

DIN EN ISO 10414-2

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**Erdöl- und Erdgasindustrie –
Feldprüfung von Bohrflüssigkeiten –
Teil 2: Flüssigkeiten auf Ölbasis (ISO 10414-2:2011);
Englische Fassung EN ISO 10414-2:2011, nur auf CD-ROM**

Petroleum and natural gas industries –
Field testing of drillings fluids –
Part 2: Oil-based fluids (ISO 10414-2:2011);
English version EN ISO 10414-2:2011, only on CD-ROM

Industries du pétrole et du gaz naturel –
Essais in situ des fluides de forage –
Partie 2: Fluides à base d'huiles (ISO 10414-2:2011);
Version anglaise EN ISO 10414-2:2011, seulement en CD-ROM

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Nationales Vorwort

Dieses Dokument (EN ISO 10414-2:2011) wurde vom ISO/TC 67 „Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries“, Subkomitee SC 3 „Drilling and completion fluids, and well cements“ in Zusammenarbeit mit dem Technischen Komitee CEN/TC 12 „Materialien, Ausrüstungen und Offshore-Bauwerke für die Erdöl-, petrochemische und Erdgasindustrie“ (Sekretariat: AFNOR, Frankreich) erarbeitet.

Für Deutschland hat hieran der Arbeitskreis NA 109-00-01-03 AK „Bohrspülung und Zemente — Spiegelausschuss zu ISO/TC 67/SC 3“ im Normenausschuss Erdöl- und Erdgasgewinnung (NÖG) des DIN Deutsches Institut für Normung e. V. mitgearbeitet.

Diese Europäische Norm enthält unter Berücksichtigung des DIN-Präsidialbeschlusses 1/2004 nur die englische Originalfassung der ISO Norm.

Diese Norm enthält neben den gesetzlichen Einheiten auch die Einheiten „°F“, „bbl“, „ft“, „gal“, „in“, „lb“, „lb/ft²“, „lb/ft³“, „lb/gal“, „lbf“, „psi“, und „r/min“, die im Deutschen Normenwerk nicht zugelassen sind. Es wird ausdrücklich darauf hingewiesen, dass die Anwendung dieser Einheiten im nationalen amtlichen und geschäftlichen Verkehr aufgrund des Gesetzes über Einheiten im Messwesen nicht zulässig ist.

Umrechnung:

Nicht-SI-Einheit	SI-Einheit	Umrechnung	
°F	°C	°C	= (5/9) (°F-32)
bbl	dm ³	1 bbl	= 158,987 dm ³
ft	m	1 ft	= 0,304 8 m
gal (galone)	l	1 gal	= 3,785 l
in (inch)	mm	1 in	= 25,4 mm
lb (pound)	kg	1 lb	= 0,453 592 37 kg
lb/ft ²	kg/m ²	1 lb/ft ²	= 4,882 kg/m ²
lb/ft ³	kg/m ³	1 lb/ft ³	= 4,882 kg/m ³
lb/gal	kg/m ³	1 lb/gal	= 0,118 kg/dm ³
lbf	N	1 lbf	= 4,448 222 N
psi (psig)	kPa	1 psi	= 6,894 757 kPa
r/min	min ⁻¹	1 r/min	= 1 min ⁻¹

Für die in diesem Dokument zitierten Internationalen Normen wird im Folgenden auf die entsprechenden Deutschen Normen hingewiesen:

ISO 10414-1 siehe DIN EN ISO 10414-1
ISO 13501 siehe DIN EN ISO 13501

ISO 10414, *Petroleum and natural gas industries — Field testing of drilling fluids* besteht aus den folgenden Teilen:

— *Part 1: Water-based fluids*

— *Part 2: Oil-based fluids*

Nationaler Anhang NA (informativ)

Begriffe, Symbole und Abkürzungen

Die Benennung der folgenden Begriffe, Symbole und Abkürzungen ist identisch mit der Benennung in der englischen Fassung.

3 Begriffe

Für die Anwendung dieses Dokuments gelten die folgenden Begriffe.

3.1

Analysenreinheit nach ACS

Grad der Chemikalie, die den von der American Chemical Society (ACS) festgelegten Reinheitsstandards entspricht und in der Datenbank des Chemical Abstracts Service (CAS) aufgelistet ist

3.2

Dichte des Wassers

Dichte von 1 g/ml (8,334 lb/gal) für entionisiertes oder destilliertes Wasser und 1 g/ml (8,345 lb/gal) für reines Leitungswasser

ANMERKUNG Für die Kalibrierung sämtlicher Geräte wird entionisiertes oder destilliertes Wasser verwendet. Leitungswasser ist für chemische Verfahren geeignet. Für die Anwendung des vorliegenden Teils der ISO 10414 entspricht das Volumen von 1 kg Wasser 1 l, und das Volumen des Wasser entspricht numerisch dem Volumen des Wassers, das in Kubikzentimetern oder –millimetern gemessen wird, d. h. 1 g = 1 ml.

3.3

Filtrationsverlust

Flüssigkeitsvolumen, das durch das Filtriermittel hindurchgeht, bevor sich ein Filterkuchen bildet

3.4

Pound

gebräuchliche US-amerikanische Einheit der Masse in Pound (Masse), im Gegensatz zur Einheit der Kraft in Pound (Scher-/Schubspannung)

3.5

volumenbezogene Masse

dimensionsloses Verhältnis der Masse eines Volumens einer Objektschubstanz zur Masse desselben Volumens einer Vergleichsschubstanz, d. h. das Verhältnis der jeweiligen Dichten zueinander

ANMERKUNG 1 Im Allgemeinen ist die Vergleichsschubstanz reines Wasser.

ANMERKUNG 2 Volumenbezogene Masse ist gewöhnlich bekannt als relative Dichte.

4 Symbole und Abkürzungen

4.1 Symbole

a_{DF}	Maß des chemischen Potentials oder des chemischen Reaktionsvermögens der Bohrflüssigkeit
a_W	Maß des chemischen Potentials oder des chemischen Reaktionsvermögens der Wasserlösungen von Standardsalzen
a_C	Maß des chemischen Potentials oder des chemischen Reaktionsvermögens von erbohrtem Bohrklein
b	Anstieg der Ringraumgeschwindigkeit und der Scherspannung an der Wand in einer laminaren Strömung, wie in O.7.2.8 festgelegt
B_{VSST}	Menge des sich abgesetzten Beschwerungsmaterials, angegeben in Pounds (Masse) je Gallone ³⁾
C	Korrekturwert, der zum Ablesewert des Thermometers zu addieren ist
$c_{Ca^{+2},DF}$	Calciumkonzentration der Bohrflüssigkeit insgesamt, angegeben in Milligramm je Liter
c_{Ca^{+2},H_2O}	Calciumkonzentration der Wasserphase je Volumen reines Wasser, angegeben in Milligramm je Liter
$c_{CaCl_2,AQ}$	Calciumchloridkonzentration der Wasserphase, angegeben in Milligramm je Liter
$c_{CaCl_2,DF,A}$	Calciumchloridkonzentration der Bohrflüssigkeit insgesamt, angegeben in Milligramm je Liter
$c_{CaCl_2,DF,B}$	Calciumchloridkonzentration der Bohrflüssigkeit insgesamt, angegeben in Pounds je Barrel
$c_{CaCl_2,DF,C}$	Calciumchloridkonzentration der Bohrflüssigkeit insgesamt, angegeben in Kilogramm je Kubikmeter
$c_{Ca(OH)_2,\%}$	Feingehaltswert von Kalk, angegeben als ein Massenanteil
$c_{Ca(OH)_2,DF,B}$	Gesamt-Kalkkonzentration der Bohrflüssigkeit insgesamt, angegeben in Pounds je Barrel
$c_{Ca(OH)_2,DF,C}$	Gesamt-Kalkkonzentration der Bohrflüssigkeit insgesamt, angegeben in Kilogramm je Kubikmeter
$c_{Ca(OH)_2,F}$	Kalkkonzentration angegeben in Kilogramm je Kubikmeter oder in Pounds je Barrel
$c_{Cl^-,DF}$	Chloridkonzentration der Bohrflüssigkeit insgesamt, in Milligramm je Liter

3) Die Verwendung von Gallone bezieht sich im gesamten vorliegenden Teil der ISO 10414 auf die US-amerikanische Gallone, die 3,7854 Liter entspricht.

$c_{\text{Cl}^- (\text{CaCl}_2), \text{DF}}$	Chloridkonzentration der Bohrflüssigkeit insgesamt als Calciumchlorid, angegeben in Milligramm je Liter
$c_{\text{Cl}^- (\text{NaCl}), \text{DF}}$	Chloridkonzentration der Bohrflüssigkeit insgesamt aus Natriumchlorid, angegeben in Milligramm je Liter
$c_{\text{NaCl}, \text{AQ}}$	Natriumchloridkonzentration der Wasserphase, angegeben in Milligramm je Liter
$c_{\text{LG}, \text{B}}$	Konzentration der Leicht-Feststoffe, angegeben in Pounds je Barrel
$c_{\text{LG}, \text{C}}$	Konzentration der Leicht-Feststoffe, angegeben in Kilogramm je Kubikmeter
$c_{\text{NaCl}, \text{DF}, \text{A}}$	Natriumchloridkonzentration der Bohrflüssigkeit insgesamt, angegeben in Milligramm je Liter
$c_{\text{NaCl}, \text{DF}, \text{B}}$	Natriumchloridkonzentration der Bohrflüssigkeit insgesamt, angegeben in Pounds je Barrel
$c_{\text{NaCl}, \text{DF}, \text{C}}$	Natriumchloridkonzentration der Bohrflüssigkeit insgesamt, angegeben in Kilogramm je Kubikmeter
$c_{\text{NaCl}, \text{DF}, \text{INSOL}, \text{A}}$	Konzentration des unlöslichen Natriumchlorids der Bohrflüssigkeit insgesamt, angegeben in Milligramm Liter
$c_{\text{NaCl}, \text{DF}, \text{INSOL}, \text{B}}$	Konzentration des unlöslichen Natriumchlorids der Bohrflüssigkeit insgesamt, angegeben in Pounds je Barrel
$c_{\text{NaCl}, \text{DF}, \text{SOL}, \text{A}}$	Konzentration des löslichen Natriumchlorids der Bohrflüssigkeit insgesamt, angegeben in Milligramm Liter
$c_{\text{NaCl}, \text{DF}, \text{SOL}, \text{B}}$	Konzentration des löslichen Natriumchlorids der Bohrflüssigkeit insgesamt, angegeben in Pounds je Barrel
$c_{\text{NaCl}, \text{DF}, \text{SOL}, \text{C}}$	Konzentration des löslichen Natriumchlorids der Bohrflüssigkeit insgesamt, angegeben in Kilogramm je Kubikmeter
$c_{\text{S}-2}$	Konzentration der aktiven Sulfide, angegeben in Milligramm je Liter
$c_{\text{WM}, \text{B}}$	Konzentration von Beschwerungsmaterial, angegeben in Pounds je Barrel
$c_{\text{WM}, \text{C}}$	Konzentration von Beschwerungsmaterial, angegeben in Kilogramm je Kubikmeter
d_1	Abstand zur Außenwand, angegeben in Zoll
D	Außenrohrdurchmesser oder Innendurchmesser eines offenen Bohrlochs, angegeben in Zoll
d	Innenrohrdurchmesser, angegeben in Zoll
D_{TVD}	Seigerteufe, angegeben in Fuß
E	Pumpenwirkungsgrad, angegeben in Prozent
f	Rohrfaktor, aus Tabelle I.2
G'	Speichermodul, angegeben in Newton je Quadratmeter
G''	Verlustmodul, angegeben in Newton je Quadratmeter

k_C	Konsistenzzahl, angegeben in Pounds (Kraft) je Sekunde
L	Länge des hydraulischen Abschnitts, angegeben in Fuß
L_A	Länge, angegeben in Fuß
l	Eingetauchte Länge des Scherrohrs, angegeben in Zentimeter
l_A	Eingetauchte Länge des Scherrohrs, angegeben in Zoll
l_D	Eingefärbte Länge (Farblänge) des Träger-Röhrchens, in Maßeinheiten auf dem Röhrchen gekennzeichnet
m_1	Masse des leeren Retortenaufbaus (Behälter, Deckel und Grundkörper gefüllt mit Stahlwolle), angegeben in Gramm
m_2	Masse des befüllten Retortenaufbaus (Behälter mit Probe, Deckel und Grundkörper gefüllt mit Stahlwolle), angegeben in Gramm
m_3	Masse des trockenen, leeren Behälters zum Auffangen der Flüssigkeit, angegeben in Gramm
m_4	Masse des gekühlten Behälters zum Auffangen der Flüssigkeit einschließlich kondensierter Flüssigkeiten, angegeben in Gramm
m_5	Masse des gekühlten Retortenaufbaus (Grundkörper gefüllt mit Stahlwolle), angegeben in Gramm
m_d	Masse des getrockneten Bohrkleins in der Retorte, angegeben in Gramm
m_{F1}	Masse der Bohrflüssigkeit nach Scherung bei 100 U/min, angegeben in Gramm
m_{F2}	dem Senkschuh entnommene Masse der Bohrflüssigkeit nach Scherung bei 100 U/min, angegeben in Gramm
m_{F3}	dem Senkschuh entnommene Masse der Bohrflüssigkeit nach Scherung bei 600 U/min, angegeben in Gramm
m_L	Masse der kondensierten Flüssigkeit (Öl und Wasser), angegeben in Gramm
m_O	Masse des Öls, angegeben in Gramm
m_S	Masse der Bohrflüssigkeitsprobe im flüssigen Zustand, angegeben in Gramm
m_{st}	Masse des Scherrohrs, angegeben in Gramm
m_{tot}	Gesamte Schermasse (Summe der Plattform und der Gewichte), angegeben in Gramm
m_{WC}	Masse des feuchten Bohrkleins, angegeben in Gramm
m_W	Masse des Wassers, angegeben in Gramm
P	gemessener Druck, angegeben in Überdruck je Quadratzoll
ΔP	erwarteter Druckanstieg, angegeben in Überdruck je Quadratzoll

