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

AMENDMENT 1: Barite 4,1

Industries du pétrole et du gaz naturel — Produits pour fluides de forage — Spécifications et essais

AMENDEMENT 1: Baryte 4,1

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Foreword

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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.

Amendment 1 to ISO 13500:2008 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*.

Amendment 1 to ISO 13500:2008 1) covers the physical properties and testing of the newly available drilling fluid product, Barite 4,1. It constitutes an alternative to the specifications of Clause 7 2), which cover the physical properties and testing of Barite, and is not intended to replace Clause 7.

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1) Amendment 1 to ISO 13500:2008 also constitutes Amendment 1 to API Specification 13A.

2) ISO 13500:2008, Clause 7, is equivalent to API Specification 13A, Section 7.

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AMENDMENT 1: Barite 4,1

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Add the following new clause immediately after Subclause 19.8.3.

20 Barite 4,1

20.1 Principle

20.1.1 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 dolomite, and metallic oxide and sulfide compounds. Although these minerals are normally insoluble, they can, under certain conditions, react with other components in some types of drilling fluids and cause adverse changes in the drilling fluid properties. (See Annex A for more details.)

20.1.2 Drilling grade barium sulfate meeting the Barite 4,1 physical and chemical specifications listed in Table 19 is not intended to meet the requirements of performance tests described in ISO and API publications. Additionally, it is not intended to be used as a reference or calibration material as specified in ISO and API publications.

20.1.3 End users should be aware that the material described in this clause has a lower density than Barite as specified in Clause 7. Drilling fluid formulations should be developed to accommodate this lower density material. Maximum practical drilling fluid density for specific formulations may be lower than attainable with Barite as specified in Clause 7.

20.1.4 End users should review fluid engineering calculations to determine where density differences between Barite 4,1, as specified in this clause, and Barite, as specified in Clause 7, will require revisions to these calculations.

20.1.5 Barite 4,1 shall be deemed to meet the requirements of this International Standard if a composite sample representing no more than one day's production conforms to the chemical and physical specifications of Table 19, represents the product produced, and is controlled by the manufacturer.

Table 19 — Barite 4,1 physical and chemical requirements

Requirement	Standard
Density	4,10 g/ml, minimum
Water-soluble alkaline earth metals, as calcium	250 mg/kg, maximum
Residue greater than 75 µm	Maximum mass fraction, 3,0 %
Particles less than 6 µm in equivalent spherical diameter	Maximum mass fraction, 30 %

20.2 Reagents and apparatus — Density by Le Chatelier flask

20.2.1 Kerosene or **mineral spirits**.

20.2.2 Oven, regulated to 105 °C ± 3 °C (220 °F ± 5 °F).

20.2.3 Desiccator, with calcium sulfate (CAS No. 7778-18-9) desiccant or equivalent.

20.2.4 Le Chatelier flask, with graduations of 0,1 ml, clamped or weighted to prevent flotation in water bath.

20.2.5 Constant-temperature bath, transparent at 32 °C ± 0,5 °C regulated to ±0,1 °C (90 °F ± 1,0 °F regulated to ±0,2 °F) [e.g. approximately 40 l aquarium (fish tank) with heater/circulator attachment, or functional equivalent].

20.2.6 Balance, with an accuracy of 0,01 g.

20.2.7 Pipette, volumetric, of 10 ml capacity.

20.2.8 Magnifying glass.

20.2.9 Dowel, wooden, approximately 8 mm (1/3 in) in diameter and 30 cm (12 in) in length, or a functional equivalent.

20.2.10 Tissue paper, absorbent.

NOTE Laboratory grade tissues are non-absorbent and thus unsuitable for use in this test procedure.

20.2.11 Weighing dish, low-form, with spout, of approximately 100 ml capacity, or a functional equivalent**.**

20.2.12 Brush, small, fine bristle.

20.3 Procedure — Density by Le Chatelier flask

20.3.1 Take approximately 100 g of Barite 4,1 which has been oven dried for at least 2 h and cooled to room temperature in a desiccator.

