INTERNATIONAL STANDARD

ISO

11171

Fifth edition
2022-03

Hydraulic fluid power — Calibration of automatic particle counters for liquids

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

COPYRIGHT PROTECTED DOCUMENT

Contents

Page

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 fifth edition cancels and replaces the fourth edition (ISO 11171:2020), which has been technically revised.

The main changes are as follows:

— in term 3.8, particle size distribution, Note 1 to entry has been amended;

— in 6.12 c), 0,72 has been changed to 0,56;

— in 6.12 d) 1,32 has been changed to 1,56;

— in B.4, 3rd line, "mass concentrate" has been changed to "mass concentration";

— B.8 has been amended to read: ……"calculate the theoretical number concentration of particles for each mass concentration, X_t.";

— in Table B.1, the units for "X_t (particles/L)" have been corrected to "X_t (particles/mL)";

— in D.3 c), 0,72 has been changed to 0,56;

— in D.3 d), 1,32 has been changed to 1,56;

— in D.7 a), 0,72 has been changed to 0,56;

— in D.7 e), 1,32 has been changed to 1,56;

— in G.6, the subscript in the denominator of Formula G.3 has been corrected from "V_0" to "V_S";

— in Reference [6] in the Bibliography, the RM 8631 batch identifier has been changed from "a" to "b";

— in Reference [7] in the Bibliography, the RM 8632 batch identifier has been changed from "b" to "a".

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 glass and plastic ware — 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 terminology databases for use in standardization at the following addresses:

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

— IEC Electropedia: available at https://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 values for Diameter [μm(c)] and the corresponding Cumulative Particle Concentration (part/mL) given in 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 8632x 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 re-adjustment of an APC refers to service or repair procedures that affect the ability of the APC to accurately size and count particles.
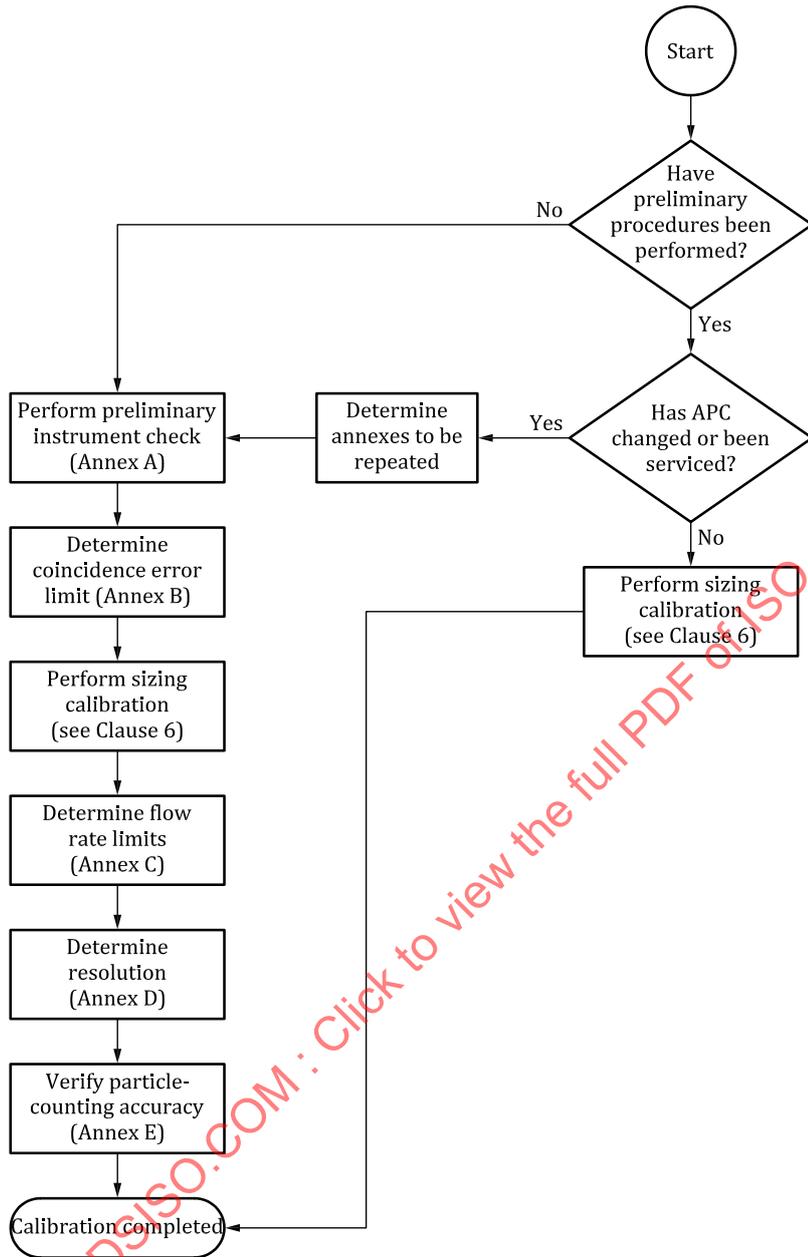
Figure 1 — Recommended sequence of APC calibration procedures

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 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 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 an 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 <u>Figure 2</u> 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 <u>4.4</u>) for primary calibrations or secondary calibration suspensions (<u>4.15</u>) prepared in accordance with <u>Annex F</u> for secondary calibrations. For particle sizes larger than 30 μm(c), use polystyrene latex spheres (see <u>4.1</u>) for primary calibrations or use secondary calibration suspensions prepared in accordance with <u>Annex F</u> 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 <u>Annex C</u>. 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 <u>5.2</u>. If a different volume is used, repeat the procedure in <u>5.2</u> using the new sample volume to avoid volume measurement errors.

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

Proceed to <u>6.2</u> 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 <u>Annex F</u>. The procedure described in <u>6.2</u> through <u>6.10</u> shall not be used for primary calibration at particle sizes larger than 30 μm(c). Proceed to <u>6.11</u> for primary calibration at particle sizes larger than 30 μm(c). The procedure described in <u>6.11</u> through <u>6.14</u> shall not be used for particle sizes 30 μm(c) and smaller.

