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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

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 of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

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

This fourth edition cancels and replaces the third edition (ISO 11171:2016), which has been technically revised.

The main changes compared to the previous edition are as follows:

— Clause 1 and 4.4: SRM 2806b are not used for sizing calibration purposes with this document;

— 3.1: the definition of an automatic particle counter (APC) is clarified;

— 3.8 (Note 1 to entry): the particle size distribution for primary calibration suspension samples is found in Table 3 of the SRM 2806x Certificate of Analysis;

— 4.6: ISO medium test dust (MTD) or other test dust conforming to ISO 12103-1 for secondary calibration suspension is permitted;

— 4.8: APC are required to have a minimum of 8 channels that can be set instead of only 6;

— 6.1: latex spheres are required for primary calibration at particle sizes greater than 30 µm(c);

— 6.1: secondary calibration suspensions can be used for secondary calibration at particle sizes greater than 30 µm(c);

— 6.2: both reference and certified data from the SRM 2806x particle size distribution are used for primary sizing calibration;

— 6.2: data from at least 16 different particle sizes taken from the certified particle size distribution are used to create the APC calibration curve;

— 6.3: data obtained from at least 12 different APC threshold voltage settings are used to relate particle concentrations to threshold settings;

— 6.6: the data acceptance criteria are based upon the mean number of particles counted rather than particle concentration;

— 6.6: dilution of calibration suspensions is permitted to allow the calibration of APCs at sizes that would otherwise be in coincidence error for calibration suspensions;

— 6.9: the constrained cubic spline method of interpolation is specified and a tool for its use to relate threshold voltage setting to particle size is provided;

— 6.9: the standard uncertainty in particle concentration at each threshold setting is calculated and reported;

— 6.11 – 6.14: the modified differential half-count method for relating particle size and threshold setting using latex spheres is specified for primary calibration of particle sizes greater than 30 µm(c);

— 6.15: the constrained cubic spline method of interpolation is specified for relating threshold voltage setting to particle size and a tool for its use to relate threshold voltage setting to particle size and to construct an APC calibration curve is provided;

— Clause 7: the only acceptable way of reporting particle size using this document is using the unit of µm(c);

— Table A.1: the median, upper and lower acceptable particle concentration limits have been updated based on the results of interlaboratory testing using RM 8632a test dust and calculated based upon the logarithm of the observed particle counts and 98 % confidence level;

— Table C.2: acceptable values for D_Q are based upon the mean number of particles counted rather than particle concentration;

— F.2: use of NIST RM 8631x, ISO MTD, or other test dust conforming to ISO 12103-1 for secondary calibration suspensions is permitted and the maximum allowable concentration for secondary suspensions is increased from 75 % to 100 times the coincidence error limit of the sensor;

— F.4 and F.7: data are obtained from at least 16 different particle sizes and reported in the certificate of analysis for the resultant secondary calibration suspensions;

— Annex G: this new annex specifies the method of dilution for calibration suspension samples for use in 6.6 for samples that would otherwise be in coincidence error;

— Annex H, Sample calculations, from ISO 11171:2016: deleted. Replaced by Annex H, Verification of particle size distribution of calibration samples.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 (APC) are an accepted means of determining the concentration and size distribution of the contaminant particles. Individual APC accuracy is established through calibration.

This document is a standard calibration procedure for APC that are used for determining particle sizes and counts. The primary particle-sizing calibration is conducted using NIST SRM 2806x suspensions with particle size distribution certified by the United States National Institute of Standards and Technology (NIST) for particle sizes 30 µm(c) and smaller, and using polystyrene latex spheres at larger sizes.

A secondary calibration method uses suspensions of NIST RM 8631x, ISO MTD, or other test dust conforming to ISO 12103-1, which are independently analysed using an APC calibrated by the primary method. Minimum performance specifications are established for the APC coefficient of variation (CV) of sample volume, CV of flow rate, resolution and particle counting accuracy. The operating limits of an APC, including its threshold noise level, coincidence error limit and flow rate limits are determined.

