

Recommended Practice Standard Procedure for Laboratory Testing Drilling Fluids

API RECOMMENDED PRACTICE 131
SIXTH EDITION, MAY 2000



**Helping You
Get The Job
Done Right.SM**

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Upstream Segment

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FOREWORD

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Standard Procedure for Laboratory Testing Drilling Fluids

1 scope

The purpose of this recommended practice is to provide standard procedures for the testing of drilling fluids. It is not a detailed manual on mud control procedures. It should be remembered that the agitation history and temperature of testing have a profound effect on mud properties.

Metric SI unit equivalents have been included in this publication in parentheses following the U.S. customary units.

2 References

2.1 STANDARDS

Unless otherwise specified, the most recent editions or revisions of the following standards and specifications shall, to the extent specified herein, form a part of this standard.

API

Spec 13A	<i>Specification for Drilling Fluid Materials</i>
RP 13B-1	<i>Recommended Practice Standard Procedure for Field Testing Water-Based Drilling Fluids</i>
RP 13K	<i>Recommended Practice for Chemical Analysis of Barite</i>
Publ 2547	<i>Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method, equivalent to ASTM D 1298-80 and IP 160/68</i>
Std 2540	<i>Petroleum Measurement Tables; equivalent to ASTM D 1250 and IP 200</i>

ASTM¹

A 526	<i>Specification for Steel Sheet, Zinc-Coated (Galvanized) by the Hot-Dip Process, Commercial</i>
D 86-82	<i>Method for Distillation of Petroleum Products</i>
D 93-80	<i>Test Methods for Flash Point by Pensky-Martens Closed Tester</i>
D 97-66	<i>Test Methods for Pour Point of Petroleum Oils</i>
D 323	<i>Test Method for Vapor Pressure of Petroleum Products (Reid Method)</i>
D 422-63	<i>Method for Particle-Size Analysis of Soils</i>
D 445-83	<i>Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)</i>

D 61 1-82	<i>Test Method for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents</i>
D 1250-80	<i>Petroleum Measurement Tables (description only)</i>
D 1298-80	<i>Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method</i>
D 2540	<i>Test Method for Drop Weight Sensitivity of Liquid Monopropellants</i>
D 4052-81	<i>Test Method for Density and Relative Density of Liquids by Digital Density Meter</i>
D 5186	<i>Test Method for the Determination of the Aromatic Content of Diesel Fuels by Supercritical Fluid Chromatography</i>

IP²

2/76	<i>Test Method for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents; equivalent to ASTM D 61 1-82</i>
15/67	<i>Test Methods for Pour Point of Petroleum Oils; equivalent to ASTM D 97-66</i>
34/75	<i>Test Methods for Flash Point by Pensky-Martens Closed Tester; equivalent to ASTM D 93-80</i>
69	<i>Test Method for Vapor Pressure of Petroleum Products (Reid Method); equivalent to ASTM D 323</i>
71/79	<i>Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity); equivalent to ASTM D 445-83</i>
123/76	<i>Method for Distillation of Petroleum Products; equivalent to ASTM D 86-82</i>
160/68	<i>Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method; equivalent to ASTM D 1298-80</i>
200/52	<i>Petroleum Measurement Tables (Description only); equivalent to ASTM D 1250-80 and API Std 2540</i>
391/88	<i>Test Method for the Determination of the Aromatic Content of Diesel Fuels by Supercritical Fluid Chromatography, equivalent to ASTM D 5 186</i>

¹American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428.

²Institute of Petroleum, 61 New Cavendish Street, London, England, W1M 8AR.

Bingham and Jackson, *Bur, Standard Bull.* 14, 75 (1918).

2.2 OTHER REFERENCES

Bingham, *Fluidity and Plasticity*, McGraw-Hill, New York (1922).

3 Particle Size Analysis for Fines in Barite

3.1 DESCRIPTION

Fines are the particles in the range of 2-10 microns equivalent Stokes' diameter and are considered to be of limited value in a weighting material.

3.2 EQUIPMENT

The following equipment is needed:

- a. Dispersant solution: 40 g sodium hexametaphosphate and approximately 3.6 g sodium carbonate/100 cm³ of solution. The sodium carbonate is used to adjust the pH of the solution to 9.0 or slightly less. After the initial pH adjustment, check the pH each day the solution is used. When the pH falls below 8.0, discard the solution.
- b. Oven: 220° ± 5°F (105 ± 3°C).
- c. Mixer: 11,000 ± 300 rpm under load, with single corrugated impeller approximately 1 in. (25 mm) in diameter (e.g., Multi-mixer Model 9B with 9B29 impeller, or equivalent).
- d. Container for mixing: 7 in. (180 mm) deep, 3¹³/₁₆ in. (97 mm) ID at top and 2³/₄ in. (70 mm) ID at bottom (e.g., Hamilton Beach Mixer Cup No. M110-D, or equivalent).
- e. Sieves: 75-micrometer, 45-micrometer and 30-micrometer sieves, having a diameter of 3 in. (76 mm) and a depth of 2¹/₂ in. (63 mm) from top of the frame to the wire cloth.
- f. Electric stopwatch: with direct reading counter, 1/100–1000 minutes.
- g. Interval timer: 1/4–120 minutes with signal bell. This timer is not a necessity, but is very convenient.
- h. Clock or watch.
- i. Rubber stopper: number 13.
- j. Wash bottles: one containing 125 cm³ dispersant solution diluted to 1000 cm³ with deionized water, and one with deionized water.
- k. Laboratory balance: sensitivity 0.01 g.
- l. Thermometer: 60°–90°F (16°–32°C), with accuracy of 1°F (0.5°C).
- m. Beaker: 250 cm³.
- n. Water bath or constant temperature room: capable of maintaining a convenient constant temperature at or near 68°F (20°C).
- o. Glass sedimentation cylinder: 18 in. (457 mm) high and 2¹/₂ in. (63 mm) diameter, and marked for a volume of 1000 cm³ (ASTM D 422-63).

- p. Hydrometer: ASTM 151H, graduated to read specific gravity of the suspension.
- q. Evaporating dishes.

3.3 PROCEDURE-SAMPLING

Obtain four samples of approximately 10 g and one sample of approximately 80 g of the barite by tube sampling and quartering for use in this particle size analysis.

3.4 PROCEDURE-MOISTURE

3.4.1 Weigh 10 ± 0.01 g of barite obtained by 3.3.

3.4.2 Dry to a constant weight at a temperature of 220° ± 5°F (105 ± 3°C).

3.4.3 Cool the sample in a desiccator and weigh.

3.5 CALCULATION-MOISTURE

$$\text{Weight \% moisture} = 100 \frac{(W_o - W)}{W_o} \quad (1)$$

where

W_o = weight of original sample, g,

W = weight of dry sample, g.

3.6 PROCEDURE-SIEVE ANALYSIS

3.6.1 Weigh 10 ± 0.01 g of barite obtained in 3.3, and place in mixing container. Add 44 cm³ of dispersant solution; hand-stir the sample and dilute to approximately 350 cm³ with deionized water. Stir 5 minutes on Multimixer.

3.6.2 Wash the sample with the diluted dispersant solution onto a 75-micrometer sieve. Continue to wash with approximately 400 cm³ of the dilute dispersant solution using a wash bottle. Then wash the material on the screen with tap water at 10 psig (170 kPa) from a spray nozzle (e.g., Spraying Systems Company No. TG 6.5 tip with 1/4 'IT body, or equivalent) for 2 minutes. While washing, allow the elbow bend of the nozzle to rest on the rim of the sieve and move the spray of water repeatedly over the surface of the screen. After tap water washing, wash the sample at least twice with deionized water, then transfer the residue from the screen to a tared evaporating dish deionized water to remove the residue from the screen.

3.6.3 Dry the residue in the oven to a constant weight, cool in a desiccator, and weigh to ± 0.01 g.

3.6.4 Repeat 3.6.1, 3.6.2, and 3.6.3 using 45-micrometer and 30-micrometer sieves with separate barite samples.

3.7 CALCULATION-SIEVE ANALYSIS

$$W = W_o \frac{100 - \text{weight \% moisture}}{100} \quad (2)$$

$$\% \text{ Residue} = 100 \frac{(W_r)}{W} \quad (3)$$

$$\% \text{ Finer} = 100 - \% \text{ residue} \quad (4)$$

where

w = weight of dry sample, g,

W_o = weight of original sample, g,

W_r = weight of residue, g.

3.8 PROCEDURE-SEDIMENTATION ANALYSIS

3.8.1 Weigh 80 ± 0.01 g of barite obtained by 3.3, and place in mixing container. Add 125 cm^3 of dispersant solution, hand-stir the sample and dilute to approximately 40 cm^3 with deionized water. Stir 5 minutes on Multimixer.

3.8.2 Transfer the mixture to a 1000 cm^3 sedimentation cylinder, washing all the sample into the cylinder and adding deionized water to the 1000 cm^3 mark. Mix the contents thoroughly by constantly changing the cylinder from the

upright to the inverted position and back for 60 seconds while holding a No. 13 rubber stopper in the top of the cylinder.

3.8.3 As the cylinder is set on the countertop, start the timer immediately. Hang the thermometer in the suspension.

3.8.4 Take hydrometer and thermometer readings at intervals of 5, 10, 20, 40, 90, 180 and 360 minutes. (This should give particle size ranges from less than 2 to over 10 microns.)

When taking a hydrometer reading, carefully insert the hydrometer about 20 – 25 seconds before the reading is due to approximately the depth at which the reading is taken. As soon as the reading is taken, carefully remove the hydrometer, clean in distilled or deionized water, and dry.

3.9 CALCULATION—SEDIMENTATION ANALYSIS

3.9.1 The amount and particle diameter of the fine material can be determined from the hydrometer data as follows (see Fig. 1 for example data sheet and calculation):

$$S, \% \text{ sample in suspension} = \left[\frac{(100,000SG)}{W(SG-1)} \right] (H_c - 1) \quad (5)$$

where

W = weight of dry sample, g,

SG = density of barite sample (determined according to API Specification 13A, Section entitled "Bade"),

Sample: API Barite Fines Content, April 1978 SG 4.30 Hydrometer No. 2

Time, Min.	Hydrometer Reading, SG	Temperature °C	Correction From Curve	Corrected Reading, Hc
5	1.0350	25.5	• 0.0019	1.033 1
10	1.0280	25.5	• 0.0019	1.0261
20	1.0210	25.5	• 0.0019	1.0191
40	1.0140	25.5	• 0.0019	1.0121
90	1.0120	25.5	• 0.0019	1.0101
180	1.0085	25.0	• 0.0020	1.0065
360	1.0070	25.0	0.0020	1.0050

EXAMPLE CALCULATION

$$\begin{aligned} \% \text{ Sample in suspension} &= \frac{(100,000)(4.30)(1.0261 - 1)}{(80)(4.30 - 1)} \\ &= 4.25\% \text{ (for the 10 min point)} \end{aligned}$$

$$\begin{aligned} \text{Particle Size D, microns} &= 17.5 \sqrt{\frac{vL}{(SG-1)T}} \\ &= 17.5 \sqrt{\frac{(0.8837)(8.9)}{(4.30-1)(10)}} \\ &= 8.5 \text{ (for the 10 min point)} \end{aligned}$$

Figure I-Example Data Sheet and Example Calculation

Table 1-Viscosity of Water at Various Temperatures

Temperature °F	Viscosity cP or mPa	Temperature °F (°C)	Viscosity cP or mPa
60 (15.6)	1.1225	72 (22.2)	0.9533
61 (16.1)	1.1061	73 (22.8)	0.9399
62 (16.7)	1.0911	74 (23.3)	0.9291
63 (17.2)	1.0773	75 (23.9)	0.9163
64 (17.8)	1.0611	76 (24.4)	0.9058
65 (18.3)	1.0479	77 (25.0)	0.8937
66 (18.9)	1.0324	78 (25.6)	0.8815
67 (19.4)	1.0197	79 (26.1)	0.8717
68 (20.0)	1.0050	80 (26.7)	0.8601
69 (20.6)	0.9904	81 (27.2)	0.8507
70 (21.1)	0.9785	82 (27.8)	0.8396
71 (21.7)	0.9646	83 (28.3)	0.8305

Values were calculated by the formula:

Viscosity, cP =

$$0.021482 \left[(t + 8.435) + \sqrt{8078.4 + (t - 8.435)^2} \right] - 1.2$$

where

t = temperature in Celsius.

Bingham and Jackson, *Bur. Standard Bull.* **14, 75 (1918)**. Bingham, *Fluidity and Plasticity*, p. 340, McGraw-Hill, New York (1922).

H_c = corrected hydrometer reading,

= hydrometer reading-composite correction
(see Notes).

$$D, \text{ Particle Diameter} = 100 \sqrt{\frac{30vL}{980(SG-1)T}} \quad (6)$$

where

D = equivalent **Stoke's diameter**, (μM),

v = viscosity of water at **test temperature**, cP (see Table 1),

L = effective depth of hydrometer, cm (see Table 2),

T = time interval from start of sedimentation to time reading is taken, minutes.

3.9.2 Plot Percent Sample in Suspension (S) or arithmetic scale as the ordinate (labeled "Cumulative Percent Finer") and the Particle Diameter (D) on a logarithmic scale as the abscissa.

Note: The composite correction is read from a curve drawn for each hydrometer. (Notice that this is a negative number.) It is necessary because:

Table 2-Values of Effective Depth Based on Readings on Hydrometer No. 151 H used in Specific Sedimentation Cylinder

Uncorrected Hydrometer Reading	Effective Depth L, cm	Uncorrected Hydrometer Reading	Effective Depth L, cm
1.000	16.3	1.020	11.0
1.001	16.0	1.021	10.7
1.002	15.8	1.022	10.5
1.003	15.5	1.023	10.2
1.004	15.2	1.024	10.0
1.005	15.0	1.025	9.7
1.006	14.7	1.026	9.4
1.007	14.4	1.027	9.2
1.008	14.2	1.028	8.9
1.009	13.9	1.029	8.6
1.010	13.7	1.030	8.4
1.011	13.4	1.031	8.1
1.012	13.1	1.032	7.8
1.013	12.9	1.033	7.6
1.014	12.6	1.034	7.3
1.015	12.3	1.035	7.0
1.016	12.1	1.036	6.8
1.017	11.8	1.037	6.5
1.018	11.5	1.038	6.2
1.019	11.3		

a. Equations for percentages of barite remaining in suspension, as given in 3.9.1 are based on the use of distilled or deionized water. A dispersing agent is used in the water, however, and the specific gravity of the resulting solution is greater than that of water.

b. Hydrometers are calibrated at 68°F (20°C). and variations in temperature from this standard temperature produce inaccuracies in the actual hydrometer reading. The amount of inaccuracy increases as the variation from standard temperature increases.

c. Hydrometers are graduated by the manufacturer to be read at the bottom of the meniscus formed by the liquid on the stem. Since it is not possible to secure readings of barite suspensions at the bottom of the meniscus, readings must be taken at the top and a correction applied.

Note 2: The net amount of the corrections for the three items listed in the above note is designated as the composite correction, and may be determined experimentally as follows:

a. For convenience, a graph or table of composite corrections for a series of 1-degree temperature differences for the range of expected test temperatures may be prepared and used as needed. Measurement of the composite corrections may be made at several temperatures spanning the range of expected test temperatures, and corrections for the test temperatures calculated assuming a straight-line relationship for the observed values (see Figure 2).