The procedure described in <u>6.2</u> to <u>6.15</u> 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.
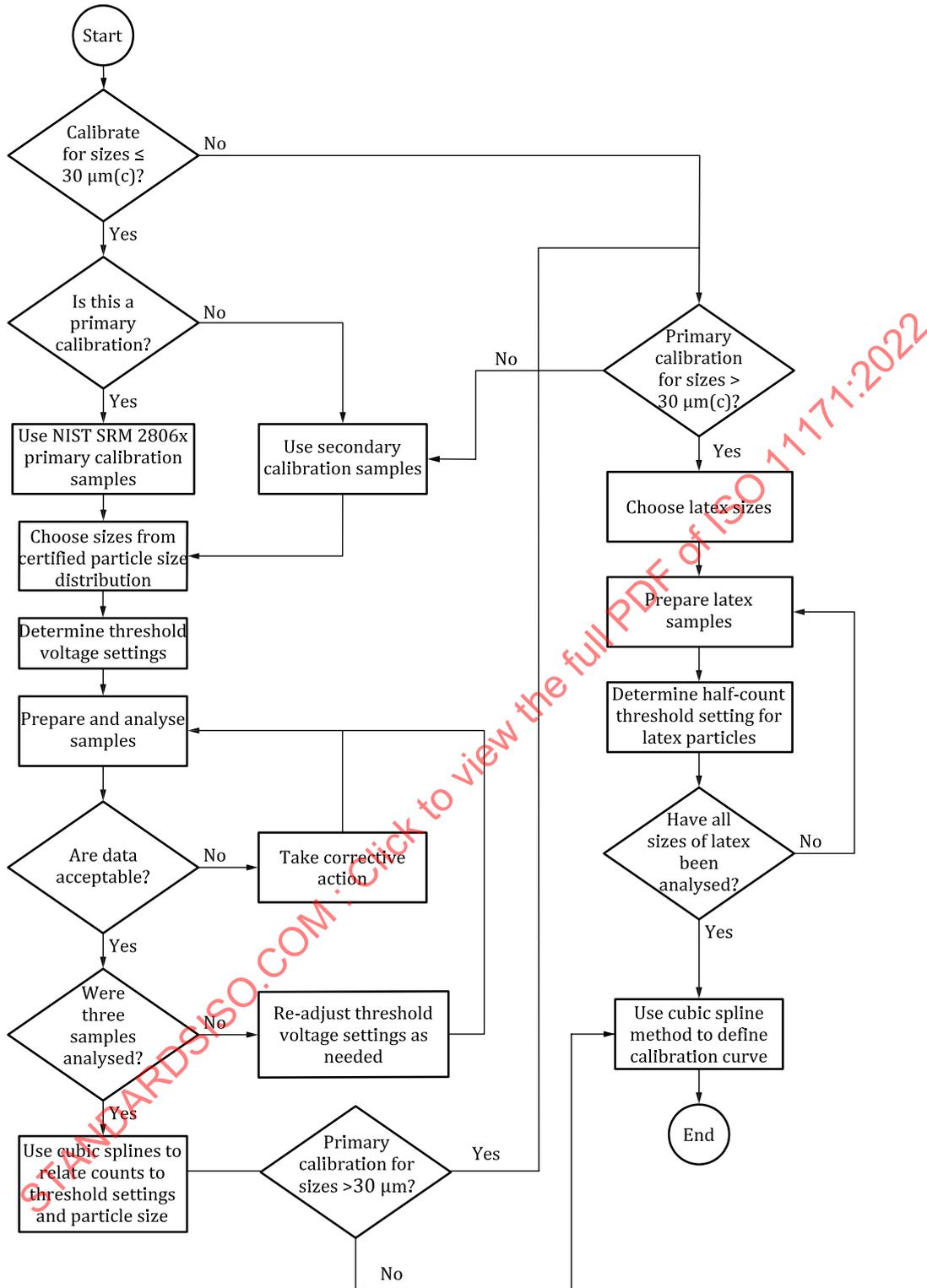
Figure 2 — Sizing calibration procedure

6.2 Choose at least 16 different particle sizes from those listed in the certified particle size distribution of the calibration suspension samples. The smallest particle size chosen shall be the smallest particle size of interest and the largest particle size shall not exceed 30 μm(c) for primary calibrations and

shall not exceed the largest reported particle size that is in conformance with Annex F for secondary calibrations.

Record these particle sizes and their corresponding particle concentrations from the certified particle size distribution in the corresponding columns of the Particle Size Calibration Summary of Table 4.

6.3 Determine a minimum of 12 different threshold settings to use in constructing a calibration curve. The first (lowest) threshold setting, J, shall be 1,5 times the threshold noise level of the APC. The highest threshold setting, H, shall correspond to a particle size of approximately 30 μm(c) or less for primary calibrations and shall correspond to a size that does not exceed the largest reported particle size that is in conformance with Annex F for secondary calibrations. The value of constant K shall be calculated using Formula (1):

$$K = 10^{(\log H - \log J)/(G-1)} \qquad (1)$$

where G is the number of threshold settings to be used to construct the calibration curve. Intermediate threshold settings between J and H shall be determined such that the value of each intermediate threshold setting shall be equal to the value of its preceding channel times K. It is permissible to round off the values of all threshold settings to the nearest value attainable by the APC, but the values of consecutive channels shall not be the same. Record the values determined for the threshold settings in the first column of Table 3.

6.4 Set the APC to the cumulative mode. Using at least eight different channels, set the channels such that the threshold settings are arranged in order of increasing voltage. The settings for these channels shall be selected from the list of threshold settings previously determined (6.3) and shall be distributed over this range of settings.

6.5 Prepare a calibration suspension sample for analysis. Shake the sample vigorously by hand. Agitate the sample ultrasonically for at least 30 s 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. Degas the sample under vacuum or ultrasonically until no surfacing bubbles are observed then analyse immediately. Obtain five consecutive particle counts, each consisting of at least 10 mL and 10 000 particles at the smallest threshold setting. Record the particle concentrations obtained for each threshold setting for each of the five counts in the appropriate cells of Table 2.

6.6 Calculate the mean observed number of particles counted, X, using Formula (2):

$$X = \overline{X} \cdot V \qquad (2)$$

where

$\overline{X}$ is the mean particle concentration, in particles per millilitre, of all acceptable counts from the sample for a particular channel;

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

Calculate the total number, N, of particles counted for each channel using Formula (3):

$$N = 5 X \qquad (3)$$

The value of N shall be greater than or equal to 1 000 in order to ensure statistically significant results for that particular channel. The value of $\overline{X}$ shall not exceed the coincidence error limit of the APC (5.3). If the value of $\overline{X}$ exceeds the coincidence error limit, prepare dilutions of the calibration samples as in accordance with Annex G and analyse them as described in 6.5.

Calculate D_Q, which is the difference expressed as a percentage between the minimum, X_{min}, and maximum, X_{max}, observed particle concentration for each channel in particles per millilitre, using Formula (4):

$$D_Q = \frac{X_{max} - X_{min}}{\overline{X}} \times 100 \qquad (4)$$

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

Using Table C.2, find the maximum allowable D_Q corresponding to the value of 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 eight channels with acceptable data, proceed to 6.7. If not, examine the results of any unacceptable channels as follows.

Calculate D_0 using Formula (5):

$$D_0 = \frac{X_{max} - X_{min}}{|X_0 - X_N|} \qquad (5)$$

where

D_0 is the outlier test parameter;

X_0 is the observed particle concentration of the suspected outlier (either X_{max} or X_{min}) in particles per millilitre;

X_N is the observed particle concentration closest in value to X_0 in particles per millilitre.

If D_0 for a particular channel is less than 1,44, discard the related outlier data point, X_0, from Table 2, then 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, all data from this channel are not acceptable and shall be discarded. If there are at least eight channels of acceptable data (using the D_Q and D_0 criteria), proceed to 6.7. If not, take appropriate corrective action and repeat 6.5 to 6.6.

If N is less than 1 000 for any channel, do not use the data for that channel. If sufficient numbers of particles counted is the only quality criterion that is not met, change the threshold setting to correspond to a smaller particle size that yields sufficient counts, or repeat 6.5 to 6.6 using a larger sample volume.

Do not collect and re-use primary and secondary calibration samples. Avoid the use of magnetic stirrers.

NOTE   Other failures to meet the quality criteria can arise from a number of sources, including contaminated diluent or glassware, volumetric errors, calculation errors, operating too closely 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. Excessive agitation or turbulence introduces bubbles into samples.