Hydraulic fluid power — Calibration of automatic particle counters for liquids

1 Scope

This document specifies procedures for the following:

a) primary particle-sizing calibration for particle sizes 1 µm(c) and larger, sensor resolution and counting performance of liquid automatic particle counters that are 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 test dust;

e) determining coincidence and flow rate limits.

This document is applicable for use with hydraulic fluids, aviation and diesel fuels, engine oil and other petroleum-based fluids. This document is not applicable to particle-sizing calibration using NIST SRM 2806b primary calibration suspensions.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 4787, *Laboratory glassware — Volumetric instruments — Methods for testing of capacity and for use*

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

ISO 12103-1, *Road vehicles — Test contaminants 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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
automatic particle counter
APC
instrument that automatically:

a) senses individual particles suspended in a controlled volume of fluid using optical light extinction or light scattering principles;

b) measures the size of particles;

c) sorts or compiles particles into size ranges;

d) counts particles in each size range;

e) reports the number of particles in each size range per unit volume; and

f) facilitates instrument calibration according to this document.

Note 1 to entry: APC used for *particle size* (3.7) determination with hydraulic fluids, aviation and diesel fuels, engine oil and other petroleum-based fluids shall be calibrated in accordance with Clause 5 of this document.

3.2
threshold noise level
minimum voltage setting of an *APC* (3.1) 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)

Note 1 to entry: The Brownian motion of any detectable particles in the sensing zone during performance of subclause A.2 can result in erratic results.

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 *APC* (3.1) to distinguish between particles of similar, but different, sizes as determined in accordance with Annex D of this document

3.5
coincidence error limit
highest concentration of NIST RM 8632x that can be counted with an *automatic particle counter* (3.1) with an error of less than 5 % resulting from the presence of more than one particle in the *sensing volume* (3.3) at one time

3.6
working flow rate
flow rate through the sensor used for sample analysis

3.7
particle size
projected area equivalent diameter of particles as determined by NIST using scanning electron microscopy traceable to SI units through a NIST length standard or using a liquid optical single particle *APC* (3.1) calibrated according to this document

Note 1 to entry: NIST uses scanning electron microscopy to determine the projected area equivalent diameter of particles in NIST standard reference material 2806x, where *x* is the letter used by NIST to designate the batch number of the certified *primary calibration* (3.9) suspension.

3.8
particle size distribution
cumulative number concentration of particles larger than a specified size, expressed as a function of *particle size* (3.7)

Note 1 to entry: A certified particle size distribution is one provided by a producer of *primary* (3.9) or *secondary calibration* (3.10) suspensions, and certifies that the particle size distribution reported for the suspensions was determined by NIST or determined according to Annex F of this document. The particle size distribution of SRM 2806x used for *primary calibrations* (3.9) shall consist of the Diameter [µm(c)] and corresponding Number of Particles > Diameter (part/mL) given in columns 1 and 3 of Table 3 of the Certificate of Analysis for Standard Reference Material® 2806x[5].

Note 2 to entry: Verification of particle size distribution of calibration samples is detailed in Annex H of this document.

3.9
primary calibration
sizing calibration conducted according to the sizing calibration procedure specified in Clause 6 of this document using NIST standard reference material 2806x for *particle sizes* (3.7) 30 µm(c) and smaller, and using polystyrene latex spheres at larger sizes

Note 1 to entry: For details of NIST standard reference material 2806x, see 4.4.

3.10
secondary calibration
sizing calibration conducted using secondary calibration suspensions

Note 1 to entry: The sizing calibration procedure is specified in Clause 6 and the preparation of secondary calibration suspensions is set out in Annex F.