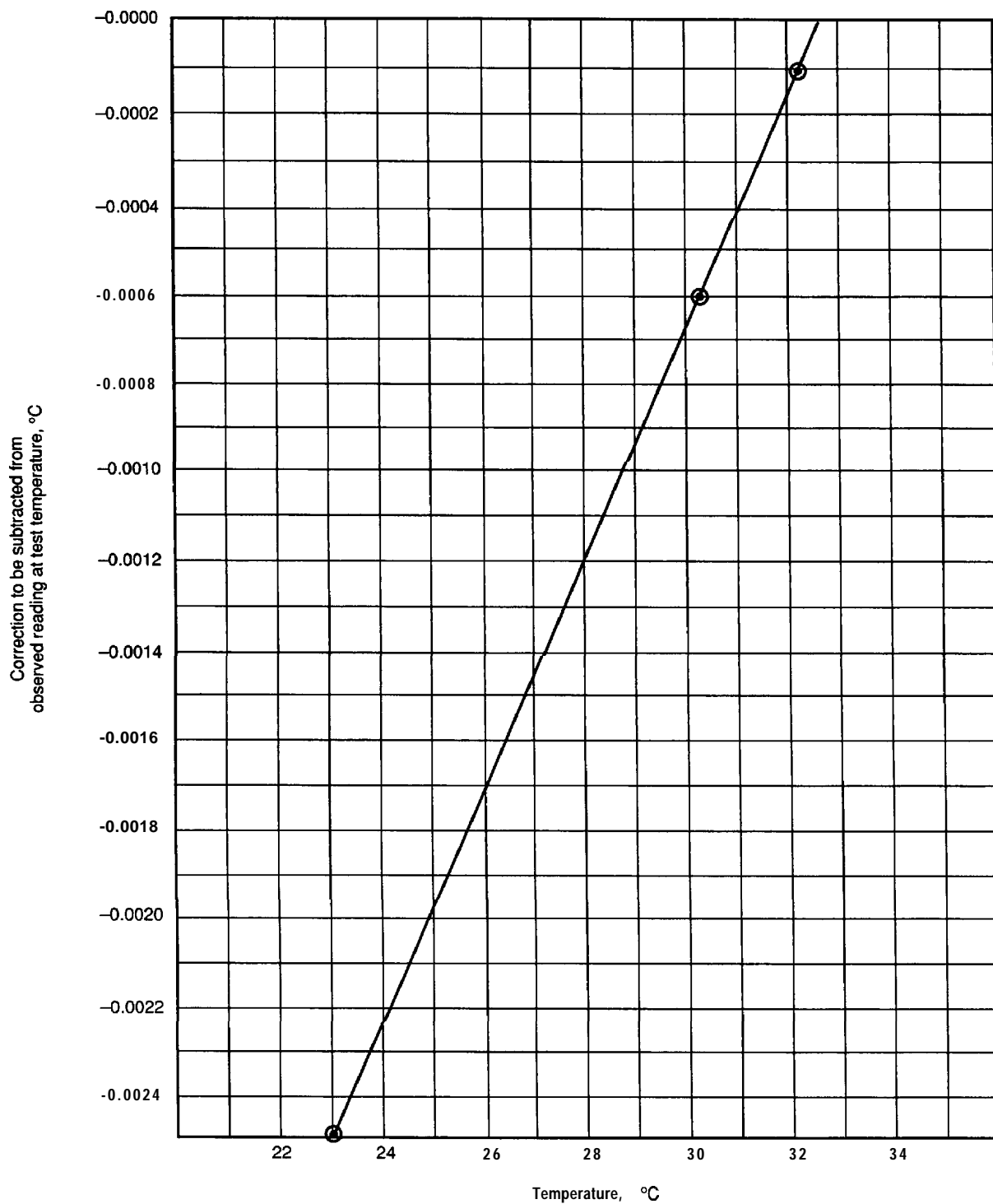


Figure 2-Example Graph of Composite Corrections for a Hydrometer-See 3.9.2

b. Prepare 1000 cm^3 of liquid composed of deionized water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test. Place the liquid in a sedimentation cylinder and the cylinder in the constant-temperature bath, set for one of the two temperatures to be used. When the temperature of the liquid becomes constant, insert the hydrometer, and after a short interval to permit the hydrometer to come to the temperature of the liquid, read the hydrometer at the top of the meniscus formed on the stem. For hydrometer 151H, the composite correction is one minus this reading. Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

4 Barite Performance

4.1 DESCRIPTION

4.1.1 The rheological properties of a weighted drilling fluid will vary based on the density, impurities, and fines in the barite. This test compares the rheological properties of a drilling fluid using a barite to a similar system using an API test calibration barite or a barite of known satisfactory performance.

4.1.2 The drilling fluid is a fresh water, prehydrated bentonite, lignosulfonate slurry. A good indicator of undesirable performance due to excessive impurities and fines is when the test sample has significantly higher rheological properties than the comparison barite.

4.1.3 This test will not reveal all undesirable impurities and should be used in conjunction with the API Barite Specification tests in API Specification 13A, and the Recommended Practice for Chemical Analysis of Barite in API Recommended Practice 13K.

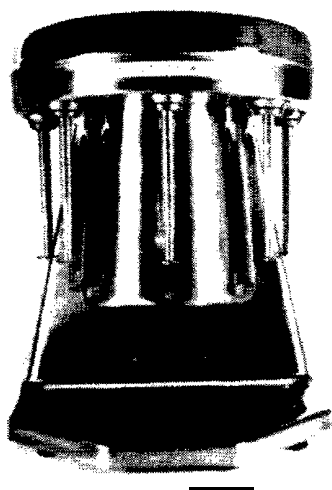


Figure 3—Multimixer Model 9B with 9B29X Impeller

4.2 EQUIPMENT

The following equipment is needed:

- a. API test calibration barite.
- b. API reference bentonite.

Note: Stocks of API test calibration barite and API reference bentonite have been set aside by API. Requests for this material should be directed to the API Upstream Department, 1220 L Street, NW, Washington, DC 20005, which will forward them to the designated custodian for all further handling. Accuracy of results for use of this calibration material is strictly the responsibility of the laboratories using it.

Note 2: For locations without ready access to the API test calibration barite and API reference bentonite, tests using local material are possible by adjusting the amount of bentonite to give proper base-mud viscosity and using a reference barite of proven performance. When non-API test calibration materials are used, comparisons between laboratories may be very difficult or impossible.

- c. Sodium hydroxide: (CAS #13 10-73-2) 5N NaOH solution.
- d. Ferrochrome or chrome lignosulfonate.

Note: For between laboratory comparisons, a common sample of the same high quality lignosulfonate should be divided between laboratories for all testing.

- e. Oven: roller type. $225^{\circ} \pm 5^{\circ}\text{F}$ ($105^{\circ} \pm 3^{\circ}\text{C}$).
- f. pH meter.
- g. Aging cell: 2 or more, 500 cm^3 , stainless steel.
- h. Viscometer: 3,300, and 600 rpm, as described in API Recommended Practice 13B-1, section entitled "Viscosity and Gel Strength."
- i. Dispersator or equivalent high-shear mixer, equipped with duplex blade.
- j. Variable transformer speed control for dispersator.
- k. Beaker: one 1500 to 2000- cm^3 .
- l. Volumetric flask: one 1-liter,
- m. Container: one 2-gallon capacity.
- n. Balance: 0 – 3000 g, precision of ± 1 g.
- o. Mixer: 11,000 ± 300 rpm under load, with single corrugated impeller approximately 1 in. (25 mm) in diameter; (e.g., Multimixer Model 9B with 9B29X impeller, or equivalent, as shown in Figure 3. The 9B29X impeller should be replaced when approximately $\frac{1}{8}$ weight loss occurs. The original weight is about 5.5 g).
- p. Mixer cup: two or more cups, depending on number of samples to be evaluated. 7 in. (180 mm) deep, $3\frac{13}{16}$ in. (97 mm) ID top, $2\frac{3}{4}$ in. (70 mm) bottom (e.g., Hamilton Beach mixer cup No. M1 10-D, or equivalent).

4.3 PROCEDURE-BASE MUD PREPARATION

4.3.1 Prepare approximately 10-barrel equivalents of bentonite slurry having a pH of 11.8 – 11.9 and a 600 rpm dial reading on the viscometer of 20 – 24 at $80 \pm 5^{\circ}\text{F}$ ($27 \pm 3^{\circ}\text{C}$).

Note: Larger or smaller batches may be prepared. Use a suitably sized container and proportional quantities of bentonite, water, lignosulfonate, and sodium hydroxide solution for a 21 **lb/bbl bentonite** slurry containing 4 **lb/bbl** lignosulfonate and 1.2 **lb/bbl NaOH**.

4.3.2 To make lo-barrel equivalent, add 3500 **cm³** distilled or deionized water into a 2-gallon (8-liter) container. While stirring with dispersator at 8500 rpm ± 1000 , slowly add 210 g of bentonite. Position dispersator shaft well off-center in container for maximum mixing shear. Continue stirring for 30 minutes.

4.3.3 Age a minimum of 16 hours in a sealed container at room temperature.

4.3.4 After aging, **restir** 5 minutes with dispersator at 8500 ± 1000 rpm. Slowly add 40 g of lignosulfonate, then add 55 ± 10 **cm³** of 5N NaOH. Stir 30 minutes after the addition of the NaOH.

4.3.5 Age a minimum of 4 hours in a sealed container at room temperature.

Note: Although the base mud can be used after only 4 hours, aging 16 hours is recommended.

4.3.6 Stir the base mud 5 minutes at 8500 ± 1000 rpm. Measure the pH of the mud and adjust to 11.8 – 11.9 with 5N NaOH.

4.3.7 Read the dial reading of the base mud at 600 rpm on the viscometer at 80° $\pm 5^\circ\text{F}$ (27° $\pm 3^\circ\text{C}$). The 600 rpm dial reading should be 20-24. If the reading is 18-19, additional high shear stirring will usually increase the reading to the acceptable range. If a second trial fails to give the proper dial reading, check materials and techniques used. If the reading is in the 24-28 range, readjust by adding distilled or deionized water in approximately 50 **cm³** increments. After each addition, stir 10 minutes and check the 600 rpm dial reading. If it is still greater than 24, add more water but do not exceed 250 **cm³**. When the 600 rpm dial reading is within the specified range, measure and record the pH and proceed to 4.4.

4.4 PROCEDURE-RHEOLOGY TEST

4.4.1 Measure 240 **cm³** of base mud into two or more mixer cups (one cup for each barite to be tested and one cup for the API test calibration barite).

4.4.2 While stirring on the mixer, slowly add 480 g of the sample to be tested to the base mud. Stir for five minutes. Scrape the sides to ensure that all the barite is well mixed. Stir an additional 10 minutes.

4.4.3 Repeat 4.4.2 using the API test calibration barite.

4.4.4 Transfer each of the weighted slurries to an aging cell. Seal and weigh cells. Record weight to the nearest gram.

4.4.5 Age the samples while hot rolling for 16 hours at 225° $\pm 5^\circ\text{F}$ (107° $\pm 3^\circ\text{C}$).

4.4.6 Cool the cells to 75° $\pm 5^\circ\text{F}$ (24° $\pm 3^\circ\text{C}$) and weigh. If weight change exceeds 5 g from 4.4.4, discard and repeat test.

4.4.7 Open aging cells and stir the aged mud 5 minutes on mixer.

4.4.8 Within 30 seconds after stirring and with the sample temperature at 80° $\pm 5^\circ\text{F}$ (27° $\pm 3^\circ\text{C}$), measure the rheological properties. Determine and record the plastic viscosity, yield point, 10 second and 10 minute gels and pH as described in API Recommended Practice 13B-1, *Field Testing Water-Based Drilling Fluids*, Sections entitled "Viscosity and Gel Strength" and "pH." Also record the sample temperature.

4.5 CALCULATION

4.5.1 Significant differences between the rheological properties of the API test calibration barite slurry and the sample barite slurries are the best indicators of undesirable performance effects. A barite with fewer impurities and fines will normally have a lower plastic viscosity, yield point and gels than the API test calibration barite.

4.5.2 One means of comparison is to determine the Performance Index (PI) of the various slurries.

$$PI = YP + PV + 3(\text{10-second gel} + \text{10-minute gel}) \quad (7)$$

In most cases improved performance can be expected if the Performance Index of the test sample is equivalent to or less than the API test calibration barite.

Example: If the PI = 150 for the API test calibration barite, a better performing barite will have a PI = 150 or less. In the same test sequence, a test barite with a PI = 200 may add excessive fines and give high viscosities in a field drilling fluid.

5 Abrasiveness of Weighting Materials

5.1 DESCRIPTION

5.1.1 Drilling fluid weighting materials may vary considerably in relative abrasivity. This laboratory test is designed to measure and evaluate this relative abrasiveness.

5.1.2 The test is performed using a standard test blade attached to a high speed mixer to mix a base mud containing the weight material. The weight loss of the blade is used to calculate the abrasiveness of the weight material in mg/min.

5.1.3 This test is only intended to give a relative index of wear for weighting materials. It should not be used to infer whether or not these materials would cause abrasion problems in a field mud system.

5.2 EQUIPMENT

The following equipment is needed:

- a. Constant Speed Mixer: 11,000 rpm \pm 300 rpm under load with single sine-wave impeller approximately 1 in. (2.54 cm) in diameter (e.g. Multimixer Model 9B with 9B29X impeller, or equivalent as shown in Figure 3). The impeller shall be replaced when its weight falls below 5.1 g. The original weight is about 5.5 g.
- b. Container: Approximate dimensions-7 in. (180 mm) deep, $3^{13}/16$ in. (97 mm) ID top, $2^{3}/4$ in. (70 mm) ID bottom (e.g., Hamilton Beach Mixer Cup No. M110-d, or equivalent).
- c. Balance: Sensitive to ± 0.0001 g.
- d. Balance: 500 g capacity, sensitive to ± 0.1 g.
- e. Abrasion Test Blade:

Specifications for the Abrasion Blade

Diameter: 1.5 in. ($1^{7}/16$ inches flat side)

Thickness: 0.056 in.

Hardness: 16 (Rockwell scale)

Wave Depth: 0.25 in.

Wave Width: 0.60 in.

No. Waves: 4

Size Center Hole: $9/32$ in.

Type of Steel: ASTM-A526

Coating Material: G-90

Approximate Weight: 12 g

Each blade may be used up to a cumulative weight loss of 200 mg (approximately 4 runs).
- f. Screw: $1/2$ in. (13 mm) size 10, round head machine screw, 32 threads/in. used to fasten abrasion test blade securely onto the shaft of the abrasion mixer.
- g. Tachometer: 11,000 \pm 50 rpm.
- h. Graduated Cylinder (TD): 500 \pm 2.5 cm³.
- i. API Reference Bentonite.

Note: Stocks of API reference bentonite for the test have been set aside by the API. Request for this material should be directed to the API Washington Office which will forward the request to the designated custodian for all further handling. Accuracy of results from use of this material is strictly the responsibility of the laboratory using it. See note following 4.2 for address.

- j. Detergent.
- k. Small Brush.
- l. Deionized or distilled water.
- m. Direct indicating viscometer: As described in API Recommended Practice 13B-1, Section entitled "Viscosity and Gel Strength."
- n. Timer: Interval, mechanical or electrical, precision to ± 0.1 minutes.

5.3 PROCEDURE

5.3.1 Prepare a base suspension by adding 15.0 \pm 0.1 g API Reference Bentonite to 350 \pm 2.5 cm³ distilled water in a container while stirring on the base suspension mixer. At least two of these suspensions will be needed for the test.

5.3.2 After about 5 minutes stirring time on the mixer, remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any clay adhering to the container. Be sure all clay clinging to the spatula is incorporated into the suspension.

5.3.3 Replace the container on the mixer and continue stirring for about 15 minutes. The container should be removed from the mixer and the side scraped to dislodge any clay clinging to the container walls at 5-minute intervals. Total stirring time after adding the clay should be 20 minutes.

5.3.4 Pour the base suspension into the cup provided with the direct indicating viscometer. Note the dial reading at the 600 rpm, rotor speed setting when it reaches a constant value. The 600 rpm dial reading should not exceed 10 at a test temperature of 77 \pm 2°F (25 \pm 1°C). If necessary, the 600 rpm dial reading of the base suspension can be lowered by adding a small amount of water.

5.3.5 Pour 300 \pm 2.5 cm³ of base suspension into the mixing container (use 280 \pm 2.5 cm³ when testing barite).

5.3.6 Add 300 \pm 1 g of hematite into the base suspension in the test jar.

5.3.7 Immediately prior to use, clean the blade by washing with detergent and a small brush. Rinse thoroughly and dry. Weigh a freshly cleaned and dried abrasion test blade to the nearest 0.1 mg. Record as **W₁** in milligrams (mg).

5.3.8 Disconnect the mixer power cord to avoid accidental operation during blade installation. Center and fasten the abrasion test blade with the waves into the downward position onto the abrasion mixer spindle with the lock washer and screw.

5.3.9 With Multimixer off, place the mixing container into position on the mixer, such that the rim engages with trip switch. Start the Multimixer using a series of quick on-off flips of the switch so as to bring the spindle up to speed gently. (An abrupt start will sling some suspension out of the cup.)

Note: Blades should be polished after manufacture to remove rough edges and blades should be installed on mixer with waves in downward position.

5.3.10 Run the test for 20 \pm 0.1 min.

5.3.11 Turn the mixer off, disconnect the power cord, remove the mixing container and the abrasion test blade.

5.3.12 Clean and dry the abrasion test blade and weigh to the nearest 0.1 mg. Record as **W₂** in milligrams (mg).

5.4 CALCULATION

$$\text{Abrasion, mg/min} = \frac{W_1 - W_2}{20} \quad (8)$$

where

W_1 = initial blade weight, mg,

W_2 = final blade weight, mg.

5.5 TEST PRECISION

The test precision for this procedure has been determined to be:

Repeatability
Within-Lab
 $r=0.45$

Reproducibility
Between-Lab
 $R=0.78$

where

r = The maximum expected difference between two test results on the same sample by the same lab at the 95% confidence level, and

R = The maximum expected difference between test results by two labs on the same sample at the 95% confidence level.

Because this test is not an absolute measurement, test results should be evaluated cautiously. Best evaluations are obtained by comparing sample test results to test results of a hematite of proven performance rather than using the test result value obtained as an absolute.

6 Standard Method for the Determination of Mercury in Drilling Fluid Barite

6.1 DESCRIPTION

6.1.1 This method covers the determination of mercury (Hg) in drilling fluid barite. The cold vapor atomic absorption technique is used for the analysis following sample digestion and oxidation to ensure that most of the Hg in the sample is dissolved in aqueous media and converted to the mercuric ion.

6.1.2 The cold vapor atomic absorption technique is based on the absorption of light energy at 253.7nm by Hg vapor. Hg is reduced to the elemental state and purged from solution in a closed system. The Hg vapor passes through a cell positioned in the light path of an Hg source lamp. Absorbance (peak height) is measured as a function of Hg content.

