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ISO 11171

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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

Reference number
ISO 11171:1999(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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

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

This first editon of ISO 11171 cancels and replaces ISO 4402:1991, of which it forms a technical revision.

Annexes A to F form a normative part of this International Standard. Annexes G, H and I are for information only.

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

This document establishes a recommended standard calibration procedure for determining particle size and counting accuracy. The primary particle-sizing calibration is conducted using suspensions of ISO medium test dust (ISO 12103-A3 or ISO MTD) with particle size distribution certified by the National Institute of Standards and Technology (NIST). A secondary method with traceability to NIST uses suspensions of the same ISO MTD as the primary method but which are independently analysed using a particle counter calibrated by the primary method. Concentration limits are determined through the use of serial dilutions of a concentrated suspension.

Operation and performance limits are also established using this document.

Hydraulic fluid power — Calibration of automatic particle counters for liquids

1 Scope

This International Standard contains procedures for:

a) primary particle-sizing, sensor resolution and counting performance;

b) secondary particle-sizing calibration using suspensions prepared with NIST reference materials;

c) establishing acceptable operation and performance limits;

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

e) determining coincidence and flow rate limits.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

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

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

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

3 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 5598 apply, plus the following:

3.1
threshold noise level
the minimum voltage setting of the particle counter at which the observed pulse-counting frequency does not exceed 60 counts/min due to electrical noise

3.2
sensing volume
the 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

3.3

resolution

a measure of the ability of an instrument to distinguish between particles of different sizes

3.4

coincidence error limit

the highest concentration of ISO ultrafine test dust (ISO 12103-A1 or ISO UFTD) that can be counted with an automatic particle counter with less than 5 % error resulting from the presence of more than one particle in the sensing volume at a time

3.5

working flow rate

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

3.6

particle size

the projected area equivalent diameter of particles as determined by NIST using scanning electron microscopy or as determined using a liquid automatic optical single-particle counter (APC) calibrated in accordance with this International Standard, unless otherwise noted

3.7

particle size distribution

the number concentration of particles, expressed as a function of particle size

3.8

primary calibration

sizing calibration conducted in accordance with clause 6 of this International Standard using NIST standard reference material 2806 (see 4.4)

3.9

secondary calibration

sizing calibration conducted in accordance with clause 6 of this International Standard using calibration suspensions prepared in accordance with annex F of this International Standard

4 Materials and equipment

4.1 Latex spheres, nearly monodispersed, with a nominal diameter of 10 µm, suspended in aqueous suspension, are required in annex D for resolution determination. In certain situations, it may also be useful to use additional sphere sizes. Regardless, the coefficient of variation shall be less than 5 %. The supplier of the latex spheres shall provide a certificate of analysis with each batch that indicates the latex particle size was obtained using techniques with traceability to national or international standards.

Latex suspensions older than one year shall not be used unless the size distribution and cleanliness of the suspension has been verified. The size distribution and cleanliness of latex spheres can be verified using the method described in clause D.13 of this International Standard.

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

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

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

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

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

4.4 NIST standard reference material 2806 (SRM 2806) primary-calibration suspension samples, available from NIST. SRM 2806 is a suspension of ISO MTD in clean dilution fluid with a size distribution certified by NIST.

4.5 NIST reference material 8631 (RM 8631) dust, prepared by drying the dust for at least 18 h at 110 °C to 150 °C, required if secondary calibration is to be performed (see 6.1). RM 8631 is ISO MTD from the same lot of dust used to prepare SRM 2806 (4.4).

4.6 ISO MTD, dried for at least 18 h at 110 °C to 150 °C before use.

4.7 ISO UFTD, dried for at least 18 h at 110 °C to 150 °C before use.

Due to potential variation in particle size distribution among different batches of test dust, it is recommended that samples prepared in annexes A, B, C and E use the same batch of dust used to generate the data in Table 7. Samples of this dust are available as NIST reference material 8632 (RM 8632).

4.8 Automatic optical single-particle counter (APC) for liquids, with batch sampler.

4.9 Clean sample containers, with closures (appropriate bottle caps, for example), and **volumetric glassware** with a volume accuracy of ± 1 % or better. The cleanliness levels of the sample containers, closures and glassware shall be less than 0,5 % of the number of particles (larger than the smallest particle size of interest) expected to be observed in the samples. The cleanliness levels shall be confirmed by ISO 3722.

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

4.11 Ultrasonic cleaner, with a power density of 3 000 W to 10 000 W per 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.**

5 Sequence of APC calibration procedures

5.1 Refer to Figure 1. Conduct the procedures of this clause when a new APC is received or following the repair or readjustment of an APC or sensor (see Table 1). Proceed to clause 6 if neither APC nor 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 procedures of annexes A, B, C, D and E have previously been conducted and documented.

NOTE In this clause, repair or readjustment of an APC refers to service or repair procedures that affect the ability of the automatic particle counter to accurately size and count particles.

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

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

If the volume measurement system is repaired, replaced or readjusted, then repeat annex A.

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 particle counter, sensor or volume measurement system.

5.2 Perform the preliminary instrument 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.

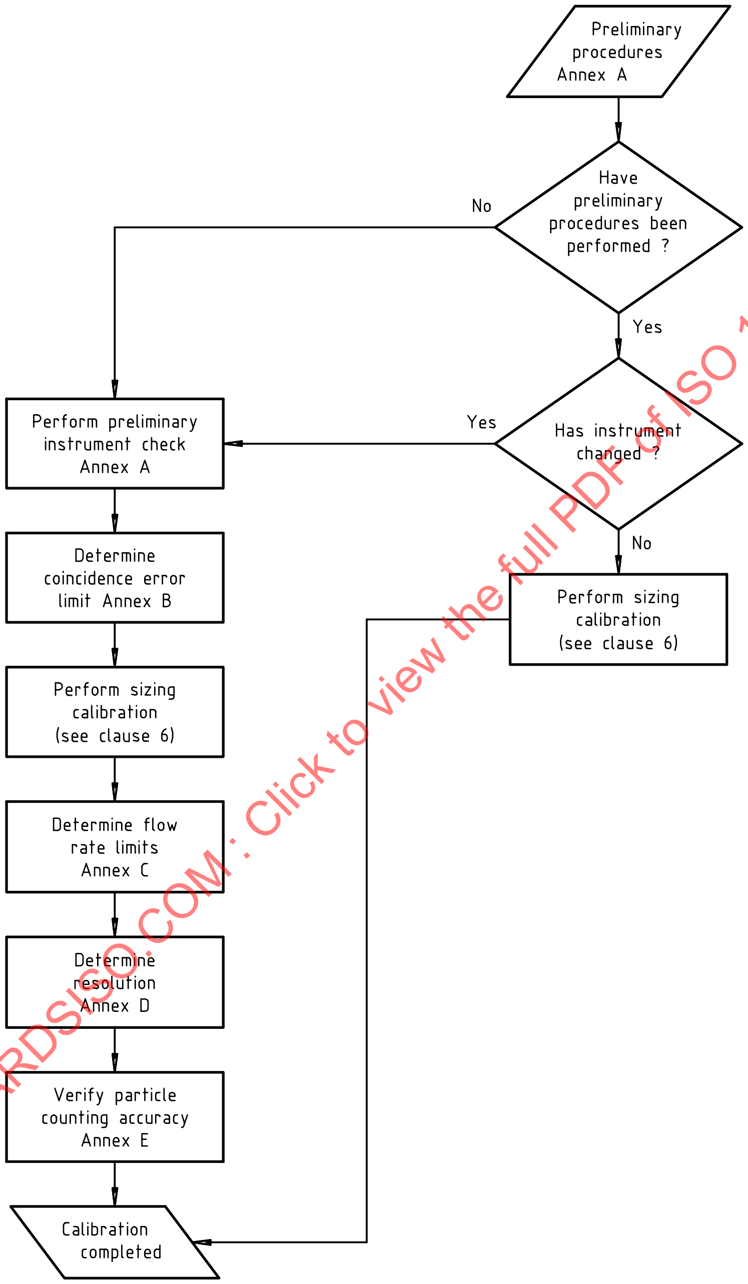


Figure 1 — Sequence of particle counter calibration procedures

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

5.6 Determine the instrument resolution in accordance with annex D.

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

5.8 In order to conform to the requirements of this International Standard, the APC shall be calibrated in accordance with 5.4 and shall meet the volume accuracy, resolution and sensor performance specifications determined in 5.2, 5.6 and 5.7, and shall be operated using the calibration curve determined in 5.4 within the coincidence error and flow rate limits determined in 5.3 and 5.5.

6 Sizing calibration procedure

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

Table 1 — Schedule of APC calibration procedures

If the APC status[a] is as described below:	**Then conduct the indicated clauses and annexes of this International Standard:**					
	Clause 6 **Sizing calibration procedure**	**Annex A** **Preliminary instrument check**	**Annex B** **Coincidence error limits**	**Annex C** **Flow rate limits**	**Annex D** **Resolution**	**Annex E** **Accuracy**
New instrument or existing APC not calibrated to this International Standard	X	X	X	X	X	X
Last calibration was more than 6 to 12 months ago	X					
Suspect calibration has changed significantly	X					
Optics (including light source) repaired or readjusted	X	X	X		X	X
Sensor or counting electronics repaired or readjusted	X	X	X	X	X	X
Volume measurement components (e.g. flow meter, burette, level detectors) repaired or readjusted		X				
Sensor cleaned	No action necessary					
Cables or peripheral equipment attached	No action necessary					
Plumbing lines and connections replaced	No action necessary					
Operation performed that does not involve disassembly of APC, sensor or volume measurement system	No action necessary					

[a] Repair or readjustment refers only to service or repair procedures that affect the ability of the automatic particle counter to accurately size and count particles. In order to verify the ability of an APC to accurately size and count particles, analyse a primary- or secondary-calibration suspension in accordance with 6.2 and 6.3, then compare the resultant particle concentration data to the corresponding particle size distribution for the sample. If the results agree within the limits given in Table 8, column 3, 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 the above table.

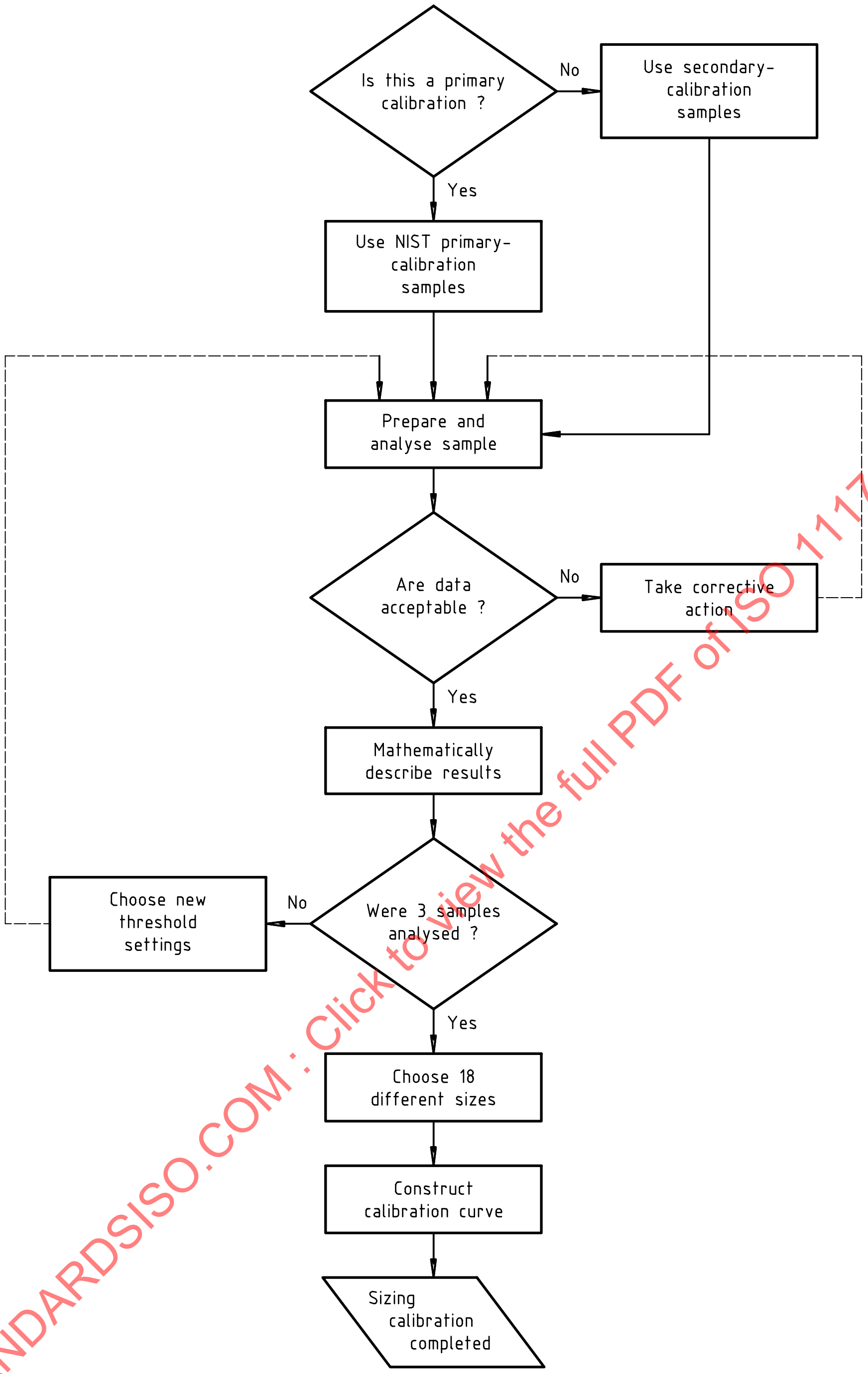


Figure 2 — Sizing calibration procedure

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

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

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

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

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

a) The lowest threshold setting shall be at least 1,5 times the threshold noise level of the instrument. This determines the minimum detectable particle size.

