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Hydraulic fluid power — Online automatic particle-counting systems for liquids — Methods of calibration and validation

Transmissions hydrauliques Systèmes de comptage automatique en ligne de particules en suspension dans les liquides — Méthode d'étalonnage et de validation

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee SC 6, *Contamination control*.

This second edition cancels and replaces the first edition (ISO 11943:1999) which has been technically revised.

This edition includes the following significant changes:

- the new SRM2806b has been taken into account for expression of μm sizes;
- there is no more intent to prepare and monitor the particle size distribution of secondary calibration suspension;
- the different validation relationships has been updated to be more severe and to make more confident the calibration of APCs;
- the round robin study is summarized in <u>Annex C</u>.

Introduction

In hydraulic fluid power systems, power is transmitted, and controlled, through a fluid under pressure within an enclosed circuit. The fluid is both a lubricant and a power-transmitting medium.

Reliable system performance requires control of the fluid medium. Qualitative and quantitative determination of particulate contaminant, in the fluid medium, requires precision in obtaining the sample and determining the size and distribution of contaminants.

Automatic Particle Counters (APC) are an accepted means for determining the size and size distribution of particulate contamination in fluids. Individual instrument accuracy is established through calibration performed with reference primary calibration suspensions or with secondary calibrations.

APCs are being utilized online to eliminate the need for sample containers, to provide increased accuracy, and to provide for a more rapid access to particle count information. A major application of online particle counting is for evaluating filtration efficiency of hydraulic filter elements during a multipass test as defined in ISO 16889. Depending upon the type of filter tested and the capabilities of the APC used, it might be necessary to dilute the samples before flowing through the sensor.

This document establishes procedures for validation of equipment for preparation of secondary calibration suspensions and for online counting of particles with of without dilution circuits, and the online calibration of APCs. It defines a procedure to match two or more particle counters that will improve the accuracy of particulate filtration efficiency as shown, for example in ISO 16889.

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Hydraulic fluid power — Online automatic particlecounting systems for liquids — Methods of calibration and validation

1 Scope

This document establishes methods to:

- validate equipment used to prepare secondary calibration suspensions for automatic particle counters;
- perform online secondary calibration of automatic particle counters;
- match two or more online particle counters, i.e. to count the same number of particles at a given size by two APCs associated on line;
- validate online particle counting systems with and without online dilution as used, for example, to measure the filtration efficiency of a hydraulic filter as described in the multipass filter test in ISO 16889.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4021, Hydraulic fluid power — Particulate contamination analysis — Extraction of fluid samples from lines of an operating system

ISO 5598, Fluid power systems and components — Vocabulary

ISO 11171:2016, Hydraulic fluid power — Calibration of automatic particle counters for liquids

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

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 5598 and ISO 11171 apply.

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

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

4 Units of measurements

The international system of units (SI) is used in accordance with ISO 80000-1.

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Throughout this document, the use of μm , [or $\mu m(b)$ or $\mu m(c)$] means that particle size measurements are carried out using an automatic particle counter that has been calibrated in accordance with either ISO 11171 or this document and particle size reported as defined in ISO 11171.

5 Test equipment

- **5.1 Liquid automatic particle counters,** requiring either calibration or verification, or a particle counter with two independent sensors.
- **5.2 A reference particle counter**, calibrated with a reference material in accordance with ISO 11171.
- **5.3 ISO Medium Test Dust (ISO MTD) concentrate,** in accordance with ISO 12103-1, category A3, dried at 110 °C to 150 °C for at least 1 h and for use in the test system, mixed in the test fluid, mechanically agitated, then dispersed ultrasonically with a power density of 3 000 W/m² to 10 000 W/m².

NOTE This standard test dust is used for filter test purposes in ISO 16889.

