1,1 times that of channel C;

e) set channel E, the fifth channel, to the threshold voltage corresponding to 1,32 times the threshold voltage of channel C.

Only differential counts are used to determine resolution.

D.8 Disperse the diluted polystyrene latex sphere sample by mechanical shaking for at least 1 min. Degas the sample under vacuum or ultrasonically until the bubbles rise to the surface. Obtain five consecutive particle counts, each consisting of at least 10 mL and 2 500 particles in the second channel. Evaluate the particle count data using the criteria described in 6.3. If D_Q is less than the appropriate value shown in Table C.2, proceed to D.9. If not, discard the data and suspect an error in the analytical technique. Repeat D.8 after taking appropriate corrective action (see Note in 6.3).

D.9 Calculate the mean differential particle concentration for each channel.

D.10 Calculate the apparent standard deviation of the APC using Formulae (D.3) and (D.4):

$$s_\mathrm{L} = \frac{d}{6\ln\left[1+2\left(N_\mathrm{B}/N_\mathrm{A}\right)\right]} \tag{D.3}$$

$$s_\mathrm{R} = \frac{d}{6\ln\left[1+2\left(N_\mathrm{C}/N_\mathrm{D}\right)\right]} \tag{D.4}$$

where

s_L is the apparent left or negative-side standard deviation, in micrometres;

s_R is the apparent right or positive-side standard deviation, in micrometres;

d is the apparent polystyrene latex sphere size, in micrometres, obtained using the calibration curve obtained in Clause 6 and the threshold setting for the third channel;

N_A is the mean number of particles counted for channel A;

N_B is the mean number of particles counted for channel B;

N_C is the mean number of particles counted for channel C;

N_D is the mean number of particles counted for channel D.

D.11 Calculate the positive and negative-side resolution (R_R and R_L, respectively) using Formulae (D.5) and (D.6):

$$R_L = \frac{100\sqrt{s_L^2 - s_I^2}}{d} \tag{D.5}$$

$$R_R = \frac{100\sqrt{s_R^2 - s_I^2}}{d} \tag{D.6}$$

where s_I is the standard deviation of the polystyrene latex sphere size, in micrometres, stated by the supplier.

The APC resolution, R, is R_R or R_L, whichever is greater. Record d, s_R, s_L, R_R, R_L, and R in Table 3.

D.12 The resolution of the sensor is acceptable if R is less than 15 % for polystyrene latex spheres with a nominal size of 10 µm (see 4.1) and the absolute value of the difference between R_R and R_L is less than or equal to 5 %. If R equals or exceeds 15 %, or the absolute value of the difference between R_R and R_L is greater than 5 %, the APC is unacceptable and needs to be serviced or replaced. Alternatively, there may be an error in the procedure used to prepare or analyse the polystyrene latex sphere sample, or the polystyrene latex spheres themselves might not meet the requirements of 4.1. In this case, the error shall be identified and corrected, and the resolution determination procedure specified in this Annex repeated

NOTE One method of correcting poor resolution is to clean the system in accordance with the instructions of the APC manufacturer.

D.13 If the APC fails to meet the resolution requirements of D.12, check the polystyrene latex sphere size distribution using an MCA or APC in the cumulative mode as follows.

a) Determine the size range corresponding to 0,85 and 1,15 times the size of the polystyrene latex spheres as determined in D.6. Divide the size range into at least 10 equal size increments and determine the corresponding threshold settings using the calibration curve obtained in Clause 6. Set the channels to as many of these threshold settings as possible.

b) Prepare and analyse a polystyrene latex sphere sample as described in D.2 and D.4, but with the APC set to the cumulative mode. Repeat the analysis at different threshold settings until sufficient numbers of particles have been obtained for all of the size increments. When repeating the analysis, do not change the channel corresponding to the smallest size.

c) Normalize the results of each run by expressing the number of counts in each channel as a percentage of the total counts observed in the smallest channel for the same run. Compile the normalized results in order of ascending size in a table. For each size, calculate the differential percentage by subtracting the cumulative results for each size from the cumulative results for the next largest size.

d) Plot the differential results versus size and draw a smooth curve through the data.

The curve plotted in item d) should be Gaussian (bell-shaped) in appearance with no secondary peaks, and the differential percentages at the smallest and largest sizes should approach zero. If this is observed and the APC failed the resolution requirements of D.12, the APC needs to be repaired or serviced or else a larger number of size increments is needed to verify the polystyrene latex sphere size distribution. If this is not observed, suspect a problem with the polystyrene latex spheres or with the sample preparation. Regardless of why the APC failed, acceptable resolution as defined in D.12 shall be obtained for the APC to meet the requirements of this International Standard.

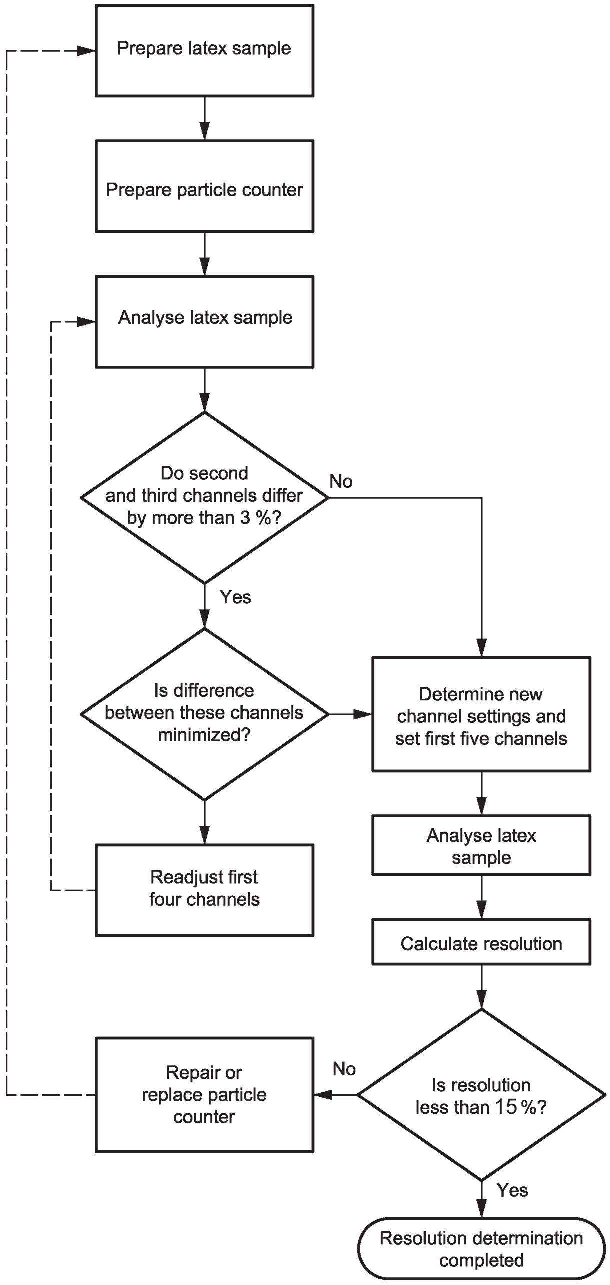


Figure D.1 — Resolution determination procedure

Annex E
(normative)

Verification of particle-counting accuracy

E.1 Figure E.1 is a flow chart of the procedure to verify particle-counting accuracy. Verify sensor performance when a new APC is received, or following the repair or readjustment of an APC or sensor.