$\frac{\Delta P}{\Delta L_A}$	Druckgefälle, angegeben in Überdruck je Quadrat Zoll je Fuß
Q	Pumpenfördermenge, angegeben in Gallonen je Minute
R_1	mittlere Ablesung am Standardthermometer, angegeben in Grad
R_2	mittlere Ablesung am Arbeitsthermometer, angegeben in Grad
R_{600}	Ablesung des Abweichungswertes bei 600 Umdrehungen je Minute, angegeben in Grad der Abweichung
R_{300}	Ablesung des Abweichungswertes bei 300 Umdrehungen je Minute, angegeben in Grad der Abweichung
R_{BPU}	berechnetes Verhältnis der Schichtabtragungsmessung, angegeben in Prozent
R_O	Verhältnis des Volumenanteils des Öls zur Summe der Volumenanteile Öl und reines Wasser aus der Retortenanalyse, angegeben als prozentualer Anteil
R_B	Verhältnis des Volumenanteils des Öls zur Summe der Volumenanteile Öl und Sole, angegeben als prozentualer Anteil
R_W	Verhältnis des Volumenanteils des Wassers zur Summe der Volumenanteile Öl und reines Wasser aus der Retortenanalyse, angegeben als prozentualer Anteil
ROC	verbleibendes Öl am Bohrklein, angegeben in Gramm je Kilogramm Bohrklein (entweder feucht oder trocken)
S	Senk-Register (en: Sag Register)
t	Zeitdauer, angegeben in Minuten
V_1	Filtrationsverlust, angegeben in Milliliter
$V_{7,5}$	Filtratvolumen nach 7,5 min, angegeben in Milliliter
V_{30}	Filtratvolumen nach 30 min, angegeben in Milliliter
V_A	Ringvolumen, angegeben in Barrel
V_{AgNO_3}	Volumen von 0,282 mol/l (0,282 N) Silbernitratreagens, angegeben in Milliliter
V_B	Grundbedarf an Alkali
V_{EDTA}	Volumen von 0,1 mol/l EDTA-Lösung, angegeben in Milliliter
V_F	Filtratvolumen
$V_{H_2SO_4}$	Volumen von 0,05 mol/l (0,1 N) Schwefelsäure, angegeben in Milliliter
V_K	Alkalität der Bohrflüssigkeit insgesamt, angegeben in Milliliter von 0,05 mol/l Schwefelsäure
V_M	Volumen des Auffangbehälters an einer bestimmten Markierung, angegeben in Milliliter
V_{NaOH}	Volumen von 0,1 mol/l (0,1 N) NaOH, angegeben in Milliliter

V_O	Volumen des Öls, angegeben in Milliliter
V_{PPT}	PPT-Volumen, angegeben in Milliliter
V_R	Gesamtvolumen der kondensierten Flüssigkeiten (Öl und Wasser), angegeben in Milliliter
V_{RC}	Volumen des Retortenbehälters, angegeben in Milliliter
V_S	Volumen der Bohrflüssigkeitsprobe, angegeben in Milliliter
V_W	Wasservolumen, angegeben in Milliliter, oder Wassermasse, angegeben in Gramm (1 ml = 1 g) (siehe 3.2)
Δv_a	Änderung der Ringraumgeschwindigkeit, angegeben in Fuß je Minute
v_a	Ringraumgeschwindigkeit, angegeben in Fuß je Minute
v_f	statische Filtriergeschwindigkeit (Strömungsgeschwindigkeit), in Milliliter je Minute ^{1/2}
w_{CaCl_2}	Massenanteil des Calciumchlorids in der Wasserphase, angegeben als prozentualer Anteil der gesamten Wasserphasenmasse
$w_{CaCl_2,SAT}$	Massenanteil des Calciumchlorids einer übersättigten Flüssigkeit in der Wasserphase, angegeben als prozentualer Anteil der gesamten Wasserphasenmasse
w_{NaCl}	Massenanteil des Natriumchlorids in der Wasserphase, angegeben als prozentualer Anteil der gesamten Wasserphasenmasse
$w_{NaCl,MAX}$	maximaler Massenanteil des löslichen Natriumchlorids in der Wasserphase, das in einem gegebenen Massenanteil von Calciumchlorid vorhanden sein kann, angegeben als prozentualer Anteil der gesamten Wasserphasenmasse
$w_{NaCl,MAX-C}$	der rückgerechnete Massenanteil von löslichem Natriumchlorid in der Wasserphase, das in einem gegebenen Massenanteil von Calciumchlorid vorhanden sein kann, angegeben als prozentualer Anteil der gesamten Wasserphasenmasse
Y_{PA}	Fließgrenze, angegeben in Pascal
Y_{PB}	Fließgrenze, angegeben in Pounds je einhundert Quadratfuß, häufig angegeben als YP
β_{10m}	Gelstärke nach 10 min, angegeben in Pounds je einhundert Quadratfuß
β_{10s}	Gelstärke nach 10 s, angegeben in Pounds je einhundert Quadratfuß
$\Gamma_{DFG,A}$	Gradient der Bohrflüssigkeit, angegeben in Kilopascal je Meter
$\Gamma_{DFG,B}$	Gradient der Bohrflüssigkeit, angegeben in Pounds je Quadratzoll je Fuß
γ_A	Gradient der Bohrflüssigkeit, angegeben in Pounds je Quadratzoll je Fuß
γ_B	Gradient der Bohrflüssigkeit, angegeben in Kilopascal je Meter
γ_i	Schergeschwindigkeit der Flüssigkeit, angegeben in reziproken Sekunden
η	Drehung des Bohrgestänges, angegeben in Umdrehungen je Minute
η_{AV}	Scheinbare Viskosität, angegeben in Millipascalsekunden (Centipoise)

η_{PV}	Plastische Viskosität, angegeben in Millipascalsekunden (Centipoise)
φ_B	Volumenanteil der Sole, angegeben als prozentualer Anteil der Bohrflüssigkeit insgesamt
φ_D	korrigierter Volumenanteil der Feststoffe, angegeben als prozentualer Anteil der Bohrflüssigkeit insgesamt
φ_d	Volumenanteil der getrockneten Retortenfeststoffe, angegeben als prozentualer Anteil des gesamten Probenvolumens
φ_{LG}	Volumenanteil der Leicht-Feststoffe, angegeben als prozentualer Anteil der suspendierten Feststoffe insgesamt
φ_O	Volumenanteil des Öls, angegeben als prozentualer Anteil der Bohrflüssigkeit insgesamt
φ_W	Volumenanteil des reinen Wassers, angegeben als prozentualer Anteil der Bohrflüssigkeit insgesamt
φ_{WM}	Volumenanteil der Feststoffe des Beschwerungsmaterials, angegeben als prozentualer Anteil der gesamten suspendierten Feststoffe
ρ	Dichte der Bohrflüssigkeit, angegeben in Pounds je Gallone
ρ_B	Dichte der Wasserphase, angegeben in Gramm je Milliliter
ρ_C	Dichte der Bohrflüssigkeit, angegeben in Kilogramm je Kubikmeter
ρ_{B1}	Dichte der Bohrflüssigkeit, angegeben in Pounds je Gallone
ρ_{B2}	Dichte der Bohrflüssigkeit, angegeben in Pounds je Kubikfuß
$\overline{\rho_d}$	durchschnittliche Dichte (volumenbezogene Masse) der suspendierten Feststoffe
$\rho_{ECD-hyd}$	Druckabfall und zusätzliche Auswirkung auf die Dichte des erbohrten Bohrkleins
$\rho_{ECD-tot}$	Erwartete äquivalente Gesamtbohrdichte
$\Delta\rho_{ECD-rot}$	Druckänderung aufgrund von Drehung
ρ_{LG}	Dichte der Leicht-Feststoffe, angegeben in Gramm je Milliliter
ρ_{max}	maximale aufgezeichnete Dichte der Bohrflüssigkeit, angegeben in Pounds je Gallone
ρ_{nom}	Nennndichte der Bohrflüssigkeit, angegeben in Pounds je Gallone
ρ_O	Dichte des verwendeten Öls, angegeben in Gramm je Milliliter
ρ_S	Dichte der Bohrflüssigkeit, angegeben in Gramm je Milliliter
ρ_w	Wasserdichte, angegeben in Gramm je Milliliter, bei Prüftemperatur (siehe Tabelle J.1)
ρ_{WM}	Dichte der Feststoffe des Beschwerungsmaterials, angegeben in Gramm je Milliliter
τ_W	Wandschubspannung, angegeben in Pounds (Kraft) je einhundert Quadratfuß
τ_Y	Fließspannung der Bohrflüssigkeit, angegeben in Pounds (Kraft) je einhundert Quadratfuß

4.2 Abkürzungen

ACS	American Chemical Society
API	American Petroleum Institute
ASTM	American Society for Testing and Materials
AV	Scheinbare Viskosität
CAS	Chemical Abstracting Services
ECD	Äquivalente Zirkulationsdichte (angegeben in Pounds je Gallone)
EDTA	Natriumsalz von Ethylendiamintetraessigsäure-Dihydrat
ERD	Abgelenkte Bohrung
ES	Elektrische Stabilität
ESD	Äquivalente statische Dichte (angegeben in Pounds je Gallone)
HTHP	hohe Temperatur, hoher Druck
OCMA	Oil Company Materials Association
OBR	Verhältnis von Öl zu Sole
OWR	Verhältnis von Öl zu Wasser
PNP	Propylenglycol- <i>n</i> -propylether
PPA	Permeabilitätsblockiergerät/Permeabilitätsmessgerät
PPT	Permeabilitätsblockierprüfung
PTFE	Polytetrafluorethylen (Beispiel: Teflon®)
PV	Plastische Viskosität
PVT	Druck-Volumen-Temperatur-Beziehung
SI	Internationales Einheitensystem
TC	IN (d. h. justiert auf Einguss)
TD	EX (d. h. justiert auf Ablauf)
TVD	Seigerteufe [angegeben in Meter (Fuß)]
USC	United States Customary (im amerikanischen Sprachraum übliche Einheiten)
VSST	Viskosimeter-Senkschuh-Prüfung (en: Viscometer Sag Shoe Test)
YP	Fließgrenze

Nationaler Anhang NB (informativ)

Literaturhinweise

DIN EN ISO 10414-1, *Erdöl- und Erdgasindustrie — Feldprüfung von Bohrspülungen — Teil 1: Flüssigkeiten auf Wasserbasis*

DIN EN ISO 13501, *Erdöl- und Erdgasindustrie — Bohrflüssigkeiten — Beurteilung von Verarbeitungssystemen*

English Version

Petroleum and natural gas industries —
Field testing of drilling fluids —
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This European Standard was approved by CEN on 28 April 2011.