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

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

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

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

6.3 Degas the sample under vacuum or ultrasonically until the bubbles rise to the surface. Obtain five consecutive particle counts each consisting of at least 10 mL and 10 000 particles at the smallest threshold setting. The mean particle concentration for the five counts ($\overline{X}$) for each channel must be greater than or equal to 100 in order to have statistically significant results. Calculate the percent difference (D_Q) between the minimum (X_{min}) and maximum (X_{max}) observed particle count for each channel using the following equation:

$$D_Q = \frac{100\,(X_{\text{max}} - X_{\text{min}})}{\overline{X}}$$

Record in Table 4 the threshold voltage setting, particle concentration data, $\overline{X}$ and D_Q for each channel.

Using Table 8, find the maximum allowable percent difference corresponding to the value of $\overline{X}$ for each channel. If the value of D_Q is less than the maximum, then the value of $\overline{X}$ for that channel is acceptable for use. If there are at least six channels with acceptable data, proceed to 6.4.

If not, then examine the results of any unacceptable channels in the following manner:

Calculate D_0 using the following equation:

$$D_0 = \frac{\overline{X}_{\text{max}} - \overline{X}_{\text{min}}}{\left| X_0 - X_N \right|}$$

where

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

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

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

If sufficient numbers of counts is the only quality criteria which is not met, change the threshold settings to correspond to particle sizes which will yield sufficient counts or repeat 6.1 to 6.3 using a greater sample volume.

NOTE Other failures to meet the quality criteria may arise from a number of sources, including contaminated dilution fluid or glassware, volumetric errors, calculation errors, operating too close to the instrument's threshold noise level, 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 may occur. If excessively high stirring rates are used, particles may be centrifuged out or bubbles may be introduced.

The collection and reuse of primary- and secondary-calibration samples is prohibited.

6.4 Plot the particle concentrations (in particles per millilitre greater than the indicated size) versus the corresponding threshold settings (in mV) on a log-log graph using only the acceptable data points (determined as in 6.3). Use appropriate mathematical techniques to define the relationship between concentration and threshold setting as recommended by the particle counter manufacturer.

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

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

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

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

6.8 Construct a calibration curve using the relationship between particle concentration and threshold setting determined in 6.7. Choose at least 18 different particle sizes from the appropriate particle size distribution data. Choose only particle sizes which fall within the size range actually observed in 6.3 to 6.7. Record in Table 3 these 18 sizes, and the corresponding concentrations and threshold settings (determined using the concentration versus threshold setting plot constructed in 6.7). Plot the corresponding threshold settings versus particle size. Consult the APC manufacturer to determine the mathematical technique appropriate for defining the calibration curve. Use this mathematical technique to define the calibration curve and for interpolation. Extrapolation to sizes outside the size range used for calibration is not permitted.

NOTE This International Standard can only be used to calibrate APCs for sizes up to 50 µm(c). Some applications may require calibration at larger sizes. For particles larger than 50 µm(c), one may consider the use of other standards such as ASTM F 658-87. Regardless, the user is cautioned that particle counting at large particle sizes is subject to many sources of error. Among the most likely sources of error are (1) the settling of large particles during all phases of sample collection, handling and analysis and (2) inherently poor particle-counting statistics resulting from the typically low concentrations of large particles in hydraulic-oil samples.

ASTM F 658-87 is a size calibration method that uses monodispersed latex particles. In contrast, the calibration method described in this International Standard is a count calibration method using a polydispersed test dust. Both methods determine the relationship between APC threshold voltage and particle size. A size calibration method, such as ASTM F 658-87, can be used for particles larger than 50 µm(c) because the NIST particle size distribution used in this International Standard is also based on the projected-area diameter of the particles. The signal detected by APCs for particles larger than 50 µm(c) is not strongly dependent on the refractive index of either the particle or the liquid.

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

7 Data presentation

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

a) as "µm" or "micrometres", with the following statement: "The sizes quoted in this document were obtained using an automatic particle counter calibrated in accordance with ISO 11171:1999";

b) as "µm(c)", where (c) indicates APC calibration to ISO 11171:1999 (where possible, this shall be defined in the text).

7.2 Retain completed Tables 2, 3, 4, 5 and 6 on file so as to be available for inspection.

8 Identification statement

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

"Calibration of liquid particle counters conforms to ISO 11171:1999, *Hydraulic fluid power — Calibration of automatic particle counters for liquids*."

Table 2 — Coincidence error data sheet

Particle counter ____________ Model ______ Date ___________ Serial No. ___________ Operator _________ Sensor _____ Model ______ Serial No. _____

Noise level ___________ Flow rate ___ Channel setting _______ Flow rate limits ________

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

Coincidence error limit (counts/mL) = ________________

$X_t = a \times (\text{concentration}) + b$

See A.2, A.7, B.5, B.7, B.8, B.9, B.10 and C.12

a = ______________

b = ______________

r = ______________

Table 3 — Particle counter calibration summary

Particle counter __________ Model __________ Date __________

Serial No. __________ Operator __________

Sensor __________ Model __________ Last calibration date __________

Serial No. __________

Noise level __________ Sample volume __________ mL Flow rate __________

COV$_V$ __________ % Flow rate limits __________

Coincidence error limit __________ mg ISO ultrafine test dust/mL __________ particles/mL

s_R __________ µm(c) R_R __________ %

s_L __________ µm(c) R_L __________ %

d __________ µm(c) R __________ %

Sizing calibration

Calibration sample __________ Lot No. __________ Concentration __________

Size µm(c)	**Threshold setting** mV	**Observed particle concentration**

Verification of particle-counting accuracy

Size µm(c)	**Expected particle concentration (see Table 7)**	**Observed particle concentration**
5		
10		

Table 4 — Particle counter sizing calibration worksheet

Particle counter ____________

Model ____________________

Date _____________________

Serial No. _________________

Operator _________________

Sensor type ______________

Model ____________________

Calibration sample _________

Serial No. _________________

Lot No. ___________________

Noise level _______________

Flow rate _________________

Concentration _____________

Initial calibration sample ___________ **Calibration sample identification number** __________

Threshold setting						
Count 1						
Count 2						
Count 3						
Count 4						
Count 5						
$\overline{X}$						
D_Q						

Second calibration sample ____________ **Calibration sample identification number** _______

Size, µm						
Threshold setting						
Count 1						
Count 2						
Count 3						
Count 4						
Count 5						
$\overline{X}$						
D_Q						

Final calibration sample ___________ **Calibration sample identification number** __________

Size, µm						
Threshold setting						
Count 1						
Count 2						
Count 3						
Count 4						
Count 5						
$\overline{X}$						
D_Q						

Table 5 — Flow rate limit worksheet

Particle counter ____________ Model ______________________ Date _______________________

Serial No. ___________________ Operator ___________________

Sensor_____________________ Model ______________________

Serial No. ___________________

Noise level _______________________

Working flow rate _________________

Flow rate limits ____________________

Coincidence error limit _____________

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

Table 6 — Secondary-calibration suspension data sheet

Particle counter ____________ Model ______________________ Date _______________________

Serial No. __________________ Operator ___________________

Sensor ____________________ Model ______________________ Lot No. ____________________

Serial No. __________________ Concentration ______________

Noise level ________________ Flow rate __________________

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

Table 7 — ISO ultrafine test dust particle size distribution for sensor performance verification

Particle size µm(c)	Particle concentration (particles/mL greater than indicated size for a 1 mg/L sample of ISO ultrafine test dust) shall be greater than or equal to:	less than or equal to:
1 [a]	70 000	100 000
2 [a]	19 000	29 000
3 [a]	8 600	13 000
4	5 400	8 100
5	3 300	4 500
6	1 500	2 500
7	660	1 400
8	280	760
9	120	410
10	58	220
11	28	120
12	14	63
13	7,4	34
14	4,1	19
15	2,3	11

[a] The concentrations shown for these particle sizes are only guidelines based on the analysis of a small number of samples. Failure to obtain results within these limits for sizes smaller than 5 µm(c) does not mean that the particle-counting system is unacceptable, unless the system also fails to obtain acceptable results for particle sizes of 5 µm(c) and larger (see E.9).

Table 8 — Maximum allowable percent differences in particle counts between runs

If $\overline{X}$ (average number of counts) is		then use these values for the maximum allowable D_Q for individual samples (see 6.3, B.5, C.8, D.4, D.8 and F.5):	and use these values for the maximum allowable COV when the number of samples analysed is (see F.5 and E.9):				
greater than or equal to:	but less than:		$n = 3$	$n = 6$	$n = 10$	$n = 20$	$n = 40$
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

Annex A
(normative)

Preliminary instrument check

A.1 Refer to Figure A.1. Conduct the preliminary instrument check when a new APC is received, or following the repair or readjustment of an APC or sensor.

A.2 Determine the threshold noise level of the APC under no-flow conditions with clean dilution fluid (4.2) in the sensor in accordance with the manufacturer's instructions. Ensure that the noise levels do not differ significantly for all the instrument's channels. If significant differences occur, readjust the instrument according to manufacturer's instructions. Record the particle counter and sensor model and serial number, the date and the threshold noise level of the first channel in Tables 2, 3, 4, 5 and 6.

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

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

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

A.4 Prepare an ISO UFTD concentrate of about 100 mg/L in the following manner. Accurately weigh out the required amount of dry ISO UFTD (±0,1 mg) and transfer it to a clean sample bottle. Fill the bottle about three-quarters full with an accurately measured amount (±1 mL) of clean dilution fluid. Cover the bottle with a clean closure and shake vigorously. Calculate the dust concentration (c_A mg/L) in the concentrate using:

$$c_A = \frac{1000\, m}{V_0}$$

where

m is the mass of ISO UFTD, in milligrams;

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

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

A.5 Vigorously shake the concentrate by hand. Ultrasonically disperse the ISO UFTD concentrate for at least 30 s and then shake it on a mechanical shaker for at least 1 min to disperse the dust.

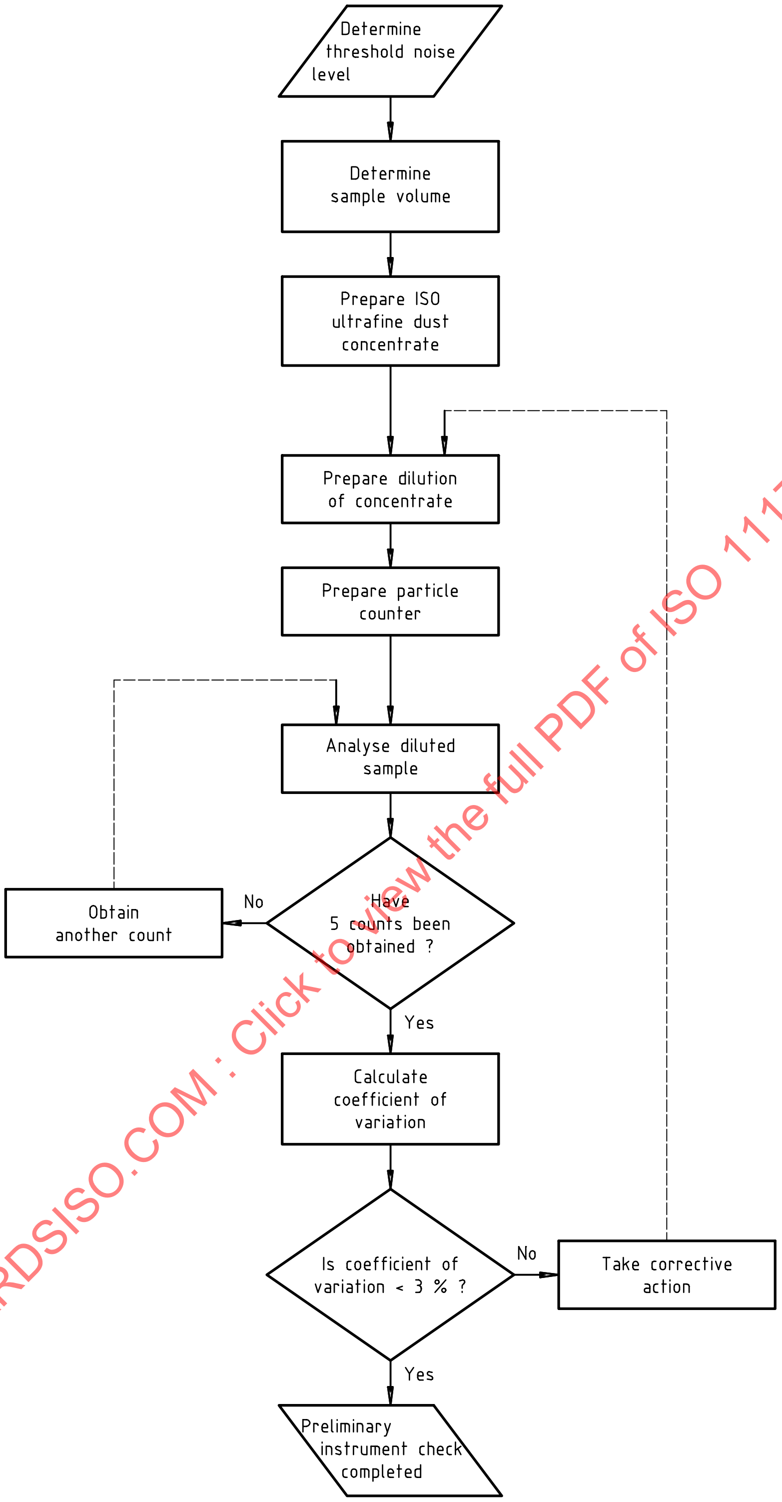


Figure A.1 — Preliminary instrument check

A.6 Calculate the amount of concentrate required to prepare a dilution that is about 25 % of the manufacturer's recommended concentration limit for the sensor. The number concentration corresponding to a particular mass concentration can be estimated from Table 7. Accurately add the required amount of concentrate and clean dilution fluid to a clean sample container in order to obtain the correct total volume of diluted ISO UFTD suspension. Put a particle-free closure on the sample container.

