



**SLOVENSKI STANDARD**  
**SIST EN ISO 13500:2000**  
**01-december-2000**

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**Petroleum and natural gas industries - Drilling fluid materials - Specifications and tests (ISO 13500:1998)**

Petroleum and natural gas industries - Drilling fluid materials - Specifications and tests (ISO 13500:1998)

Erdöl- und Erdgasindustrien - Bohrspülungen - Spezifikationen und Prüfungen (ISO 13500:1998)

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**Ta slovenski standard je istoveten z: EN ISO 13500:1998**

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**ICS:**

75.180.10	Oprema za raziskovanje in odkopavanje	Exploratory and extraction equipment
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**SIST EN ISO 13500:2000**

**en**

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EUROPEAN STANDARD  
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English version

Petroleum and natural gas industries - Drilling fluid materials -  
Specifications and tests (ISO 13500:1998)

Industries du pétrole et du gaz naturel - Fluides de forage -  
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Erdöl- und Erdgasindustrien - Bohrspülungen -  
Spezifikationen und Prüfungen (ISO 13500:1998)

This European Standard was approved by CEN on 1 July 1998.

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

### Foreword

The text of the International Standard ISO 13500:1998 has been prepared by Technical Committee ISO/TC 67 "Materials, equipment and offshore structures for petroleum and natural gas industries" in collaboration with Technical Committee CEN/TC 12 "Materials, equipment and offshore structures for petroleum and natural gas industries", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by month of January 1999, and conflicting national standards shall be withdrawn at the latest by January 1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

### Endorsement notice

The text of the International Standard ISO 13500:1998 has been approved by CEN as a European Standard without any modification.

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STANDARD PREVIEW  
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INTERNATIONAL  
STANDARD

ISO  
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First edition  
1998-07-01

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**Petroleum and natural gas industries —  
Drilling fluid materials — Specifications and  
tests**

*Industries du pétrole et du gaz naturel — Fluides de forage —  
Spécifications et essais*

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Reference number  
ISO 13500:1998(E)

## ISO 13500:1998(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.

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.

## iTeh STANDARD PREVIEW

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International Standard ISO 13500 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum and natural gas industries*, Subcommittee SC 3, *Drilling and completion fluids, and well cement*.

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Annexes A, B, and C of this International Standard are for information only.

## Introduction

This International Standard covers materials which are in common usage in petroleum and natural gas drilling fluids. These materials are used in bulk quantities, can be purchased from multiple sources, and are available as commodity products. No single-source or limited-source products are included, nor are speciality products.

International Standards are published to facilitate communication between purchasers and manufacturers, to provide interchangeability between similar equipment and materials purchased from different manufacturers and/or at different times, and to provide an adequate level of safety when the equipment or materials are utilised in the manner and for the purposes intended. This International Standard provides minimum requirements and is not intended to inhibit anyone from purchasing or producing materials to other standards.

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This International Standard is substantially based on API Spec 13A, 15<sup>th</sup> Edition, May 1, 1993. The purpose of this International Standard is to provide product specifications for barite, haematite, bentonite, nontreated bentonite, Oil Companies Materials Association (OCMA) grade bentonite, attapulgite, sepiolite, technical-grade low viscosity carboxymethylcellulose (CMC-LVT), technical-grade high viscosity carboxymethylcellulose (CMC-HVT), and starch.

The intent of the document was to incorporate all International Standards for drilling fluid materials into an ISO formatted document. A survey of the industry found that only the American Petroleum Institute (API) issued testing procedures and specification standards for these materials.

Reference to OCMA materials has been included in API work, as the OCMA and subsequent holding committees were declared defunct, and all specifications were submitted to API in 1983.



