INTERNATIONAL STANDARD

ISO 13503-3

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Petroleum and natural gas industries — Completion fluids and materials —

Part 3: Testing of heavy brines

Industries du pétrole et du gaz naturel — Fluides et matériaux de complétion —

Partie 3: Essais de saumures denses

Reference number
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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 2.

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.

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

ISO 13503-3 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*, Subcommittee SC 3, *Drilling and completion fluids, and well cements*.

ISO 13503 consists of the following parts, under the general title *Petroleum and natural gas industries — Completion fluids and materials*:

— *Part 1: Measurement of viscous properties of completion fluids*

— *Part 3: Testing of heavy brines*

The following parts are under preparation:

— *Part 2: Measurement of properties of proppants used in hydraulic fracturing and gravel packing operations*

— *Part 4: Procedure for measuring stimulation and gravel-pack fluid leakoff under static conditions*

— *Part 5: Procedure for measuring the long-term conductivity of proppants*

Introduction

This part of ISO 13503 covers heavy brines commonly used in petroleum and natural gas completion, workover and drill-in fluids. These brines can be purchased or rented from multiple sources, and are available worldwide. No single source or limited source of supply is included, either by inference or reference.

Annexes A to F are given for information.

Petroleum and natural gas industries — Completion fluids and materials —

Part 3: Testing of heavy brines

1 Scope

This part of ISO 13503 covers the physical properties, potential contaminants and test procedures for heavy brine fluids manufactured for use in oil and gas well drilling, completion and workover fluids.

This part of ISO 13503 provides methods for assessing the performance and physical characteristics of heavy brines for use in field operations. It includes procedures for evaluating the density or specific gravity, the clarity or amount of particulate matter carried in the brine, the crystallization point or the temperature (both ambient and under pressure) at which the brines make the transition between liquid and solid, the pH, and iron contamination.

It also contains a discussion of gas hydrate formation and mitigation, brine viscosity, corrosion testing, buffering capacity and a standardised reporting form.

This part of ISO 13503 is intended for the use of manufacturers, service companies and end-users of heavy brines.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments applies).

ISO 10414-1:2001, *Petroleum and natural gas industries — Field testing of drilling fluids — Part 1: Water-based fluids*

ASTM [1)] E77, *Standard Test Method for Inspection and Verification of Thermometers*

NIST [2)] SRM 185h, *Potassium Hydrogen Phthalate, pH Standard*

NIST SRM 186g, *Potassium Dihydrogen Phosphate, pH Standard*

NIST SRM 191C, *pH Standards*

NBS (NIST) Circular 555, *Testing of Hydrometers*, 22 Oct 1954

1) ASTM, American Society for Testing and Materials, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, USA. http://www.astm.org.

2) NIST, National Institute of Standards and Technology, 100 Bureau Drive, Stop 3460, Gaithersburg, MD 20899-3460, USA. http://www.nist.gov.

3 Terms, definitions and abbreviated terms

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

3.1 Terms and definitions

3.1.1
ACS reagent grade
grade of chemical that meets purity standards as specified by the American Chemical Society (ACS)

3.1.2
user
manufacturer, service company, end user or operator applying the testing of this part of ISO 13503

3.2 Symbols and abbreviated terms

ACS American Chemical Society

API American Petroleum Institute

ASTM American Society for Testing and Materials

CAS Chemical Abstracts Service

FCTA first crystal to appear

LCTD last crystal to dissolve

NIST National Institute of Standards and Technology

NTU nephelometric turbidity unit

psi pounds per square inch

TCT true crystallization temperature

TD to deliver

USC United States Customary

V volts

ρ density, in g/cm^3

ρ_1 density at lower temperature

ρ_2 density at upper temperature

ρ_{70} density at 70 °F (21 °C), in lb/gal

ρ_{21} density at 21 °C (70 °F), in g/cm^3

$\rho_{\text{A-SI}}$ average wellbore density, in g/cm^3

$\rho_{\text{A-USC}}$ average wellbore density, in lb/gal

ρ_{m} measured density

ρ_{cf} density, in lb/ft^3

ρ_{SI} density, in kg/m^3

$\rho_{SI\text{-}21}$ density at 21°C, in kg/m^3

ρ_{USC} density, in lb/gal

ρ_W density of water

θ temperature, in °C (°F)