6.2 EQUIPMENT

The following equipment is needed:

a. Atomic absorption spectrophotometer: Any atomic absorption unit equipped with background compensation having an open sample presentation area in which to mount the absorption cell is suitable. Instrument settings recommended by the particular manufacturer should be followed.

Note: Instruments designed specifically for the measurement of mercury using the cold vapor technique are commercially available and may be substituted for the atomic absorption spectrophotometer.

b. Mercury hollow cathode or electrodeless discharge lamp.

c. Recorder: any multi-range variable speed recorder compatible with the UV detection system is suitable.

d. Absorption cell: standard spectrophotometer cells 3.9 in. (100 mm) long, having quartz end windows may be used. The cell is attached to a burner for support and aligned in the light beam to give maximum transmittance.

Note: Suitable cells may be constructed from glass tubing 1 in. OD x 3.9 in. length (25 mm OD x 100 mm length) with quartz windows 1 in. diameter x 0.06 in. thickness (25 mm diameter x 1.6 cm thickness) cemented in place at the ends. Gas inlet and outlet ports (also of glass but 0.3 in. OD (0.64 cm OD) are attached approximately 0.5 in. (1.3 cm) from each end.

Note 2: Longer cells (e.g., 11.9 in. (30 cm)) are common to separable Hg systems and provide lower detection limits.

e. Gas source: nitrogen or argon.

f. Flowmeter: capable of measuring a gas flow of approximately 1 liter per minute.

g. Aeration tubing: a straight glass frit having a coarse porosity. Clear flexible plastic tubing is used for passage of the Hg vapor from the sample bottle to the absorption cell and return.

h. Drying tube: 5.9 in. x 0.8 in. (15 cm x 2 cm) diameter tube containing 20 g of magnesium perchlorate with glass wool packed at each end.

i. Reaction bottle: 250 to 300 cm³ container fitted with ground glass joint. (A gas washing bottle best serves as a reaction bottle.) The complete Hg analysis apparatus is shown in Figure 4.

j. Digestion vessel: 250 cm³ flask with a ground glass joint fitted with a water-cooled condenser.

k. Filtration cell: any apparatus capable of filtering the digested sample through #40 or #42 Whatman filter paper, or equivalent.

1. Distilled-deionized water: Reagent grade or prepared by passing distilled water through a series of cation and anion exchange resins. Use distilled-deionized water for preparation of all reagents and calibration standards and as dilution water.

Note: Special reagents low in mercury are available for the procedure described below.

m. Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated hydrochloric acid (HCl) to one volume of concentrated nitric acid (HNO₃).

n. Hydrochloric acid, 1.2 N (CAS #7647-01-0): Add 100 cm³ of concentrated HCl to 500 cm³ of water in a 1-liter volumetric flask Dilute to 1 liter with water.

o. Stannous chloride: Add 10 g stannous chloride (SnCl₂) to 50 cm³ of 1.2 N HCl in a 100 cm³ volumetric flask Dilute to volume with 1.2 N HCl (CAS #7772-99-8).

p. Hydroxylamine hydrochloride: 12 g of hydroxylamine hydrochloride in water and dilute to 100 cm³ with water (CAS #5470-11-1).

q. Potassium permanganate: 5% solution, w/v 5 g of potassium permanganate (KMnO₄) (CAS #7722-64-7) in 100 cm³ of water.

r. Potassium persulfate solution: Dissolve 50 g potassium persulfate (K₂S₂O₈) (CAS #7727-21-1) in water and dilute to 1 liter with hot [140°F (60°C)] water.

s. Stock Hg solution (1 cm³ = 1 mg Hg which is equivalent to a 1000 mg/L Hg standard): Dissolve 0.1354 g of mercuric chloride in 75 cm³ of water. Add 10 cm³ of concentrated HNO₃ and adjust the volume to 100 cm³.

Note: Commercial stock Hg solutions (1000 µg/cm³ or 1000 mg/L) are available and can be used as an alternative to preparing the stock solution.

t. Intermediate Hg solution (1 cm³ = 10 µg Hg which is equivalent to a 10 µg/cm³ or 10 mg/L Hg standard): Pipette 1 cm³ stock Hg solution into a 100 cm³ volumetric flask and bring to volume with water containing 10 cm³ concentrated HNO₃ Per liter. Solution should be stable for several weeks.

Note: Store lower concentration Hg standards (≤ 10 µg/cm³) in glass to avoid losses/gains of Hg by exchange with atmosphere.

u. Working Hg solution (1 cm³ = 0.1 µg which is equivalent to 0.1 µg/cm³ or 0.1 mg/L, Hg standard): Pipette 1 cm³ of the intermediate Hg solution into a 100 cm³ volumetric flask and bring to volume with water containing 10 cm³ concentrated HNO₃ per liter.

6.3 PROCEDURE-PREPARATION OF STANDARDS

Note: Ten times higher concentration standards are required with some Hg systems.

a. 0.005 (µg Hg/cm³): Place 5 cm³ of Hg working solution (see 6.2u) in a 100 cm³ volumetric flask and dilute to the mark with 1.2 N HCl.

b. 0.010 (µg Hg/cm³): 10 cm³ of Hg working solution and dilute as in 6.3a.

c. 0.020 (µg Hg/cm³): 20 cm³ of Hg working solution and dilute as in 6.3a.

d. 0.050 (µg Hg/cm³): 50 cm³ of Hg working solution and dilute as in 6.3a.

6.4 PROCEDURE-SAMPLE DIGESTION

Note: Extreme care should be taken to prevent loss of mercury during the digestion step.

6.4.1 Weigh a 2.00 g sample and place in the 250 cm³ flask.

6.4.2 Add 40 cm³ aqua regia, 15 cm³ KMnO₄, and 8 cm³ K₂S₂O₈ to flask and reflux for 1 hour using water-cooled condenser in a fume hood. Cool.

6.4.3 Add 6 cm³ of the hydroxylamine hydrochloride solution to reduce excess permanganate as evidenced by a loss of color.

6.4.4 Allow solids to settle and filter through Whatman #40 or #42 filter paper into a 100 cm³ volumetric flask. Wash digestion flask and residue several times into filter with water. Dilute to mark with water.

6.4.5 Prepare a procedural blank by carrying out 6.4.2 through 6.4.4 without sample.

6.5 PROCEDURE-CHECK FOR RECOVERY OF HG DURING DIGESTION

6.5.1 Transfer a 10 cm³ aliquot of the working solution (6.2u), containing 1.0 µg of Hg to one of the 250 cm³ digestion flasks.

6.5.2 Treat as in 6.4.2 through 6.4.4.

6.6 PROCEDURE-ANALYSIS OF STANDARDS AND SAMPLES

CAUTION: Because of the toxic nature of mercury vapor, precaution must be taken to avoid its inhalation. Therefore, a trap has been included in the system to pass the vapor through an absorbing medium, such as equal volumes of 0.1 N KMnO₄ and 10% H₂SO₄.

6.6.1 Turn on Hg system, adjust airflow and zero instrument according to manufacturer's specifications.

Note: Each Hg analysis system will have a slightly different physical arrangement and methodology. The system discussed below (and shown in Figure 4) serves as one useful example of a single pass arrangement.

6.6.2 Place 5 cm³ of stannous chloride solution in reaction bottle containing 100 cm³ water and allow purge gas to pass through the aeration apparatus and the absorption cell (Figure 4) until no absorbance signal is observed.

6.6.3 Divert gas flow with two-way valve and place 1 cm³ of standard, prepared in 6.3a, in reaction bottle, wait 1 minute, and then let purge gas run until a peak is observed on the recorder and the signal subsequently returns to zero. Rinse flask with 1.2 N HCl and then water between analyses. Repeat with each standard prepared in 6.3b through 6.3d.

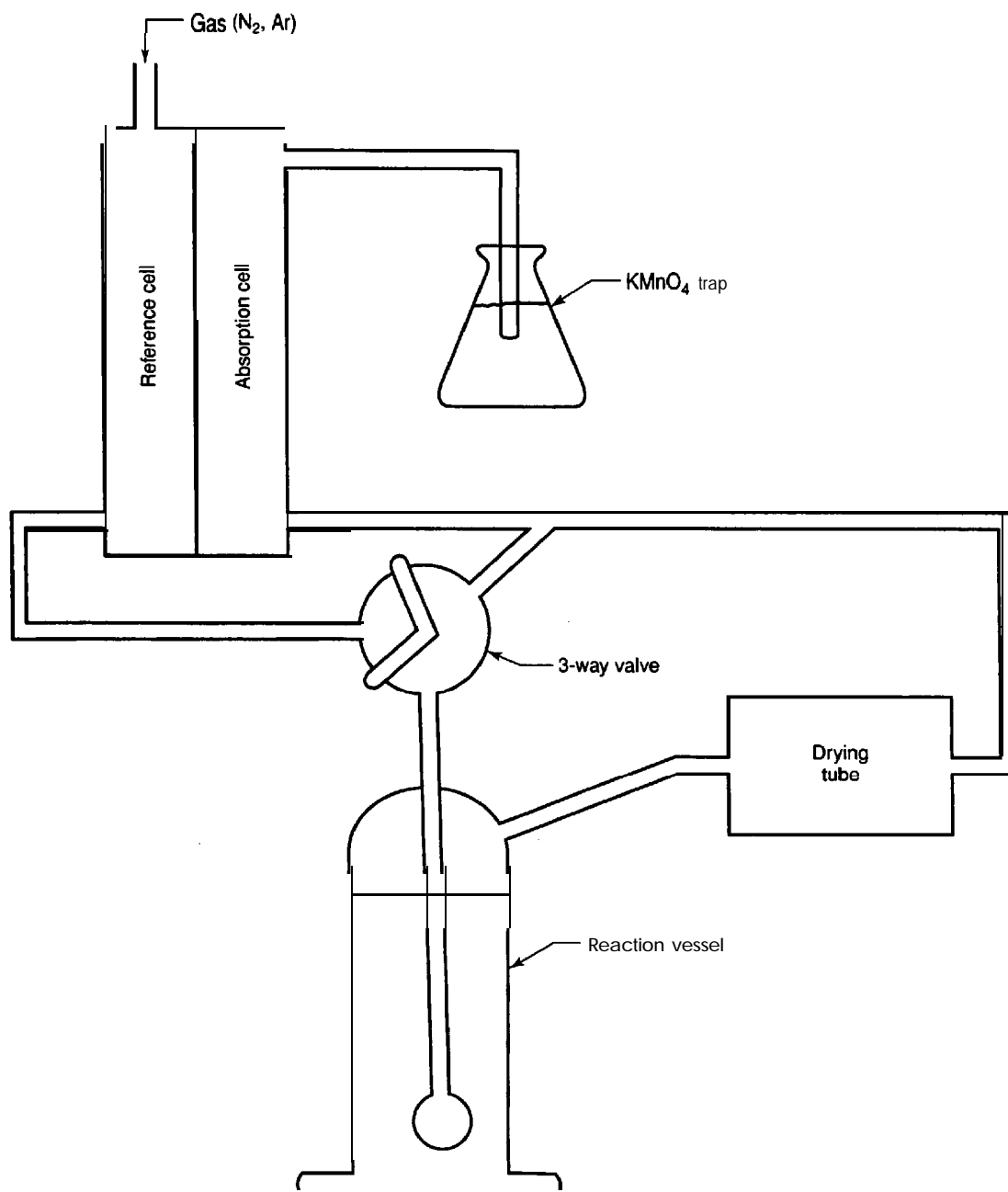


Figure 4-A Common System for Cold-Vapor Hg Analysis

Note: **With** some Hg systems, it may be necessary to take a larger aliquot of **the** standard for Hg analysis or use higher concentration standards.

6.6.4 Repeat 6.6.2 and 6.6.3 for each sample using 0.5 to 5 cm^3 aliquots (from the 100 cm^3 total).

Note: With some Hg systems, it may be necessary to use a larger sample aliquot.

6.6.5 Also analyze 1 cm^3 of the sample prepared for Hg recovery check in 6.5. Absorbance for that sample should be at least 95% of that for the standard of 6.3b.

6.7 CALCULATION

6.7.1 Following analysis of the standards, construct a standard curve by plotting peak height versus micrograms of mercury (0; 0.005; 0.010; 0.02; 0.05; 0.10 μg).

Note: With some Hg systems, **the** standard curve may use 0.05, 0.1, 0.2, 0.5 and 1.0 μg Hg values.

6.7.2 Measure the peak height of the unknown and read **the** mercury value from the standard curve.

6.7.3 Calculate the mercury concentration in the sample by the formula:

Mercury, $\mu\text{g/g}$ = (9)

$$\frac{(\text{Hg in digested sample, } \mu\text{g})(100 \text{ cm}^3 \text{ solution})}{(\text{sample weight, g})(\text{sample aliquot, cm}^3)}$$

7 Standard Method for the Determination of Cadmium and the Lead in Drilling Fluid Barite

7.1 DESCRIPTION

7.1.1 This method covers the determination of cadmium (Cd) and lead (Pb) in drilling fluid barite. Atomic absorption spectroscopy (AA) is **used** for analysis following sample digestion to ensure that most of the Cd and Pb in the sample are dissolved in an aqueous medium.

7.1.2 The aqueous sample containing the dissolved Cd and Pb ions is atomized and aspirated into a flame. During separate analyses, light beams of wavelength 228.8 nm for Cd and 283.3 nm for Pb are passed through the flame into a **mono**-chromator and onto a detector that measures the amount of light absorbed. The light energy absorbed in the flame is a measure of the concentrations of Cd and Pb in the sample.

7.2 EQUIPMENT

The following equipment is needed:

a. Atomic absorption spectrophotometer: Any commercial atomic absorption unit having an energy source, an atomizer

burner system, a **monochromator**, a detector and background compensation is suitable. Instrument settings recommended by the particular manufacturer should be followed.

b. Cd and Pb hollow cathode or electrodeless discharge lamps.

c. Fuel: commercial grade acetylene is generally acceptable.

d. Oxidation: air which may be supplied from a compressed air line, a laboratory compressor, or from a cylinder of compressed air.

e. Digestion vessel: 250 cm^3 flask with a ground-glass **joint**, fitted with a water-cooled condenser.

f. Filtration cell: any apparatus capable of filtering the digested sample through **Whatman #40** or **#42** filter paper, or equivalent.

g. Distilled-deionized water: reagent grade or prepared by passing distilled water through a series of cation and anion exchange resins. Use distilled-deionized water for the preparation of all reagents and calibration standards and as dilution water.

h. Hydrochloric acid (1 + 1) (CAS #7647-01-0): Add 1 volume concentrated hydrochloric acid (HCl) to 1 volume water.

i. Nitric acid (HNO_3) concentrated. Reagent grade (CAS #7697-37-2).

j. Stock Cd solution (1 cm^3 = 0.1 mg which is equivalent to a 100 $\mu\text{g}/\text{cm}^3$ or 100 mg/L standard): Weigh 0.2744 g of cadmium nitrate ($\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, analytical reagent grade), dissolve in 200 cm^3 water in a 1 liter volumetric flask. Add 20 cm^3 HCl (1 + 1) and dilute to mark with water.

k. Stock Pb solution (1 cm^3 = 1 mg Pb which is equivalent to a 1000 $\mu\text{g}/\text{cm}^3$ or 1000 mg/L standard): Weigh 1.599 g lead nitrate ($\text{Pb}(\text{NO}_3)_2$, analytical reagent grade), dissolve in 200 cm^3 of water, add 10 cm^3 HNO_3 (concentrated) and dilute to 1 liter with water.

Note: Commercial stock Cd and Pb (1000 $\mu\text{g}/\text{cm}^3$ or 1000 mg/L) solutions are available and recommended as an alternative to preparing **the** stock solution. For Cd, **the** 100 $\mu\text{g}/\text{cm}^3$ stock standard (7.2j) can be prepared by placing 100 cm^3 of the 1000 $\mu\text{g}/\text{cm}^3$ standard in a 1 liter volumetric flask, adding 20 cm^3 of HCl (1 + 1) and diluting to mark **with** water.

7.3 PROCEDURE-PREPARATION OF COMBINED CADMIUM AND LEAD STANDARDS

a. 0.1 $\mu\text{g}/\text{cm}^3$ Cd, 1 $\mu\text{g}/\text{cm}^3$ Pb: Transfer 1 cm^3 aliquots each of the Cd and the Pb stock solutions to a 1 liter volumetric flask, fill about $1/2$ full with distilled-deionized water, add 10 cm^3 HCl (1 + 1) and dilute with water to 1 liter and store in acid washed plastic bottles.

b. 0.2 $\mu\text{g}/\text{cm}^3$ Cd, 2 $\mu\text{g}/\text{cm}^3$ Pb: Use 2 cm^3 aliquots of each stock solution and repeat dilution as in 7.3a.

c. 0.5 $\mu\text{g}/\text{cm}^3$ Cd, 5 $\mu\text{g}/\text{cm}^3$ Pb: Use 5 cm^3 aliquots of each stock solution and repeat dilution as in 7.3a.

d. 1 $\mu\text{g}/\text{cm}^3$ Cd, 10 $\mu\text{g}/\text{cm}^3$ Pb: Use 10 cm^3 aliquots of each stock solution and repeat dilution as in 7.3a.

e. 2 $\mu\text{g}/\text{cm}^3$ Cd, 20 $\mu\text{g}/\text{cm}^3$ Pb: Use 20 cm^3 aliquots of each stock solution and repeat dilution as in 7.3a.

