BS ISO 11171:2016

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Hydraulic fluid power — Calibration of automatic particle counters for liquids

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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 It supersedes BS ISO 11171:2010 which is withdrawn.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.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Compliance with a British Standard cannot confer immunity from legal obligations.

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INTERNATIONAL STANDARD

ISO 11171

Third edition
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

Reference number
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Contents

Page

Foreword iv
Introduction v
1 Scope 1
2 Normative references 1
3 Terms and definitions 1
4 Materials and equipment 2
5 Sequence of APC calibration procedures 4
6 Sizing calibration procedure 7
7 Data presentation 13
8 Identification statement 14
Annex A (normative) **Preliminary APC check** 15
Annex B (normative) **Coincidence error procedure** 18
Annex C (normative) **Flow rate limit determination** 23
Annex D (normative) **Resolution determination** 27
Annex E (normative) **Verification of particle-counting accuracy** 32
Annex F (normative) **Preparation and verification of bottles of secondary calibration suspensions** 35
Annex G (informative) **APC calibration round robin** 38
Annex H (informative) **Sample calculations** 43
Annex I (informative) **Verification of particle size distribution of calibration samples** 49
Bibliography 51

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Document Sharing Hub
Full document details: BS ISO 11171:2016

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).

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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.

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INTERNATIONAL STANDARD ISO 11171:2016(E)

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

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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

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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.

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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.

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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.

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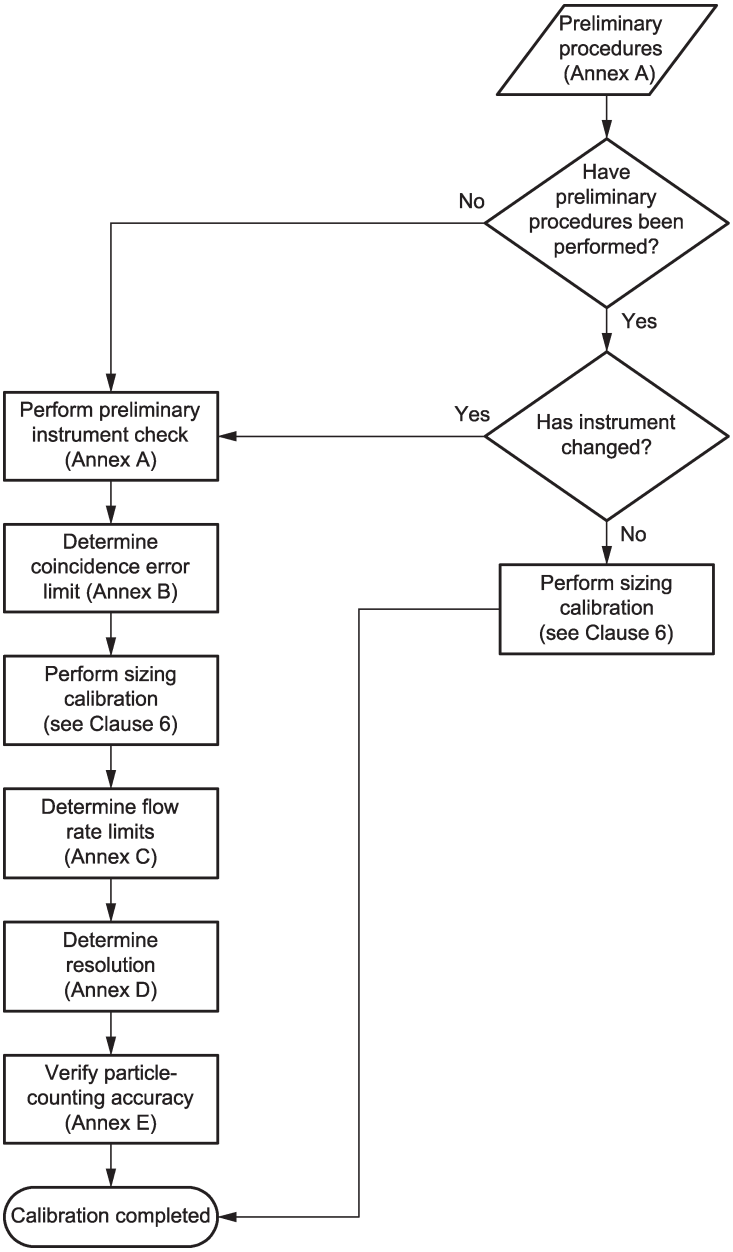

Figure 1 — Sequence of APC calibration procedures

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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

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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.