E.2 If the resolution determined in Annex D meets the acceptance criteria described in D.12, proceed to E.3. If not, the APC system is unacceptable and needs to be serviced or replaced.

E.3 Prepare three identical samples containing 1,00 mg/l of RM 8632 (4.7) suspended in clean dilution fluid.

IMPORTANT — It is critical that the dust concentration in this sample be accurate because the results obtained by analysis of it are compared to the values given in Table A.1. Inaccuracies in sample preparation or counting can contribute to deviations from the values in Table A.1 which could result in rejection of an otherwise acceptable sensor. The three 1,00 mg/l RM 8632 samples may be prepared from the concentrate prepared in A.4. When this method is used, be careful to avoid errors introduced by weighing, volume measurement and settling. Alternatively, one may wish to consider purchasing 1,00 mg/L RM 8632 samples from a reliable source that certifies its work.

E.4 Disperse the sample ultrasonically for at least 30 s and then shake it on a mechanical shaker for at least 1 min to disperse the dust. Keep shaking the sample until it is to be analysed.

E.5 Set the APC to the cumulative mode and set the threshold voltage settings to at least six different sizes between 5 µm and 15 µm (inclusive). These settings shall be greater than 1,5 times the threshold noise level of the APC and shall include the smallest particle size of interest as well as 5 µm(c) and 10 µm(c). Use the calibration curve previously determined in Clause 6 to determine the threshold voltage settings corresponding to these sizes. If the sensor is incapable of counting at one or more of these sizes, choose alternative sizes within this range.

E.6 Adjust the flow rate to the working flow rate.

E.7 Degas the sample under vacuum or ultrasonically until the bubbles rise to the surface, and then gently turn the sample bottle over at least five times, taking care not to introduce air bubbles into the liquid. Obtain five consecutive particle counts, each consisting of at least 10 mL and 10 000 particles at the smallest particle size for the sample. If the data meet the quality criteria specified in 6.3, proceed to E.8. If they do not, discard the data and suspect an error in the analytical technique. Repeat E.3 to E.7 after taking appropriate corrective action (see Note in 6.3).

E.8 Repeat E.4 to E.7 until all three samples have been analysed.

E.9 For each particle size, calculate the mean particle concentration, $\overline{X}$, and the coefficient of variation, C_V, expressed as a percentage, for the three samples using Formula (E.1):

$$C_{\mathrm{V}} = \frac{100}{\overline{X}} \sqrt{\frac{N \sum_{i=1}^{N} \overline{X}_{\mathrm{i}}^{2} - \left(\sum_{i=1}^{N} \overline{X}_{\mathrm{i}} \right)^{2}}{N(N-1)}} \tag{E.1}$$

where

$\overline{X}_{\mathrm{i}}$ is the mean particle concentration at a particular size for sample *i*;

N is (in this annex only) the total number of samples.

If the C_{V} for any particle size is smaller than the corresponding values in Table C.2, compare the mean for each size to the appropriate limits given in Table A.1. If $\overline{X}$ falls within the limits given in Table A.1 for all six sizes, sensor performance is verified. Report the published particle concentration and mean particle concentration for each size in Table 2.

If the C_{V} for any particle size is larger than the corresponding values in Table C.2, discard the data and suspect an error in the sample preparation or analytical technique. Repeat E.3 to E.9 after taking appropriate corrective action.

If $\overline{X}$ falls outside the limits given in Table A.1 for particle sizes larger than or equal to 5 µm(c), then the APC system is unacceptable and needs to be serviced or replaced. Alternatively, there may be an error in the procedure used to prepare or analyse the verification samples in the calibration. In the latter case, the error shall be identified and corrected and the procedure in Annex E repeated.

