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Hydraulic fluid power — Determination of the particulate contamination level of a liquid sample by automatic particle counting using the light-extinction principle

Transmissions hydrauliques — Détermination du niveau de pollution particulaire d'un échantillon liquide par comptage automatique des particules par absorption de lumière

Reference number
ISO 11500:2008(E)

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Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

ISO 11500 was prepared by Technical Committee ISO/TC 131, *Fluid power systems*, Subcommittee SC 6, *Contamination control*.

This second edition cancels and replaces the first edition (ISO 11500:1997), which has been technically revised, specifically with the following major differences:

a) inclusion of a “quick check” method to determine the presence of water in the sample;

b) update of the method for calibrating the automatic particle counter (APC) from ISO 4402(1) to ISO 11171;

c) elimination of requirement to analyse samples in a class 100 000 clean room;

d) improved dilution methods;

e) improved guidelines on APC operation, including guidance for detecting and overcoming coincidence error;

f) revision of how to check the validity of the reported particle count.

It also incorporates the Technical Corrigendum ISO 11500:1997/Cor. 1:1998.

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1) ISO 4402, *Hydraulic fluid power — Calibration of automatic-count instruments for particles suspended in liquids — Method using AC Fine Test Dust contaminant*. Withdrawn and revised by ISO 11171.

Introduction

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

The presence of solid contaminant particles in the liquid interferes with the ability of the hydraulic fluid to lubricate and causes wear to the components. The extent of contamination in the fluid has a direct bearing on the performance and reliability of the system and it is necessary to control solid contaminant particles to levels that are considered appropriate for the system concerned.

A quantitative determination of particulate contamination requires precision in obtaining the sample and in determining the extent of contamination. The liquid automatic particle counter (APC), which works on the light-extinction principle, has become an accepted means of determining the extent of contamination. The accuracy of particle count data can be affected by the techniques used to obtain such data.

This International Standard details procedures for the analysis of contaminated liquid samples using an automatic particle counter. Correct use of an automatic particle counter helps to reduce errors and enhances the accuracy of reproducibility in data.

Hydraulic fluid power — Determination of the particulate contamination level of a liquid sample by automatic particle counting using the light-extinction principle

1 Scope

This International Standard specifies an automatic particle counting procedure for determining the number and sizes of particles present in hydraulic-fluid bottle samples of clear, homogeneous, single-phase liquids using an automatic particle counter (APC) that works on the light-extinction principle.

This International Standard is applicable to the monitoring of

a) the cleanliness level of fluids circulating in hydraulic systems,

b) the progress of a flushing operation,

c) the cleanliness level of support equipment and test rigs,

d) the cleanliness level of packaged stock.

NOTE 1 Measurements can be made with particles suspended in the original liquid or in a sample of the liquid diluted with a compatible liquid to reduce coincidence error.

NOTE 2 The presence of a fluid interface obstructs the light beam and gives false signals.

2 Normative references

The following referenced documents are indispensable for the application 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 3722, *Hydraulic fluid power — Fluid sample containers — Qualifying and controlling cleaning methods*

ISO 4406, *Hydraulic fluid power — Fluids — Method for coding the level of contamination by solid particles*

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

ISO 11171:1999, *Hydraulic fluid power — Calibration of automatic particle counters for liquids*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 5598 and ISO 11171:1999 and the following apply.

3.1
agglomerate
two or more particles that are in intimate contact and cannot be separated by gentle stirring and the small shear forces thus generated

3.2
coincidence error limit
highest concentration of ISO ultrafine test dust (ISO 12103-1, 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

[ISO 11171:1999, 3.4]

3.3
light extinction
reduction in intensity of a light beam passing through the sensing volume caused by the interaction of the light with single particles

NOTE This is also known as light blockage or light obscuration.

3.4
sensing volume
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

[ISO 11171:1999, 3.2]

3.5
threshold noise level
minimum voltage setting of the particle counter at which the observed pulse-counting frequency does not exceed 60 counts/min due to electrical noise

[ISO 11171:1999, 3.1]

4 Materials and equipment

4.1 Automatic particle counter (APC) for liquids, based on the light-extinction principle, consisting of an instrument to size and record the electrical signals generated when a single particle passes through a sensor for counting within the specified range.

The APC shall be capable of cumulative counting. The APC shall include an automatic bottle-sampling apparatus or a means of moving the sample liquid through the sensor and then into a calibrated volumetric measuring device without altering the particle size distribution of the sample.

4.2 Bottle sampler, used to transfer the liquid being analysed through a sensor, which may be an auxiliary component or a part of the APC itself.

If gas is used to force the liquid through the sensor, the gas shall be filtered through a 0,45 µm filter and shall be free from oil and water.

4.3 Density meter, with an accuracy of 0,001 g/cm^3, if the mass-dilution method is used.

4.4 Electronic balance, calibrated, with a resolution of 0,1 mg or better.

4.5 **Hot plate**, capable of heating to 150 °C ± 2 °C.

4.6 **Sample-agitating device**, such as a **laboratory bottle roller** or **paint shaker**, capable of re-dispersing the contaminant in the liquid sample without altering the basic particle size distribution of the contaminant.

4.7 **Sample bottles**, normally cylindrical bottles made of glass or high-density polyethylene, fitted with either a non-shedding, threaded cap forming a seal with the bottle without the use of an insert, or a cap with an internal seal.

The dimensions of the bottle depend upon the type of sample-bottle facility in use with the APC, but bottles should normally have a capacity of 250 mL. The bottle should be flat-bottomed and wide-necked to facilitate cleaning. The sample bottles shall be cleaned and verified in accordance with ISO 3722.

NOTE It has been found that a cleanliness level of fewer than 10 particles > 4 µm(c) and fewer than 2 particles > 6 µm(c) per millilitre of sample-bottle volume is adequate.

4.8 **Liquid dispensers**, fitted with a 0,45 µm membrane filter directly at the outlet.

4.9 **Temperature-measuring device**, calibrated, with an accuracy of ± 1 °C or better.

4.10 **Timer**, capable of measuring minutes and seconds, calibrated, with an accuracy of 0,1 s or better.

4.11 **Ultrasonic bath**, rated at a power intensity of 3 000 W/m^2 to 11 000 W/m^2 of the base area, which has been shown to be an acceptable means of both dispersing agglomerated particles within the liquid and removing air introduced by manual agitation.

4.12 **Volumetric glassware**, consisting of a range of calibrated graduated cylinders and graduated syringes or dosing pipettes (standard total displacement volumetric ware with multiple markings or air displacement) conforming to an appropriate standard and cleaned and verified in accordance with 6.2.

NOTE Examples of appropriate standards for volumetric glassware include ISO 4788 and ISO 8655 (all parts).