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Contents

	Page
Foreword.....	7
Introduction	8
1 Scope	9
2 Normative references	10
3 Terms and definitions	10
4 Symbols and abbreviated terms	11
4.1 Symbols	11
4.2 Abbreviated terms	16
5 Determination of drilling fluid density (mud weight)	17
5.1 Principle	17
5.2 Apparatus	17
5.3 Procedure	18
5.4 Calculation.....	18
6 Alternative method for determination of drilling fluid density.....	20
6.1 Principle.....	20
6.2 Apparatus	20
6.3 Procedure	21
6.4 Calculation.....	22
7 Viscosity and gel strength	22
7.1 Principle.....	22
7.2 Determination of viscosity using the Marsh funnel	22
7.2.1 Apparatus	22
7.2.2 Procedure	22
7.3 Determination of viscosity and gel strength using a direct-reading viscometer.....	23
7.3.1 Apparatus	23
7.3.2 Procedure	24
7.3.3 Calculation.....	24
8 Static filtration.....	26
8.1 Principle.....	26
8.2 High-temperature/high-pressure test up to 175 °C (350 °F)	26
8.2.1 Apparatus	26
8.2.2 Procedure	27
8.2.3 Calculation.....	29
8.3 High-temperature/high-pressure test 175 °C (350 °F) up to and including 230 °C (450 °F)	29
8.3.1 Apparatus	29
8.3.2 Procedure	30
8.3.3 Calculation.....	31
9 Retort test for oil, water and solids concentrations.....	31
9.1 Principle.....	31
9.2 Apparatus	32
9.2.1 Retort instrument, as specified below.	32
9.3 Procedure — Volumetric method.....	33
9.4 Calculation — Volumetric method	34
9.5 Procedure — Gravimetric method	35
9.6 Calculation — Gravimetric method.....	38
9.7 Calculation — Volume fractions of oil, water and solids	39
10 Chemical analysis of oil-based drilling fluids.....	40
10.1 Principle.....	40
10.2 Reagents and apparatus	42

	Page
10.3 Base alkalinity demand, V_B	42
10.3.1 Procedure	42
10.3.2 Calculation	43
10.4 Whole-drilling-fluid alkalinity, V_K	43
10.4.1 Procedure	43
10.4.2 Calculation	44
10.5 Whole-drilling-fluid chloride concentration	45
10.5.1 Procedure	45
10.5.2 Calculation	46
10.6 Whole-drilling-fluid calcium concentration	46
10.6.1 Procedure	46
11 Electrical stability test	47
11.1 Principle	47
11.2 Apparatus	47
11.3 Equipment calibration/performance test	48
11.4 Procedure	48
12 Lime, salinity and solids calculations	49
12.1 Principle	49
12.2 Apparatus	50
12.3 Whole-drilling-fluid calculations	50
12.3.1 Whole-drilling-fluid lime concentration	50
12.3.2 Whole-drilling-fluid calcium chloride and sodium chloride concentrations	50
12.4 Aqueous phase calculations	52
12.5 Soluble and insoluble whole-drilling-fluid sodium chloride calculations	56
12.6 Calculation — Solids in the whole drilling fluid	57
12.6.1 Corrected solids concentration	57
12.6.2 Calculation — Oil-to-water ratio	58
12.6.3 Calculation — Oil-to-brine ratio	59
12.6.4 Calculation — Solids concentration and density	59
12.6.5 Calculation — Volume fraction and concentration of weighting material and low-gravity solids	60
Annex A (informative) Measurement of shear strength using shearometer tube	62
A.1 Principle	62
A.2 Apparatus	62
A.3 Procedure	62
A.4 Calculation	63
Annex B (informative) Determination of oil and water retained on cuttings	64
B.1 Principle	64
B.2 Apparatus	64
B.3 Procedure	65
B.4 Calculations	67
Annex C (informative) Determination of aqueous-phase activity of emulsified water using an electrohygrometer	69
C.1 Principle	69
C.2 Reagents and apparatus	69
C.2.1 Standard saturated salt solutions	69
C.3 Procedure	70
C.3.1 Calibration of hygrometer	70
C.3.2 Measurement of oil-based drilling fluid activity, a_{DF}	71
C.4 Calculation — Drilling fluid activity, a_{DF}	71
Annex D (informative) Determination of aniline point	72
D.1 Principle	72
D.2 Reagents and apparatus	72
D.3 Procedure	73

Annex E (informative) Lime, salinity and solids calculations.....	75
E.1 Principle.....	75
E.2 Test data used in examples	75
E.3 Drilling fluid containing only CaCl_2 salt (drilling fluid A).....	76
E.4 Drilling fluid containing only NaCl salt (drilling fluid B).....	78
E.5 Drilling fluid containing both CaCl_2 and NaCl salts (drilling fluid C).....	80
E.6 Drilling fluid containing both CaCl_2 and excess NaCl salts (drilling fluid D).....	83
E.7 Calculation in spreadsheet — Drilling fluid A to drilling fluid D.....	86
Annex F (informative) Sampling, inspection and rejection of drilling materials	96
F.1 Principle.....	96
F.2 Sampling of powdered material in packages.....	96
F.3 Sampling powdered material in bulk	96
F.4 Preparation of test sample.....	96
F.5 Test.....	97
F.6 Inspection	97
F.7 Rejection.....	97
F.8 Retesting.....	97
Annex G (informative) Rig-site sampling	98
G.1 Principle.....	98
G.2 Bottom-flow sampling (preferred).....	98
G.2.1 Apparatus	98
G.2.2 Procedure	98
G.3 Grab sampling.....	99
G.3.1 Apparatus	99
G.3.2 Procedure	99
Annex H (informative) Determination of cutting activity by the Chenevert method	101
H.1 Principle.....	101
H.2 Reagents and apparatus	101
H.2.1 Standard saturated salt solutions.....	101
H.3 Procedure	102
H.3.1 Calibration of hygrometer.....	102
H.3.2 Measurement of oil-contaminated cuttings activity, a_C	103
H.4 Calculation — Cuttings activity, a_C	103
Annex I (informative) Chemical analysis of active sulfides by the Garrett gas train method	104
I.1 Principle.....	104
I.2 Reagents and apparatus	104
I.3 Procedure	106
I.4 Calculation.....	108
Annex J (informative) Calibration and verification of glassware, thermometers, viscometers, retort kit cup and drilling fluid balances.....	109
J.1 Principle.....	109
J.2 Calibration of apparatus	109
J.2.1 Volumetric glassware.....	109
J.2.2 Thermometers	109
J.2.3 Viscometers.....	109
J.2.4 Drilling fluid balances	109
J.2.5 Retort kit cups.....	109
J.3 Procedure — Calibration of graduated cylinders.....	110
J.4 Procedure — Calibration of thermometers	110
J.5 Procedure — Calibration of viscometers	112
J.6 Procedure — Calibration of retort kit cups	113
J.7 Procedure — Calibration of filter press gasket and O-rings.....	113

Annex K (informative) High-temperature/high-pressure filtration testing of oil-based drilling fluids using the permeability plugging apparatus and cells equipped with set-screw secured end caps	114
K.1 Principle.....	114
K.2 Safety considerations	114
K.3 Apparatus — Permeability plugging apparatus (PPA) with set-screw-secured end caps	116
K.4 Procedure for high-temperature, high-pressure (HTHP) filtration	118
K.4.1 Preheating the heating jacket	118
K.4.2 Loading the filtration cell.....	118
K.4.3 Pressurizing the cell.....	119
K.4.4 Conducting the filtration test	121
K.5 Test conclusion and disassembly	121
K.6 Calculation — Data reporting	123
K.6.1 Filtrate reporting.....	123
K.6.2 Spurt loss	123
K.6.3 Calculation	123
K.6.4 Filter cake reporting	123
Annex L (informative) High-temperature/high-pressure filtration testing of oil-based drilling fluids using the permeability plugging apparatus and cells equipped with threaded end caps	124
L.1 Principle.....	124
L.2 Safety considerations	124
L.3 Apparatus — Permeability plugging apparatus (PPA) with threaded end caps	126
L.4 Procedure for high-temperature, high-pressure (HTHP) filtration	128
L.4.1 Preheating the heating jacket	128
L.4.2 Loading the filtration cell.....	128
L.4.3 Pressurizing the cell.....	129
L.4.4 Conducting the filtration test	130
L.5 Test conclusion and disassembly	130
L.6 Calculating — Data reporting	132
L.6.1 Filtrate reporting.....	132
L.6.2 Spurt loss	132
L.6.3 Calculation	132
L.6.4 Filter cake reporting	132
Annex M (informative) Compatibility of elastomeric materials with non-aqueous-based drilling fluids	133
M.1 Principle.....	133
M.2 Apparatus and reagents	134
M.2.1 Standard reference elastomers.....	134
M.2.2 Standard reference test liquids.....	134
M.3 Procedure — Test specimen preparation	134
M.4 Procedure — Elastomer physical changes after immersion	134
M.4.1 Principle.....	134
M.4.2 Summary of immersion procedure.....	135
M.4.3 Summary of change in volume procedure.....	135
M.4.4 Summary of changes in tensile strength, elongation and hardness procedures	136
M.5 Procedure — Effect of immersion on a stressed elastomer specimen	136
Annex N (informative) Sand content procedure for non-aqueous fluids	137
N.1 Principle.....	137
N.2 Apparatus	137
N.3 Procedure	137
N.4 Calculation	137
Annex O (informative) Identification and monitoring of weight-material sag	138
O.1 Principle.....	138
O.2 Guidelines on using sag monitoring methods	138

	Page
O.3 Surface monitoring of weight-material sag	139
O.3.1 Principle	139
O.3.2 Apparatus	140
O.3.3 Procedure	140
O.3.4 Calculation	140
O.3.5 Data reporting	141
O.4 Field sag monitoring based on downhole density	143
O.4.1 Principle	143
O.4.2 Measurements of downhole pressure	143
O.4.3 Weight-material signatures using downhole pressure tools	144
O.4.4 Downhole pressure changes that are not caused by weight-material sag	146
O.4.5 Theoretical downhole density excluding effects in O.4.4	149
O.4.6 Weight-material sag events caused by drillpipe rotation only	149
O.5 Dynamic weight-material sag test — VSST method	150
O.5.1 Principle	150
O.5.2 Apparatus	151
O.5.3 Procedure	152
O.5.4 Calculation	153
O.6 Rheological measurements of drilling fluids exhibiting weight-material sag	154
O.6.1 Principle	154
O.6.2 Scope	154
O.6.3 Apparatus	154
O.6.4 Sample preparation	155
O.6.5 Potential rheological tests	155
O.6.6 Data interpretation	156
O.7 Field sag monitoring based on critical wall shear stress	158
O.7.1 Principle	158
O.7.2 Predictive model	158
O.7.3 Comparison of laboratory and field data	160
O.8 Additional resource literature	160
Annex P (informative) Oil-based drilling fluid report form	161
Bibliography	162

Foreword

This document (EN ISO 10414-2:2011) has been prepared by Technical Committee ISO/TC 67 "Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries" in collaboration with Technical Committee CEN/TC 12 "Materials, equipment and offshore structures for petroleum, petrochemical 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 December 2011, and conflicting national standards shall be withdrawn at the latest by December 2011.

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

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of ISO 10414-2:2011 has been approved by CEN as a EN ISO 10414-2:2011 without any modification.

Introduction

This part of ISO 10414 is based on API RP 13B-2:2005, *Recommended practice for field testing of oil-based drilling fluids*.

As with any laboratory procedure requiring the use of potentially hazardous chemicals and equipment, the user is expected to have received proper training and knowledge in the use and disposal of these potentially hazardous materials. 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, quantities expressed in the International System (SI) of units are also, where practical, expressed in United States Customary (USC) units in parentheses for information. The units do not necessarily represent a direct conversion of SI units to USC units, or USC units to SI units. Consideration has been given to the precision of the instrument making the measurement. For example, thermometers are typically marked in one degree increments, thus temperature values have been rounded to the nearest degree.

Calibrating an instrument refers to ensuring the accuracy of the measurement. Accuracy is the degree of conformity of a measurement of a quantity to its actual or true value. Accuracy is related to precision, or reproducibility, of a measurement. Precision is the degree to which further measurements or calculations will show the same or similar results. Precision is characterized in terms of the standard deviation of the measurement. The results of calculations or a measurement can be accurate but not precise, precise but not accurate, neither accurate nor precise, or both accurate and precise. A result is valid if it is both accurate and precise.

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 concentrations;
- e) alkalinity, chloride concentration and calcium concentration;
- f) electrical stability;
- g) lime and calcium concentrations, calcium chloride and sodium chloride concentrations;
- h) low-gravity solids and weighting material concentrations.

The annexes provide additional test methods or examples that can optionally be used for the determination of:

- shear strength (Annex A);
- oil and water concentrations from cuttings (Annex B);
- drilling fluid activity (Annex C);
- aniline point (Annex D);
- lime, salinity and solids concentration (Annex E);
- sampling, inspection and rejection (Annex F);
- rig-site sampling (Annex G);
- cuttings activity (Annex H);
- active sulphides (Annex I);
- calibration and verification of glassware, thermometers, viscometers, retort kit cups and drilling fluid balances (Annex J);
- permeability plugging apparatus with set-screw secured end cap (Annex K);
- permeability plugging apparatus with threaded end cap (Annex L);
- elastomer compatibility (Annex M);
- sand content of oil-based fluid (Annex N);
- identification and monitoring of weight-material sag (Annex O);
- oil-based drilling fluid test report form (Annex P).