Note: All standards should be stored in polyethylene bottles and should be stable for several months.

f. Prepare an acid blank by the above procedure without adding Cd or Pb stock solution.

7.4 PROCEDURE--SAMPLE DIGESTION

7.4.1 Weigh out a 10 g or smaller sample and place in the 250 cm^3 flask.

7.4.2 Add 50 cm^3 (1 + 1) HCl to the flask and reflux for 1 hour using the water-cooled condenser.

7.4.3 Allow flask and contents to cool.

7.4.4 Let solids settle and decant through a Whatman #40 or #42 filter (or equivalent) into a 100 cm^3 volumetric flask. Wash residue and digestion flask with water, let settle, and decant through filter. Dilute to mark with water. Aliquots of the well-mixed, filtered digest solution may be stored in smaller plastic containers for several weeks.

7.4.5 Prepare a procedural blank by performing 7.4.1 through 7.4.4 without sample.

Note: No losses of Cd or Pb were found during acid digestion.

7.5 PROCEDURE-ANALYSIS OF STANDARDS AND SAMPLES

Note: One alternate procedure for determining Cd or Pb in the extract is using a graphic furnace, or flameless atomic absorption spectrophotometry. This process uses a heated graphite tube to rapidly volatilize the Cd or Pb containing solution; the vapor thus generated is measured for Cd or Pb using an atomic absorption spectrophotometer.

Note 2: A second alternate method for determining Cd or Pb in the digest solution is using a plasma spectrophotometer. These are available as either direct current plasma (DCP) or inductively coupled plasma (ICP). These instruments aspirate the extract into the plasma, where the solution is volatilized. The Cd or Pb atoms thus generated are excited to a high energy level by the plasma, and then emit radiation which is characteristic of the atomic structure of Cd or Pb. This radiation is then separated from other emitted radiation by a monochromator and is measured quantitatively by a photomultiplier.

7.5.1 Turn on atomic absorption instrument and set up according to manufacturer's instructions for flame atomization for Cd (228.8 nm).

7.5.2 Aspirate each of the standards in the AA and record absorbances and Cd concentration of standard. Aspirate water between each standard. Repeat until each of the standards gives a uniform absorbance.

7.5.3 Proceed with aspiration of acid (7.3f) and procedural blanks (7.4.5) and samples, recording absorbances, again aspirating water between each sample.

7.5.4 Rerun the standards after every 6 to 10 samples and at the conclusion of the sample set.

7.5.5 Repeat 7.5.1 through 7.5.4 for Pb at 283.3 nm.

7.6 CALCULATION

7.6.1 Prepare separate calibration curves for Cd and Pb by plotting absorbance versus concentration of each standard.

7.6.2 Using the sample absorbances and the Cd and Pb graphs from 7.6.1, separately determine the digest solution Cd and Pb concentrations, subtracting any procedural blank (should be essentially zero).

7.6.3 Calculate the Cd and Pb concentrations in the sample by the formula:

$$\text{Cadmium, } \mu\text{g/g} = \quad (10)$$

$$\frac{\text{Cd in digested sample, } \mu\text{g}/\text{cm}^3 (100 \text{ cm}^3 \text{ solution})}{\text{Sample weight, g}}$$

$$\text{Lead, } \mu\text{g/g} = \quad (11)$$

$$\frac{\text{Pb in digested sample, } \mu\text{g}/\text{cm}^3 (100 \text{ cm}^3 \text{ solution})}{\text{Sample weight, g}}$$

8 Standard Method for the Determination of Arsenic in Drilling Fluid Barite

8.1 DESCRIPTION

8.1.1 This method covers the determination of arsenic (As) in drilling fluid barite. The gaseous hydride atomic absorption method is used for analysis following sample digestion and reduction to ensure that most of the As in the sample is dissolved in aqueous media and converted to the trivalent form.

8.1.2 The gaseous hydride method is based on the absorption of light energy at 193.7 nm by As. As in the trivalent form is converted to gaseous arsine (AsH_3) using sodium borohydride (NaBH_4) in an acid medium. The gaseous hydride is swept through a heated quartz tube or into an argonhydrogen flame of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of As concentration.

Note: An alternate procedure for determining As in the extract is flameless atomic absorption spectrophotometry. This process uses a heated graphite tube to rapidly volatilize the As-containing solution; the vapor thus generated is measured for As using the atomic absorp

tion spectrophotometer. This method of analysis eliminates the need for the **arsine** generator, and is also more sensitive to As. Graphite furnaces are commercially available from most manufacturers of atomic absorption instruments; instrument settings for As recommended by the manufacturer should be followed, however, slight modifications in these settings may be needed to optimize the sensitivity and reproducibility of As analysis.

8.2 EQUIPMENT

The following equipment is needed:

a. Atomic absorption spectrophotometer: any commercial atomic absorption unit having an energy source, a heated quartz tube (preferred) or an atomizer burner system, a **monochromator**, a detector, and background compensation is suitable. Instrument settings for As recommended by the manufacturer should be followed.

Note: With the heated quartz tube, hydride generation system, **background** correction was not found to be required.

b. Arsenic hollow cathode or electrodeless discharge lamp.
c. Arsine generator: a reaction flask of about 12 cm^3 capacity fitted with inlet-outlet tubes for argon flow through the solution and a device for adding sodium borohydride in a closed system.

Note: Arsine generators are available commercially from instrument manufacturers and may have slightly different configurations from that described above.

d. Digestion vessel: 25 cm^3 PTFE-lined digestion bomb. Such bombs, which are commercially available from several manufacturers, should be leak-proof and air tight when sealed, and should be capable of withstanding operating temperatures of at least 230°F (110°C) and pressures of at least 200 psi. Typical capacities are in the range of 21 to 25 cm^3 .

e. Heating apparatus: water bath controllable to 212 $\pm 2^\circ\text{F}$ (100 $\pm 1^\circ\text{C}$).

f. **Filtration** cell: any apparatus capable of filtering the digested sample through #40 or #42 **Whatman** filter paper, or equivalent.

g. Distilled-deionized water: reagent grade or prepared by passing distilled water through a series of cation and anion exchange resins. Use distilled-deionized water for the preparation of all reagents and calibration standards and as dilution water.

h. Nitric acid (sp. gr. 1.42): concentrated redistilled nitric acid (**HNO₃**). Use analytical grade with an arsenic content not greater than 1×10^{-6} percent (10 μg As/L).

i. Nitric acid (3.2 N): dilute 200 cm^3 of concentrated **HNO₃** to 1 liter with water (CAS #7697-37-2).

j. Potassium iodide solution (150 g/L): dissolve 15 g of potassium iodide (KI) (CAS #7681-11-0) in 100 cm^3 of water. Store in amber bottle.

k. Potassium thiocyanate solution (50 g/L): dissolve 5 g of potassium thiocyanate (KSCN) (CAS #333-20-0) in 100 cm^3 of water.

1. Sodium borohydride pellets: (CAS #16940-66-2) 0.8 cm pellets weighing about 0.25 g each, or,

Sodium borohydride solution: dissolve 30 g **NaBH₄** in a 1% **NaOH** solution and dilute to 1 liter in a volumetric flask.

m. Stock As solution (1 cm^3 = 1 mg As, which is equivalent to 1000 $\mu\text{g}/\text{cm}^3$ or 1000 mg/L As): dissolve 1.320 g arsenic trioxide (**As₂O₃**) in 100 cm^3 water containing 4 g **NaOH** and dilute to 1 liter with water.

Note: Commercial stock As solutions (1000 $\mu\text{g}/\text{cm}^3$) are available and recommended as an alternative to preparing the stock solution.

n. Intermediate As solution (1 cm^3 = 10 μg As, which is equal to 10 $\mu\text{g}/\text{cm}^3$ or 10 mg/L As): Pipette 1 cm^3 of stock arsenic solution into a 100 cm^3 volumetric flask and bring to volume with 3.2 N **HNO₃**.

o. Working As solution (1 cm^3 = 1 μg As, which is equivalent to 1 $\mu\text{g}/\text{cm}^3$ or 1 ppm As): Pipette 10 cm^3 intermediate arsenic solution into a 100 cm^3 volumetric flask and bring to volume with 3.2 N **HNO₃**.

8.3 PROCEDURE-PREPARATION OF STANDARDS

a. 0.025 μg As/ cm^3 : Transfer 2.5 cm^3 of the working As solution (see 8.20) to a 100 cm^3 volumetric flask and bring to volume with 3.2 N **HNO₃**.

b. 0.05 μg As/ cm^3 : Use 5 cm^3 of working As solution and dilute as in 8.3a.

c. 0.075 μg As/ cm^3 : Use 7.5 cm^3 of working As solution and dilute as in 8.3a.

d. 0.10 μg As/ cm^3 : Use 10 cm^3 of working As solution and dilute as in 8.3a.

8.4 PROCEDURE-SAMPLE DIGESTION

8.4.1 Weigh a 250 mg sample and place in the 25 cm^3 Teflon (**TFE**) bomb. Add 10 cm^3 concentrated **HNO₃**, tightly stopper the bomb, and heat for 1.5 hours in the water bath at 176°F (80°C).

8.4.2 Cool for 1 hour at ambient temperature.

8.4.3 Reheat for 1.5 hours at 176°F (80°C).

8.4.4 Cool to ambient temperature.

8.4.5 Remove cover carefully and decant into a 50 cm^3 beaker.

8.4.6 Rinse the bomb and lid several times with water and add rinse to the beaker.

8.4.7 Add water to the beaker to bring volume to about 40 cm^3 .

8.4.8 Filter through a **Whatman** #40 or #42 filter (or equivalent) into a 50 cm^3 volumetric flask. Wash the beaker with small portions of water into the filter. Bring flask to volume with water.

8.4.9 To obtain a procedural blank, repeat 8.4.1 through 8.4.8 without any sample.

8.5 PROCEDURE-ANALYSIS OF STANDARDS AND SAMPLES

CAUTION: To minimize As absorption losses on glassware, run the analysis on the standards and sample immediately upon preparation.

8.5.1 **Turn** on atomic absorption instrument and set up according to manufacturer's directions.

8.5.2 For each analysis, add 1 cm^3 aliquots of the standard solutions (8.3) and blanks (8.4.9) to the arsine generator. Add 2 cm^3 of the KSCN solution 2 cm^3 of the KI solution and 15 cm^3 of 3.2 N HNO_3 .

Note: Larger aliquots than 1 cm^3 may be required with some systems.

8.5.3 Allow 10 minutes for the As to be reduced to the trivalent state.

8.5.4 Connect the generator and add 1 NaBH_4 pellet or pump NaBH_4 solution until maximum signal is obtained according to manufacturer's specifications. Record the peak height. When the recorder **returns** to the baseline, disconnect the generator.

8.5.5 Prepare a standard curve by plotting peak height versus μg As for each standard.

8.5.6 Run sample solutions using OS to 5 cm^3 or more (from the total of 100 cm^3) in the same manner as standards (8.5.2 through 8.5.4).

8.6 CALCULATION

8.6.1 Determine the μg As in the digested sample from the calibration curve prepared in 8.5.5.

8.6.2 Calculate the As concentration in the sample by the formula:

$$\text{Arsenic, } \mu\text{g/g} = \quad (12)$$

$$\frac{(\text{As in digested sample, } \mu\text{g})(50 \text{ cm}^3 \text{ solution})}{(\text{Sample weight, g})(\text{Sample volume added to generator, cm}^3)}$$

9 Bridging Materials for Regaining Circulation

9.1 DESCRIPTION

The ability of a material to seal a lost circulation zone is of importance in the determination of the proper material. The size of the opening to be sealed or bridged varies with forma-

tion, and the particle of sealing material must be sized to adequately bridge that opening.

9.2 EQUIPMENT

The following equipment is needed:

- Test cell for testing bridging materials (see Figures 5 and 6). Fitted with the following:
 - Stainless-steel disks: 0.25 in. (6.4 mm) thick by 1.88 in. (47.5 mm) diameter. Disks have square-edged slots 1.38 in. (35 mm) in length and widths of 0.04 in. (1.0 mm); 0.08 in. (2.0 mm); 0.12 in. (3.0 mm); 0.16 in. (4.0 mm); and 0.20 in. (5.0 mm).
 - Sleeve 2.87 in. (73 mm) in diameter, 2.25 in. (57 mm) high with perforated base plate containing approximately 32 0.25 in. (6.4 mm) holes.
 - Brass or stainless-steel marbles: 95 or more, 0.56 in. (14.3 mm) in diameter (enough to just fill bed volume).
 - Brass-clad or stainless-steel BB shot: 1200 grams of shot 0.173 in. (4.39 mm) diameter and a 10-mesh stainless-steel screen 2.88 in. (73 mm) in diameter.
- Nitrogen: pressure regulated.
- Plastic container: 3500 cm^3 , fitted with inlet and outlet suitable to accommodate the sudden discharge of mud from the cell.
- Mixer: 11,000 \pm 300 rpm under load, with single corrugated impeller approximately 1 in. (25 mm) in diameter (e.g., Multimixer Model 9B with 9B29 impeller, or equivalent).
- Container for mixing: 7 in. (180 mm) deep, 3¹³/₁₆ in. (97 mm) ID at top and 2³/₄ in. (70 mm) ID at bottom (e.g., Hamilton Beach Mixer Cup No. MI 10-D, or equivalent).

9.3 PROCEDURE-PREPARATION OF BASE MUD

9.3.1 Base mud consists of 5 to 8% by weight Wyoming bentonite, aged for a minimum of 72 hours and adjusted to an Apparent Viscosity of 25 \pm 2cP after stirring 10 minutes on a Multimixer.

9.3.2 To 3500 cm^3 (10 laboratory barrels) of the base mud, add a weighed amount of the material to be tested. The additive concentration is expressed in pounds per barrel (lb/bbl).

9.4 PROCEDURE-STATIC SLOT TEST

9.4.1 Select a disk, preferably having a small slot, and place it in the valve outlet half-union with the perforated plate and sleeve used to support BB or marble beds removed from the cell.

9.4.2 Open the cylinder bleed valve and place the graduated container under the outlet.

9.4.3 Pour the mud containing the material to be tested into the cell with cell outlet valve open. Record the volume of mud which flows out.

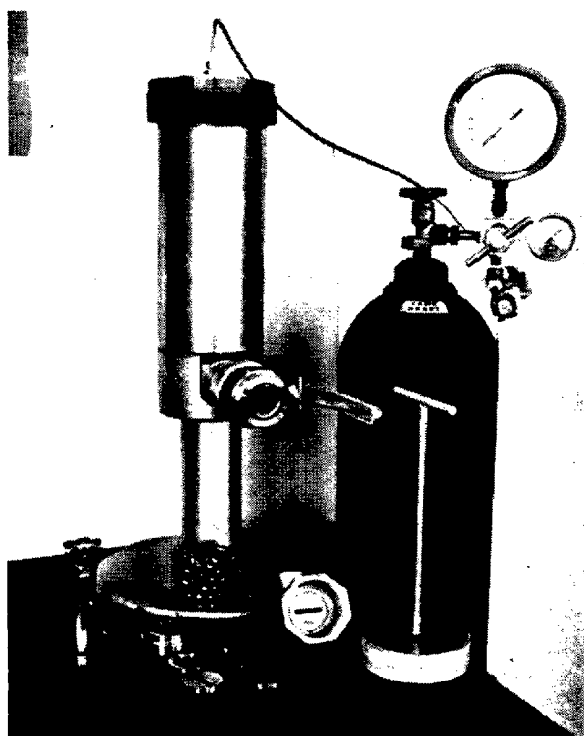


Figure 5—Bridging Material Test Equipment
(See Par. 9.2)

9.4.4 Screw the cap on the cell. The free piston may be placed on the mud in the cell, if desired.

9.4.5 Start the timer and apply pressure at a rate of 2 psi/second (13.8 kPa/sec), until a pressure of 100 psig (790 kPa) is reached. Record the volume of mud discharged. The minimum pressure at which a seal occurs may or may not be observed: if observed, it should be recorded.

9.4.6 Increase pressure at a rate of 10 psi/second (69 kPa/sec) to 1000 psi (7000 kPa), or until the seal fails and the cylinder empties. Record the volume through or the maximum pressure obtained. If a seal is obtained, maintain the pressure for 10 minutes and record the final volume.

