INTERNATIONAL STANDARD

ISO 11500

First edition
1997-09-01

Hydraulic fluid power — Determination of particulate contamination by automatic counting using the light extinction principle

Transmissions hydrauliques — Détermination de la pollution particulaire par comptage automatique à absorption de lumière

This material is reproduced from ISO documents under International Organization for Standardization (ISO) Copyright License number IHS/ICC/1996. Not for resale. No part of these ISO documents may be reproduced in any form, electronic retrieval system or otherwise, except as allowed in the copyright law of the country of use, or with the prior written consent of ISO (Case postale 56, 1211 Geneva 20, Switzerland, Fax +41 22 734 10 79), IHS or the ISO Licensor's members.

Reference number
ISO 11500:1997(E)

Licensed to POLARIS Laboratories, LLC/Jeffrey White
ANSI Store order #X110223 Downloaded: 11/21/2003 9:36:13 PM ET
Single user license only. Copying and networking prohibited.

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

Annex A of this International Standard is for information only.

© ISO 1997

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central
Printed in Switzerland

Licensed to POLARIS Laboratories, LLC/Jeffrey White
ANSI Store order #X110223 Downloaded: 11/21/2003 9:36:13 PM ET
Single user license only. Copying and networking prohibited.

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 needs to be controlled to levels which are considered appropriate for the system concerned.

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 contaminant. The accuracy of particle count data can be affected by the techniques used.

This International Standard details procedures for the analysis of contaminated liquid samples using an automatic particle counter. Use of an automatic particle counter will help to reduce errors and will enhance the accuracy of reproducibility in data.

Licensed to POLARIS Laboratories, LLC/Jeffrey White
ANSI Store order #X110223 Downloaded: 11/21/2003 9:36:13 PM ET
Single user license only. Copying and networking prohibited.

Hydraulic fluid power — Determination of particulate contamination by automatic counting using the light extinction principle

1 Scope

1.1 This International Standard specifies a liquid automatic particle counting procedure for determining the particle size distribution and number of particles present in hydraulic fluid bottle samples taken in accordance with an approved procedure. Measurements are generally made with the particles suspended in the original liquid.

1.2 In general, this method of quantitative analysis is suitable for monitoring

— the contamination level in hydraulic systems;

— the progress of a flushing operation;

— support equipment and test rigs;

— packaged stock.

1.3 The use of this technique is only applicable to clear, homogeneous single-phase liquids; the presence of a fluid interface will obstruct the light beam and give false signals.

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 3448:1992, *Industrial liquid lubricants — ISO viscosity classification.*

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

ISO 3938:1986, Hydraulic *fluid power — Contamination analysis — Method for reporting analysis data.*

ISO 4402:1991, *Hydraulic fluid power — Calibration of automatic-count instruments for particles suspended in liquids — Method using classified AC Fine Test Dust contaminant.*

ISO 4406:1987, *Hydraulic fluid power — Fluids — Code for defining the level of contamination by solid particles.*

ISO 4407:1991, *Hydraulic fluid power — Fluid contamination — Determination of particulate contamination by the counting method using a microscope.*

ISO 4572:— [1], *Hydraulic fluid power — Filters — Multi-pass method for evaluating filtration performance of a filter element.*

---

[1] To be published. (Revision of ISO 4572:1981)

Licensed to POLARIS Laboratories, LLC/Jeffrey White
ANSI Store order #X110223 Downloaded: 11/21/2003 9:36:13 PM ET
Single user license only. Copying and networking prohibited.

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 agglomerates: Two or more particles which are in intimate contact and cannot be separated by gentle stirring and the small shear forces thus generated.

3.2 coincidence: Presence of more than one particle in the sensing volume at the same time.

NOTE — Coincidence causes an overcount of larger particles and an undercount of smaller particles. The coincidence limit of the counter (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. This concentration is normally given by the instrument manufacturer with a note indicating the probability of coincidence. Coincidence is reduced by dilution (see 6.7).

3.3 light extinction: Reduction in intensity of a light beam passing through the sensing volume caused by absorption and/or scattering of the light by single particles.

3.4 "noise" level (of the instrument): Minimum voltage setting of the detection circuit below which spurious signals of electrical noise become significant and are counted as particles.