Table 2 — APC particle sizing calibration worksheet (see 6.6)

APC _____   Model _____   Date _____

Serial number _____   Operator _____

Sensor type _____   Model _____   Noise level _____ mV

Serial number _____   Flow rate _____ mL/min

First calibration suspension worksheet

First calibration suspension identification number

	Channel 1	Channel 2	Channel 3	Channel 4	Channel 5	Channel 6	Channel 7	Channel 8
Threshold setting (mV)								
Count 1 (particles/mL)								
Count 2 (particles/mL)								
Count 3 (particles/mL)								
Count 4 (particles/mL)								
Count 5 (particles/mL)								
$\bar{X}$ (particles/mL)								
D_Q								
$\bar{X}_N$ (particles/mL)								

Second calibration suspension worksheet

Second calibration suspension identification number

	Channel 1	Channel 2	Channel 3	Channel 4	Channel 5	Channel 6	Channel 7	Channel 8
Threshold setting (mV)								
Count 1 (particles/mL)								
Count 2 (particles/mL)								
Count 3 (particles/mL)								
Count 4 (particles/mL)								
Count 5 (particles/mL)								
$\bar{X}$ (particles/mL)								
D_Q								
$\bar{X}_N$ (particles/mL)								

Third calibration suspension worksheet

Third calibration suspension identification number

	Channel 1	Channel 2	Channel 3	Channel 4	Channel 5	Channel 6	Channel 7	Channel 8
Threshold setting (mV)								
Count 1 (particles/mL)								
Count 2 (particles/mL)								
Count 3 (particles/mL)								
Count 4 (particles/mL)								
Count 5 (particles/mL)								
$\bar{X}$ (particles/mL)								
D_Q								
$\bar{X}_N$ (particles/mL)								

Table 3 — Calibration curve worksheet

Threshold setting	$\bar{X}_N$ (particles/mL)			Mean (particles/mL)	N_C	Standard uncertainty (particles/mL)
mV	Sample 1	Sample 2	Sample 3			

6.7 Normalize the values for $\bar{X}$ recorded in Table 2 for each channel for a particular sample, using Formula (6):

$$\bar{X}_N = \bar{X} \cdot D_R \tag{6}$$

where

$\bar{X}_N$ is the mean normalized particle concentration, in particles per millilitre, of the undiluted calibration suspension sample for a particular channel;

$\bar{X}$ is the mean particle concentration, in particles per millilitre, of all acceptable counts from the sample for a particular channel (6.6);

D_R is the actual dilution ratio for the sample (G.8).

If the sample was not diluted, the value of D_R is 1. If the sample was diluted according to Annex G, use the value of D_R for the sample obtained in G.8. Record the value of $\bar{X}_N$ for each channel in the appropriate cells in Tables 2 and 3.

6.8 Repeat 6.4 to 6.7 two more times using different calibration suspension samples and approximately the same D_R as the first sample. Choose threshold settings for each sample such that at least two different samples are analysed for each of the threshold settings listed in Table 3 (6.3).

Note that a different undiluted calibration sample shall be used each time 6.4 to 6.7 is repeated to ensure that the APC calibration curve is based on data from 3 different primary or secondary calibration samples.

6.9 Define the relationship between particle size and threshold voltage setting using the constrained cubic spline technique. A spreadsheet to carry out this interpolation is provided at the following URN: https://standards.iso.org/iso/11171/ed-5/en.

To use the spreadsheet, open the worksheet labelled 6.9 and enter the threshold voltage settings and corresponding values of $\bar{X}_N$ for each calibration suspension sample taken from the first four columns of Table 3 (6.7) in the white data cells of columns A, B, C and D in order of decreasing value of threshold voltage setting. Enter only values of $\bar{X}_N$ in cells for threshold settings that were actually used for a given sample. Leave all other cells empty. Record in the fifth column of Table 3 the mean $\bar{X}_N$ for each threshold setting which is displayed in column E of the 6.9 worksheet. Calculate the standard deviation of all acceptable counts for all the samples listed in Table 2 (6.6) for each threshold setting. Calculate the standard uncertainty for each threshold setting using Formula (7):

$$s_N = \frac{s D_R}{\sqrt{N_C}} \tag{7}$$

where

s_N is the standard uncertainty in normalized particle concentration, in particles per millilitre, of the undiluted calibration suspension sample for a particular channel;

s is the standard deviation, in particles per millilitre, of all acceptable counts from all the samples (6.6) obtained from a particular channel;

D_R is the actual dilution ratio for the sample (G.8);

N_C is the total number of acceptable particle counts from all samples for a particular channel (i.e. 8 to 15).

Record in the appropriate cells of Table 3, the values of N_C and s_N for each threshold setting.

NOTE   Failure to enter the threshold voltage settings in the subclause 6.9 worksheet in the proper order results in error. The worksheet will display values of 0 for the interpolated threshold voltage settings in column H if the threshold voltage settings in column A are not consecutive or not in order of decreasing voltage. A value of 0 for the interpolated threshold voltage setting will also be displayed for any particle concentration that falls outside the range of the data in column E.

6.10 Determine the threshold voltage settings that correspond to the particle concentrations for each size selected in 6.2 using the same subclause 6.9 worksheet. Enter, in order of increasing particle size,

the size and corresponding particle concentrations from Table 4 in the blue data cells of column F and G. The worksheet displays the interpolated threshold voltage setting corresponding to each size in yellow column H. The values of extrapolated points are displayed as "0". Record the interpolated threshold voltage settings for each of these sizes in the right column of the Particle Size Calibration Summary of Table 4 that corresponds to this size. The use of extrapolated data is forbidden. Proceed to 6.11 to continue calibration for particle sizes larger than 30 μm(c) for primary calibrations or for particle sizes greater than the largest size listed on the certificate of analysis for secondary calibrations. If the APC is not to be calibrated for larger particle sizes, proceed to 6.15.

6.11 Obtain polystyrene latex spheres (4.1) with nominal diameters larger than 30 μm and spanning the particle size range of interest. The particle size of the smallest polystyrene latex sphere (4.1) chosen shall be between 35 μm and 45 μm. The particle size of the next largest latex spheres shall be approximately equal to the size of the smallest latex sphere times a constant with a value between 1,1 and 1,5. Subsequent larger latex particle sizes shall be approximately equal to the largest preceding latex particle size times a constant with a value between 1,1 and 1,5. Prepare the polystyrene latex suspensions for each size of latex spheres as described in D.2.

6.12 Set the APC to the differential mode and set the first four channels of the APC at threshold settings, 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 expected to correspond to the polystyrene latex sphere size;

c) set the second channel to the threshold voltage setting corresponding to 0,56 times the threshold voltage setting of the third channel;

d) set the fourth channel to the threshold voltage setting corresponding to 1,56 times the threshold voltage setting of the third channel.

Use only the differential particle counts of the second and third channels determined in 6.13 through 6.15 to determine the threshold setting corresponding to the polystyrene latex sphere size.

6.13 Prepare and analyse the diluted polystyrene latex sphere sample as described in 6.5, but obtain at least 2 500 particles in the second channel, instead of 10 000 particles at the smallest threshold setting. Evaluate the particle count data using the criteria described in 6.6. If D_Q is less than the appropriate value shown in Table C.2 for the second and third channels, proceed to 6.14. If not, discard the data and suspect an error in the analytical technique. Repeat 6.11 to 6.13 after taking appropriate corrective action (see NOTE in 6.6).

NOTE   To determine the half-count setting more rapidly, it may be desirable to perform 6.12 to 6.14, but obtain only one count (instead of five as indicated in 6.13) 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 6.14 is achieved. Following this, repeat 6.12 to 6.14 using five counts as described in 6.13.

6.14 Calculate the difference in observed particle counts, D, expressed as a percentage, between the second and third channels using Formula (8):

$$D = \left(1 - \frac{N_2}{N_3}\right) \times 100 \tag{8}$$

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. If not, and if the value of D is negative, the threshold settings selected in 6.12 are too high. If D is positive, the threshold settings selected in 6.12 are too low. Re-adjust the second, third and fourth channel settings while maintaining the relationship among these three channels as described in 6.12. Repeat 6.12 to 6.14 using the re-adjusted threshold settings. 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.

For each latex sphere size, record the mean latex particle diameter in the particle size column and the corresponding mean threshold setting in the threshold setting column of the Particle Size Calibration Summary of Table 4. Do not record a particle concentration and leave this data cell empty.

Repeat 6.12 to 6.14 using the next size of latex spheres until all latex samples prepared in 6.11 have been analysed and then proceed to 6.15.

6.15 Define the APC calibration curve by interpolation of the particle size data and corresponding mean threshold voltage settings listed in the Particle Size Calibration Summary of Table 4 using the constrained cubic spline technique. A spreadsheet to carry out this interpolation is provided at the following URN: https://standards.iso.org/iso/11171/ed-5/en.