A.7 Set the APC to the cumulative mode. Set the lowest threshold setting of the instrument to 1,5 times the threshold noise level of the instrument. Using clean dilution fluid, adjust the flow rate to the working flow rate. Record the flow rate in Tables 2, 3, 4, 5 and 6.

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

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

$$\mathrm{COV_V} = \frac{100}{\overline{X}} \sqrt{\frac{n \sum_{i=1}^{n} X_i^2 - \left(\sum_{i=1}^{n} X_i \right)^2}{n(n-1)}}$$

where

n is the number of consecutive particle counts performed (i.e. 5);

$\overline{X}$ is the mean number of particles observed;

X_i is the number of particles observed for count i.

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

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

Annex B
(normative)

Coincidence error procedure

B.1 Refer to Figure B.1. Determine the coincidence error limit when a new instrument is received, or following the repair or readjustment of an APC or sensor.

The magnitude of the coincidence error at a given concentration is governed by the physical dimensions of the sensing volume as well as the sample's particle size distribution, including those particles too small to be counted. The coincidence error limit determined in this annex shall be regarded as a benchmark indicator that is useful for routine analyses. For other work, multiple dilutions of a sample shall be analysed to establish that the sample's size distribution does not yield a concentration limit significantly different from that determined with 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 instrument or corresponding to the smallest particle size of interest. Set the remaining channels to higher settings arranged in ascending order.

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

B.4 Prepare dilutions of the concentrate prepared as in A.4 which are 0 %, 10 %, 20 %, 30 % ... to 150 % of the manufacturer's recommended concentration limit for the sensor. Table 7 can be used to estimate the mass of ISO UFTD necessary to obtain the required particle concentration. Calculate the required amount of concentrate (V_1 mL) needed for each dilution using:

$$V_1 = \frac{c_\mathrm{M} V_\mathrm{S} L}{100 c_\mathrm{A}}$$

where

c_M is the manufacturer's concentration limit, in particles per millilitre;

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

L is the percentage of the manufacturer's limit;

c_A is the concentration of the concentrate, in particles per millilitre.

Disperse the ISO UFTD concentrate as described in A.5. Accurately add the required amounts of concentrate (V_1) and clean dilution fluid (see 4.2) containing antistatic additive to the sample container in order to attain the correct total volume.

Put particle-free closures on the sample containers.

B.5 Samples shall be analysed in order of increasing concentration and disperse particles as described in A.5.

Prior to counting, degas the diluted sample under vacuum or ultrasonically until the bubbles rise to the surface. Obtain five consecutive particle counts of at least 10 mL for each measured volume.

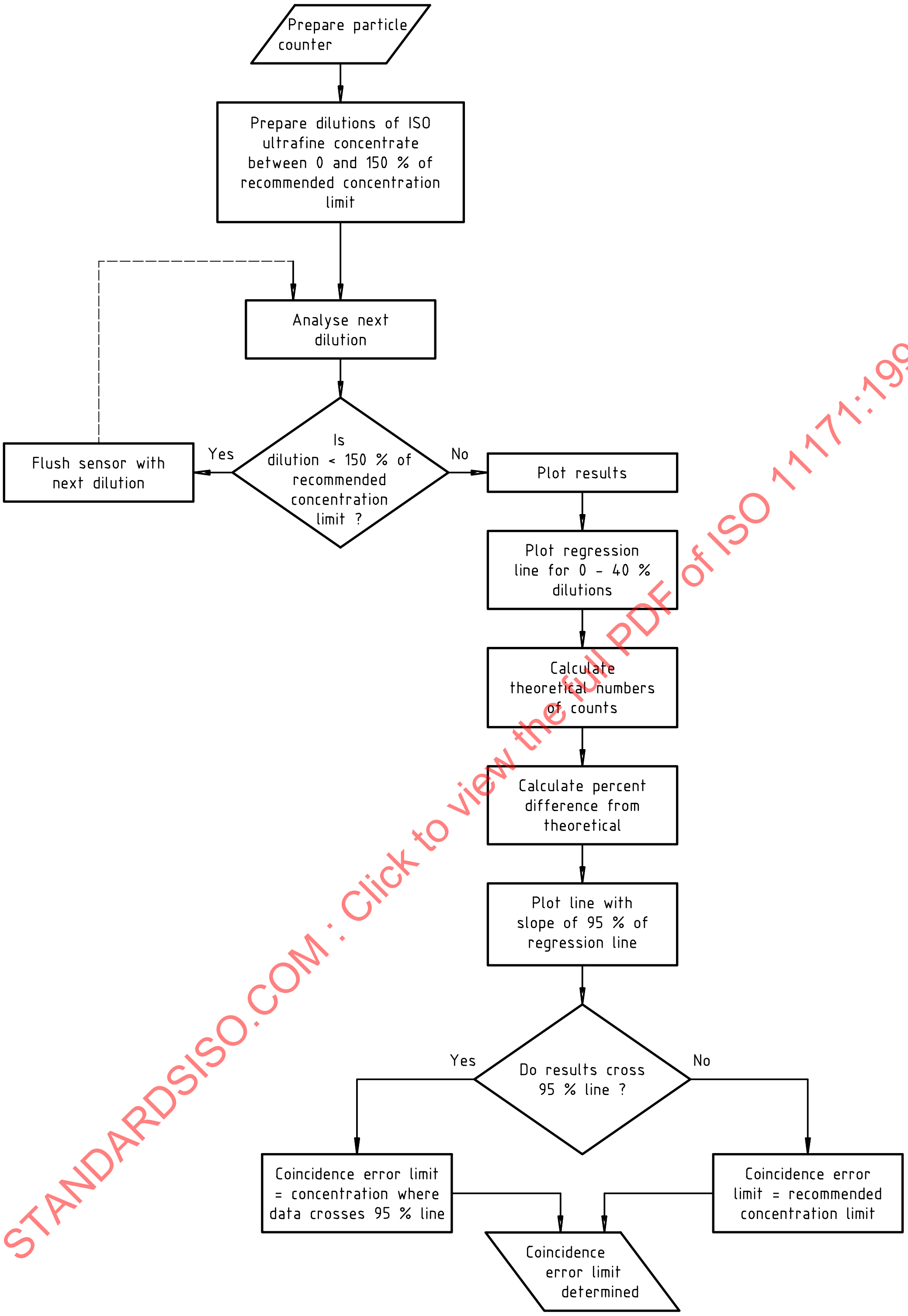


Figure B.1 — Coincidence error procedure

Evaluate the particle count data using the criteria described in 6.3. If D_Q is acceptable, record in Table 2 the concentration of the diluted sample (in mg/L), corresponding observed particle concentrations and the mean particle concentration ($\overline{X}$) before proceeding to B.6. If D_Q is too large, discard the data and suspect an error in the analytical technique. In this case, repeat B.4 and B.5 after taking appropriate corrective action (see note in 6.3).

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 number of counts as a function of particle concentration on a linear-linear graph. Determine the linear regression line for the results from the 0 to 40 % concentration samples using the least-squares method. Plot the regression line on the graph for concentrations ranging from 0 to 150 % of the manufacturer's recommended limit. This line defines the theoretical relationship between the number of particles and the concentration. Record in Table 2 the slope (a), intercept (b) and correlation coefficient (r) obtained by regression analysis.

B.8 Use the regression equation obtained in B.7 to calculate the theoretical number of counts for each concentration (X_t). Record the value of X_t for each concentration in the appropriate space in Table 2.

B.9 Calculate the percent difference (E) between the theoretical (X_t) and mean ($\overline{X}$) particle concentration for each dilution using the following equation:

$$E = 100\,\frac{(\overline{X} - X_t)}{X_t}$$

Record in Table 2 the value of E for each concentration.

B.10 Determine the coincidence error limit of the sensor by plotting a line with a slope of 95 % of the theoretical relationship (see B.7) and the same intercept (b) as the theoretical relationship on the graph constructed in B.7. The lowest concentration (expressed in terms of particles/mL) at which the data line crosses the 95 % line is the coincidence error limit of the sensor. If the data line does not cross the 95 % line, report the manufacturer's limit as the coincidence error limit of the sensor and use this value for all subsequent work. Record the coincidence error limit of the sensor in Tables 2 and 3 in terms of number of particles per mL.

Annex C
(normative)

Flow rate limit determination

C.1 Refer to Figure C.1. Perform the flow rate determination when a new instrument is received, or following the repair or readjustment of an APC or sensor.

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

C.2 Record the date, operator, APC and sensor model and serial numbers in Table 5.

C.3 Set the APC to the cumulative mode. Use the calibration curve determined in 6.8 to set the first channel to 5 µm(c) or the smallest particle size of interest. The data from other channels are ignored during the flow rate determination. Let the flow rate number (n) equal 1.

Unless otherwise recommended by the APC manufacturer, all channels shall be arranged in order of increasing threshold setting.

C.4 Calculate the flow rate (q) to be used as follows:

$$q = \frac{n\,Q_\mathrm{m}}{5}$$

where

n is the flow rate number;

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

Establish a flow rate approximately equal to q using clean dilution fluid.

C.5 Prepare a sample as described in A.4 to A.6.

C.6 Vigorously shake the sample by hand. Ultrasonically disperse the ISO UFTD sample for at least 30 s and then shake it on a mechanical shaker for at least 1 min to disperse the dust.

C.7 Degas the sample under vacuum or ultrasonically until the bubbles rise to the surface. Obtain five consecutive particle counts for volumes of at least 10 mL each for the dust sample prepared in C.5. Record the flow rate and the number of counts observed at 5 µm(c) or the smallest particle size of interest for each value of n shown in Table 5.

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

C.9 Calculate the mean observed particle concentration ($\overline{X}$ particles/mL) and record this result in Table 5.

C.10 If n equals or exceeds 8, proceed to C.11. If not, increment n by 1 and repeat C.4 to C.10.

C.11 Plot the number of counts as a function of the flow rate on a linear-linear graph and connect the data points by a smooth curve.

C.12 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 instrument. Record the flow rate limits in Tables 2, 3 and 5.

Ideally, the instrument will always be used at the working flow rate. Particle counters shall be calibrated at each flow rate for which they will be used. The instrument shall always be used within the flow rate limits just determined. If any of the data used for size calibration were obtained using flow rates outside these limits, the size calibration shall be repeated within these flow rate limits.

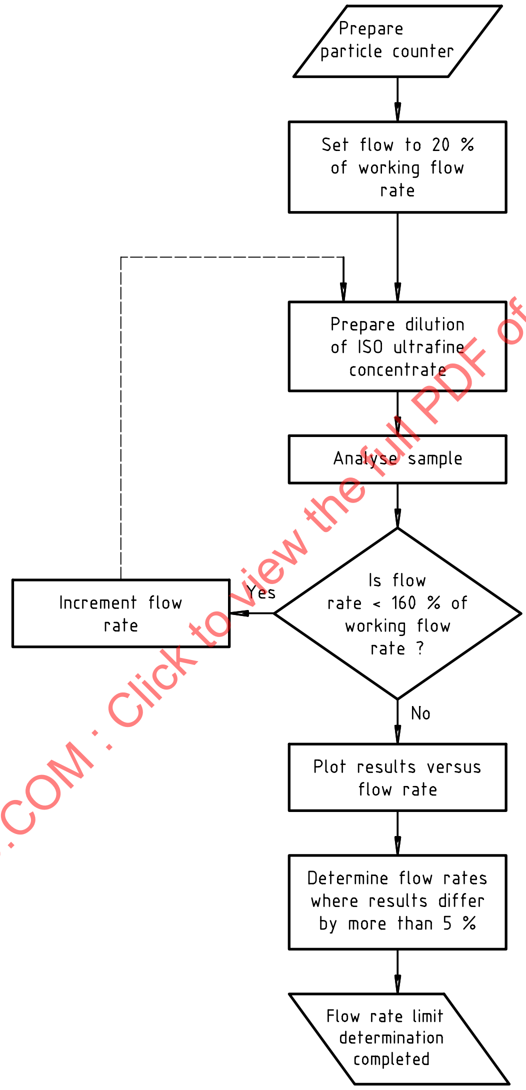


Figure C.1 — Flow rate limit determination

Annex D
(normative)

Resolution determination

D.1 Refer to Figure D.1. Perform the resolution determination when a new instrument is received, or following the repair or readjustment of an APC or sensor.