θ_1 temperature at lower reading, in °C (°F)

θ_2 temperature at upper reading, in °C (°F)

θ_m measured temperature, in °C (°F)

∇_{cf} mud gradient, in psi/foot calculated from density expressed in pounds per cubic foot

∇_{SI} mud gradient, in kPa/m

∇_{USC} mud gradient, in psi/foot calculated from density expressed in pounds per gallon

B_c intercept of hydrometer-correction curve

C density correction factor for temperature, in g/cm^3 per °C

C_θ temperature correction factor, in lb/gal per 100 °F

C_p pressure correction factor, in lb/gal per 1 000 psi

C_{SI} density correction factor for temperature, in kg/m^3 per °C

C_{USC} density correction factor for temperature, in lb/gal per °F

h depth (height) of well, in feet

h_2 depth (height) of well at true vertical depth, in feet

R hydrometer reading, specific gravity

R_1 hydrometer reading at lower temperature

R_2 hydrometer reading at higher temperature

V volume, in cm^3

V_1 volume of the original sample, in cm^3

Z_c slope of hydrometer-correction curve

4 Requirements

4.1 Quality control instructions

All quality control work shall be controlled by documented instructions which include appropriate methodology and quantitative acceptance criteria.

4.2 Records retention

All records specified in this part of ISO 13503 shall be maintained for a minimum of one year from the date of preparation.

5 Calibration of equipment

5.1 General requirements

5.1.1 Laboratory equipment and reagents shall be calibrated at periodic intervals, and by specified calibration procedures. For laboratory items not listed, the user shall develop procedures where deemed appropriate.

5.1.2 The user shall control, calibrate, verify and maintain the laboratory equipment and reagents used in this part of ISO 13503 for measuring product conformance.

5.1.3 The user 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 user 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 user shall establish and document responsibility for administration of the calibration programme and responsibility for corrective action.

5.1.6 The user 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 Reagents and materials for calibration

5.2.1 Chemicals and solutions

ACS reagent grade, or equivalent, is recommended. Shelf life shall not exceed the manufacturer's recommendation or six months after opening, if no recommendation is stated.

5.2.2 Distilled or de-ionized water

The user shall develop, document and implement a method to determine hardness of water, such as ASTM D1126 or equivalent. The water shall not be used if hardness is greater than 5 μg/cm^3 calcium ion concentration.

5.3 General requirements for calibration of particular apparatus

5.3.1 Volumetric glassware

Laboratory volumetric glassware used for testing, including flasks and pipettes, are usually calibrated by the supplier. Users of products in accordance with this part of ISO 13503 shall document evidence of glassware calibration prior to use. Supplier certification is acceptable. Calibration may be checked gravimetrically. Periodic recalibration is not required.

5.3.2 Laboratory temperature-measuring devices

The user shall calibrate all laboratory temperature-measuring devices used in measuring product conformance against a secondary reference temperature-measuring device. The secondary reference temperature-measuring device shall show evidence of calibration as performed against NIST certified master instruments in accordance with ASTM E77 and NBS (NIST) Circular 555.

5.3.3 Laboratory balances

The user shall calibrate laboratory balances periodically in the range of use with ASTM Class 1, 4 or 6 or better weights, depending on balance accuracy and in accordance with good laboratory practices, good management practices or ISO quality management standards, and shall service and adjust balances whenever calibration indicates a problem.

5.3.4 Hydrometers

The user shall check the calibration of each hydrometer with fluids of known density referenced to a standard.

5.3.5 Densitometer

The user shall calibrate each densitometer to a NIST standard according to the equipment manufacturer's recommendations.

5.4 Frequency of calibration verification

5.4.1 General

Any instrument subjected to movement that will affect its calibration shall be recalibrated prior to use.

5.4.2 Temperature-measuring devices

Calibrate each temperature-measuring device before it is put into service. After calibration, mark each temperature-measuring device with an identifying number that ties it to its corresponding correction chart. Check calibration as required and stated by manufacturer, against the secondary reference temperature-measuring device.