To use the spreadsheet, open the 6.15 worksheet and enter all of the particle sizes and corresponding mean threshold settings from Table 4 into the white data cells of column A and B in order of increasing particle size. Enter the values for all particle sizes of interest into the blue data cells of column C of the worksheet in order of increasing size. The worksheet displays the interpolated threshold setting corresponding to each size in yellow column D and the actual calibration curve. Extrapolation is forbidden and the spreadsheet displays extrapolated value as "0".

Table 4 — APC Calibration summary

APC Sensor	Model _____	Date _____
	Serial Number _____	Operator _____
	Model _____	Calibration type (primary or secondary) _____
	Serial Number _____	Calibration sample identification numbers _____

Noise level (Annex A) _____mV	Coincidence error limit (Annex B) _____ particles/mL
Sample volume (Annex A) _____mL	Working flow rate (Annex C) _____ mL/min
C_v, vol (Annex A) _____ %	Flow rate range (Annex C) _____ mL/min

Resolution (Annex D)

S_R	_____ µm	R_R _____ %
S_L	_____ µm	R_L _____ %
d	_____ µm	R _____ %

Verification of Particle Counting Accuracy (Annex E)		
Particle Size	Expected particle concentration	Observed particle concentration
µm(c)	Particles/mL	Particles/mL

Particle Size Calibration Summary (Clause 6)		
Particle Size	Particle Concentration	Threshold Setting
µm(c)	Particles/mL	mV

7 Data presentation

7.1 Report all particle sizes obtained using an APC calibrated in accordance with this document 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 2806x.

7.2   Retain completed Table 2, Table 3, Table 4, Table B.1 and Table C.1 on file for inspection.

8   Identification statement

Use the following statement in test reports, catalogues and sales literature when an APC is calibrated in accordance with this document:

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

Annex A
(normative)

Preliminary APC check

A.1   Conduct the preliminary APC check as set out in the flow chart in <u>Figure A.1</u> when a new APC is received, following the repair or re-adjustment of an APC or sensor.
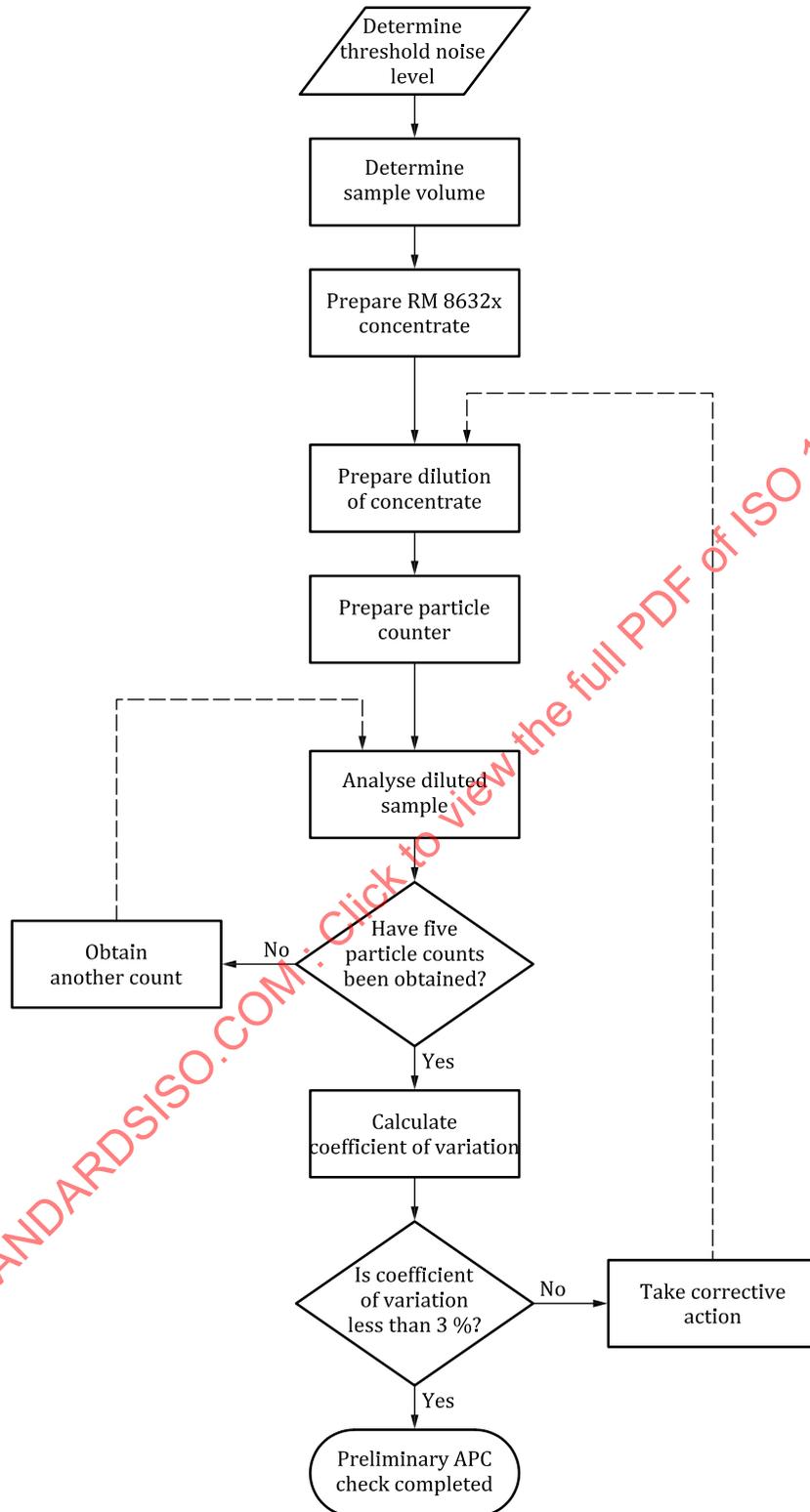

Figure A.1 — Preliminary APC check procedure

A.2   Determine the threshold noise level of the APC under no-flow conditions with clean diluent (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, re-adjust the APC. Record the APC and sensor model and serial number, the date, operator and the threshold noise level of the first channel in Tables 2, 4, 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.

A.3   Determine the sample volume actually measured during a particle-counting run using a method with traceability to a national or ISO standard. Record this value in Table 4 and use it to calculate particle concentrations in all subsequent work.

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

A.4   Prepare an RM 8632x concentrate of approximately 100 mg/L as follows:

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

b)   fill the bottle approximately 75 % full with an accurately measured amount (±1 mL) of clean diluent.

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 8632x;

V_0 is the volume, in millilitres, of clean diluent.

The RM 8632x concentrate, prepared in accordance with this subclause, is used to determine the coincidence error limit (Annex B) and flow rate limits (Annex C) of the APC, as well as to verify particle-counting accuracy (Annex E). Take special care in determining the dust concentration of the concentrate and 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 8632x concentrate ultrasonically for at least 30 s and then shake it on a mechanical shaker for at least 60 s to disperse the dust. De-gas the concentrate under vacuum or ultrasonically until no surfacing bubbles are observed and proceed immediately to A.6.

A.6   Calculate the amount of concentrate required to prepare a dilution that is about 25 % of the coincidence error limit for the sensor recommended by the manufacturer. Estimate the number concentration corresponding to a particular mass concentration using Table A.1.

Add accurately the required amount of concentrate and clean diluent (4.2) to a clean sample container in order to obtain the correct total volume of diluted RM 8632x suspension. Put a particle-free closure on the sample container.