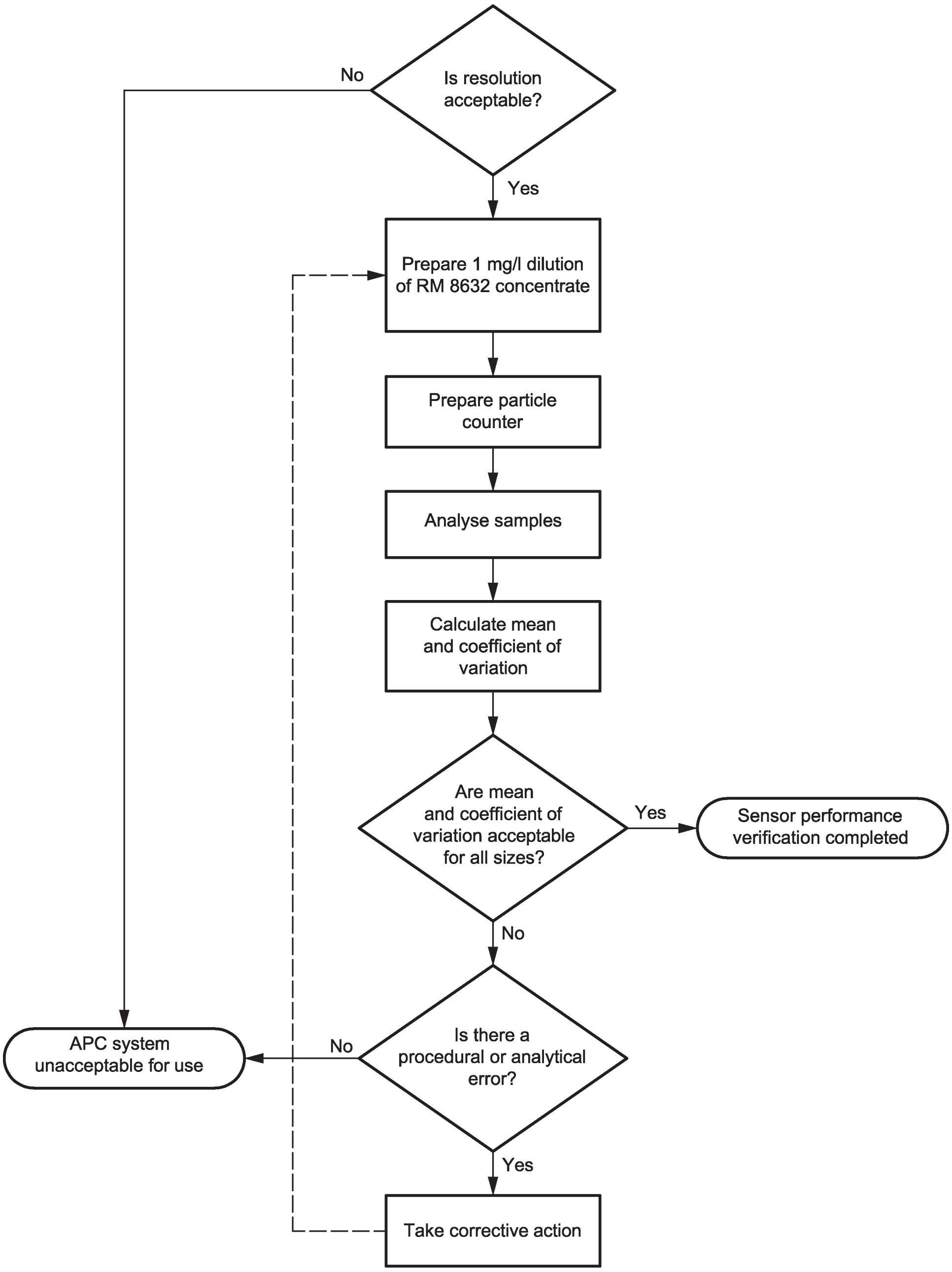


Figure E.1 — Verification of particle-counting accuracy

Annex F
(normative)

Preparation and verification of bottles of secondary calibration suspensions

F.1 Figure F.1 is a flow chart of the procedure to prepare secondary calibration suspensions. This Annex shall be used to prepare secondary calibration suspensions when, for economic or other reasons, it is desirable to utilize secondary calibration suspensions instead of NIST calibration suspensions. For example, if more than one APC needs to be calibrated, the use of secondary calibration suspensions may be more practical.

To prepare secondary calibration suspensions, one APC, calibrated using NIST primary calibration suspensions that conform to Clauses 5 and 6, is needed for reference. Calibration of the reference APC with secondary calibration samples is unacceptable. Bottles of secondary calibration suspensions prepared in accordance with this Annex can be used for sizing calibration instead of NIST primary calibration suspensions employed for other purposes.

F.2 Prepare a minimum of 30 bottles of secondary calibration samples using a properly validated multi-pass test system in accordance with ISO 16889 or a similar system capable of mixing a total volume of suspension of at least 8 l. Use ISO MTD (4.6) to prepare the samples. Use clean dilution fluid (4.2) as the fluid. The dust concentration shall be such that the particle concentration does not exceed 75 % of the coincidence error limit for the sensor. Collect and store samples prepared in this manner in clean, sealed containers with closures (4.9).

Due to possible batch-to-batch variability in test dusts, the use of NIST RM 8631 is recommended.

NOTE The shelf-life of the secondary calibration samples is the same as that of the NIST primary calibration suspensions.

F.3 Calibrate an APC in accordance with Clauses 5 and 6 using NIST primary calibration suspensions (4.4).

The APC used to verify the bottles of secondary calibration suspension samples shall meet all APC performance specifications described in this International Standard. Where a choice of APCs is available, the APC with the best performance, in terms of coincidence error characteristics, threshold noise level, resolution, etc., shall be used.

F.4 Select particle sizes and corresponding threshold voltage settings using the criteria specified in 6.2.

F.5 Analyse in accordance with 6.2 and 6.3 every fifth sample bottle prepared in F.2.

Evaluate the particle count data for each sample bottle using the criteria described in 6.3. If D_Q is less than the appropriate value shown in Table C.2, consider the data from that sample to be acceptable. If D_Q is greater than the appropriate value shown in Table C.2, suspect an error in the analytical technique, discard the data and analyse the next consecutive sample bottle only after taking appropriate corrective action (see Note in 6.3).

For each threshold setting, calculate the mean, standard deviation and coefficient of variation using Formula (E.1). If the C_V for each threshold setting is less than the corresponding value taken from Table C.2, then the mean, standard deviation and corresponding particle size data define the particle size distribution for these secondary calibration suspensions and can be used for secondary calibration of other APCs. Complete Table F.1 by filling in the required data. If the C_V for each threshold setting is greater than the corresponding value taken from Table C.2, re-evaluate the sample preparation procedure, take appropriate corrective action and repeat F.2 to F.5.

Primary and secondary calibration samples shall not be collected and reused.

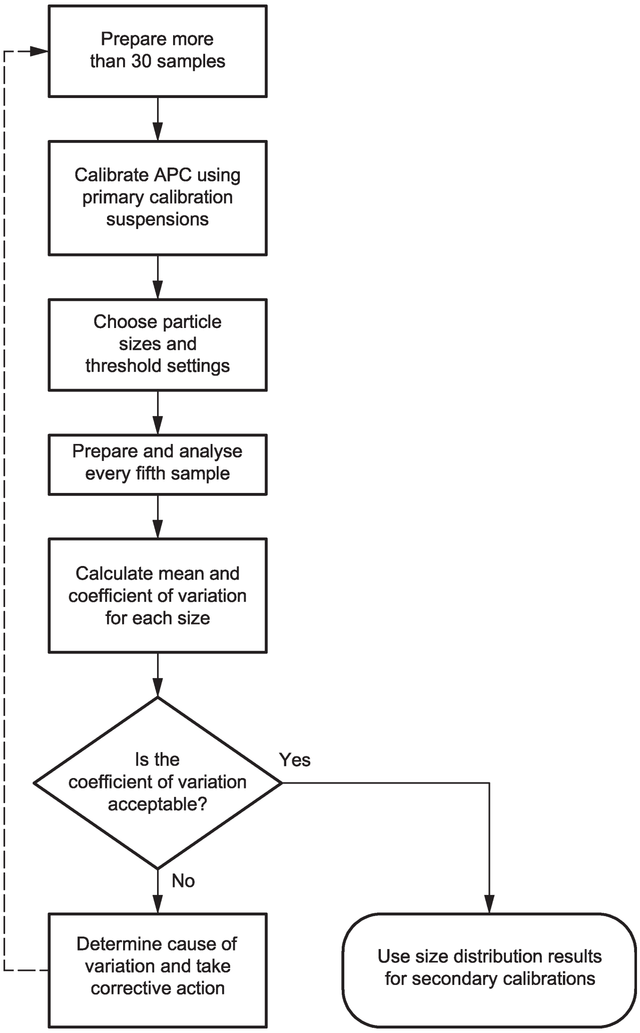


Figure F.1 — Procedure for preparation and verification of secondary calibration suspensions

Table F.1 — Secondary calibration suspension validation data sheet

APC ______ Model ______ Date ______
Serial no. ______ Operator ______
Sensor ______ Model ______ Lot no. ______
Serial no. ______ Concentration ______
Noise level ______ Flow rate ______