- **5.4 Test fluid,** as specified in ISO 16889.
- **5.5 Hydraulic equipment**, comprising:
- a) a reservoir, pump, liquid temperature control system and instrumentation which are capable of meeting the validation requirements of <u>Clause 8</u>;
- b) a clean-up filter capable of providing an initial fluid contamination level less than 50 particles/mL at the smallest particle size that will be validated or less than 2 % of the expected number of counts;
- c) a configuration which does not alter the contaminant distribution over the anticipated test duration (refer to ISO 16889);
- d) fluid sampling sections in accordance with \$50 4021;
- e) a configuration which will supply contaminated liquid to the particle counters under constant flow and temperature within the limits of <u>Table 1</u>.
- NOTE 1 A multipass test rig (see 150 16889) can be used provided it has been validated in accordance with Clause 8 of this procedure.
- NOTE 2 An alternative typical configuration which has proved to be satisfactory is given in Annex A.
- **5.6 Hydraulic circuit,** containing dilution equipment, if required, for online counter adaptation to a filter multipass test stand.

For typical hydraulic circuit configurations, which have proven to be satisfactory, refer to Annex B.

6 Accuracy of measuring equipment and test conditions

6.1 Utilize measuring equipment with an accuracy within the limits in <u>Table 1</u>.

Table 1 — Accuracy of measuring equipment and test conditions

Test conditions SI unit		Instrument accuracy (± of reading)	Allowed test condition variations	
Flow	L/min	0,5 %	2 %	
Kinematic viscosity	mm ² / _s	1 %	2 mm ² / _s	
Pressure	kPa	1 %	2 %	
Temperature	°C	0,5 °C	1 %	
Time	S	0,05 s	0 ,1 s	
Volume	L	1 %		
Mass	g	0,1 mg	2 %	

CAUTION — Maintaining the accuracy of test conditions, within the limits of <u>Table 1</u>, does not imply that by doing so the validation limits are satisfied. It has been proven that the most useful way in attaining the validation requirements, is by maintaining the accuracy of test conditions given in <u>Table 1</u>, along with using the proper particle counting procedures and correctly designed equipment.

7 Off-line APC calibration procedure

7.1 Conduct a sizing calibration on a particle counter when new or after major service as suggested by the particle counter manufacturer in accordance with ISO 11171.

NOTE The calibration is a primary calibration if the calibration suspension is NIST SRM 2806x where "x" is the SRM 2806 batch identification letter of the primary calibration samples. The APC then is called a 'Reference APC'.

7.2 Use the procedures specified in ISO 11171 to determine particle coincidence error limits of the particle counter and sensor, or use the manufacturer's stated levels, provided that it has been obtained in a similar manner.

8 Validation of online hydraulic equipment

- **8.1** This procedure of validation demonstrates whether:
- Particle size distribution of the suspension circulating within the equipment is stable within stated limits over time.
- The sampling or bottle filling ports gives representative samples. The complete and following procedure is given in <u>Figure 1</u>.
- **8.2** Connect a single particle counter with a valid calibration as defined in <u>Clause 7</u> and set it to the cumulative mode with at least six different threshold settings over the particle size range of interest. Sizes outside of this range cannot be reported as being in accordance with this document.

NOTE Since this procedure only aims at verifying the stability of particle counts over time, the use of a reference APC with primary calibration is not necessary.

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- **8.3** Adjust the total fluid volume, expressed in L, in the suspension preparation equipment to the maximum volume it is designed to prepare and measure it within ± 1 %. Maintain fluid viscosity at $(15 \pm 1,0)$ mm²/s.
- **8.4** Circulate the fluid at a flow rate through the clean-up filter until the fluid contamination level is < 5 particles > 5 μ m per mL.
- **8.5** Determine the mass of ISO MTD to be introduced in the system to achieve a concentration of $3 \text{ mg/L} (\pm 0.3)$. Record the ISO MTD lot number.

NOTE Any other concentration can be used provided it does not produce a particle count at the lowest size that is in excess of 75 % of the particle concentration limit of the instrument determined in 7.2.