5.4.3 Laboratory balances

Calibrate each balance prior to being put into service. Check calibration at least annually, or more frequently as stated by manufacturer.

5.4.4 Hydrometers

Calibrate each hydrometer prior to being put into service. After calibration, note and record each hydrometer identifying number that ties it to its correction chart. Before each use, inspect for damage, and if needed recalibrate or discard.

5.5 Calibration verification

5.5.1 Temperature-measuring devices

5.5.1.1 Place the temperature-measuring devices to be calibrated side-by-side with a secondary reference temperature-measuring device in a constant-temperature water bath or suitable container of 4 l (1 gal) or more, filled with water, on a counter in a constant-temperature room. Allow to equilibrate for at least 1 h.

5.5.1.2 Read and record the temperature of all temperature-measuring devices.

5.5.1.3 Repeat the readings at 1 h intervals to obtain a minimum of four readings.

5.5.1.4 Calculate the average and the range of readings for each temperature-measuring device. The difference between the range of readings for each temperature-measuring device shall not exceed 0,1 °C (0,2 °F), or the smallest scale division on the temperature-measuring device being calibrated.

5.5.1.5 If the manufacturer of the temperature-measuring device recommends test calibration at multiple temperatures, repeat 5.5.1.1 through 5.5.1.4 in water baths set at various temperatures.

5.5.1.6 Calculate the average deviation of each temperature-measuring device reading from the secondary reference temperature-measuring device reading. Calculate and document correction for each temperature-measuring device. Discard the temperature-measuring device, if appropriate.

5.5.2 Hydrometers

5.5.2.1 Calibrate each hydrometer to be used using the same solution as the NIST traceable standard hydrometer, at temperatures spanning the anticipated test temperatures, and by reading the bottom of the meniscus. Calibrate each hydrometer using the method given in 5.5.2.2 through 5.5.2.9.

5.5.2.2 Prepare 1 l of test solution of the relevant density.

5.5.2.3 Place the test solution into 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 ± 0,2 °C (± 0,5 °F). Insert the NIST standard hydrometer and wait at least 5 min for the hydrometers and solution to reach the bath temperature, as measured by equilibrium of the solution temperature.

5.5.2.4 Read and record the bottom of the meniscus as the hydrometer reading. Read and record the reading obtained by the temperature-measuring device. Repeat readings at least 5 min apart to obtain a minimum of four readings.

5.5.2.5 Repeat 5.5.2.3 through 5.5.2.4 using the hydrometer to be calibrated.

5.5.2.6 Calculate the average hydrometer reading and designate this R_1. Calculate the average temperature reading and designate it as θ_1.

5.5.2.7 Repeat 5.5.2.3 through 5.5.2.4, with the bath temperature set to the highest expected test temperature. Calculate average hydrometer and temperature readings, and designate these readings as R_2 and θ_2.

5.5.2.8 Calculate the hydrometer-correction curve slope as follows.

$$Z_c = 1000 \frac{(R_1 - R_2)}{\theta_2 - \theta_1} \quad (1)$$

where

Z_c is the hydrometer-correction curve slope;

R_1 is the average hydrometer reading at the lower bath temperature;

R_2 is the average hydrometer reading at the higher bath temperature;

θ_1 is the average temperature at the lower bath temperature;

θ_2 is the average temperature at the higher bath temperature.

Temperature may be measured in either degrees Celsius or degrees Fahrenheit as long as all measurements and calculations are consistent in units.

5.5.2.9 Calculate the hydrometer-correction curve intercept as follows.

$$B_c = (Z_c \cdot \theta_1)[1000(R_1 - 1)] \tag{2}$$

where

B_c is the hydrometer-correction curve intercept;

Z_c is the hydrometer-correction curve slope;

R_1 is the average hydrometer reading at the lower bath temperature;

θ_1 is the average temperature at the lower bath temperature;