Particle size, µm(c)							
Threshold setting							
	Mean particle concentration for indicated sample bottle (particles/mL)						
Sample bottle 1							
Sample bottle 6							
Sample bottle 11							
Sample bottle 16							
Sample bottle 21							
Sample bottle 26							
Sample bottle 31							
Sample bottle 36							
Sample bottle 41							
Sample bottle 46							
Sample bottle 51							
Sample bottle 56							
Sample bottle 61							
Sample bottle 66							
Sample bottle 71							
Sample bottle 76							
Sample bottle 81							
Sample bottle 86							
Sample bottle 91							
Sample bottle 96							
Sample bottle 101							
$\overline{X}$ (particles/mL)							
Standard deviation							
C_V							

Annex G
(informative)

APC calibration round robin

G.1 Background

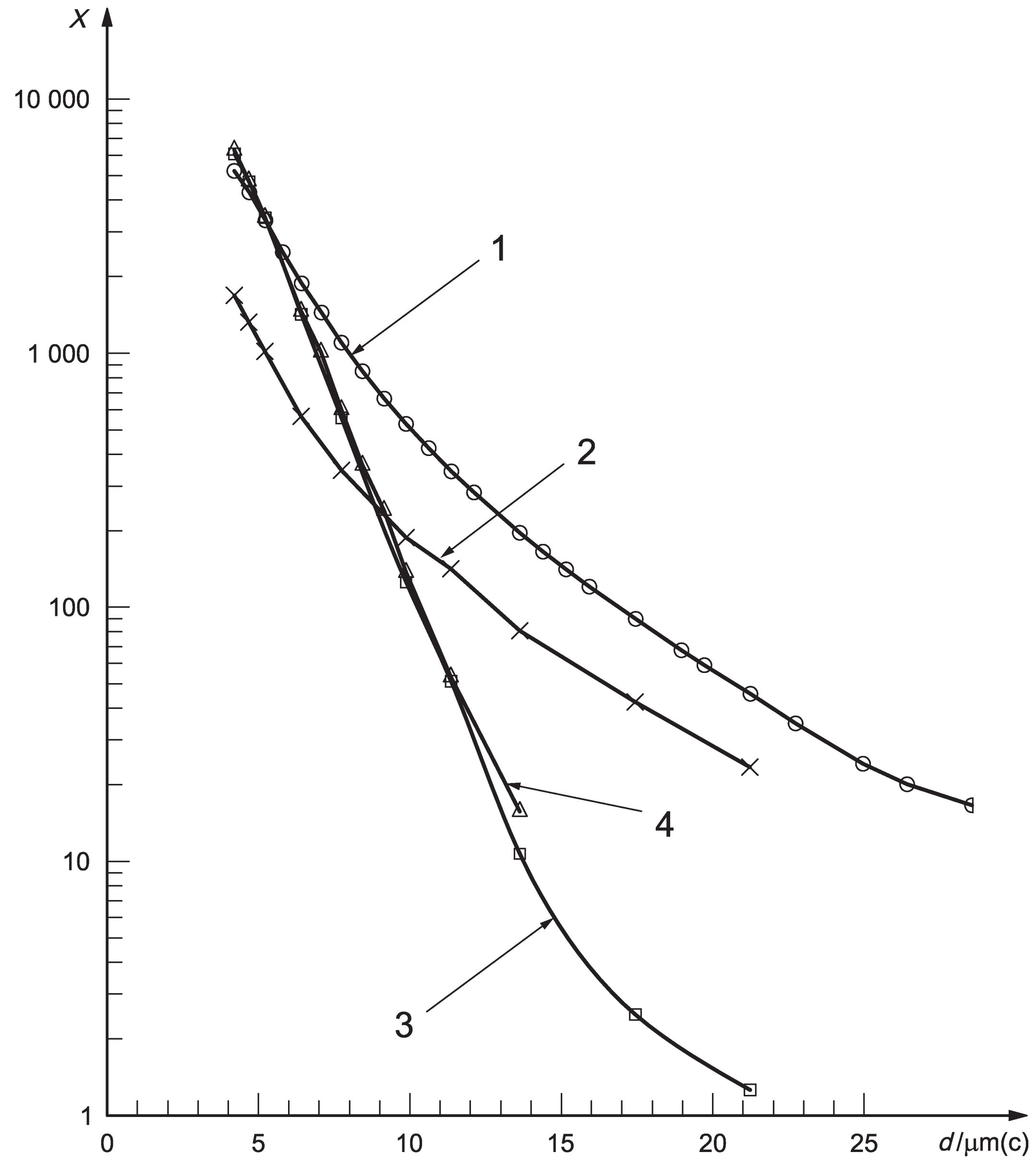
A round robin was conducted to evaluate an earlier draft of this International Standard. Only minor technical differences exist between the earlier draft and this International Standard. Laboratories participated in the round robin numbered 27. Results were obtained from 29 APCs, including 24 light extinctions sensors and five light-scattering sensors. Together, the round robin included APCs produced by six different particle counter manufacturers and 12 different models of particle sensors.

Participants were supplied with samples of calibration fluid, 10 µm polystyrene latex spheres suspended in oil, and dry ISO UFTD. Because the round robin was conducted prior to NIST completing certification of SRM 2806a, participants were provided with a size distribution of the calibration fluid determined using the ISO 4402 AC Fine Test Dust (ACFTD) calibration method. When the NIST certification of the primary calibration samples was complete, the ACFTD particle sizes originally obtained in the round robin were converted to the new ISO 11171 particle sizes. To establish a correlation between particle sizes obtained using ISO 4402 and ISO 11171, three laboratories from the original round robin calibrated five APCs in accordance with ISO 4402 and ISO 11171. APCs representing three different manufacturers and five different types of sensor, including two light-scattering and three light-extinction sensors, were involved. Table G.1 shows the resultant correlation between ISO 4402 ACFTD particle sizes and ISO 11171 NIST traceable particle sizes. This table was used to convert the ACFTD sizes obtained in the original round robin to NIST sizes.

Table G.1 — Correlation between particle sizes obtained using APCs calibrated in accordance with ISO 11171 (NIST suspension) and the withdrawn ISO 4402:1991 (ACFTD)

Particle size determined using an APC calibrated in accordance with	
ISO 11171 (NIST suspension) µm(c)	**ISO 4402:1991 (withdrawn) (ACFTD)** µm
4	< 1
5	2,7
6	4,3
7	5,9
8	7,4
9	8,9
10	10,2
15	16,9
20	23,4
25	30,1
30	37,3

Figure G.1 shows the particle size distributions of the four types of dust sample: calibration sample, verification sample, sample A and sample B. The lines represent the mean results reported by the participating laboratories for the type of sample indicated. The calibration samples were suspensions of ISO MTD in clean dilution fluid. These were prepared by the same laboratory that prepared the NIST primary calibration suspension samples. ISO MTD is also used in the multi-pass filter test procedure of ISO 16889. Verification samples were prepared from dry ISO UFTD by participating laboratories as described in Annex E. The size distribution of ISO UFTD resembles that of downstream samples taken during a multi-pass filter test. Like the verification samples, sample A was a 1,00 mg/l suspension of ISO UFTD in clean dilution fluid, except that these samples were all prepared by the same laboratory. Sample B was a suspension of 0 µm to 30 µm dust[1] in clean dilution fluid. Its particle size distribution resembles that of upstream samples taken during a multi-pass filter test.