- **8.6** Prepare the test dust concentrate in accordance with <u>5.3</u>. By-pass the clean-up filter element and add the required quantity of ISO MTD into the reservoir and allow it to circulate for approximately 15 min.
- **8.7** Start the test by conducting online automatic particle counts on samples with a minimum volume of 10 mL at least at 2-min intervals for 1 h, or, at least 30 intervals spaced evenly throughout the longest period of time that the system is to be used.
- **8.8** Complete Table 2 by filling in the required data. For each particle-size setting, calculate the mean \bar{x} , also the standard deviation " σ " of all the counts using the following formula:

$$\sigma = \sqrt{\frac{n\sum_{i=1}^{n}(x_i^2) - (\sum_{i=1}^{n}x_i)^2}{n(n-1)}}$$
(1)

where

- x_i is the particles per mL for each threshold setting for sample i;
- *n* is the total number of particle counts recorded.
- **8.9** Calculate the acceptable standard deviation for each particle size by using the following formula:

$$\sigma Acceptable = \sqrt{x} + 0,0004x$$
 (2)

NOTE This acceptable standard deviation is based upon the average standard deviation obtained in the round robin study discussed in $\underline{\text{Annex C}}$.

- **8.10** Accept the validation if the standard deviation for each particle size is less than or equal to the acceptable standard deviation for that size.
- **8.11** If the standard deviation for a given particle size exceeds the acceptable standard deviation, then re-evaluate the hydraulic equipment and procedures, the flow rates through the APC sensor and dilution system, and particle count volumes for the online particle counting equipment. Take appropriate action and repeat the procedure from <u>8.3</u> to <u>8.10</u>. If these actions do not improve the standard deviation to an acceptable level, then the APC sensor may require a service.

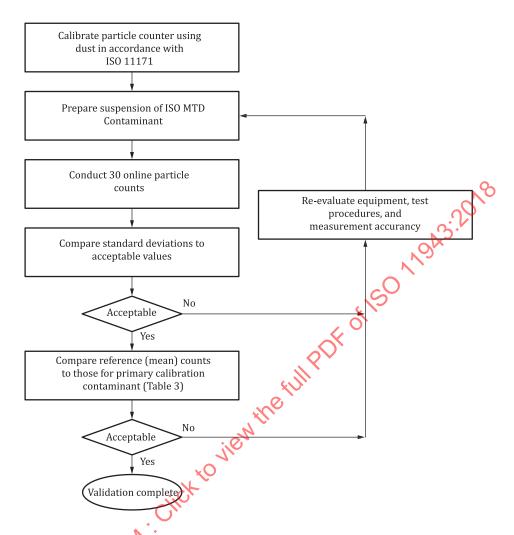


Figure 1 — Flowchart for the validation procedure of online hydraulic equipment

$Table\ 2-Secondary\ calibration\ dust\ data\ sheet$

Laboratory:							
Operator:	Date:		IS(O MTD Lot nu	mber:		
Concentration mg/L:	Particle coun	t volume:	mL Partio	cle counter n	nodel numbe	er:	
Particle counter serial nu	mber:	Senso	r model:				
Sensor serial number:		ISO 13	1171 primar	y calibration	n date:		
Size			er of partic	les per mL >	> stated size	e	
μm							
Count 1							
Count 2							10
Count 3						.?)
Count 4						ν. σ.	
Count 5						1/2	
Count 6							
Count 7					S		
Count 8							
Count 9							
Count 10				. ?			
Count 11				11/13			
Count 12				0			
Count 13			X				
Count 14			No:				
Count 15			7/1				
Count 16			XO.				
Count 17		Cilo					
Count 18		.0					
Count 19		W.					
Count 20	\sim)					
Count 21	~0.						
Count 22							
Count 23	~O ⁵						
Count 24	<u> </u>						
Count 25							
Count 26							
Count 27							
Count 28							
Count 29							
Count 30							
Mean							
σ							
Acceptable σ							

9 Online secondary calibration of automatic particle counter

- **9.1** At best the online secondary calibration of an APC has to be performed without dilution because any errors are magnified by the dilution ratio. Connect the reference APC onto the hydraulic equipment validated according to <u>Clause 8</u>, prepare the test dust according to <u>8.3</u>, <u>8.4</u>, <u>8.5</u> and <u>8.6</u>. Perform at least three consecutive online counts of at least 25 mL sample volume (after the counts have been stabilized) at several particle sizes covering the range over which the counter is to be utilized and report data in column 2 of <u>Table 3</u>.
- **9.2** Calculate and record, in column 3 of <u>Table 3</u>, the acceptable calibration limits for each particle size using the following formula:

Calibration limit = 0,185 (instrument counts in column 2 of Table 3)0,85

NOTE These calibration limits are based upon results issued from the round obin study (see <u>Annex C</u>).