20.3.2 Fill a clean Le Chatelier flask to approximately 20 mm (0,8 in) below the zero mark with kerosene.

20.3.3 Place the flask upright in the constant-temperature bath. The level of water in the bath shall be higher than the 24 ml graduation of the flask, but below the stopper level. Ensure flask is stabilized by use of clamps or weights.

20.3.4 Allow the flask and contents to equilibrate for a minimum of 1 h. Using the magnifying glass with care to keep eyes at meniscus level, read the volume at the lowest portion of the curved interface, and record the initial volume to the nearest 0,05 ml without removing the flask from the constant-temperature bath. Record as V_1.

If the kerosene level is above or below the −0,2 ml to +1,2 ml volume range after equilibrating, use the 10 ml pipette to add or remove kerosene in order for it to come within this range. Allow the flask to equilibrate for at least 1 h and record the initial volume as in 20.3.4.

20.3.5 Remove the Le Chatelier flask from the bath, wipe dry, and remove the stopper. Roll several lengths of tissue paper diagonally along the length of the dowel, and use this assembly as a swab to dry the inside neck of the flask. Do not allow the swab to come into contact with the kerosene in the flask.

20.3.6 Weigh 80 g ± 0,05 g dried Barite 4,1 into the weighing dish and carefully transfer to the Le Chatelier flask. Take care to avoid splashing the kerosene or plugging the flask with Barite 4,1 at the bulb. This is a slow process, requiring repeated transfers of small amounts of Barite 4,1. Use a brush to transfer any residual Barite 4,1 into the flask, then replace the stopper. Record the mass as m.

20.3.7 If necessary, carefully tap the neck of the flask with the wooden dowel, or agitate carefully from side to side, to dislodge any Barite 4,1 clinging to the walls. Do not allow kerosene to come into contact with the ground glass stopper joint of the flask.

20.3.8 Gently roll the flask along a smooth surface at no more than 45° from vertical, or twirl the upright flask at the neck vigorously between the palms of both hands, to remove entrained air from the Barite 4,1 sample. Repeat this procedure until no more bubbles can be seen rising from the Barite 4,1.

20.3.9 Return the flask to the bath and let stand for at least 0,5 h.

20.3.10 Remove the flask from the bath and repeat 20.3.8 to remove any remaining air from the Barite 4,1 sample.

20.3.11 Immerse the flask in the bath again for at least 1 h.

20.3.12 Record the final volume in the same manner as described in 20.3.4. Record the volume as V_2.

20.4 Calculation — Density by Le Chatelier flask

Calculate the density, ρ, in grams per millilitre, according to Equation (53):

$$\rho = \frac{m}{(V_2 - V_1)} \tag{53}$$

where

m is the sample mass, expressed in grams;

V_1 is the initial volume, expressed in millilitres;

V_2 is the final volume, expressed in millilitres.

Record the calculated density.

20.5 Reagents and apparatus — Water-soluble alkaline earths as calcium

20.5.1 Aqueous EDTA solution, composed of 3,72 g ± 0,01 g of the disodium salt of ethylenediaminetetraacetic acid dihydrate [disodium salt of (ethylenedinitrilo)tetraacetic acid dihydrate] (CAS No. 6381-92-6) diluted to a final volume of 1 000 ml with deionized water in a volumetric flask.

20.5.2 Buffer solution, comprising 67,5 g ± 0,01 g of ammonium chloride (CAS No. 12125-02-9) and 570 ml ± 1 ml of 15 mol/l ammonium hydroxide (CAS No. 1336-21-6) solution diluted to a final volume of 1 000 ml with deionized water in a volumetric flask.

20.5.3 Hardness indicator solution, comprising 1,00 g ± 0,01 g of Calmagite (CAS No. 3147-14-6), or equivalent [1-(1-hydroxy-4-methylphenylazo)-2-naphthol-4-sulfonic acid] diluted to a final volume of 1 000 ml with deionized water in a volumetric flask.