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 10414-1:2008¹⁾, *Petroleum and natural gas industries — Field testing of drilling fluids — Part 1: Water-based fluids*

ISO 13501²⁾, *Petroleum and natural gas industries — Drilling fluids — Processing equipment evaluation*

API RP 13D:2010, *Recommended practice on the rheology and hydraulics of oil-well drilling fluids*

3 Terms and definitions

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

3.1
ACS reagent grade
grade of chemical meeting the purity standards specified by the American Chemical Society (ACS) and listed in the Chemical Abstracting Service (CAS)

3.2
density of water
density of 1 g/ml (8,334 lb/gal) for deionized or distilled water and 1 g/ml (8,345 lb/gal) for clean tap water

NOTE Deionized or distilled water is used for all equipment calibration. The volume of 1 kg of water is 1 l for the purposes of this part of ISO 10414, and the volume of water is numerically equivalent to the volume of the water measured in cubic centimetres or millilitres, i.e. 1 g = 1 ml.

3.3
spurt loss
volume of fluid that passes through the filtration medium before a filter cake is formed

3.4
pound
U.S. customary unit used to indicate pound-mass (weight), as opposed to pound-force (shear stress)

3.5
volumic mass
dimensionless ratio of the mass of a volume of an object substance to the mass of the same volume of a reference substance, i.e. the ratio of their respective mass densities

NOTE 1 Generally speaking, the reference substance is pure water.

NOTE 2 Volumic mass is commonly known as specific gravity.

¹⁾ For the purposes of this part of ISO 10414, API RP 13B-1:2009, *Recommended practice for field testing water-based drilling fluids*, is equivalent.

²⁾ For the purposes of this part of ISO 10414, API RP 13C, *Recommended practice on drilling fluids processing systems evaluation*, is equivalent.

4 Symbols and abbreviated terms

4.1 Symbols

a_{DF}	measure of the chemical potential or reaction availability of drilling fluid
a_W	measure of the chemical potential or reaction availability of water solutions of standard salts
a_C	measure of the chemical potential or reaction availability of drilled cuttings
b	slope of the annular velocity and shear stress at the wall in laminar flow, as defined in O.7.2.8
B_{VSST}	amount of weight-material sag, expressed in pounds-mass per gallon ³⁾
C	correction value to add to thermometer reading
$c_{Ca^{+2},DF}$	whole-drilling-fluid calcium concentration, expressed in milligrams per litre
c_{Ca^{+2},H_2O}	aqueous-phase calcium concentration per volume of pure water, expressed in milligrams per litre
$c_{CaCl_2,AQ}$	aqueous-phase calcium chloride concentration, expressed in milligrams per litre
$c_{CaCl_2,DF,A}$	whole-drilling-fluid calcium chloride concentration, expressed in milligrams per litre
$c_{CaCl_2,DF,B}$	whole-drilling-fluid calcium chloride concentration, expressed in pounds per barrel
$c_{CaCl_2,DF,C}$	whole-drilling-fluid calcium chloride concentration, expressed in kilograms per cubic metre
$c_{Ca(OH)_2,\%}$	lime assay value, expressed as a weight fraction
$c_{Ca(OH)_2,DF,B}$	whole-drilling-fluid total lime concentration, expressed in pounds per barrel
$c_{Ca(OH)_2,DF,C}$	whole-drilling-fluid total lime concentration, expressed in kilograms per cubic metre
$c_{Ca(OH)_2,F}$	lime concentration of field lime, expressed in kilograms per cubic metre or pounds per barrel
$c_{Cl^-,DF}$	whole-drilling-fluid chloride concentration, expressed in milligrams per litre
$c_{Cl^-(CaCl_2),DF}$	whole-drilling-fluid chloride concentration as calcium chloride, expressed in milligrams per litre
$c_{Cl^-(NaCl),DF}$	whole-drilling-fluid chloride concentration from sodium chloride, expressed in milligrams per litre
$c_{NaCl,AQ}$	aqueous-phase sodium chloride concentration, expressed in milligrams per litre

³⁾ Gallon as used throughout this part of ISO 10414 refers to the U.S. gallon of 3,785 4 litres.

$c_{LG,B}$	low-gravity solids concentration, expressed in pounds per barrel
$c_{LG,C}$	low-gravity solids concentration, expressed in kilograms per cubic metre
$c_{NaCl,DF,A}$	whole-drilling-fluid sodium chloride concentration, expressed in milligrams per litre
$c_{NaCl,DF,B}$	whole-drilling-fluid sodium chloride concentration, expressed in pounds per barrel
$c_{NaCl,DF,C}$	whole-drilling-fluid sodium chloride concentration, expressed in kilograms per cubic metre
$c_{NaCl,DF,INSOL,A}$	whole-drilling fluid insoluble sodium chloride concentration, expressed in milligrams per litre
$c_{NaCl,DF,INSOL,B}$	whole-drilling-fluid insoluble sodium chloride concentration, expressed in pounds per barrel
$c_{NaCl,DF,SOL,A}$	whole-drilling-fluid soluble sodium chloride concentration, expressed in milligrams per litre
$c_{NaCl,DF,SOL,B}$	whole-drilling-fluid soluble sodium chloride concentration, expressed in pounds per barrel
$c_{NaCl,DF,SOL,C}$	whole-drilling-fluid soluble sodium chloride concentration, expressed in kilograms per cubic metre
c_{S-2}	concentration of active sulfides, expressed in milligrams per litre
$c_{WM,B}$	weighting material concentration, expressed in pounds per barrel
$c_{WM,C}$	weighting material concentration, expressed in kilograms per cubic metre
d_1	distance from the outer wall, expressed in inches
D	outer pipe diameter or inner diameter of open hole, expressed in inches
d	inner pipe diameter, expressed in inches
D_{TVD}	true vertical depth, expressed in feet
E	pump efficiency, expressed as percentage
f	tube factor, taken from Table I.2
G'	storage modulus, expressed in Newtons per square metre
G''	loss modulus, expressed in Newtons per square metre
k_C	consistency factor, expressed in pounds-force per second
L	length of the hydraulic section, expressed in feet
L_A	length, expressed in feet
l	submerged length of shear tube, expressed in centimetres
l_A	submerged length of shear tube, expressed in inches
l_D	Dräger tube darkened length (stained length), marked in units on the tube

m_1	mass of the empty retort assembly (cup, lid and body packed with steel wool), expressed in grams
m_2	mass of the filled retort assembly (cup with sample, lid and body packed with steel wool), expressed in grams
m_3	mass of the empty, dry liquid receiver, expressed in grams
m_4	mass of the cooled liquid receiver with condensed liquids, expressed in grams
m_5	mass of the cooled retort assembly (body packed with steel wool), expressed in grams;
m_d	mass of the dried retort cuttings, expressed in grams
m_{F1}	mass of drilling fluid following shear at 100 r/min, expressed in grams
m_{F2}	mass of drilling fluid taken from Sag Shoe following shear at 100 r/min, expressed in grams
m_{F3}	mass of drilling fluid taken from Sag Shoe following shear at 600 r/min, expressed in grams
m_L	mass of the liquid condensed (oil and water), expressed in grams
m_O	mass of the oil, expressed in grams
m_S	mass of the liquid drilling fluid sample, expressed in grams
m_{st}	mass of shear tube, expressed in grams
m_{tot}	total shear mass (sum of platform and weights), expressed in grams
m_{WC}	mass of the wet cuttings, expressed in grams
m_W	mass of water, expressed in grams
P	measured pressure, expressed in pounds-gauge per square inch
ΔP	anticipated pressure increase, expressed in pounds-gauge per square inch
$\frac{\Delta P}{\Delta L_A}$	pressure gradient, expressed in pounds-gauge per square inch per foot
Q	pump rate, expressed in gallons per minute
R_1	average reading for the standard thermometer, expressed in degrees
R_2	average reading for the working thermometer, expressed in degrees
R_{600}	dial reading at 600 revolutions per minute, expressed in degrees deflection
R_{300}	dial reading at 300 revolutions per minute, expressed in degrees deflection
R_{BPU}	calculated bed pickup measurement ratio, expressed as a percentage

R_O	ratio of the volume fraction of oil to the sum of the volume fractions of oil and pure water from the retort analysis, expressed as a percentage
R_B	ratio of the volume fraction of oil to the sum of the volume fractions of oil and brine, expressed as a percentage
R_W	ratio of the volume fraction of water to the sum of the volume fractions of oil and pure water from the retort analysis, expressed as a percentage
ROC	retained oil on cuttings, expressed in grams per kilogram of cuttings (either wet or dry)
S	Sag Register
t	time, expressed in minutes
V_1	spurt loss, expressed in millilitres
$V_{7,5}$	filtrate volume after 7,5 min, expressed in millilitres
V_{30}	filtrate volume after 30 min, expressed in millilitres
V_A	annular volume, expressed in barrels
V_{AgNO_3}	volume of 0,282 mol/l (0,282 N) silver nitrate reagent, expressed in millilitres
V_B	base alkalinity demand
V_{EDTA}	volume of 0,1 mol/l EDTA solution, expressed in millilitres
V_F	filtrate volume
$V_{H_2SO_4}$	volume of 0,05 mol/l (0,1 N) sulfuric acid, expressed in millilitres
V_K	whole-drilling-fluid alkalinity, expressed in millilitres of 0,05 mol/l sulfuric acid
V_M	receiver volume at specific mark, expressed in millilitres
V_{NaOH}	volume of 0,1 mol/l (0,1 N) NaOH, expressed in millilitres
V_O	volume of oil, expressed in millilitres
V_{PPT}	volume of filtrate from PPT, expressed in millilitres
V_R	total volume of condensed liquids (oil and water), expressed in millilitres
V_{RC}	volume of retort cup, expressed in millilitres
V_S	drilling fluid sample volume, expressed in millilitres;
V_W	water volume, expressed in millilitres, or water mass, expressed in grams (1 ml = 1 g) (see 3.2)
Δv_a	change in annular velocity, expressed in feet per minute

v_a	annular velocity, expressed in feet per minute.
v_f	static filtration rate (velocity of flow), expressed in millilitres per minute ^{1/2}
w_{CaCl_2}	aqueous-phase mass fraction of calcium chloride, expressed as a percentage of the total aqueous-phase mass
$w_{CaCl_2,SAT}$	aqueous-phase mass fraction of calcium chloride of a super-saturated fluid, expressed as a percentage of the total aqueous-phase mass
w_{NaCl}	aqueous-phase mass fraction of sodium chloride, expressed as a percentage of the total aqueous phase mass
$w_{NaCl,MAX}$	maximum aqueous-phase mass fraction of soluble sodium chloride that can exist for a given mass fraction of calcium chloride, expressed as a percentage of the total aqueous phase mass
$w_{NaCl,MAX-C}$	recalculated maximum aqueous-phase mass fraction of soluble sodium chloride that can exist for a given mass fraction of calcium chloride, expressed as a percentage of the total aqueous phase mass
Y_{PA}	yield point, expressed in pascals
Y_{PB}	yield point, expressed in pounds per one hundred square feet, often expressed as YP
β_{10m}	gel strength at 10 min, expressed in pounds per one hundred square feet
β_{10s}	gel strength at 10 s, expressed in pounds per one hundred square feet
$\Gamma_{DFG,A}$	drilling fluid gradient, expressed in kilopascals per metre
$\Gamma_{DFG,B}$	drilling fluid gradient, expressed in pounds per square inch per foot
γ_A	shear strength of the drilling fluid, expressed in pounds per square inch per foot
γ_B	shear strength of the drilling fluid, expressed in pascals
γ_i	fluid shear rate, expressed in reciprocal seconds
η	drillpipe rotation, expressed in revolutions per minute
η_{AV}	apparent viscosity, expressed in millipascal seconds (centipoises)
η_{PV}	plastic viscosity, expressed in millipascal seconds (centipoises)
φ_B	volume fraction of brine, expressed as a percentage of the whole drilling fluid
φ_D	corrected volume fraction of solids, expressed as a percentage of the whole drilling fluid
φ_d	volume fraction of dried retort solids, expressed as a percentage of the total sample volume
φ_{LG}	volume fraction of the low-gravity solids, expressed as a percentage of the total suspended solids