- **9.3** Connect the APC to be calibrated onto the same hydraulic equipment as below, give the same conditions of circulation according to <u>9.1</u> and obtain a minimum of three consecutive online particle counts on 25 mL sample volumes (after the counts have stabilized). Average the counts obtained at each size, divide by the volume analysed and record the values obtained in column 4 of <u>Table 3</u>.
- **9.4** Accept the current calibration if all particle counts obtained in <u>9.3</u> are equal to the counts given by the Reference APC (in case of secondary calibration of the APC) or the secondary calibrated APC (in case of working verification of the APC) given in column 2 of <u>Table 3</u> within the limits in column 3 of <u>Table 3</u>.

Table 3 — Online particle counts of either calibration or verification suspension

Column 1	Column 2	Column 3	Column 4
Particle size, μm	Reférence or secondary instrument counts (N/mL)	± Calibration limits	Particle counts of APC under either calibration or verification
>3			
>42			
, > 5			
>6			
>7			
>10			
>12			
>14			
>15			
>20			
>30			

9.5 If the particle counts obtained in $\underline{9.3}$ are outside the limits set, take corrective action and repeat the verification procedure described in $\underline{9.1}$ to $\underline{9.4}$.

Ensure that:

- a) the proper sensor flowrate is being used;
- b) the particle size threshold settings are correct;
- c) the fluid is completely degassed;
- d) the sample weights and volumes are correct;
- e) at the end there is some possibility to interpolate the thresholds settings between **verif**ied points but it should be avoided. Extrapolation is not allowed.

10 Matching of two or more particle counters

10.1 This procedure shall be used when two or more APC are to be used upstream and downstream of a filter to measure its filtration efficiency at various sizes.

NOTE The "master" system (sensor 1) in the matching process is the system with the lower signal to noise ratio. This allows the "slave" sensor (sensor 2) a wider matching range.

- **10.2** In itself this procedure is not suited to performing or verifying the calibration of one or the two instruments. For calibration refer to <u>Clause 7</u>, and for online calibration refer to <u>Clause 9</u>.
- **10.3** Connect the APC calibrated in accordance with <u>Clause 9</u> (online calibration) and another one to be matched to the sampling points described in 5 of a closed loop system validated in accordance with <u>Clause 8</u>.

The two instruments have to match together without using the dilution system.

- **10.4** Set the instruments to the cumulative mode and to at least six different threshold settings over the particle size range of interest.
- **10.5** Adjust total fluid volume, expressed in L in the hydraulic test equipment to the desired level and measure within ± 1 %. Maintain fluid viscosity at $(15 \pm 1,0)$ mm²/s. The concentration of particles at any size should be statistically significant and particle counts should be at any size >10.
- 10.6 Circulate the fluid through the clean-up filter until the fluid contamination level is <5 particles $^{>5}$ μm per mL.
- **10.7** Determine the ISO MTD concentration to be used for the matching so that the maximum particle count at the lowest particle size can be approximately 75 % of the APC concentration limits determined in 7.2. Prepare in accordance with 5.3. Record the ISO MTD batch/lot number.
- **10.8** By-pass the clean-up filter, add the contaminant to the reservoir and mix by circulation for about 15 min or until the counts have stabilized across size range.
- **10.9** Start the APC matching procedure by conducting online automatic counts at 1-min intervals for the period of, at least, 30 min.

10.10 Calculate the allowable variation between sensors or counters for each particle size based upon the following formula and report in <u>Table 5</u>.

Allowable variation between sensors = 4,925.6 + 0,013.2 (average counts of sensor 1)

NOTE The variation between counters has been validated as well as ISO MTD, as with ISO UFTD during the last round robin study (see $\underline{\text{Annex C}}$).

The maximum allowable particle count difference between counters shall be less than 10 % those given in Table 4, whatever the size lower than 30 μ m.

