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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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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 11171 was prepared by Technical Committee ISO/TC 131, *Fluid power systems*, Subcommittee SC 6, *Contamination control*.

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This second edition of ISO 11171 cancels and replaces the first edition (ISO 11171:1999), of which it constitutes a technical revision, including the following modifications to clarify the steps to be performed when using this International Standard and to minimize several potential sources of variability. Specifically:

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a) A definition of an automatic particle counter (APC) has been added as 3.1.

b) In 5.1, the specific sequence of steps to be followed in calibration is not longer mandated.

c) In 5.1, Note 2 has been added to provide guidance on how to detect a change in the calibration of an APC.

d) 6.3 now explicitly requires that at least 5 000 particles be counted in order to have statistically valid data for a particular channel setting.

e) B.4 no longer requires that a 0 % sample be prepared and analysed; instead, the regression equation for coincidence error determined in B.7 is forced through the origin.

f) Annex C now distinguishes between fixed flow rate and adjustable flow rate bottle samplers. Fixed flow rate instruments simply require verification of their ability to consistently deliver a constant flow rate within 3 %. For adjustable flow rate instruments, this International Standard requires that their working flow rate and flow rate limits be determined, in addition to verification of their ability to consistently deliver a constant flow rate within 3 %.

g) D.12 increases the acceptable resolution of an APC from 10 % to 15 %.

h) E.3 requires that NIST RM 8632 dust be used for verification of counting accuracy. Use of ISO UFTD is no longer acceptable.

i) Annex H now includes an example of how to construct a calibration curve.

It also incorporates the Technical Corrigendum ISO 11171:1999/Cor.1:2001.

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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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Hydraulic fluid power — Calibration of automatic particle counters for liquids

1 Scope

This International Standard specifies procedures for:

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.

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2 Normative references

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The following referenced documents are indispensable for the application of this document. 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*

ISO 21501-3, *Determination of particle size distribution — Single particle light interaction methods — Part 3: Light extinction liquid-borne particle counter*

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

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NOTE 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

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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

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

NOTE 2 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 2806

NOTE 1 The procedure is specified in Clause 6.

NOTE 2 For details of NIST standard reference material 2806, see 4.4.

3.10
secondary calibration
sizing calibration conducted using calibration suspensions

NOTE The procedure is specified in Clause 6 and the calibration suspensions are prepared in accordance with Annex F.

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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.

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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 have 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[3] 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.

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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

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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:

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; or

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.

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NOTE 2 For the purposes of this clause, 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.

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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:

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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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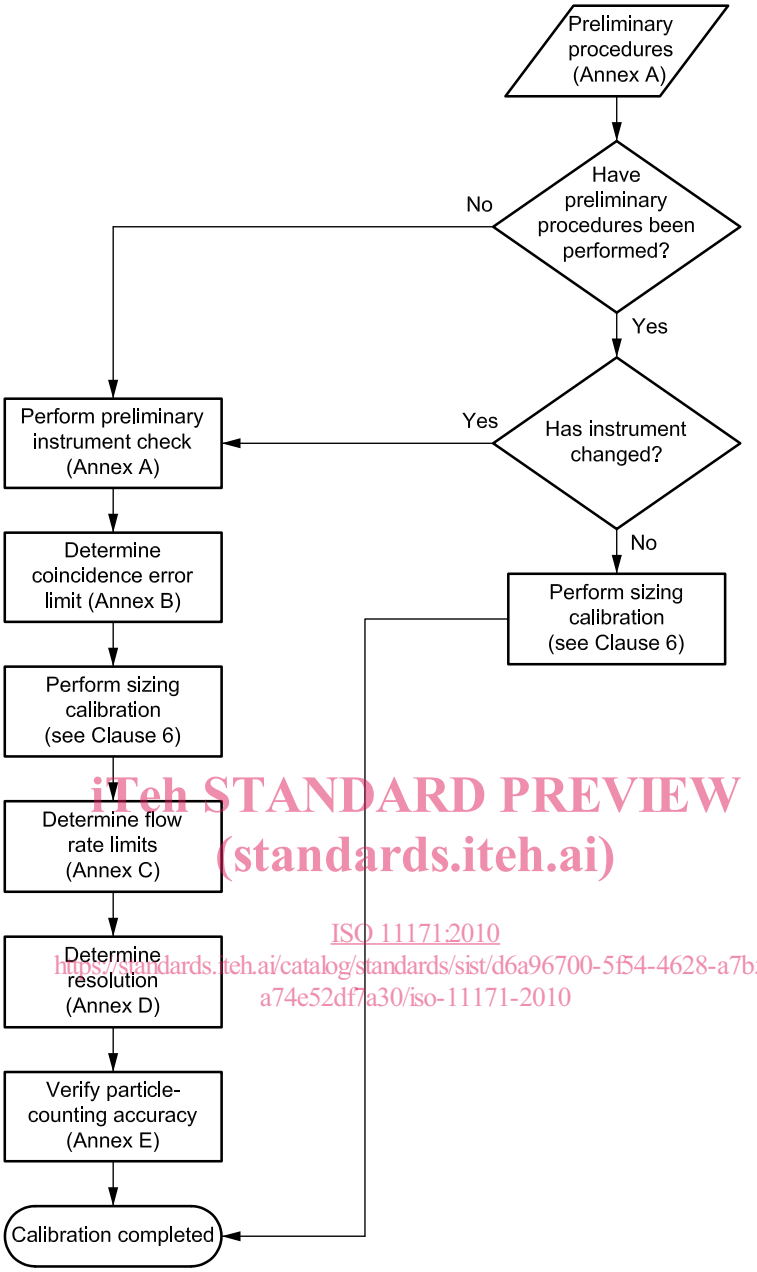

