INTERNATIONAL STANDARD

ISO 10414-2

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Petroleum and natural gas industries — Field testing of drilling fluids —

Part 2: Oil-based fluids

Industries du pétrole et du gaz naturel — Essais in situ des fluides de forage —

Partie 2: Fluides à base d'huiles

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Contents

Page

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 3.

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 part of ISO 10414 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 10414-2 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 cements*.

ISO 10414 consists of the following parts, under the general title *Petroleum and natural gas industries — Field testing of drilling fluids*:

— *Part 1: Water-based fluids*

— *Part 2: Oil-based fluids*

Annexes A to J of this part of ISO 10414 are for information only.

Introduction

This part of ISO 10414 is based on API RP 13B-2, third edition, February 1998.

As with any laboratory procedure requiring the use of potentially hazardous chemicals, the user is expected to have received proper knowledge and training in the use and disposal of these chemicals. The user is responsible for compliance with all applicable local, regional, and national requirements for worker and local health, safety and environmental liability.

In this part of ISO 10414, where practical, US customary units are included in brackets for information.

Petroleum and natural gas industries — Field testing of drilling fluids —

Part 2: Oil-based fluids

1 Scope

This part of ISO 10414 provides standard procedures for determining the following characteristics of oil-based drilling fluids:

a) drilling fluid density (mud weight);

b) viscosity and gel strength;

c) filtration;

d) oil, water and solids contents;

e) alkalinity, chloride content and calcium content;

f) electrical stability;

g) lime and calcium contents, calcium chloride and sodium chloride contents;

h) low-gravity solids and weighting material contents.

Annexes A, B, C, D, H and I provide additional test methods that may optionally be used for the determination of

i) shear strength,

j) oil and water contents from cuttings,

k) drilling fluid activity,

l) aniline point,

m) cuttings activity,

n) active sulfides.

Annexes F, G and J provide procedures that may optionally be used for

o) sampling, inspection and rejection,

p) rig-site sampling,

q) calibration and verification of glassware, thermometers, viscometers, retort kit cups and drilling fluid balances.

Annex E provides examples of calculations for

r) lime, salinity and solids content.

2 Term and definition

For the purposes of this part of ISO 10414, the following term and definition applies:

2.1
ACS reagent grade
grade of chemical meeting the purity standards specified by the American Chemical Society (ACS)

3 Abbreviated terms

ACS American Chemical Society

CAS Chemical Abstracts Service

EDTA ethylenediaminetetraacetic acid

ES electrical stability

HT/HP high temperature, high pressure

OCMA Oilfield Chemical Manufacturer's Association

PNP propylene glycol normal-propyl ether

PTFE polytetrafluoroethylene

TC to contain

TD to deliver

USC United States Customary (units)

4 Determination of drilling fluid density (mud weight)

4.1 Principle

A procedure is given for determining the mass of a given volume of liquid (= density). The density of drilling fluid is expressed as grams per cubic centimetre, or kilograms per cubic metre.

4.2 Apparatus

4.2.1 Any **density-measuring instrument** having an accuracy of ± 0,01 g/cm^3 or 10 kg/m^3.

The mud balance is the instrument generally used for drilling fluid density determinations. The mud balance is designed such that the drilling fluid holding cup, at one end of the beam, is balanced by a fixed counterweight at the other end, with a sliding-weight rider free to move along a graduated scale. A level-bubble is mounted on the beam to allow for accurate balancing. Attachments for extending the range of the balance may be used when necessary.

The instrument should be calibrated frequently with fresh water. Fresh water should give a reading of 1,00 g/cm^3 or 1 000 kg/m^3 at 21 °C (70 °F). If it does not, adjust the balancing screw or the amount of lead shot in the well at the end of the graduated arm as required.

4.2.2 **Thermometer**, with a range of 0 °C to 105 °C (32 °F to 220 °F).

4.3 Procedure

4.3.1 The instrument base should be set on a flat, level surface.

4.3.2 Measure the temperature of the drilling fluid and record.

4.3.3 Fill the clean, dry cup with drilling fluid to be tested; put the cap on the filled drilling-fluid holding cup and rotate the cap until it is firmly seated. Ensure that some of the drilling fluid is expelled through the hole in the cap, in order to free any trapped air or gas.