Table 4 — Allowed deviation between two online APC when mismatchin	able 4 — Allowed deviation between two onl	lline APC when mismatchin
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Particle size	% allowed deviation
μm	70 anowed deviation
>4	1,5
>5	1,5
>6	1,9
>8	3,2
>10	4,2
>12	4,5
>15	5,4
>20	7
>25	10,8
>30	15

10.11 Adjust the threshold settings of the second counter (sensor 2) such that the average counts per mL match the average counts for the Reference counter (sensor 1) within the allowable variation given in Table 4 for each particle size counted. Repeat for other counters requiring matching.

Table 5 — Data sheet for calibration verification

Laboratory:						
Operator: Date:	ISO MTD L	ot number:	_			
Concentration mg/L:	_ Particle coun	nt volume:	_mL			
Particle counter model nu				ial number:		
Sensor model:				er:		
ISO 11171 primary calibr	ation date:					
Size			er of particles	per mL > stat	ed size	.8
μm			-			00
Sensor 1:					719A	5.1
5611301 1.					, OA	
M 11 1 11					N'S	
Model and serial number:					60	
number.					100	
0						
Count 1				,(QV		
Count 2				WIII		
Count 3				`		
Average			111.			
Average			:014			
			7,			
Sensor 2:		V				
		click				
Model and serial		.0.				
number:		an.				
		Ö,				
	-0.					
Count 1	25					
Count 2	-0°					
Count 3	8-1					
Average						
Average						
6						
Other units:						

11 Validation of an online dilution and particle counting system

- **11.1** Perform a validation of the online dilution system, where used, at the same frequency as calibration verification. The procedure is described in <u>Figure 2</u>.
- **11.2** Use dilution fluid which has been filtered to a cleanliness level of <5 particles >5 μ m per mL, unless it can be established that a higher level does not add more than 1 % error to the resulting particle counts.
- **11.3** Validate first at the minimum dilution factor to be used.
- **11.4** Circulate the fluid through the clean-up filter until the fluid contamination level is <5 particles >5 μ m per mL.
- **11.5** Prepare an ISO MTD secondary calibration suspension in accordance with 8.3 to 8.6 but at a level equal to 75 ± 10 % of the concentration limits in 7.2, multiplied by the dilution factor selected. Record the batch number of ISO MTD.

EXAMPLE For a 2 x dilution factor (1 part diluent: 1 part suspension), use a sample concentration equal to 2 times 50 % of the counter concentration limit.

- **11.6** By-pass the clean-up filter, add the contaminant to the reservoir and mix by circulation for about 15 min or until the counts have stabilized across the size range, whichever occurs first.
- **11.7** Set the particle counter at a minimum of six particle size threshold settings covering the range of interest.
- **11.8** Using the dilution factor selected obtain a minimum of three 25 mL particle counts (after the counts have been stabilized) for each sensor and calculate the average count of the diluted sample for each particle size.
- **11.9** Calculate the average counts per mL for each particle size threshold setting dividing the average count by the fluid volume counted in mL. Record in <u>Table 6</u>. Use separate data sheets for both sensors 1 and 2 where used.
- **11.10** All particle counts obtained in <u>11.7</u> should be equal to the Reference counts, ± the calibration limits in column 3 of <u>Table 3</u>, for each particle size counted. In addition, when sensors 1 and 2 are being used the average counts from the two counters (sensors) shall agree within the allowable variation given in <u>10.10</u> for each particle size counted.
- **11.11** Repeat the procedures given in <u>11.4</u> to <u>11.10</u> at the maximum dilution factor to be used and at least two other intermediate dilution factors within the total dilution range of the system.

It is recommended that the most common dilution ratios that will be used for the equipment be checked as described in this clause.