5.5.2.10 Record Z_c, B_c and hydrometer serial number in a permanent calibration record.

6 Density

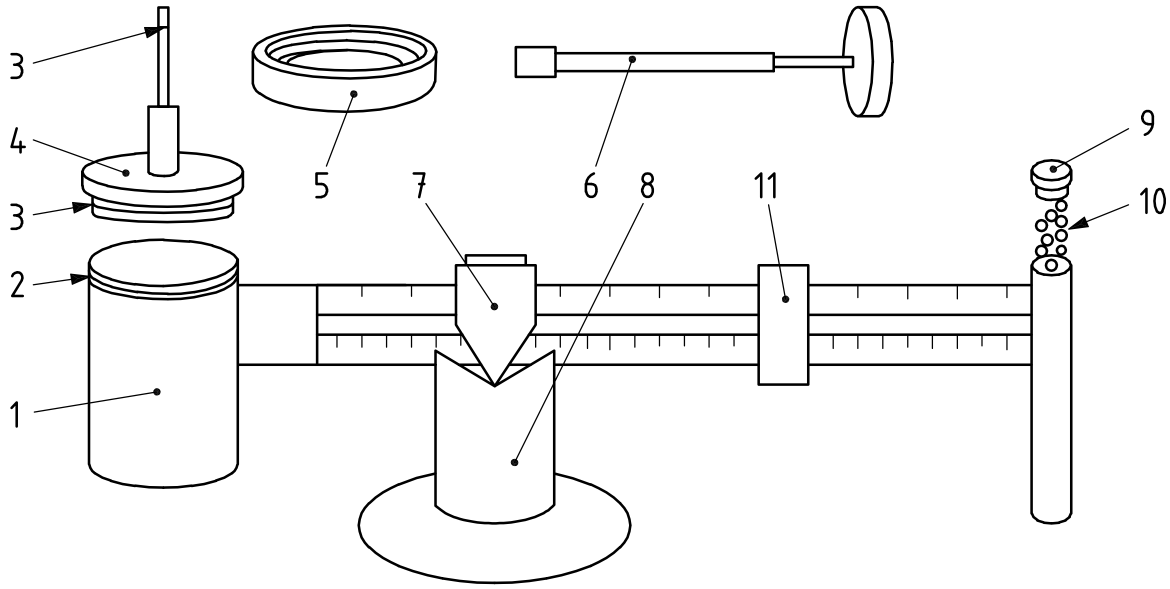
6.1 General

6.1.1 The density of a brine is determined by its salt type and concentration. Moreover, the brine density decreases as the temperature increases. The amount of decrease depends on the brine composition. Conversely, the brine density increases as the pressure on the brine increases. Therefore, the brine density at ambient atmospheric conditions is not a reliable indicator of brine density downhole because the brine density changes with temperature, hydrostatic pressure and applied pressure.

6.1.2 The density of a brine is the mass of brine per unit of volume. Density is generally reported as grams per cubic centimetre, kilograms per cubic metre, or pounds mass per gallon. Density may also be expressed in terms of specific gravity or pressure gradient. Specific gravity is the ratio of the mass of a specific volume of material at a specified temperature when compared to an equal volume of reference material at the same (or another) temperature. For brines, the reference material is water. Pressure gradient is the hydrostatic pressure exerted by the fluid per unit depth.

6.1.3 The reference temperature used to report density of heavy brines is 21 °C (70 °F). The corrected density is not used to determine hydrostatic pressure. Instead, the actual density at ambient temperature is used to calculate hydrostatic pressure.

6.1.4 The measurement of the density of brines is affected by entrapped gasses. If gas entrainment is a problem, it is recommended that the procedures outlined in ISO 10414-1 be used. These procedures use a pressurised fluid density (mud) balance and/or de-aerator to aid in the density measurement. See Figure 1 for an example of a mud balance.