Table A.1 — Particle size distribution for sensor performance verification (see A.6, B.4, E.4 and E.9)[a]

Particle size μm(c)	Median particle concentration (particles/mL greater than indicated size for a 1,00 mg/L sample of RM 8632x)	Particle concentration (particles/mL greater than indicated size for a 1,00 mg/L sample of RM 8632x) shall be	
		greater than or equal to	less than
2	20 210	14 180	28 820
3	10 700	7 928	14 440
4	6 300	4 553	8 713
5	3 475	2 330	5 183
6	1 631	938,2	2 834
7	769,8	413,6	1 433
8	379,1	179,7	799,5
9	196,2	76,57	502,6
10	88,90	27,89	283,4
[a] The particle concentrations in Table A.1 are for RM 8632a and can change if a different batch of RM 8632x is used.			

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 diluent, adjust the flow rate to the working flow rate. Record the flow rate in Tables 2, 4, B.1, C.1, and F.1.

Conduct all of the procedures at the same 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 appropriate flow rate.

A.8 Prepare and analyse the diluted RM 8632x suspension sample prepared in A.6 as described in 6.5. Calculate the coefficient of variation for volume measurement, $C_{V,vol}$, using Formula (A.2):

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

where

N_C is the number of consecutive particle counts performed on the sample (i.e. five);

$\overline{X}$ is the mean particle concentration, in particles per millilitre, for the five counts;

X_i is the particle concentration, in particles per millilitre, observed for count i.

A.9 The $C_{V,vol}$ shall be 3 % or less in order for the APC to be acceptable. Record the $C_{V,vol}$ in Table 4.

Although the $C_{V,vol}$ 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, only proceed with the calibration where the $C_{V,vol}$ is 3 % or less.

Annex B
(normative)

Coincidence error procedure

B.1   Determine the coincidence error limit as set out in Figure B.1 when a new APC is received or after the repair or re-adjustment 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 work requiring a higher degree of accuracy, analyse multiple dilutions of a sample 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.

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. Only data from this channel is used in this annex. 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 diluent (see 4.2). Flush the sensor with at least two 150 mL batches of clean diluent.

B.4   Prepare dilutions of the concentrate (see 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 concentration of RM 8632x 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 8632x concentrate in accordance with A.5. Accurately add the required amounts of concentrate, V_1, and clean diluent (4.2) to the sample container in order to attain the correct total volume. Put particle-free closures on the sample containers.
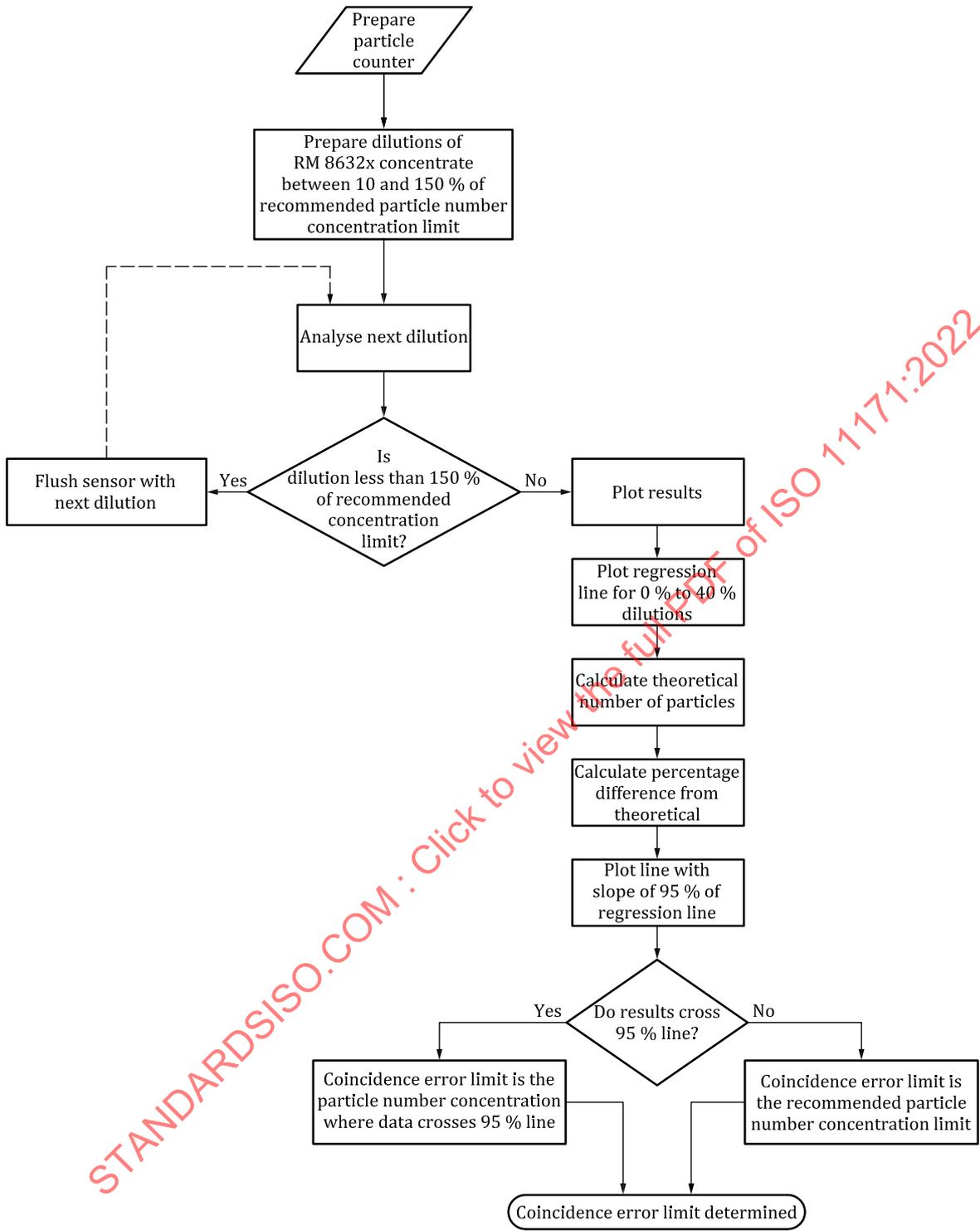
Figure B.1 — Coincidence error determination procedure

B.5   Analyse the samples prepared in B.4 as described in 6.5 in order of increasing particle number concentration.

Evaluate the particle count data using the criteria described in 6.6. If D_Q is acceptable, record in Table B.1 the mass concentration, in milligrams per litre, of the diluted sample, the corresponding observed particle number concentrations for each of the five counts, and the mean particle number concentration, $\bar{X}$, 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 to 6.6).

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 8632x, γ, on a linear-graph. Determine the linear regression line for the results from the 10 % to 40 % particle number concentration samples using the least squares method with the intercept set to 0. The regression equation shall pass through the origin and be of the form given in Formula (B.2):

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

where a is the slope of the regression line.

Plot the regression line just determined 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 concentration 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 equation obtained in B.7 to calculate the theoretical number concentration of particles for each mass concentration, X_t. Record the value of X_t for each mass 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 the value of E for each mass concentration in Table B.1.

B.10 Determine the coincidence error limit of the sensor by plotting a line passing through the origin with a slope of 95 % of the theoretical relationship (see B.7). The lowest particle number concentration, in particles per millilitre, at which the data line (B.7) 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 4 and Table B.1.

Table B.1 — Coincidence error data sheet

Particle counter _____ Model _____ Serial no. _____ Date _____ 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)											
$\bar{X}$ (particles/mL)											
X_t (particles/mL)											
E (%)											

Coincidence error limit (particle counts/mL) = _____

$X_t = a\, \gamma$

$a =$ _____

$r =$ _____

Annex C
(normative)

Flow rate limit determination

C.1   Perform the flow rate limit determination, as set out in Figure C.1, when a new APC is received or following the repair, or re-adjustment of an APC or sensor.