φ_O	volume fraction of oil, expressed as a percentage of the whole drilling fluid
φ_W	volume fraction of pure water, expressed as a percentage of the whole drilling fluid
φ_{WM}	volume fraction of the weighting material solids, expressed as a percentage of the total suspended solids
ρ	drilling fluid density, expressed in pounds per gallon
ρ_B	aqueous-phase density, expressed in grams per millilitre
ρ_C	drilling fluid density, expressed in kilograms per cubic metre
ρ_{B1}	drilling fluid density, expressed in pounds per gallon
ρ_{B2}	drilling fluid density, expressed in pounds per cubic foot
$\overline{\rho_d}$	average density (volumic mass) of the suspended solids.
$\rho_{ECD-hyd}$	pressure drop and extra density effects of drilled cuttings
$\rho_{ECD-tot}$	total predicted equivalent circulating density
$\Delta\rho_{ECD-rot}$	change in pressure due to rotation
ρ_{LG}	density of the low-gravity solids, expressed in grams per millilitre
ρ_{max}	maximum recorded drilling fluid density, expressed in pounds per gallon
ρ_{nom}	nominal drilling fluid density, expressed in pounds per gallon
ρ_O	density of the oil being used, expressed in grams per millilitre
ρ_S	drilling fluid density, expressed in grams per millilitre
ρ_W	water density, expressed in grams per millilitre, at the test temperature (see Table J.1)
ρ_{WM}	density of the weighting material solids, expressed in grams per millilitre
τ_W	wall shear stress, expressed in pounds-force per hundred square feet
τ_Y	drilling fluid yield stress, expressed in pounds-force per hundred square feet

4.2 Abbreviated terms

ACS	American Chemical Society
API	American Petroleum Institute
ASTM	American Society of Testing Materials
AV	apparent viscosity
CAS	Chemical Abstracting Services

ECD	equivalent circulating density [expressed in kilograms per cubic metre (pounds per gallon)]
EDTA	sodium salt of ethylenediaminetetraacetic acid dihydrate
ERD	extended reach drilling
ES	electrical stability
ESD	equivalent static density [expressed in kilograms per cubic metre (pounds per gallon)]
HTHP	high temperature, high pressure
OCMA	Oil Company Materials Association
OBR	oil-to-brine ratio
OWR	oil-to-water ratio
PNP	propylene glycol normal-propyl ether
PPA	permeability plugging apparatus
PPT	permeability plugging test
PTFE	polytetrafluoroethylene (e.g. Teflon®)
PV	plastic viscosity
PVT	pressure, volume and temperature relationship
SI	International System of units
TC	to contain
TD	to deliver
TVD	true vertical depth [expressed in metres (feet)]
USC	United States Customary units
VSST	Viscometer Sag Shoe Test
YP	yield point

5 Determination of drilling fluid density (mud weight)

5.1 Principle

A procedure is given for determining the mass of a given volume of liquid (i.e. density). The density of drilling fluid is expressed as grams per millilitre, or kilograms per cubic metre (pounds per gallon or pounds per cubic foot).

5.2 Apparatus

5.2.1 Any density-measuring instrument having an accuracy of $\pm 0,01$ g/ml or ± 10 kg/m³ (0,1 lb/gal or 0,5 lb/ft³).

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, e.g. bi-weekly or weekly. Fresh water should give a reading of 1,00 g/ml or 1 000 kg/m³ (8,345 lb/gal or 62,4 lb/ft³) 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. A calibration of the upper density should be performed as specified by the manufacturer, and done on a less frequent basis, e.g. annually.

5.2.2 Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F) and an accuracy of ±0,1 °C (±0,2 °F).

5.3 Procedure

5.3.1 The mud balance instrument shall be set on a flat, level surface.

5.3.2 Measure the temperature of the drilling fluid and record.

5.3.3 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.

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

5.3.5 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.

5.3.6 Read the drilling fluid density from one of the four calibrated scales on the arrow side of the sliding weight. As it is considered that the water density is 1 g/ml (see definitions 3.2 and 3.5), the density can be read directly in units of grams per millilitre using specific gravity scale, pounds per gallon and pounds per cubic foot, or as a drilling fluid gradient in pounds per square inch per 1 000 ft.

5.4 Calculation

5.4.1 Report the drilling fluid density to the nearest 0,01 g/ml or 10 kg/m³ (0,1 lb/gal or 0,5 lb/ft³).

5.4.2 To convert the reading, ρ , to other units, use Equations (1) to (7) and Tables 1 and 2.

Equations (1) to (3) are used to convert the density, ρ_S , expressed in grams per millilitre, to other units:

$$\rho_C = 1000 \times \rho_S \quad (1)$$

where ρ_C is the drilling fluid density, expressed in kilograms per cubic metre.

$$\rho_{B1} = 8,345 \times \rho_S \quad (2)$$

where ρ_{B1} is the drilling fluid density, expressed in pounds per gallon.

$$\rho_{B2} = 62,4 \times \rho_S \quad (3)$$

where ρ_{B2} is the drilling fluid density, expressed in pounds per cubic foot.

Table 1 provides the multiplication factor for conversion from one density unit to another.

Table 1 — Conversion of density units

Measured in	Multiply to get			
	g/ml	kg/m ³	lb/gal	lb/ft ³
g/ml	1	1 000	8,345	62,43
kg/m ³	0,001	1	0,008 3	0,062 43
lb/gal	0,120	120	1	7,480 5
lb/ft ³	0,016 0	16,02	0,133 7	1

Equations (4) to (7) are used to convert the density to the drilling fluid gradient, Γ_{DFG} , expressed in kilopascals per metre (pounds per square inch per foot):

$$\Gamma_{\text{DFG,A}} = 9,81 \times \text{g/ml} \quad (4)$$

$$\Gamma_{\text{DFG,A}} = 0,022\ 6 \times \text{psi/ft} \quad (5)$$

$$\Gamma_{\text{DFG,B}} = 0,052\ 0 \times \text{lb/gal} \quad (6)$$

$$\Gamma_{\text{DFG,B}} = 0,006\ 94 \times \text{lb/ft}^3 \quad (7)$$

where

$\Gamma_{\text{DFG,A}}$ is the drilling fluid gradient, expressed in kilopascals per metre;

$\Gamma_{\text{DFG,B}}$ is the drilling fluid gradient, expressed in pounds per square inch per foot.

A list of density conversions is given in Table 2.

Table 2 — Density conversion

Grams per cubic centimetre ^a g/ml	Kilograms per cubic metre kg/m ³	Pounds per gallon (lb/gal)	Pounds per cubic foot (lb/ft ³)
0,70	700	5,8	43,7
0,80	800	6,7	49,9
0,90	900	7,5	56,1
1,00	1 000	8,345 ^b	62,4
1,10	1 100	9,2	68,7
1,20	1 200	10,0	74,9
1,30	1 300	10,9	81,1
1,40	1 400	11,7	87,4
1,50	1 500	12,5	93,6
1,60	1 600	13,4	99,9
1,70	1 700	14,2	106,1
1,80	1 800	15,0	112,4
1,90	1 900	15,9	118,6
2,00	2 000	16,7	124,8
2,10	2 100	17,5	131,1
2,20	2 200	18,4	137,3
2,30	2 300	19,2	143,6
2,40	2 400	20,0	149,8
2,50	2 500	20,9	156,1
2,60	2 600	21,7	162,3
2,70	2 700	22,5	168,5
2,80	2 800	23,4	174,8
2,90	2 900	24,2	181,0

^a Same value as relative density as specific gravity in grams per cubic centimetre or kilogram per litre.

^b Accurate conversion factor.

6 Alternative method for determination of drilling fluid density

6.1 Principle

6.1.1 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 drilling fluid sample is placed in a fixed-volume sample cup under pressure.

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

6.2 Apparatus

6.2.1 Any **density-measuring instrument** having an accuracy of $\pm 0,01$ g/ml or 10 kg/m³ ($0,1$ lb/gal or $0,5$ lb/ft³).

A 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.

The instrument should be calibrated frequently with fresh water, e.g. bi-weekly or weekly. Fresh water should give a reading of 1,00 g/ml or 1 000 kg/m³ (8,345 lb/gal or 62,4 lb/ft³) 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. A calibration of the upper density should be performed as specified by the manufacturer, and done on a less frequent basis, e.g. annually.

6.2.2 Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F) and an accuracy of ± 1 °C (± 2 °F).

6.3 Procedure

6.3.1 Measure the temperature of the drilling fluid and record.

6.3.2 Fill the sample cup of the pressurized mud balance to a level approximately 6,5 mm (0,25 in) below the upper edge of the cup.

6.3.3 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 drilling fluid 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.

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

6.3.5 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 force the piston rod inside. A force of approximately 225 N (50 lb-force) or greater should be maintained on the piston rod.

6.3.6 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.

6.3.7 The pressurized drilling fluid sample is now ready for weighing. Rinse the exterior of the cup and wipe dry. Place the 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. As it is considered that the water density is 1 g/ml (see definitions 3.2 and 3.5), the density can be read directly in units of grams per millilitre using specific gravity scale, pounds per gallon, and pounds per cubic foot, or as a drilling fluid gradient in pounds per square inch per 1 000 feet.

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

6.3.9 Clean the cup, lid and pump assembly. Rinse thoroughly with base oil.

6.4 Calculation

Report the drilling fluid density to the nearest 0,01 g/ml or 10 kg/m³ (0,1 lb/gal or 0,5 lb/ft³).

For conversions, use the formulae given in 5.4.2.

7 Viscosity and gel strength

7.1 Principle

7.1.1 Viscosity and gel strength are measurements that relate to the flow properties (rheology) of drilling fluids.

7.1.2 The following instruments are used to measure viscosity and gel strength of drilling fluids for elevated and lower temperature applications. The lower temperature rheology may be anticipated in the riser annulus of deepwater drilling at elevated pressures. The equipment is as follows:

- a) Marsh funnel: a simple device for indicating viscosity on a routine basis;
- b) direct-indicating viscometer: a mechanical device for measuring viscosity at varying shear rates.

NOTE Information on the rheology of drilling fluids can be found in API RP 13D:2010.

7.2 Determination of viscosity using the Marsh funnel

7.2.1 Apparatus

7.2.1.1 Marsh funnel, calibrated to deliver 946 ml (1 quart) of fresh water at a temperature of 21 °C ± 3 °C (70 °F ± 5 °F) in 26 s ± 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 = 3/16 in);
- c) screen, with 1,6 mm (0,063 in = 1/16 in) openings (12 mesh); fixed at 19,0 mm (0,75 in) below top of funnel.

7.2.1.2 Graduated cup, with a capacity of at least 946 ml (1 quart).

7.2.1.3 Stopwatch.

7.2.1.4 Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F) and an accuracy of ±1 °C (±2 °F).

7.2.2 Procedure

7.2.2.1 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.

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

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

7.2.2.4 Report the time (7.2.2.2), to the nearest second, with the volume, as the Marsh funnel viscosity. Report the temperature (7.2.2.3) of the fluid to the nearest degree Celsius (degree Fahrenheit).

7.3 Determination of viscosity and gel strength using a direct-reading viscometer

7.3.1 Apparatus

7.3.1.1 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 – R1:

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
sleeve surface:	surface roughness average 16 to 32 cross-hatch honed

b) Bob – B1, closed, with flat base and tapered top:

diameter:	34,49 mm (1,358 in)
cylinder length:	38,0 mm (1,496 in)
rotor surface:	surface roughness average 16 to 32 cross-hatch honed

c) Torsion spring constant – F1,0:

torsional stiffness:	10,54 N·m/rad (386 dyne-cm/degree deflection)
shear stress constant:	29,3 pascals per radian deflection (0,511 pascals per degree of deflection) (1,065 lb/100 ft ² and degree of 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.

7.3.1.2 Stopwatch.

7.3.1.3 Thermostatically controlled viscometer cup:

a) greater than room temperature: thermostatically controlled direct-heated viscometer cup;

- b) less than room temperature: double-walled viscometer cup connected to a thermostatically controlled refrigerated circulating bath.

7.3.1.4 Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F) and an accuracy of ± 1 °C (± 2 °F).

7.3.2 Procedure

7.3.2.1 Place a sample of the drilling fluid in a thermostatically controlled viscometer cup. Leave enough empty volume (approximately 50 ml to 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 $50\text{ °C} \pm 1\text{ °C}$ ($120\text{ °F} \pm 2\text{ °F}$) or $65\text{ °C} \pm 1\text{ °C}$ ($150\text{ °F} \pm 2\text{ °F}$) for reference comparisons to historical data. Testing at a lower temperature, such as $4\text{ °C} \pm 1\text{ °C}$ ($40\text{ °F} \pm 2\text{ °F}$), is recommended for low temperature effects. The place of sampling should be stated in the report.

CAUTION — The maximum recommended operating temperature is 90 °C (190 °F). If it is necessary to test fluids above this temperature, a solid metal bob or a hollow metal bob with a completely dry interior should be used. Liquid trapped inside a hollow bob can vaporize when immersed in high-temperature fluid and cause the bob to explode.