4.3.4 Holding the cap firmly on the drilling-fluid holding cup (with cap hole covered), wash or wipe the outside of the cup clean and dry.

4.3.5 Place the beam on the base support and balance it by moving the rider along the graduated scale. Balance is achieved when the bubble is under the centreline.

4.3.6 Read the drilling fluid density at the edge of the rider toward the drilling-fluid cup. Make appropriate corrections when a range extender is used.

4.4 Calculation

4.4.1 Report the drilling fluid density, ρ_s, to the nearest 0,01 g/cm^3 or 10 kg/m^3.

4.4.2 To convert the reading to other units, use the following:

$$\rho_s = 1\,000 \times \text{g/cm}^3 \quad (1)$$

$$\rho_s = 16 \times \text{lb/ft}^3 \quad (2)$$

$$\rho_s = 119{,}8 \times \text{lb/US gal} \quad (3)$$

where ρ_s is the density, expressed in kilograms per cubic metre.

$$\nabla\rho_s = 9{,}81 \times \text{g/cm}^3 \quad (4)$$

$$\nabla\rho_s = 0{,}0226 \times \text{psi/1 000 ft} \quad (5)$$

where $\nabla\rho_s$ is the drilling fluid density gradient, expressed in kilopascals per metre.

A list of density conversions from SI to USC units is given in Table 1.

Table 1 — Density conversions between SI and USC units

Grams per cubic centimetre[a] g/cm^3	Kilograms per cubic metre kg/m^3	Pounds per US gallon (lb/US gal)	Pounds per cubic foot (lb/ft^3)
0,70	700	5,8	43,6
0,80	800	6,7	49,8
0,90	900	7,5	56,1
1,00	1 000	8,345[b]	62,3
1,10	1 100	9,2	68,5
1,20	1 200	10,0	74,8
1,30	1 300	10,9	81,0
1,40	1 400	11,7	87,2
1,50	1 500	12,5	93,5
1,60	1 600	13,4	99,7
1,70	1 700	14,2	105,9
1,80	1 800	15,0	112,1
1,90	1 900	15,9	118,4
2,00	2 000	16,7	124,6
2,10	2 100	17,5	130,8
2,20	2 200	18,4	137,1
2,30	2 300	19,2	143,3
2,40	2 400	20,0	149,5
2,50	2 500	20,9	155,8
2,60	2 600	21,7	162,0
2,70	2 700	22,5	168,2
2,80	2 800	23,4	174,4
2,90	2 900	24,2	180,7

[a] Same value as relative density.

[b] Accurate conversion factor.

5 Alternative method for determination of drilling fluid density

5.1 Principle

The pressurized mud balance provides a more accurate method for determining the density of a drilling fluid containing entrained air or gas than does the conventional mud balance. The pressurized mud balance is similar in operation to the conventional mud balance, the difference being that the slurry sample is placed in a fixed-volume sample cup under pressure.

The purpose of placing the sample under pressure is to minimize the effect of entrained air or gas upon slurry density measurements. By pressurizing the sample cup, any entrained air or gas is decreased to a negligible volume, thus providing a slurry density measurement more closely in agreement with that obtained under downhole conditions.

5.2 Apparatus

5.2.1 Any **density-measuring instrument** having an accuracy of ± 0,01 g/cm^3 or 10 kg/m^3.

The pressurized mud balance is the instrument generally used for density determinations of pressurized drilling fluids. The pressurized mud balance is designed such that the drilling-fluid holding cup and screw-on lid, at one end of the beam, is balanced by a fixed counterweight at the other end, with a sliding-weight rider free to move along a graduated scale. A level-bubble is mounted on the beam to allow for accurate balancing.

Calibrate the instrument frequently with fresh water. Fresh water should give a reading of 1,0 g/cm^3 or 1 000 kg/m^3 at 21 °C (69,8 °F). If it does not, adjust the balancing screw or the amount of lead shot in the well at the end of the graduated arm as required.

