

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

The European Standard EN ISO 13500:1998 has the status of a British Standard

ICS 75.180.10

National foreword

This British Standard is the English language version of EN ISO 13500:1998. It is identical with ISO 13500:1998

The UK participation in its preparation was entrusted by Technical Committee PSE/17, Materials and equipment for the petroleum and natural gas industries, to Subcommittee PSE/17/3, Drilling and completion fluids and well cements, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

Conversion factor

For the users of equipment calibrated in oilfield (US) customary units, the following conversion factor should be noted:

1 pound force per square inch (p.s.i.) = 6.894757 kilopascals (kPa).

For example, a water pressure regulator capable of regulation to 10 p.s.i. ± 1 p.s.i. can be considered equivalent to a water pressure regulator capable of regulation to 69 kPa ± 7 kPa, as given in **7.8.9** (barite), **8.8.10** (haematite), **9.7.9** (bentonite), **11.7.9** (OCMA grade bentonite), **12.4.9** (attapulgitite) and **13.4.9** (sepiolite).

Hazard warning (clause 12 - Attapulgitite)

Attapulgitite fibres bear similarities to asbestos fibres, which present a potential health risk. As a result, some countries and various operators have banned attapulgitite from their drilling operations. However, although the most commonly encountered forms of attapulgitite are fibrous, some are granular. If attapulgitite is to be used in drilling operations, then appropriate health and safety precautions should be employed for its handling, usage and disposal.

Cross-references

Attention is drawn to the fact that CEN and CENELEC Standards normally include an annex which lists normative references to international publications with their corresponding European publications. The British Standards which implement these international or European publications may be found in the BSI Standards Catalogue under the section entitled “International Standards Correspondence Index”, or by using the “Find” facility of the BSI Standards Electronic Catalogue.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, the EN ISO title page, the EN ISO foreword page, the ISO title page, pages ii to iv, pages 1 to 66, an inside back cover and a back cover.

Amendments issued since publication

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Descriptors: see ISO document

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 -
Spécifications et essais (ISO 13500:1998)

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

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

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.



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.

INTERNATIONAL STANDARD

**ISO
13500**

First edition
1998-07-01

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

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



Reference number
ISO 13500:1998(E)



Documents
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Descriptors: petroleum industry, natural gas, petroleum extraction, natural gas extraction, drilling, fluids, specifications, physical properties, tests, physical tests.

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 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*.

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.

This International Standard is substantially based on API Spec 13A, 15th 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.

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)¹⁾

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*

¹⁾ 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 API²⁾. 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.

²⁾ 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 9B³⁾ with 9B29X impeller blades or equivalent, mounted flash side up): The manufacturer shall verify that all spindles rotate at 11 500 r/min $\pm$ 300 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.

³⁾ 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:

- a) Take at least three samples of approximately 10 g Test Calibration Bentonite per 9.8.
- b) Test each sample per 9.8 using the certified sieve described in 5.5.1.
- c) 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.

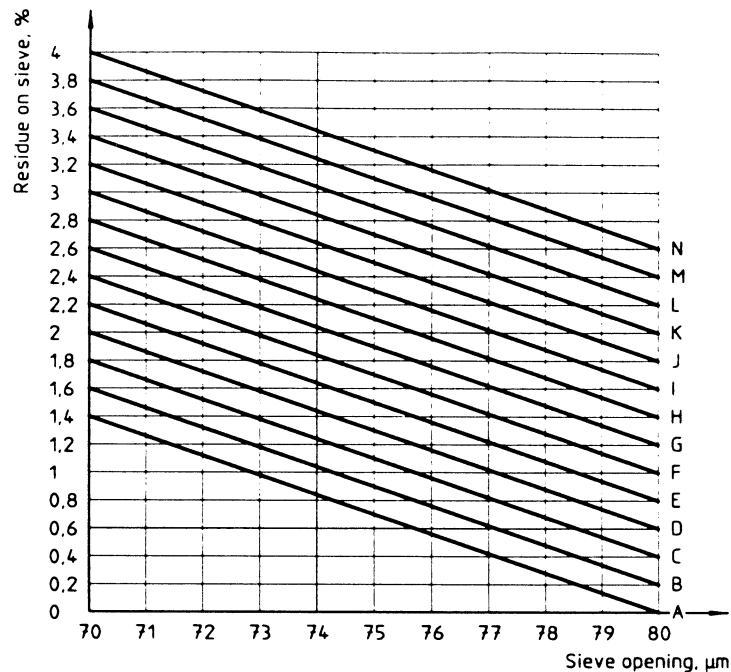


Figure 1 — Sieve calibration graph, 75 µm sieve

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

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

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

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), $C = + 0,4$ %

5.5.10.2 Example of sieve correction application:

Sieve correction value, $C = + 0,4 \%$

Test sample % residue, $R_s = 2,8 \%$

Corrected test sample % residue, $R_c = 2,8 \% + 0,4 \% = 3,2 \%$

These correction values are valid from 0 % to 4 % residue retained on sieve.