Key

X	concentration, particles/mL	2	sample B
d	particle size	3	sample A
1	calibration sample	4	verification sample

Figure G.1 — Particle size distribution of round-robin samples

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[1] Product available from Powder Technology Inc. This information is given for the convenience of users of this document, and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

G.2 Results

The round-robin data for the verification samples, for sample A and for sample B were statistically analysed. The results are summarized in Tables G.2 to G.4. The mean, standard deviation and 95 % confidence intervals were calculated based on the logarithm of the cumulative particle concentration data. Variability in the data is assumed to be primarily the result of differences in calibration. The actual sizes determined by the laboratories should be normally distributed. Because cumulative concentration varies with size in a logarithmic fashion, the logarithm of the corresponding observed cumulative concentrations is expected to be normally distributed. The mean, $\overline{X}_{\log}$, standard deviation, s, and coefficient of variation, $C_{\mathrm{V,log}}$, are defined by Formulae (G.1), (G.2), and (G.3):

$$\overline{X}_{\log} = \frac{\sum_{i=1}^{N} \log \overline{X}_i}{N} \tag{G.1}$$

$$s = \sqrt{\frac{\sum_{i=1}^{N} \left(\log \overline{X}_{\mathrm{i}} - \overline{X}_{\log} \right)^2}{N-1}} \tag{G.2}$$

$$C_{\mathrm{V,log}} = \frac{100s}{\overline{X}_{\log}} \tag{G.3}$$

where

$\overline{X}_i$ is the mean particle concentration data reported by laboratory *i* at a specific size;

N is the number of APCs submitting data for the indicated size and type of sample.

Values of $\overline{X}_{\log}$, s, and $C_{\mathrm{V,log}}$ are reported in the statistics sections of Tables G.2, G.3, and G.4.

Using $\overline{X}_{\log}$ and s, the mean and 95 % confidence intervals for the particle concentration and size for each type of sample were estimated. The mean, $\overline{X}_{\mathrm{G}}$, upper, $\overline{X}_{\mathrm{U}}$, and lower, $\overline{X}_{\mathrm{L}}$, 95 % confidence intervals for the particle concentration are defined by Formulae (G.4), (G.5), and (G.6):

$$\overline{X}_{\mathrm{G}} = 10^{\overline{X}_{\log}} \tag{G.4}$$

$$\overline{X}_{\mathrm{U}} = 10^{2s} \times \overline{X}_{\mathrm{G}} \tag{G.5}$$

$$\overline{X}_{\mathrm{L}} = 10^{-2s} \times \overline{X}_{\mathrm{G}} \tag{G.6}$$

Using the $\overline{X}_{\mathrm{G}}$ and s results, the 95 % confidence intervals for particle size, d_{U} and d_{L}, respectively, were obtained for each type of sample. The particle size distributions were determined by linear regression of the corresponding $\overline{X}_{\mathrm{G}}$ and particle size data. To avoid statistical artefacts, only data for sizes with $n > 5$ were used for regression. Tables G.2 to G.4 report the mean diameters calculated using the regression formula, d, d_{U} and d_{L}.

Table G.2 — Sample A results

Parameter	**Particle size,** µm(c)									
	4,2	4,6	5,1	6,4	7,7	9,8	11,3	13,6	17,5	21,2
Statistics										
$\overline{X}_{\log}$	3,79	3,68	3,53	3,15	2,75	2,10	1,71	1,03	0,40	0,10
s	0,02	0,03	0,04	0,06	0,10	0,14	0,16	0,17	0,15	0,17
$C_{V,\log}$	0,45	0,87	1,01	2,02	3,64	6,84	9,18	16,35	37,44	168
n	10	19	6	27	22	28	4	28	15	26
95 % **confidence intervals for particle concentration,** particles/mL										
$\overline{X}_{G}$	6 105	4 739	3 414	1 420	558	125,64	51,27	10,74	2,50	1,26
$\overline{X}_{U}$	6 601	5 489	4 021	1 905	884	243,31	105,67	23,35	4,98	2,75
$\overline{X}_{L}$	5 647	4 092	2 899	1 059	352	64,88	24,88	4,94	1,26	0,58
95 % **confidence intervals in particle sizing,** µm(c)										
d	4,2	4,6	5,1	6,4	7,8	9,8	11,1	13,6	17,6	21,1
d_{U}	4,1	4,4	4,8	6,0	7,1	8,9	10,1	12,2	15,4	17,2
d_{L}	4,3	4,8	5,4	6,9	8,4	10,7	12,2	15,4	21,1	30,3

Table G.3 — Sample B results

Parameter	**Particle size,** µm(c)									
	4,2	4,6	5,1	6,4	7,7	9,8	11,3	13,6	17,5	21,2
Statistics										
$\overline{X}_{\log}$	3,23	3,12	3,01	2,75	2,54	2,27	2,15	1,91	1,63	1,37
s	0,03	0,03	0,04	0,04	0,04	0,04	0,01	0,06	0,02	0,07
$C_{V,\log}$	0,81	0,86	1,26	1,30	1,63	1,94	0,37	3,40	1,49	5,24
n	10	19	5	27	22	28	3	28	11	26
95 % **confidence intervals for particle concentration data,** particles/mL										
$\overline{X}_{G}$	1 692	1 326	1 020	563	346	188	141	80,65	42,38	23,52
$\overline{X}_{U}$	1 909	1 501	1 214	663	418	231	147	108,66	47,38	32,75
$\overline{X}_{L}$	1 500	1 171	856	477	286	154	136	59,86	37,91	16,89
95 % **confidence intervals in particle sizing,** µm(c)										
d	4,2	4,6	5,1	6,4	7,8	9,8	11,0	13,6	17,5	21,2
d_{U}	4,0	4,4	4,8	6,0	7,2	9,1	10,8	12,1	15,5	18,8
d_{L}	4,4	4,9	5,5	6,9	8,4	10,6	11,1	15,2	19,7	23,8