5.2.2 **Thermometer**, with a range of 0 °C to 105 °C (32 °F to 220 °F).

5.3 Procedure

5.3.1 Measure the temperature of the drilling fluid and record.

5.3.2 Fill the sample cup to a level slightly (approximately 6 mm) below the upper edge of the cup.

5.3.3 Place the lid on the cup with the attached check-valve in the down (open) position. Push the lid downward into the mouth of the cup until surface contact is made between the outer skirt of the lid and the upper edge of the cup. Any excess slurry will be expelled through the check-valve. When the lid has been placed on the cup, pull the check-valve up into the closed position, rinse off the cup and threads with water, and screw the threaded cap on the cup.

5.3.4 The pressurizing plunger is similar in operation to a syringe. Fill the plunger by submersing its end in the slurry with the piston rod completely inside. Then draw the piston rod upward, thereby filling the cylinder with slurry. This volume should be expelled with the plunger action and refilled with fresh slurry sample to ensure that this plunger volume is not diluted with liquid remaining from the last clean-up of the plunger mechanism.

5.3.5 Push the nose of the plunger onto the mating O-ring surface of the cap valve. Pressurize the sample cup by maintaining a downward force on the cylinder housing in order to hold the check-valve down (open) and at the same time to force the piston rod inside. A force of approximately 225 N (50 lbf) or greater should be maintained on the piston rod.

5.3.6 The check-valve in the lid is pressure-actuated; when the inside of the cup is pressurized, the check-valve is pushed upward into the closed position. To close the valve gradually ease up on the cylinder housing while maintaining pressure on the piston rod. When the check-valve closes, release pressure on the piston rod before disconnecting the plunger.

5.3.7 The pressurized slurry sample is now ready for weighing. Rinse the exterior of the cup and wipe dry. Place instrument on the knife edge. Move the sliding weight right or left until the beam is balanced. The beam is balanced when the attached bubble is centred between the two black marks. Read the density from one of the four calibrated scales on the arrow side of the sliding weight. The density can be read directly in units of grams per cubic centimetre, pounds per gallon, and pounds per cubic foot, or as a drilling fluid gradient in pounds per square inch per 1 000 feet.

5.3.8 To release the pressure inside the cup, reconnect the empty plunger assembly and push downward on the cylinder housing.

5.3.9 Clean the cup and rinse thoroughly with base oil.

5.4 Calculation

Report the drilling fluid density to the nearest 0,01 g/cm^3 or 10 kg/m^3.

For conversions, use the formulas given in 4.4.2.

6 Viscosity and gel strength

6.1 Principle

Viscosity and gel strength are measurements that relate to the flow properties (rheology) of drilling fluids. The following instruments are used to measure viscosity and/or gel strength of drilling fluids:

a) Marsh funnel — a simple device for indicating viscosity on a routine basis;

b) direct-indicating viscometer — a mechanical device for measurement of viscosity at varying shear rates.

NOTE Information on the rheology of drilling fluids can be found in API RP 13D.

6.2 Determination of viscosity using the Marsh funnel

6.2.1 Apparatus

6.2.1.1 **Marsh funnel**, calibrated to deliver 946 ml (1 quart) of fresh water at a temperature of (21 ± 3) °C [(70 ± 5) °F] in (26 ± 0,5) s, with a graduated cup as a receiver.

The Marsh funnel shall have the following characteristics:

a) **funnel cone**, length 305 mm (12,0 in), diameter 152 mm (6,0 in) and a capacity to bottom of screen of 1 500 ml (1,6 quarts);

b) **orifice**, length 50,8 mm (2,0 in) and inside diameter 4,7 mm (0,185 in);

c) **screen**, with 1,6 mm (0,063 in) openings (12 mesh); fixed at 19,0 mm (0,748 in) below top of funnel.

6.2.1.2 **Graduated cup**, with capacity at least 946 ml (1 quart).

6.2.1.3 **Stopwatch**.

6.2.1.4 **Thermometer**, with a range of 0 °C to 105 °C (32 °F to 220 °F).