NOTE Correction values are rounded to the nearest 0,1.

Table 1 — Correction values (C) for 75 μm^4 sieves

Working sieve size Average opening ^a , μm	Correction size ^b :	
	Barite/Haematite	Bentonite
70	−0,7	−0,3
71	−0,6	−0,2
72	−0,4	0
73	−0,3	0
74	−0,1	0
75	0	0
76	+ 0,1	0
77	+ 0,3	0
78	+ 0,4	0
79	+ 0,6	+ 0,2
80	+ 0,7	+ 0,3

^a Determined from sieve calibration graph, Figure 1.

^b Value to be added to test result of sample tested on sieve to convert results to equivalent 75 μm (NOTE Negative values are subtracted).

5.6 Calibration procedure — Sieve 45 μm (5.2.4) for haematite

5.6.1 Obtain a 45- μm sieve with a *certified centreline value*.

5.6.2 Obtain a suitable quantity of uniform haematite sufficient to last six months or longer. Mix thoroughly and store in a closed container. Identify this as "uniform haematite for 45- μm sieve calibration." Take at least three samples of approximately 50 g dry haematite.

5.6.3 Test each of the samples per 8.9 using the certified sieve described in 5.6.1.

5.6.4 Calculate % residue, R , for each sample by:

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

⁴) ASTM sieve specifications allow $\pm 5 \mu\text{m}$ variation.

5.6.5 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 $\pm 0,5$ of their average. If not, review test procedure technique and equipment operation for sources of error. Make corrections where needed and repeat.

5.6.6 From the intersection of % residue and certified sieve opening size on the Sieve Calibration Graph, Figure 2, 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.

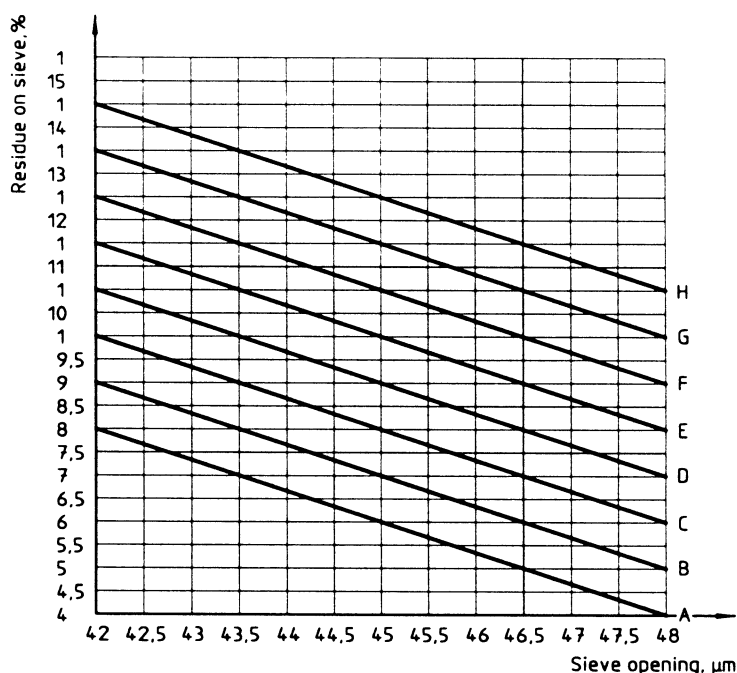


Figure 2 — Sieve calibration graph, 45 µm sieve

5.6.7 Repeat 5.6.2 through 5.6.4 except substitute the working sieve to be calibrated from the certified sieve.

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

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

Individual sample values shall agree within $\pm 0,5$ of their average. If not, review test procedure technique and equipment operation for sources of error. Make corrections where needed and repeat, beginning at 5.6.7.

5.6.9 From Sieve Calibration Graph, Figure 2, determine the working sieve opening size to the nearest whole value from intersection of % residue and the calibration line from 5.6.6 above. Record the working sieve opening size and identify the calibrated sieve and test calibration material container.