IMPORTANT — It is essential that the flow rate limits determined in accordance within this annex are followed in all APC calibration and sample analysis work. APC calibration is sensitive to flow rate. At high flow rates, the electronics may not determine the size of the particle correctly. The time interval separating successive particles can be so small that the electronics are unable to distinguish them as individual particles.
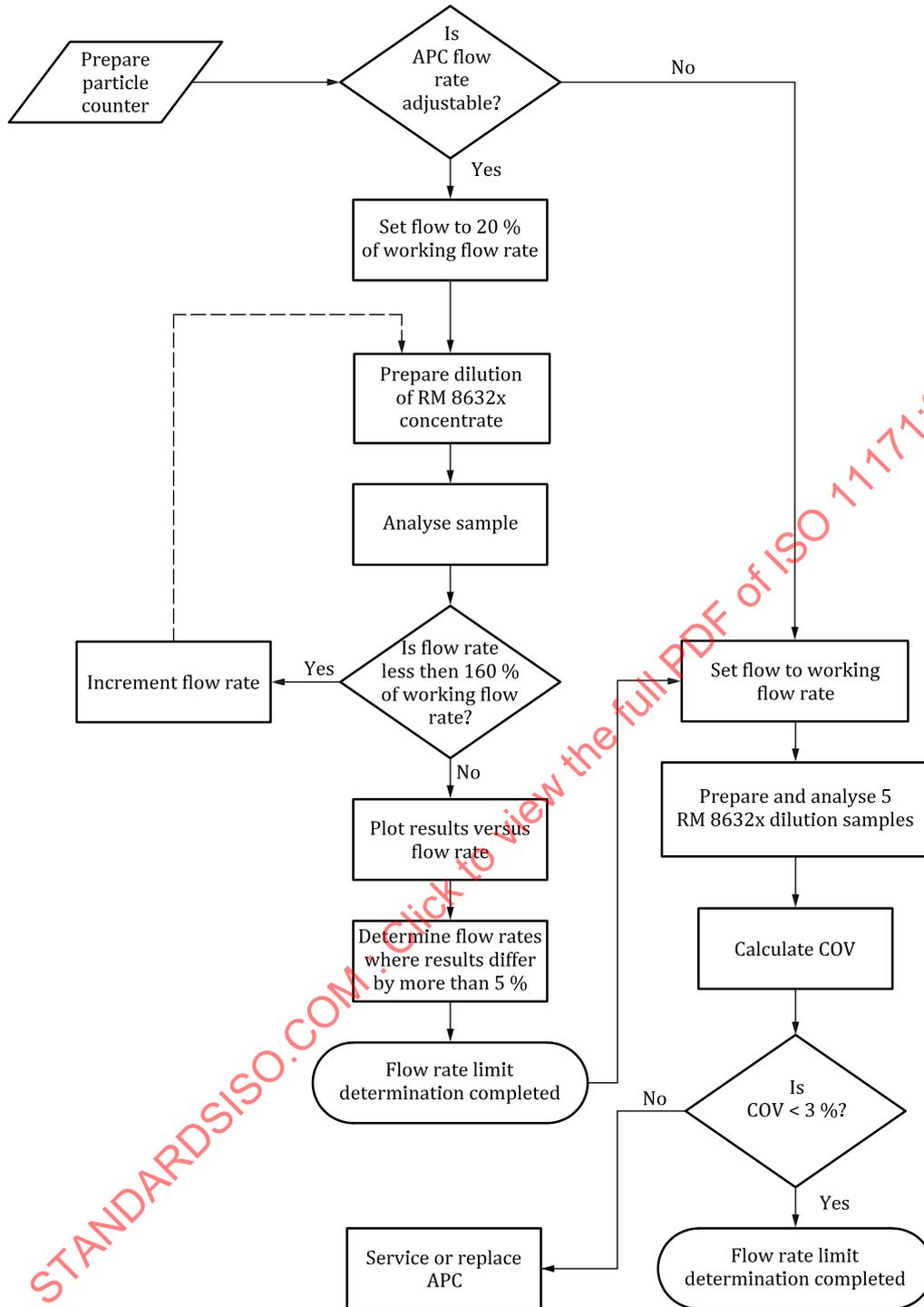
Figure C.1 — Flow rate limit determination procedure

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).

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 6.15 to set the first channel to 1,5 times the threshold noise level of the APC or the smallest particle size of interest. Ignore the data from other channels during the flow rate limit determination. Let the flow rate number, $n_{\text{fr}} = 1$.

NOTE   All channels to be in order of increasing threshold setting, unless otherwise recommended by the APC manufacturer.

C.5   Calculate the flow rate, q, in millilitres per minute, to be used according to Formula (C.1):

$$q = \frac{n_{\text{fr}}\, q_{\text{m}}}{5} \tag{C.1}$$

where

  n_{fr} is the flow rate number;

  q_{m} is the working flow rate, in millilitres per minute.

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

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

C.7   Prepare and analyse the RM 8632x sample prepared in C.6 as described in 6.5.

C.8   Record the flow rate and the number of particles observed at the channel corresponding to 1,5 times the threshold noise level of the APC or the smallest particle size of interest for each count in the appropriate column in Table C.1.

C.9   Evaluate the particle count data using the criteria described in 6.6. If D_{Q} is less than or equal to the appropriate value shown in Table C.2, proceed to C.10. 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.9 after taking appropriate corrective action (see NOTE to 6.6).

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 _____

_____

n_{fr}	Particle counts (C.8 and C.10)							
	1	2	3	4	5	6	7	8
Flow rate								
Count 1								
Count 2								
Count 3								
Count 4								
Count 5								
$\bar{X}$								

	Particle counts (C.14 and C.15)					
	Count 1	Count 2	Count 3	Count 4	Count 5	$\bar{X}$
Sample 1						
Sample 2						
Sample 3						
Sample 4						
Sample 5						
Mean	—	—	—	—	—	
Standard deviation	—	—	—	—	—	
C_V	—	—	—	—	—	

Table C.2 — Maximum allowable differences, expressed as percentages, in particle counts between runs (see 6.6, 6.13, B.5, C.9, D.4, D.9, E.6, E.9, F.5 and F.6)

If X (mean observed number of particles counted) is		Use these values for the maximum allowable D_Q for an individual sample	Use these values for the maximum allowable C_V when the number of samples analysed is (see E.9 and F.6)				
greater than or equal to	but less than	(see 6.6, 6.13, B.5, C.9, D.4, D.9 E.6 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.5 to C.10.

C.12 Plot the mean observed particle concentration, $\overline{X}$, in particles per millilitre, 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 Table 4, Table B.1 and Table C.1.

C.14 Establish a flow rate 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 the channel corresponding to 1,5 times the threshold noise level of the APC or the smallest particle size of interest for each count of each sample in Table C.1. Calculate the mean observed particle concentration, $\overline{X}$, in particles per millilitre, for each sample and record this result in Table C.1.

C.15 Calculate and record, in Table C.1, the mean, standard deviation and coefficient of variation of the five $\overline{X}$ for the samples analysed in C.14. If the coefficient of variation is less than or equal to 3 %, then the APC meets the flow rate specifications of this document. If the coefficient of variation is greater than 3 %, the APC is unacceptable and requires servicing or replacement.

Ideally, always use the APC at the working flow rate. Calibrate APCs at each flow rate for which they are used. Always use the APC within the flow rate limits determined in accordance with C.2 to C.13. If any of the data used for size calibration was obtained using flow rates outside these limits, repeat the size calibration within these flow rate limits.