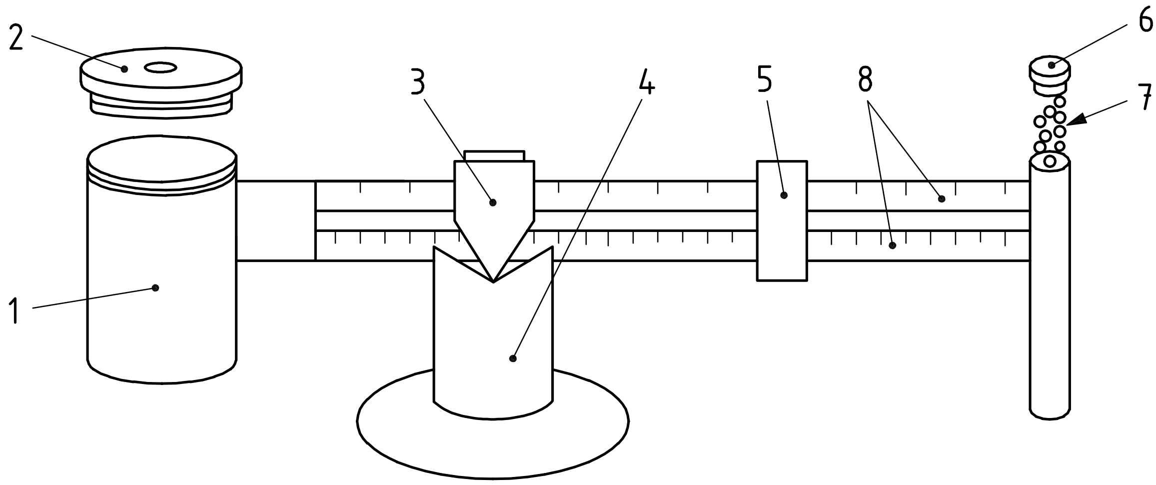
Key

1 sample cup
2 threaded edge to sample cup
3 O-ring seals
4 pressurized lid
5 retaining ring for securing lid
6 pressurization device
7 knife edge with level
8 base with fulcrum
9 weight adjustment screw cover
10 scale calibration material
11 rider for reading scales

Figure 1 — Pressurised mud balance

6.1.5 The atmospheric (see Figure 2) and pressurised mud balances are useful for determining density trends (increases or decreases) in the field, but are not sufficiently accurate for absolute determination of brine density. For purposes of absolute density determination, a set of calibrated hydrometers shall be used instead of a mud balance. For the use and calibration of mud balances, follow the procedures specified in ISO 10414-1.

6.1.6 Any instrument of sufficient precision to permit measurement to within ± 5 kg/m^3 (± 0,005 g/cm^3; ± 0,05 lb/gal) and reportable to within ± 10 kg/m^3 (± 0,01 g/cm^3; ± 0,1 lb/gal) may be used. Generally, this requires the use of hydrometers for fluid density measurements. An atmospheric or pressurised mud balance may be used, but lacks sufficient precision to meet the requirements of this clause for reporting brine density (see 6.2.1 through 6.2.3).



Key

1 sample cup
2 lid for cup
3 knife edge with level
4 base with fulcrum
5 rider for reading scale
6 weight adjustment screw cover
7 scale calibration material
8 scales

Figure 2 — Atmospheric mud balance

6.2 Apparatus

6.2.1 Set of glass hydrometers, covering the range of specific gravities encountered in heavy brine application with graduations in specific gravity units (dimensionless) not greater than 0,002.

The hydrometer is a weighted bulb with a graduated stem. The depth to which the hydrometer sinks in a fluid is determined by the density of the fluid and temperature. The specific gravity is read directly from the graduated stem [5]. Any oil present in the sample will interfere with the measurement, and the use of freshly filtered samples is recommended.

If hydrometers with built-in thermometers are used, it is recommended that the sample be brought to 21 °C (70 °F) to avoid conversion errors.

Most hydrometers used in the oilfield are calibrated to water at 60 °F and sample at 60 °F. Therefore, the calculations reported herein are referenced to the specific gravity based on 60 °F/60 °F hydrometers.

6.2.2 Cylindrical sample container, plastic or glass.

For convenience in pouring, the cylinder may have a lip on the rim. The inside diameter of the cylinder shall be at least 2,54 cm (1,0 in) greater than the outside diameter of the hydrometer used. The height of the cylinder shall be such that the hydrometer floats in the sample fluid with at least 2,54 cm (1,0 in) clearance between the bottom of the hydrometer and the bottom of the cylinder.

6.2.3 Temperature-measuring device, range 0 °C to 105 °C (32 °F to 220 °F), with accuracy of ± 1 °C (± 2 °F).

6.2.4 Filter paper.

6.2.5 Constant temperature bath, regulated to 21 °C ± 1 °C (70 °F ± 2 °F).