7.3.2.2 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.

7.3.2.3 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} .

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

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

7.3.2.6 Stop the rotor and allow the 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, β_{10s} , at 10 s, in pounds per one hundred square feet.

NOTE Gel strength value is calculated from dial reading. 1 degree deflection is equal to 1,065 Pa in SI units and to 0,511 lbf/100 ft² in USC units.

7.3.2.7 Restir the drilling fluid sample at 600 r/min for 10 s, stop the motor and then allow the drilling fluid to stand undisturbed for 10 min. Repeat the measurements as in 7.3.2.6 and report the maximum reading as β_{10m} , the 10 m gel, in pounds per one hundred square feet (lbf/100 ft²).

NOTE Gel strength value is calculated from dial reading. 1 degree deflection is equal to 1,065 Pa in SI units and to 0,511 lbf/100 ft² in USC units.

7.3.3 Calculation

7.3.3.1 The dimensions for the rotor, bob and spring constant, as described in 7.3.1.1, determine the following:

- 1° deflection of the bob equals a shear stress of approximately 1 lbf/100 ft², or more exactly $1,065\text{ }100\text{ lbf}/100\text{ ft}^2 = 0,511\text{ Pa}$;
- 1 r/min of the rotor equals a shear rate of $1,702\text{ }3\text{ s}^{-1}$.

The viscosity, defined as shear stress in millipascal divided by shear rate in reciprocal seconds, will be expressed in millipascal seconds (which is equivalent to centipoises). At 300 r/min (shear rate of 511 s^{-1}), the degree deflection will correspond to millipascal seconds (or centipoises).

7.3.3.2 The calculation for the plastic viscosity, η_{PV} , expressed in millipascal seconds (centipoises), is given in Equation (8):

$$\eta_{PV} = R_{600} - R_{300} \quad (8)$$

where

η_{PV} is the plastic viscosity, expressed in millipascal seconds (centipoises);

R_{600} is the dial reading at 600 revolutions per minute, expressed in degrees deflection;

R_{300} is the dial reading at 300 revolutions per minute, expressed in degrees deflection.

NOTE 1 Plastic viscosity is commonly known in the industry by the abbreviation PV.

NOTE 2 1 cP = 1 mPa.s.

7.3.3.3 The calculation for yield point, Y_{PA} , expressed in pascals, is given in Equation (9):

$$Y_{PA} = 0,48 \times (R_{300} - \eta_{PV}) \quad (9)$$

where

Y_{PA} is the yield point, expressed in pascals.

7.3.3.4 When calculating values in USC units, the yield point in pounds per one hundred square feet is calculated as follows:

$$Y_{PB} = R_{300} - \eta_{PV} \quad (10)$$

where

Y_{PB} is the yield point, expressed in pounds per one hundred square feet.

NOTE Yield point, in 100 lb/ft², is commonly known in the industry by the abbreviation YP.

7.3.3.5 The calculation for apparent viscosity, η_{AV} , expressed in millipascal seconds (centipoises), is given in Equation (11):

$$\eta_{AV} = \frac{R_{600}}{2} \quad (11)$$

where

R_{600} is the dial reading at 600 r/min, expressed in degrees deflection;

η_{AV} is the apparent viscosity, expressed in millipascal seconds (centipoises).

NOTE Apparent viscosity, in millipascal seconds, is commonly known in the industry by the abbreviation AV.

Report the plastic viscosity, yield point, 10 s gel, 10 min gel, and apparent viscosity.

8 Static filtration

8.1 Principle

8.1.1 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, aqueous phase or emulsion concentration.

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

8.1.3 Filtration tests are performed at high-temperature conditions and under static conditions. 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 1 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.

NOTE 2 For the purposes of this provision, API RP 13B-1 is equivalent.

8.1.4 The 175 ml, 250 ml, or 500 ml unit may be used for static filtration testing up to and including 175 °C (350 °F).

8.1.5 For testing above 175 °C (350 °F), only the 500 ml unit shall be used. It shall be equipped with a thermocouple in direct contact with the fluid contained in the cell to more accurately measure the temperature, and it shall use a porous filter stainless-steel media.

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

8.2.1 Apparatus

8.2.1.1 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 with temperature controller or thermostat, to heat to 175 °C (350 °F);
- d) high-pressure filtrate collection vessel, maintained at proper back-pressure (see Table 3) 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.

CAUTION — Strict adherence to manufacturer's recommendations as to sample volumes, equipment temperatures and pressures is essential. Failure to do so could result in serious injury.

Do not use nitrous oxide cartridges as pressure sources for HTHP 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, in accordance with ISO 10414-1:2008, Clause A.5.

NOTE For the purposes of this provision, API RP 13B-1:2009, Clause A.5, is equivalent.

Table 3 — Recommended minimum back-pressure

Test temperature		Vapour pressure		Minimum back-pressure	
°C	°F	kPa	psi	kPa	psi
100	212	101	14,7	690	100
120	250	207	30	690	100
150	300	462	67	690	100
Limit of "normal" field testing					
175	350	932	135	1 104	160
200	400	1 704	247	1 898	275
230	450	2 912	422	3 105	450

8.2.1.2 Filter medium:

- a) Whatman No. 50 or S&S 576⁴⁾, or equivalent filter paper, for temperatures up to 200 °C (400 °F);
- b) porous disc, Dynalloy X-5⁵⁾ or equivalent, for temperatures above 200 °C (400 °F): a new disc is required for each test.

8.2.1.3 Mechanical or electronic timer, with at least a 30 min interval.

8.2.1.4 Thermometer, with a range up to 260 °C (500 °F), and with a 12,5 cm (5 in) or longer stem, or a thermocouple with a range up to 260 °C (500 °F), preferred.

8.2.1.5 Graduated cylinder (TC), long, slender glass tube, with a capacity of 10 ml or 20 ml.

8.2.1.6 Graduated cylinder (TC), optional, with a capacity of 25 ml.

8.2.1.7 Field mixer, cup type, to operate at 10 r/min, 1 000 r/min and 15 000 r/min.

8.2.1.8 Ruler, graduated in millimetres (inches), to measure filter cake thickness.

8.2.2 Procedure

8.2.2.1 Place the thermometer in the well of the heating jacket. Preheat the jacket to approximately 6 °C (10 °F) above the desired test temperature. Adjust the thermostat to the desired test temperature.

If the filtration unit is equipped with a thermocouple in direct contact with the fluid to measure drilling fluid temperature inside the cell (test temperature), then that temperature should be monitored and reported during the filtration test. Filtration results reported for temperatures so measured may differ from results based on cell wall temperature. Under the "Comments" section, record if the results were based on fluid temperature measured with a direct contact thermocouple.

⁴⁾ Whatman® No. 50 and S&S® 576 are examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO 10414 and does not constitute an endorsement by ISO of these products.

⁵⁾ Dynalloy® X-5 disc is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 10414 and does not constitute an endorsement by ISO of this product.

8.2.2.2 Stir the drilling fluid sample for 10 min using the field mixer set at the 1 000 r/min speed. Pour the fluid sample into the filter cell, leaving at least a 2,5 cm (1 in) space in the cell to allow for fluid expansion. Install the porous disk [or the filter paper if the test temperature is below 200 °C (400 °F)] in the cell.

8.2.2.3 Complete the assembly of the filter cell, with both top and bottom valves closed, and place it in the heating jacket. Transfer the thermometer from the heating jacket into the well of the filter cell.

8.2.2.4 Connect the high-pressure filtrate collection vessel onto the lower valve stem and lock it in place. Ensure that the collection vessel is completely free of water or oil.

8.2.2.5 Connect the regulated pressure source to the upper valve. Connect a similar regulated pressure source to the filtrate collection vessel and lock these connections in place.

8.2.2.6 Keeping the two valve stems closed, adjust the pressure on the upper pressure regulator to 690 kPa (100 psi) higher than the minimum back-pressure value, as shown in Table 3. Open the upper valve stem, and readjust the upper pressure regulator, if required, to maintain the pressure 690 kPa (100 psi) higher than the minimum back-pressure shown in Table 3 for the test temperature. Maintain both pressures until the test temperature is reached.

NOTE If the time required to reach the test temperature exceeds 1 h, the heater might be defective and the validity of the test is questionable.

8.2.2.7 When the sample reaches the selected test temperature, as indicated by the thermocouple, set the lower pressure regulator at the pressure ("minimum back-pressure") shown for the temperature given in Table 3. Open the lower valve stem and immediately increase the pressure on the upper regulator to 3 450 kPa (500 psi) higher than the back-pressure. This will start the filtration process. Start the timer. Maintain the test temperature to within ± 3 °C (± 5 °F) during the test, as indicated by the thermometer in the filter cell. If the back-pressure rises above the selected back-pressure during the test, cautiously draw off and collect a portion of the filtrate to reduce the back-pressure.

8.2.2.8 Collect the filtrate in the long slender graduated cylinder (or optional graduated cylinder). Record the volume of the 30 min total (water plus oil) filtrate. Also record the volumes of solid and aqueous phases, if present.

NOTE The long slender glass cylinder allows more accurate detection and measurements of volumes of oil, water and solids in the filtrate. Heating of the cylinder near an emulsion interface can improve separation of water, solids and oil in the filtrate.

8.2.2.9 Immediately after collecting the 30 min filtrate, turn off to stop heating and unplug the heating jacket from the electrical outlet. Close the upper, then lower valve stems to contain the pressure. Following the manufacturer's detailed instructions, bleed pressure off the regulators and hoses, then disconnect the pressurization system. Remove the cell from the heating jacket and allow cell to cool to below 50 °C (125 °F). Keep the cell upright during cooling, depressurization and disassembly.

CAUTION — Pressure in the filter cell can be 6 200 kPa (950 psi), even after the cell is cooled. To avoid possible serious injury, keep cell upright and cool to room temperature, then bleed pressure from cell before disassembling.

8.2.2.10 Bleed pressure from the filter cell by slowly opening the upper valve stem. Avoid spraying drilling fluid as gas exits the stem. Ensure that pressure is fully released before dislodging the cap. Carefully disassemble the cell.

8.2.2.11 Pour the liquid from the cell.

8.2.2.12 Remove the filter cake on the porous disk (or the filter paper). Measure the filter cake thickness, at its centre, to the nearest millimetre (1/32 in).

8.2.2.13 Settling of solids onto the filter cake may have occurred during the test. Observe indications of this, such as an abnormally thick cake or coarse texture. Record these cake characteristics under the "Comments" on the Oil Mud Report Form (see Annex P). To minimize settling, the times for heat-up and cool-down should be minimized and the cake should be recovered and examined promptly.

8.2.3 Calculation

8.2.3.1 The filtrate volume, V_F , should be corrected to a filter area of 4 516 mm² (7,0 in²). HTHP filter cells usually have half the standard filter area (2 258 mm²) (3,5 in²), thus double the observed volume before reporting.

8.2.3.2 Report the cake thickness to the nearest millimetre (1/32 in), its texture and the presence of any emulsion or water in the filtrate.

8.3 High-temperature/high-pressure test 175 °C (350 °F) up to and including 230 °C (450 °F)

8.3.1 Apparatus

8.3.1.1 High-temperature/high-pressure filter press, consisting of the following components:

a) 500 ml volume cell, only;

NOTE For safety reasons, it is advisable that only the 500 ml cell be used for testing up to and above 230 °C (450 °F).

b) filter cell, to contain working pressures up to 15 500 kPa (2 250 psi) at a temperature of 230 °C (450 °F);

c) pressurized gas source, nitrogen with regulators (preferred);

d) heating system with temperature controller or thermostat, to heat to 260 °C (500 °F);

e) high-pressure filtrate collection vessel, maintained at proper back-pressure (see Table 3), to avoid flashing or evaporation of the filtrate;

f) filter cell, equipped with an internal thermocouple to monitor temperature of a drilling fluid sample near its centre in the cell, with removable end fitted with oil-resistant seals.

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

CAUTION — Not all manufacturers' equipment can be used above 150 °C (300 °F). Failure to know the pressure/temperature rating of equipment in use can result in serious injury. Testing at high temperature and high pressure calls for added safety precautions. The 175 ml and 250 ml filtration cells are not recommended for use at these higher temperatures and pressures.

CAUTION — Do not use nitrous oxide cartridges as pressure sources for HTHP 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, in accordance with ISO 10414-1:2008, Clause A.5.

NOTE For the purposes of this provision, API RP 13B-1:2009, Clause A.5, is equivalent.

8.3.1.2 Filter medium, Dynalloy® X-5 or equivalent porous disc, for temperatures above 200 °C (400 °F). A new disc is required for each test.