3.5 saturation level: Saturation of the electronic circuitry when it ceases to function correctly due to an excessive particle concentration.

NOTE — Saturation may also arise when analysing samples containing a very large number of particles whose size is smaller than the minimum size detected by the instrument. Most particle distributions cause a coincidence problem before the instrument experiences saturation.

3.6 sensing volume: Illuminated volume through which particles pass and are detected.

4 Apparatus

4.1 Liquid automatic particle counter (APC), based on the light extinction principle, consisting of an instrument to size and record the electrical signals generated by the passage of single particles through a sensor suitable for counting within the specified range. The particle counter shall include an automatic bottle sampling apparatus or a similar means of allowing for the passage of liquid directly to the sensor and then into a measuring vessel, without altering the contaminant distribution.

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.

4.2 Electronic balance, calibrated, with a resolution of 0,1 mg.

4.3 Volumetric glassware, consisting of a range of graduated dosing pipettes and graduated cylinders conforming to an appropriate standard. All glassware used shall be cleaned and verified in accordance with ISO 3722.

4.4 Optical microscope, to check the suitability of the sample for particle counting using the automatic particle counter. A binocular microscope comprising:

- a magnification of up to × 200,
- a mechanical x — y stage,
- a calibrated eye-piece graticule,
- an internal incident or oblique lighting source,

is considered appropriate.

Licensed to POLARIS Laboratories, LLC/Jeffrey White
ANSI Store order #X110223 Downloaded: 11/21/2003 9:36:13 PM ET
Single user license only. Copying and networking prohibited.

4.5 Sampling agitating device, suitable for redispersing the contaminant in the liquid sample. The device, such as a laboratory bottle roller or paint shaker, shall not alter the basic size distribution of the contaminant.

4.6 Ultrasonic bath, rated at 4 000 W/m^2 of the base area has been shown to be an acceptable means of both dispersing agglomerated particles within the liquid, and removing air introduced by manual agitation. However, experience has shown that particles can be liberated from the container, particularly if the period of immersion in the bath is prolonged (> 30 s). This can affect the validity of the results obtained from the analysis of clean samples, typically ISO 4406 code 16/14 and below.

4.7 Sample containers

The containers are normally cylindrical glass or polypropylene bottles, fitted with either a suitable non-shedding threaded cap forming a seal with the bottle without the use of an insert, or a cap with a suitable internal seal. The dimensions of the bottle will depend upon the type of sample bottle facility in use with the counter, but should normally be 250 ml capacity. The bottle should be flat-bottomed and wide-necked to facilitate cleaning.

The sample containers shall be cleaned and verified in accordance with ISO 3722.

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

4.9 Vacuum filtration apparatus, suitable for filtering the various liquids used in the procedure. Filtering is generally through a 0,45 µm membrane filter, which shall be compatible with the liquids used.

5 Materials

5.1 The following materials are required for cleaning glassware:

a) a supply of distilled or deionised water;

b) a liquid detergent which is water soluble;

c) propan-2-ol, reagent grade, 0,45 µm filtered;

d) petroleum spirit (boiling point between 100°C and 120°C) or a suitable solvent with similar degreasing properties, 0,45 µm filtered.

5.2 Standardized test dust

Calibrated in accordance with ISO 4402.

5.3 Diluent liquid

A suitable liquid for diluting the sample shall be filtered through a 0,8 µm membrane filter or a suitable cartridge filter having an ISO 4572 multi-pass rating of $\beta_1 \geqslant 75$. The diluent liquid shall be compatible with both the sample liquid and the apparatus used.

It is recommended that the refractive index of the diluent liquid should not differ from that of the sample liquid by more than 10 %. An ISO VG5 base mineral oil, as defined in ISO 3448, is considered appropriate for oil samples.

The cleanliness level of the diluent liquid shall be checked before use. A level of less than 2 particles greater than 2 µm and less than 0,5 particles greater than 5 µm per millilitre of liquid is required.

Licensed to POLARIS Laboratories, LLC/Jeffrey White
ANSI Store order #X110223 Downloaded: 11/21/2003 9:36:13 PM ET
Single user license only. Copying and networking prohibited.

