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Hydraulic fluid power — Procedures used to certify the standard reference material SRM 2806d used in ISO 11171

Transmissions hydrauliques — Procédures utilisées pour certifier le matériau de référence normalisé SRM 2806d utilisé dans l'ISO 11171

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Foreword

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

NIST Standard Reference Material (SRM) 2806d is intended for use as a reference material for calibrating liquid particle sizing instruments, especially automatic optical particle counters. The fluid power industry estimates that about 80 % of all hydraulic failures are due to particulate contamination in the hydraulic fluid. Equipment failure is costly in terms of repairs, lost productivity, safety, and waste generation. Particle counters are routinely used to monitor fluid cleanliness in operating systems and to assess cleanliness of equipment coming off production lines as well as of individual components. SRM 2806d is a particle counter calibrant for liquid suspensions and a required reference material for ISO 11171[1]. Industries impacted by SRM 2806d include hydraulics industry, aerospace, automotive, ship, petroleum and lubricant, gas, power generation, filter manufacturers and the military.

SRM 2806d is a National Institute of Standards and Technology (NIST) certified standard reference material. "NIST certified value is a value for which NIST has the highest confidence in that all known or suspected sources of bias and imprecision have been considered. For NIST to issue an SRM, the reference material at a minimum must have certain properties:

a) the material must be homogenous;

b) the material must be stable over a suitable length of time;

c) some property of the material to be certified by NIST, a measurand, must be determined with utmost care, usually by employing two or more measurement techniques;

d) the certified value must be traceable to a primary standard, i.e. the International System of Units (the SI).

The measurand values and associated uncertainty in the certified value must be given.[2] Enough information must be given to provide users with confidence that the certified value is fit for the purpose specified in the documents supplied to the user.

SRM 2806d – Medium Test Dust in Hydraulic Fluid is composed of MIL-PRF-5606 hydraulic fluid (Exxon-Mobil)[19][1)] with an added trace amount of SAE 5-80 Medium Test Dust[1)] (Particle Technology Inc., Arden Hills, MN)[3]. The polydisperse medium test dust came from the same batch of dry dust, 4390C, that was used to make past SRM 2806, SRM 2806a, SRM 2806b, RM 8631, RM 8631a and RM8631b. The SRM has been manufactured in approximately 200 l batches with a specified dust level of approximately 6 mg/l, and split into 400 to 800 individual bottles, each bottle sequentially labelled to denote its place in the production sequence.

The measurand for method-dependent values is operationally defined by the procedure used to obtain the value. The values associated with SRM 2806d was partially derived by a method dependent process - consensus in that an interlaboratory study (ILS) was conducted using automatic particle counters to achieve this goal. However, SRM 2806d does have SI traceability through SRM 2806b which was certified by SEM microscopy traceable to the meter through the NIST Line Scan Interferometer.

"A consensus value is derived from suitable number of valid measurement results and represents the best estimate of the value based on agreement between multiple measurement results obtained from the ILS. The ILS could be administered by NIST or another organization that has experience in directing the study and the results and procedures are given with enough detail so that the ILS can be reproduced. There must be an associated uncertainty in the derived value(s) calculated from accepted statistical means and the value(s) and uncertainty needs to be fit for the purpose of the CRM[2]."

For SRM 2806d, the most relevant characteristics of the materials are the following:

— projected area particle diameter that is traceable to a recognized reference and cumulative particle size concentration that is obtained by an accepted method;

— material is homogenous – low variation of cumulative particle size concentration from bottle to bottle;

1) This information represents trademarks for the respective companies and is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named.

— material is stable over the normal use time. This means the hydraulic fluid does not degrade nor does the particle population change due to say agglomeration or particle formation;

— material is free of any extraneous contaminant;

— production and testing are documented well enough to provide assurance and confidence in the material and to provide a basis of quality in case something changes within the material.