# Petroleum and natural gas industries — Drilling fluid materials — Specifications and tests

## 1 Scope

This International Standard covers physical properties and test procedures for materials manufactured for use in oil- and gas-well drilling fluids. The materials covered are barite, haematite, bentonite, nontreated bentonite, OCMA grade bentonite, attapulgite, sepiolite, technical grade low-viscosity carboxymethylcellulose (CMC-LVT), technical grade high-viscosity carboxymethylcellulose (CMC-HVT), and starch. This International Standard is intended for the use of manufacturers of named products.

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## 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 6780:1988, *General-purpose flat pallets for through transit of goods — Principal dimensions and tolerances*

API RP 13B-1, *Recommended Practice Standard Procedure for Field Testing Water-Based Drilling Fluids* (second edition, 1997)<sup>1)</sup>

API RP 13K, *Recommended Practice for Chemical Analysis of Barite* (second edition, 1996)

APME 1993 (Association of Plastic Manufacturers in Europe)

ASTM D422, *Standard Test Method for Particle-Size Analysis of Soils* (1963)

ASTM E11, *Standard Specification for Wire-Cloth Sieves for Testing Purposes* (1995)

ASTM E77, *Standard Test Method for Inspection and Verification of Liquid-in-Glass Thermometers* (1992)

ASTM E691, *Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method*. (1992)

ASTM E177, *Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods* (1990)

NIST (NBS) *Monograph 150*

<sup>1)</sup> ISO 10414-1 under preparation.

### 3 Definitions and abbreviations

#### 3.1 Definitions

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

##### 3.1.1

##### **ACS reagent grade**

chemicals which meet purity standards as specified by the American Chemical Society (ACS)

##### 3.1.2

##### **flash side**

side containing residue ("flash") from stamping; also, the side with concave indentations

#### 3.2 Abbreviations

ACS	American Chemical Society
API	American Petroleum Institute
ASTM	American Society for Testing and Materials
EDTA	Ethylenediaminetetraacetic acid
CAS	Chemical Abstracts Service
CMC-HVT	Carboxymethylcellulose — High viscosity technical grade
CMC-LVT	Carboxymethylcellulose — Low viscosity technical grade
OCMA	Oil Companies Materials Association
NBS	National Bureau of Standards
NIST	National Institute of Standards and Technology
TC	To contain
TD	To deliver

### 4 Requirements

#### 4.1 Quality control instructions

All quality control work shall be controlled by manufacturer's documented instructions, which include appropriate methodology and quantitative or qualitative acceptance criteria.

#### 4.2 Use of test calibration materials in checking testing procedures

**4.2.1** Test Calibration Barite Lot 001 and Lot 002, and Test Calibration Bentonite can be obtained by contacting the API<sup>2)</sup>. The calibration test materials are shipped in a 7,6 litre (2 gallon) plastic container.

NOTE Test Calibration Barite 001 will be routinely supplied until quantities are exhausted, at which time Test Calibration Barite 002 will take its place.

<sup>2)</sup> American Petroleum Institute, 1220 L Street NW, Washington, D.C. 20005-4070, USA.

**4.2.2** The API office will forward the request to the designated custodian for further handling. The test calibration products will be furnished with a certificate of calibration giving the established values for each property and the confidence limits within which a laboratory's results shall fall.

**4.2.3** The custodian shall furnish a certificate of analysis for each sample.

**4.2.4** For calibration requirements of API Test Calibration Materials, refer to 5.2.11 and 5.3.10.

**4.2.5** API Standard Evaluation Base Clay (formerly OCMA Base Clay - Not OCMA Grade Bentonite): Stocks of API Standard Evaluation Base Clay have been set aside and can be ordered through the API.

### 4.3 Records retention

All records specified in this International Standard shall be maintained for a minimum of five years from the date of preparation.

## 5 Calibration

### 5.1 Coverage

**5.1.1** This clause covers calibration procedures and calibration intervals for laboratory equipment and reagents specified. For laboratory items not listed, the manufacturer shall develop procedures where deemed appropriate.