6 Procedures

6.1 Precautions

6.1.1 Instrument location

The instrument shall be located in a clean environment to prevent the ingress of airborne particles into the sample during the analysis procedures.

An appropriate environment is considered to be that with a maximum particle count per cubic metre of

— 100 000 for size $\geqslant$ 0,3 µm;

— 35 000 for size $\geqslant$ 0,5 µm;

— 200 for size $\geqslant$ 5 µm; and

— 1 for size $\geqslant$ 10 µm.

NOTE — A list of standards for clean room environments is given in annex A.

6.1.2 Electrical interference

As the APC is typically a high-sensitivity device, it can be affected by radio frequency interference (RFI) or electromagnetic interference (EMI). Precautions should be taken to ensure that the test area does not exceed the RFI and EMI capabilities of the instrument.

In addition, the voltage supply to the instrument shall be stable and free of "noise". A constant voltage transformer is considered appropriate.

6.1.3 Chemicals

Chemicals used in the procedures can be harmful, toxic or flammable. Good laboratory practices should be observed in their preparation and use. Care shall be taken to ensure chemical compatibility with the materials used.

6.1.4 Instrument operation

The instrument shall be used in accordance with the instrument manufacturer's recommendations. All measurements shall be made at particle concentrations which are below 50 % of the instrument manufacturer's stated coincidence limit (see 3.2) and at a size which is at least 1,5 times above the "noise" level of the instrument.

Ensure that the instrument has been switched on for long enough to become stabilised.

6.2 Calibration procedure

6.2.1 Calibration of the APC should be carried out at least every six months, or until enough history with the instrument has been obtained to warrant longer intervals. It shall also be re-calibrated when any change is made to the instrument, or its operation is suspect.

6.2.2 The calibration of the instrument shall be carried out in accordance with ISO 4402.

6.2.3 An interim counting check on the calibration status of the APC should be made regularly.

6.3 Sample bottle preparation

Sample bottles shall be cleaned and verified in accordance with ISO 3722.

Licensed to POLARIS Laboratories, LLC/Jeffrey White
ANSI Store order #X110223 Downloaded: 11/21/2003 9:36:13 PM ET
Single user license only. Copying and networking prohibited.

6.4 Magnetic stirrer

The use of a magnetic stirrer in the bottle sampler is not recommended for samples containing ferrous or other magnetic particles. If such a stirrer is fitted as standard equipment, it may be necessary to remove or negate the drive magnet.

6.5 Preparation of sample before counting

6.5.1 Liquid samples which are found on visual examination to contain contamination which is likely to affect the performance of the sensor shall not be counted by this method.

6.5.2 Remove any visible contamination from the exterior of the bottle using a lint-free cloth.

6.5.3 If the presence of water or other immiscible liquids is seen in the sample, it shall not be analysed by this method as it will interfere with the operation of the instrument. It shall be counted using the optical microscope in accordance with ISO 4407.

6.5.4 It is preferable to analyse the sample in an undiluted form. However, dilution is often necessary to reduce either the optical density of the sample, its viscosity or the particle populations. Guidance on dilution is given in 6.7.

6.5.5 The sample should be checked for suitability for automatic particle counting. Use apparatus for performing an optical particle count as described in ISO 4407, and clean the apparatus accordingly.

6.5.6 Redisperse the contaminant within the contents of the sample in accordance with the procedure described in 6.6.2.

6.5.7 Vacuum filter a 50 ml volume of the sample through a 1,2 µm gridded analysis membrane, pass an equal volume of filtered solvent through to remove residual sample liquid, and allow to dry.

6.5.8 Examine the membrane using an optical microscope fitted with internal incident or oblique lighting at a magnification of × 200, for the presence of either large numbers of small particles (< 5 µm), non-particulate "silt" layers, gels or large particles which may block the sensor. If these are present in significant quantities, then it is recommended that the sample be counted using the optical microscope.

NOTE — The presence of large numbers of small particles (< 5 µm – "silt"), non-soluble additive material and gels will interfere with the operation of the APC and unrepresentative particle counts will result due to coincidence and/or saturation.