5.6.10 Determine correction value (C) for working sieve from Table 2. Record this value and identify it with the calibrated sieve and specified test calibration material container.

NOTE The sieve correction value obtained from Table 2 as specified is a number to be added to the residue value obtained on a test sample. (Negative values are subtracted).

5.6.10.1 Example of haematite sieve correction value determination:

Certified sieve size = 46,5 µm

Haematite average % residue on certified sieve, $S = 7,0 \%$

Calibration line = C

Haematite average % residue on working sieve, $R_a = 9,7 \%$

Working sieve size average opening (determined from sieve calibration graph, Figure 2) = 42,5 µm

Correction value (from Table 2), $C = -1,7 \%$

5.6.10.2 Example of sieve correction application:

Sieve correction value, $C = -1,7 \%$

Test sample residue, $R = 8,8 \%$

Corrected test sample residue, $R_c = 8,8 \% + (-1,7 \%) = 7,1 \%$

Table 2 — Correction value (C) for 45 µm⁵⁾ sieves

Working sieve size Average opening ^a µm	Correction size ^b Haematite
42,0	-2,0
42,5	-1,7
43,0	-1,3
43,5	-1,0
44,0	-0,7
44,5	-0,3
45,0	0,0
45,5	+ 0,3
46,0	+ 0,7
46,5	+ 1,0
47,0	+ 1,3
47,5	+ 1,7
48,0	+ 2,0
NOTE Correction values are rounded to the nearest 0,1.	
^a Determined from sieve calibration graph, Figure 2.	
^b Value to be added to test result of sample tested on sieve to convert results to equivalent 45 µm. (NOTE Negative values are subtracted).	

⁵⁾ ASTM sieve specifications allow $\pm 3 \mu\text{m}$ variation.

5.7 Calibration procedure — Hydrometers

5.7.1 Calibrate each hydrometer to be used using the same concentration dispersant solution as is used in the test, at temperatures spanning the anticipated test temperatures, and by reading the top rather than the bottom of the meniscus. Calibrate *each* hydrometer using the procedure below.

5.7.2 Prepare one litre of dispersant solution as follows:

5.7.2.1 Place $125\text{ cm}^3 \pm 2\text{ cm}^3$ ($125\text{ g} \pm 2\text{ g}$) of dispersant solution from test procedure (7.11.1) into a 1-litre volumetric flask.

5.7.2.2 Dilute to the 1 000-cm³ mark with deionized water. Mix thoroughly.

5.7.3 Place the dispersant solution in a sedimentation cylinder. Then place the cylinder in a constant-temperature bath. Set bath temperature to the lowest expected temperature for any actual test. Allow to reach equilibrium $\pm 0,2\text{ }^\circ\text{C}$. Insert the hydrometer to be calibrated and wait at least 5 min for the hydrometer and solution to reach bath temperature.

5.7.4 Take a hydrometer reading at the top of the meniscus formed by the stem, and take a thermometer reading. Repeat readings at least 5 min apart so as to obtain a minimum of four readings each.

5.7.5 Calculate the average hydrometer reading and designate as $R1$. Calculate the average temperature reading and designate as $T1$.

5.7.6 Repeat 5.7.3 through 5.7.4 except set bath temperature to highest expected test temperature, calculate average hydrometer and temperature readings, and designate these readings as $R2$ and $T2$.

5.7.7 Calculate the hydrometer correction curve slope (Mc) as follows:

$$Mc = 1000 \frac{(R1 - R2)}{(T2 - T1)} \quad (4)$$

where

$R1$ is the average hydrometer reading at lower temperature;

$R2$ is the average hydrometer reading at higher temperature;

$T1$ is the average temperature reading at lower temperature;

$T2$ is the average temperature reading at higher temperature.

NOTE Temperature may be measured in either $^\circ\text{C}$ or $^\circ\text{F}$ so long as all measurements and calculations are consistent in units (including subsequent use of hydrometer in routine test situations).

5.7.8 Calculate the hydrometer correction curve intercept (Bc) as follows:

$$Bc = (Mc \times T1) + [(R1 - 1) \times 1000] \quad (5)$$

where

Mc is the hydrometer correction curve slope;

$T1$ is the average thermometer reading at lower temperature;

$R1$ is the average hydrometer reading at lower temperature.

5.7.9 Record *Mc*, *Bc* and hydrometer serial number in permanent calibration record and on test data sheet used in test 7.13 and 8.13.