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

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

D.2 Prepare a sample of latex suspension as follows. Calculate the volume of latex concentrate (4.1) that must be added to clean Aerosol OT dilution fluid (4.3) to achieve a particle concentration that is approximately 25 % of the concentration limit for the sensor. Estimate the volume of concentrate from the equation:

$$V_0 = \frac{(V_S \, c_L \, \pi \, d^3)}{6 \times 10^{10} \, S}$$

where

V_0 is the volume of concentrate needed to make the latex suspension, in millilitres;

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

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

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

S is the percent solids on a mass basis in the latex concentrate (this value is typically supplied by the latex manufacturer).

Vigorously shake the concentrate by hand. Ultrasonically disperse it for 30 s and then mechanically shake it for at least 1 min to suspend the latex particles. Prepare a latex suspension by adding the required amount of latex concentrate (V_0) to a sample container approximately 75 % full of clean Aerosol OT dilution fluid (see 4.3).

Disperse the sample by mechanical shaking for at least 1 min.

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

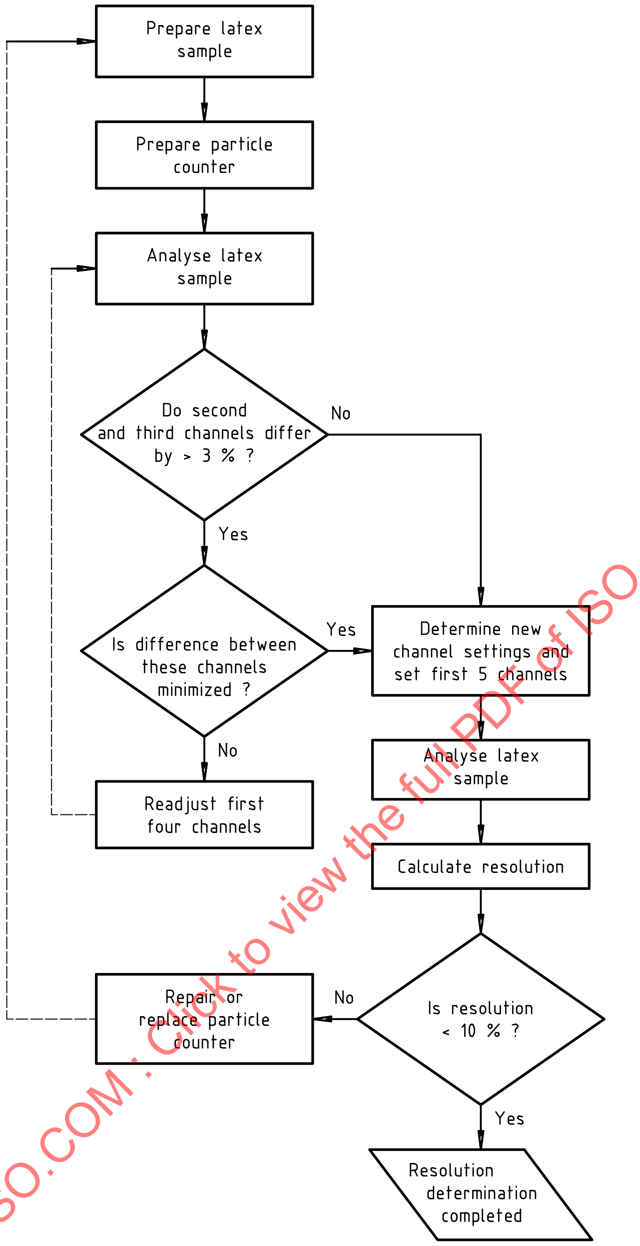


Figure D.1 — Resolution determination

D.3 Set the automatic particle counter to the differential mode. Use the calibration curve (see clause 6) to estimate the threshold setting that corresponds to the latex particle size, as follows:

- set the first channel to 1,5 times the threshold noise level of the instrument;
- set the third channel to the threshold voltage setting corresponding to the latex particle size;
- set the second channel to the threshold voltage setting corresponding to 0,72 times the threshold voltage setting of the third channel;
- set the fourth channel to the threshold voltage setting corresponding to 1,32 times the threshold voltage setting of the third channel.

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

NOTE 1 D.3 to D.5 describe the moving-window differential half-count method for determining the threshold setting which corresponds to the median latex particle size. These data are critical to determining sensor resolution. Inaccurate determination of the threshold setting corresponding to the median latex size can result in apparently poor sensor resolution and failure of the instrument to pass the sensor performance specification.

NOTE 2 Due to differences in the optical properties of the latex and the calibration dust (4.4), the apparent particle size obtained using a particle counter calibrated in accordance with this International Standard will differ from the size stated by the latex supplier. As a first approximation, choose a particle size in D.3 that is 10 % to 50 % larger than the particle size stated by the latex supplier.

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

D.4 Disperse the diluted latex sample by mechanical shaking for at least 1 min. Degas the sample under vacuum or ultrasonically until the bubbles rise to the surface. Obtain five consecutive particle counts, each consisting of at least 10 mL and 2 500 particles in the second channel. Evaluate the particle count data using the criteria described in 6.3. If D_Q is less than the appropriate value shown in Table 8, proceed to D.5.

If not, discard the data and suspect an error in the analytical technique. Repeat D.2 to D.4 after taking appropriate corrective action (see note in 6.3).

D.5 Calculate the percent difference (D) between the second and third channels using the following equation:

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

where

N_2 is the mean number of counts for the second channel;

N_3 is the mean number of counts 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 latex. In this case, proceed to D.6. If not and if the value of D is negative, the threshold settings in D.3 are too high. If D is positive, the threshold settings in D.3 are too low. Repeat D.3 to D.5 using threshold settings for the second, third and fourth channel which have been readjusted. If no threshold setting yields an absolute value for D less than 3 %, the threshold setting of the third channel yielding the minimum absolute value of D corresponds to the size of the latex.

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

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

- set channel A, the first channel, to the threshold voltage corresponding to 0,72 times the threshold voltage of channel C;
- set channel B, the second channel, to correspond to a size 0,9 times that of channel C;
- keep channel C, the third channel, at the threshold setting corresponding to the size of the latex (determined in D.5);
- set channel D, the fourth channel, to correspond to a size 1,1 times that of channel C;
- set channel E, the fifth channel, to the threshold voltage corresponding to 1,32 times the threshold voltage of channel C.

Only differential counts will be used to determine resolution.

D.8 Disperse the diluted latex sample by mechanical shaking for at least 1 min. Degas the sample under vacuum or ultrasonically until the bubbles rise to the surface. Obtain five consecutive particle counts, each consisting of at least 10 mL and 2 500 particles in the second channel. Evaluate the particle count data using the criteria described in 6.3. If D_Q is less than the appropriate value shown in Table 8, proceed to D.9.

If not, discard the data and suspect an error in the analytical technique. Repeat D.8 after taking appropriate corrective action (see note in 6.3).

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

D.10 Calculate the apparent standard deviation of the instrument using the following equations:

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

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

where

s_{L} is the apparent left or negative-side standard deviation, in micrometres;

s_{R} is the apparent right or positive-side standard deviation, in micrometres;

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

N_{A} is the mean number of counts for channel A;

N_{B} is the mean number of counts for channel B;

N_{C} is the mean number of counts for channel C;

N_{D} is the mean number of counts for channel D.

D.11 Calculate the positive- and negative-side resolution (R_R and R_L, respectively) using the following equations:

$$R_L = \frac{100\sqrt{s_L^2 - s_l^2}}{d}$$

$$R_R = \frac{100\sqrt{s_R^2 - s_l^2}}{d}$$

where s_l is the standard deviation of the latex stated by the supplier, in micrometres.

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

D.12 The resolution of the sensor is acceptable if R is less than or equal to 10 % for latex with a nominal size of 10 µm (see 4.1). If R exceeds 10 %, the particle counter is unacceptable and needs to be serviced or replaced. Alternatively, there may be an error in the procedure used to prepare or analyse the latex sample, or the latex itself may not meet the requirements of 4.1. In this case, the error shall be identified and corrected, and annex D repeated.

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

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

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

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.

Plot the differential results versus size and draw a smooth curve through the data. The curve should be Gaussian (bell-shaped) in appearance with no secondary peaks, and the differential percentages at the smallest and largest sizes should approach zero.

If this is observed and the instrument failed the resolution requirements of D.12, the particle counter needs to be repaired or serviced or else a larger number of size increments is needed to verify the latex particle size distribution. If this is not observed, suspect a problem with the latex or with the sample preparation. Regardless of why the instrument failed, acceptable resolution as defined in D.12 shall be obtained for the instrument to meet the requirements of this International Standard.

Annex E
(normative)

Verification of particle-counting accuracy

E.1 Refer to Figure E.1. Verify sensor performance when a new APC is received, or following the repair or readjustment of an APC or sensor.

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

E.3 Prepare three identical samples containing 1,00 mg/L of ISO UFTD (4.7) suspended in clean dilution fluid.

NOTE It is critical that the dust concentration in this sample be accurate since the results obtained by analysis of it will be compared to those in Table 7. Inaccuracies in sample preparation or counting can contribute to deviations from Table 7 that could result in rejection of an otherwise acceptable sensor. The three 1,00 mg/L ISO UFTD samples may be prepared from the concentrate prepared in A.4. When this method is used, be careful to avoid errors introduced by weighing, volume measurement and settling. Alternatively, one may wish to consider purchasing 1,00 mg/L ISO UFTD samples from a reliable source which certifies its work.

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

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

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

E.7 Degas the sample under vacuum or ultrasonically until the bubbles rise to the surface. Obtain five consecutive particle counts, each consisting of at least 10 mL and 10 000 particles at the smallest particle size for the sample. If the data meet the quality criteria described in 6.3, proceed to E.8.

If not, discard the data and suspect an error in the analytical technique. Repeat E.3 to E.7 after taking appropriate corrective action (see note in 6.3).

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

E.9 For each particle size, calculate the mean particle concentration ($\overline{X}$) and the coefficient of variation (COV in percent) for the three samples using:

$$\mathrm{COV} = \frac{100}{\overline{X}} \sqrt{\frac{n \sum_{i=1}^{n} \overline{X}_i^{2} - \left(\sum_{i=1}^{n} \overline{X}_i \right)^2}{n(n-1)}}$$

where

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

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

If the COV for each size is less than the corresponding values in Table 8, then compare the mean for each size to the appropriate limits given in Table 7. If $\overline{X}$ falls within the limits given in Table 7 for all six sizes, sensor performance has been verified. Report the published particle concentration and mean particle concentration for each size in Table 3.

If the COV is too large, discard the data and suspect an error in the sample preparation or analytical technique. Repeat E.3 to E.9 after taking appropriate corrective action.

If $\overline{X}$ falls outside the limits given in Table 7 for particle sizes greater than or equal to 5 µm(c), then the particle counter system is unacceptable and needs to be serviced or replaced. Alternatively, there may be an error in the procedure used to prepare or analyse the verification samples in the calibration. In the latter case, the error shall be identified and corrected and annex E repeated.