5 Diluent liquid

NOTE Observe standard laboratory safety practices and regulations when handling diluents.

5.1 The diluent liquid shall be cleaned to contain fewer than 15 particles > 4 µm(c) per millilitre.

5.2 The diluent liquid shall be physically and chemically compatible with both the sample liquid and the apparatus used.

NOTE See Annex B for examples.

5.3 Care shall be taken to ensure that the diluent does not affect the particle counts.

NOTE See Annex C for information about a method for pre-cleaning the diluent.

6 Pre-test requirements and procedures

6.1 Precautions

6.1.1 Chemicals

Good laboratory practices should be observed in the preparation and use of chemicals used in these procedures, as they can be harmful, toxic or flammable. Take care to ensure compatibility of the chemicals with the materials used. Refer to the material safety data sheet (MSDS) for each chemical and follow the precautions for safe handling and usage described therein.

6.1.2 Electrical interference

Precautions should be taken to ensure that the test area does not exceed the radio-frequency interference (RFI) and electro-magnetic interference (EMI) capabilities of the APC.

NOTE 1 An APC is typically a high-sensitivity device and can be affected by RFI or EMI.

The voltage supply to the instrument shall be stable and free of electrical noise.

NOTE 2 The use of a constant-voltage transformer is considered appropriate.

6.1.3 Use of magnetic stirrer

Do not use a magnetic stirrer for samples containing ferrous or other magnetic particles. If such a stirrer is fitted as standard equipment, it can be necessary to remove or negate the drive magnet.

6.1.4 Relative humidity

The relative humidity of the test area should be controlled within the range of 40 % RH to 70 % RH.

NOTE Relative humidity can affect the particle counts.

6.1.5 Sample storage

Store samples susceptible to bacterial growth under refrigerated conditions (at 5 °C ± 2 °C). Bring refrigerated samples to room temperature prior to evaluation and analyse within 1 h of reaching room temperature.

6.2 Glassware cleaning procedure

6.2.1 Clean all glassware using a validated cleaning procedure. Validate cleanliness in accordance with ISO 3722. The final solvent liquid used for the flush should be

a) filtered petroleum spirit or equivalent, if the samples being analysed are petroleum-based or synthetic liquids,

b) either propan-2-ol (2-propanol) or filtered demineralized water, if the samples being analysed are water-based liquids.

6.2.2 The required cleanliness level (RCL) of all glassware shall be such that contaminant thereon cannot significantly contribute to the overall result.

NOTE It has been found that a cleanliness level of fewer than 10 particles > 4 µm(c) and fewer than 2 particles > 6 µm(c) per millilitre of the glassware's volume is adequate.

6.2.3 Filter all liquids used for cleaning and rinsing through a 1 µm or finer membrane filter.

6.3 APC calibration procedure

Calibration of the APC shall be maintained in accordance with ISO 11171.

6.4 APC operation

6.4.1 Use the APC in accordance with its manufacturer's instructions. Make all measurements at particle concentrations that are below 80 % of the manufacturer's stated coincidence error limit (see 3.2) and at a particle size that is at least 1,5 times above the threshold noise level (see 3.5) of the instrument.

6.4.2 Ensure that the APC has been switched on for long enough to become stabilized.

6.4.3 Clean the sensor and associated pipework prior to use by flushing them with filtered solvent (see 6.2.1).

NOTE This cleaning can be achieved by filling a clean sample bottle with filtered solvent and flushing the solvent through the sensor and associated pipework at a flow rate that is approximately 50 % higher than the flow rate used during analysis.

Ensure that the sampling probe is dried before analysing a sample, otherwise errors can result from the creation of an optical interface between the liquids.

6.4.4 If the sensor has been used previously to analyse a liquid that is not miscible with the liquid being analysed, clean the sensor using the cleaning procedure in 7.4.

6.4.5 Inspect the sensing volume on a regular basis for the presence of particles either in the sensing volume itself or in the entry to the sensing volume.

6.4.6 Verify the cleanliness level of the particle-counting system, including the APC, diluent and glassware, by analysing a volume of filtered diluent that conforms to the requirements given in Clause 5.

6.5 Sample inspection and preparation before counting

6.5.1 Outline

See Figure 1 for a flowchart that illustrates the procedure for the preparation of a liquid sample for automatic particle counting.

6.5.2 Initial preparation and inspection

Remove any visible contamination from the exterior of the closed sample bottle using a lint-free cloth, and visually inspect the sample for

a) cloudiness (which can be an indication of excessive particles or free water),

b) macroscopic particles,

c) free water,

d) inappropriate containers (i.e. leaking or damaged containers or containers not conforming to 4.7),

e) excessive volume (i.e. the sample fills more than 80 % of the sample bottle volume).

A sample exhibiting the phenomena described in items a) through d) shall not be counted using the method specified in this International Standard, as these conditions are likely to affect the performance of the sensor. Record the results of visual inspection in the test report [see Clause 8, item p)].

If sample volume is excessive, proceed to 6.5.3.

If the sample does not exhibit items a) through d), proceed to 6.5.4.

While this International Standard does not require a microscopic examination of the sample, one may be conducted, if desired.