20.5.4 Deionized (or distilled) water.

20.5.5 Balance, of capacity exceeding 100 g with an accuracy of 0,01 g.

20.5.6 Erlenmeyer flask, of 250 ml nominal capacity, equipped with a tight-fitting stopper.

20.5.7 Graduated cylinder, of 100 ml to 150 ml capacity (TC) with 1 ml graduations.

20.5.8 Titration vessel, e.g. beaker, of 100 ml to 150 ml capacity.

20.5.9 Serological pipettes or **burette**, with graduations of 0,1 ml.

20.5.10 Volumetric pipettes (TD), of 10 ml capacity or equivalent.

20.5.11 Filter press, low-pressure/low-temperature, in accordance with ISO 10414-1:2008, Clause 7, or filtration funnel.

20.5.12 Filter paper, Whatman 50, or equivalent.

20.5.13 Glass container, small.

20.5.14 Wrist-action shaker, optional.

20.5.15 Volumetric flask, of 1 000 ml capacity.

20.5.16 Stirring rod.

20.6 Procedure — Water-soluble alkaline earth metals as calcium

20.6.1 Weigh 100 g ± 0,05 g of Barite 4,1. Transfer to the Erlenmeyer flask and add 100 ml ± 1 ml of deionized water. Stopper the flask and shake for at least 5 min during an approximate 1 h interval or by an optional mechanical shaking apparatus for 20 min to 30 min.

20.6.2 After shaking, filter the suspension through the low-pressure filter cell or funnel using two sheets of filter paper and collect filtrate in a suitable glass container.

20.6.3 Add 50 ml ± 1 ml deionized water to the titration vessel. Add about 2 ml of hardness buffer and sufficient hardness indicator to achieve a distinct blue colour. Swirl to mix.

A solution whose colour is other than distinct blue at this point indicates contamination of the equipment and/or water. Find and eliminate the source of contamination and rerun the test.

20.6.4 Using the volumetric pipette, measure 10 ml of the filtrate into the titrating vessel. Swirl to mix. A blue colour indicates an absence of calcium hardness and the test is then complete. A wine-red colour will develop if calcium and/or magnesium are present. Record as V_4.

20.6.5 If hardness is present, begin stirring and titrate with EDTA solution to the blue endpoint. The endpoint of the titration is best described as the point at which additional EDTA produces no further red to blue change. The EDTA volume used to produce the blue endpoint will be used in the calculation in 20.7. Record as V_3.

If the endpoint is unclear or unobtainable, other tests shall be performed. Results and methodology of these tests shall be recorded.

20.7 Calculation — Water-soluble alkaline earths as calcium

Calculate the soluble alkaline earth metals as calcium, w_{AEM}, in milligrams per kilogram, according to Equation (54).

$$w_{\mathrm{AEM}} = 400\left(\frac{V_3}{V_4}\right) \tag{54}$$

where

V_3 is the volume of EDTA used, expressed in millilitres;

V_4 is the volume of filtrate used, expressed in millilitres.

Record the calculated value.

20.8 Reagents and materials — Residue of diameter greater than 75 µm

20.8.1 Sodium hexametaphosphate (CAS No. 10124-56-8).

20.8.2 Oven, regulated to 105 °C ± 3 °C (220 °F ± 5 °F).

20.8.3 Desiccator, with calcium sulfate (CAS No. 7778-18-9) desiccant or equivalent.

20.8.4 Balance, with an accuracy of 0,01 g.

20.8.5 Mixer (e.g. Multimixer® Model 9B with 9B29X impellers or equivalent), having each spindle fitted with a single sine-wave impeller, approximately 25 mm (1 in) in diameter, mounted flash side up.

20.8.6 Container, of approximate dimensions 180 mm (7,1 in) deep, 97 mm (3-5/6 in) d top, 70 mm (2,75 in) d bottom (e.g. Hamilton Beach® mixer cup No. M110-D[3]), or equivalent).

20.8.7 Sieve, 75 µm, conforming to the requirements of ASTM E161, of approximate dimensions 76 mm (3,0 in) diameter and 69 mm (2,75 in) from top of frame to wire cloth.

NOTE Supplier's verification that sieve conforms to ASTM E161 is satisfactory evidence of compliance.

20.8.8 Spray nozzle with 1/4 TT body (Spraying Systems Co., No. TG 6.5 tip with 1/4 TT body[4]), or equivalent), attached to water line with 90° elbow.