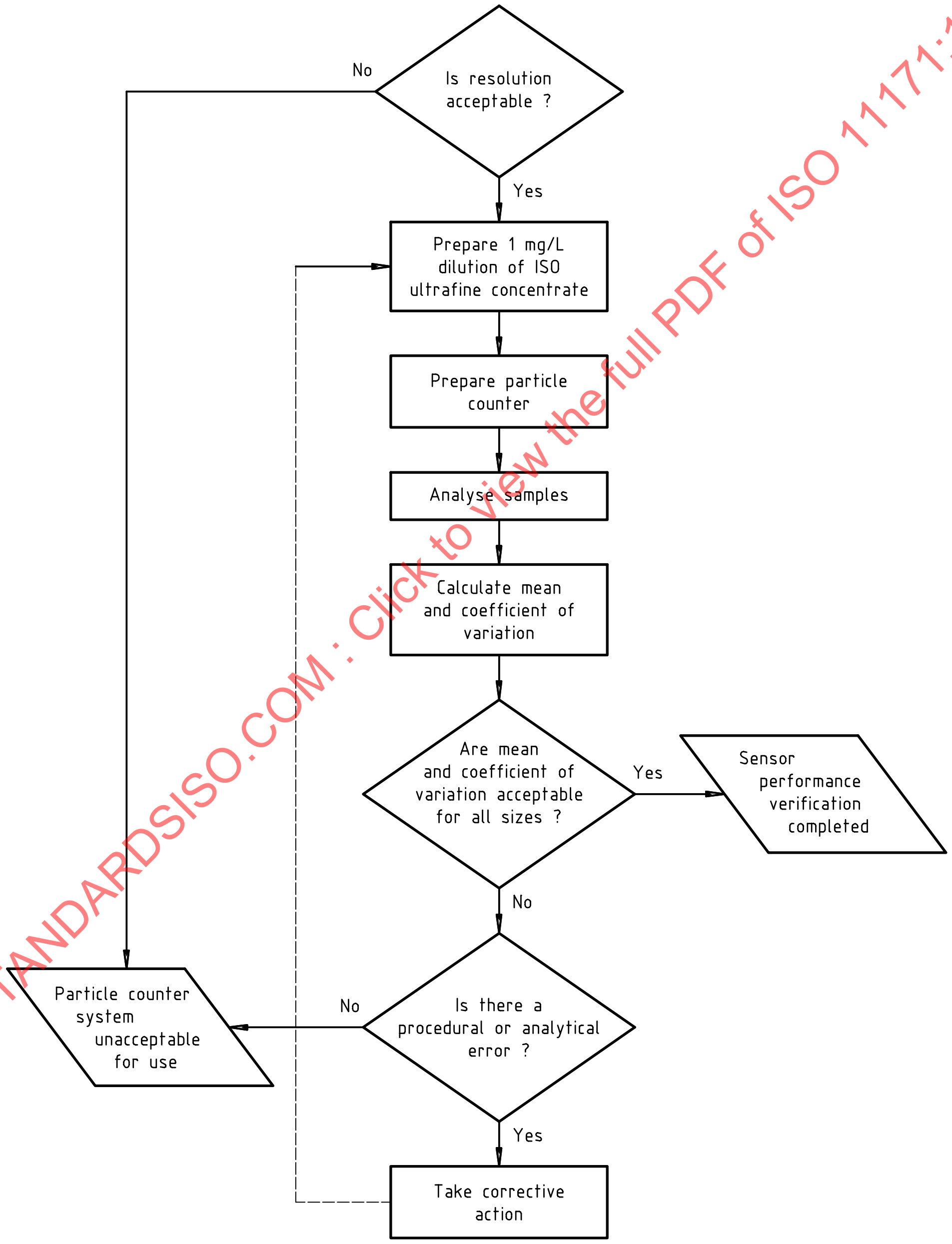


Figure E.1 — Verification of particle-counting accuracy

Annex F
(normative)

Secondary-calibration suspensions

F.1 Refer to Figure F.1. To prepare secondary-calibration suspensions, one APC, calibrated using NIST primary-calibration suspensions conforming with clauses 5 and 6, is needed for reference. Calibration of the reference particle counter with secondary-calibration samples is unacceptable. Secondary-calibration suspensions prepared in accordance with this annex can be used for sizing calibration instead of NIST primary-calibration suspensions for other purposes.

This annex shall be used to prepare secondary-calibration suspensions when, for economic or other reasons, it is desirable to utilize secondary-calibration suspensions instead of NIST calibration suspensions. For example, if more than one instrument needs to be calibrated, the use of secondary-calibration suspensions may be more practical.

F.2 Prepare a minimum of 30 secondary-calibration samples using a properly validated multipass test system (see ISO 16889) or a similar system capable of mixing a total volume of suspension of at least 8 L. Use ISO MTD (4.6) to prepare the samples. Use clean dilution fluid (4.2) as the fluid. The dust concentration shall be about 2 mg/L but the particle concentration shall not exceed 25 % of the coincidence error limit for the sensor. Collect and store samples prepared in this manner in clean, sealed containers with closures (4.9).

Due to possible batch-to-batch variability in test dusts, it is recommended that dust from the same batch used to generate the NIST primary-calibration suspensions be used to prepare secondary suspensions. This dust is available as NIST RM 8631.

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

F.3 Calibrate an APC in accordance with clauses 5 and 6 using NIST primary-calibration suspension samples (4.4).

The APC used to prepare the secondary-calibration suspension samples shall meet all particle counter performance specifications described in this document. Where a choice of instruments is available, the instrument with the best performance, in terms of coincidence error characteristics, threshold noise level, resolution, etc., shall be used.

F.4 Choose particle sizes and corresponding threshold voltage settings using the criteria described in 6.2.

F.5 Analyse, as per 6.2 and 6.3, every fifth sample prepared in F.2.

Evaluate the particle count data for each sample using the criteria described in 6.3. If D_Q is less than the appropriate value shown in Table 8, consider the data from that sample to be acceptable. If D_Q is too large, suspect an error in the analytical technique, discard the data and analyse the next consecutive sample only after taking appropriate corrective action (see note in 6.3).

For each threshold setting, calculate the mean, standard deviation and coefficient of variation using the equation shown in E.9.

If the COV for each threshold setting is less than the corresponding value taken from Table 8, then the mean, standard deviation and corresponding particle size data define the particle size distribution for these secondary-calibration suspensions and can be used for secondary calibration of other particle counters. Complete Table 6 by filling in the required data.

If not, reevaluate the sample preparation procedure, take appropriate corrective action and repeat F.2 to F.5.

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

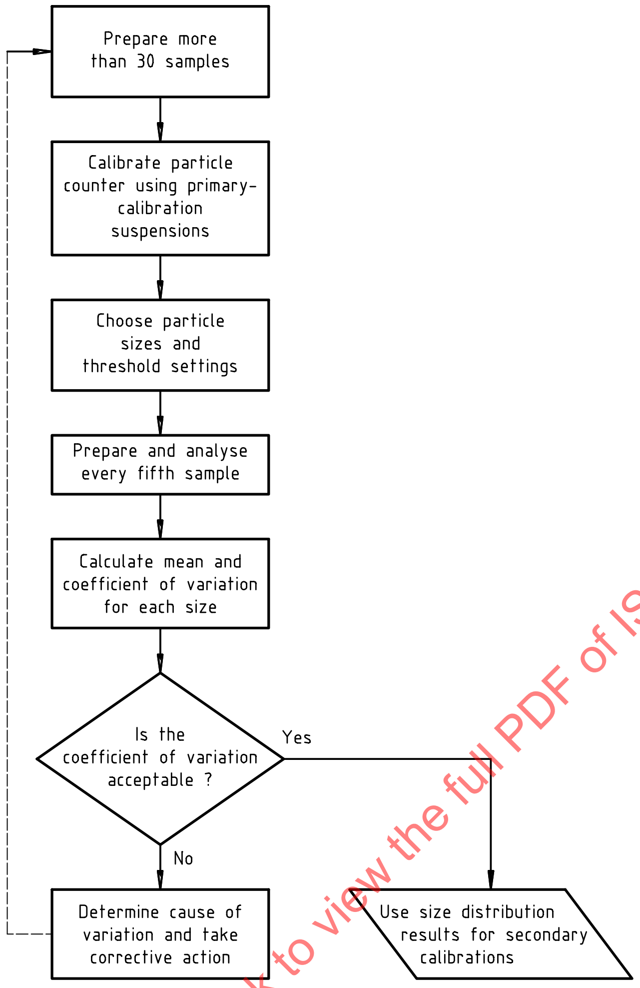


Figure F.1 — Secondary-calibration suspension preparation

Annex G
(informative)

Particle counter calibration round robin

G.1 Background

A round robin was conducted to evaluate this International Standard. Document ISO/TC 131/SC 8/WG 9 N111, an earlier draft of this International Standard, was used as the basis for the round robin. Only minor technical differences existed between the N111 draft and this International Standard. Twenty-seven laboratories participated in the round robin. Participants were supplied with samples of calibration fluid, 10 µm latex spheres suspended in oil, dry ISO ultrafine dust (the primary-calibration samples specified in 4.4 of this document, except that a NIST-certified size distribution was not available at the time of the round-robin. To facilitate the round robin, participants were provided a size distribution of these samples determined using the ISO 4402:1991 AC fine test dust (ACFTD) calibration method. When the certification of the primary-calibration samples was complete, the ACFTD sizes originally obtained in the round robin were converted to the new ISO 11171 sizes. Results were obtained from 29 different instruments, including light extinction and scattering sensors. Table G.1 summarizes the characteristics of the instruments used by each laboratory and any reported deviations from the procedure.

In order to establish a correlation between particle sizes obtained using the ACFTD and new calibration methods, three labs from the original round robin calibrated five particle counters conforming to ISO 4402:1991 and to this International Standard. Particle counters representing three different manufacturers and five different types of sensor, including two light-scattering and three light-extinction sensors were used. Table G.2 shows the resultant correlation between ACFTD particle sizes and the new NIST sizes obtained using this International Standard. This table was used to convert the ACFTD sizes obtained in the original round robin to NIST sizes.

Figure G.1 shows the particle size distributions of the four types of dust sample: calibration sample, verification sample, sample A and sample B. The lines represent the mean results reported by the participating labs for the type of sample indicated. The calibration samples were suspensions of ISO MTD in clean dilution fluid (4.2). These were prepared by the same lab that prepared the NIST primary-calibration suspension samples (4.4). ISO MTD is used in the multipass filter test procedure, ISO 16889. Verification samples were prepared from dry ISO ultrafine test dust by participating labs as described in annex E. The size distribution of ISO ultrafine resembles that of downstream samples taken during a multipass filter test. Like the verification samples, sample A was a 1,00 mg/L suspension of ISO ultrafine dust in clean dilution fluid, except that these samples were all prepared by the same laboratory. Sample B was a suspension of Powder Technology Inc. 0 to 30 µm dust in clean dilution fluid. Its particle size distribution resembles that of upstream samples taken during a multipass filter test.

G.2 Data analysis techniques

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

$$\overline{X}_{\log} = \frac{\sum_{i=1}^{n} \log \overline{X_i}}{n}$$

$$s = \frac{\sqrt{\sum_{i=1}^{n} \left(\log \overline{X}_i - \overline{X}_{\log}\right)^2}}{n-1}$$

$$\mathrm{COV}_{\log} = \frac{100s}{\overline{X}_{\log}}$$

where

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

n is the number of instruments submitting data for the indicated size and type of sample;

$\overline{X}_{\log}$, s and $\mathrm{COV}_{\log}$ are reported in the “statistics” sections of Tables G.3, G.4 and G.5. The results are shown in graphical form in Figures G.2, G.3 and G.4.

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

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

$$\overline{X}_{\mathrm{U}} = 10^{2s}\ \overline{X}_{\mathrm{G}}$$

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

Using the ($\overline{X}_{\mathrm{G}}$) and s results, the 95 % confidence intervals for particle size, d_{U} and d_{L}, respectively, were obtained for each type of sample. The particle size distributions were determined by linear regression of the corresponding ($\overline{X}_{\mathrm{G}}$) and particle size data. To avoid statistical artifacts, only data for sizes with $n > 5$ were used for regression. Tables G.3 to G.5 report the mean diameters calculated using the regression equation, d, d_{U} and d_{L}.

G.3 Sizing calibration procedure

Refer to figures G.2 to G.6. No problems were encountered with this part of the procedure. Several labs found the data quality test useful for rejecting outliers, without the need to analyse additional samples. Others reported using this test to identify and isolate analytical problems. The data quality (D_Q) test also aided operators to determine a sample volume appropriate for calibration and sample analysis work. For example, sample volumes larger than 10 mL must be used to calibrate for sizes larger than about 25 µm(c). Most labs calibrate at the particle sizes of greatest interest to them. Several generated calibration curves by repeating the process three times using exactly the same threshold settings each time. Since this makes interpolation to intermediate sizes less accurate, this International Standard now requires that different threshold settings be used each time 6.1 to 6.5 is repeated. This ensures that the particle counter calibration curve will be based on data representing at least 18 different sizes. When attempting to define a calibration curve mathematically, consult the particle counter manufacturer. Curve-fitting software is available to simplify the process.