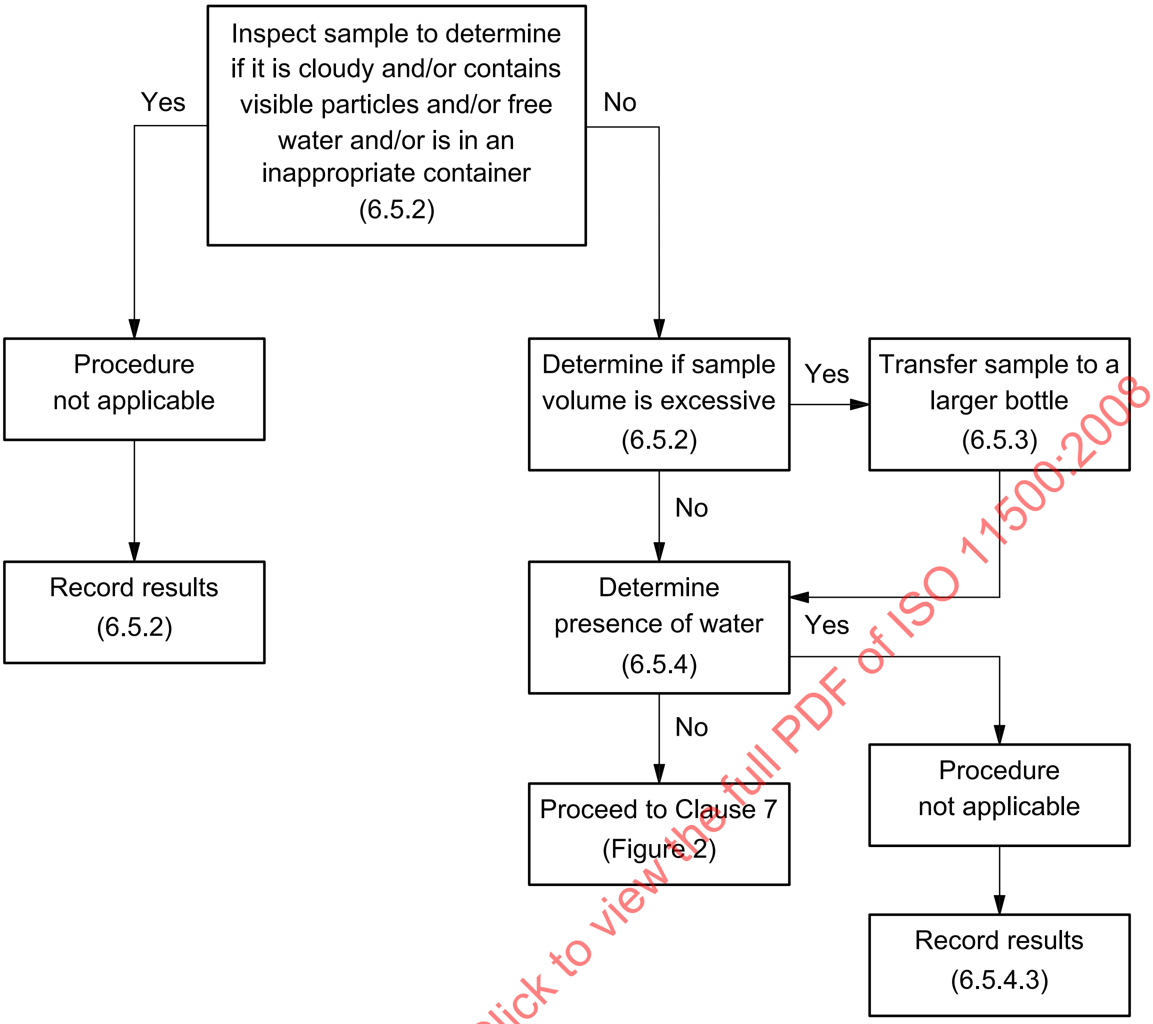


Figure 1 — Flowchart for inspection and preparation of a liquid sample before automatic particle counting

6.5.3 Preparation of sample of excessive volume

6.5.3.1 Estimate the fluid volume in the sample bottle. If the sample takes up less than 80 % of the bottle volume, it may be used. If it takes up more than 80 % of the bottle volume, reduce the volume, following the procedure given in 6.5.3.2 through 6.5.3.4.

NOTE It is difficult to achieve re-dispersion of particulate contaminant in sample bottles that are filled to more than 80 % of the bottle volume, which results in a non-homogenous suspension. Use of the procedure specified in 6.5.3.2 and 6.5.3.4 corrects this problem.

Use caution to ensure that contamination is not added by using the procedure in 6.5.3.2 and 6.5.3.4.

6.5.3.2 Estimate the volume of the sample and select a clean sample bottle (secondary bottle) such that it is about 50 % to 80 % full when the complete sample is transferred into it. This bottle should fit into the sample-agitating device and the bottle-sampling apparatus.

6.5.3.3 Transfer the sample into the secondary sample bottle as follows.

a) Pour approximately half of the sample into the secondary bottle.

b) Vigorously shake the remaining sample by hand.

c) Immediately pour the remaining sample into the secondary sample bottle.

Extreme care should be taken not to spill any of the sample. If any of the sample is spilled or lost during the transfer, the sample should not be counted.

6.5.3.4 Cap the secondary sample bottle.

6.5.4 Determination of presence of water

6.5.4.1 Determine that the amount of water in the sample is not too great to allow the use of the method in this International Standard by either of the following methods.

a) The hot plate method is as follows.

   1) Preheat the hot plate to 140 °C ± 2 °C.

   2) Agitate the sample in the sample-agitating device for 5 min.

   3) Place the sample in an ultrasonic bath and energize the bath for 30 s or until no surfacing air bubbles are observed.

   4) Place 1 mL to 2 mL of the sample on the hot plate and observe the reaction of the liquid. If the sample spits or bubbles, it contains water. If the sample spreads out in a thin film without spitting, it does not contain water.

b) Use another device for measuring relative water content, with the result that the water content shall not exceed 70 % of the saturation level of the hydraulic fluid.

NOTE ISO 760 and ISO 12937 are alternate methods of determining the presence of water.

6.5.4.2 If it is determined that the sample does not contain water in quantities that can affect the particle count provided by the APC, the sample may be used for particle counting in accordance with this International Standard.

6.5.4.3 If it is determined that the sample contains water in quantities that can affect the particle count, the sample shall not be evaluated by the procedure specified in this International Standard. Record the results.

6.6 Determination of need for sample dilution

It is preferable to analyse the sample in an undiluted form. However, dilution is often necessary to reduce the optical density of the sample, its viscosity or the particle concentration. Guidance on when dilution is necessary is given in 7.2.1.

7 Procedure for determining the particulate contamination level by automatic counting

7.1 Outline

Figure 2 shows a flowchart that illustrates the procedure for determining particulate contamination in liquid samples by automatic counting.