20.8.9 Water pressure regulator, capable of regulating to 69 kPa ± 7 kPa (10 psi ± 1 psi).

20.8.10 Evaporating dish or functional equivalent.

20.8.11 Wash bottle.

20.9 Procedure — Residue of diameter greater than 75 µm

20.9.1 If required, equilibrate approximately 60 g of dried Barite 4,1 in a desiccator.

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[3] Hamilton Beach® mixer cup No. M110-D 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.

[4] Spraying Systems Co., No. TG 6.5 tip with 1/4 TT body 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.

20.9.2 Weigh 50 g ± 0,01 g of dried Barite 4,1. Record the mass as m. Add the weighed sample to approximately 350 ml of water containing about 0,2 g of sodium hexametaphosphate. Stir on the mixer for 5 min ± 1 min.

20.9.3 Transfer the sample to the 75 µm sieve. Use wash bottle to remove all material from the container to the sieve. Wash the material on the sieve with water controlled to 69 kPa ± 7 kPa (10 psi ± 1 psi) from a spray nozzle for 2 min ± 15 s. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.

20.9.4 Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.

20.9.5 Dry the residue in the oven to a constant mass. Record the residue mass as m_2 and the total drying time.

20.10 Calculation — Residue of diameter greater than 75 µm

Calculate the mass fraction residue of particles greater than 75 µm, w_1, as a percentage, according to Equation (55):

$$w_1 = 100\left(\frac{m_2}{m}\right) \quad (55)$$

where

m is the sample mass, expressed in grams;

m_2 is the residue mass, expressed in grams.

Record the calculated value.

20.11 Reagents and apparatus — Particles less than 6 µm in equivalent spherical diameter by sedimentation method

20.11.1 Dispersant solution, comprising 40 g ± 0,1 g sodium hexametaphosphate and 3,60 g ± 0,1 g anhydrous sodium carbonate (CAS No. 497-19-8) per 1 000 ml of solution. The sodium carbonate is used to adjust the pH of the solution to approximately 9,0.

20.11.2 Oven, regulated to 105 °C ± 3 °C (220 °F ± 5 °F).

20.11.3 Desiccator, with calcium sulfate (CAS No. 7778-18-9) desiccant or equivalent.

20.11.4 Balance, with an accuracy of 0,01 g.

20.11.5 Mixer (e.g. Multimixer Model 9B with 9B29X impellers or equivalent), having each spindle fitted with a single sine-wave impeller of approximately 25 mm (1 in) in diameter, mounted flash side up.

20.11.6 Container, of approximate dimensions 180 mm (7,1 in) deep, 97 mm (3-5/6 in) d top, 70 mm (2,75 in) d bottom (e.g. Hamilton Beach mixer cup No. M110-D, or equivalent).

20.11.7 Sedimentation cylinder, glass, approximately 457 mm (18 in) in height and 63 mm (2,5 in) in diameter, marked for a volume of 1 000 ml (ASTM D422).

20.11.8 Rubber stopper, No. 13.

20.11.9 Water bath or **constant-temperature room**, capable of maintaining a convenient constant temperature at 24 °C ± 4 °C (75 °F ± 7 °F).

20.11.10 Thermometer, including the range 16 °C ± 0,5 °C to 32 °C ± 0,5 °C (60 °F ± 1,0 °F to 90 °F ± 1,0 °F).

20.11.11 Hydrometer, ASTM 151H, graduated to read specific gravity of suspension.

20.11.12 Timer, mechanical or electrical, with an accuracy of 0,1 min over the test period.

20.12 Procedure — Particles less than 6 µm in equivalent spherical diameter by sedimentation method

20.12.1 Weigh 80 g ± 0,1 g of dry Barite 4,1 and place in a container. Record the mass as m.

20.12.2 Add 125 ml ± 2 ml (127 g ± 2 g) of dispersant solution (20.11.1). Dilute to approximately 400 ml with deionized water. Rinse all adhering particles from the spatula into the suspension.

20.12.3 Stir 5 min ± 0,5 min in the mixer.

20.12.4 Transfer the suspension to the sedimentation cylinder. Rinse the container with deionized water to ensure that all sample particles are transferred to the sedimentation cylinder.

20.12.5 Add deionized water to the 1 000 ml mark. Mix the contents thoroughly by constantly changing the cylinder from the upright to the inverted position and back for 60 s ± 5 s while holding a No. 13 rubber stopper in the top of the cylinder.