6.5.9 Estimate the numbers of particles greater than 5 µm; this will be used to check the validity of the APC count.

6.6 Sample analysis procedure

6.6.1 Sensor system cleanliness

6.6.1.1 It is essential that the sensor and associated pipework be cleaned prior to use by flushing with filtered solvent. This can be achieved by using the pre-cleaned sample bottle filled with filtered solvent and flushing through at a flow rate approximately 50 % higher than the analysis flow rate.

6.6.1.2 If the sensor has previously been used to analyse a liquid which is not miscible with the liquid to be analysed, the sensor will need special cleaning (see 6.8).

6.6.1.3 It is recommended that the sensing volume be inspected on a regular basis for the presence of particles, either in the sensing volume itself or in the entry to it.

6.6.1.4 Verify the cleanliness level of the particle counting system by analysing a suitable volume of filtered solvent or diluent liquid. A cleanliness level corresponding to less than 10 particles greater than 2 µm and less than 2 particles greater than 5 µm per millilitre is required.

Licensed to POLARIS Laboratories, LLC/Jeffrey White
ANSI Store order #X110223 Downloaded: 11/21/2003 9:36:13 PM ET
Single user license only. Copying and networking prohibited.

6.6.2 Redispersion of contaminants

6.6.2.1 If the sample has been standing for a period of time, particle settlement, and hence agglomeration, will occur. It is essential that the agglomerates be broken up, and the contaminant particles be re-dispersed evenly throughout the contents of the sample before analysis.

6.6.2.2 Shake the sample vigorously by hand for at least 1 min, or use an acceptable method of mixing (see 4.5). If ultrasonics is used, stand the sample container so that the level of the liquid in the bath is either just below the fluid level in the sample bottle, or 3/4 up the side of the container, whichever is least. The period of immersion shall not exceed 30 s.

6.6.2.3 Degas the sample by either applying a vacuum of at least 53,5 kPa (535 mbar) to the uncapped sample bottle, or by placing the capped bottle in an ultrasonic bath until no surfacing air bubbles are observed.

6.6.3 Sample analysis

6.6.3.1 It is essential that the particle populations of the sample are below the coincidence limit of the sensor. An assessment will have been made in 6.5.

This can also be verified by extracting an aliquot of the sample to be analysed, diluting it using a high dilution ratio, e.g. 20:1, and analysing the suspension. This will determine whether the sample can be analysed directly and should indicate the optimum dilution ratio.

The final dilution ratio shall be selected to give a particle count of less than 50 % of the stated coincidence as defined in 3.2.

6.6.3.2 The sample shall be analysed immediately after degassing and the sensor shall be flushed with a small volume of the test sample before counting.

6.6.3.3 Count the sample in accordance with the instrument manufacturer's instructions, at the flow rate used during calibration.

6.6.3.4 The sample should be counted in at least three equal volumes to verify the stability of the suspension. These counts should agree within 10 % at the smallest size range. If agreement is not attained, the reason should be investigated.

6.6.3.5 Check the validity of the particle count by comparing this count with either

a) the 5 µm count obtained by optical counting (see 6.5);

b) the exploratory particle count obtained from 6.6.3.1;

c) analysis of a further sample using a dilution ratio of twice that used for analysis. The counts should reduce in number as the dilution ratio increases; if they do not, a further dilution will be necessary.

6.6.3.6 After each test, the sensor and associated pipework should be flushed with pre-filtered solvent.

6.7 Sample dilution

6.7.1 Sample dilution may 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 limit of the instrument;

c) reduce the optical density of the sample; with very dark liquids, the instrument may not operate correctly;

d) perform an exploratory automatic particle count or to verify the validity of the particle count data.

Licensed to POLARIS Laboratories, LLC/Jeffrey White
ANSI Store order #X110223 Downloaded: 11/21/2003 9:36:13 PM ET
Single user license only. Copying and networking prohibited.

6.7.2 The process of dilution can introduce errors in particle count data because of the addition of extraneous contamination, which will be 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 thoroughly cleaned using approved procedures.

6.7.3 An appropriate solvent may be used but experience has shown that the refractive index of the sample can be changed and this can affect particle characterisation.

Single-step dilution is recommended for dilution ratios less than or equal to 10:1, and serial dilution above this ratio. There are two proven methods used to perform dilution. The first is the mass or gravimetric method, the second is the volumetric method which requires high accuracy glassware. High accuracy single-step dilutions are possible using mass dilution techniques.