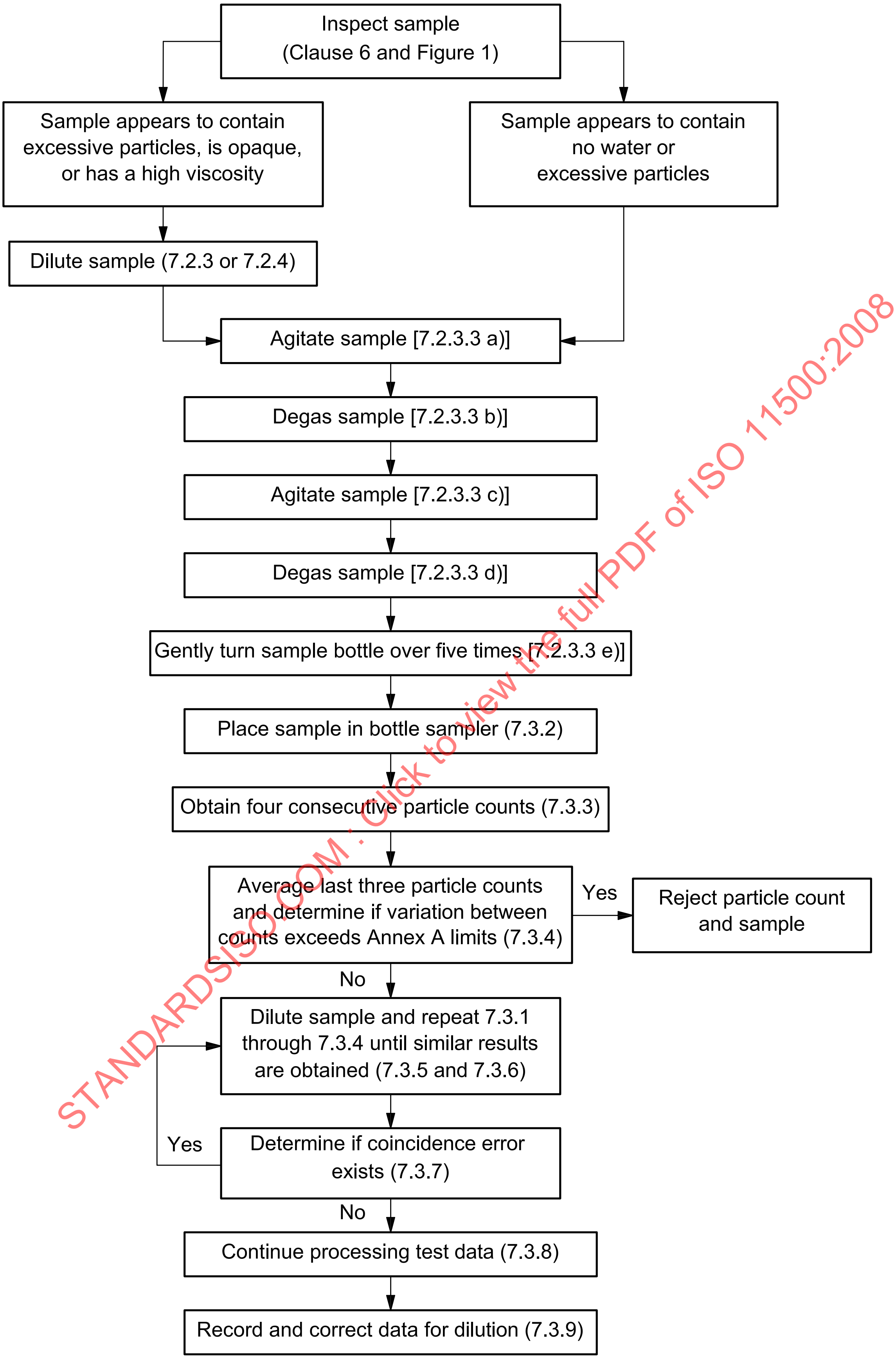


Figure 2 — Flowchart illustrating the procedure for determining particulate contamination in liquid samples by automatic counting

7.2 Sample dilution

7.2.1 Use of sample dilution

Sample dilution can be necessary to

a) reduce the viscosity of the sample commensurate with the design of the bottle sampler,

b) reduce the particle population below the coincidence error limit of the APC,

c) reduce the optical density of the sample; for example, with very dark liquids, the APC might not operate correctly,

d) perform an exploratory count to optimize the dilution ratio or to verify the validity of the particle count data.

Initial treatment of the sample is based on its opacity. If the sample looks clear (e.g. similar to the clarity of new MIL-H-5606) and a concentration of particles is not visible to the eye, the first particle count may be made without dilution. If the sample is opaque, it should be diluted with clean diluent in the ratio of 25 % sample to 75 % diluent before particle counting.

7.2.2 Precautions

The process of dilution can introduce errors in particle count data because of the addition of extraneous contamination, which is magnified by the dilution ratio. To reduce such errors, dilution should be performed under clean air conditions and all apparatus used in the procedure should be cleaned to the cleanliness level specified in 6.2.

NOTE Dilution with solvent can change the water saturation characteristics of the sample. When diluting with solvents with a water saturation (solubility of water in the solvent) of less than approximately 100 mg/L, care should be taken to ensure that the water saturation point of the diluted samples is not exceeded.

7.2.3 Volumetric dilution method

7.2.3.1 Use volumetric glassware that conforms to the requirements of 4.12.

7.2.3.2 Select the dilution ratio (for example, 10 % sample to 90 % diluent).

7.2.3.3 Prepare the sample as follows.

a) Vigorously shake the sample bottle for a minimum of 60 s.

b) Sonicate the sample by placing the sample bottle in an ultrasonic bath and energize the bath for 30 s.

c) Repeat 7.2.3.3 a).

d) Degas the sample either by

    1) placing the sample bottle in the vacuum chamber of the bottle sampler or auxiliary device and applying vacuum until no surfacing air bubbles are observed, or

    2) placing the sample bottle in an ultrasonic bath and energize the bath for 30 s or until no surfacing air bubbles are observed. When taking the sample bottle out of the ultrasonic bath, remove the water from the outside of the bottle using a lint-free wiper.

e) Remove the bottle from the equipment used to degas the sample, and gently turn the sample bottle over at least five times, taking care not to introduce air bubbles into the liquid. Proceed without delay to the next step.

NOTE A commercially available roller bed can be used to turn sample bottles over. Rolling the bottle continuously until it is analysed prevents sedimentation.

7.2.3.4 Measure the quantity of clean diluent necessary to achieve the dilution ratio selected in 7.2.3.2, record the volume as V_d, then transfer approximately 50 % of the diluent to a clean sample bottle (secondary sample bottle).

7.2.3.5 Measure the sample volume required to achieve the dilution ratio selected in 7.2.3.2.

7.2.3.6 Add the sample collected in 7.2.3.5 to the clean diluent in the secondary sample bottle.

7.2.3.7 Rinse the container used to measure the sample in 7.2.3.5 with remaining diluent, and add all to the secondary sample bottle. Measure and record the total volume as V_t.

7.2.3.8 Calculate the actual dilution ratio, D_R, using Equation (1):

$$D_R = \frac{V_t}{V_t - V_d} \tag{1}$$

where

V_t is the total volume, expressed in millilitres;

V_d is the volume of diluent, expressed in millilitres.

7.2.4 Mass dilution method

7.2.4.1 Determine and record the density of the sample and the diluent. Care should be taken so that the density meter does not add contamination to the sample.

7.2.4.2 Place an empty clean sample bottle (secondary sample bottle) on the balance and either tare the balance or record the mass of the clean sample bottle.

7.2.4.3 Follow steps 7.2.3.1 through 7.2.3.3 to prepare the sample for dilution.

7.2.4.4 Estimate the sample volume required. Using a pre-cleaned pipette (see 6.2), extract that volume from the original sample bottle immediately after agitation, ensuring that there is enough fluid left in the original sample bottle to determine the sample density, transfer the sample to the secondary sample bottle, and measure and record the mass of the sample in the secondary sample bottle as M_s.