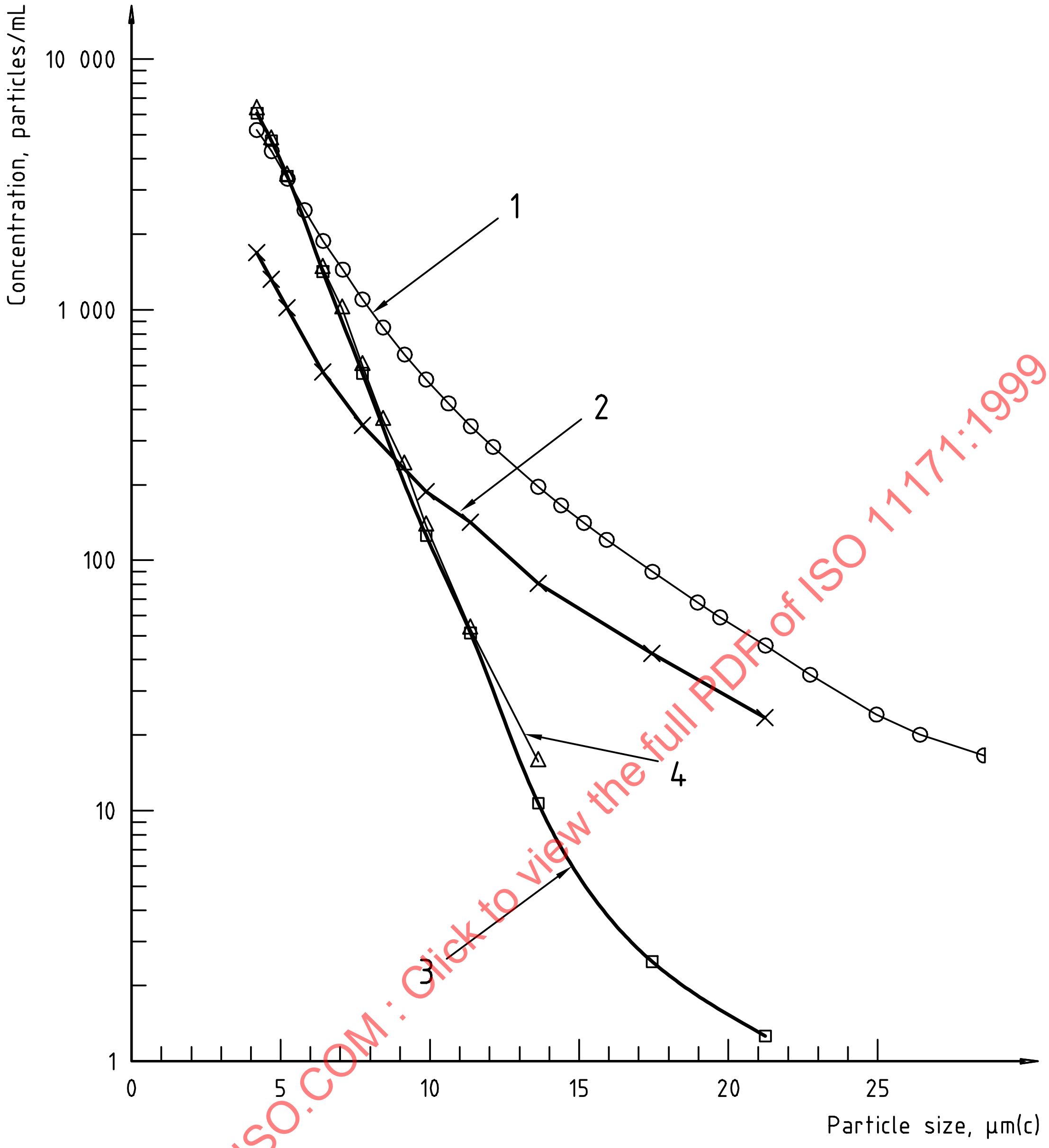
Due to the redefinition of particle size with this International Standard (see Table G.2), most particle-counting systems utilizing light-extinction sensors and purchased prior to 1993 will be unable to count particles smaller than about 4 µm(c). Three light-extinction models purchased after 1993 were able to count particles as small as 2,5 µm(c) to 3,0 µm(c). On the other hand, particle-counting systems with light-scattering sensors were able to count smaller particles. Some of these were able to count particles smaller than 1,0 µm in size using this International Standard.

The results show that particle-sizing variability among laboratories is a function of the particle concentration and the particle counter used. The results are summarized in Tables G.2 to G.4. Figure G.5 shows the relationship between sizing variability, defined here as the ratio of d_U (or d_L) to d, and particle concentration. The variability is typically better than ±10 % at concentrations exceeding 10 particles/mL. Under these conditions, at least 500 particles were actually counted. At lower concentrations, variability increased. This is an artifact of the statistics of small populations. One way to decrease variability, especially at larger sizes, is to increase the sample volume used for calibration.

Sizing variability is also a function of the instrument being used. As shown in Figure G.6, sizing variability for sample A improves by approximately 5 % when results from a single class of instruments (manufacturer B and sensor L, P, Q or S) are considered. Since this type was the most common used in the round robin, it was chosen for illustrative purposes only. The results demonstrate that instrument design can have a significant influence on variability. The purpose of the sensor performance verification (annex E) part of this International Standard is to minimize the effects of radical instrument design changes that may produce excessive variability between instrument types.

It is uncertain which instrument factors contribute to the variability. There was no significant difference between light-extinction and light-scattering sensors. Past work (Verdegan, 1989; and Verdegan, Schwandt, Holm, 1993) suggests that resolution may be an important source of variability, particularly for dusts with steep particle size distributions, such as sample A and downstream multipass samples. Referring to Table G.3, it is noteworthy that three labs (1, 22 and 30) contribute to much of the variability. There is reason to suspect that two of these have poor resolution characteristics. Lab 30 reported a resolution of 32 %, which is the poorest resolution reported. Lab 1 was unable to determine resolution, but experience suggests that this model of instrument has poor resolution characteristics. Instrument factors other than resolution, including the signal-to-noise ratio, nature of the light source, geometry of the optics, and type of batch sampler also influence variability.

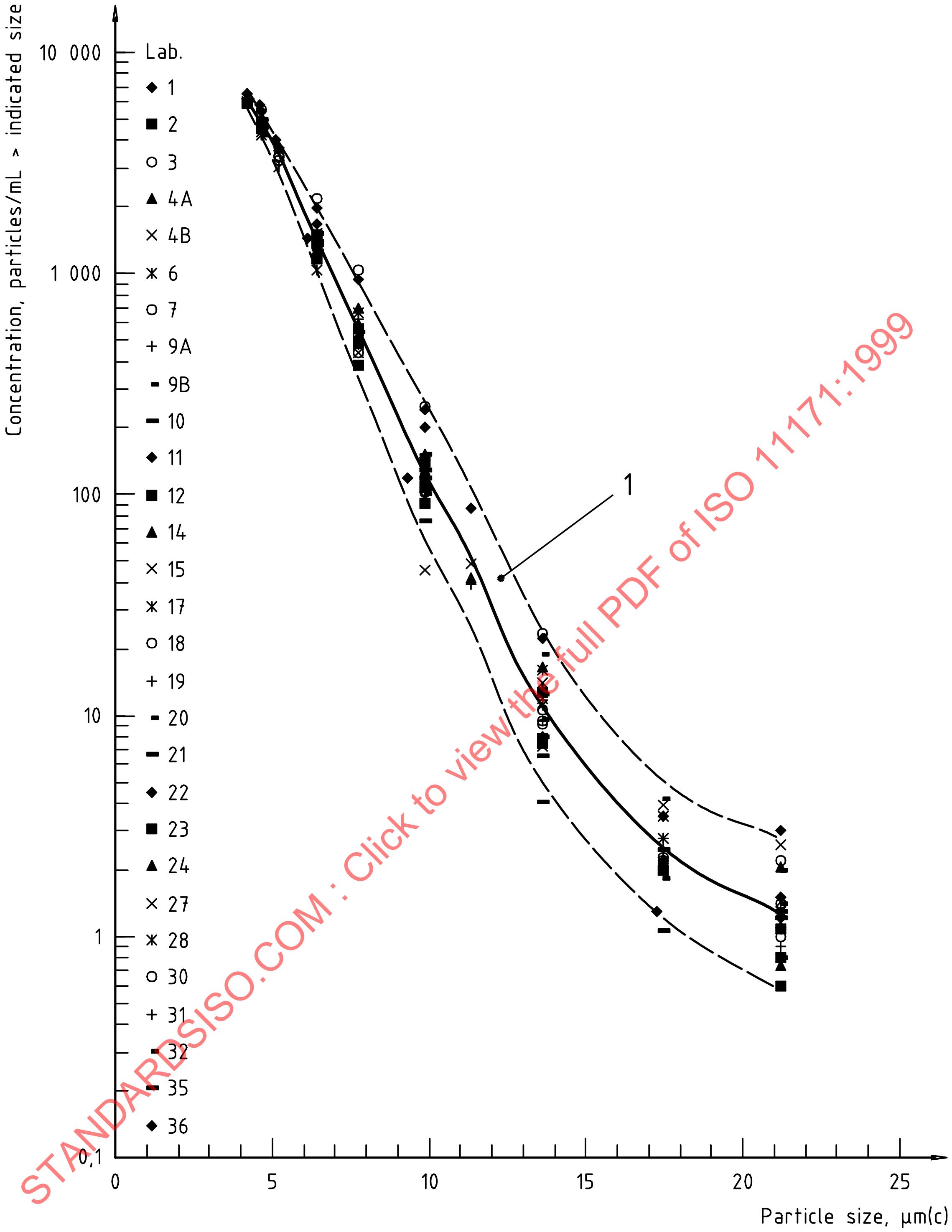
As shown in Figure G.7, sizing variability is not a function of particle size with this International Standard, while variability increases with decreasing size using ISO 4402:1991, the ACFTD calibration method. At large sizes, the variability is increased, regardless of the calibration method used, due to particle statistics, as previously discussed. For sizes smaller than 5 µm, variability increases with decreasing particle size when ISO 4402:1991 is used due to an artifact of errors in the ACFTD size distribution. As a result of limitations inherent in the optical microscopy techniques available at the time that the ACFTD size distribution was determined, the numbers of particles smaller than 5 µm were under-represented. The current revision, however, uses calibration samples with a particle size distribution certified using modern scanning electron microscopy techniques and rigorous statistical analysis. A direct consequence of this is that particle-sizing variability does not increase with decreasing size when this International Standard is used. The ACFTD variability is due to small changes in observed concentration that are interpreted as relatively large, but non-existent, changes in size. This increases sizing variability at small sizes when ISO 4402:1991 is used. For example, a 5 % error in the observed number of counts using ISO 4402:1991 would produce a 10 % error in size measurement at 2 µm, but less than a 4 % error when this International Standard is used. These results suggest that this International Standard provides a more accurate sizing calibration than ISO 4402:1991.



Key

1 Calibration sample
2 Sample B
3 Sample A
4 Verification sample

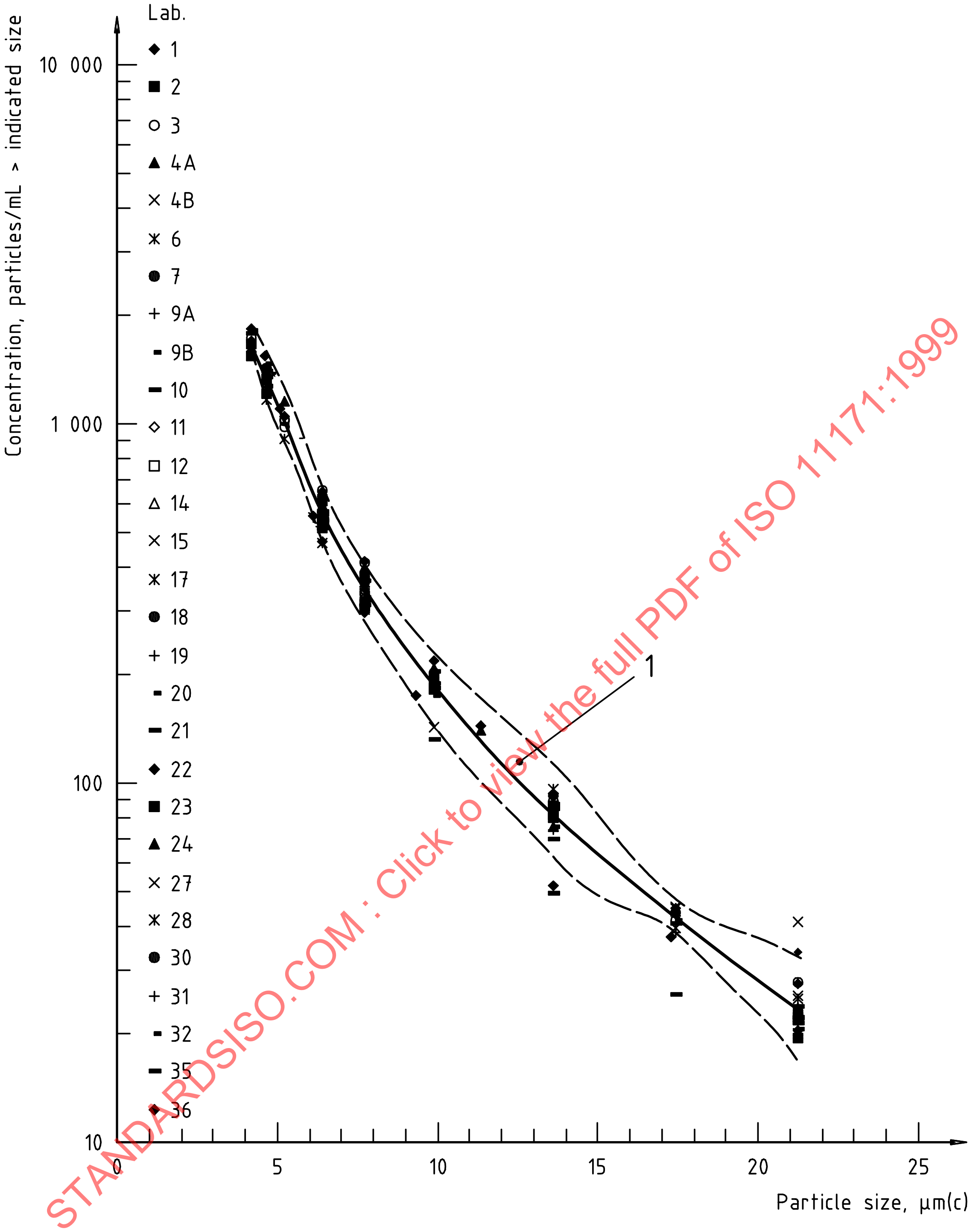
Figure G.1 — Particle size distribution of round-robin samples