**5.1.2** The manufacturer shall control, calibrate, verify, and maintain the laboratory equipment and reagents used in this standard for measuring product conformance to standard requirements.

**5.1.3** The manufacturer shall maintain and use laboratory equipment and reagents in a manner such that measurement uncertainty is known and meets required measurement capability.

**5.1.4** The manufacturer shall document and maintain calibration procedures, including details of laboratory equipment and reagent type, identification number, frequency of checks, acceptance criteria, and corrective action to be taken when results are unsatisfactory.

**5.1.5** The manufacturer shall establish and document responsibility for administration of the calibration program, and responsibility for corrective action.

**5.1.6** The manufacturer shall document and maintain calibration records for laboratory equipment and reagents; shall periodically review these records for trends, sudden shifts or other signals of approaching malfunction; and shall identify each item with a suitable indicator or approved identification record to show calibration status.

### 5.2 Apparatus and reagents

#### 5.2.1 Volumetric glassware

Laboratory volumetric glassware used for final acceptance, including Le Chatelier flasks, pipettes, and burettes, are usually calibrated by the supplier. Manufacturers of products to this International Standard shall document evidence of glassware calibration prior to use. Supplier certification is acceptable. Calibration may be checked gravimetrically. Periodic recalibration is not required.

#### 5.2.2 Laboratory thermometers

The manufacturer shall calibrate all laboratory thermometers used in measuring product conformance to standards against a secondary reference thermometer. The secondary reference thermometer shall show evidence of calibration as performed against NIST certified master instruments in accordance with the procedures outlined by ASTM E77-92 and NBS (NIST) Monograph 150.

### 5.2.3 Laboratory balances

The manufacturer shall calibrate laboratory balances periodically in the range of use with NIST Class P, Grade 3, or better weights; and shall service and adjust balances whenever calibration indicates a problem.

### 5.2.4 Sieves conforming to ASTM E11

Approximate dimensions are 76 mm diameter and 69 mm from top of frame to wire cloth. Barite (clause 7) and haematite (clause 8) manufacturers shall calibrate 75 µm sieves using API Test Calibration Barite with established values for residue retained. Haematite (clause 8) manufacturers shall calibrate 45 µm sieves using a suitable quantity of uniform haematite. Bentonite (clause 9), OCMA grade bentonite (clause 11), attapulgite (clause 12) and sepiolite (clause 13) manufacturers shall calibrate 75 µm sieves using API Test Calibration Bentonite. No sieve calibration is available for CMC-Low Viscosity Technical Grade, CMC-High Viscosity Technical Grade and starch, as no reference material and sieve calibration has been established.

### 5.2.5 Hydrometer

The manufacturer shall calibrate each hydrometer with the dispersant solution used in the sedimentation procedure.

### 5.2.6 Motor-driven direct-indicating viscometer

The manufacturer shall calibrate each meter with 20 mPa·s and 50 mPa·s, certified standard silicone fluids.

### 5.2.7 Laboratory pressure-measuring device

The manufacturer shall document evidence of laboratory pressure-measuring device calibration prior to use.

### 5.2.8 Mixer

(e.g. Multimixer® Model 9B<sup>3)</sup> with 9B29X impeller blades or equivalent, mounted flash side up): The manufacturer shall verify that all spindles rotate at  $11\,500\text{ r/min} \pm 300\text{ r/min}$  under no load with one spindle operating. Each spindle will be fitted with a single sine-wave impeller approximately 25 mm in diameter mounted flash side up. New impellers shall be weighed prior to installation, with mass and date recorded.

### 5.2.9 Chemicals and solutions

Shall meet ACS or international equivalent reagent grade if available.

### 5.2.10 Deionized (or distilled) water

Manufacturer shall develop, document, and implement a method to determine hardness of water. The water shall not be used if hardness is indicated.

### 5.2.11 API Test Calibration Materials

Manufacturer shall perform in-house verification of API Test Calibration Barite and/or (where applicable) API Test Calibration Bentonite for properties listed with their Certificates of Analysis.