9.4.7 Repeat the test with increasing sizes of slots until no permanent seal is achieved at 1000 psi (7000 kPa).

9.5 PROCEDURE-DYNAMIC SLOT TEST

9.5.1 Prepare base mud as described in 9.3 and apparatus as outlined in 9.4.1.

9.5.2 With the outlet valve closed, pour the test mud into the cell. The free piston may be placed on top of the mud.

9.5.3 Close the cap and set the gas regulator to deliver at the test pressure, 100 psig (790 kPa).

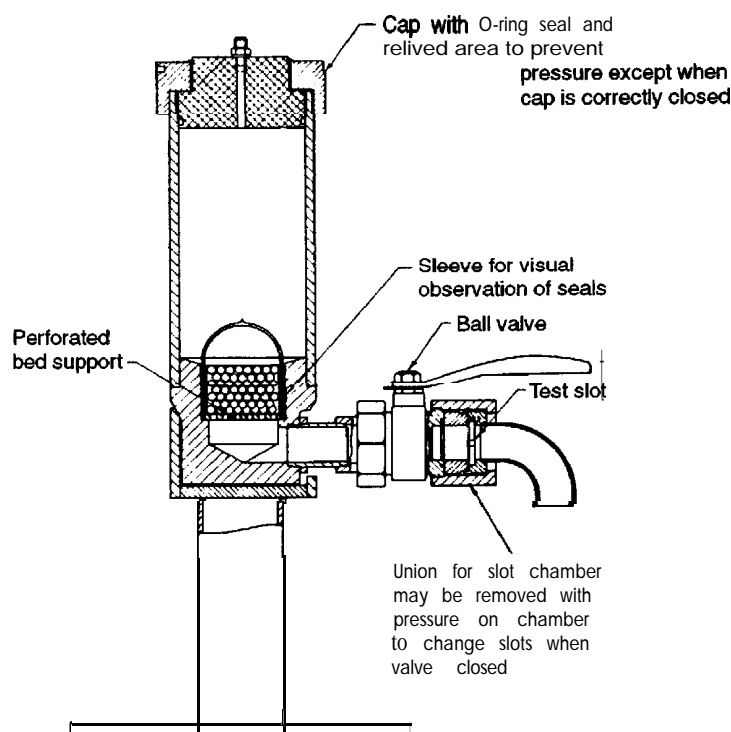


Figure 6—Bridging Material Test Cell 1000 PSIG
(7000 kPa) Working Pressure (See Par. 9.2)

9.5.4 Open the cell outlet valve and start the timer. Record the volume of mud which flows through the slot and the time required to seal.

9.5.5 Pressure can then be increased to 1000 psig (7000 kPa) at the rate of 10 psi/second and maintained for 10 minutes as in 9.4.6 (69 kPa/sec).

9.5.6 Repeat the test with increasing sizes of slots until no permanent seal is achieved.

9.6 PROCEDURE-STATIC MARBLE BED TEST

9.6.1 Prepare the marble bed by pouring the 0.56 in. (14.3 mm) marbles into the sleeve so as to form a bed 2.25 in. (57 mm) thick above the perforated plate (just to top of the container). Place the full-bore ring in the slot groove.

9.6.2 With the cell outlet valve open and the graduated container under the outlet, pour the test mud into the cell. Record the volume through the bed under the hydrostatic head.

9.6.3 Place the free piston on top of the mud and the cap on the cell. Close the cylinder bleed valve. and start the timer. Apply pressure and record results as in 9.4.5 and 9.4.6.

9.6.4 On completion of the test, release the pressure. Remove the marble bed and examine the appearance of the seal and the depth of penetration of the sealing material.

9.7 PROCEDURE-DYNAMIC MARBLE BED TEST

9.7.1 Prepare the marble bed as in 9.6.1.

9.7.2 With the cell outlet valve closed, pour untreated base mud into the cell to fill the void spaces under and within the marble bed until the top of the untreated mud is level with the top of the sleeve.

9.7.3 Carefully pour the test mud into the cell so as not to disturb the mud in the bed. The free piston may be placed on top of the mud.

9.7.4 Close the cell and apply pressure with the gas regulator adjusted to 100 psig (790 kPa). Open the cell outlet valve and start the timer. Record the volume of mud which flows through the bed and the time to obtain a seal.

9.7.5 Continue the test as in 9.4.6. After 10 minutes at 1000 psig (7000 kPa) or after failure, inspect the bed as in 9.6.4.

9.8 PROCEDURE-STATIC BB BED TEST

9.8.1 Prepare the BB shot bed by placing the 10-mesh stainless-steel screen on the perforated plate and pouring the brass-clad or stainless-steel BB shot into the sleeve so as to form a bed 2.25 in. (57 mm) thick above the screen (just to the top of sleeve). The thickness of the shot bed can be varied from a minimum of 1 in. (25 mm) to 2.25 in. (57 mm). If less than 2.25 in. (57 mm) record the thickness.

9.8.2 Place the full-bore ring in the slot groove. With the cell outlet valve open and the graduated cylinder under the outlet, pour the test mud into the cell.

9.8.3 Proceed with the test according to 9.6.2 through 9.6.4.

9.9 PROCEDURE-DYNAMIC BB BED TEST

9.9.1 Prepare the shot bed as in 9.8.1.

9.9.2 With the cell outlet valve closed, pour untreated base mud into the cell to fill the void spaces under and within the shot bed until the top of the untreated mud is level with the top of the sleeve.

9.9.3 Proceed with the test according to 9.7.3 through 9.7.5.

10 Filtration Control Agents

10.1 DESCRIPTION

10.1.1 Organic filtration control agents can vary greatly in chemical composition and their performance characteristics in different drilling fluid environments. Judgments are made by comparing performance of test additives to that of known materials that have been accepted for field use.

10.1.2 In specific cases, comparisons may be made in the actual held mud where the material will be used, but in general, evaluations are performed in laboratory prepared muds.

10.1.3 It is recommended that materials considered for broad applications be screened in different mud systems. While filtration control is the property being evaluated, the effect of the test material on rheology, pH or other properties may be of concern.

10.2 EQUIPMENT

The following equipment is needed:

- a. Balance: precision of ± 0.01 g.
- b. Sodium chloride: ACS reagent grade, or equivalent (CAS #7647-14-5).
- c. Distilled or deionized water.
- d. Mixer: 11,000 \pm 300 rpm under load, with single corrugated impeller approximately 1 in. (25 mm) in diameter (e.g., Multimixer Model 9B with 9B29X impeller, or equivalent, as shown in Figure 3. The 9B29X impeller should be replaced when approximately $\frac{1}{8}$ weight loss occurs. The original weight is about 5.5 g).
- e. Container: 7 in. (180 mm) deep, $3\frac{13}{16}$ in. (97 mm) ID top, $2\frac{3}{4}$ in. (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M 110-D or equivalent).
- f. Clock or timer.
- g. Carboy: large, covered (e.g., gallon glass jar with screw-type lid).
- h. Spatula.
- i. Attapulgate-API specification material.
- j. Simulated drilled solids, for example:
 1. Rev-Dust-available from The Milwhite Company, 5601 Clinton Drive. Houston, TX 77020.
 2. Martin's #5 Ball Clay-available from Kentucky-Tennessee Clay Company, Box 449, Mayfield, Tennessee 42066.
 3. Standard Evaluation Base Clay-request for clay should be directed to the API Upstream Segment, Washington, D.C., which will forward the request to a designated custodian for all further handling.
- k. Filter press: API low temperature-low pressure, as described in API Recommended Practice 13B-1, Section entitled "Filtration."

Note: The API Low Temperature/Low Pressure filter press *must* have a filter area of 4520 to 4640 mm² which is a diameter of 75.86 to 76.86 mm. The filter press gasket is the determining factor of the filter area. It is recommended that the filter press gasket used to be tested by a conical gage that has the maximum (76.86 mm) and the minimum (75.86 mm) marked on it. Any filter press gasket found out of these ranges (either larger or smaller than the markings) will be discarded.

- l. Calcium chloride: ACS reagent grade, or equivalent (CAS #10043-52-4).

- m. Magnesium chloride: ACS reagent grade, or equivalent (CAS #7786-30-3).
- n. Potassium chloride: ACS reagent grade, or equivalent (CAS #7447-40-7).
- o. Xanthan gum: drilling fluid grade dry powder.
- p. Viscometer: direct indicating, as described in API Recommended Practice 13B-1, Section entitled "Viscosity and Gel Strength."
- q. API Reference Bentonite.
- r. Sea salt-available from Lake Products Company, Box 498, Ballwin, Missouri 63001, or equivalent ocean water.
- s. Sodium hydroxide solution: standardized 10 normal (10N) (CAS #1310-73-2).
- t. Lignosulfonate (chrome or ferrochrome): drilling fluid grade.
- u. Lime: ACS reagent grade or equivalent.
- v. Roller oven: regulated to $150 \pm 5^\circ\text{F}$ ($66 \pm 3^\circ\text{C}$).
- w. pH meter: as described in API Recommended Practice 13B-1, Section entitled "pH."
- x. Defoamer.
- y. Jar: small, covered (e.g., pint glass jar with screw-type lid).
- z. Oven: regulated to $220 \pm 5^\circ\text{F}$ ($105 \pm 3^\circ\text{C}$).

aa. Aging cells.

bb. Filter cell: high temperature-high pressure, as described in API Recommended Practice 13B-1, Section entitled "Filtration".

10.3 PROCEDURE-GENERAL INSTRUCTIONS FOR PREPARATION OF BASE MUDS

10.3.1 Prepare sufficient quantity of base mud for a series of tests. This can be achieved by preparing a large batch or by combining small batches for uniformity prior to aging.

10.3.2 Composition, order of addition, mixing time, shear and aging time should be consistent from batch to batch.

10.3.3 Stir all base muds prior to testing to insure uniformity.

10.3.4 Test a sample of the untreated base mud each time a new series of tests is begun. Subject a sample of the untreated base mud to the same amount of stirring, heat aging and testing as the treated muds.

10.4 PROCEDURE--SATURATED SALT MUD

10.4.1 Prepare saturated salt water by adding 400 g of sodium chloride/liter of water (to exceed the solubility of sodium chloride in water at room temperature).

10.4.2 Mix 30 minutes, age overnight at room temperature, and decant into a large covered carboy.

10.4.3 Prepare a suspension of attapulgite clay and simulated drilled solids using 8 g of attapulgite and 30 g of simu-

lated drilled solids/338 cm^3 (401 g) saturated salt water in a container. Sift the clay and solids into the water while stirring on the mixer set at high shear,

10.4.4 After five minutes, remove container from mixer and scrape sides to dislodge any clay and solids adhering to container.

10.4.5 Replace on mixer and continue stirring for an additional 25 minutes (total mixing time 30 minutes).

10.4.6 Store the suspension 24 hours in a sealed carboy at room temperature.

10.4.7 Stir the suspension for five minutes on mixer.

10.4.8 Measure the filtrate volume using a low temperature-low pressure API filtrate cell, as described in API Recommended Practice 13B-1, Section entitled "Filtration." Filtrate volume should be 90 – 105 cm^3

10.4.9 For the purpose of testing, the weight equivalent is 439 g/350 cm^3 .

10.5 PROCEDURE-HIGH HARDNESS CONTENT SATURATED SALT MUD

10.5.1 Prepare a brine with a high hardness content by adding 125 g calcium chloride and 22 g magnesium chloride/liter of saturated salt solution, as prepared in 10.4.1.

10.5.2 Mix 30 minutes, age overnight at room temperature, and decant into a large covered carboy.

10.5.3 Prepare a suspension of attapulgite clay and simulated drilled solids using 8 g of attapulgite and 30 g of simulated drilled solids/338 cm^3 (429 g) high hardness content saturated salt mud in a container. Sift the clay and solids into the water while stirring on the mixer set at high shear.

10.5.4 After five minutes, remove container from mixer and scrape sides to dislodge any clay and solids adhering to container.

10.5.5 Replace on mixer and continue stirring for an additional 25 minutes (total mixing time 30 minutes).

10.5.6 Store the suspension 24 hours in a sealed carboy at room temperature.

10.5.7 Stir the suspension for 5 minutes on mixer.

10.5.8 Measure the filtrate volume using a low temperature-low pressure API filtrate cell, as described in API Recommended Practice 13B-1, Section entitled "Filtration." Filtrate volume should be 90 – 105 cm^3 .

10.5.9 For the purposes of testing, the weight equivalent is 467 g/350 cm^3 .

10.6 PROCEDURE-10% POTASSIUM CHLORIDE MUD

10.6.1 Prepare 10% potassium chloride solution by adding 111 g of potassium chloride/liter of water in a container.

10.6.2 Sift 1.0 g of Xanthan gum slowly into 360 g (340 cm³) of 10% potassium chloride solution while stirring on a mixer set at high shear.

10.6.3 After five minutes, remove container from mixer and scrape sides to dislodge any polymer adhering to container.

10.6.4 Replace on mixer and continue stirring for an additional 10 minutes (total mixing time 15 minutes).

10.6.5 Add 30 g of simulated drilled solids while continuing to stir in a mixer set at high shear.

10.6.6 After five minutes, remove container from mixer and scrape sides to dislodge any solids adhering to container.

10.6.7 Replace on mixer and continue stirring for an additional 10 minutes (total mixing time 30 minutes).

10.6.8 Measure the rheological properties using the rheometer, as described in API Recommended Practice 13B- 1, Section entitled "Viscosity and Gel Strength."

Properties should be:

Plastic Viscosity: cP	3 - 7
Yield Point: lb/100 ft ²	6 - 10
10-second Gel: lb/100 ft ²	2 - 4
10-minute Gel: lb/100 ft ²	3 - 5

10.6.9 Measure the filtrate volume using a low temperature-low pressure API filtrate cell, as described in API Recommended Practice 13B- 1, Section entitled "Filtration." Filtrate volume should be 30 - 50 cm³.

10.6.10 For the purpose of testing, the weight equivalent is 39.1 g/350 cm³.

10.7 PROCEDURE-PREHYDRATED BENTONITE SLURRY

10.7.1 In procedures requiring prehydrated bentonite in a base mud, prepare and age the bentonite in advance.

10.7.2 Prepare a suspension of the bentonite using 26 g bentonite (as received)/350 cm³ water (6.67% by weight solids) for each sample to be tested in a container. Sift the clay into the water while stirring on the mixer set at high shear.

10.7.3 After 5 minutes, remove container from mixer and scrape sides to dislodge any bentonite adhering to container.

10.7.4 Replace on mixer and continue stirring for an additional 10 minutes (total mixing time 15 minutes).

10.7.5 Store the suspension approximately 24 hours (overnight) in a sealed carboy at room temperature.

10.7.6 Stir the suspension for 5 minutes on a mixer before using.

10.8 PROCEDURE-MODIFIED SEA WATER MUD

10.8.1 Prepare simulated sea water by adding 41.45 g sea salt/liter of water.

10.8.2 Prepare a suspension of bentonite clay using 150 g (144 cm³) of prehydrated bentonite slurry, as prepared in 10.7, in a container. Add 193 cm³ of simulated sea water, as prepared in 10.8.1, while stirring on the mixer set at high shear.

10.8.3 Continue stirring while adding 2.5 cm³ of 10 N NaOH and sift in 30 g of simulated drilled solids.

10.8.4 After 5 minutes, remove container from mixer and scrape sides to dislodge any clay and solids adhering to container.

10.8.5 Replace on mixer and continue stirring for an additional 10 minutes (total mixing time 15 minutes.)

10.8.6 Store the suspension 24 hours in a sealed carboy at room temperature.

10.8.7 Stir the suspension for 5 minutes on mixer.

10.8.8 Measure the filtrate volume using a low temperature-low pressure API filtrate cell, as described in API Recommended Practice 13B- 1, Section entitled "Filtration." Filtrate volume should be 60 - 70 cm³.

10.8.9 For the purpose of testing, the weight equivalent is 382 g/350 cm³.

10.9 PROCEDURE-LOW SALINITY MUD

10.9.1 Prepare low salinity solution by adding 42 g sodium chloride/liter of water (4% sodium chloride brine).

10.9.2 Prepare a suspension of bentonite clay using 150 g (144 cm³) of prehydrated bentonite slurry, as prepared in 10.7, in a container. Add 193 cm³ of low salinity water to the suspension of bentonite while stirring on the mixer set on high shear.

10.9.3 Continue stirring while adding 2.5 cm³ of 10 N NaOH and sift in 30 g of simulated drilled solids.

10.9.4 After 5 minutes, remove container from mixer and scrape sides to dislodge any clay and solids adhering to container.

10.9.5 Replace on mixer and continue stirring for an additional 10 minutes (total mixing time 15 minutes).

10.9.6 Store the suspension 24 hours in a sealed **carboy** at room temperature.

10.9.7 Stir the suspension for 5 minutes on mixer.

10.9.8 Measure the filtrate volume using a low temperature-low pressure API filtrate cell, as described in API **13B-1**, Section entitled "Filtration." Filtrate volume should be 35 – 45 cm^3 .

10.9.9 For the purpose of testing, the weight equivalent is 382 g/350 cm^3 .

10.10 PROCEDURE-LIME TREATED MUD

10.10.1 Prepare a suspension of bentonite clay using 260 g (250 cm^3) of prehydrated bentonite slurry, as prepared in Par. 10.7, in a container. Add 73 cm^3 of water, 4 g of chrome lignosulfonate and 2.5 cm^3 of 10 normal (10N) **NaOH** while stirring on the mixer set at high shear.

10.10.2 Stir 30 minutes.

10.10.3 Continue stirring and sift in 3 g of **lime** and 30 g of simulated drilled solids.

10.10.4 After 5 minutes, remove container from mixer and scrape sides to dislodge any clay and solids adhering to container.