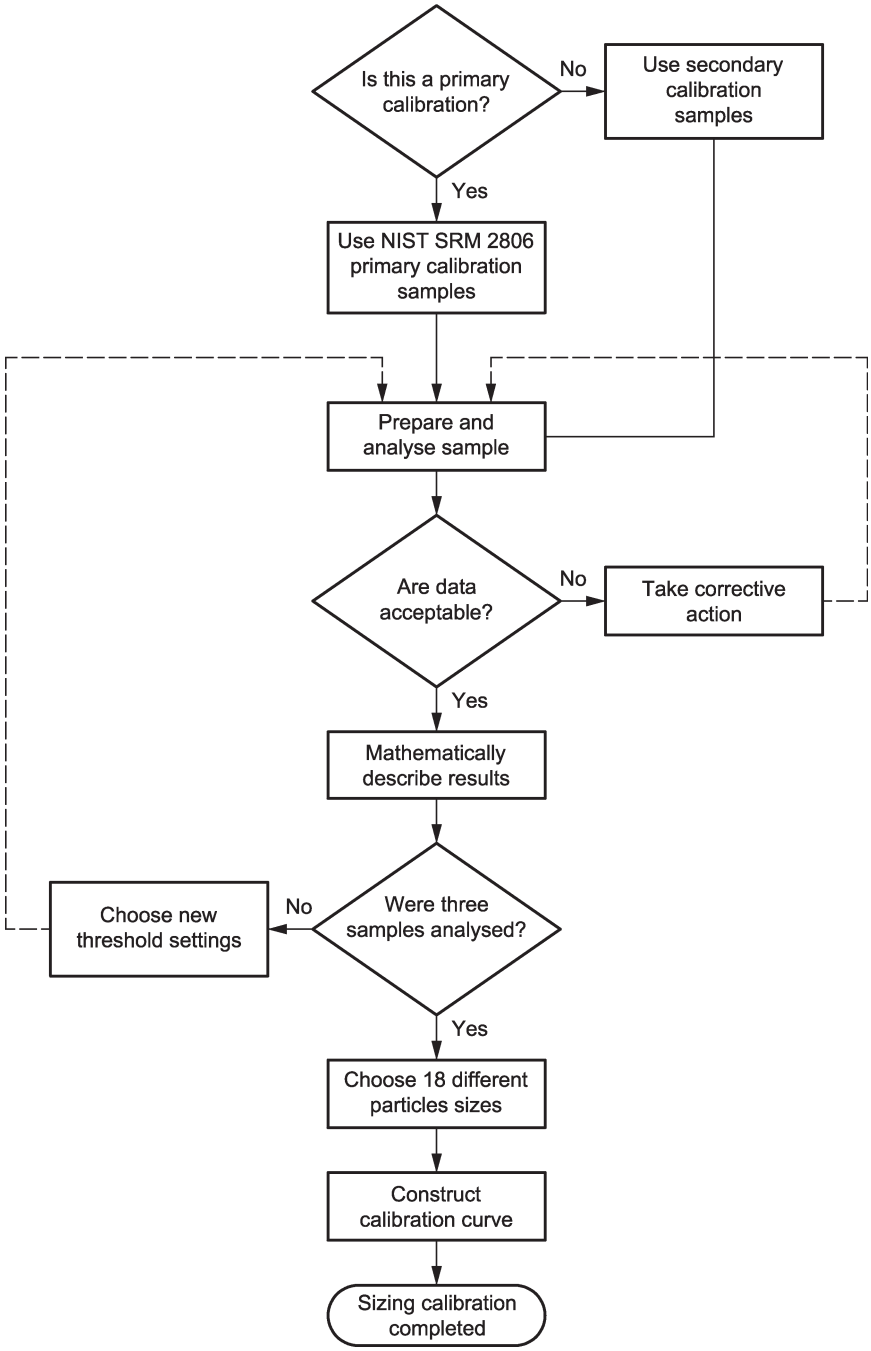

Figure 2 — Sizing calibration procedure

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.

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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

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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.

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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						
$\bar{X}$ (particles/mL)						
D_Q						

Second calibration suspension _____ **Calibration suspension identification number** _____

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

Third calibration suspension _____ **Calibration suspension identification number** _____

Threshold setting						
Count 1						
Count 2						
Count 3						
Count 4						
Count 5						
$\bar{X}$ (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

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