For “Hydrometer calibration. Example data sheet and calculation”, see C.1.

5.8 Calibration — Motor-driven direct-indicating viscometers

5.8.1 Apparatus and materials.

5.8.1.1 **Standard thermometer**, with an accuracy of $\pm 0,1^{\circ}\text{C}$, e.g., ASTM 90c or 91c.

5.8.1.2 **Certified calibration fluid**, of viscosity 20 mPa·s, with chart (viscosity vs. temperature).

5.8.1.3 **Certified calibration fluid**, of viscosity 50 mPa·s, with chart (viscosity vs. temperature).

5.8.1.4 **Magnifying glass**, approximately 3× magnification.

5.8.2 Procedure

5.8.2.1 Allow the viscometer and the calibration fluids to stand on counter-top a minimum of 2 h to approach temperature equilibrium.

5.8.2.2 Operate viscometer without fluid a minimum of 2 min to loosen bearing and gears.

5.8.2.3 Clean and dry viscometer cup. Fill the viscometer cup to scribed line with 20 mPa·s calibration fluid and place on meter stage. Raise stage until fluid level is to inscribed line on rotor sleeve.

5.8.2.4 Place thermometer into the fluid and hold or tape to the side of viscometer to prevent breakage.

5.8.2.5 Operate viscometer at 100 r/min setting until thermometer reading is stable to within $\pm 0,1^{\circ}\text{C}$. Record temperature reading.

5.8.2.6 Using magnifying glass, take dial readings at 300 r/min and 600 r/min settings. Estimate readings to nearest 0,5 dial unit and record.

5.8.2.7 Compare 300 r/min dial reading to certified viscosity at test temperature from fluid calibration chart. Record readings and deviation from certified calibration fluid viscosity as furnished by supplier. Divide 600 r/min reading by 1,98 to obtain viscosity value at 600 r/min. Compare this value to certified fluid.

5.8.2.8 Repeat 5.8.2, 5.8.2.3 through 5.8.2.7 using 50 mPa·s fluid.

5.8.2.9 Compare deviations to values in Table 3. Tolerances shall not exceed values in Table 3.

Table 3 — Dial reading tolerances with various calibration fluids, F-1 spring (or equivalent) in motor-driven viscometer

Calibration fluid	Acceptable tolerance	
	300 r/min	600 r/min/1,98
20 mPa·s	$\pm 1,5$	± 2
50 mPa·s	$\pm 1,5$	± 2

5.9 Calibration — Laboratory pressure-measuring devices

5.9.1 Type and accuracy: Pressure-measuring devices shall be readable to at least 2,5 % of full-scale range.

5.9.2 Pressure-measuring devices shall be calibrated to maintain $\pm 2,5$ % accuracy of full-scale range.

5.9.3 Usable range: Pressure measurements shall be made at not less than 25 % nor more than 75 % of the full-pressure span of pressure gauges.

5.9.4 Pressure-measuring devices shall be recalibrated with a master pressure-measuring device or a dead-weight tester at 25 %, 50 %, and 75 % of full scale.

5.10 Calibration — Standardization of EDTA solution

5.10.1 Reagent

5.10.1.1 Standard calcium chloride solution, $c(\text{CaCl}_2) = (0,010\,0 \pm 0,000\,1) \text{ mol/l}$.

5.10.2 Procedure

5.10.2.1 To a suitable flask, add $50 \text{ cm}^3 \pm 0,05 \text{ cm}^3$ deionized water and $50 \text{ cm}^3 \pm 0,05 \text{ cm}^3$ of standard CaCl_2 solution.

5.10.2.2 Proceed as in 7.6.1 through 7.6.5, but without adding barite or additional water. (Use the 100 cm^3 solution prepared above in place of the 100 cm^3 deionized water specified in 7.6.1.)

5.10.2.3 Calculate calibration correction (C_c) as follows:

$$C_c = C_m - 200 \quad (6)$$

where C_m is the $40 \times$ (EDTA volume, cm^3)

NOTE The calibration correction as determined by this procedure results in a number to be subtracted from the sample test value, S_s .

a) Example of calibration correction determination:

EDTA volume for CaCl_2 solution = $4,8 \text{ cm}^3$

$$C_m = 40 \times 4,8 = 192$$

$$C_c = 192 - 200$$

$$C_c = -8$$

a) Example of calibration correction use:

EDTA for sample = $6,1 \text{ cm}^3$

Test value for sample, $S_s = 244 \text{ mg/kg}$

Corrected test value, $S_c = S_s - C_c = 244 - (-8) = 252 \text{ mg/kg}$

6 Packaged materials

6.1 Description

6.1.1 Packaging of palletized goods should safeguard the means of safe handling, transport, storage, and identification, and minimize damage and spillage. Packed material should be inside the dimensions of the pallet although some overhang is allowed.