6.7.4 Single-step dilution

6.7.4.1 Mass or gravimetric method

6.7.4.1.1 Verify the cleanliness level of the diluent immediately before use.

6.7.4.1.2 Place an empty sample bottle on the balance and record its weight.

6.7.4.1.3 Add a suitable volume of diluent liquid, and record its weight.

6.7.4.1.4 Redisperse the contents of the test sample bottle in accordance with 6.6.2. Extract an aliquot and add to the analysis bottle to give approximately the desired dilution ratio. Record the total weight.

6.7.4.1.5 Cap the bottle before remixing.

6.7.4.1.6 Measure the density of the diluent and sample liquids at the test temperature and calculate the actual volumetric dilution ratio. Multiply all subsequent counts by the dilution ratio.

6.7.4.2 Volumetric method

6.7.4.2.1 Verify the cleanliness level of the diluent immediately before use.

6.7.4.2.2 Measure a suitable volume of diluent and add to a pre-cleaned sample bottle.

6.7.4.2.3 Redisperse the contents of the test sample bottle in accordance with 6.6.2, extract a suitable volume of the test sample and add to the analysis bottle.

6.7.4.2.4 Cap the sample bottle before remixing.

NOTE — A glass analysis bottle calibrated in successive volumes has been shown by experience to be a suitable alternative to the use of pipettes and graduated cylinders. It reduces the number of stages in the procedure, and hence the extent of cross-contamination.

6.7.5 Serial dilution

Serial dilution is a two-step dilution procedure to achieve higher dilution ratios without the use of small volumes of sample liquid. The procedure described above is followed, and a further dilution is made on the diluted sample.

6.8 Analysis of different liquids

Care shall be taken when the sensor is used to analyse non-compatible liquids. It is possible that films or droplets of the previous liquid remain on the transparent windows of the sensor, and this will lead to erratic 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 might be:

a) flush with distilled or de-ionised water;

Licensed to POLARIS Laboratories, LLC/Jeffrey White
ANSI Store order #X110223 Downloaded: 11/21/2003 9:36:13 PM ET
Single user license only. Copying and networking prohibited.

b) flush with propan-2-ol;

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

d) dry carefully with dry, oil-free, compressed air.

7 Test report

The test report shall be established in accordance with ISO 3938 for automatic particle counting, with additional comments regarding the condition of the sample being added, if appropriate.

Licensed to POLARIS Laboratories, LLC/Jeffrey White
ANSI Store order #X110223 Downloaded: 11/21/2003 9:36:13 PM ET
Single user license only. Copying and networking prohibited.

Annex A
(informative)

Bibliography

[1] ISO 4021:1992, *Hydraulic fluid power — Particulate contamination analysis — Extraction of fluid samples from lines of an operating system.*

[2] ISO 14644-1:—[2], *Cleanrooms and associated controlled environments — Part 1: Classification of airborne particulate cleanliness for clean rooms and clean zones.*

[3] United States FED-STD-209E. *Clean room and work station requirements, controlled environment.*

[4] BS 5295-1:1989, *Environmental cleanliness in enclosed spaces — Part 1: Specification for clean rooms and clean air devices.*

[5] NF X 44-101:1981, *Définition et classification de la propreté particulaire de l'air et d'autres gaz.*

[6] NF X 44-102:1983, *Enceintes a empoussièrement contrôlé — Définitions — Classifications — Introduction à la procédure de réception et de contrôle périodique.*

---

2) To be published.

Licensed to POLARIS Laboratories, LLC/Jeffrey White
ANSI Store order #X110223 Downloaded: 11/21/2003 9:36:13 PM ET
Single user license only. Copying and networking prohibited.

© ISO

ICS 23.100.01

Descriptors: hydraulic fluid power, hydraulic fluids, contamination, suspended matters, tests, determination, particle density (concentration), size classification.

Price based on 9 pages

Licensed to POLARIS Laboratories, LLC/Jeffrey White
ANSI Store order #X110223 Downloaded: 11/21/2003 9:36:13 PM ET
Single user license only. Copying and networking prohibited.