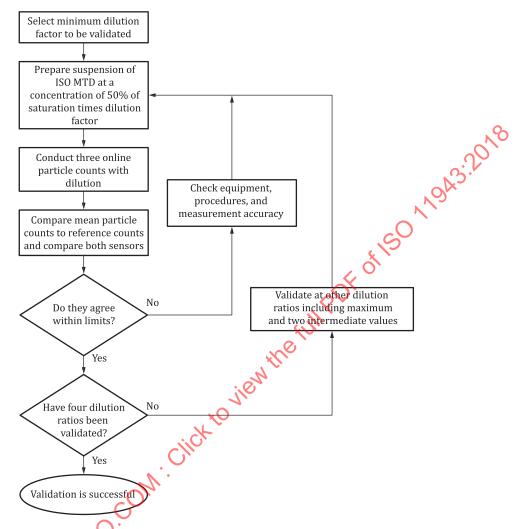


Figure 2 — Flowchart of the procedure for online dilution system validation

Table 6 — Data sheet for validation of online dilution equipment

Laboratory:	
Operator:	Date:
ISO MTD Lot number: Cond	centration mg/L: Particle count volume: mL
Particle counter model number:	Particle counter serial number:
Sensor model:	Sensor serial number:
Is this sensor to be located upstream o	or downstream of the test filter?
ISO 11171 primary calibration date:	
Size	Number of particles per mL > stated size
μm	(2).V
Concentration:mg/L	NOW.
Dilution factor:	
Count 1	5
Count 2	S. C.
Count 3	
Average	
Concentration:mg/L	
Dilution factor:	
Count 1	1,4/10
Count 2	
Count 3	
Average	
Concentration:mg/L	
Dilution factor:	$\cdot P^{r}$
Count 1	
Count 2	
Count 3	
Average	
Concentration:mg/L	
Dilution factor:	
Count 1	
Count 2	
Count 3	
Average	

12 Precautions

- **12.1** Online particle counting using ISO MTD for particle sizes >40 μ m requires extreme caution to ensure against particle settling. Calibration at the maximum particle sizes to be used shall be verified online (see <u>Clause 10</u>).
- **12.2** Testing coarse filters with online counters requires higher dilution factors which necessitates the use of higher-accuracy flow measurement.

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- **12.3** Dilution is required when particle counter concentration limits are exceeded. High concentrations of undetected particles can influence particle counts at measured sizes. Higher dilution factors shall be used under these conditions.
- **12.4** When online dilution is required, the dilution fluid shall be the same fluid type as the test fluid to be counted.
- **12.5** The presence of free water, or air, in the test fluid to be analysed has a detrimental effect on results. Precautions shall be taken to eliminate these variables.
- **12.6** Isolate the particle counter sensors from other large equipment to prevent electrical noise influences.
- **12.7** Flow pulsations due to pump ripple can cause erroneous particle counts. Damping can be provided by small accumulators if they are included in the system verification.
- **12.8** Minimize all line lengths and maximize flow rates to result in low lag times (the time from the sample being extracted from the multipass test stand, until sensed by the particle counter). A count lag time of less than 30 s should be obtainable. Lag times of sensors should be within 10 s of one another.
- **12.9** Valve adjustment shall be kept to a minimum during operation to reduce errors due to particle generation by the valve.

13 Identification statement

Online particle counting systems calibrated and verified in accordance with ISO 11943:2018, *Hydraulic fluid power* — *Online automatic particle-counting systems for liquids* — *Methods of calibration and validation.*

Annex A

(informative)

Typical online calibration and validation system design information guide

A.1 General

- **A.1.1** Online calibration and validation requires a validation procedure to determine the acceptability of the equipment to perform the desired function.
- **A.1.2** It is intended that Annex A provides basic guidance in constructing equipment that meets the validation requirements of this document.
- **A.1.3** The reader is cautioned that Annex A provides only guidelines for construction and does not guarantee successful validation of the equipment.

A.2 Online sample preparation equipment

A.2.1 General

The schematic of a typical set-up is shown in Figure A.1.

A.2.2 Lines

All lines should be sized for turbulent mixing flow and long straight runs should be avoided.

A.2.3 Fittings

Fittings should not have internally exposed threads or lips that can be contaminant traps.

A.2.4 Lines and fittings

Lines and fittings should be arranged to eliminate dead flow zones and where possible, vertical runs are preferable to horizontal.

A.2.5 Valves

Ball valves are preferable to other types of valves because ball valves are not contaminant traps and have a self-cleaning action. The valves should only be used in the full on or off positions and not for flow control.

A.2.6 Reservoir

The reservoir with a small volume, typically less than 10 L, should be constructed with a conical bottom displaying an included angle of not more than 90° with the entering test fluid diffused below the fluid surface.

NOTE Small reservoir volumes can cause a problem in accurately weighing the required quantity of test dust due to its low mass.