7.2.4.5 Add clean diluent to the secondary sample bottle to obtain the desired dilution ratio, and record the total mass as M_t (that is, the mass of the sample, the diluent and the secondary sample bottle, if the balance had not been tared for the bottle's mass).

7.2.4.6 Calculate the actual dilution ratio, D_R, using Equation (2):

$$D_R = \frac{\dfrac{M_t - M_s}{\rho_d} + \dfrac{M_s}{\rho_s}}{\dfrac{M_s}{\rho_s}} \tag{2}$$

where

M_t is the total mass, expressed in grams;

M_s is the mass of the sample, expressed in grams;

ρ_d is the density of the diluent, expressed in grams per cubic centimetre;

ρ_s is the density of the sample, expressed in grams per cubic centimetre.

7.3 Analysis procedure

7.3.1 Prepare the sample as specified in 7.2.3.3, omitting steps b) and c) if the sample has been recently diluted in accordance with 7.2.3 or 7.2.4.

CAUTION — Elapsed time after final sonication until particle counting should be kept to a minimum; a maximum of 1 min is recommended.

7.3.2 Place the sample in the bottle sampler.

7.3.3 Obtain four consecutive particle counts at the required particle size ranges.

NOTE The following particle sizes are commonly used in reporting such data and also allow approximate comparison with data obtained using APCs calibrated in accordance with ISO 11171:1999 > 4 µm(c), 6 µm(c), 10 µm(c), 14 µm(c), 21 µm(c), 38 µm(c) and 70 µm(c).

Record the volume analysed and approximate flow rate through the sensor.

7.3.4 Discard the first count and average the next three counts. Reject the count and the sample if the variation between counts for all particle size ranges analysed exceeds the limits given in Annex A.

If the sample is rejected, consideration should be given to repeating the test using a sample volume that will ensure that a minimum of 20 particles of the largest particle size of interest can be counted.

7.3.5 If the sample was not diluted before the first set of particle counts performed in 7.3.3, dilute it with clean diluent to a ratio of 10 % sample to 90 % diluent. If the sample was diluted before the first set of particle counts performed in 7.3.3, dilute it at a higher dilution ratio than that selected in 7.2.3.8 or 7.2.4.6.

7.3.6 Repeat 7.3.1 through 7.3.4 until two sets of data showing similar results are obtained. Results shall be considered as similar if the variation between the particle count reported and the particle count obtained at the next higher dilution ratio is within the limits given in Annex A.

If the initial particle count was carried out without dilution, a dilution of 10 % sample and 90 % diluent is recommended. If the initial sample contained 90 % diluent, a dilution of 5 % sample and 95 % diluent is suggested.

7.3.7 Determine whether the particle count at the smallest particle size of interest exceeds the coincidence error limit of the APC. If coincidence error exists (e.g. normative particle counts obtained at the last two dilutions do not give the same results, or the APC indicates an over-concentration of particles), select a higher dilution ratio and repeat 7.3.1 through 7.3.4.

Coincidence causes an overcount of larger particles and an undercount of smaller particles. The coincidence error limit of the APC (not to be confused with the saturation limit) is the maximum acceptable concentration of all particles larger than the size of the smallest particle detectable by the instrument. All measurements should be taken at particle concentrations that are below 80 % of the APC manufacturer's stated coincidence limit. This concentration is normally given by the APC manufacturer with a note indicating the probability of coincidence. Coincidence is reduced by dilution (see 7.2).

7.3.8 If coincidence error does not exist, continue by processing the test data.

7.3.9 Record the particle counts obtained using the lowest acceptable dilution ratio. Correct the test data for the dilution ratio to provide the actual number of particles per millilitre in the sample.

7.4 Analysis of different liquids

Take care when the sensor is used to sequentially analyse non-compatible liquids.

NOTE It is possible that films or droplets of the previous liquid remain on the transparent windows of the sensor, which leads to inaccurate counting. The procedure for changing liquids involves flushing with a succession of solvents, each one compatible with the preceding one. For example, to change from water to oil, a typical sequence can be as follows.

a) Flush with distilled or de-ionized water.

b) Flush with propan-2-ol (2-propanol).

c) Flush with petroleum spirit (recommended boiling point between 100 °C and 120 °C) or a solvent with similar properties.

d) Dry carefully with dry, oil-free compressed air that has passed through a compressed-air filter that is 99,99 % efficient at 2 µm with a pressure rating that is suitable for the application.

e) Flush with liquid of the actual sample, if sufficient sample is available.

8 Test report

The following minimum information shall be recorded (the form given in Annex D may be used to record and report data):

a) laboratory identification;

b) date of analysis;

c) sample identification;

d) sample liquid;

e) APC manufacturer and model;

f) sensor model;

g) sensor flow rate;

h) sensor coincidence error limit;

i) method of APC calibration;

j) date of APC calibration;

k) dilution ratio;

l) diluent used;

m) volume counted per run;

n) particle count data with a minimum of three digits per millilitre at the particle sizes analysed;

o) if desired, contamination level code of the average of each particle count, expressed in accordance with ISO 4406;

p) any comments pertaining to the analysis.

9 Identification statement (reference to this International Standard)

It is strongly recommended to manufacturers who have chosen to conform to this International Standard that the following statement be used in test reports, catalogues and sales literature:

"Determination of particulate contamination level of a hydraulic liquid sample by automatic particle counting performed in accordance with ISO 11500:2008, *Hydraulic fluid power — Determination of the particulate contamination level of a liquid sample by automatic particle counting using the light-extinction principle*."

Annex A
(normative)

Procedure for performing a statistical check of an automatic particle counter

A.1 After obtaining four particle counts on a particular sample, discard the first particle count, average the next three particle counts and record as $\overline{X}$.

A.2 Calculate the percentage difference, ΔX, between the highest and lowest measured particle count, X_{max} and X_{min}, respectively, for each particle size using Equation (A.1):

$$\Delta X = \frac{100(X_{\text{max}} - X_{\text{min}})}{\overline{X}} \tag{A.1}$$

A.3 Compare the calculated percentage differences to those shown in Table A.1 for the corresponding average number of particle counts. If the calculated percentage difference is less than or equal to the maximum allowable percentage difference value in Table A.1, correct the data for dilution ratio and volume to determine the number of particles per millilitre of the sample. If the calculated percentage difference exceeds the value given in Table A.1, the data should be rejected and appropriate steps taken to correct for analytical errors. If particle counts exceed the maximum allowable percentage difference, repeat the analysis.

