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STANDARD

**ISO**  
**4402**

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**Hydraulic fluid power — Calibration of  
automatic-count instruments for particles  
suspended in liquids — Method using classified  
AC Fine Test Dust contaminant**

*Transmissions hydrauliques — Étalonnage des compteurs automatiques  
de particules en suspension dans les liquides — Méthode utilisant une  
fine poussière d'essai (ACFTD)*



Reference number  
ISO 4402:1991(E)

## 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.

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 4402 was prepared by Technical Committee ISO/TC 131, *Fluid power systems*, Sub-Committee SC 8, *Product testing and contamination control*.

This second edition cancels and replaces the first edition (ISO 4402:1977), table 3 of which has been technically revised.

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## **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 nature and extent of contamination.

Liquid automatic particle-counters are an accepted means for determining the nature and extent of contamination. Individual instrument accuracy is established through calibration.

# Hydraulic fluid power — Calibration of automatic-count instruments for particles suspended in liquids — Method using classified AC Fine Test Dust contaminant

## 1 Scope

This International Standard defines a procedure for the calibration of automatic-count instruments for particles suspended in liquids when used for the determination of particle size distribution of contaminants encountered in hydraulic fluid power applications.

It also establishes a uniform and realistic basis for particle sizing and counting.

**NOTE 1** It is assumed that users of this procedure are competent in the operation of their particular particle-counter and that proper sample-handling techniques will be used throughout the procedure. Sample-handling techniques which allow particle settling, erroneous suspension concentration, etc., will result in incorrect calibration.

## 2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3722:1976, *Hydraulic fluid power — Fluid sample containers — Qualifying and controlling cleaning methods*.

ISO 5598:1985, *Fluid power systems and components — Vocabulary*.

## 3 Definitions

For the purposes of this International Standard, the definitions given in ISO 5598:1985 apply.

## 4 Apparatus

The following materials and equipment shall be used.

**4.1 AC Fine Test Dust contaminant (ACFTD)<sup>1)</sup>**, processed and counter verified.

**4.2 Filter membrane**, which is compatible with the suspension fluid (4.3) and has a pore size of less than 1  $\mu\text{m}$  diameter.

**4.3 Suspension fluid**, compatible with the particle-counter and prefiltered through the membrane (4.2).

**4.4 Graduated cylinder**.

**4.5 Analytical balance**, accurate to within  $\pm 0,1$  mg.

1) ACFTD may be obtained from:

AC Spark Plug, General Motors Corporation, 1300 North Dort Highway, Flint, MI 48556, USA

or

AC Spark Plug, Sales Department, Milton Keynes, Bucks., UK.

**4.6 Sample bottles**, with screw caps and, if necessary, thin plastic film, all cleaned to a cleanliness level of no more than five particles greater than 5 µm per ml of bottle volume, in accordance with ISO 3722.

**4.7 Sample agitating device**, that will not alter the basic distribution of the calibration dust during agitation.

**4.8 Automatic-count instrument**, for particles suspended in liquids..

## 5 Concentration saturation procedure

**5.1** Carry out the following procedure in a basically still atmosphere to ensure there is no significant extraneous contamination of the sample.

**5.2** Place an accurately ( $\pm 0,1$  mg) weighed amount (for example 100 mg) of dry ACFTD into a sample bottle (4.6) and add an accurately ( $\pm 1\%$ ) measured volume (for example 1 litre) of filtered suspension fluid (4.3). Replace the clean plastic film (if used) between the container opening and cap.

**5.3** Disperse and suspend the dust in this concentrated standard mixture (for example 100 mg/l) by violent agitation (for example, 5 min with a paint shaker, 30 s in an ultrasonic bath and 15 min with a paint shaker).

**5.4** Prepare at least six suspensions with different concentration levels (for example 3 mg/l, 6 mg/l, 9 mg/l, 12 mg/l, 15 mg/l, 18 mg/l) by using the standard suspension from 5.3 as follows.

- Place the correct amount of the concentrated standard suspension into a clean sample bottle.
- Add the correct amount of filtered suspension fluid to the sample bottle.
- Replace the clean plastic film (if used) over the bottle opening.
- Cap the bottle and shake to obtain a uniform particle suspension.

**5.5** Adjust the automatic particle-counter to its most sensitive noise-free level and set any other channels arbitrarily at progressively higher levels. Use only the counts from the most sensitive channel for the saturation procedure.

**5.6** Maintain a convenient flow rate within the limits suggested by the manufacturer of the particle-counter. Use this flow rate for all counting unless the instrument is recalibrated for a new flow rate.