## 5.3 Calibration intervals

### 5.3.1 General

Any instrument subjected to movement which can affect its calibration shall be recalibrated prior to use.

<sup>3)</sup> Multimixer® Model 9B is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

### 5.3.2 Thermometers

Calibrate each thermometer before being put into service. After calibration, mark each thermometer with an identifying number that ties it to its corresponding correction chart. Check calibration annually against the secondary reference thermometer.

### 5.3.3 Laboratory balances

Calibrate each balance prior to being put into service. Check calibration at least once per month for six months, then at least once per six months *if* required measurements capability is being maintained. If not, service and recalibrate, then check at least once per month until required measurement capability is maintained for six months, then once per six months.

### 5.3.4 Sieves

Calibrate each sieve (where required: see 5.2.4) prior to being put into service. Check calibration at least once per 40 tests. After calibration, mark each sieve with an identifying number that ties it to its correction record. Since sieve calibration will change with use, maintain an up-to-date correction record.

### 5.3.5 Hydrometer

Calibrate each hydrometer prior to its being put into service. After calibration, note and record each hydrometer identifying number that ties it to its correction chart. Periodic recalibration is not required.

### 5.3.6 Motor-driven direct-indicating viscometers

Calibrate each viscometer prior to its being put into service. Check calibration at least once per week for three months, then at least once per month if required measurement capability is being maintained.

### 5.3.7 Mixer

(e.g. Multimixer® Model 9B with 9B29X impellers or equivalent): Check and record mixer spindle speed at least once every 90 days to ensure operation within the prescribed r/min range, using a phototachometer or similar device. Remove, clean, dry, and weigh each impeller blade in service at least once every 90 days. Record masses, and replace blades when mass drops below 90 % of its original value.

### 5.3.8 Deionized (or distilled) water

Manufacturer shall determine hardness of water whenever a new batch of water is prepared or purchased, or whenever deionizing cartridges are replaced.

### 5.3.9 Laboratory pressure-measuring devices

Manufacturer shall document evidence of laboratory pressure-measuring device calibration prior to placing into service and annually thereafter.

### 5.3.10 API Test Calibration Materials

Manufacturer shall test the applicable API Test Calibration Material(s) at least once per three months.

## 5.4 Calibration procedure — Thermometers

**5.4.1** Place thermometer to be calibrated side by side with secondary reference thermometer into a constant-temperature water bath (or suitable container of 4 litres or more, filled with water, on a counter in a constant-temperature room) and allow to equilibrate for at least 1 h.

**5.4.2** Read both thermometers and record readings.

**5.4.3** Repeat readings throughout at least a 1-h interval to obtain a minimum of four readings.

**5.4.4** Calculate the average and the range of readings for each thermometer. The difference between the range of readings for each thermometer shall not exceed 0,1 °C, or the smallest scale division on the thermometer being calibrated.

**5.4.5** Calculate average deviation of thermometer reading from secondary reference thermometer reading. Calculate and document correction for each thermometer.

## 5.5 Calibration procedure — Sieve 75 µm (5.2.4) for barite, haematite, bentonite, attapulgite and sepiolite

NOTE Bentonite is tested by this calibration procedure with the following changes noted:

- Take at least three samples of approximately 10 g Test Calibration Bentonite per 9.8.
- Test each sample per 9.8 using the certified sieve described in 5.5.1.
- Continue procedure outlined in 5.5.4 through 5.5.10.