There are two main steps to certifying a reference material that NIST can provide to its customer base. The first step is very important because it determines the quality of the material that will be certified by NIST. In the production and acceptance phase, NIST tests the material to verify that it is stable and homogenous and that there are no anomalous properties or materials present in the material. Whether or not the material is acceptable for certification is the critical question to be answered in the first phase. After the material passes the acceptance criteria, some measurand associated with the material that NIST can both determine with high confidence and can assign an associated uncertainty in that value is measured. If NIST cannot have confidence in its determination of the measurand, the material cannot be certified, and cannot become a NIST SRM, but can become an uncertified reference material. In the case of SRM 2806, the material has been certified in the past by microscopy (SEM), but more recently by consensus determination as part of an interlaboratory study. SRM 2806d has both certified and uncertified values associated with it.

Historically, SRM 2806 was certified by determining the cumulative particle size distribution of the medium test dust particles using scanning electron microscopy to obtain electron micrograph images followed by image analysis to determine particle area used to calculate equivalent circular diameter of the particles traceable to the SI.[4] The historic values of the cumulative particle concentration determined by microscopy are given in this document for SRM 2806a, SRM 2806b and SRM 2806d. For the first time, in 2019, a NIST issue of SRM 2806, SRM 2806d, was certified as a consensus standard based on analysis of measurements from an interlaboratory study (ILS) that included 13 laboratories and 22 automatic particle counter (APC) measurements with contributions from 5 countries – China, France, Germany, United Kingdom and the United States. This is the 4th generation of this SRM, but the first based on consensus measurements. The measurements were made traceable to the meter through a secondary standard that was compared to SRM 2806b, the SRM traceable to the meter. Ideally, it would be highly desirable to obtain traceability to a higher, more fundamental standard for each consensus determination.

In summary, the steps taken to assure the quality to certify SRM 2806d are the following:

— production and acceptance testing of candidate material;

— homogeneity testing using a statistical experimental design to select bottles and automatic particle counting by both manufacturer and NIST;

— automatic particle count data subjected to a battery of statistical tests;

— scanning electron microscopy (SEM) measurements on selected samples to verify hydraulic fluid cleanliness and lack of contamination;

— accelerated ageing process at high and low temperatures to confirm stability;

— certification process of accepted candidate material;

— consensus through interlaboratory study with NIST doing the final data analysis;

— verification using SEM micrograph image collection and image analysis to derive projected area particle diameter and cumulative particle concentration to compare with the ILS results;

— annual verification by automatic particle counter to confirm that the SRM continues to perform consistent with the certified values.

The document covers main components of the NIST certification process for SRM 2806 and is divided into the following main sections:

— production and NIST acceptance testing of the candidate material.

— certification process which has been accomplished by either of two methods:

1) Interlaboratory Study (ILS).

2) Microscopy and image analysis.

Hydraulic fluid power — Procedures used to certify the standard reference material SRM 2806d used in ISO 11171

1 Scope

This document describes the criteria and procedures used by the United States National Institute of Standards and Technology (NIST) to certify the calibration material SRM 2806d, which is used in the primary calibration of liquid automatic particle counters (APCs).

SRM 2806d is a suspension of SAE 5 - 80 µm test dust in hydraulic fluid with a cumulative particle concentration determined through a consensus measurement processes, interlaboratory study (ILS), utilizing APCs. The original projected area equivalent diameters for SRM 2806 were certified using a scanning electron microscope (SEM) and image analysis techniques traceable to the meter through the NIST line scan interferometer.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO online browsing platform: available at https://www.iso.org/obp

— IEC electropedia: available at https://www.electropedia.org/

3.1
National Institute of Standards and Technology Standard Reference Material
reference material certified and issued by the National Institute of Standards and Technology (NIST) that provides a measurand with its associated uncertainty used for calibration or comparison with other materials.

Note 1 to entry: The common and recognized abbreviation for this is NIST SRM.