3.11
multi-channel analyser
MCA
electronic device capable of sorting incoming electric pulses according to their amplitude

4 Materials and equipment

4.1 Polystyrene latex spheres that:

a) are in aqueous suspension;

b) have a nominal diameter of:

   1) 10 µm if used for resolution determination in accordance with Annex D;

   2) larger than 30 µm for particle size calibration in accordance with Clause 6 for particle sizes of larger than 30 µm;

c) have a coefficient of variation that is less than 5 % where the coefficient of variation is the ratio of the standard deviation of the latex particle diameters in the suspension to their mean particle diameter;

d) have a certificate of analysis that indicates that the latex spheres mean particle diameter and coefficient of variation were determined using techniques with traceability to national 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 of polystyrene latex spheres can be verified using the method described in D.14.

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 diluent, consisting of the test liquid used in ISO 16889 and containing:

a) an antistatic additive at such a concentration that resultant conductivity of the clean diluent is (2 500 ± 1 000) pS/m at room temperature; and

b) less than 0,5 % of the number concentration 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 diluent, for use in calibration for particle sizes greater than 30 µm(c) and to determine sensor resolution in Annex D (the clean diluent specified in 4.2 is used for all other operations in this document), which:

a) is prepared from a concentrate made by adding 120 g of aerosol OT to each litre of clean diluent (4.2), which is:

   1) heated to approximately 60 °C and stirred until no undissolved aerosol OT is visible to the eye; and

   2) diluted with clean diluent (4.2) to a final concentration of 12 g of aerosol OT per litre; and

b) meets the same cleanliness levels as the diluent specified in 4.2**.**

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

NOTE In 4.3 a) 1), it is critical that all of the aerosol OT be dissolved prior to proceeding to 4.3 a) 2). Depending upon the local conditions, complete dissolution can require in excess of 6 hours of heating and stirring as described.

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

4.4 NIST standard reference material 2806x (SRM 2806x) 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, for use in primary calibrations. SRM 2806b shall not be used for calibrations according to this document.

4.5 NIST reference material 8631x (RM 8631x) dust, where *x* is the letter used by NIST to designate the batch number of the reference material, available from NIST, prepared by drying the dust for at least 18 h at a temperature between 110 °C and 150 °C, for use if secondary calibration is to be performed (see 6.1).

4.6 ISO medium test dust (MTD) or other test dust conforming to ISO 12103-1, dried for at least 18 h at a temperature between 110 °C and 150 °C before use, for use if secondary calibration is to be performed (see 6.1).

4.7 NIST reference material 8632x (RM 8632x) dust, where *x* is the letter used by NIST to designate the batch number of the reference material, prepared by drying the dust for at least 18 h at a temperature between 110 °C and 150 °C, before use, required for determination of coincidence error limit and in Annexes A, B, C and E.

NOTE The reference materials specified in 4.4, 4.5, 4.6 and 4.7 can change as new batches are produced. Such a change does not affect the particle sizing calibration (Clause 6), but the ability of an APC to meet the counting accuracy requirements of E.9 can be affected if the batch of RM 8632*x* used to prepare the samples differs from the batch used to create Table A.1.

4.8 Automatic particle counter for liquids, with bottle sampler with at least 8 channels that can be set at various threshold settings.

4.9 Clean sample containers, with closures (appropriate bottle caps, for example), and **volumetric glassware** of at least class B accuracy as defined in ISO 4787, with cleanliness levels lower than 0,5 % of the number of concentration of particles (larger than the smallest particle size of interest) expected to be observed in the samples, confirmed in accordance with ISO 3722.

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

4.11 Ultrasonic bath, 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: 0,01 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.

4.15 Secondary calibration suspension, prepared according to Annex F for use in secondary calibrations. Secondary calibration samples shall not be used for primary calibrations.