8.3.1.3 Mechanical or electronic timer, with at least a 30 min interval.

8.3.1.4 Thermometer, with a range up to 260 °C (500 °F), and with a 12,5 cm (5 in) or longer stem, or a thermocouple with a range up to 260 °C (500 °F), preferred.

8.3.1.5 **Graduated cylinder (TC)**, long, slender with a volume of 10 ml or 20 ml.

8.3.1.6 **Graduated cylinder (TC)**, optional, with a volume of 25 ml.

8.3.1.7 **Field mixer**, cup type, to operate at 10 r/min, 1 000 r/min and 15 000 r/min.

8.3.1.8 **Ruler**, graduated in millimetres (inches), to measure filter cake thickness.

8.3.2 Procedure

8.3.2.1 Place the thermometer in the well of the heating jacket. Preheat the jacket to approximately 6 °C (10 °F) above the desired test temperature. Adjust the thermostat to the test temperature.

If the filtration unit is equipped with a thermocouple in direct contact with the fluid to measure drilling fluid temperature inside the cell (test temperature), then that temperature should be monitored and reported during the filtration test. Filtration results reported for temperatures so measured may differ from results based on cell wall temperature. Under the "Comments" section, record if the results were based on fluid temperature measured with a direct contact thermocouple.

8.3.2.2 Stir the drilling fluid sample for 10 min using the field mixer set at the 1 000 r/min speed. Pour the fluid sample into the filter cell, leaving at least a 2,5 cm (1 in) space in the cell to allow for fluid expansion. Install the filter paper in the cell.

8.3.2.3 Complete the assembly of the filter cell. Install the thermocouple into the cell to monitor the temperature of the fluid near the centre of the cell. Place the cell, with upper and lower valve stems closed, in the heating jacket. Connect the thermocouple to the temperature-readout instrument and determine that it is reading correctly.

8.3.2.4 Connect the high-pressure filtrate collection vessel onto the lower valve stem and lock it in place. Ensure that the filtrate collection vessel is free of residual water or oil.

8.3.2.5 Connect the pressurized gas source to the upper valve. Connect a similar pressurized gas source to the lower collection vessel and lock these connections in place.

8.3.2.6 Keeping the two valve stems closed, adjust the pressure on the upper pressure regulator to 690 kPa (100 psi) higher than the minimum back-pressure value, as shown in Table 3. Open the upper valve stem, and readjust the upper pressure regulator, if required, to maintain the pressure 690 kPa (100 psi) higher than the minimum back-pressure shown in Table 3 for the test temperature. Maintain both pressures until the test temperature is reached.

NOTE If the time required to reach the test temperature exceeds 1 h, the heater might be defective and the validity of the test is questionable.

8.3.2.7 When the sample reaches the selected test temperature, as indicated by the thermocouple, set the lower pressure regulator at the pressure ("minimum back-pressure") shown for the temperature given in Table 3. Open the lower valve stem and immediately increase the pressure on the upper regulator to 3 450 kPa (500 psi) higher than the back-pressure. This will start the filtration process. Start the timer. Maintain the test temperature to within ± 3 °C (± 5 °F) during the test, as indicated by the thermometer in the filter cell. If the back-pressure rises above the selected back-pressure during the test, cautiously draw off and collect a portion of the filtrate to reduce the back-pressure.

8.3.2.8 Collect the filtrate in the long slender graduated cylinder (or optional graduated cylinder). Record the volume of the 30 min total (water plus oil) filtrate. Also record the volumes of solid and water phases, if present.

8.3.2.9 Immediately after collecting the 30 min filtrate, turn off to stop heating and unplug the heating jacket from the electrical outlet. Close the upper, then lower valve stems to contain the pressure. Following the manufacturer's detailed instructions, bleed pressure off the regulators and hoses, then disconnect the pressurization system. Remove the cell from the heating jacket and allow the cell to cool to below 50 °C (125 °F). Keep the cell upright during cooling, depressurization and disassembly.

CAUTION — Pressure in the filter cell can be 6 200 kPa (950 psi), even after the cell is cooled. To avoid possible serious injury, keep cell upright and cool to room temperature, then bleed pressure from cell before disassembling.

8.3.2.10 Bleed pressure from the filter cell by slowly opening the upper valve stem. Avoid spraying drilling fluid as gas exits the stem. Ensure that pressure is fully released before dislodging the cap. Carefully disassemble the cell.

8.3.2.11 Pour the liquid from the cell.

8.3.2.12 Remove the filter cake on the filter paper. Measure the filter cake thickness, at its centre, to the nearest millimetre (1/32 in).

8.3.2.13 Settling of solids onto the filter cake may have occurred during the test. Observe indications of this, such as an abnormally thick cake or coarse texture. Record these cake characteristics. To minimize settling, the times for heat-up and cool-down should be minimized and the cake should be recovered and examined promptly.

8.3.3 Calculation

8.3.3.1 The filtrate volume, V_F , should be corrected to a filter area of 4 580 mm² (7,0 in²). HTHP filter cells usually have half the standard filter area (2 258 mm²) (3,5 in²), thus double the observed volume before reporting.

8.3.3.2 Report the cake thickness to the nearest millimetre (1/32 in), the texture and the presence of any emulsion or water in the filtrate.

9 Retort test for oil, water and solids concentrations

9.1 Principle

9.1.1 A retort test measures water and oil released from an oil-based drilling fluid sample when heated in a calibrated and properly operating retort instrument. Included in this clause are procedures for performing a retort analysis using either a volumetric or gravimetric method.

NOTE The gravimetric procedure will provide more accurate values than the standard volumetric approach.

9.1.2 Knowledge of water, oil and solids concentrations is fundamental to proper control of drilling fluid properties such as oil-to-water ratio, rheology, density, filtration and salinity of the aqueous (water) phase. Given that knowledge of solids in an oil-based drilling fluid is essential to the evaluation of solids control equipment, reference shall be made to ISO 13501.

NOTE For the purposes of this provision, API RP 13C is equivalent.

9.1.3 In a retort test, a known volume or mass of oil-based drilling fluid is heated in a retort instrument to vaporize the liquid components. These vapours are then condensed and collected in a precision-graduated liquid receiver.

9.1.4 For the volumetric method, the volume fractions expressed as percentages of oil, water and solids are calculated from the total starting volume of oil-based drilling fluid and the condensed liquid volumes of water and oil collected in a precision-graduated liquid receiver.

9.1.5 For the gravimetric method, the volume fractions expressed as percentages of oil, water and solids are calculated from the mass of retorted oil-based drilling fluid, mass of dry solids after retorting, density of the water, oil, and oil-based drilling fluid plus the measured volume of condensed water collected in a precision-graduated receiver. If the density of the oil used is not known, optional procedures are given for either calculating the density of the oil from mass measurements or for using handheld density-measuring devices.

NOTE This gravimetric method is based on the mass lost during retorting and differs from the volumetric method which uses the recovered volume. This might result in the volume fraction solids values being lower than in the volumetric method, especially with oil-based drilling fluids or where volatile components are not fully condensed with the volumetric method.

9.2 Apparatus

9.2.1 Retort instrument, as specified below.

- a) **Retort assembly**, including a retort body, cup and lid constructed of 303 stainless steel, or equivalent.

Standard cup sizes are 10 ml (precision $\pm 0,05$ ml), 20 ml (precision $\pm 0,1$ ml), and 50 ml (precision $\pm 0,25$ ml). When using the volumetric procedure, the retort cup volume with lid shall be verified gravimetrically in accordance with the procedure and calculations given in ISO 10414-1:2008, Annex H.

NOTE For the purposes of this provision, API RP 13B-1:2009, Annex H, is equivalent.

- b) **Condenser**, capable of cooling the oil and water vapours below their vaporization temperature.
- c) **Heating jacket**, nominal power 350 W.
- d) **Temperature controller**, capable of limiting the temperature of the retort to $500\text{ }^{\circ}\text{C} \pm 40\text{ }^{\circ}\text{C}$ ($930\text{ }^{\circ}\text{F} \pm 70\text{ }^{\circ}\text{F}$).

9.2.2 Liquid receiver (TC), specially designed cylindrical glassware with a rounded bottom to facilitate cleaning and a funnel-shaped top to catch falling drops, meeting the following specifications:

- **precision**: see Table 4;
- **calibration (TC)**: at $20\text{ }^{\circ}\text{C}$ ($68\text{ }^{\circ}\text{F}$);
- **scale**: millilitre or volume fraction (as a percentage);
- **material**: transparent and inert to oil, water and salt solutions at temperatures up to $32\text{ }^{\circ}\text{C}$ ($90\text{ }^{\circ}\text{F}$).

Table 4 — Precision of liquid receiver

		Total volume			
		10 ml	20 ml	50 ml	50 ml tapered
Precision	(0 % to 100 %)	$\pm 0,05$ ml	$\pm 0,10$ ml	$\pm 0,25$ ml	—
Frequency of graduation marks	(0 % to 100 %)	0,10 ml	0,10 ml	0,50 ml	—
	(0 % to 5 %)	—	—	—	0,05 ml
	(5 % to 100 %)	—	—	—	0,25 ml

When using the volumetric procedure, the receiver volume shall be verified gravimetrically in accordance with the procedure and calculations in ISO 10414-1:2008, Annex H.

NOTE For the purposes of this provision, API RP 13B-1:2009, Annex H, is equivalent.

9.2.3 Fine steel wool, oil-free.

Liquid steel wool or coated steel wool substitutes should not be used for this application.

9.2.4 High-temperature-resistant silicone grease, to be used as a thread seal and lubricant.

9.2.5 Pipe cleaners.

9.2.6 Putty knife or spatula, with blade shaped to fit the inside dimensions of the sample cup of the retort.

9.2.7 Corkscrew.

9.2.8 Syringe, of capacity 10 ml, 20 ml, or 50 ml, to fill retort cup.

9.2.9 Marsh funnel (7.2.1.1).

9.2.10 Top-loading balance, capable of weighing 2 000 g with an accuracy of $\pm 0,01$ g.

9.2.11 Mud balance (5.2.1).

9.3 Procedure — Volumetric method

9.3.1 Ensure that the retort sample cup, condenser passage and liquid receiver are clean, dry and cooled from previous use. Thoroughly clean the inside of the sample cup and lid with a putty knife or spatula prior to each test. Periodically, the interior of the sample cup should also be lightly polished with steel wool. The condenser passage should also be cleaned and dried before each test using pipe cleaners. A build-up of material in the condenser passage can decrease condensation efficiency which can cause erroneous liquid readings in the test and, for some types of equipment, may present a safety hazard.

CAUTION — A moist or partially clogged condenser passage may be a safety hazard.

9.3.2 The heating jacket should be cooled to less than 93 °C (200 °F).

9.3.3 Pack the retort body with steel wool.

9.3.4 Collect a representative sample of oil-based drilling fluid and allow it to cool to approximately 27 °C (80 °F). Screen the test sample through the 1,68 mm (0,066 in or 12-mesh) screen of the Marsh funnel to remove lost circulation material, large cuttings or debris.

9.3.5 Mix the drilling fluid sample thoroughly to ensure it is homogeneous. Be careful not to entrain any air, and ensure that no solids remain on the bottom of the container.

NOTE Air or gas entrapment in the retort sample will result in erroneously high retort solids, due to a reduced volume of drilling fluid sample.

9.3.6 Fill the retort sample cup slowly to avoid air entrapment. Lightly tap the side of the cup to expel air. Place the lid on the cup. Rotate the lid to obtain a proper fit. Ensure that a small excess of drilling fluid flows out of the hole in the lid. Wipe excess sample from the lid; avoid wicking out the drilling fluid through the hole.

9.3.7 Apply lubricant/sealant sparingly to the threads of the retort sample cup. With lid in place, hand-tighten the retort sample cup onto the body.

9.3.8 Apply lubricant/sealant sparingly to the threads on the condenser passage stem and attach to the condenser body. Place the retort assembly into the heating jacket. Close the insulating lid.

9.3.9 Place the clean, dry liquid receiver below the condenser passage outlet.

NOTE 1 To improve the ability to make accurate readings of the volume of oil and water, it is possible to pre-wet the inside of the glass liquid receiver with propylene glycol normal-propyl ether (PNP). PNP is the product used to break the oil-based mud emulsion during chemical titration of oil-based drilling fluid (see Clause 10). The basic method for wetting the glass liquid receiver is to add approximately 0,5 ml of PNP to the liquid receiver, then tilt and roll the liquid receiver to allow the solvent to coat the inside. After coating the inside of the liquid receiver with solvent, turn the receiver upside down long enough to empty all of the excess solvent.

NOTE 2 PNP degrades easily. It is advisable that only fresh chemical, within the expiration period noted, be used, and that proper disposal in accordance with local, state and federal regulations be ensued. It is readily biodegradable.

