
**Hydraulic fluid power — Calibration of
liquid automatic particle counters —
Procedures used to certify the standard
reference material SRM 2806**

*Transmissions hydrauliques — Étalonnage des compteurs automatiques
de particules en suspension dans les liquides — Procédures utilisées pour
certifier le matériau de référence normalisé SRM 2806*

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Foreword

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In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

Attention is drawn to the possibility that some of the elements of this Technical Report may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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Introduction

Solid particulates are a major contributor to wear in hydraulic systems. The fluid power industry, the aerospace industry and the military sector utilize optical automatic particle counter (APC) technologies to assess the level of hydraulic oil contamination by suspended particulate. The amount of contamination is often related to the integrity of the system and the usage of the fluid. APCs are also employed in various oil filter testing operations by the manufacturers and the users. The standard method ISO 4402^{[1]1)} has been used for nearly 30 years to calibrate optical particle counters in terms of particle size as a function of particle concentration.

The calibration material used in ISO 4402:1991 is Air Cleaner Fine Test Dust (ACFTD) produced in the past by a division of General Motors Corporation. This material consists of a polydisperse dust having the largest number of particles, as indicated in ISO 4402:1991, with the size range of 1 µm to 80 µm diameter (particle concentration increases with decreasing diameter). There is a low concentration of particles reported to extend out to approximately 100 µm. Some problems have arisen with the use of ACFTD in such calibration procedures. Firstly, there has been ongoing concern that the particle size distribution is not accurate in the small particle size regime (< 10 µm) of the distribution^{[2], [3], [4], [5]}. Many researchers have noted that there are more sub-10 µm particles in ACFTD than reported by ISO 4402:1991. Secondly, but not less importantly, the production of ACFTD has been discontinued by the supplier.

Thus there is a need to investigate, design and devise a new standard method (*Hydraulic fluid power — Calibration method for liquid automatic particle counters*) using a new Standard Reference Material (SRM)^[6]. The National Institute of Standards and Technology (NIST) was requested to develop an SRM for use by the fluid power industry. Users will benefit from improved precision since there is a central source of only one material and increased accuracy resulting from the size characterization^[7]. The new SRM, designated as SRM 2806, is composed of ISO Medium Test Dust (ISO MTD) suspended in MIL-H-5606 hydraulic fluid. The number of particles per millilitre greater than specified sizes has been determined for this material.

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1) Cancelled in 1999 and replaced by ISO 11171:1999.

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Hydraulic fluid power — Calibration of liquid automatic particle counters — Procedures used to certify the standard reference material SRM 2806

1 Scope

This Technical Report describes the procedures used by the United States National Institute of Standards and Technology (NIST) for the certification of the calibration material SRM 2806, which is used in the primary calibration of liquid automatic particle counters.

SRM 2806 is a suspension of ISO MTD in hydraulic fluid with a number size distribution certified using a scanning electron microscope (SEM) and image analysis techniques.

2 Equipment and material

2.1 Test powder

2.1.1 Standard reference material SRM 2806

The particulate material used is a silica powder made from Arizona desert sand by jet milling and then air classifying to a consistent particle size distribution. Several grades with different size ranges are available and their properties are specified in ISO 12103-1^[8].

The powder used to prepare SRM 2806 is an ISO 12103-A3 grade, also called ISO MTD, with supplier batch number 4390C.

2.1.2 Reference materials RM 8631 and RM 8632

Reference materials RM 8631 and RM 8632 are composed of ISO MTD and ISO ultra fine test dust lot numbers 4390C (same lot as the SRM 2806) and 4476 J, respectively. These RMs provide materials to make secondary standards used in support of ISO 11171^[9] and SRM 2806^[10]. The RM was received in 3,6 kg bottles. This dust was dried and spin-riffled into 147 aliquots, each of 20 mg. The material was examined for homogeneity using optical particle counters after suspension in clean oil.

2.2 Test fluid

Test fluid in which ISO MTD is suspended is a hydraulic fluid widely used worldwide for filter testing. This oil is defined in American national standards as MIL-H 5606 and in French national standards as AIR 3520, and in the NATO specification H 515.

Its physical-chemical properties are defined in annex A of ISO 16889:1999^[11].

To ease particle dispersion, a small quantity (50 µg/g) of an antistatic agent is added to the oil so that its conductivity is 1 500 pS/m ± 100 pS/m.