6.1.2 This procedure applies to products covered by this International Standard. The main intention is to improve the possible recycling of all packaging materials, including dry powdered or granular materials, not covered under this International Standard, used in drilling fluids, completion fluids and oil well cements.

6.2 Apparatus — Pallets

6.2.1 The preferred pallet design and construction should be in accordance with ISO 6780 or APME 1993.

6.2.2 Preferred sizes for wooden pallets include:

- a) 1200 mm × 1000 mm (47 in × 39 in) CP6;
- b) 1140 mm × 1140 mm (45 in × 45 in) CP8/CP9/CP3;
- c) 1219 mm × 1219 mm (48 in × 48 in);
- d) 1118 mm × 1321 mm (44 in × 52 in);
- e) 1067 mm × 1321 mm (42 in × 52 in) equivalent to CP4/CP7;
- f) 1016 mm × 1219 mm (40 in × 48 in).

NOTE CP is the size in accordance with ISO 6780.

6.2.3 Other pallet sizes and details concerning design and construction shall be agreed upon by the manufacturer and the customer.

6.2.4 The maximum outside dimensions of the total package shall be in accordance with the applicable pallet size plus a maximum overhang of 3 cm (1,2 in). The overall height shall not exceed 203,2 cm (80 in).

6.2.5 The maximum net mass should not exceed 2 000 kg (4 409 lb).

6.3 Apparatus — Bags

6.3.1 The manufacturer filling the bag shall take reasonable steps to ensure bag construction capable of safe handling, transport and storage.

6.3.2 The manufacturer shall take reasonable steps to select bags that will minimize waste and provide recycling possibilities of the packaging material.

6.3.3 The manufacturer shall consider humidity-barrier capabilities of the bags against the needs of the particular product when selecting bags.

6.4 Marking — Pallets

Markings shall include the following where applicable and as specified by individual contracts:

- a) product name;
- b) gross/net mass, in kilograms or pounds.

6.5 Marking — Bags

Markings shall include the following where applicable and as specified by individual contracts:

- a) name of the material in print script at least 13 mm in height;
- b) mass of the material in letters, or numbers and letters, at least 6 mm in height. The mass shall be listed in kilograms;

- c) lot/batch number in print script and/or numbers at least 3 mm in height, traceable to manufacturer's country of origin;
- d) identification as recyclable;
- e) safety information.

6.6 Pallet covers

6.6.1 Each pallet may have a cover made of at least one of the following:

- a) Polyethylene (PE) shrink or wrapped film.
- b) PE bonnet type.
- c) Polypropylene (PP) bonnet type.

6.6.2 All plastics shall be UV-stabilized, unless otherwise requested. Cardboard, carton, or wood covers may be used in place of the above. If appropriate, a bottom layer of cardboard, PE sheet or plywood may be connected to the cover to unitize the overall package.

6.7 Storage

The manufacturer shall advise on storage upon request.

6.8 Recycling

6.8.1 General

If appropriate, recycling of the remaining materials after using the contents may be done in accordance with the guidelines given below. All recycling should be done in accordance with local instructions as well as with the administration concerned

6.8.2 Pallets

General recovery and recycling, provided that pallet description is in accordance with ISO 6780 or APME 1993.

6.8.3 Cover

Selection for PE, PP or carton and recycle accordingly.

6.8.4 Bags

Use of high performance paper quality results in less packaging materials and less waste for recycling. After separation of the various components, recycle accordingly.

NOTE When handling chemicals, reduction in the volume of packaging materials can be obtained by application of containers in a dedicated container scheme.

7 Barite

7.1 Description

7.1.1 Drilling grade barite is produced from commercial barium sulfate-containing ores. The manufacturer shall retain certificates of analysis or similar documentation on these commercial barium sulfate ores. It may be produced from a single ore or a blend of ores and may be a straight-mined product or processed by beneficiation methods, i.e., washing, tabling, jigging, or flotation. It may contain accessory minerals other than the barium sulfate (BaSO_4) mineral. Because of mineral impurities, commercial barite may vary in colour from off-white to grey to red or brown. Common accessory minerals are silicates such as quartz and chert, carbonate compounds such as siderite and