5 Sequence of APC calibration procedures

5.1 Conduct the procedures of this Clause when a new APC is received or following the repair or re-adjustment of an APC or sensor as shown in Table 1. See Figure 1 for a recommended sequence of steps to be followed when performing a full calibration on a new APC. Annexes A and B shall be performed prior to proceeding to Clause 6. 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, and 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 given in Figure 1 and Table 1 are recommendations. The operator may follow a different order, as long as all required parts are performed.

Annexes A, B, C, D and E may be performed by an individual laboratory or by the manufacturer of the APC prior to delivery. If these are performed prior to delivery, it is not always required to repeat these annexes upon receipt of the APC, depending upon the manufacturer's recommendations.

NOTE 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.

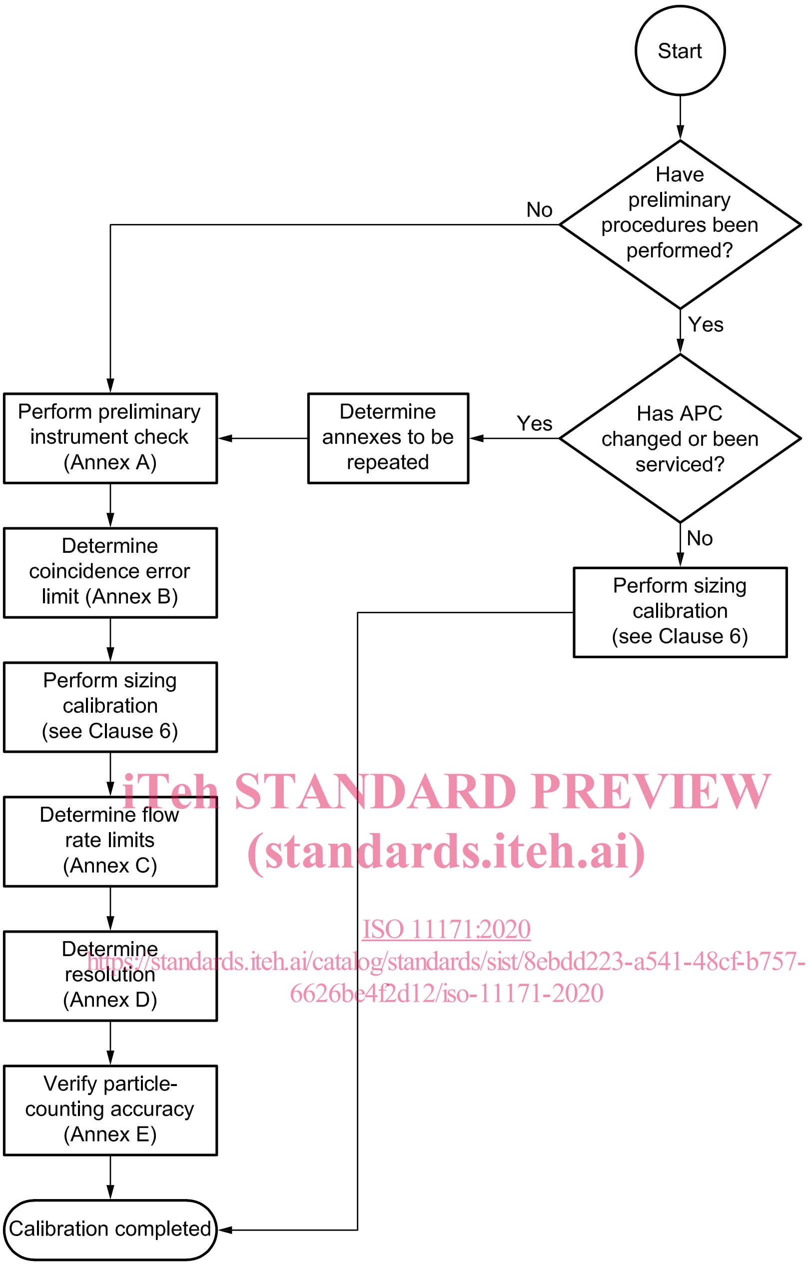

Figure 1 — Recommended sequence of APC calibration procedures

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 individual moving range (IMR) chart, to detect significant changes in calibration;

b) comparing calibration curves reporting particle size in units of µm(c) 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 6.5 through 6.7, then comparing the resulting particle concentration data to the corresponding particle size distribution for the sample:

   1) if the results agree within the limits for the maximum allowable D_Q (6.6) given in Table C.2, the ability of the APC to size and count particles has not been significantly affected;

   2) if the results do not agree, a significant change has occurred, so proceed as indicated in Table 1.

e) determining the threshold noise level of the APC in accordance with A.2, then comparing the resulting noise level to previously obtained results. If the threshold noise level has increased 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. If the threshold noise level has not changed, this is not proof that the APC's operating characteristics are unchanged.

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

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

If the volume measurement system is repaired, replaced or re-adjusted, the procedures of A.3 to A.9 and of Annex C shall be performed.

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 document, the APC shall:

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 accordance with 5.4 within the coincidence error and flow rate limits determined in accordance with 5.3 and 5.5.

Table 1 — Schedule of APC calibration procedures

APC status[a]	Relevant clause or annex of this document to be performed					
	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 document	Perform procedure	Perform procedure	Perform procedure	Perform procedure	Perform procedure	Perform procedure
Last calibration was more than 6 m to 12 m ago	Perform procedure	Not required	Not required	Not required	Not required	Not required
Suspicion that calibration has changed significantly	Perform procedure	Not required	Not required	Not required	Not required	Not required
Optics (including light source) repaired or readjusted	Perform procedure	Perform procedure	Perform procedure	Not required	Perform procedure	Perform procedure
Sensor or counting electronics repaired or readjusted	Perform procedure	Perform procedure	Perform procedure	Perform procedure	Perform procedure	Perform procedure
Volume measurement components (e.g. flow meter, burette, level detectors) repaired or readjusted	Not required	Perform procedure	Not required	Perform procedure	Not required	Not required
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 re-adjustment 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 6.5 through 6.7, 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 Conduct the sizing calibration set out in Figure 2 every three to six months, when a new APC is received, or after the repair or re-adjustment of an APC or sensor. After a suitable calibration history for an APC and sensor has been developed, the time interval between successive calibrations can be increased, but shall not exceed one year. For particle sizes 30 µm(c) and smaller, use NIST calibration suspensions (see 4.4) for primary calibrations or secondary calibration suspensions (4.15) prepared in accordance with Annex F for secondary calibrations. For particle sizes larger than 30 µm(c), use polystyrene latex spheres (see 4.1) for primary calibrations or use secondary calibration suspensions prepared in accordance with Annex F for secondary calibrations.

Conduct all phases of the calibration at the same working flow rate. Determine the flow rate limits of the APC in accordance with Annex C. Discard any data obtained at flow rates outside these limits and repeat the corresponding part of the procedure using the proper flow rate.

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

Determine the threshold noise level of the APC using the method in A.2 before proceeding to 6.2.

Proceed to 6.2 to calibrate for particle sizes smaller than or equal to 30 µm(c) and for secondary calibrations for particles larger than 30 µm(c) using samples prepared in full conformance with Annex F. The procedure described in 6.2 through 6.10 shall not be used for primary calibration at particle sizes larger than 30 µm(c). Proceed to 6.11 for primary calibration at particle sizes larger than 30 µm(c). The procedure described in 6.11 through 6.14 shall not be used for particle sizes 30 µm(c) and smaller.

The procedure described in 6.2 to 6.15 assumes manual calibration of an APC with at least 8 channels that can be set at various threshold settings. Alternatively, calibration can be performed using a multi-channel analyser (MCA) or software that follows the same procedure. If an MCA is used, the relationship between the measured voltage of the MCA and the APC threshold setting shall be established. In general, software and MCA methods tend to be faster and more accurate than manual methods.