A.2.7 Clean-up filter

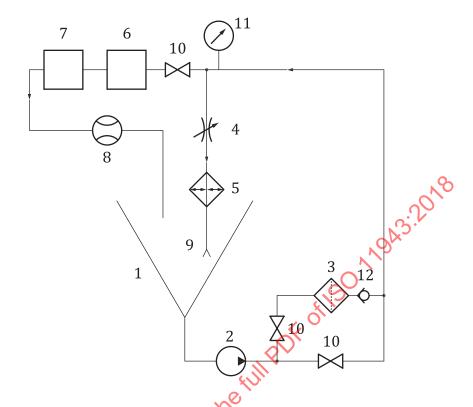
The system clean-up filter shall be capable of providing the required initial system contamination level. A filter with a filtration ratio of $\beta > 1\,000$ at the smallest particle size of interest is recommended.

A.2.8 Heat exchanger/heater

Depending upon the system design, cooling or heating of the system fluid may be necessary but take care as any heat exchanger may cause sedimentation. That is the reason why a conventional shell tubetype heat exchanger with oil flowing through the tubes, or a reservoir with a double jacket in which a temperature-controlled fluid is circulated, is recommended for cooling or heating the test fluid.

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Key

- conical reservoir (60° to 90° included angle) with precisely controlled volume
- 2 centrifugal pump
- 3 clean-up filter
- 4 back-up pressure valve
- 5 heater/heat exchanger
- 6 automatic particle counter sensor
- 7 automatic particle counter sensor
- 8 flow meter
- 9 flow diffuser
- 10 ball valve (is not to be used for flow regulation)
- 11 pressure gauge
- 12 check valve

Figure A.1 — Example of a system for online calibration and validation

NOTE Calibration stand can be made portable (on wheels) so it can be brought to the online automatic particle counters as required for calibration or verification.

 $CAUTION - Two \ sensors \ in \ series \ can \ cause \ problems \ due \ to \ cavitation, if \ the \ pressure \ drop \ in \ the \ first \ sensor \ is \ too \ high.$

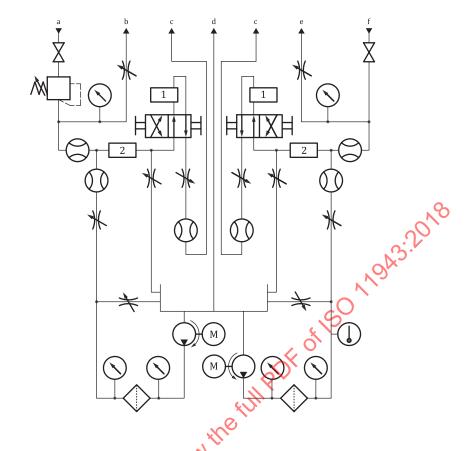
Annex B

(informative)

Hydraulic circuit design guide for online counter adaptation to a multipass test stand

B.1 General

- **B.1.1** Online particle counting (with or without dilution) during a multipass test requires adaptation to the multipass test stand and pre-test validation of the set-up to determine the acceptability of the equipment to perform the desired function.
- **B.1.2** It is intended that Annex B provides some basic guidance in constructing equipment that meets the validation requirements of this document.
- **B.1.3** The reader is cautioned that Annex B provides only guidelines for construction and does not guarantee successful validation of the equipment.
- **B.1.4** Schemes of three typical set-ups, which have been proven to be successful, are shown in Figures B.1, B.2 and B.3.
- **B.1.5** The system component design guide for the online sample preparation equipment, given in Annex A, should be followed for the online counting and dilution system.



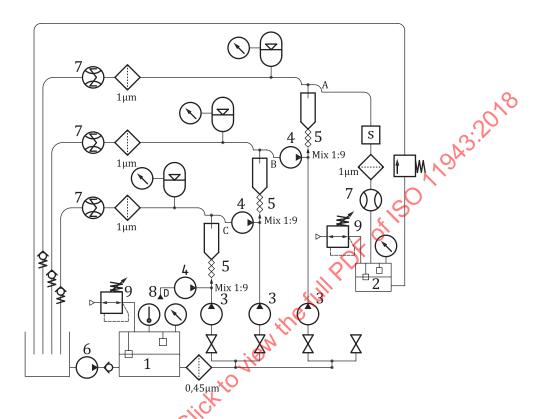
Key

- 1 sensor
- 2 mixer
- a Upstream sample.
- b Upstream return.
- c Sensor return.
- d Overflow.
- e Downstream return.
- f Downstream sample.