Annex D
(normative)

Resolution determination

D.1   Perform the resolution determination as set out in Figure D.1 when a new APC is received or following the repair or re-adjustment of an APC or sensor.
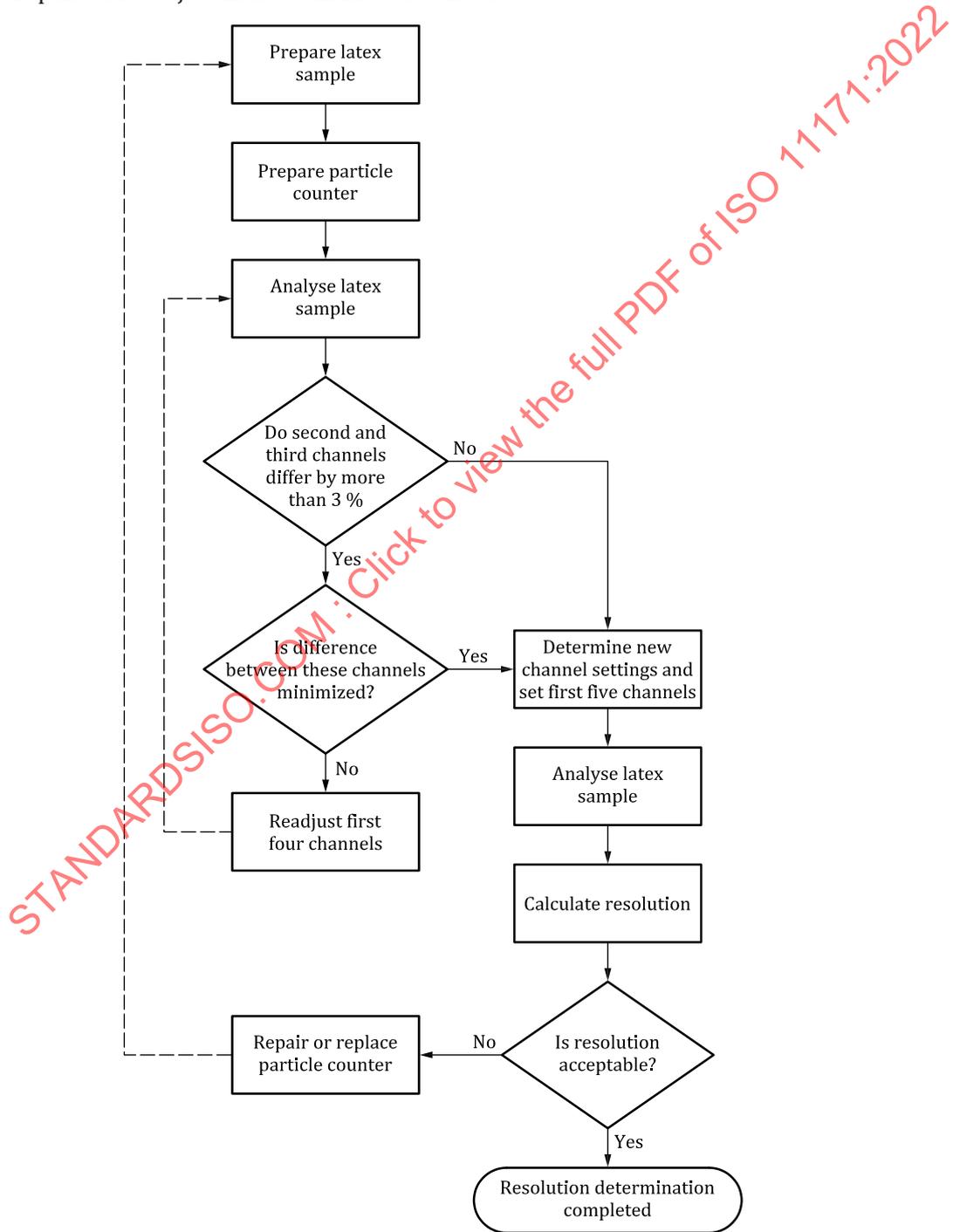

Figure D.1 — Resolution determination procedure

Determine resolution using a polystyrene latex sphere suspension with a nominal particle size of 10 μm (4.1). Measure the resolution at not less than 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 specified in D.3. Be aware that optical artefacts can occur that yield poor resolution results. If poor resolution is obtained contact the APC manufacturer to determine the reason and discuss the appropriate action.

Note that the procedures may be carried out manually or by the use of appropriate software for those APCs with microprocessor controls. Alternatively, resolution may be determined through use of an MCA. Such practice is acceptable, provided the relationship between the measured voltage of the MCA and the APC threshold setting is 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), V_0, in millilitres, that needs to be added to clean aerosol OT diluent (4.3) to achieve a particle concentration that is approximately 25 % of the coincidence error limit for the sensor 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 its certificate of analysis (see 4.1);

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

b) Shake polystyrene latex sphere 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;

c) 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 diluent (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 counted, shall be clear in appearance. Before proceeding, correct any cloudiness in the sample, which indicates insufficient dilution.

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 0,56 times the threshold voltage setting of the third channel;

d) Set the fourth channel to the threshold voltage setting corresponding to 1,56 times the threshold voltage setting of the third channel.

Use only the differential particle counts of the second and third channels to determine the threshold setting corresponding to the polystyrene latex sphere size.

     35

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 Clauses 5 and 6 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 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 mean polystyrene latex sphere size. These data are critical to determining sensor resolution. Inaccurate determination of the threshold setting corresponding to the mean polystyrene latex sphere size can result in apparently poor sensor resolution and failure of the APC to pass the sensor performance specification.

NOTE 2   To determine the half-count setting more rapidly, some labs first 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, and then re-adjust the channels as necessary and repeat the process until the half-count condition described in D.5 achieved. Following this, they repeat D.3 to D.6 using five counts as described in D.4.

D.4   Prepare and analyse the diluted polystyrene latex sphere sample as described in 6.5, but obtain at least 2 500 particles in the second channel, instead of 10 000 particles at the smallest threshold setting. Evaluate the particle count data using the criteria described in 6.6. If D_Q is less than the appropriate value shown in Table C.2 for the second and third channels, 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 6.6).

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

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

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. Re-adjust the second, third and fourth channel settings while maintaining the relationship among these three channels as described in D.3. Repeat D.3 to D.5 using threshold settings for the second, third and fourth channel which have been re-adjusted. 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 6.15, determine the particle size that corresponds to the threshold setting of the third channel. Using the calibration curve obtained in 6.15, 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 setting corresponding to 0,56 times the threshold setting of the third channel, channel C;

b)   Set channel B, the second channel, to the threshold voltage setting corresponding to a size 0,9 times the size of the polystyrene latex sphere determined in D.6;

c)   Keep channel C, the third channel, at the threshold voltage setting corresponding to the size of the polystyrene latex sphere determined in D.6;

d) Set channel D, the fourth channel, to the threshold voltage setting corresponding to a size 1,1 times the size of the polystyrene latex sphere determined in D.6;

e) Set channel E, the fifth channel, to the threshold voltage setting corresponding to 1,56 times the threshold setting of channel C.

Use only differential counts to determine resolution.

D.8 Prepare and analyse the diluted polystyrene latex sphere sample as described in 6.5, but obtain at least 2 500 particles in the second channel, instead of 10 000 particles at the smallest threshold setting.

D.9 Evaluate the particle count data using the criteria described in 6.6. If D_Q is less than the appropriate value shown in Table C.2 for the second and third channels, proceed to D.10. If not, discard the data and suspect an error in the analytical technique. Repeat D.8 after taking appropriate corrective action (see NOTE in 6.6).

D.10 Calculate the mean differential particle concentration for each channel.

D.11 Calculate the apparent standard deviation of the APC using Formulae (D.3) and (D.4):

$$s_L = \frac{d}{6\ln\left[1 + 2(N_B / N_A)\right]} \tag{D.3}$$

$$s_R = \frac{d}{6\ln\left[1 + 2(N_C / N_D)\right]} \tag{D.4}$$

where

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

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

d is the apparent polystyrene latex sphere size, in micrometres, obtained using the calibration curve obtained in 6.15 for the threshold setting of 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.12 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 4.