Table G.4 — Verification sample results

Parameter	**Particle size,** μm(c)										
	4,2	4,6	5,1	6,4	7,1	7,7	8,4	9,1	9,8	11,3	13,6
Statistics											
$\bar{X}_{\log}$	3,80	3,68	3,53	3,16	3,00	2,78	2,56	2,38	2,14	1,73	1,19
s	0,04	0,04	0,04	0,07	0,17	0,11	0,13	0,18	0,13	0,11	0,13
$C_{\mathrm{V,log}}$	1,06	1,18	1,19	2,09	5,60	3,97	4,98	7,61	6,20	6,49	10,83
n	8	20	17	25	3	20	8	5	24	5	8
95 % **confidence intervals for particle concentration data,** particles/mL											
$\bar{X}_{\mathrm{G}}$	6 286	4 769	3 415	1 458	1 009	600	361	241	137	53,28	15,66
$\bar{X}_{\mathrm{U}}$	7 570	5 827	4 146	1 977	2 190	997	649	555	251	89,26	28,42
$\bar{X}_{\mathrm{L}}$	5 221	3 903	2 812	1 076	465	361	201	104	74	31,80	8,63
95 % **confidence intervals in particle sizing,** μm(c)											
d	4,2	4,7	5,2	6,5	7,0	7,7	8,4	9,1	9,9	11,5	13,6
d_{U}	3,9	4,3	4,9	6,0	5,9	7,0	7,5	7,8	8,9	10,6	12,9
d_{L}	4,5	5,0	5,5	6,9	8,1	8,4	9,4	10,4	10,9	12,4	14,3

G.3 Conclusions

Good agreement between laboratories was obtained for all three types of sample. Agreement deteriorated when the number of particles counted fell below 500. Agreement improved when data from similar types of APC were compared. In order to reduce variability in the results, it is critical that APCs meet the minimum performance levels defined in Annexes A, D, and E and that they be operated within the limits established in Annexes A, B, and C.

Annex H
(informative)

Sample calculations

H.1 Example 1 — Data quality criteria calculations

H.1.1 A calibration sample was analysed and five counts obtained as specified in 6.3. In one channel, counts of 35 710, 35 283, 35 345, 35 389, and 43 522 were obtained. Using the data quality criteria given in 6.3, are the results acceptable for use in calibration?

H.1.2 Find X_{max}, X_{min}, and $\overline{X}$.

X_{max} = 43 522 and X_{min} = 35 283

$$\overline{X} = \frac{35\,710 + 35\,283 + 35\,345 + 35\,389 + 43\,522}{5} = 37\,050 \qquad \text{(H.1)}$$

H.1.3 Calculate D_Q.

$$D_Q = \frac{X_{max} - X_{min}}{\overline{X}} \times 100 = \frac{43\,522\; - \;35\,283}{37\,050} \times 100 = 22{,}24 \qquad \text{(H.2)}$$

H.1.4 Compare D_Q to the maximum allowable percentage difference; see Table C.2. Because $\overline{X}$ = 37 050, the maximum allowable percentage difference should be taken from the row for values of $\overline{X}$ greater than 10 000 and from the column for 6.3. Thus, the maximum allowable value of D_Q is 11,0. Since the calculated value of D_Q is greater than this, the data are not acceptable and shall be tested for outliers.

H.1.5 Inspect the data for possible outliers. Inspection of the raw data shows that the first four counts are relatively close to one another (35 710, 35 283, 35 345, and 35 389), but the last count (43 522) appears to be much higher. Since the last count is the suspected outlier, X_0 = 43 522. The count closest to the suspected outlier is 35 710, so X_N = 35 710.

H.1.6 Calculate D_0.

$$D_0 = \frac{X_{max} - X}{\left| X_0 - X_N \right|} = \frac{43\,522 - 35\,283}{43\,522 - 35\,710} = 1{,}05$$

H.1.7 Determine whether the outlier can be eliminated from the data set. Since the calculated value of D_0 is less than 1,44, the suspected outlier can be discarded.

H.1.8 Recalculate D_Q using the remaining four counts.

Now X_{max} = 35 710 and X_{min} = 35 283, thus

$$\overline{X} = \frac{35\,710 + 35\,283 + 35\,345 + 35\,389}{4} = 35\,432 \qquad \text{(H.3)}$$

$$D_Q = \frac{X_{\max} - X_{\min}}{\overline{X}} \times 100 = \frac{35\,710 - 35\,283}{35\,432} \times 100 = 1{,}20 \qquad \text{(H.4)}$$

H.1.9 Compare D_Q to the maximum allowable percentage difference; refer to Table C.2. Because $\overline{X}$ = 35 432, the maximum allowable percentage difference should be taken from the row for values of $\overline{X}$ greater than 10 000 and from the column for 6.3. Thus, the maximum allowable value of D_Q is 11,0. Because the calculated value of D_Q is less than this, the data are now acceptable. Proceed with the calibration procedure using this new value for $\overline{X}$.

H.2 Example 2 — Calibration curve construction

H.2.1 See 6.8.

Using the data obtaining by analysing calibration samples in 6.3, determine the relationship between particle concentration and threshold setting. This is done in 6.4 by plotting particle concentrations versus the corresponding threshold settings on a $\log_{10}$-$\log_{10}$ graph using only the acceptable data points. This is illustrated in Figure H.1 b). Use appropriate mathematical techniques to construct a smooth curve (Figure H.1, label 1) between the data points.