Table A.1 — Maximum allowable percentage differences in particle counts between runs

Average number of particles counted per run $\overline{X}$	Maximum allowable difference %
$10\,000 \leqslant \overline{X} < \infty$	11,0
$5\,000 \leqslant \overline{X} < 10\,000$	11,3
$2\,000 \leqslant \overline{X} < 5\,000$	11,9
$1\,000 \leqslant \overline{X} < 2\,000$	13,4
$500 \leqslant \overline{X} < 1\,000$	15,6
$200 \leqslant \overline{X} < 500$	19,3
$100 \leqslant \overline{X} < 200$	27,5
$50 \leqslant \overline{X} < 100$	37,4
$20 \leqslant \overline{X} < 50$	51,8

A.4 If fewer than 20 particles of the particle size of interest are counted, repeat the analysis with a sample volume that ensures that a minimum of 20 particles of the largest particle size of interest can be counted.

Annex B
(informative)

Acceptable diluents

CAUTION — Observe standard laboratory safety practices and regulations when handling diluents.

For the purposes of this International Standard, the following liquids are considered acceptable for use as diluents:

a) MIL-H-5606 (mineral oil);

b) toluene;

c) mineral oil in the HL-XX category (as classified in accordance with ISO 6743-4);

d) propan-2-ol (2-propanol);

e) demineralized water (only if sample is water-based);

f) petroleum spirit, 100 °C to 120 °C grade;

g) others to suit.

NOTE 1 The diluent is considered acceptable if its refractive index is similar to the refractive index of the hydraulic liquid sample. This is to ensure that the diluent does not affect the particle counts.

NOTE 2 The use of a volatile solvent can cause erroneous particle counts due to the generation of air bubbles, i.e. “boiling”, if its vapour pressure is reached in the sensor. This can be alleviated by ensuring that the pressure in the sensor is always above the vapour pressure of any liquids used.

NOTE 3 It is necessary to take care when diluting to ensure that the sample and the diluent are mutually miscible and to ensure that the additive packages of the sample oil and diluent are compatible.

Annex C
(informative)

Method for pre-cleaning diluent and method for incorporating additives into the diluent to eliminate the influence of static electricity on particle counts

CAUTION — Observe standard laboratory safety practices and regulations during the processing of samples.

C.1 Diluent pre-cleaning system

C.1.1 The diluent pre-cleaning system should be designed so as to provide diluent that meets or exceeds the cleanliness requirements of Clause 5. A typical diluent pre-cleaning system is shown in Figure C.1.

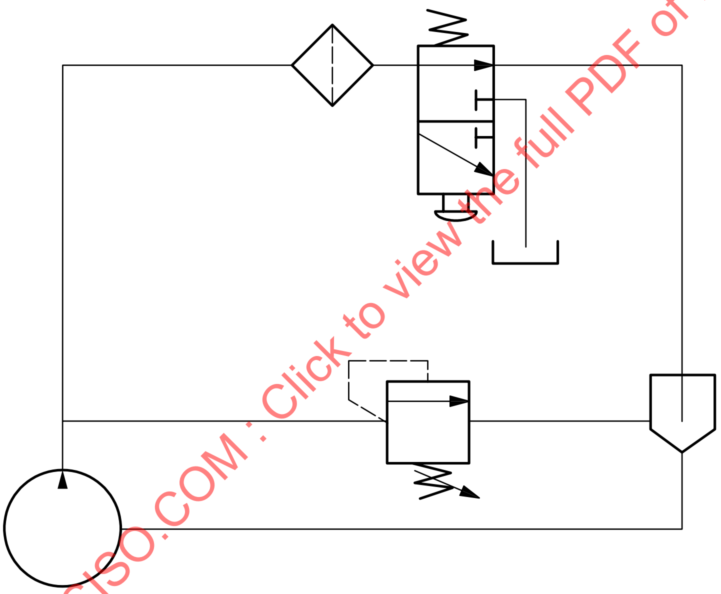


Figure C.1 — Circuit diagram for a typical diluent pre-cleaning system

C.1.2 The reservoir in the diluent pre-cleaning system should have a conical-shaped bottom with an included angle no greater than 90°, and the liquid that goes to the pump should be drawn from the lowest point of the cone. The conical-shaped bottom prevents the heavier particles from accumulating on the bottom of the reservoir and randomly entering the system flow. Piping may be made from polymeric material, stainless steel or copper. Any polymeric material should be compatible with the liquid used. Iron or rubber should not be used as they can contain contaminant generators. The length of the pipe delivering fluid to the valve should be as short as possible and positioned downwards to assist flushing.

C.1.3 A motor-driven impeller pump and a high-efficiency filter capable of cleaning the liquid to the required cleanliness level should be used.

C.1.4 If the diluent pre-cleaning system uses a valve to control the dispensing of the liquid, a ball valve should be used. A needle valve should not be used. The ball valve should not be used to meter the flow rate and should be used in either the fully open or fully closed position. Avoid the use of valves that have graphite-filled stem packing materials or silicone lubricants. Position the valve to prevent particles from settling in the valve.

C.1.5 Before taking a diluent sample, circulate the diluent for a period of time, t, expressed in minutes encompassing a minimum of 20 passes through the filter, in accordance with Equation (C.1):

$$t = \frac{20 \times V_{\text{ts}}}{q} \qquad (C.1)$$

where

V_{ts} is the volume of the test stand, expressed in litres;

q is the flow rate, expressed in litres per minute.

Open the valve and flush out the valve and delivery piping before taking a sample.

C.2 Procedure for determining the cleanliness level of pre-cleaned diluent

C.2.1 Open the sampling valve and operate the diluent pre-cleaning system to establish a homogeneous liquid.

C.2.2 Using a clean bottle of known volume, collect a sample from the diluent pre-cleaning system to fill the bottle to approximately 75 % of the bottle's volume.

C.2.3 Place a cap on the bottle with the sample and place it on the sample-agitating device.

C.2.4 Conduct an analysis of the diluent by following the procedure in 7.3.1 through 7.3.4.

C.2.5 If the desired cleanliness level is not met, reject the diluent and continue cleaning it until the desired cleanliness level is achieved. Unsatisfactory results can mean that the diluent pre-cleaning system requires repair or maintenance.

C.3 Procedure for incorporating additives into the diluent to eliminate the influence of static electricity on particle counts

Prepare the diluent by adding and blending an appropriate amount of antistatic additive to the clean diluent in the reservoir. Operate the clean-up circuit for the time determined in Equation (C.1) to obtain a homogenous mixture.

Monitor the conductivity of the diluent and add further antistatic additive to bring the conductivity to between 1 000 pS/m and 10 000 pS/m.