Key

1 95 % confidence interval

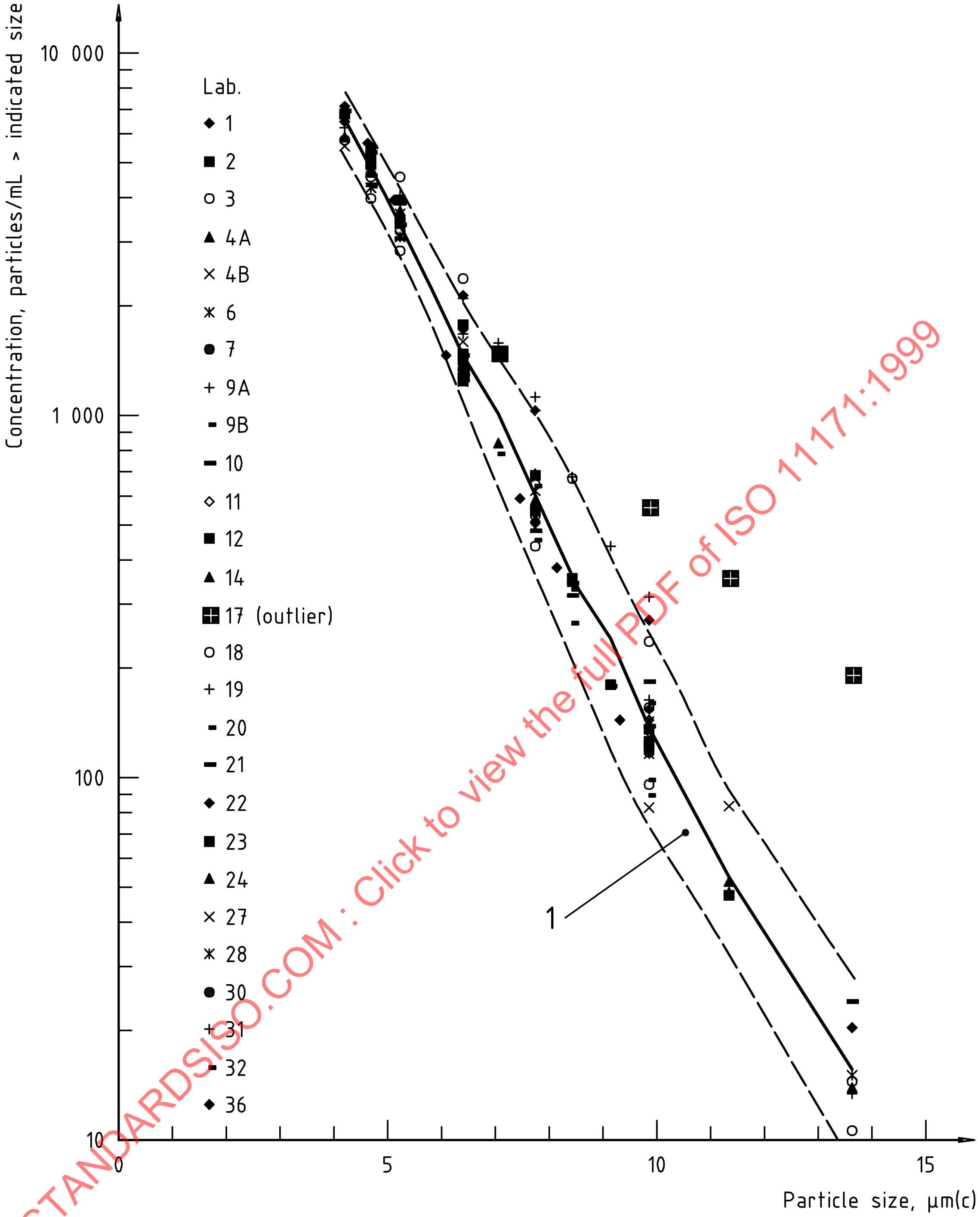
Figure G.2 — Sample A results



Key

1 95 % confidence interval

Figure G.3 — Sample B results



Key

1 95 % confidence interval

Figure G.4 — Verification sample results

Table G.1 — Equipment summary

Lab	Particle counter			Operating principle	Noise level	Coincidence error limit	Flow rate mL/min			R	Comments
	Manuf	Model	Sensor		mV	particles/mL	working	min.	max.	%	
1	B	c	N	E	2,50	9 200	50	46	60	NR	Resolution not done: unable to achieve $D < 3$ %, flow rate range overestimated
2	C	f	U	E	9,00	8 500	100	20	180	6,1	Underestimates lower flow rate limit
3	B	b	L	E	12,8	6 400	60	26	76	5,1	
4A	B	d	Q	E	12,90	9 323	24	9	33	13,5	
4B	B	d	Q	E	13,50	9 500	25	13	36	17,57	
6	B	b	L	E	13,20	21 064	20	15	35	P	Unable to measure left-side resolution due to instrument problem
7	B	b	L	E	16,50	16 500	40	36	45	10,8	
9A	B	b	Q	E	10,00	10 800	25	9	36	10,1	Flow rate range not calculated
9B	B	c	Q	E	12,50	10 800	25	8	36	11,73	Old latex used for resolution
10	B	b	Q	E	14,67	11 000	25	10	34	18,8	
11	C	f	V	s	5,00	>2 410	100	77	120	4,3	Coincidence error limit exceeds maximum concentration used
12	A	a	J	E	3,70	24 700	20	12	25	4,042	
14	F	l	K	E	31,00	3 755	50	47	59	8,23	Underestimates lower flow rate limit
15	B	b	L	E	13,60	8 000	60	48	60	P	Unable to determine left-side resolution, did not find upper flow rate limit
17	B	c	N	E	7,50	11 400	50	45	54	NR	Insufficient latex for resolution, outlier for verification sample
18	B	c	P	E	4,60	5 120	30	24	36	9,3	
19	B	e	M	E	8,40	3 200	100	94	117	9,534	Old latex used for resolution
20	B	e	R	E	11,50	4 000	20	20	20	NR	Latex concentration too low for resolution, flow rate limit not determined, coincidence error limit overestimated due to high noise level
21	D	i	W	E	25,00	28 500	25	23	28	NR	
22	C	g	V	S	4,00	6 700	50	44	60	11,8	Old latex used for resolution
23	B	b	Q	E	6,20	>27 000	25	11	32	16,8	Coincidence error limit exceeds maximum concentration made up
24	A	a	H	E	3,00	4 900	15	13	17	9,6	
27	D	j	X	E	130,00	8 000	50	40	65	NR	Resolution and calibration data not reported
28	D	i	Y	E	358,00	8 000	25	18	29	4,6	Old latex used for resolution
30	E	k	Z	E	150,00	22 500	25	10	40	31,97	Old latex used for resolution
31	C	f	T	S	2,00	7 000	25	17	29	6,96	
32	B	c	S	E	50,00	9 283	20	17	29	8,259	Old latex used for resolution, no antistatic additive in verification samples, mechanical shaker not used
35	C	h	V	S	7,00	5 000	50	30	60	<20	Insufficient sample to complete resolution, exceeded coincidence error limit for sample A
36	C	f	V	S	4,00	8 500	50	40	60	7,6	Old latex used for resolution but with MCA, interpolated values reported for samples so not included in statistical analysis
				E = Extinction		S = Scattering				P = Instrument problem; NR = Not reported	

Table G.2 — Correlation between particle sizes obtained using ACFTD (ISO 4402:1991) and NIST (ISO 11171) calibration methods

Particle size obtained using: ACFTD size (ISO 4402:1991) µm	NIST (ISO 11171) size µm(c)
1	4,2
2	4,6
3	5,1
4	5,8
5	6,4
6	7,1
7	7,7
8	8,4
9	9,1
10	9,8
11	10,6
12	11,3
13	12,1
14	12,9
15	13,6
16	14,4
17	15,2
18	15,9
19	16,7
20	17,5
21	18,2
22	19
23	19,7
24	20,5
25	21,2
26	22
27	22,7
28	23,5
29	24,2
30	24,9
31	25,7
32	26,4
33	27,1
34	27,9
35	28,5
36	29,2
37	29,9
38	30,5
39	31,1
40	31,7

NOTE This table is only a guideline. The exact relationship between ACFTD sizes and the NIST sizes may vary from instrument to instrument, depending on the characteristics of the particle counter and original ACFTD calibration.

Table G.3 — Sample A results

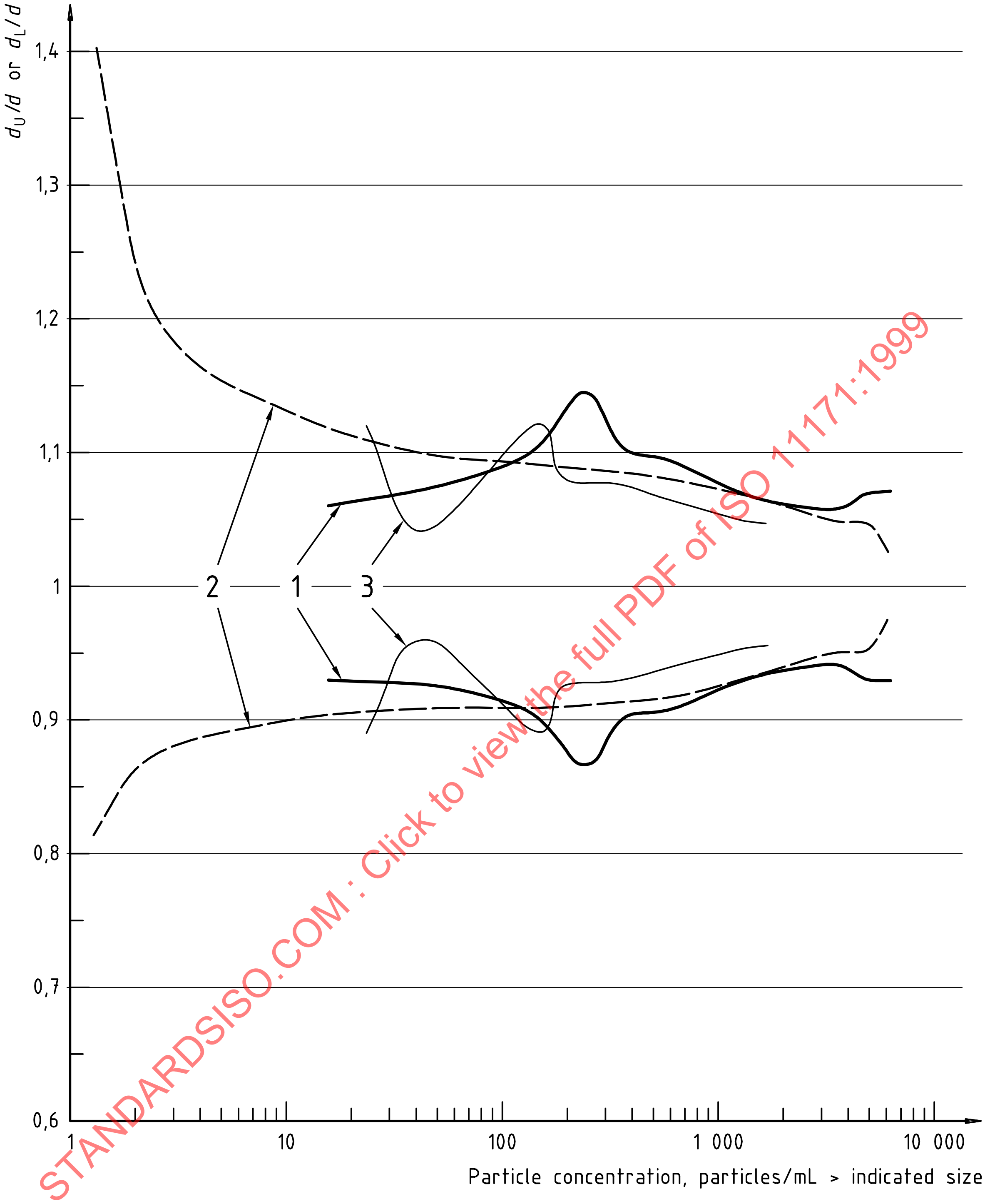
Lab	**Concentration,** particles per mL greater than indicated size									
	Particle size, µm(c)									
	4,2	**4,6**	**5,1**	**6,4**	**7,7**	**9,8**	**11,3**	**13,6**	**17,5**	**21,2**
1				1 317	569	202,00		8,00		3,00
2				1 163	386	91,76		7,91		1,09
3	5 903	4 614	3 262	1 307	442	101,50		9,50		1,40
4A	5 962,02	4 792,04		1 456,3	595,26	153,12		16,7		2,06
4B	5 983	4 586		1 518	564	113,00		12,00		0,80
6		4 199	3 029	1 420	477	97,05		7,57	2,17	1,15
7		4 852	3 392	1 406	524	115,65		10,55	2,27	1,08
9A		4 714		1 407		152,20		12,70	2,80	1,30
9B	6 076	4 548		1 225	548	153,40		18,80	4,20	1,40
10		4 582		1 348	482	119		12,6	2,47	1,22
11	6 556	5 441		1 674		146,10		12,94		1,21
12	5 925	4 610		1 487	562	107,40		7,50		0,60
14				1 273	523	118,70		13,20	2,26	0,74
15		4 365		1 293	530	124,66	48,26	14,14	3,96	2,62
17					663	141,70		11,10	2,80	1,30
18			3 374	1 481		121,70		9,20	2,20	1,00
19				1 569	618	151,10	39,10	9,50	2,50	1,20
20				1 350	484	105,55		8,00	1,84	0,80
21		5 011		1 399	496	130,00		6,60		1,30
22	6 527	5 004	3 736	1 974	941	243,00	86,80	22,20	3,50	1,50
23	5 917	4 542		1 399	485,16	139,38		12,70	2,00	0,80
24			3 749	1 474	701	124,76	42,20	7,76		
27		4 776		1 455	437	45,90		7,30		1,44
28				1 031	562	134,70		16,10	3,50	1,30
30		5 536		2 171	1 033	249,00		23,40		2,20
31	6 174	4 987		1 380		141,50		11,70		0,90
32		4 202		1 267		100,70		9,60		2,00
35	6 073	4 917		1 528		76,39		4,09	1,07	
36		5 500	4 800	1 300	700	102,00		50,00		0,20
Statistics, excluding interpolated values (lab 36)										
$\overline{X}_{\log}$	3,79	3,68	3,53	3,15	2,75	2,10	1,71	1,03	0,40	0,10
s	0,02	0,03	0,04	0,06	0,10	0,14	0,16	0,17	0,15	0,17
$\mathrm{COV}_{\log}$	0,45	0,87	1,01	2,02	3,64	6,84	9,18	16,35	37,44	168
n	10	19	6	27	22	28	4	28	15	26
95 % Confidence intervals for particle concentration, particles/mL										
$\overline{X}_{G}$	6 105	4 739	3 414	1 420	558	125,64	51,27	10,74	2,50	1,26
$\overline{X}_{U}$	6 601	5 489	4 021	1 905	884	243,31	105,67	23,35	4,98	2,75
$\overline{X}_{L}$	5 647	4 092	2 899	1 059	352	64,88	24,88	4,94	1,26	0,58
95 % Confidence intervals in particle sizing, µm(c)										
d	4,2	4,6	5,1	6,4	7,8	9,8	11,1	13,6	17,6	21,1
d_U	4,1	4,4	4,8	6,0	7,1	8,9	10,1	12,2	15,4	17,2
d_L	4,3	4,8	5,4	6,9	8,4	10,7	12,2	15,4	21,1	30,3
Statistics for similiar instruments: manufacturer B and sensor L,P,Q or S										
$\overline{X}_{\log}$	3,78	3,66	3,51	3,14	2,71	2,09	1,68	1,07	0,42	0,12
s	0,00	0,02	0,02	0,03	0,04	0,07		0,11	0,12	0,16
$\mathrm{COV}_{\log}$	0,13	0,57	0,64	0,91	1,51	3,45		10,56	28,91	135
n	5	11	4	12	9	12	1	12	8	12
95 % Confidence intervals for particle concentration data, particles/mL										
$\overline{X}_{G}$	5 968	4 540	3 261	1 374	514,36	122,71	48,26	11,80	2,66	1,31
$\overline{X}_{U}$	6 106	4 996	3 619	1 568	621,36	171,04		19,86	4,68	2,75
$\overline{X}_{L}$	5 833	4 127	2 938	1 204	426	88		7,00	1,51	0,63
95 % Confidence intervals in particle sizing, µm(c)										
d	4,2	4,7	5,2	6,4	7,7	9,9	11,4	13,6	17,5	21,2
d_U	4,1	4,5	5,0	6,2	7,5	9,4	3,8	12,8	15,6	17,2
d_L	4,2	4,9	5,4	6,6	7,9	10,4	3,8	14,6	20,3	28,2