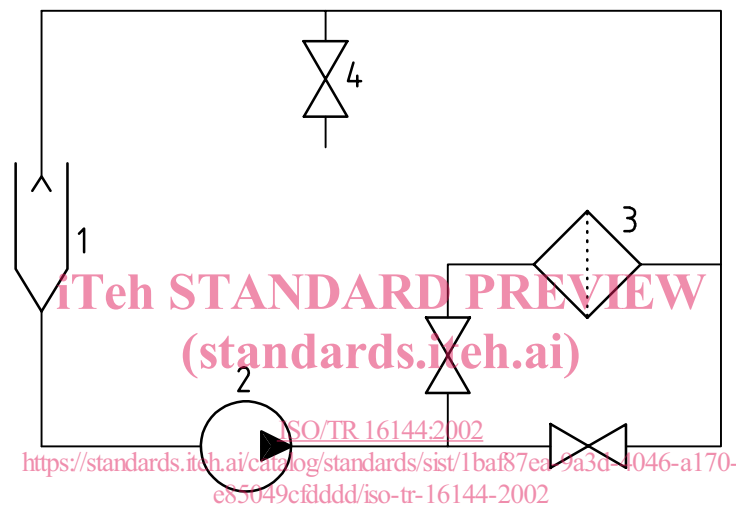
2.3 Sample preparation loop

In view of supplying worldwide demand for several years with the SRM 2806 (supplied in bottles of 400 ml), it was necessary to prepare and store a great number of bottles for further sales.

Because of the settling velocity of the larger silica grains, a special mixing loop was built with mechanical and hydraulic components which were used to eliminate grinding the powder in suspension. It was designed according to the recommendations of ISO 11943^[12].

To guarantee bottle sample homogeneity, a supplementary volume of oil was necessary to allow sampling of control bottles used as described in 3.1.2.

The schematic of the sample preparation loop is given in Figure 1.



Key

- 1 Fluid reservoir (200 l)
- 2 Circulating pump
- 3 Clean-up filter
- 4 Sampling tap

Figure 1 — Schematic of calibration suspension preparation loop

2.4 Membrane preparation equipment

Particles are filtered on 25 mm diameter polycarbonate membranes, 0,2 µm pore diameter using the equipment commonly used for determining hydraulic fluid particulate contamination by gravimetry according to ISO 4405^[13] or by microscopic counting according to ISO 4407^[14].

2.5 Scanning electron microscope and image analyser

The scanning electron microscope used to examine particles is a JEOL 840. The images were produced by electron backscattering and collected on a MicroVax and analysed using LISPIX, a public domain image processing software developed at NIST. LISPIX currently runs on any computer.

3 Equipment validation

3.1 Sample preparation validation

3.1.1 General

Quality assurance for both production and testing was developed by a task force composed of North American members from two filter manufacturers, a particle counter manufacturer, an independent laboratory and NIST. APC measurements were made by both the independent laboratory and NIST, with NIST performing the data analysis.

3.1.2 Homogeneity testing/batch screening

An experimental sampling design was developed and implemented at NIST to measure the bottle-to-bottle homogeneity and, at the same time, to identify possible systematic errors in the instrumental measurements. In the production process, four bottles (a, b, c, d) were filled at any one time. There were 320 bottles per batch and bottles were numerically labelled sequentially from 1 (a, b, c, d), 2 (a, b, c, d), ..., to 80 (a, b, c, d) as they were produced. Selected bottles from each batch were tested for homogeneity at both the independent laboratory and NIST using APCs with extinction sensors calibrated according to ISO 4402:1991. Four bottles (a, b, c, d) were sampled and analysed from approximately the following four points in the production cycle: 5 %, 30 %, 60 %, and 95 %. Another set of four bottles that were produced directly adjacent to the first four were then analysed. For example, the first 16 bottles 5 a, 5 b, 5 c, 5 d, 25 (a, b, c, d), 50 (a, b, c, d) and 75 (a, b, c, d) were analysed in that order. Then bottles 6 (a, b, c, d), 26 (a, b, c, d), 51 (a, b, c, d), and 76 (a, b, c, d) were analysed all by the same calibrated APC. With three replicates for each bottle, this totalled 96 measurements. Each batch of 320 bottles was subjected to this procedure or a modified version of this test. A batch of material was deemed homogeneous if the coefficient of variation for the number of particles larger than 5 µm, 10 µm, 20 µm and 30 µm did not exceed 4 %, 4 %, 4 %, 5 % and 7 % respectively and there were no systematic variations in the batch. The cumulative particle size distribution was determined for the nominal size range of 1 µm to 80 µm particle diameter and measurements were compared for the same batch of materials.

