SLOVENSKI STANDARD

# SIST ISO/TR 16144:2003

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**Fluidna tehnika - Hidravlika - Kalibriranje naprav za avtomatsko ģtetje delcev, izloļ enih v tekoļ inah - Uporabljeni postopki za certificiranje standardnih referenļ nih materialov po SRM 2806**

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



**Ta slovenski standard je istoveten z: ISO/TR 16144:2002**

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# TECHNICAL REPORT

# ISO/TR 16144

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2002-09-15

# 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

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 3.

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.

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.



ISO/TR 16144 was prepared by Technical Committee ISO/TC 131, *Fluid power systems*, Subcommittee SC 6, *Contamination control*.



## 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.



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

# 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.

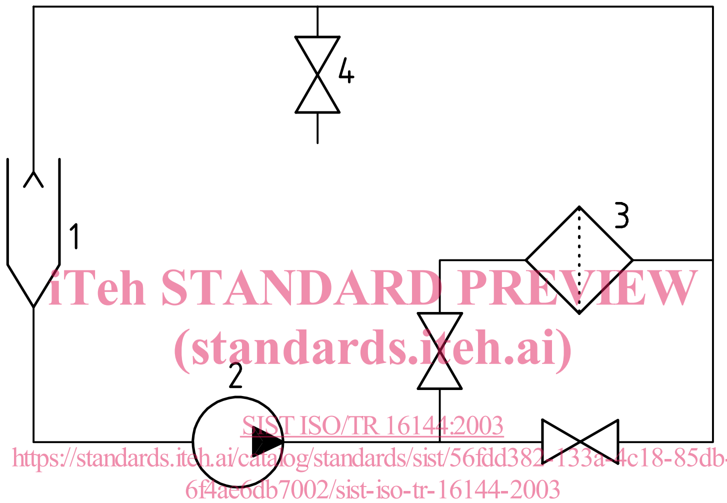
## 2.3 Sample preparation loop

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, 7 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.



### 3.1.3 Homogeneity

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<br>µm | Relative standard deviation<br>% |
|---|---|
| 5 | 1,1 |
| 10 | 1,3 |
| 15 | 2,0 |
| 20 | 3,8 |
| 30 | 6,7 |