Annex D
(informative)

Form for reporting particulate contamination level in a hydraulic liquid sample as determined by automatic counting

Laboratory identification: ____________________ **Date of analysis:** ____________________

Sample identification: ____

Sample liquid: ____

APC manufacturer/model: ____________________ **Sensor model:** ____________________

Sensor flow rate: ____________________ mL/min **Sensor coincidence error limit:** ____________________

Method of APC calibration: ____________________ **Date of APC calibration:** ____________________

Dilution ratio: ____________________ **Diluent used:** ____________________

Volume counted per run: ______________ mL

Particle count data

Sample	Run number	Number of particles per millilitre > particle size					ISO 4406 code [a]
		> µm(c)	> µm(c)	> µm(c)	> µm(c)	> µm(c)	
Diluent	1						
	2						
	3						
	4						
	Average of run numbers 2, 3 and 4						
Sample number Dilution ratio: ______________	1						
	2						
	3						
	4						
	Average of run numbers 2, 3 and 4						
	Total						
Sample number Dilution ratio: ______________	1						
	2						
	3						
	4						
	Average of run numbers 2, 3 and 4						
	Total						

[a] If desired.

Comments:

Annex E
(informative)

Data from round robin test programme conducted to verify the procedure specified in ISO 11500

Initially, the revision of ISO 11500:1997 was designed to incorporate a procedure that would enable performing particle-count analysis of hydraulic fluids containing as much as 1 % water.

Two round robins have been conducted to verify the revised procedure. The data from the first round robin test programme had excessive scatter and were abandoned. The data indicated that either the participants did not understand the procedure or they did not follow the revised procedure and continued to process the samples as they had been doing in the past.

A second round robin test programme was conducted and the results were somewhat better; but the objective of establishing a procedure that had the ability to perform particle-count analysis on hydraulic fluids containing water was not confirmed.

The samples used in the round robin test programme were prepared as follows.

a) Secondary calibration samples were provided by a single company.

b) Samples labelled RR1 were cleaned to fewer than 40 particles > 4 µm(c) per millilitre.

c) Samples labelled RR2 had the same cleanliness level as RR1 with 0,1 % water added.

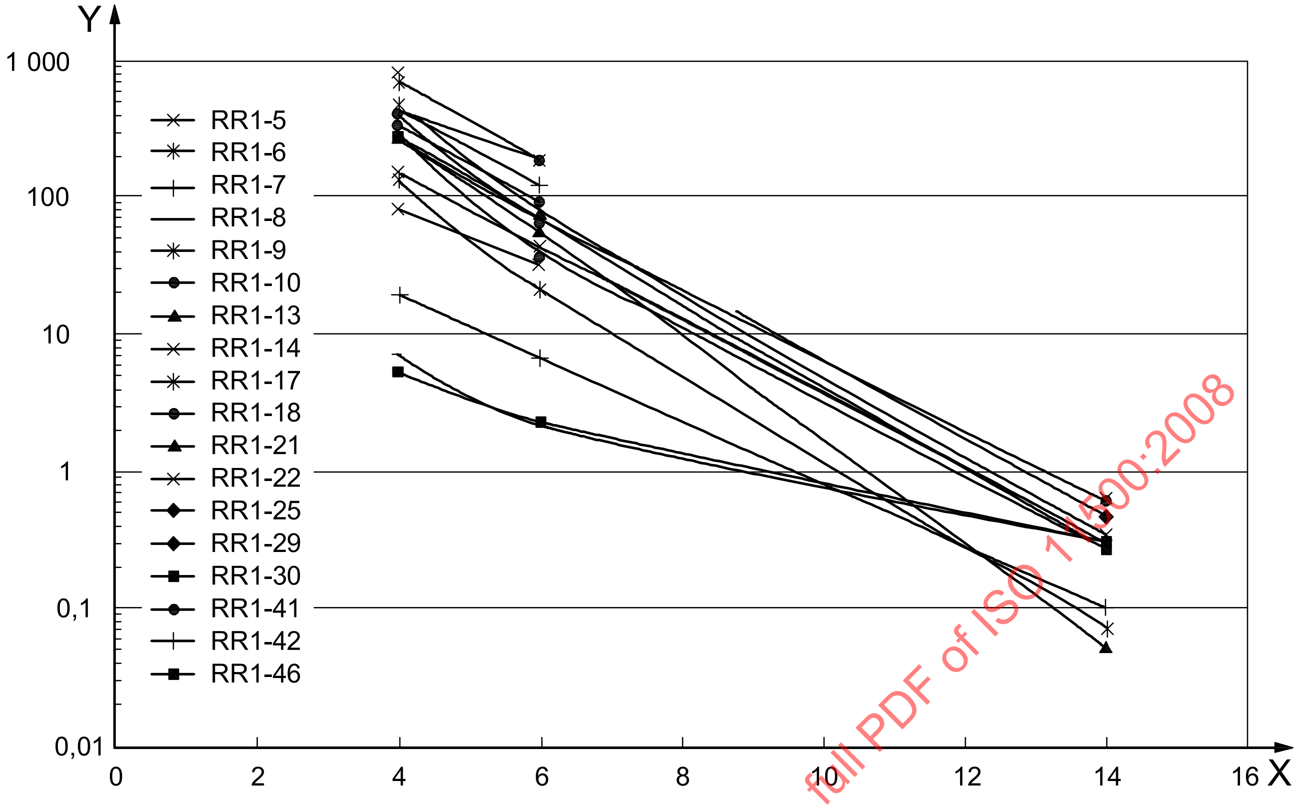
d) Samples labelled RR3 had a solid contamination level of 22/20/16 in accordance with ISO 4406.

e) Samples labelled RR4 had a solid contamination level of 20/19/15 in accordance with ISO 4406.

The data indicated that some participants did not follow the provided procedure and that was believed to be the cause of the inability to process samples containing water. Only two of the 11 participants were able to produce the expected data.

The revision of ISO 11500 was rewritten to remove content pertaining to the analysis of samples that contained water. The data from the prior round robin that related to the samples that contained water were removed from consideration, and the remaining data are provided in this annex (see Figures E.1 to E.3).

Following the publication of ISO 11500:2008, it is anticipated that work will be initiated to conduct another smaller round robin test programme to see if agreement can be reached on a method for analysing samples containing water.