Table G.4 — Sample B results

Lab	**Concentration,** particles per mL greater than indicated size									
	Particle size, µm(c)									
	4,2	**4,6**	**5,1**	**6,4**	**7,7**	**9,8**	**11,3**	**13,6**	**17,5**	**21,2**
1				471	299	187		52,00		34,00
2				514	307	183		86,40		22,00
3	1 730	1 356	1 019	566	337	196		81,30		22,20
4A	1 618	1 319		574	361	200		87,8		23,34
4B	1 636	1 281		611	373	194		89,80		25,60
6		1 162	910	561	331	188		81,81	39,64	20,02
7		1 295	976	545	335	192		89,04	43,59	23,26
9A		1 379		567		205		86,50	42,50	22,50
9B	1 822	1 467		561	366	204		87,10	41,60	22,20
10		1 227		546	322	183		84,47	40,68	21,54
11	1 703	1 435		634		190		80,82		20,46
12	1 675	1 321		613	375	197		83,60		22,40
14				594	370	208		86,06	44,12	23,72
15		1 254		529	333	194	140	89,22	45,22	22,82
17					355	195		85,90	44,80	25,00
18			1 025	567		185		80,20	41,50	22,40
19				615	353	193		74,40	38,20	19,90
20				552	330	189		85,50	43,60	22,40
21		1 351		538	314	184		70,00		24,00
22	1 848	1 398	1 051	647	413	220	145	93,70	45,00	27,70
23	1 542	1 212		537	309	182		79,80	38,30	19,50
24			1 151	585	399	195	140	75,62		
27		1 372		554	305	142		81,30		41,00
28				465	339	197		96,60	48,20	23,20
30		1 428		656	411	203		83,60		28,00
31	1 586	1 296		535		187		82,80		20,70
32		1 279		535		176		75,50		20,60
35	1 785	1 403		569		132		49,15		25,92
36		1 500		510	330	168		101,00		24,00
Statistics, excluding interpolated values (lab 36)										
$\overline{X}_{\log}$	3,23	3,12	3,01	2,75	2,54	2,27	2,15	1,91	1,63	1,37
s	0,03	0,03	0,04	0,04	0,04	0,04	0,01	0,06	0,02	0,07
$\mathrm{COV}_{\log}$	0,81	0,86	1,26	1,30	1,63	1,94	0,37	3,40	1,49	5,24
n	10	19	5	27	22	28	3	28	11	26
95 % Confidence intervals for particle concentration data, particles/mL										
$\overline{X}_G$	1 692	1 326	1 020	563	346	188	141	80,65	42,38	23,52
$\overline{X}_U$	1 909	1 501	1 214	663	418	231	147	108,66	47,38	32,75
$\overline{X}_L$	1 500	1 171	856	477	286	154	136	59,86	37,91	16,89
95 % Confidence intervals in particle sizing, µm(c)										
d	4,2	4,6	5,1	6,4	7,8	9,8	11,0	13,6	17,5	21,2
d_U	4,0	4,4	4,8	6,0	7,2	9,1	10,8	12,1	15,5	18,8
d_L	4,4	4,9	5,5	6,9	8,4	10,6	11,1	15,2	19,7	23,8

Table G.5 — Verification sample results

Lab	**Concentration,** particles per mL greater than indicated size										
	Particle size, µm(c)										
	4,2	**4,6**	**5,1**	**6,4**	**7,1**	**7,7**	**8,4**	**9,1**	**9,8**	**11,3**	**13,6**
1				1 292		510			144		
2		5 160	3 368	1 237		546		181	122		
3	5 754	4 516	3 209	1 296		436			95		7
4A	5 816	4 688	3 385	1 393		543			125		
4B	5 508	4 224	3 114	1 371		488			83		
6		4 245	3 089	1 478		518			116		15,05
7		4 861	3 390	1 412		531			117		10,64
9A		5 491	4 038	1 673					164		
9B	6 878	5 277	3 842	1 457		634			160		
10		4 329	3 063	1 290			319		139		24,1
11	6 469	5 343		1 715					155		20,4
12	6 781	5 347	3 921	1 780		681			125		
14				1 342	836	583	347		120	48,1	
15											
17					1 492				559	356,3	193,7
18		4 528				670		237		43,9	
19				2 098	1 576	1 124	678	436	315		
20				1 249	780	453	267	179	89		
21		4 596	3 363	1 336		478	342		183		
22	7 082	5 508		2 146		1 028			272		
23		4 885	3 442	1 480			356		136	47,3	
24			3 668	1 417		691			124	51,4	14
27		4 807	3 590	1 591		616			129	83,6	
28				1 249		601			143		15,6
30		3 948	2 827	1 393		640			156		
31	6 192	5 032	3 566	1 424					137		13,4
32		4 254		1 296			329		98		
35											
36		5 300	2 730	1 250		510	322	180			
Statistics, excluding outlier (lab 17) and interpolated values (lab 36)											
$\overline{X}_{\log}$	3,80	3,68	3,53	3,16	3,00	2,78	2,56	2,38	2,14	1,73	1,19
s	0,04	0,04	0,04	0,07	0,17	0,11	0,13	0,18	0,13	0,11	0,13
$\mathrm{COV}_{\log}$	1,06	1,18	1,19	2,09	5,60	3,97	4,98	7,61	6,20	6,49	10,83
n	8	20	17	25	3	20	8	5	24	5	8
95 % Confidence intervals for particle concentration data, particles/mL											
$\overline{X}_{G}$	6 286	4 769	3 415	1 458	1 009	600	361	241	137	53,28	15,66
$\overline{X}_{U}$	7 570	5 827	4 146	1 977	2 190	997	649	555	251	89,26	28,42
$\overline{X}_{L}$	5 221	3 903	2 812	1 076	465	361	201	104	74	31,80	8,63
95 % Confidence intervals in particle sizing, µm(c)											
d	4,2	4,7	5,2	6,5	7,0	7,7	8,4	9,1	9,9	11,5	13,6
d_{U}	3,9	4,3	4,9	6,0	5,9	7,0	7,5	7,8	8,9	10,6	12,9
d_{L}	4,5	5,0	5,5	6,9	8,1	8,4	9,4	10,4	10,9	12,4	14,3



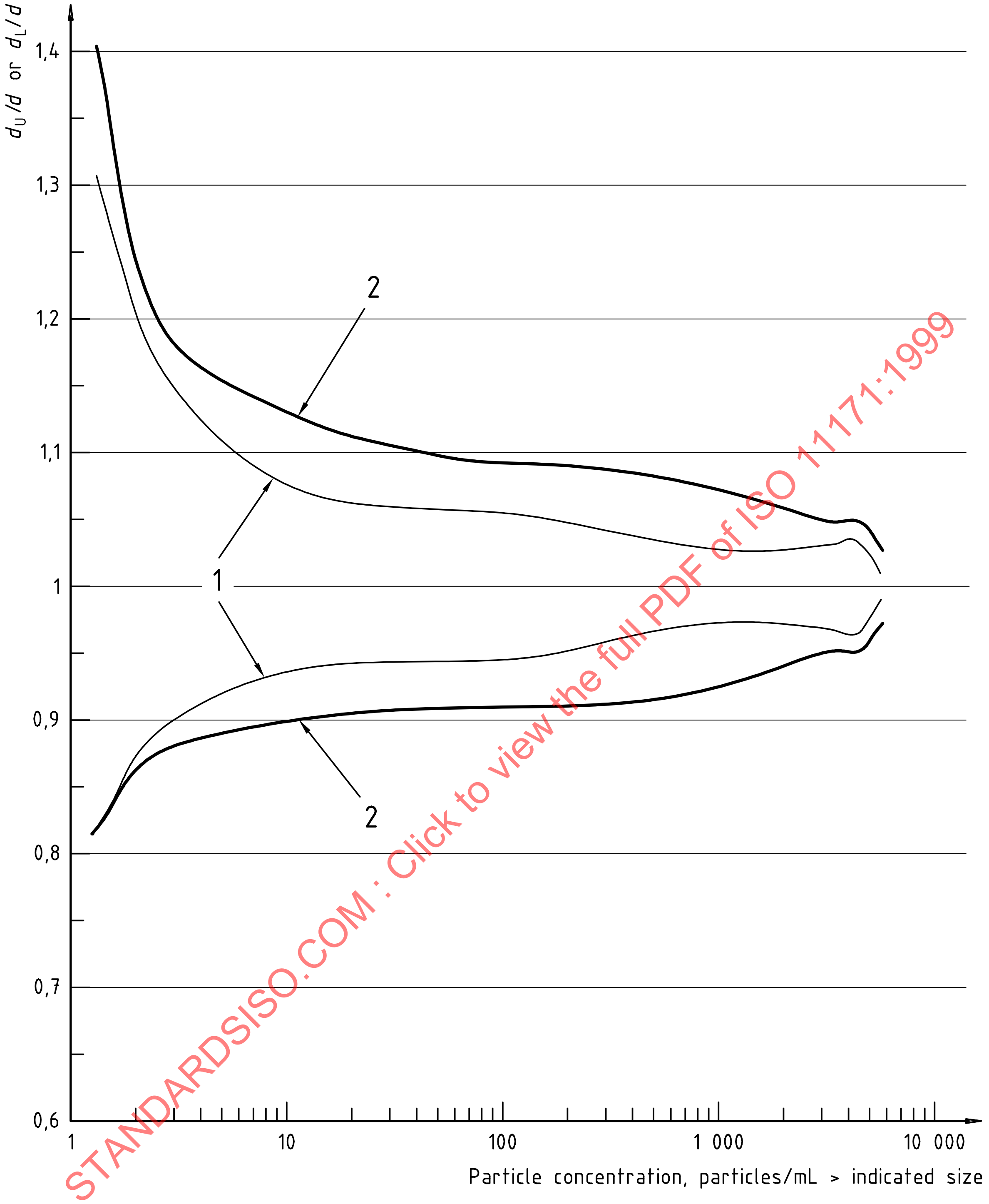
Key

1 Verification sample

2 Sample A

3 Sample B

Figure G.5 — Sizing variability as a function of concentration



Key

1 Sample A — same particle counter manufacturer

2 Sample B — all particle counter manufacturers

Figure G.6 — Sizing variability as a function of particle counter manufacturer