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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
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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.

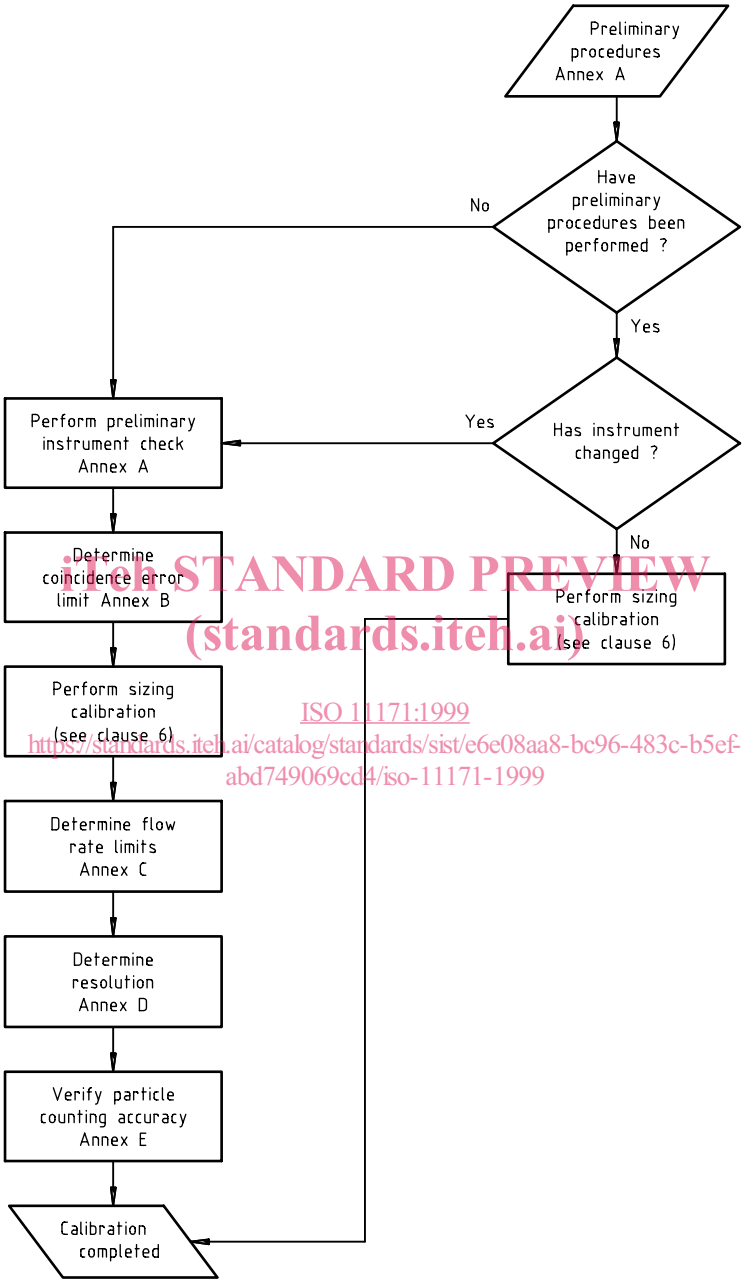

Figure 1 — Sequence of particle counter calibration procedures

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.

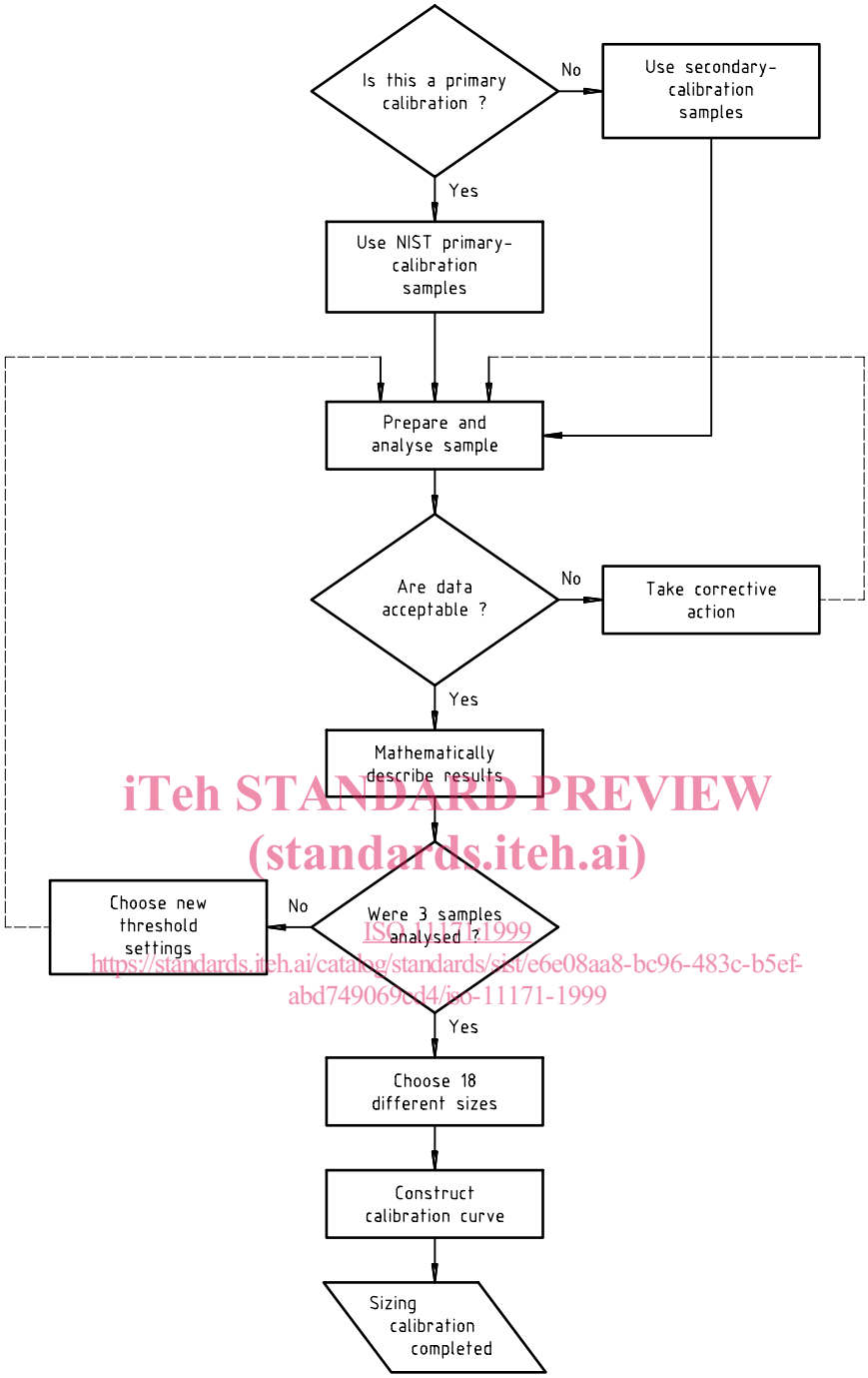

Figure 2 — Sizing calibration procedure

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:

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

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:

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

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.*"