**5.7** Obtain five consecutive cumulative particle counts from the most sensitive channel for each concentration.

**5.8** Record the counts (see 5.7) in a table such as table 1 for each sample.

NOTE 2 Column 1 in the tables should represent the sample having the lowest concentration.

**5.9** Determine the average of the five counts and record on line 7 of table 1.

**5.10** Divide the average (see 5.9) by the corresponding concentration,  $\rho$ , in milligrams per litre, and enter on line 8 of table 1.

**5.11** Calculate the moving averages ( $\bar{Z}$ ) for the ( $\bar{Y}$ ) values recorded on line 8 of table 1 using the following equation:

$$\bar{Z}_n = \frac{\bar{Y}_1 + \bar{Y}_2 + \dots + \bar{Y}_n}{n}$$

where

$\bar{Z}_n$  is the moving average (see table 1, line 9);

$\bar{Y}$  is the average count per milligram per litre =  $\bar{X}/\rho$ ;

$\bar{X}$  is the arithmetic average of the cumulative particle counts (see table 1, line 7);

$n$  is the number of samples.

**5.12** Calculate the errors by subtracting the value of  $\bar{Z}$  from  $\bar{Y}$  for each sample and record these values on line 10 of table 1.

**5.13** Calculate the ranges by subtracting the lowest particle count value for a sample from the highest particle count value for the same sample and record these values on line 11 of table 1.

**5.14** Compute the average of all ranges by dividing the sum of the range values by the number of samples.

**5.15** Record this average range on line 12 of the last sample column of table 1.

**5.16** Using the  $D$ -factor directly below the recorded average range (see table 1), calculate the upper range limit and the settling tolerance utilizing the expressions specified below table 1. An examination of the error values (line 10) in the order of increasing sample concentration will yield a point where all samples of a higher concentration exhibit an error value greater than the settling tolerance. This point

represents the saturation limit of the counter for the corresponding channel setting.

**5.17** If the range on line 11 of any count below the saturation limit exceeds the upper range limit, re-count that concentration and evaluate the table with the new counts.

**5.18** If more than one sample exceeds the upper range limit or if any sample error below the saturation level exceeds the setting tolerance, review the counting technique.

## 6 Particle-size calibration procedure

**6.1** Prepare a calibration suspension by diluting a portion of the standard suspension (5.3) with filtered suspension fluid (4.3) to obtain a single concentration which is below the saturation level (see 5.16) of the counter.

**6.2** Fill a minimum of three sample bottles with the calibration suspension.

**Table 1 — Example of particle count worksheet**

Line No.	Sample No.	1	2	3	4	5	6	7	8	9	10		
1	Concentration, $\rho$ , mg/l												
2	Count 1												
3	Count 2												
4	Count 3												
5	Count 4												
6	Count 5												
7	Average, $\bar{X}$												
8	$\bar{Y} = \bar{X}/\rho$ <sup>1)</sup>												
9	Moving average, $\bar{Z}$												
10	Error 8 - 9												
11	Range <sup>2)</sup>												
12	Average range <sup>3)</sup>	→											
(factor for $n > 10 = 2,12$ )							D-factor →		1,88	1,89	1,90	1,91	1,92
							Upper range limit = average range × D-factor		→				
							Setting tolerance = ± average range × 0,55		→				
<p>1) Average count per milligram per litre.                  2) Highest count minus lowest count obtained.                  3) Average of all ranges recorded in line 11.</p>													

**6.3** Adjust the channel setting(s) of the instrument to some arbitrary value(s), for example, the manufacturer's suggested setting(s) within the size range of interest.

**6.4** Obtain five consecutive particle counts at the setting(s) specified in 6.3 from each of the bottles specified in 6.2.

**6.5** Repeat the procedure given in 6.3 until a minimum of four different channel settings of the instrument have been evaluated over the desired particle-size counting range.

**6.6** Calculate the average of the five particle counts from each bottle.

**6.7** Calculate the average counts of all the bottles for each channel setting.

**6.8** Calculate the average particle count per millilitre per milligram per litre concentration for each channel setting ( $\bar{A}$ ) by dividing the average count for all the bottles specified in 6.6 by the product of the gravimetric content of the calibration suspension (in milligrams per litre) and the sample volume, in millilitres.

**6.9** Plot on a log-log graph the averages ( $\bar{A}$ ), found as described in 6.8, versus the corresponding channel settings.

**6.10** Select the desired particle size to be counted by each channel of the automatic particle-counter. From the particle size distribution shown in table 2 for the calibration dust, obtain the number of particles per millilitre greater than the desired particle size selected for the channel.

**6.11** Select settings corresponding to the desired particle counts utilizing the plot of  $\bar{A}$  versus the channel settings (see 6.9).

