INTERNATIONAL STANDARD ISO 4402

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Hydraulic fluid power — Calibration of liquid automatic particle-count instruments — Method using Air Cleaner Fine Test Dust contaminant

Transmissions hydrauliques — Étalonnage des compteurs automatiques de particules en suspension dans les liquides — Méthode utilisant une fine poussière d'essai ("Air Cleaner Fine Test Dust")

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FOREWORD

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4402 was developed by Technical Committee ISO/TC 131, *Fluid power systems and components*, and was circulated to the member bodies in September 1975.

It has been approved by the member bodies of the following countries :

Austria	India	South Africa, Rep. of
Belgium	Italy	Spain
Brazil	Japan	Sweden
Czechoslovakia	Mexico	Turkey
Egypt, Arab Rep. of	Netherlands	United Kingdom
Finland	Poland	U.S.A.
Germany	Romania	Yugoslavia

The member body of the following country expressed disapproval of the document on technical grounds :

France

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0 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.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a procedure for the calibration of automatic counting 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 — 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., can result in incorrect calibration.

2 REFERENCES

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

ISO 5598, *Fluid power — Vocabulary.*[1]

3 DEFINITIONS

For definitions of terms used, see ISO 5598.

4 MATERIALS AND EQUIPMENT

Use the materials and equipment specified in table 1.

TABLE 1 — **Materials and equipment and their requirements**

Item	Requirements
Calibration dust[1]	Processed, count-verified and available per note
Membrane filter disk	1,0 μm mean pore size or less
Suspension liquid	Compatible with the particle-counter and prefiltered through the membrane filter disk
Analytical balance	Accurate within ± 0,1 mg
Graduated cylinder	Accurate within ± 1 % of volume measured
Sample containers	Screw-cap bottles with plastic film cleaned to cleanliness level of no more than 3 particles greater than 10 μm per millilitre of bottle volume, in accordance with ISO 3722
Sample agitating device	One that will not alter the basic distribution of the calibration dust during agitation
Liquid automatic particle-counter	

1) To obtain standard Air Cleaner Fine Test Dust, contact :

Equipment Sales Department
AC Spark Plug Division
General Motors Corporation
1300 North Dort Highway
Flint, Michigan 48556, U.S.A.

1) In preparation.

5 CONCENTRATION SATURATION PROCEDURE

5.1 Place an accurately (± 0,1 mg) weighed amount (for example 100 mg) of dried Air Cleaner Fine Test Dust into a sample container as specified in table 1 and add an accurately (± 1 %) measured volume (for example 1 l) of filtered suspension fluid. Replace the clean plastic film between the container opening and cap.

5.2 *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).

5.3 Prepare at least six different concentration levels by using the standard suspension from 5.2 (for example 3, 6, 9, 12, 15, 18 mg/l) as follows :

5.3.1 Place the correct amount of the concentrated standard suspension into a clean sample container.

5.3.2 Add the correct amount of filtered suspension fluid to the sample container.

5.3.3 Replace the clean plastic film over the container opening.

5.3.4 Cap the container and shake to obtain a uniform particle suspension.

5.4 Adjust the automatic particle-counter to its most sensitive noise-free level and set any other channels arbitrarily at progressively higher levels. Only the counts from the most sensitive channel will be used for the saturation procedure.

5.5 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.

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

5.7 Record the counts (see 5.6) in table 2 for each sample (column 1 should represent the sample having the lowest concentration).

5.8 Determine the average of the five counts and record on line 7 of table 2.

5.9 Divide the average (see 5.8) by the corresponding concentration in milligrams per litre and enter on line 8 of table 2.

5.10 Calculate the moving averages ($\overline{Z}$) for the $\overline{Y}$ values recorded on line 8 of table 2 using the following equation :

$$\overline{Z}_n = \frac{\overline{Y}_1 + \overline{Y}_2 + \ldots + \overline{Y}_n}{n}$$

where

$\overline{Z}_n$ is the moving average (see table 2, line 9);

$\overline{Y}$ is the average count per milligram per litre $= \dfrac{\overline{X}}{\text{mg/l}}$

$\overline{X}$ being the arithmetic average of the cumulative particle counts (see table 2, line 7);

n is the number of samples.

5.11 Calculate the errors by subtracting the value of $\overline{Z}$ from $\overline{Y}$ for each sample and record these values on line 10.

5.12 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.

5.13 Compute the average of all ranges using the following expression :

$$\frac{\text{sum of range values}}{\text{number of samples}} = \text{average range}$$

5.14 Record this average range on line 12 of the last sample column.

5.15 Using the *D*-factor directly below the recorded average range (see table 2), calculate the upper range limit and the setting tolerance utilizing the expressions specified below table 2. 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 setting tolerance. This point represents the saturation limit of the counter for the corresponding channel setting.

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

5.17 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 with filtered suspension fluid to obtain a single concentration which is below the saturation level (see 5.15) of the counter.