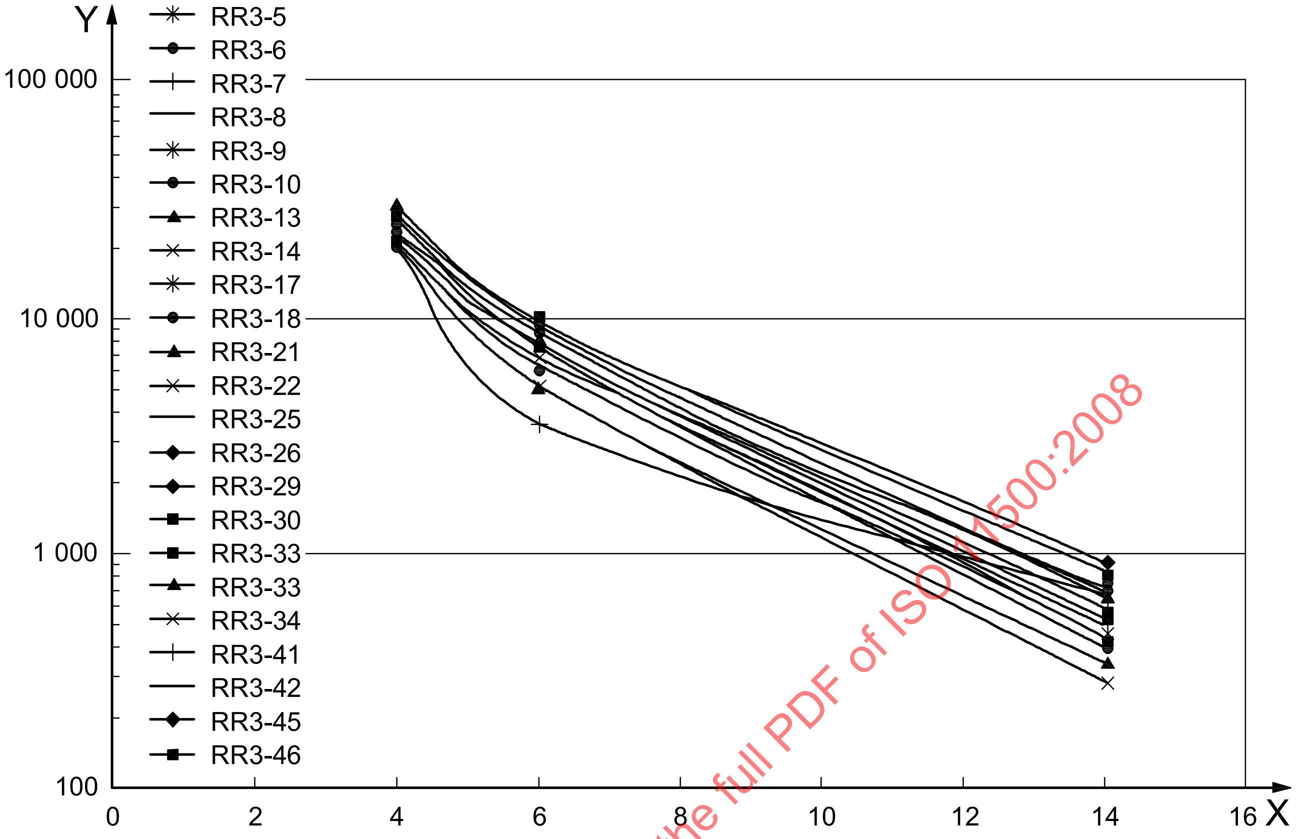


Key

X particle size, µm(c)

Y particles per millilitre greater than the indicated size

Figure E.1 — Number of particles per millilitre vs. particle size for sample RR1

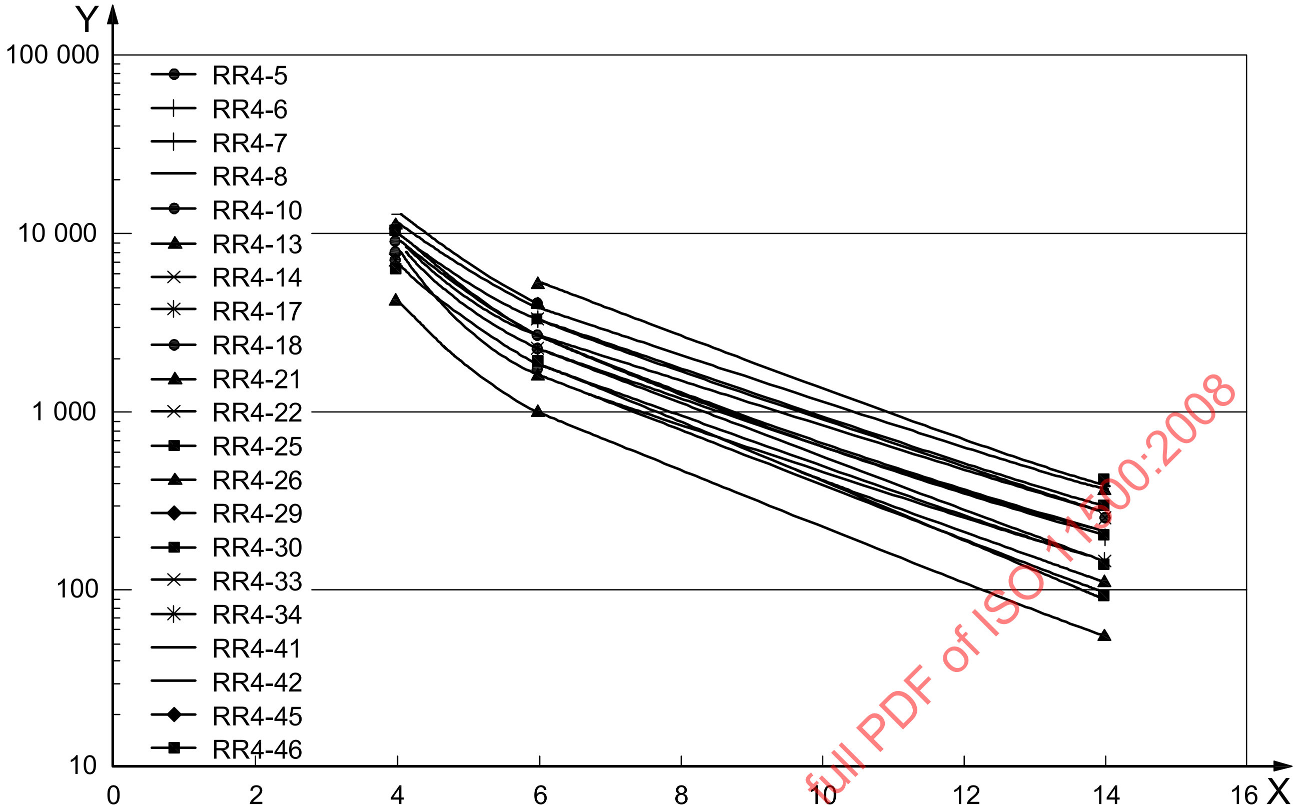


Key

X particle size, µm(c)

Y particles per millilitre greater than the indicated size

Figure E.2 — Number of particles per millilitre vs. particle size for sample RR3



Key

X particle size, µm(c)

Y particles per millilitre greater than the indicated size

Figure E.3 — Number of particles per millilitre vs. particle size for sample RR4