3.2
interlaboratory study
ILS
collaborative study of a material that provides enough technically valid measurement results obtained from appropriate portions of homogenous stable material to enable generation of a consensus value (and associated uncertainty) that is fit for purpose of the material

Note 1 to entry: In this document, the material is an automatic particle counter calibration fluid.

[SOURCE: Reference [2], 2,4, modified — “NIST” deleted before “material”, “obtained from appropriate portions of homogenous stable material” added, “(and associated uncertainty) that is fit for purpose of the material” replaced “whose associated uncertainty is fit for purpose”. Note 1 to entry added.]

3.3
automatic particle counter
APC
instrument that automatically

a) senses individual particles suspended in a controlled volume of fluid using optical light extinction or light scattering principles,

b) provides electrical response that correlates to the size of particles,

c) sorts or compiles responses (particles) into size ranges, and

d) counts particles in each size range.

3.4
scanning electron microscopy
SEM
microscopy using a scanning electron beam to produce micrograph images

3.5
image analysis
process of performing measurements on the morphology of digital micrograph representations of objects

3.6
statistical analysis
data analysis employing accepted mathematical methods to describe the properties of the data, such as patterns and trends, and characterize the data based on overall or average behaviour, as well as provide a description of variation

3.7
equivalent projected area particle diameter
equivalent diameter of a circular particle in two dimensions that has the same area as the particle measured

4 Equipment and material

4.1 Test powder

4.1.1 Reference material for SRM 2806d

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[3].

The powder used to prepare SRM 2806d is SAE 5-80 µm test dust with supplier batch number 4390C in the approximate size range as an ISO 12103-A3 grade, also called ISO MTD.

4.1.2 Reference materials RM 8361 and RM 8632

NIST reference materials RM 8631 and RM 8632 are composed of SAE 5-80 and ISO ultra-fine test dust, lot numbers 4390C (same lot as the SRM 2806d) and 13458D ultra fine test dust, respectively. These RMs provide materials to make secondary standards used in support of ISO 11171 and SRM 2806d[5][6][8].

4.2 Test fluid

Test fluid in which ISO MTD is suspended is a hydraulic fluid widely used worldwide for filter testing. This fluid is defined in American National Standards as Exxon Mobil MIL-PRF-5606 hydraulic fluid. Fluid density measured at 22 °C (at NIST) was 0,869 624 g/cm^3 with an uncertainty of 0,0008 g/cm^3. The index of refraction was found to be 1,470 5.

4.3 Instrumentation

The ILS employed a diverse set of automatic particle counters normally used by the user group of the standard. The current ILS contained four different automatic particle counters models that are widely used in the community.

The NIST laboratory analysis was conducted using two techniques:

a) automatic optical particle counting (APC) using an HRLD 600 sensor (for the initial acceptance) and a HRLD 150JA (for final ILS) both with an auto sampler operating in the volumetric mode, and

b) microscopy using a commercial available scanning electron microscope, TESCAN MIRA-3[1)] automated using SEMantics[8][9] to image the particles and NIST Lispix software to image analyze to determine particle diameter and particle concentration[10].

4.4 Statistical data analysis

Statistical data analysis was conducted at NIST using a graphical Exploratory Data Analysis approach coded in DATAPLOT.[11][12] Overall description of the data and statistical analysis associated with SEM particle analysis process is available[4].