**6.12** Adjust the channel(s) of the particle-counter to the settings selected in 6.11.

**6.13** Verify the settings at the desired particle sizes by performing particle counts on the calibration suspension obtained as described in 6.1.

**6.14** Make minor adjustments to the channel setting(s) if the average particle count value obtained from five consecutive counts of the calibration suspension differs from the correct value for the calibration dust.

**6.15** For final verification of the channel settings obtained as described in 6.13, prepare a minimum of six sample bottles with the calibration suspension (6.1).

**6.16** Using each of the channel settings obtained (6.13), establish five consecutive particle counts from each of the bottles (6.15). For a multichannel particle-counter, more than one channel setting may be verified simultaneously.

**6.17** Prepare a table similar to table 1 for each of the channel settings.

**6.18** Enter the five consecutive cumulative particle counts obtained as described in 6.16 for each sample in the proper locations in the table.

**6.19** Record the arithmetic average of the counts from each bottle on line 7 of each table.

**6.20** Calculate the ranges by subtracting the lowest particle count value for a sample from the highest particle count value for the same sample.

**6.21** Record the values (see 6.20) on line 11 of each table.

**6.22** Calculate the average of all ranges by dividing the sum of the range values by the number of samples.

**6.23** Record this average range on line 12 of the last sample column of each table.

**6.24** Using the appropriate *D*-factor given in table 1, calculate and record the upper range limit and the setting tolerance for each channel setting utilizing the expressions given in table 1.

**6.25** If the range for any sample at any of the channel settings exceeds the upper range limit for that setting, prepare another sample to be counted at that setting and evaluate the table with the new counts.

**6.26** If any of the average counts from 6.19 for a channel setting differs from the correct value for the ACFTD by more than the setting tolerance for that channel setting, adjust the channel setting and repeat the procedures given in 6.16 to 6.26 for the new channel setting.

Table 2 — Optical particle counts for 1 mg/l ACFTD

Diameter $\mu\text{m}$	Number of particles per millilitre of size > diameter	Diameter $\mu\text{m}$	Number of particles per millilitre of size > diameter
1	$1,8 \times 10^3$	51	1,2
2	$1,4 \times 10^3$	52	1,1
3	$0,99 \times 10^3$	53	1,0
4	$0,71 \times 10^3$	54	0,97
5	$0,52 \times 10^3$	55	0,90
6	$0,39 \times 10^3$	56	0,84
7	$0,29 \times 10^3$	57	0,79
8	$0,23 \times 10^3$	58	0,74
9	$0,18 \times 10^3$	59	0,69
10	$0,14 \times 10^3$	60	0,65
11	$0,12 \times 10^3$	61	0,61
12	95	62	0,57
13	79	63	0,54
14	66	64	0,50
15	55	65	0,47
16	47	66	0,45
17	40	67	0,42
18	34	68	0,40
19	29	69	0,37
20	25	70	0,35
21	22	71	0,33
22	19	72	0,32
23	17	73	0,30
24	15	74	0,28
25	13	75	0,27
26	12	76	0,25
27	10	77	0,24
28	9,3	78	0,23
29	8,4	79	0,22
30	7,5	80	0,21
31	6,8	81	0,20
32	6,1	82	0,19
33	5,5	83	0,18
34	5,0	84	0,17
35	4,5	85	0,16
36	4,1	86	0,15
37	3,8	87	0,14
38	3,4	88	0,14
39	3,1	89	0,13
40	2,9	90	0,13
41	2,6	91	0,12
42	2,4	92	0,11
43	2,2	93	0,11
44	2,1	94	0,10
45	1,9	95	0,10
46	1,7	96	0,10
47	1,6	97	0,09
48	1,5	98	0,09
49	1,4	99	0,08
50	1,3	100	0,08



**6.27** Determine the saturation limit of the counter for the lowest desired particle size setting by using the saturation procedure given in clause 5. Use the channel setting corresponding to the lowest desired particle size (5.5) instead of the lowest noise-free setting.

## **7 Precautions**

**7.1** If accurate counting is desired, do not exceed the saturation level of the instrument which is obtained as part of the calibration procedure.

**7.2** Any variation in flow rate or instrument adjustment from that used during calibration may result in erroneous particle counts.

**7.3** The presence of moisture or air in oil samples to be evaluated with an automatic particle-counter may have a detrimental effect on the results. Take precautions to exclude moisture from oil samples.

## **8 Identification statement (reference to this International Standard)**

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 4402, *Hydraulic fluid power — Calibration of automatic-count instruments for particles suspended in liquids — Method using classified AC Fine Test Dust contaminant.*"

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