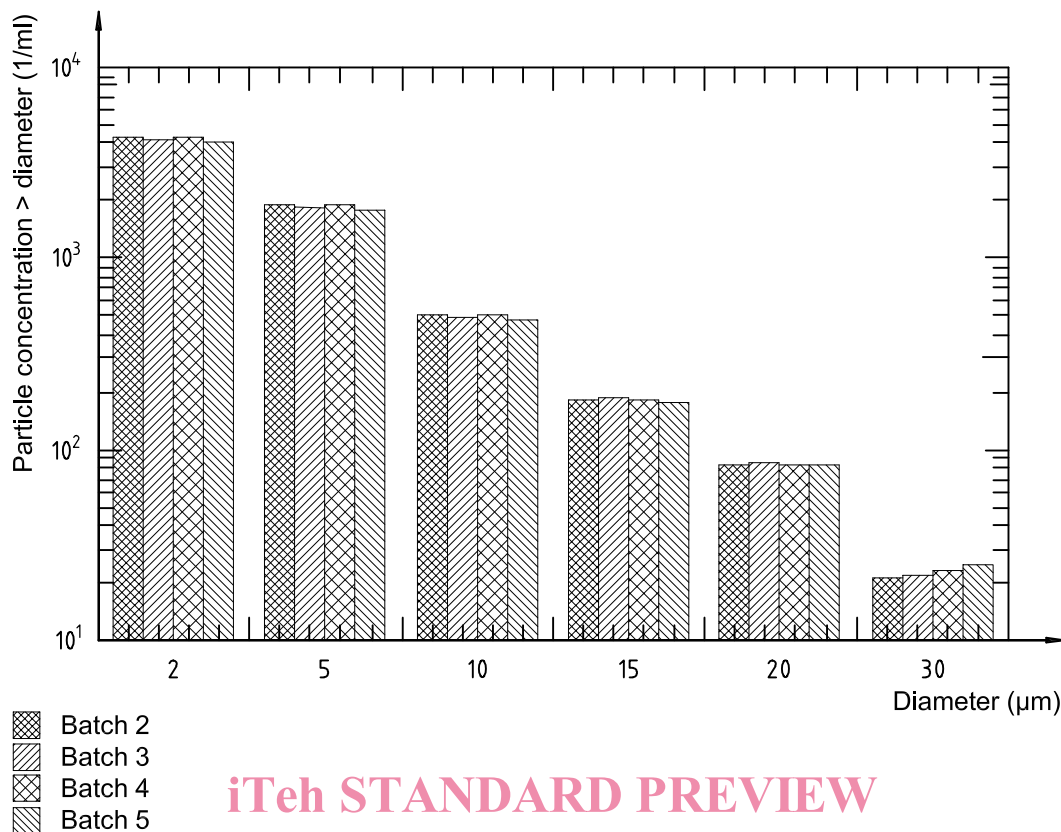
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3.1.3 Homogeneity <https://standards.iteh.ai/catalog/standards/sist/1baf87ea-9a3d-4046-a170-e85049cf1ddd/iso-tr-16144-2002>

To provide high precision measurement capability for a user community, a standard reference material should be as homogeneous as possible. Special efforts were made to assure that this material was made with a low bottle-to-bottle variation within the batch. Within batch variability for the SRM is presented in Table 1 expressed as relative standard deviation for within batch measurements. Figure 2 shows the batch-to-batch comparison in histogram form. The histogram is composed of the mean values of the cumulative particle counts for the same volume of fluid analysed.

Table 1 — Variability found within a batch of material

Greater than size µm	Relative standard deviation %
5	1,1
10	1,3
15	2,0
20	3,8
30	6,7



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Figure 2 — Particle counts in four batches of ISO MTD to verify bottle-to-bottle homogeneity

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3.2 Microscope calibration validation

3.2.1 Microscope calibration

SRM 484d, a NIST scanning electron microscope magnification standard, is mounted in the x and y direction (orthogonal) on the SEM sample stage and used in conjunction with each sample to calibrate the x-y length for particle sizing. SRM 1960, 1 µm polystyrene spheres, were examined by the same procedures used for the dust particles, in order to verify the procedure. Elemental analysis is conducted for a subset of dust particles in the filter sample using energy dispersive X-ray spectroscopy to assure, within the limits of the experiment, that only mineral dust is analysed and that other contaminating particulate material is not present.