6.2.2 Procedure

6.2.2.1 Cover the funnel orifice with a finger and pour freshly sampled drilling fluid through the screen into the clean, upright funnel. Fill until fluid reaches the bottom of the screen.

6.2.2.2 Remove finger and start the stopwatch. Measure the time for drilling fluid to fill to the 946 ml (1 quart) mark of the cup.

6.2.2.3 Measure the temperature of the fluid, in degrees Celsius (degrees Fahrenheit).

6.2.2.4 Report the time (6.2.2.2), to the nearest second, with the volume, as the Marsh funnel viscosity. Report the temperature (6.2.2.3) of the fluid to the nearest degree Celsius (degree Fahrenheit).

6.3 Determination of viscosity and/or gel strength using a direct-indicating viscometer

6.3.1 Apparatus

6.3.1.1 **Direct-indicating viscometer,** powered by an electric motor or a hand crank.

Drilling fluid is placed in the annular space between two concentric cylinders. The outer cylinder or rotor sleeve is driven at a constant rotational velocity. The rotation of the rotor sleeve in the fluid produces a torque on the inner cylinder or bob. A torsion spring restrains the movement of the bob, and a dial attached to the bob indicates

displacement of the bob. Instrument constants should be adjusted so that plastic viscosity and yield point are obtained by using readings from rotor sleeve speeds of 300 r/min and 600 r/min.

The components shall meet the following specifications.

a) **Rotor sleeve**

Inside diameter	36,83 mm (1,450 in)
Total length:	87,0 mm (3,425 in)
Scribed line:	58,4 mm (2,30 in) above the bottom of sleeve, with two rows of 3,18 mm (0,125 in) holes spaced 120° (2,09 rad) apart, around rotor sleeve just below scribed line.

b) **Bob**, closed, with flat base and tapered top

Diameter:	34,49 mm (1,358 in)
Cylinder length:	38,0 mm (1,496 in)

c) **Torsion spring constant:**

386 dyne-cm/degree deflection

d) **Rotor sleeve speeds**

High speed:	600 r/min
Low speed:	300 r/min

NOTE Other rotor speeds are available in viscometers from various manufacturers.

6.3.1.2 Stopwatch.

6.3.1.3 Thermostatically controlled viscometer cup.

6.3.1.4 Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F).

6.3.2 Procedure

6.3.2.1 Place a sample of the drilling fluid in a thermostatically controlled viscometer cup. Leave enough empty volume (approximately 100 ml) in the cup for displacement of fluid due to the viscometer bob and sleeve. Immerse the rotor sleeve exactly to the scribed line. Measurements in the field should be made with minimum delay from the time of drilling fluid sampling. Testing should be carried out at either (50 ± 1) °C [(120 ± 2) °F] or (65 ± 1) °C [(150 ± 2) °F]. The place of sampling should be stated on the report.

The maximum recommended operating temperature is 90 °C (200 °F). If fluids have to be tested above this temperature, either a solid metal bob, or a hollow metal bob with a completely dry interior should be used.

WARNING — Liquid trapped inside a hollow bob may vaporize when immersed in high-temperature fluid and cause the bob to explode.

6.3.2.2 Heat (or cool) the sample to the selected temperature. Use intermittent or constant shear at 600 r/min to stir the sample while heating (or cooling) to obtain a uniform sample temperature. After the cup temperature reaches the selected temperature, immerse the thermometer into the sample and continue stirring until the sample reaches the selected temperature. Record the temperature of the sample.

6.3.2.3 With the sleeve rotating at 600 r/min, wait for the viscometer dial reading to reach a steady value (the time required is dependent on the drilling fluid characteristics). Record the dial reading R_{600} in pascals for 600 r/min.

6.3.2.4 Reduce the rotor speed to 300 r/min and wait for the dial reading to reach steady value. Record the dial reading R_{300} in pascals for 300 r/min.

6.3.2.5 Stir the drilling fluid sample for 10 s at 600 r/min.

6.3.2.6 Allow drilling fluid sample to stand undisturbed for 10 s. Slowly and steadily turn the hand-wheel in the appropriate direction to produce a positive dial reading. Record the maximum reading as the initial gel strength. For instruments having a 3 r/min speed, the maximum reading attained after starting rotation at 3 r/min is the initial gel strength. Record the initial gel strength (10-second gel) in pascals (pounds per 100 square feet).