This is a critical step. Suspension shall be homogeneous at the start of sedimentation. This is difficult to obtain because of the high density of Barite 4,1.

20.12.6 Set the cylinder into the water bath (or on the counter top of the constant-temperature room) and, simultaneously, start the timer. Hang the thermometer in the water bath.

20.12.7 Take hydrometer readings at intervals of 10 min ± 0,1 min, 20 min ± 0,1 min, 30 min ± 0,1 min and 40 min ± 0,1 min (or until the first point below the 6 µm value is reached). To take a hydrometer reading, carefully and slowly lower the hydrometer to approximately the 1,020 reading before releasing. Once the hydrometer stabilizes, read the top of the meniscus at the prescribed time. Carefully and slowly, remove the hydrometer, rinse with deionized water, and dry after each reading. The hydrometer shall be removed immediately after each reading to eliminate particle build-up on the shoulders, which causes erroneous results. All hydrometer readings shall be done with a minimum of fluid disturbance to preserve the suspension-settling equilibrium.

20.12.8 Record the time, t, expressed in minutes, the temperature, θ, expressed in degrees Celsius (degrees Fahrenheit) and the hydrometer reading, R, on the data sheet.

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

20.12.9 For each time interval, determine the water viscosity, η, and the effective hydrometer depth, L, from Tables 3 and 4. Record on the data sheet.

20.13 Calculation — Particles less than 6 µm in equivalent spherical diameter by sedimentation method

20.13.1 From the hydrometer calibration (5.2.5.2), enter the hydrometer correction slope, M_c, and hydrometer correction intercept, B_c, onto the data sheet.

20.13.2 Calculate the sample constant, K_s, as given in Equation (56) (or determine from Table 20) and enter into the data sheet:

$$K_\mathrm{s} = 100 \frac{\rho}{m(\rho - 1)} \tag{56}$$

where

ρ is the sample density, expressed in grams per millilitre;

m is the sample mass, expressed in grams.

20.13.3 Calculate and enter into the data sheet the equivalent spherical diameter, D_e, in micrometres, for each time interval as given in Equation (57):

$$D_\mathrm{e} = 17{,}5\sqrt{\frac{\eta L}{(\rho - 1)t}} \tag{57}$$

where

η is the viscosity of the water, expressed in millipascal seconds;

ρ is the sample density, expressed in grams per millilitre;

t is the time, expressed in minutes;

L is the effective depth (see Table 4), expressed in centimetres.

20.13.4 Calculate and enter into the data sheet the cumulative finer value percentage, w_a, for the equivalent particle diameter, D_e, as given in Equation (58):

a) for the cumulative percent particle diameter equivalent immediately greater than 6 µm, w_2, using D_1 determined in Equation (57);

b) for the cumulative percent particle diameter equivalent immediately less than 6 µm, w_3, using D_2 determined in Equation (57);

$$w_\mathrm{a} = K_\mathrm{s}\left[\left(M_\mathrm{c} \cdot \theta\right) - B_\mathrm{c} + \left(R - 1\right)1\,000\right] \tag{58}$$

where

K_s is the sample constant;

M_c is the hydrometer correction slope, determined in 5.2.5.2.7, Equation (1);

θ is the suspension temperature, expressed in degrees Celsius or degrees Fahrenheit (see 20.12.8);

B_c is the hydrometer correction intercept, as determined in 5.2.5.2.8, Equation (2);

R is the hydrometer reading.

20.13.5 Calculate and enter into the data sheet the cumulative percentage below 6 µm, w_4, as given in Equation (59):

$$w_4 = \left[\left(\frac{w_2 - w_3}{D_1 - D_2}\right)\left(6 - D_2\right)\right] + w_3 \tag{59}$$

where

w_2 is the cumulative percentage for the point immediately greater than 6 µm;

w_3 is the cumulative percentage for the point immediately below 6 µm;

D_1 is the equivalent particle diameter immediately greater than 6 µm, determined in Equation (57);

D_2 is the equivalent particle diameter immediately below 6 µm, determined in Equation (57).

For an example of calculation for particles below 6 µm in equivalent spherical diameter, see Clause C.2.