3.2.2 Traceability

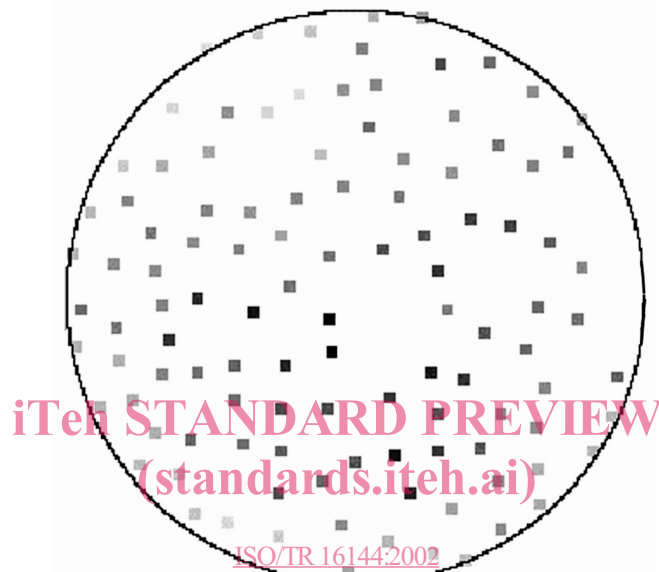
Particle size is traceable to a primary measurement, optical interferometry, through the NIST SRM 484d^[15]. This SRM standard was imaged for each data set collected and for each magnification. From the certified lengths and the measured number of pixels, a pixel-to-length relation was derived and was used to convert the particle images represented in pixels to area in square micrometres. Uncertainties were determined for these conversions (reported as length uncertainty). They are composed of a combination of uncertainty in the pixel determination and the reported uncertainty in the SRM^{[10], [15]}.

3.3 Membrane preparation validation

3.3.1 Sampling from the filter

One component of the total measurement uncertainty results from sampling. Analysis of a large number of fields indicated a non-uniform particle coverage on the filter. Non-uniform particle deposition was observed on many of

the filters after particle separation from the hydraulic oil. Presumably, this was the result of vigorous rinsing with the clean solvent that caused particles to be preferentially relocated toward the centre of the filter. To overcome this sampling problem, the SEM was made to select randomly fields-of-view of particles on the filter surface. Figure 3 shows a schematic of a filter with the regions from which micrograph field of view images were sampled and their respective particle counts represented in grey-level format. The darkest fields contain the largest counts and are generally located in the interior region of the filter, while the low-count fields are found closer to the edge of the filter. In this case, a non-random sampling of only interior fields would give an elevated particle concentration. Note that the edge fields of view that overlap the particle-free boundary are included in the sample population. These edge regions have their area corrected by extracting the particle-free area observed in the micrographs.



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NOTE The darker squares correspond to fields with the highest number of particles counted and the lighter squares indicate low particle coverage.

Figure 3 — Schematic of a filter surface showing the location from which fields were sampled and micrographs obtained

3.3.2 Image analysis

The image analysis was carried out on the original images; none of the features of the particles was altered or enhanced. Thresholding was accomplished visually for each image to maximize the particle thresholded area without introducing background pixels or false particles into the analysis. Once thresholded, the software determined the number of pixels comprising the particles, i.e. the areas. Each magnification that spans a portion of the particle size distribution is analysed separately. The particles have brighter edge regions when compared to the particle interiors as illustrated by Figure 4. The pixel scan across the horizontal line is shown as an insert in the figure. This is observed in electron micrographs because the scattering beam electrons can more readily escape from the particle edges and be detected whereas electrons penetrating the central part of the particle have less probability of detection^[16]. Since the edges are bright, they are almost always above threshold and included in the particle. The software “fills” all hollow particles, i.e. particles having pixels below threshold in their central region. Thus, the critical step in determining accurate particle area is to identify the particle boundary or edge. There is a complication in that the true particle edge can never be known to be better than a pixel width. Edge determinations for linear microstructures by electron microscopy for metrology are discussed at length^[17]. To minimize the area uncertainty, particles need to be represented by a large number of pixels. For example, a 1 µm sphere is represented by approximately 270 pixels at 3 300 × magnification. One would not want to analyse the same particle at 300 × where the particle would be represented by only 2 or 3 pixels. The lowest number of pixels used in the design analysis is, in most cases, between 13 and 50 pixels.