TABLE 2 — **Particle count work sheet**

Line No.	Sample No.	1	2	3	4	5	6	7	8	9	10
1	concentration mg/l										
2	count 1										
3	count 2										
4	count 3										
5	count 4										
6	count 5										
7	average ($\bar{X}$)										
8	$\bar{Y} = \frac{\bar{X}}{\text{mg/l}}$ *										
9	$\bar{\bar{Z}}$ = moving average										
10	error 8 – 9										
11	range**										
12	average range***					→					
	(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			→	

* Average count per mg/l.

** Highest minus lowest count obtained.

*** Average of all ranges recorded in line 11.

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

6.3 Adjust the channel setting(s) of the instrument to some arbitrary value(s), for example 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 containers specified in 6.2.

6.5 Repeat 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 container.

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

6.8 Calculate the average particle count per millilitre per milligram per litre concentration for each channel setting using the average count for all the containers specified in 6.6, the gravimetric level (milligrams per litre) of the calibration suspension, and the following expression :

$$\bar{A} = \frac{\text{average count}}{(\text{sample volume, ml})\ (\text{suspension gravimetric, mg/l})}$$

6.9 Plot on a log-log graph the averages ($\bar{A}$) from 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 3 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 channel settings (see 6.9).

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

6.13 Check the settings at the desired particle sizes by performing particle counts on the calibration suspension obtained in 6.1.

6.14 Establish 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 in 6.13, prepare a minimum of six sample containers with the calibration suspension from 6.1.

6.16 Using each of the channel settings obtained in 6.13, establish five consecutive particle counts from each of the containers in 6.15. For a multi-channel particle-counter, more than one channel setting may be verified simultaneously.

6.17 Prepare a table similar to table 2 for each of the channel settings.

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

6.19 Record the arithmetic average of the counts from each container 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.

6.22 Calculate the average of all ranges using the following expression :

$$\frac{\text{sum of range values}}{\text{number of samples}} = \text{average range}$$

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 below table 2, calculate and record the upper range limit and the setting tolerance for each channel setting utilizing the expressions given below table 2.

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 the table evaluated 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 Air Cleaner Fine Test Dust by more than the setting tolerance for that channel setting, adjust the channel setting and repeat 6.16 through 6.26 for the new channel setting.

6.27 Determine the saturation limit of the counter for the lowest desired particle size setting by using the saturation procedure in clause 5. Use the channel setting corresponding to the lowest desired particle size in 5.4 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 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 liquid automatic particle-count instruments — Method using Air Cleaner Fine Test Dust contaminant.*"

TABLE 3 — **Optical particle counts for 1 mg/l Air Cleaner Fine Test Dust**

Diameter µm	No. part./ml > Diameter	Diameter µm	No. part./ml > Diameter
1,00	1 751,943	51,00	1,198 6
2,00	1 396,884	52,00	1,115 1
3,00	991,813	53,00	1,038 6
4,00	708,078	54,00	0,968 2
5,00	516,688	55,00	0,903 5
6,00	385,724	56,00	0,843 9
7,00	293,984	57,00	0,789 0
8,00	228,183	58,00	0,738 3
9,00	179,953	59,00	0,691 4
10,00	143,913	60,00	0,648 1
11,00	116,515	61,00	0,607 9
12,00	95,369	62,00	0,570 7
13,00	78,823	63,00	0,536 2
14,00	65,721	64,00	0,504 2
15,00	55,230	65,00	0,474 4
16,00	46,748	66,00	0,446 7
17,00	39,828	67,00	0,420 9
18,00	34,136	68,00	0,396 8
19,00	29,419	69,00	0,374 4
20,00	25,483	70,00	0,353 5
21,00	22,178	71,00	0,333 9
22,00	19,386	72,00	0,315 6
23,00	17,015	73,00	0,298 5
24,00	14,990	74,00	0,282 5
25,00	13,254	75,00	0,267 5
26,00	11,758	76,00	0,253 4
27,00	10,464	77,00	0,240 3
28,00	9,340	78,00	0,227 9
29,00	8,360	79,00	0,216 2
30,00	7,503	80,00	0,205 3
31,00	6,751	81,00	0,195 0
32,00	6,089	82,00	0,185 3
33,00	5,504	83,00	0,176 2
34,00	4,986	84,00	0,167 6
35,00	4,526	85,00	0,159 5
36,00	4,117	86,00	0,151 9
37,00	3,751	87,00	0,144 7
38,00	3,425	88,00	0,137 9
39,00	3,132	89,00	0,131 5
40,00	2,869	90,00	0,125 4
41,00	2,632	91,00	0,119 6
42,00	2,418	92,00	0,114 2
43,00	2,225	93,00	0,109 0
44,00	2,051	94,00	0,104 2
45,00	1,892	95,00	0,099 5
46,00	1,748	96,00	0,095 2
47,00	1,617	97,00	0,091 0
48,00	1,498	98,00	0,087 1
49,00	1,389	99,00	0,083 3
50,00	1,290	100,00	0,079 8