5 Material production and description of acceptance testing

5.1 Production of SRM 2806d

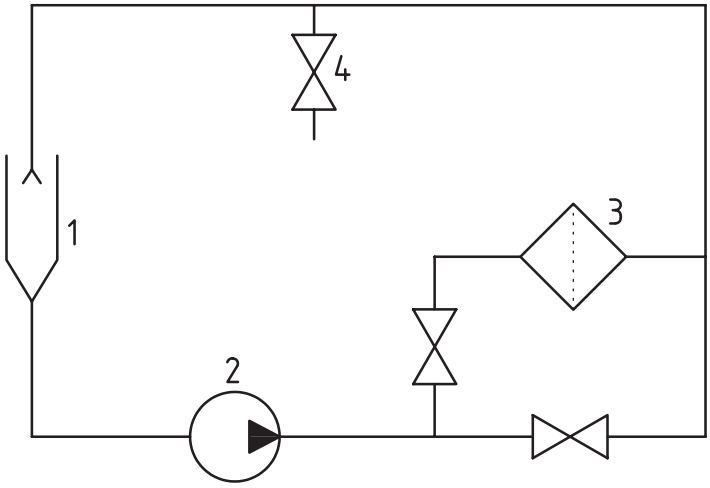
Creation and test procedures that were used to assess the quality and suitability of the new material to become a NIST SRM. The candidate SRM 2806d material was made and tested according to the Specifications for Manufacturing and Acceptance of New SRM 2806 Material document given in the Annex A.

Hydraulic fluid suspension was made using a test stand (with four delivery nozzles A, B, C, and D) that circulates the mineral dust - hydraulic fluid suspension to mix the material thoroughly and produce ideally a large homogeneous suspension. Sequentially 100 bottles were filled from 4 nozzles for a production run of 400 bottles labelled 1A to 100A, 1B to 100B, …, 1D to 100D. Bottles analysed by manufacturer and by NIST were chosen by a statistical design. Bottles were taken from the global collection 400 bottles as specified in Annex A, designed to sample the material over the complete production sequence, but randomized for possible systematic problems related to filling nozzle or circulation flow rate.

From Annex A:

The manufacturer analysed one bottle out of each ten bottles produced for within batch homogeneity as described in ISO 11171:2022, F.3, F.4 and F.5, except that only a total of 10 % bottle samples were analysed (10 % total number). NIST received and tested eight bottles for within batch for homogeneity using an extinction particle counter calibrated to the existing SRM 2806b. The eight bottles consist of two bottles taken from each quartile of the production cycle. The data is compared to analogous data from the manufacturer.

The schematic of the test stand used to prepare the bottles of SRM 2806d is shown in Figure 1, taken from the withdrawn ISO/TR 16144.



Key

1	fluid reservoir (200 l)
2	circulating pump
3	clean-up filter
4	sampling tap

Figure 1 — Schematic of the preparation loop used to mix and fill bottles of SRM 2806d. Sampling tap (4) has four nozzles

5.2 Automatic optical particle counter data

Liquid borne automatic particle counters are used to qualify the candidate material and ultimately provide the data to certify this reference material through a multi-lab measurement process, the above-mentioned ILS.

The procedure for analysis of a single bottle followed the recommended approach described in ISO 11171. The sensor was calibrated using NIST SRM 2806b (the current valid SRM) to determine the particle number distribution of candidate SRM. This single calibration was used throughout the measurements.

5.3 Scanning electron microscope

Selected bottles of candidate SRM 2806d were subjected to the resuspension procedure described in ISO 11171. Approximately 10 mL to 50 mL of hydraulic fluid were poured and then filtered from each sample using 0,4 µm diameter pore size polycarbonate filters. The mass of the aliquot of filtered hydraulic fluid was determined by weighing the fluid bottle sample before and after filtration. The filtered samples were washed three to four times each with a clean, filtered (0,2 µm) reagent grade heptane solvent to remove the oil residue. The filters were evacuated over 48 h to remove any volatiles. Filters were gold coated with ca. 10 nm thick gold delivered by an Ar plasma coater and were mounted on a metal stage and analysed using a TESCAN MIRA-3 SEM[8][9]. The goal of this analysis was to verify the cleanliness of the hydraulic oil samples, to verify that the hydraulic fluid has not degraded and to verify that there is no contaminate materials in the suspension.

SEM images are taken of filtered particles from selected bottles of candidate SRM 2806d. A typical electron backscatter image is shown in Figure 2. The procedure is detailed elsewhere[4].