10.10.5 Replace on mixer and continue stirring for an additional 10 minutes (total mixing time 45 minutes).

10.10.6 Roll the suspension 16 hours in a sealed **carboy** in an oven set at 150°F (65°C).

10.10.7 Stir the suspension for 5 minutes on mixer.

10.10.8 Measure the filtrate volume using a low **temperature-low** pressure API filtrate cell, as described in API Recommended Practice **13B-1**, Section entitled "Filtration." Filtrate volume should be 12 – 20 cm^3 .

10.10.9 Measure the P_m of the mud using the procedure in API Recommended Practice **13B-1**, Section entitled "Chemical Analysis." The P_m of the mud should be 10 – 20 cm^3 .

10.10.10 For the purpose of testing, the weight equivalent is 373 g/350 cm^3 .

10.11 PROCEDURE-LOW SOLIDS, **NON-DISPERSED** MUD

10.11.1 Prepare a suspension of bentonite clay using 150 g (144 cm^3) of **prehydrated** bentonite **slurry**, as prepared in 10.7, in a container. Add 200 cm^3 of water and 15 g of simulated drilled solids while stirring on the mixer set at high shear.

10.11.2 After 5 minutes, remove container from mixer and scrape sides to dislodge any clay and **solids** adhering to container.

10.11.3 Replace container on mixer and continue stirring for an additional 10 minutes (total mixing time 15 minutes).

10.11.4 Store the suspension for 24 hours in a sealed **carboy** at room temperature.

10.11.5 Stir the suspension for 5 minutes on mixer.

10.11.6 Measure the filtrate volume using a low temperature-low pressure API filtrate cell, as described in API Recommended Practice **13B-1**, Section entitled "Filtration." Filtrate volume should be 20 – 30 cm^3 .

10.11.7 For the purpose of testing, the weight equivalent is 365 g/350 cm^3 .

10.12 PROCEDURE-FRESHWATER **LIGNOSULFONATE** MUD

10.12.1 Prepare a suspension of bentonite clay using 225 g (216 cm^3) of prehydrated bentonite slurry, as prepared in 10.7, in a container. Add 127 cm^3 of water, 15 g of simulated drilled solids, 1 g of chrome lignosulfonate, and 1.2 cm^3 of 10 normal (10N) **NaOH** while stirring on the mixer set at high shear.

10.12.2 After 5 minutes, remove container from mixer and scrape sides to dislodge any clay and solids adhering to container.

10.12.3 Replace on mixer and continue stirring for an additional 10 minutes (total mixing time 15 minutes).

10.12.4 Store the suspension 24 hours in a sealed **carboy** at room temperature.

10.12.5 Stir the suspension for 5 minutes on mixer.

10.12.6 Measure the filtrate volume using a low temperature-low pressure API filtrate **cell**, as described in API Recommended Practice **13B-1**, Section entitled "Filtration." Filtrate volume should be 9 – 15 cm^3 .

10.12.7 Measure the **pH** of the suspension using a **pH** meter or strips, as described in API Recommended Practice **13B-1**, Section entitled, "**pH**." **pH** should be 9.5 – 10.5.

10.12.8 For the purpose of testing, the weight equivalent is 369 g/350 cm^3 .

10.13 PROCEDURE-INITIAL PERFORMANCE TEST

10.13.1 Select concentrations of filtration control agent so that a wide range can be covered (e.g., 0.1, 0.5, 1.0, 3.0 and 5.0 g/350 cm^3). Include an untreated sample of the base mud.

Note: If a comparison to a known field-accepted material is being made performance tests should be run on equivalent range of known field-accepted material concentrations.

10.13.2 Weigh into each of several mixing containers the weight equivalent of 350 cm³ of the desired base mud. Place the containers on the mixer set at high shear.

10.13.3 Weigh out the desired amounts of filtration control agents and add uniformly over a period of 30 seconds while stirring on the mixer.

10.13.4 Mix 30 minutes removing the containers twice to scrape sides to dislodge any clay and solids adhering to container.

Note: If foaming occurs while stirring and remains when the sample is removed from the mixer, attempt to remove the entrained air by stirring slowly with a spatula. Add defoamer if necessary. Defoamer additions can change filtration results. Record the amount of defoamer added.

10.13.5 Measure the pH of the suspensions, by procedure described in API Recommended Practice 13B-1, Section entitled "Filtration." If the pH is lower than the untreated test mud by more than 0.5 pH units, adjust the pH to that of the untreated test mud with 10 normal (1 ON) NaOH. If the pH is higher than the untreated test mud, record the pH, but do not make any adjustment.

10.13.6 Determine the low temperature-low pressure API filtrate volume, as described in API Recommended Practice 13B-1, Section entitled "Filtration." Record the filtrate volume, cake thickness, and cake consistency.

Note: The API filtrate volume should be run immediately after preparation.

10.14 PROCEDURE-PERFORMANCE AFTER HEAT AGING

10.14.1 Dynamically age the samples prepared and tested in 10.13 by rolling or rotating in sealed jars for at least 16 hours in an oven set at 150°F (65°C).

10.14.2 Cool to room temperature.

10.14.3 Measure the pH of the suspensions and adjust the pH with 10N NaOH to the initial pH as indicated in 10.135.

10.14.4 Stir for 10 minutes on the mixer set at high shear.

10.14.5 Determine the low temperature-low pressure API filtrate volume. Record the filtrate volume, cake thickness, and cake consistency.

10.14.6 If the high temperature stability is desired, age the suspensions either statically or dynamically at the higher temperature.

10.14.7 Cool to room temperature. Stir 5 minutes on the mixer set at high shear.

10.14.8 Determine the high temperature-high pressure filtrate volume by the procedure described in API Recom-

mended Practice 13B-1, Section entitled "Filtration." Record the filtrate volume, cake thickness, and cake consistency.

11 Methylene Blue Test for Drill Solids and Commercial Bentonite

11.1 METHYLENE BLUE CAPACITY OF DRILL SOLIDS-DESCRIPTION

11.1.1 The methylene blue capacity of drill solids can be determined by a procedure that is similar to that used for the drilling fluid. The methylene blue capacity is an estimate of the cation exchange capacity of the drill solids. The true cation exchange capacity of the drill solids will probably be somewhat higher than the methylene blue capacity.

11.1.2 Methylene blue solution is used to titrate a sample of drill solids that has been dried, ground to a powder, and treated with a strong dispersant and an oxidizing agent. The dispersed drill solids sample is titrated with methylene blue solution until a blue "halo" appears around a drop of dyed drill solids on filter paper.

11.2 METHYLENE BLUE CAPACITY OF DRILL SOLIDS-EQUIPMENT

The following equipment is needed:

a. Methylene blue solution: 3.20 g reagent grade methylene blue (C₁₆H₁₈N₃SCl)/L (1 cm³ = 0.01 milliequivalent) (CAS #61-73-4).

Note: The moisture content of reagent grade methylene blue must be determined each time the solution is prepared. Dry a 1.000 g portion of methylene blue to a constant weight at 200 ± 5°F (93 ± 3°C). Make the appropriate correction in the weight of methylene blue to be taken to prepare the solution as follows:

Weight of sample to be taken, g = (13)

$$\frac{3.20}{\text{weight of dried sample}}$$

b. Tetrasodium pyrophosphate: 2% (w/v) solution (CAS #7722-88-5).

c. Hydrogen peroxide: 3% solution (CAS #7722-84-1).

d. Dilute sulfuric acid: approximately 5N.

e. Erlenmeyer flask: 250 cm³

f. Burette: 10 cm³, micropipette: 0.5 cm³ or graduated pipette: 1 cm³.

g. Graduated cylinder (TD): 50 cm³.

h. Stirring rod.

i. Hot plate.

j. Magnetic stirrer and stirring bar coated.

k. Whatman No. 1 filter paper, or equivalent.

l. Drying oven: 220 ± 5°F (105 ± 3°C).

m. Mortar and pestle.

- n. 75 micrometer sieve.
o. Balance: sensitivity 0.05 g.

11.3 METHYLENE BLUE CAPACITY OF DRILL SOLIDS-PROCEDURE

11.3.1 Clean all traces of drilling fluid from the drill solids sample and dry at $220 \pm 5^\circ\text{F}$ ($10.5 \pm 3^\circ\text{C}$) for 2 hours.

11.3.2 Grind the drill solids to a fine powder using the mortar and pestle and pass through the **200-mesh** sieve.

11.3.3 Weigh 1.0 g of the less than 75 micrometers drill solids and add to the Erlenmeyer flask containing 25 cm^3 of 2% tetrasodium pyrophosphate.

11.3.4 Mix well and boil gently for 10 minutes. Do not allow to boil to dryness.

11.3.5 Add 15 cm^3 hydrogen peroxide and 1 cm^3 of sulfuric acid. Boil gently for an additional 10 minutes. Cool to room temperature and dilute to above 50 cm^3 with water

11.3.6 Add methylene blue solution to the flask in increments of 1.0 cm^3 while stirring on the magnetic stirrer. If the approximate amount of methylene blue solution necessary to reach the endpoint is known from previous tests, somewhat larger increments can be used at the beginning of the titration. After each addition, mix the contents of the flask for 30 seconds and remove 1 drop of suspension with the stirring rod and place on the filter paper. The initial end point of the titration is reached when dye appears as a faint blue ring or halo surrounding the dyed solids as shown in Figure 7.

11.3.7 When the initial end point is detected, stir the flask for an additional 2 minutes and place another drop on the test paper. If the blue ring is again evident, the two minute end point has been reached. If the ring does not appear, continue as in 11.3.6 until a drop taken after stirring 2 minutes shows the blue tint.

11.3.8 Continue stirring an additional 10 minutes and place another drop on the test paper. If the blue ring is again evident the 10 minute endpoint has been reached. If the ring does not appear, continue as in 11.3.6 until a drop taken after stirring 30 seconds shows the blue tint.

11.4 METHYLENE BLUE CAPACITY OF DRILL SOLIDS-CALCULATION

The methylene blue capacity of the **drill** solids (MBT) is given as follows:

$$\text{Drill solids MBT, meq/100 g} = \quad (14)$$

$$\frac{\text{methylene blue solution, cm}^3}{\text{weight of drill solids sample, g}}$$

11.5 METHYLENE BLUE CAPACITY OF COMMERCIAL BENTONITE- DESCRIPTION

The methylene blue capacity of commercial bentonite can be determined using a procedure similar to that used for the drill solids. The methylene blue capacity is an estimate of the cation exchange capacity of the bentonite. The true cation exchange capacity of the bentonite will probably be somewhat higher than the methylene blue capacity. A well dispersed sample of the bentonite is titrated with methylene blue solution until a blue or turquoise "halo" appears around a drop of dyed bentonite solids on filter paper.

11.6 METHYLENE BLUE CAPACITY OF COMMERCIAL BENTONITE- EQUIPMENT

The following equipment is needed:

- Methylene blue solution: $1 \text{ cm}^3 = 0.01$ milliequivalent (See 11.2a).
- Tetrasodium pyrophosphate: 2% (w/v) solution. (See 11.2b).
- Erlenmeyer flask: 250 cm^3 .
- Burette: 50 cm^3 .
- Graduated cylinder (TD): 50 cm^3 .
- Stirring rod.
- Hot plate.
- Magnetic stirrer and stirring bar (coated).
- Whatman** No. 1 filter paper, or equivalent.
- Balance: sensitivity 0.05 g.

11.7 METHYLENE BLUE CAPACITY OF COMMERCIAL BENTONITE- PROCEDURE

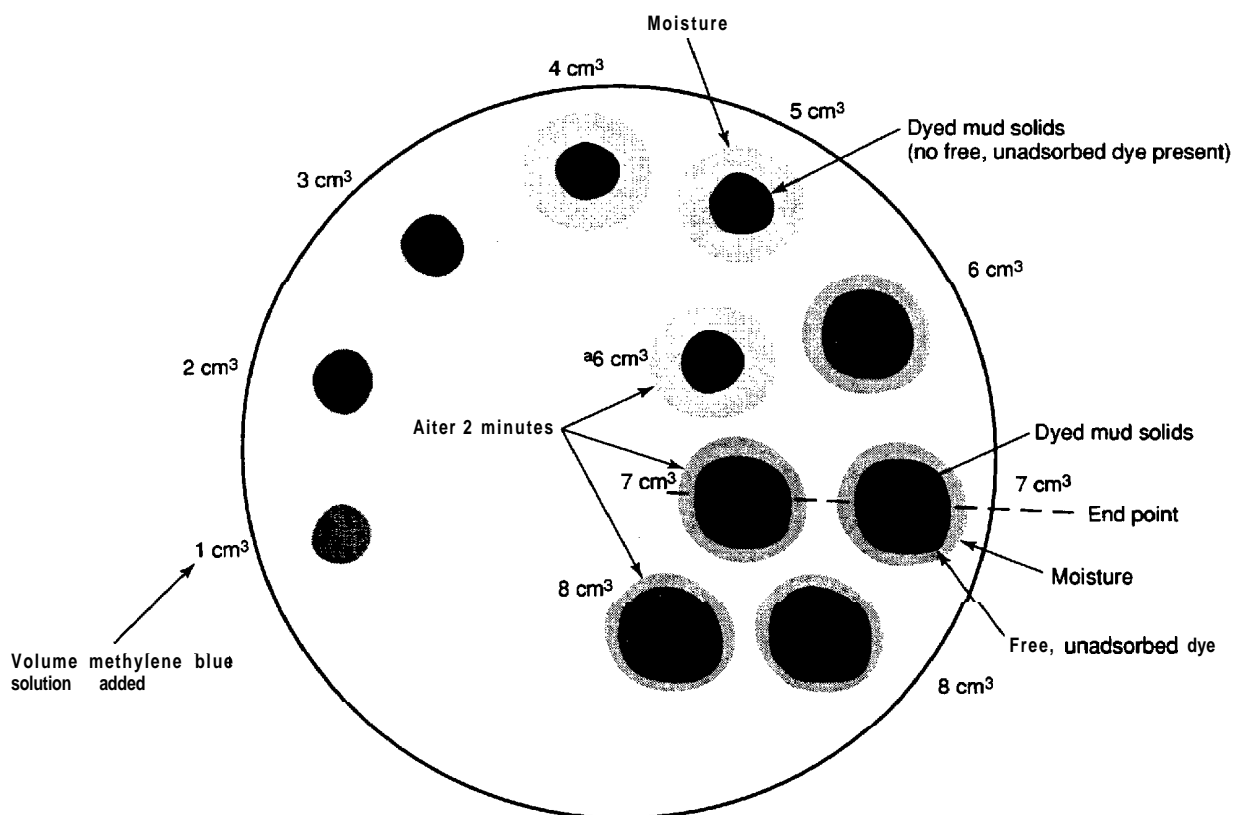
11.7.1 Accurately weigh 0.5 g bentonite and add to the Erlenmeyer flask containing 50 cm^3 of 2% **tetrasodium pyrophosphate** solution.

11.7.2 Mix well and boil gently for 10 minutes. Do not allow to boil to dryness.

11.7.3 Cool to room temperature and bring volume to about 50 cm^3 with water.

11.7.4 Add methylene blue solution to the flask with the burette in one cm^3 increments while stirring on the magnetic stirrer. Somewhat larger increments can be used at the beginning of the titration as the endpoint should only be reached after 25 or more cm^3 have been added. After each addition, mix the contents of the flask for 30 seconds and remove one drop of the suspension with the stirring rod and place on the filter paper. The initial endpoint of the titration is reached when dye appears as a faint blue ring or halo surrounding the dyed solids as shown in Figure 7.

Note: For the best accuracy it is recommended that increments no larger than 1 cm^3 be used.



Note: ^aFree dye detected immediately after adding sixth cm^3 is adsorbed after two minutes and indicates that end point has not quite been reached.

Figure 7—Spot Tests for End Point of Methylene Blue Titration

11.7.5 When the initial endpoint is detected, stir the flask for an additional 2 minutes and place another drop on the test paper. If the blue ring is again evident the 2 minute endpoint has been reached. If the ring **does** not appear, continue as in 11.7.4 until a drop taken after stirring 2 minutes shows the blue tint or halo.

11.7.6 Continue stirring an additional 10 minutes and place another drop on the test paper. If the blue ring is again evident, the 10 minute endpoint has been reached. If the ring does not appear, continue as in 11.7.4 until a drop taken after stirring 30 seconds shows the blue tint.