NOTE 3 Liquid or vapour PNP can cause moderate eye irritation and moderate corneal injury. Prolonged skin contact can cause slight irritation with local redness, but is unlikely to result in absorption of harmful amounts. Repeated exposure can cause drying and flaking of the skin, irritation, or a burn. Brief inhalation of PNP (minutes) is not likely to cause adverse effects. Excessive inhalation can cause irritation to the nose and throat. Excessive exposure can cause lethargy. PNP has low toxicity if swallowed. Swallowing large amounts can cause injury. Repeated exposure can cause liver, kidney and eye effects. Birth defects were not observed in laboratory animals.

NOTE 4 The length of the liquid receiver might require that it be angled out from the retort condenser passage and perhaps supported off the edge of the worktable.

9.3.10 Turn on the heating jacket and allow the retort assembly to run a minimum of 1 h. Collect the condensate into the glass liquid receiver. If drilling fluid boils over into the liquid receiver, cool and clean the equipment and rerun the test using a larger amount of steel wool packed into the retort body.

9.3.11 Remove the liquid receiver and allow it to cool.

CAUTION — The retort body is still extremely hot and will cause severe burns if contacted.

NOTE 1 If an emulsion interface is present between the oil and water phases, heating the interface might break the emulsion. As a suggestion, remove the retort assembly from the heating jacket by grasping the condenser. Carefully heat the glass liquid receiver along the emulsion band by gently touching the receiver for short intervals with the hot retort assembly. Avoid boiling the liquid. After the emulsion interface is broken, allow the liquid receiver to cool.

Record the total liquid volume, V_R , and water volume, V_W , collected in the liquid receiver.

NOTE 2 Reading the meniscus correctly is extremely important for accuracy. Firstly, always read the meniscus with the interface at eye level. Secondly, for the air-to-liquid meniscus, read the volume at the “lowest point” of the meniscus which is in the middle of the liquid receiver at the very bottom of the liquid. For opaque liquids it might be necessary to estimate the top of the liquid in the middle of the cylinder. Thirdly, for the water-to-oil meniscus, read the water volume at the lowest point.

9.3.12 Turn off the heating jacket. Remove the retort assembly and condenser from the heating jacket and allow them to cool. Remove the condenser. Clean the retort assembly and condenser.

9.4 Calculation — Volumetric method

9.4.1 Using the measured volumes of oil and water, and the volume of the original whole drilling fluid sample (10 ml, 20 ml, or 50 ml), calculate, as percentages, the volume fractions of water, oil and total solids in the drilling fluid.

9.4.2 Calculate the volume of oil in the condensed sample.

$$V_O = V_R - V_W \quad (12)$$

where

V_O is the volume of oil, expressed in millilitres;

V_R is the total volume of condensed liquids (oil and water), expressed in millilitres;

V_W is the water volume, expressed in millilitres, or water mass, expressed in grams (1 ml = 1 g) (see 3.2).

9.4.3 Calculate the volume fraction of oil in the total sample.

$$\varphi_O = 100 \times \frac{V_O}{V_S} \quad (13)$$

where

φ_O is the volume fraction of oil, expressed as a percentage of the total sample volume;

V_O is the volume of oil, expressed in millilitres;

V_S is the drilling fluid sample volume, expressed in millilitres.

9.4.4 Calculate the volume fraction of water.

$$\varphi_W = 100 \times \frac{V_W}{V_S} \quad (14)$$

where

φ_W is the volume fraction of water, expressed as a percentage;

V_W is the water volume, expressed in millilitres, or water mass, expressed in grams (1 ml = 1 g) (see 3.2);

V_S is the drilling fluid sample volume, expressed in millilitres.

9.4.5 Calculate the volume fraction of solids remaining in the retort.

$$\varphi_d = 100 - \varphi_W - \varphi_O \quad (15)$$

where

φ_d is the volume fraction of dried retort solids, expressed as a percentage of the total sample volume;

φ_W is the volume fraction of water, expressed as a percentage;

φ_O is the volume fraction of oil, expressed as a percentage of the total sample volume.

NOTE The calculated solids overestimate the volume of suspended solids because of dissolved salts. A volumetric correction for salinity can be made using known volume factors for salt solutions (see Clause 12). Calculation for determining the oil-to-water ratio and the corrected solids concentration and density are also found in Clause 12.

9.5 Procedure — Gravimetric method

9.5.1 Ensure that the retort sample cup, condenser passage and liquid receiver are clean, dry and cooled from previous use. Thoroughly clean the inside of the sample cup and lid with a putty knife or spatula prior to each test. Periodically, the interior of the sample cup should also be lightly polished with steel wool. The condenser passage should also be cleaned and dried before each test using pipe cleaners. A build-up of material in the condenser can decrease condensation efficiency which can cause erroneous liquid readings in the test and, for some types of equipment, may present a safety hazard.

CAUTION — A moist or partially clogged condenser passage may be a safety hazard.

9.5.2 The heating jacket should be cooled to less than 93 °C (200 °F).

9.5.3 Pack the retort body with steel wool.

9.5.4 Apply lubricant/sealant sparingly to the threads of the retort cup. With lid in place, hand-tighten the retort cup onto the body.

9.5.5 Apply lubricant/sealant sparingly to the threads on the condenser passage stem and attach to the condenser body.

9.5.6 Weigh and record the total mass of the empty retort sample cup, lid, and retort body packed with steel wool. Record this as m_1 , expressed in grams.

NOTE All weights are recorded to the nearest 0,01 g.

9.5.7 Collect a representative sample of oil-based drilling fluid and allow it to cool to approximately 27 °C (80 °F). Screen the test sample through the 1,68 mm (0,066 in or 12-mesh) screen of the Marsh funnel to remove lost circulation material, large cuttings or debris.

9.5.8 Mix the drilling fluid sample thoroughly to ensure it is homogeneous. Be careful not to entrain any air, and ensure that no solids remain on the bottom of the container.

NOTE Air or gas entrapment in the retort sample will result in erroneously high retort solids, due to a reduced volume of drilling fluid sample.

9.5.9 Measure and record the density of the oil-based drilling fluid using a mud balance as described in Clause 5 or Clause 6 (more accurate). Alternative gravimetric methods, such as a volumetric flask or cup, are also acceptable.

Record the drilling fluid density as ρ_S , to the nearest 0,01 g/ml, 10 kg/m³ (0,1 lb/gal or 0,5 lb/ft³).

Alternatively, there are several small-volume portable handheld density-measuring devices that can be used to accurately measure the density of the drilling fluid. Examples of such devices are Anton Paar® DNA 35N and Mettler® Densito.⁶⁾

9.5.10 If the density of the oil being used is not known, measure the density of the base oil using a mud balance, as described in Clause 5 or 6 (more accurate). Alternative gravimetric methods, such as a volumetric flask or cup, are also acceptable.

Record the oil density as ρ_O , to the nearest 0,01 g/ml, 10 kg/m³ (0,1 lb/gal or 0,5 lb/ft³).

Alternatively, there are several small-volume portable handheld density-measuring devices that can be used to accurately measure the density of the condensed oil. Example of such devices are Anton Paar® DNA 35N and Mettler® Densito.

9.5.11 Remove the retort cup from the retort body. Fill the retort sample cup slowly to avoid air entrapment. Lightly tap the side of the cup to expel air. Place the lid on the cup. Rotate the lid to obtain a proper fit. Ensure that a small excess of drilling fluid flows out of the hole in the lid. Wipe excess sample from the lid; avoid wicking out the drilling fluid through the hole.

⁶⁾ Anton Paar® DNA 35N and Mettler® Densito are examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO 10414 and does not constitute an endorsement by ISO of these products.

9.5.12 Screw the retort sample cup with lid onto the retort body. Weigh the retort sample cup filled with drilling fluid, lid, and retort body (packed with steel wool). Record this as m_2 , expressed in grams.

NOTE All weights are recorded to the nearest 0,01 g.

9.5.13 Attach the condenser. Place the retort assembly into the heating jacket. Close the insulating lid.

9.5.14 Weigh an empty, clean, dry liquid receiver. Record this as m_3 , expressed in grams. Place the receiver below the condenser passage outlet.

NOTE 1 To improve the ability to make accurate readings of the volume of oil and water, it is possible to pre-wet the inside of the glass liquid receiver with PNP. PNP is the product used to break the oil-based mud emulsion during chemical titration of oil-based drilling fluid (see Clause 10). The basic method for wetting the glass liquid receiver is to add approximately 0,5 ml of PNP to the liquid receiver, then tilt and roll the liquid receiver to allow the solvent to coat the inside. After coating the inside of the liquid receiver with solvent, turn the receiver upside down long enough to empty all of the excess solvent.

NOTE 2 PNP degrades easily. It is advisable that only fresh chemical, within the expiration period noted, be used.

NOTE 3 Due to the rounded bottom of the liquid receiver, it might be helpful to place the liquid receiver in a 100 ml graduated cylinder to hold it on the top-loading balance while being weighed.

NOTE 4 The length of the liquid receiver might require that it be angled out from the retort condenser passage and perhaps supported off the edge of the worktable.

9.5.15 Turn on the heating jacket and allow the retort assembly to run a minimum of 1 h. Collect the condensate into the glass liquid receiver. If drilling fluid boils over into the liquid receiver, cool and clean the equipment and rerun the test using a larger amount of steel wool packed into the retort body.

9.5.16 Remove the liquid receiver and allow it to cool.

CAUTION — The retort body is still extremely hot and will cause severe burns if contacted.

NOTE 1 If an emulsion interface is present between the oil and water phases, heating the interface might break the emulsion. As a suggestion, remove the retort assembly from the heating jacket by grasping the condenser. Carefully heat the glass liquid receiver along the emulsion band by gently touching the receiver for short intervals with the hot retort assembly. Avoid boiling the liquid. After the emulsion interface is broken, allow the liquid receiver to cool.

Record the total condensed liquid volume, V_R , and water volume, V_W , collected in the liquid receiver. These are converted to mass, expressed in grams, as described in 9.6.

NOTE 2 Reading the meniscus correctly is extremely important for accuracy. Firstly, always read the meniscus with the interface at eye level. Secondly, for the air-to-liquid meniscus, read the volume at the “lowest point” of the meniscus which is in the middle of the liquid receiver at the very bottom of the liquid. For opaque liquids it might be necessary to estimate the top of the liquid in the middle of the cylinder. Thirdly, for the water-to-oil meniscus, read the water volume at the lowest point.

9.5.17 Weigh the glass liquid receiver and its liquid content (oil and water). Record as m_4 , expressed in grams.

9.5.18 Turn off the heating jacket. Remove the retort assembly and condenser from the heating jacket and allow them to cool. Remove the condenser.

9.5.19 Remove the condenser and passage stem. Weigh the cooled retort assembly (retort sample cup, lid, and retort body with steel wool packing) without the condenser. Record this as m_5 , expressed in grams.

9.5.20 Clean the retort assembly and condenser.

9.6 Calculation — Gravimetric method

9.6.1 Calculate the mass balance factor of the retorted oil-based drilling fluid, dry retorted solids, and condensed liquids to be certain that they are within $\pm 3\%$ for an accurate test.

9.6.2 Calculate the mass of the liquid drilling fluid sample before retorting.

$$m_S = m_2 - m_1 \quad (16)$$

where

m_S is the mass of the liquid drilling fluid sample, expressed in grams;

m_2 is the mass of the filled retort assembly (cup with sample, lid and body packed with steel wool), expressed in grams;

m_1 is the mass of the empty retort assembly (cup, lid and body packed with steel wool), expressed in grams.

9.6.3 Calculate the mass of the dry solids after retorting.

$$m_d = m_5 - m_1 \quad (17)$$

where

m_d is the mass of the dried retort cuttings, expressed in grams;

m_5 is the mass of the cooled retort assembly (body packed with steel wool), expressed in grams;

m_1 is the mass of the empty retort assembly (cup, lid and body packed with steel wool), expressed in grams.

9.6.4 Calculate the mass of liquids (oil and water) condensed after retorting.

$$m_L = m_4 - m_3 \quad (18)$$

where

m_L is the mass of the liquid condensed (oil and water), expressed in grams;

m_4 is the mass of the cooled liquid receiver with condensed liquids, expressed in grams;

m_3 is the mass of the empty, dry liquid receiver, expressed in grams.

9.6.5 Calculate the mass balance of the retorting method. The sum of the mass of dried retorted solids, m_d , and condensed liquids, m_L , after retorting divided by the mass of the beginning oil-based drilling fluid sample, m_S , shall be greater than 0,97 but less than 1,03.

$$0,97 \leq \frac{m_d + m_L}{m_S} \leq 1,03 \quad (19)$$

Repeat the procedure if this requirement is not met.