Figure B.1 — Example of the circuit number 1 for an online counter adaption to the multipass test stand

Serial dilution 1 000 × (as shown in Figure B.2) Suggested:

pumps 4 (20 to 50) mL/min – Sample pumps 3 (180 to 240) mL/min – Dilution



Key

- 1 automatic levelled chambers (pressurized)
- 2 automatic levelled chambers (pressurized)
- 3 tandem peristaltic dilution pumps
- 4 tandem peristaltic sample pumps
- 5 static mixers and expanded chamber

- 6 transfer pump (autmatic demand)
- 7 totalizing micro-oval flow meters
- 8 from multipass
- 9 regulated air
- S sensor

Figure B.2 — Example of the hydraulic circuit number 2 for online counter adaptation to multipass test stand

Systems, as shown, are required for upstream and downstream sampling. Pick-off point A can be moved to point B or C to give $100 \times or 10 \times dilution$. Pressure points A, B, C and D to be the same.

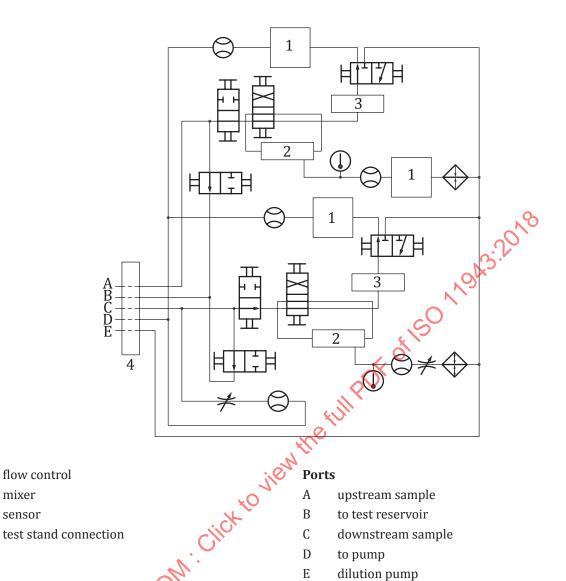


Figure B.3 — Example of the hydraulic circuit number 3 for online counter adaptation to multipass test stand

Key

1

2

3

4

mixer

Annex C

(informative)

Summary of ISO TC 131/SC 6 round robin study for online calibration and validation

Annex C outlines the round robin study conducted during 2015, relative to this document.

The purpose of the round robin was to measure the intra-company repeatability and inter-company reproducibility of particle count data obtained using automatic particle counters calibrated with ISO 11943 with traceability to ISO 11171:2010 and the new batch of NIST SRM2806 calibration fluid (referred to as SRM2806b).

Procedure:

- 1) Validate the calibration test rig according to <u>Clause 8</u> at particle sizes of 4, 5, 6, 7, 8, 10, 12, 15 and $20 \mu m(b)$;
- 2) Calibrate APC n°1 online as <u>Clause 9</u> with a reference APC which had been calibrated according to ISO 11171 using SRM2806b [expression of particle size in μm(b)],
- 3) Match APC n°1 and APC n°2 according to Clause 10;
- 4) Validate the online dilution and particle counting system according to Clause 11;
- 5) Validate the impact of the online calibration with particle counts at sizes smaller than 10 μ m(b) using ISO UFTD.

Nine (9) laboratories participated in the round robin; 5 from USA, 2 from Germany, 1 from France and 1 from China.

Results issued from the round robin test:

Relative to Item 1:

The standard deviation measured, based upon 30 counts for each 9 laboratories, is far lower than the previous value given in ISO 11943:1999 (see Figure C.1).

It has been proposed to divide by 2 the previous standard deviation according to the following formula:

$$\sigma = \sqrt{\overline{x} + 0.0004 \cdot x^2} \tag{C.1}$$

With \bar{x} being the average value of 30 counts/mL, this formula will be applied in 8.9.