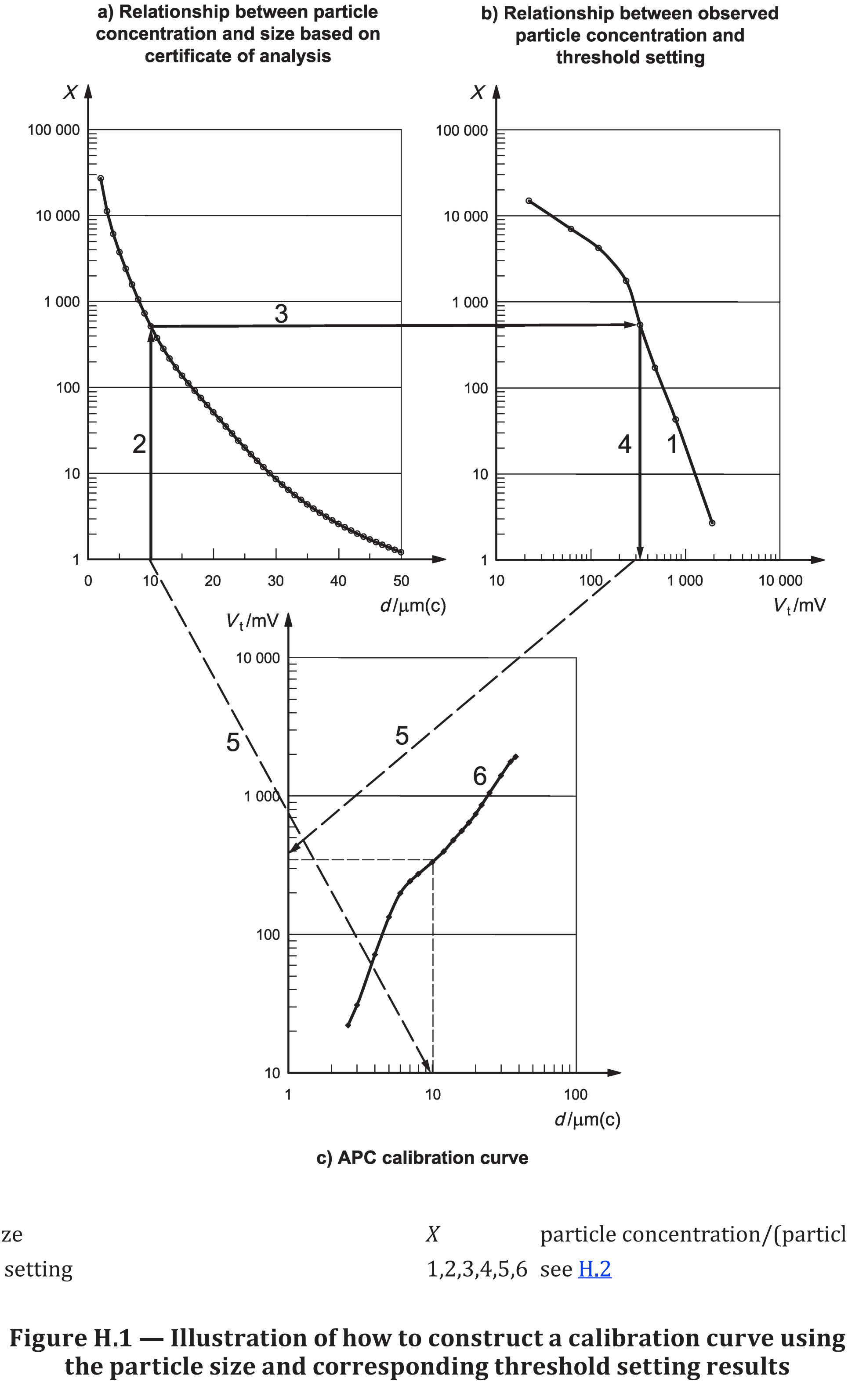
H.2.2 Choose particle sizes for calibration. For illustration purposes, a particle size of 10 µm(c) is used in this example.

H.2.3 Determine the particle concentrations corresponding to the previously chosen particle sizes. Figure H.1 a) shows the relationship between particle concentration and particle size for SRM 2806 traceable calibration samples used in this example. The vertical arrow (Figure H.1, label 2) shows that at 10 µm(c), the NIST certificate of analysis for the samples reports that there are 513,7 particles larger than 10 µm(c)/mL.

H.2.4 Determine the threshold settings corresponding to the previously determined particle concentrations. Referring to the horizontal arrow connecting the two plots (Figure H.1, label 3), it can be seen that 513,7 particles/mL corresponds to a threshold setting of 539 mV (Figure H.1, label 4).

H.2.5 Determine the threshold settings corresponding to the previously chosen particle sizes. In H.2.3 and H.2.4, it was determined that 513,7 particles/mL corresponds to a 10 µm(c) particle size according to the certification of analysis for the samples and to a threshold setting of 539 mV. Thus, a threshold setting of 539 mV corresponds to a particle size of 10 µm(c) (Figure H.1, label 5).

H.2.6 Repeat H.2.2 to H.2.5 for other particle sizes. Use the particle size and corresponding threshold setting results determined in this manner to construct a calibration curve (Figure H.1, label 6).



c) APC calibration curve

Key

d	particle size	*X*	particle concentration/(particles/mL) > size
V_t	threshold setting	1,2,3,4,5,6	see H.2

Figure H.1 — Illustration of how to construct a calibration curve using the particle size and corresponding threshold setting results

H.3 Example 3 — Regression equation for coincidence error procedure

H.3.1 See B.7.

The coincidence error procedure was performed at 1,5 times the threshold noise level as specified in Annex B. Table H.1 is a partial listing of the results.

Table H.1 — Data for use in example 3

Concentration limit recommended by the manufacturer %	**Dust concentration** γ mg/l	**Observed concentration** $\bar{n}$ particles/mL
0	0	6,6
10	0,12	1 654
20	0,25	3 347
30	0,28	3 857
40	0,40	5 235
50	0,48	6 286

H.3.2 The number of data points that can be used to determine the theoretical relationship between actual and observed concentration is five ($N = 5$, i.e. representing the 0 %, 10 %, 20 %, 30 %, and 40 % data), since only the 0 % to 40 % data can be used for regression.

Calculate $\Sigma \bar{X}$, $\Sigma\gamma$, $\Sigma\gamma \bar{X}$, $\Sigma\gamma^2$ and $\Sigma \bar{X}^2$.

$$\Sigma\bar{X} = 6{,}6 + 1\,654 + 3\,347 + 3\,857 + 5\,235 = 14\,099{,}6$$

$$\Sigma\gamma = 0 + 0{,}12 + 0{,}25 + 0{,}28 + 0{,}40 = 1{,}05$$

$$\Sigma\gamma\bar{X} = (0 \times 6{,}6) + (0{,}12 \times 1\,654) + (0{,}25 \times 3\,347) + (0{,}28 \times 3\,857) + (0{,}40 \times 5\,235) = 4\,209$$

$$\Sigma\gamma^2 = (0{,}00)^2 + (0{,}12)^2 + (0{,}25)^2 + (0{,}28)^2 + (0{,}40)^2 = 0{,}315\,3$$

$$\Sigma\bar{X}^2 = (6{,}6)^2 + (1\,654)^2 + (3\,347)^2 + (3\,857) + (5\,235)^2 = 56\,219\,843$$

H.3.3 Calculate *a* and *r* for the regression formula.

$$a = \frac{N\left(\sum\gamma\bar{X}\right) - \left(\sum\gamma\sum\bar{X}\right)}{N\sum\gamma^2 - \left(\sum\gamma\right)^2} = \frac{(5\times 4\,209) - (1{,}05\times 14\,099{,}6)}{(5\times 0{,}315\,3) - (1{,}05)^2} = 13\,167 \qquad \text{(H.5)}$$

$$r = \frac{N\sum\gamma\bar{X} - \left(\sum\gamma\sum\bar{X}\right)}{\sqrt{\left[N\sum\gamma^2 - \left(\sum\gamma\right)^2\right]\left[N\sum\bar{X}^2 - \left(\sum\bar{X}\right)^2\right]}} = \frac{(5\times 4\,209) - (14\,009{,}6\times 1{,}05)}{\left[(5\times 0{,}315\,3) - 1{,}05^2\right]\left[(5\times 56\,219\,843) - 14\,009{,}6^2\right]} = 0{,}999$$

(H.6)

H.3.4 Use the regression formula to calculate the theoretical concentration, X_t, for the 50 % (0,48 mg/l) concentration:

$$X_t = a\,\gamma = 13\,167 \times 0{,}48 = 6\,320{,}16 \qquad \text{(H.7)}$$

H.4 Example 4 — Determination of half-count setting

H.4.1 See D.5.