NOTE To convert the dial reading to pounds per 100 square feet: 1 Pa = 0,48 lb/100 ft^2.

6.3.2.7 Restir the drilling fluid sample at 600 r/min for 10 s and then allow the drilling fluid to stand undisturbed for 10 min. Repeat the measurements as in 6.3.2.6 and report the maximum reading as the 10-minute gel in pascals (pounds per 100 square feet).

NOTE To convert the dial reading to pounds per 100 square feet: 1 Pa = 0,48 lb/100 ft^2.

6.3.3 Calculation

$$\eta_P = R_{600} - R_{300} \quad (6)$$

$$YP = 0{,}48 \times (R_{300} - \eta_P) \quad (7)$$

$$\eta_A = R_{600}/2 \quad (8)$$

where

η_P is the plastic viscosity, in millipascal seconds;

NOTE Plastic viscosity is commonly known in the industry by the abbreviation PV.

YP is the yield point, in pascals;

η_A is the apparent viscosity, in millipascal seconds;

R_{600} is the dial reading at 600 r/min, in pascals (pounds per 100 square feet);

R_{300} is the dial reading at 300 r/min, in pascals (pounds per 100 square feet).

NOTE 1 To convert to CGS units of centipoise, 1 mPa·s = 1 cP.

NOTE 2 When calculating values in USC units, the yield point (in pounds per 100 square feet) is calculated as follows:

$$YP = R_{300} - \eta_P$$

7 Filtration

7.1 Principle

7.1.1 Measurement of the filtration behaviour and the filter cake characteristics of an oil-based drilling fluid are fundamental to the treatment and control of a drilling fluid, as are the characteristics of the filtrate, such as the oil, water or emulsion content.

7.1.2 Filtration characteristics of an oil-based drilling fluid are affected by the quantity, type and size of solid particles and emulsified water in the drilling fluid, and by properties of the liquid phase. Interactions of these various components can be influenced by temperature and pressure.

7.1.3 Filtration tests are performed at ambient (low) temperature and at high-temperature conditions to provide data for comparison purposes. Two filtration procedures are given: one for testing up to 175 °C (350 °F) and one for testing from 175 °C (350 °F) to 230 °C (450 °F). Use only the filtration equipment and procedure specified for the temperature required.

NOTE No low-temperature filtration test procedure for oil-based drilling fluids is specified herein, but it can be performed much like the water-based drilling fluid test provided in ISO 10414-1.

7.1.4 Either the 175 ml, 250 ml, or 500 ml unit can be used for testing filtration up to and including 175 °C (350 °F). For testing above 175 °C (350 °F), only the 500 ml unit shall be used. It shall be equipped with a thermocouple to measure the temperature of drilling fluid in the cell, and it shall use a porous filter media.

7.2 High temperature/high pressure test up to 175 °C (350 °F)

7.2.1 Apparatus

7.2.1.1 **High-temperature/high-pressure filter press**, consisting of:

a) **filter cell**, to contain working pressures up to 9 000 kPa (1 300 psi) at temperature;

b) **pressurized gas source**, such as carbon dioxide or nitrogen, with regulators;

NOTE Nitrogen is preferred.

c) **heating system**, to heat to 175 °C (350 °F);

d) **high-pressure filtrate collection vessel**, maintained at proper back-pressure (see Table 2) to avoid flashing or evaporation of the filtrate;

e) **filter cell**, containing a thermometer well, fitted with a removable end, a filter-media support and with oil-resistant seals.

NOTE Valve stems on each end of the cell can be opened or closed during the test.

WARNING — Not all manufacturers' equipment is capable of achieving the same temperatures and pressures. Rigid adherence to manufacturer's recommendations as to sample volumes, temperatures and pressures is essential. Failure to do so could result in serious injury.

Do not use nitrous oxide cartridges as pressure sources for HT/HP filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous materials. Nitrous oxide cartridges shall be used only for Garrett gas train carbonate analysis (see annex I)**.**