11.8 METHYLENE BLUE CAPACITY OF COMMERCIAL BENTONITE—CALCULATION

The methylene blue capacity of commercial bentonite (MBT) is calculated as follows:

$$\text{Bentonite MBT, meq/1 00g} = \quad (15)$$

$$\frac{\text{methylene blue solution, cm}^3}{\text{weight of bentonite sample, g}}$$

11.9 SOLIDS CALCULATIONS

The following procedure can be used to give a rough estimate of the **amount** of drill solids and commercial bentonite in a drilling fluid using the low gravity solids (LGS) in **lb/bbl** calculated in **API Recommended Practice 13B-1**, Section entitled “Water, Oil, and Solids,” and the methylene blue capacities for **the drilling fluid (API Recommended Practice 13B-1**, Section entitled “Methylene Blue Capacity”), drill

solids (11.4) and commercial bentonite (11.8). The amounts of each type of low gravity solid can be calculated as follows:

$$\text{Average MBT, meq/100 g} = \quad (16)$$

$$\frac{70 \text{ (Bentonite equivalent, lb/bbl)}}{\text{LGS, lb/bbl}}$$

$$\text{Bentonite, lb/bbl} = \quad (17)$$

$$\frac{\text{Average MBT} - \text{Drill Solids MBT}}{\text{Bentonite MBT} - \text{Drill Solids MBT} \times \text{LGS, lb/bbl}}$$

$$\text{Drill Solids, lb/bbl} = \text{LGS, lb/bbl} - \text{Bentonite, lb/bbl} \quad (18)$$

Note: If the MBT for bentonite is not known, use a value of 70 meq/100 g, as is assumed in the calculation of the bentonite equivalent of the drilling fluid (API Recommended Practice 13B-1, Section entitled "Water, Oil and Solids"). The solids composition calculated assuming an MBT of 70 meq/100 g for bentonite will not be as accurate as if the full procedure above is used but the calculated amounts of bentonite and drill solids can still be useful as a monitoring tool.

12 Deflocculation Tests for Thinner Evaluation

12.1 DESCRIPTION

12.1.1 The following laboratory test is designed to evaluate the relative effectiveness of a thinner in a high solid environment. The data obtained using this procedure should be compared to data using a reference thinner in the same series of tests and should not be interpreted as thinner performance in actual drilling fluid formulations.

12.1.2 A 28.0% by weight suspension of Neutral Panther Creek bentonite is prepared with distilled water and aged at room temperature for one week or more. A laboratory barrel portion of the suspension is treated with five lb/bbl of chrome or ferrochrome lignosulfonate for uncontaminated tests and 8 lb/bbl of the reference thinner for contaminated (5 lb/bbl gypsum and 4 lb/bbl sodium chloride) tests and the pH adjusted to 9.5 with sodium hydroxide. Viscometric properties are measured initially and after hot-rolling 16 hours at 150°F (66°C). API filtration is measured after hot-rolling. After completing the measurements on the hot-rolled samples, the pH of the suspension is adjusted to 9.5 and aged 16 hours in a stainless steel cell at 350°F (177°C). The cell is pressured to 500 psi (3450 kPa) with nitrogen prior to heating. Viscometric properties of the suspension are measured after high temperature aging as removed from the cell. The pH of the suspension is readjusted to 9.5 and Viscometric properties are remeasured and the API filtration is determined.

12.1.3 The effectiveness of the test sample is evaluated by comparing the performance of a reference sample of lignosulfonate or other thinner.

12.2 EQUIPMENT

The following equipment is needed:

- a. Neutral Panther Creek bentonite (air-floated): available from American Colloid Company, Skokie, Illinois.
- b. Sodium chloride: (CAS #7647-14-5) 99% minimum purity.
- c. Gypsum: (CAS #7778-18-9) 85% through 100 mesh — 92% minimum $\text{Ca}_2\text{SO}_4 \times 2\text{H}_2\text{O}$.
- d. Sodium hydroxide: (CAS #1310-73-2) CP, used to make solution where 1 cm³ contains 0.25 g NaOH.
- e. Distilled or demineralized water.
- f. Standard or reference thinner.
- g. Mixer: capable of operating at 10,000 ± 100 rpm under load and designed so that the bearings are not exposed to the test fluid (e.g., the Hamilton Beach Mixer Model 936, or equivalent).
- h. Mixing container with air tight cover.
- i. Balance: 3 kg capacity.
- j. Mud balance.
- k. Viscometer (e.g., Fann VG Meter, Model 35, or equivalent), as described in API Recommended Practice 13B-1, Section entitled "viscosity and Gel Strength."
- l. Powerstat: Type 116, or equivalent.
- m. pH Meter: accuracy 0.1 pH unit.
- n. Minute timer.
- o. API filter press, as described in API Recommended Practice 13B-1, Section entitled "Filtration."
- p. No. 50 Whatman filter paper, or equivalent.
- q. Spatula.
- r. High temperature aging cells: stainless steel with pressure connector.
- s. Gravity convection oven-capable of 150 ± 5°F (66 ± 3°C).
- t. Pint jar: Mason or Kerr, or equivalent.
- u. Burette: graduated to 0.1 cm³.
- v. Nitrogen source: 500 psi or greater.
- w. Syringe: 10 cm³, graduated to 0.2 cm³.
- x. Mixer: Hamilton Beach Model 30, or equivalent.

12.3 PROCEDURE-MOISTURE CONTENT

12.3.1 Determine the moisture content of the Panther Creek bentonite as follows:

12.3.2 Weigh 10 ± 0.01 g of clay and dry to a constant weight at a temperature of 220 ± 5°F (105 ± 3°C). Cool the sample in a desiccator and weigh.

12.4 CALCULATION-MOISTURE CONTENT

Percent Moisture = (19)

$$\frac{(\text{Weight Original Sample} - \text{Weight Dry Sample})}{\text{Weight Original Sample}} \times 100$$

12.5 PROCEDURE-PREPARATION OF MUD BASE

12.5.1 Prepare 500 g suspension by dispersing 140 g of Panther Creek bentonite (dry basis) in distilled or demineralized water. The amount of water required will be 350 g minus the amount of water in the clay as determined in 12.4. The clay should be added to the water slowly while stirring the suspension with the Hamilton Beach mixer.

12.5.2 Shear the suspension for $2\frac{1}{2}$ hours, seal the container air-tight so that no moisture will escape, and age overnight at room temperature. On the following day stir for 30 minutes and determine the solids content of the suspension as follows.

12.5.3 Weigh 10 ± 0.01 g of suspension and dry at a temperature of $220 \pm 5^\circ\text{F}$ ($105 \pm 3^\circ\text{C}$). Cool the sample in a desiccator and weigh. Perform this test in duplicate until results agree within 0.1 weight percent.

12.5.4 After correcting the solids content to within $\pm 0.1\%$, determine the specific gravity of the suspension to ± 0.1 unit by use of the mud balance. Calculate the weight of 350 cm^3 of suspension (specific gravity ± 350 = weight in g) and immediately bottle this amount of suspension in individual pint jars. Be sure that the lids are sealed air-tight and store for future use. Base mud should age in jars at room temperature for a minimum of 7 days before using and should be discarded if not used within 90 days of preparation.

12.5.5 Solids content shall be $28.0 \pm 0.1\%$. If the solids are too low, let the suspension stand overnight exposed to the atmosphere so that some of the moisture can evaporate. Then redetermine solids content as above. Repeat until solids content is correct. If solids are too high, see 12.6.2.

12.6 CALCULATION

12.6.1 Solid Content, etc.

$$\text{Percent Solids} = \frac{\text{Weight of Dry Sample}}{\text{Weight of Original Sample}} \times 100 \quad (20)$$

12.6.2 If the solids are too high, add demineralized water. Calculate the amount of water to add by the following formula:

$$W_a = W_t \frac{W_{sol}}{S} (100) \quad (21)$$

where

W_a = Weight of water required for solids adjustment, g,

W_t = Specified weight of suspension = 500 g,

W_{sol} = Total weight of solids added = 140 g Panther Creek bentonite (dry basis),

S = % solids determined.

Example: Solids too high

Assumption: 29.1% solids determined

$$W_a = 500 - \frac{140}{29.1} (100)$$

$$W_a = 500 - 481.1$$

$$W_a = 18.9 \text{ g}$$

12.7 PROCEDURE-FLOW PROPERTIESTEST

12.7.1 To 350 cm^3 of the Panther Creek suspension, add 1.75 g salt and stir for 3 minutes at 750 ± 50 r/min with a Hamilton Beach mixer or equivalent.

12.7.2 Add five grams of the deflocculant being tested. Stir the sample for 1 minute.

12.7.3 Add sufficient caustic soda (1 $\text{cm}^3 = 0.25$ g NaOH) so that the pH measures 9.5 ± 0.1 after 20 minutes total stirring time.

12.7.4 Scrape down the sides of the jar at least once to insure proper mixing. The mixing speed shall be kept at $7,500 \pm 500$ rpm. After 20 minutes total stirring time, remove the mud from the mixer. Check to see that the pH is 9.5 ± 0.1 .

12.7.5 Stir for one minute and measure the flow properties with a Fann Model 35 VG meter, or equivalent.

12.7.6 Record the 600, 300, 200, 100, 6 and 3 rpm viscometer readings, initial gel, pH, and total cm^3 of NaOH used.

12.7.7 Cap the jar tightly and hot-roll for 16 hours at 105°F (66°C).

12.7.8 Cool the hot-rolled mud to room temperature. Re-adjust the pH.

12.7.9 Take the 600, 300, 200, 100, 6 and 3 rpm viscometer readings, initial gel, pH, lo-minute gel, and API filtrate.

12.7.10 Place the mud in a stainless steel high temperature aging cell. Pressure the aging cell to 500 psi (3450 kPa) with nitrogen and test for leaks by immersing in water. Place the cell in a 350°F (177°C) oven for 16 hours.

12.7.11 Remove the cell, cool, and transfer the mud to a pint jar.

12.7.12 Measure and record all viscometer readings. Readjust the **pH** to 9.5 ± 0.1 and remeasure all viscometric properties and the API filtrate.

12.7.13 To 350 cm^3 of the base mud, add 5 g gypsum, 4 g sodium chloride, and 8 g of the deflocculant to be evaluated.

12.7.14 Stir into the mud for 20 to 30 seconds by raising, lowering, and turning the jar by hand while the mixer is running at 7,000 to 8,000 rpm with the toggle switch in the high speed position.

12.7.15 Place the jar into the jar holder. Set the timer for 20 minutes stirring time. After an elapsed time of 1 minute, add a sufficient volume of **NaOH** solution ($1 \text{ cm}^3 = 0.25 \text{ g NaOH}$) rapidly with a syringe to give a **pH** of 9.5 ± 0.1 at the end of the 20 minute stirring time. The quantity of **NaOH** varies with each type of deflocculant used and must be determined by trial and error.

12.7.16 After 5 minutes stirring, remove the sample from the mixer and scrape down the sides of the jar.

12.7.17 Replace the sample on the mixer and continue stirring for a total elapsed time of 20 minutes at 7,000 to 8,000 rpm.

12.7.18 At the end of 20 minutes elapsed time, remove the mud from the mixer, measure the **pH**; and if the **pH** is 9.5 ± 0.1 , stir for one minute at 7,000 to 8,000 rpm and measure the flow properties with the viscometer. If the **pH** is not 9.5 ± 0.1 , discard the sample and repeat the test.

12.7.19 Measure and record the **600, 300, 200, 100, 6** and 3 rpm viscometer readings, initial gel, **pH** and total **NaOH** used.

12.7.20 Cap the jar tightly and hot-roll for 16 hours at 150°F (66°C).

12.7.21 Cool the hot-rolled mud to room temperature and stir for five minutes at 7,000 to 8,000 rpm.

12.7.22 Record the **600, 300, 200, 100, and 6** rpm readings, the initial gel, the **pH**, and the API filtrate.

12.7.23 Adjust the **pH** to 9.5 with **NaOH** and place in a stainless steel high temperature aging cell. Pressure the aging cell to 500 psi (3450 kPa) with nitrogen and test for leaks by immersing in water. Place the cell in an oven at 350°F (177°C) for 16 hours.

12.7.24 Remove the cell, cool and transfer the mud to a pint jar, stir 5 minutes at 7,000 to 8,000 rpm on a Hamilton Beach mixer, or equivalent.

12.7.25 Measure and record all viscometer readings. Record the **pH**.

12.7.26 Then adjust the **pH** to 9.5 with **NaOH** and remeasure all the viscometric properties and the API filtrate.

12.8 CALCULATION-THINNER EFFICIENCY

The effectiveness of the test sample is evaluated by comparing its performance to the performance of a reference sample of lignosulfonate or other thinner.

(22)

$$\text{Efficiency, \%} = \frac{\text{Performance (test sample)}}{\text{Performance (reference)}} \times 100$$

13 Standard Methods for Testing Base Oils Used in Drilling Fluids: Density, Viscosity, Distillation, Aniline Point, Pour Point and Flash Point

13.1 DESCRIPTION

13.1.1 A base oil, as defined in this Section, is a solids-free and water-free hydrocarbon oil commonly used in the drilling fluid industry for construction and/or dilution of an oil-based drilling or completion fluid. Commonly used base oils are often termed "mineral oils" or "solvent oils" or "absorber oils" and would also include the "diesel oils."

13.1.2 This section references and gives a brief summary of American Society for Testing and Materials (ASTM) testing methods which API Subcommittee 13 on Drilling Fluids Materials has reviewed and found to be acceptable for the testing of base oils commonly used in oil base drilling and completion fluids. Most of these procedures are also standard IP (Institute of Petroleum) tests.

13.1.3 The full, stepby-step procedures are not, reproduced herein, but can be obtained from ASTM or IP headquarters offices. When ordering copies of these **procedures**—see the addresses below—use ASTM or IP designation numbers and the latest revision date.

American Society for Testing and Materials
100 Barr Harbor Drive
West Conshohocken, PA 19428

Institute of Petroleum
61 New Cavendish Street
London W1 M 8AR, England
Telephone: 01636 1004

13.2 EQUIPMENT

Equipment required for each of the tests of this section is described in the appropriate ASTM procedure bulletin and is only mentioned in general terms herein. The specific test equipment is mandatory in order to perform the standard tests

correctly and safely and to achieve results that meet API requirements.

13.3 DENSITY, RELATIVE DENSITY (SPECIFIC GRAVITY), OR API GRAVITY-HYDROMETER METHOD-ASTM D-1298-80 (1980) AND API 2547 (1968) AND IP 160/68 (1968)

13.3.1 Description

This method covers the laboratory determination, using a glass hydrometer, of density, relative density (specific gravity), or API gravity of crude petroleum, petroleum products, or mixtures of petroleum and non-petroleum products normally handled as liquids, and having a Reid vapor pressure (Method ASTM D 323 or IP 69) of 26 lb or less. Values are measured on a hydrometer at convenient temperatures, reading of density being reduced to 60°F (15°C) and readings of relative density (specific gravity) and API gravity to 60°F (WC), by means of international standard tables. (See Note below.) By means of these same tables, values determined in any one of the three systems of measurement are convertible to equivalent values in either of the other two so that measurements may be made in the units of local convenience. Generally, the drilling fluids industry will prefer to use the relative density (specific gravity) units.

Note: The tables referred to above for conversion between the systems of density or specific gravity or API gravity are ASTM D 1250-80 (also identified as IP 200/52 (81) and API D 2540) entitled *Petroleum Measurement Tables*. These were approved in 1980 and reapproved in 1984 by ASTM, IP and API committees.

13.3.2 Summary of Method

The sample is brought to the prescribed temperature and transferred to a cylinder which is approximately at the same temperature. The appropriate hydrometer is lowered into the sample. After temperature equilibrium has been reached and the hydrometer is still, the hydrometer scale is read and the temperature of the sample is noted. If necessary, the cylinder and its contents may be placed in a constant temperature bath to avoid excessive temperature variation during the test.

13.4 DENSITY AND RELATIVE DENSITY OF LIQUIDS-DIGITAL DENSITY METER-ASTM D 4052-81 (1981)

13.4.1 Description

This method covers the determination of the density or relative density of petroleum distillates and viscous oils that can be handled in a normal fashion as liquids at test temperatures between 60°F and 95°F (WC and 35°C). Its application is restricted to liquids with vapor pressures below 600 mm mercury (80 kPa) and viscosities below about 15,000 cSt at the temperature of test. It should not be applied to samples so

dark in color that the absence of air bubbles in the sample cell cannot be established with certainty.

13.4.2 Summary of method

Approximately 0.7 cm³ of liquid sample is introduced into an oscillating sample tube, and the change in oscillating frequency caused by the change in the mass of the tube is used in conjunction with calibration data to determine the density of the sample.

13.5 KINEMATIC VISCOSITY OF TRANSPARENT AND OPAQUE OILS—CALIBRATED CAPILLARY TUBE METHOD-ASTM: D 445-83 (1983) AND IP 71/79 (1979)

13.5.1 Description

This test method covers the determination of the kinematic viscosity (a measure of the resistance to flow under gravity) of liquid petroleum products, both transparent and opaque, by measuring the time for a given volume of the liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity can be obtained by multiplying the measured kinematic viscosity by the density of the liquid.

13.5.2 Summary of Method

The time is measured in seconds for a fixed volume of the liquid sample to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer. Testing temperatures usually selected are 104°F and 158°F (40°C and 70°C) for base oils, unless these temperatures could constitute a hazard or fire or flash explosion during the testing.

13.6 DISTILLATION-ASTM D 86-82 (1982) AND IP 123/76 (1976)

13.6.1 Description

This method covers the distillation of motor gasolines, aviation gasolines, aviation turbine fuels, special boiling point spirits, naphthas, white spirit, kerosenes, gas oils, distillate fuel oils, and similar petroleum products.

13.6.2 Summary of Method

A 100 cm³ sample is distilled under prescribed conditions which are appropriate to its nature as shown in Table 1 of the ASTM procedure. Systematic observation of the thermometer readings and volumes of condensate are made, and from these data, the results of the tests are calculated and reported. Important temperature values found are: (a) initial boiling

point when the first drop is condensed, (b) final boiling point, when maximum temperature is reached and (c) dry point, the temperature just as the last drop evaporates from the lowest point in the flask.