Using the APC in the differential mode, the threshold settings for the first four channels are adjusted as specified in D.3 until the results in Table H.2 are obtained. The noise level for the APC is 3,0 mV.

Table H.2 — Data for use in example 4

Channel	**Description**	**Threshold setting** mV	**Differential counts** N_i
1	1,5 times noise level	1,5 × 3,0 = 4,5	953
2	0,72 times third channel setting	0,72 × 57,8 = 41,6	29 592
3	median size of polystyrene latex sphere	57,8	30 250
4	1,32 times third channel setting	1,32 × 57,8 = 76,3	1 774

H.4.2 Calculate D.

$$D = \left(1 - \frac{N_2}{N_3}\right) \times 100 = \left(1 - \frac{29\ 592}{30\ 250}\right) \times 100 = 2{,}18\ \% \qquad \text{(H.8)}$$

H.4.3 Because the absolute value of D is less than 3 %, the half-count condition has been achieved and the threshold setting of the third channel corresponds to the size of the polystyrene latex sphere.

H.5 Example 5 — Resolution determination

H.5.1 See D.11 and D.12.

In example 4, the half-count setting was obtained using the calibration curve determined in 6.8. This setting was found to correspond to a particle size of 13,12 µm(c) (d = 13,12 µm). The settings of the first five channels were readjusted in accordance with D.7 and the results in Table H.3 were obtained.

Table H.3 — Data for use in example 5

Channel	**Description**	**Setting** mV	**Particle size** µm(c)	**Differential counts** N_i
A	0,72 times channel C	0,72 × 57,8 = 41,6	10,91	304
B	0,9 times the size of the polystyrene latex sphere	48,1	0,9 × 13,12 = 11,81	29 946
C	half-count setting	57,8	13,12	23 879
D	1,1 times the size of the polystyrene latex sphere	68,4	1,1 × 3,12 = 14,42	4 713
E	1,32 times channel D	1,32 × 57,8 = 76,3	15,30	1 774

H.5.2 Calculate s_L and s_R, in micrometres (c), from the results, d = 13,12 µm(c), N_A = 304, N_B = 29 946, N_C = 23 879 and N_D = 4 713.

$$s_R = \frac{d}{6\ln\left[1 + 2(N_C / N_D)\right]} = \frac{13{,}12}{6\ln\left[1 + 2(23\ 879 / 4\ 713)\right]} = 0{,}91 \qquad \text{(H.9)}$$

$$s_L = \frac{d}{6\ln\left[1+2(N_B / N_A)\right]} = \frac{13{,}12}{6\ln\left[1+2(29\,926 / 304\right]} = 0{,}41 \qquad \text{(H.10)}$$

H.5.3 Calculate R_L and R_R, the standard deviation of the polystyrene latex sphere size, s_I, reported by the supplier is 0,039 µm.

$$R_L = \frac{100\sqrt{s_L^2 - s_I^2}}{d} = \frac{100\sqrt{0{,}41^2 - 0{,}039^2}}{13{,}12} = 3{,}11 \qquad \text{(H.11)}$$

$$R_R = \frac{100\sqrt{s_R^2 - s_I^2}}{d} = \frac{100\sqrt{0{,}91^2 - 0{,}039^2}}{13{,}12} = 6{,}93 \qquad \text{(H.12)}$$

H.5.4 Compare R_L and R_R to determine the resolution, R. In this example, R_R is greater than R_L, so $R = R_R$, and the APC resolution is 6,93 %, which is within the performance specifications of Annex D.

Annex I
(informative)

Verification of particle size distribution of calibration samples

I.1 Background

In order to properly calibrate automatic particle counters, Clause 6 requires that valid primary or secondary calibration samples be used. In order to verify particle-counting accuracy, NIST RM 8632 samples are used in Annex E. Whether primary calibration sample, secondary calibration sample, or NIST RM 8632 sample, it is critical that the particle size distribution of the sample be representative of the specified contaminant. Even though the specified contaminant was used to prepare the samples, the size distribution may have been altered by sample preparation methodology, deterioration of the samples over time, contamination, or other reasons. This Annex provides guidance on how the particle size distribution of samples used in conjunction with this International Standard can be verified.

I.2 Procedure

I.2.1 Calibrate an APC in accordance with Clauses 5 and 6 using NIST primary calibration suspension samples (4.4).

I.2.2 Choose particle sizes and corresponding threshold voltage settings using the criteria described in 6.2.

I.2.3 Analyse samples in accordance with 6.2 and 6.3.

I.2.4 Calculate R_d, the ratio of the mean particle concentration for each particle size to the reference mean particle concentration, using Formula (I.1):

$$R_d = \frac{X_{d,\mathrm{S}}}{X_{d,\mathrm{R}}} \tag{I.1}$$

where

$X_{d,\mathrm{S}}$ is the mean particle concentration for the sample at particle size d;

$X_{d,\mathrm{R}}$ is the mean particle concentration for the reference at the same particle size.

For primary and secondary calibration samples, $X_{d,\mathrm{R}}$ should be taken from the NIST SRM 2806 certificate of analysis. For ISO UFTD samples, $X_{d,\mathrm{R}}$ is obtained by calculating the mean of the concentration range for each size given in Table A.1.

I.2.5 Plot R_d for each size as a function of particle size d and connect the data points. If the result is a near horizontal line, this suggests that the size distribution of the sample approximates that of the reference suspension. If the result is not horizontal or exhibits significant deviations from horizontal at one or more sizes, this suggests that the sample is not representative of the reference suspensions and should not be used.

I.2.6 Calculate the ratio, T_d, of the mean particle concentration for each particle size to the corresponding mean particle concentration for the same sample at 10 μm(c) using Formula (I.2):

$$T_d = \frac{X_{d,S}}{X_{10,S}} \qquad \text{(I.2)}$$

where

$X_{d,S}$ is the mean particle concentration for the sample at particle size d;

$X_{10,S}$ is the mean particle concentration for the same sample at 10 µm(c).

I.2.7 Repeat I.2.6 for the reference sample.

I.2.8 Plot T_d for each size as a function of d and connect the data points for the sample and for the reference. If the two lines exhibit essentially the same behaviour result, this suggests that the size distribution of the sample approximates that of the reference suspension. If not, this suggests that the sample is not representative of the reference suspensions and should not be used.

I.2.9 If the sample passes the acceptance criteria given in I.2.5 and I.2.8, then the samples are acceptable for use in this International Standard. If not, the samples should be replaced or remade after correcting the underlying problem.

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[2] Withdrawn.

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