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Figure 1 — Sequence of APC calibration procedures

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Table 1 — Schedule of APC calibration procedures

APC status[a]	Relevant clause and annexes of this International Standard					
	Clause 6 Sizing calibration procedure	Annex A Preliminary APC check	Annex B Coincidence error limits	Annex C Flow rate limits	Annex D Resolution	Annex E Accuracy
New APC or existing APC not calibrated to this International Standard	×	×	×	×	×	×
Last calibration was more than 6 to 12 months ago	×	—	—	—	—	—
Suspicion that calibration has changed significantly	×	—	—	—	—	—
Optics (including light source) repaired or readjusted	×	×	×	—	×	×
Sensor or counting electronics repaired or readjusted	×	×	×	×	×	×
Volume measurement components (e.g. flowmeter, burette, level detectors) repaired or readjusted	—	×	—	—	—	—
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.

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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.

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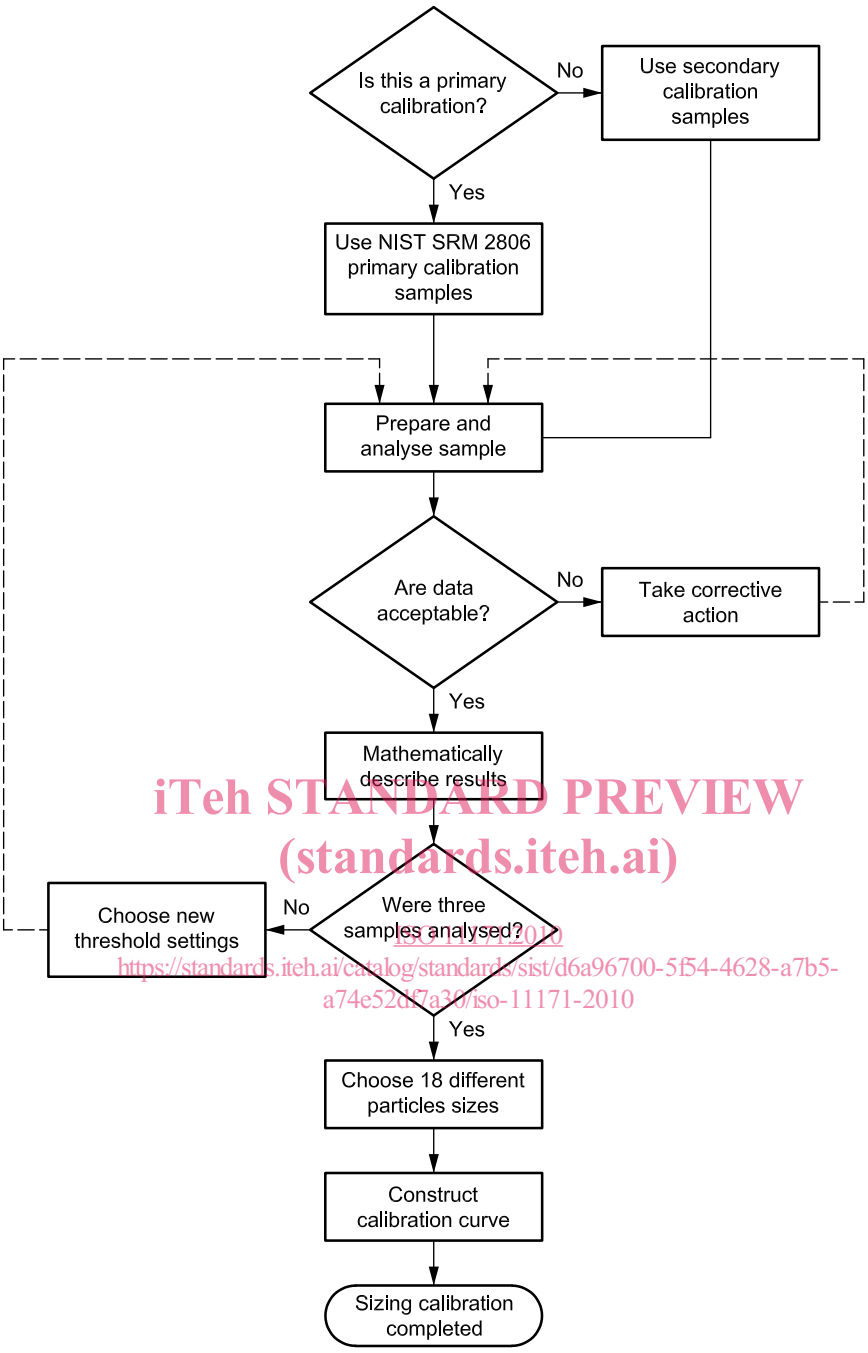

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Figure 2 — Sizing calibration procedure

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.

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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.

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

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

where

$\overline{X}$ 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 Equation (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.

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