**5.5.1** Obtain a 75-µm sieve with a *certified centreline value*.

**5.5.2** Take at least three samples of approximately 50 g dry API Test Calibration Barite (TCB).

**5.5.3** Test each of the samples per 7.9 using the certified sieve described in 5.5.1.

**5.5.4** Calculate % residue,  $R$ , for each sample by:

$$\% \text{ Residue, } R = 100 \frac{(\text{mass of residue, g})}{(\text{mass of sample, g})} \quad (1)$$

**5.5.5** Calculate average % residue,  $S$ , of test calibration material on certified sieve by:

$$S = \frac{R_1 + R_2 + R_3 + \dots}{N} \quad (2)$$

where

$R_1 + R_2 + R_3$  is the sum of each individual test result

$N$  is the number of samples tested

Individual sample values shall agree within  $\pm 0,2$  of their average. If not, review test procedure technique and equipment operation for sources of error. Make corrections where needed and repeat.

**5.5.6** From the intersection of % residue and certified sieve opening size on the Sieve Calibration Graph, Figure 1, determine the calibration line (A, B, C, etc.) for use with the specific container of test calibration material. Record this and identify the container with this value.

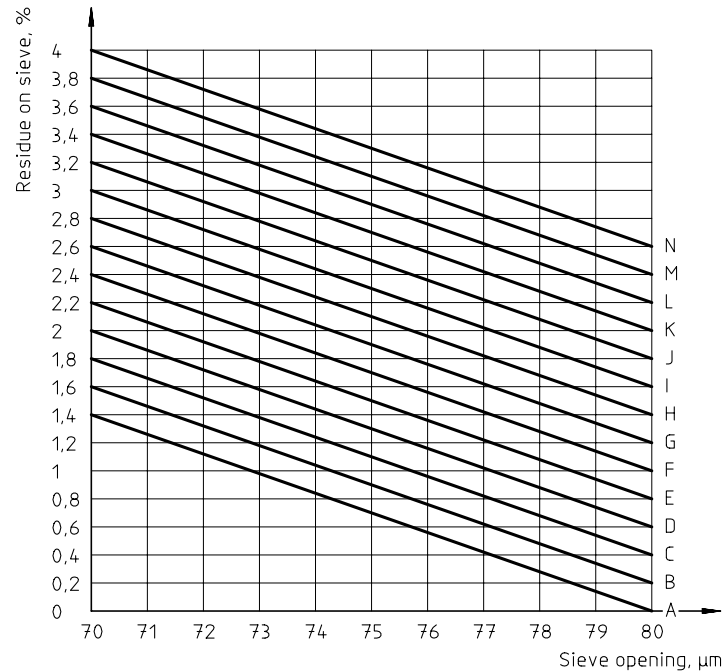


Figure 1 — Sieve calibration graph, 75 µm sieve

5.5.7 Repeat 5.5.2 through 5.5.4 except substitute the working sieve to be calibrated from the certified sieve.

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5.5.8 Calculate average % residue,  $R_a$ , of test calibration material on working sieve by:

$$R_a = \frac{R_1 + R_2 + R_3 + \dots}{N} \quad (3)$$

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Individual sample values shall agree within  $\pm 0,2$  of their average. If not, review test procedure technique and equipment operation for sources of error. Make corrections where needed and repeat, beginning at 5.5.7.

5.5.9 From Sieve Calibration Graph, Figure 1, determine the working sieve opening size to the nearest whole value from intersection of % residue and the calibration line from 5.5.6 above. Record the working sieve opening size and identify the calibrated sieve and test calibration material container.

5.5.10 Determine correction value ( $C$ ) for working sieve from Table 1. Record this value and identify it with the calibrated sieve and specified test calibration material container.

NOTE The sieve correction value obtained from Table 1 as specified is a number to be added to the residue value obtained on a test sample. (Negative values are subtracted.)

5.5.10.1 Example of barite sieve correction value determination:

Certified sieve size = 73 µm

Test calibration barite average % residue on certified sieve,  $S = 2,0$  %

Calibration line = F

Test calibration barite average % residue on working sieve,  $R_a = 1,3$  %

Working sieve size average opening (determined from sieve calibration graph, Figure 1) = 78 µm

Correction value (from Table 1),  $C = + 0,4$  %