D.13 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 %, the resolution of the sensor is acceptable. 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 requires servicing or replacement. Alternatively, there might 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, identify and correct the error, and repeat the resolution determination procedure specified in D.2 to D.12.

NOTE   Cleaning the liquid-wetted portions of the APC system in accordance with the instructions of the manufacturer can help improve resolution.

D.14 If the APC fails to meet the resolution requirements of D.13, 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 0,55 and 1,45 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 6.15. 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 are 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 fit 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 observed and the APC failed the resolution requirements of D.13, the APC needs to be repaired or serviced or else a larger number of size increments are needed to verify the polystyrene latex sphere size distribution. If not observed, suspect a problem with the polystyrene latex spheres or with the sample preparation. Regardless of why the APC failed, obtain an acceptable resolution for the APC as defined in D.13. If resolution cannot be obtained, then the APC cannot be calibrated in accordance with this document.

Annex E
(normative)

Verification of particle-counting accuracy

E.1   Verify sensor particle-counting accuracy as set out in Figure E.1 when a new APC is received, or following the repair or re-adjustment of an APC or sensor.

E.2   Prepare three identical samples containing 1,00 mg/L of RM 8632x (4.7) suspended in clean diluent (4.2).

IMPORTANT — It is critical that the dust concentration in this sample is accurate, because the results obtained by analysis 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 8632x samples may be prepared in accordance with A.4 through A.6. When this method is used, be careful to avoid errors introduced by weighing, volume measurement and settling. Alternatively, consider purchasing 1,00 mg/L RM8632x samples from a reliable source that certifies its work.

E.3   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.4   Set the APC to the cumulative mode and set the threshold voltage settings to at least six different sizes selected from those particle sizes listed in Table A.1. 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 4 μm(c), 6 μm(c) and 10 μm(c). Use the calibration curve previously determined in 6.15 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 the range of sizes listed in Table A.1.

E.5   Adjust the flow rate to the working flow rate.

E.6   Analyse the samples as described in 6.5. If the data meet the quality criteria specified in 6.6, proceed to E.8. If they do not, discard the data and suspect an error in the analytical technique.

E.7   Repeat E.2 to E.6 after taking appropriate corrective action (see NOTE to 6.6).

E.8   Repeat E.3 to E.6 until three samples have been analysed.

```
        ┌─────────────────────┐
        │ Prepare 1 mg/L      │
        │ dilution of RM 8632x│
        │ concentrate         │
        └─────────────────────┘
                  │
        ┌─────────────────────┐
        │ Prepare particle    │
        │ counter             │
        └─────────────────────┘
                  │
        ┌─────────────────────┐
        │ Analyse sample      │
        └─────────────────────┘
                  │
              ╱───────╲
         No  ╱ Were three ╲
        ◄───╱ samples      ╲
            ╲ analysed?    ╱
             ╲───────────╱
                  │ Yes
        ┌─────────────────────┐
        │ Calculate mean      │
        │ and coefficient of  │
        │ variation           │
        └─────────────────────┘
                  │
              ╱───────╲
             ╱ Are mean ╲        Yes   ╭──────────────────────╮
            ╱ and coefficient ╲───────►│ Sensor performance   │
            ╲ of variation    ╱        │ verification completed│
            ╲ acceptable for  ╱        ╰──────────────────────╯
             ╲ all sizes?    ╱
              ╲───────────╱
                  │ No
              ╱───────╲
 ╭──────────╮  No ╱ Is there a ╲
 │ APC system│◄──╱ procedural or ╲
 │unacceptable│   ╲ analytical   ╱
 │ for use   │    ╲ error?      ╱
 ╰──────────╯     ╲───────────╱
                  │ Yes
        ┌─────────────────────┐
        │ Take corrective     │
        │ action              │
        └─────────────────────┘
```
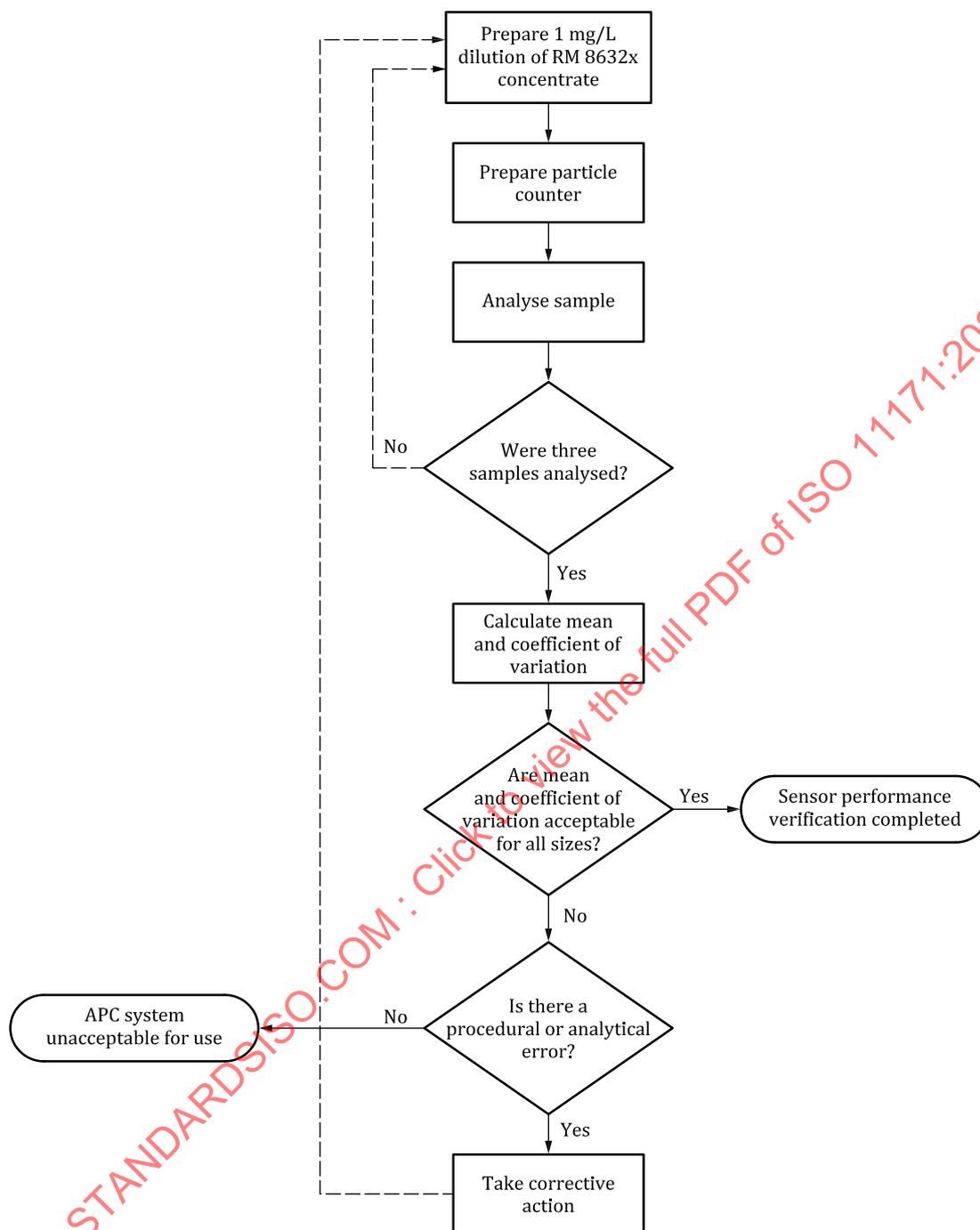
Figure E.1 — Verification of particle-counting accuracy