13.7 ANILINE POINT AND MIXED ANILINE POINT-ASTM D 611-82 (1982) AND IP: 2/76 (1976)

13.7.1 Description

These test methods cover the determination of the aniline point of petroleum products and hydrocarbon solvents. Method A is suitable for transparent samples with an initial boiling point above room temperature and where the aniline point is below the bubble point and above the solidification point of the aniline-sample mixture. Method B, a thin-film method is suitable for samples too dark for testing by Method A. Methods C and D are for samples that may vaporize appreciably at the aniline point. Method D is particularly suitable where only small samples are available. Method E describes a procedure using an automatic apparatus suitable for the range of liquids covered by Methods A and B.

These test methods also cover the determination of the mixed aniline point of petroleum products and hydrocarbon solvents having aniline points below the temperature at which aniline will crystallize from the aniline-sample mixture.

Note: This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

13.7.2 Summary of Methods

Method A, and perhaps B, are the methods most likely to be applicable to drilling fluid industry use for testing base oils. The specified volumes of aniline and the oil sample (or aniline and sample and n-heptane) are placed in a tube and mechanically mixed. The mixture is heated at a controlled rate until the two phases become miscible. The mixture is then cooled at a controlled rate and the temperature at which two phases begin to separate and cloudiness appears is recorded as the aniline point (or mixed aniline point if n-heptane is used as a solvent).

13.8 POUR POINT-ASTM D 97-66 (1966) AND IP 15/67 (1967)

13.8.1 Description

The test for pour point is intended for use on any petroleum oil. A procedure suitable for black oils, cylinder stock, and nondistillate fuel oil is also described in this ASTM standard.

13.8.2 Summary of Method

After preliminary heating, the sample is cooled at a specified rate and examined at intervals of 5°F (3°C) for flow characteristics. The lowest temperature at which movement of the oil is observed is recorded as the pour point.

13.9 FLASH POINT BY PENSKEY-MARTENS CLOSED-TESTER-ASTM D 93-80 (1989) AND IP 34/75 (1975)

13.9.1 Description

This method covers the determination of the flash point by Pensky-Martens closed-cup tester of fuel oils, suspensions of solids, liquids that tend to form a surface film under test conditions, and other liquids. (This method is not recommended by the API for testing emulsions of water-and-oil such as drilling fluids.)

13.9.2 Summary of Method

The oil sample is heated at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame causes the vapor above the sample to ignite.

13.10 AROMATICS CONTENT-ASTM D 5186 (1991) OR IP 391 (1988)

13.10.1 Description

Two methods are provided: ASTM and IP. Both methods will measure "aromatic content" of drilling fluid oils, petroleum distillates, and diesel fuels. The user should realize that analyses of a complex oil by the two methods may not give the same values for aromatic content. The reason for variation is the two instruments separate aromatic components differently and their detectors function by different principles.

Instruments for the IP method are more widely available and less expensive than those required for the ASTM method. The ASTM method uses a column and detector system that gives a better representation of the types of aromatics found in complex oils but it does not measure low concentrations of aromatics.

13.10.1.1 IP 391

IP 391 is suitable for determining monocyclic, bicyclic and tricyclic aromatic fractions in drilling fluid oils and petroleum distillates which boil in the 284°F to 680°F (140°C- 360°C) range with Bromine numbers less than 1.0 (determined by IP 130). This method is appropriate for concentrations from 0.05 to 25 volume percent monocyclic aromatics and 0.05 to

5 volume percent bicyclic and tricyclic aromatics. Higher concentrations require appropriate dilutions.

Note: Compounds containing oxygen or nitrogen may cause erroneous results for the IP method.

13.10.1.2 ASTM D 5186

The ASTM D 5186 method is suitable for determining aromatic compounds in fuels with concentrations from 5 to 75 weight percent of aromatics.

13.10.2 Summary of the Method IP 391

A 5 – 10 microliter oil sample is injected into a liquid chromatograph which is fitted with a polar separation column with hexane as the mobile phase. The column separates the aromatic components into mono-, bi- and tricyclic fractions which appear as peaks on the chromatogram. The fractions are identified by a differential refractive-index detector as they exit the column. Percentages are determined by integrating areas under the peaks based on prior calibration with known concentrations of an aromatic compound.

13.10.3 Summary of the Method ASTM D 5186

A 0.05 – 0.2 microliter oil sample is injected into a packed silica column using supercritical carbon dioxide as the mobile phase. Aromatic components in the sample are separated from nonaromatic components by absorption in the silica column. Components are measured by flame ionization detector as they exit the column. Concentrations are calculated from peak areas based on prior calibration using known concentrations of an aromatic compound.

14 Potassium in Drilling Fluids by the ISE (Ion Selective Electrode) Method

14.1 DESCRIPTION

14.1.1 Potassium compounds, primarily the chloride and to a lesser degree the hydroxide, are used in drilling fluids to aid in the stabilization of formation clays and/or shales. The ISE method can provide accurate, quantitative data on samples having a range of potassium ion concentrations from a few milligrams per liter to saturation.

14.1.2 The measurement of potassium ion concentration by the ISE method is analogous to a pH measurement and can be performed with almost the same ease and simplicity. Both procedures rely on the measurement of the difference in potential between a sensing and a reference electrode. Depending on the instrumentation used, this signal is presented as either the actual potential, measured in millivolts, or in some form of a direct readout of concentration.

14.1.3 For monovalent ions such as hydrogen and potassium, the change in potential between a sensing and a refer-

ence electrode is theoretically 59 millivolts for a ten-fold change in the activity of the ion being measured. The actual change is normally somewhat lower, usually about 56 ± 2 millivolts for the potassium electrode system. This value is defined as the slope of an electrode system.

14.1.4 It should be noted that the actual property being measured is activity, rather than concentration. However, because the activity of any ionic species is strongly influenced by the total ionic strength of the solution, it is feasible to use the two terms interchangeably so long as the total ionic strength is held constant. This procedure provides for this by the addition of sodium chloride solution to all standards and samples to maintain a constant background ionic strength.

14.1.5 Under the conditions of this test method, none of the materials normally present in mud filtrate samples have been found to interfere. Errors arising from sodium contaminations are eliminated by providing a high constant background sodium level through the use of sodium chloride as the ISA (ionic strength adjuster). The relatively high dilution reduces all other potential interferences to a harmless level. This method is intended to be used for the analysis of filtrate samples only, not whole mud samples.

14.2 EQUIPMENT

- a. A specific ion meter or pH meter: Any one of a wide variety of commercially available instruments is suitable, so long as it can provide a millivolt readout with a resolution of at least 0.1 millivolt. Either analog or digital readout is acceptable, but the preferred type of instrument is one of the "research" models with a digital readout and controls which permit direct indication of concentration after proper calibration.
- b. Potassium ion-selective electrode (ISE): Orion Model 93-19 potassium electrode, or equivalent. This is a two-part electrode, with a reusable body and a disposable, screw-on sensing module. Only the module, not the entire electrode, requires replacement. Average service life of each module is 6 months.
- c. Reference electrode: A single-junction reference electrode, Orion Model 90-01, or equivalent (both the sensing and reference electrodes described above have durable epoxy bodies).

Note: Reference to the Orion electrodes is for information only, and is not intended to exclude from use electrodes from other manufacturers. Other electrodes are acceptable so long as the accuracy, resolution, and stability is equivalent to the Orion products. Not all commercially available electrodes have been evaluated for use in this procedure, but the Orion products have consistently provided satisfactory performance. Each analyst is responsible for verifying the performance of his electrode system before continuing with any quantitative determinations.

- d. Magnetic stirrer with stirring bars (coated).
- e. Beakers: 150 cm³, Pyrex®, its equivalent or plastic.

f. Volumetric pipettes: various sizes for preparing dilutions of samples and standards. The use of microliter pipettes with disposable plastic tips (such as those manufactured by Eppendorf or Oxford) is particularly convenient for diluting filtrate samples where the quantity of filtrate available is limited or extremely high dilutions are required.

g. Graph paper: 3- or 4-cycle semilogarithmic for preparing calibration curves.

h. Volumetric flasks: various sizes for preparing calibration standards and dilutions of filtrate samples.

i. Standard potassium solution, 1000 mg/L: this solution is readily available from most laboratory supply firms, or can be prepared by dissolving 1.907 g of reagent grade KCl in distilled or deionized water and diluting to liter. The KCl should be dried in an oven at $220 \pm 5^\circ\text{F}$ ($105 \pm 3^\circ\text{C}$) for 2 hours and stored in a desiccator prior to preparation of the standard solution.

j. Sodium chloride solution, 5 molar: Dissolve 292 g of reagent grade sodium chloride in distilled or deionized water and dilute to 1 liter. This solution is used as an ionic strength adjustor (ISA) and to provide a high, constant background sodium concentration.

k. Sodium chloride solution, 0.1 M, saturated with silver: Dilute 2 cm³ of the 5 molar sodium chloride solution to 100 cm³ and add silver nitrate solution drop-wise until a cloudiness persists. This solution is used as the internal filling solution for the single junction reference electrode.

14.3 PROCEDURE-PREPARATION OF ELECTRODES

14.3.1 If the Orion 93-19 electrode is used, remove the sensing module from the vial and screw it firmly into the electrode body, taking care to ensure that the rubber washer is in place.

14.3.2 Shake down the electrode in the manner of a thermometer.

14.3.3 Soak the electrode in distilled or deionized water for at least 10 minutes, then store in a diluted potassium solution, containing 10 – 100 mg/L potassium, until ready for use.

14.3.4 By means of either an eye dropper or syringe, fill the single-junction reference electrode with the 0.1 molar sodium chloride (saturated with silver ion).

14.3.5 If other types of sensing or reference electrodes are used, follow the instructions of the manufacturer when preparing them for use.

14.4 PROCEDURE-OPERATIONAL CHECK OF ELECTRODE SYSTEM

14.4.1 The two properties of the electrode system which will have the greatest influence on accuracy and reproducibility are the slope and the stability. The slope is defined as the

change in potential observed when the concentration changes by the factor of 10. The stability of the readings is an indication of the amount of noise (rapidly, continuously changing readings) and drift (readings slowly changing in one direction).

14.4.2 When performing the initial calibration with the standard solutions according to the instructions in either 14.5 or 14.6, record the actual measured potential for the 10 mg/L and the 100 mg/L standard solutions. For a new potassium electrode (or sensing module) the difference between these two readings should be between 54 and 59 millivolts. The slope will normally decrease with time. When the slope reaches a value of approximately 48 millivolts the electrode (or module) should be replaced.

14.4.3 If, when performing the initial calibration, there is an indication of noise or drift, refer to the troubleshooting section of the electrode manual. There are a number of possible causes for these effects with any electrode system, and the cause is normally found only by a process of elimination. If the analyst is unable to achieve a stability level which meets the manufacturer's specifications, the sensing electrode (or module) should be replaced.

14.5 PROCEDURE-MEASUREMENTS USING A METER WITH DIRECT CONCENTRATION READOUT CAPABILITY

14.5.1 Prepare a 100 mg/L potassium standard and a 10 mg/L potassium standard by serial dilution of the 1000 mg/L standard solution.

14.5.2 Add the ISA solution (5 molar sodium chloride) to each standard at the rate of 2 cm³ of ISA per 100 cm³ of standard.

14.5.3 Transfer the standards to 150 cm³ beakers. Place the 10 mg/L standard on the magnetic stirrer, place the electrodes in the standard solution, and stir at a moderate rate.

14.5.4 Wait for a stable reading (normally 1 – 2 minutes), then adjust the controls to display the value of the standard, following the procedure described in the meter instruction manual.

14.5.5 Remove the electrodes, rinse with distilled water, blot dry, and place in the 100 mg/L standard. Stir at a moderate rate.

14.5.6 Wait for a stable reading, then adjust the controls to display the value of the standard. The meter and electrode system are now calibrated.

14.5.7 Prepare a 1:1000 dilution of each mud filtrate sample.

14.5.8 Transfer 100 cm³ of diluted sample to a 150 cm³ beaker, add 2 cm³ ISA, and stir at a moderate rate.

14.59 Rise electrodes with distilled water, blot dry, immerse in sample and wait for a stable reading. The value displayed on the meter is the potassium concentration in the diluted sample. This value multiplied by the dilution factor (1000 in this case) will give the potassium concentration in the original sample.

Note: Concentration values which are above or below the value of the standards used for calibration are still reliable, since the 10 and 100 mg/L standards are near the midpoint of the region of linear response of the potassium electrode.

14.6 PROCEDURE—MEASUREMENTS WITH INSTRUMENTS WHICH PROVIDE EITHER DIGITAL OR ANALOG READOUT IN MILLIVOLTS

14.6.1 Prepare 100 mg/L, 10 mg/L, and 1 mg/L potassium standards by serial dilution of the 100 mg/L standard solution.

14.6.2 Add the ISA solution (5 molar sodium chloride) to each standard at the rate of 2 cm³ of ISA per 100 cm³ of standard.

14.6.3 Transfer the standards to 150 cm³ beakers. Place the 1 mg/L standard on the magnetic stirrer, place the electrodes in the standard solution and stir at a moderate rate.

14.6.4 Adjust the controls of the instrument so that the readout is in terms of millivolts, wait until the reading is stable, and record the reading.

14.6.5 Remove electrodes, rinse, blot dry and place in the 10 mg/L standard. When the reading is stable, record the reading.

14.6.6 Repeat 14.6.5 with the 100 mg/L standard.

14.6.7 Plot the millivolt readings on the linear axis and concentration in mg/L on the logarithmic axis of standard 3- or 4-cycle semilogarithmic paper.

14.6.8 Transfer 100 cm³ of sample which has been diluted 1:1000 to a 150 cm³ beaker, add 2 cm³ of the ISA solution, place the electrodes in the solution, and stir at a moderate rate.

14.6.9 Record the reading when stable, and determine the concentration of the diluted sample by use of the calibration curve. This value multiplied by the dilution factor gives the potassium content of the original sample.

Note: For accurate measurements, all standards and samples should be at ambient temperature. Some magnetic stirrers generate considerable heat, and it may be necessary to place a layer of insulating material (plastic or cardboard) between the stirrer and beaker. Between measurements, rinse electrodes and blot dry with tissue. After immersion in sample or standard solutions, check end of electrode for air bubbles and remove if present. Always read and carefully follow the manufacturer's instructions regarding the care,

maintenance, storage, servicing and troubleshooting of the instrument and electrodes.

15 Calcium in Drilling Fluid by the Ion-Selective Electrode (ISE) Method

15.1 DESCRIPTION

15.1.1 Calcium is present to some extent in the liquid phase of all water-based drilling fluids. The concentration level may vary from several percent by weight to a few milligrams per liter. It may be intentionally added in the form of lime, gypsum, or calcium chloride, or it may be introduced through the make-up or dilution water, or as an impurity in various products, or from drilled formations. Regardless of the concentration level or the origin of the calcium, it is nearly always necessary, or desirable, to monitor the calcium content on a quantitative basis.

15.1.2 The ISE method is a rapid, simple, and accurate method for the determination of calcium at any level in filtrate samples. Unlike the EDTA titrametric method, no sample treatment is required for highly colored or turbid samples. Under the conditions of this test method, none of the materials normally present in drilling fluids have been found to interfere. The combination of relatively high dilution, ionic strength adjustment through addition of KCl solution, and adjustment of pH to 5.0, ensure maximum conversion of calcium to ionic form and effectively eliminates or minimizes interferences.

15.1.3 This method is intended for the analysis of solution samples only and is not suitable for the analysis of whole mud samples.

15.2 EQUIPMENT

The following equipment is needed:

a. A pH meter capable of providing a millivolt readout with a resolution of at least 0.1 millivolt. There are many instruments available, ranging from battery-operated, hand-held portable units for field use to sophisticated, versatile "research" instruments programmed to provide readout in concentration units.

Readout may be either analog or digital. The preferred type for laboratory use is a research model with a digital readout and controls which provide direct indication of concentration after proper calibration.

b. Calcium ion-selective electrode (ISE): Orion Model 93-20 calcium electrode, or equivalent. This is a two-part electrode, with a reusable epoxy body and a disposable, screw-on sensing module. Average service life of the module is six months.

c. Reference electrode: A single-junction reference electrode, Orion Model 90-01, or equivalent.

- d. Magnetic stirrer: with teflon-coated stirring bars, approx. $\frac{3}{8}$ in. diameter and 1 in. length. (9.5 mm diameter, 25 mm length).
- e. Beaker: 150 cm^3 , Pyrex or PMP plastic or equivalent.
- f. Volumetric pipettes, Class A: Various sizes are required for preparing dilutions of standards and samples. The use of a good quality microliter pipette with disposable tips is particularly convenient for quantitative transfer of volumes of 1 cm^3 and smaller.
- g. Volumetric flasks, Class A: Various sizes for preparing calibration standards and dilutions of filtrate samples.
- h. Graph Paper: 3- or 4-cycle semilogarithmic for preparing calibration curves.
- i. Standard calcium solution, 1000 mg/L : Weigh 2.4972 g reagent grade calcium carbonate on an analytical balance, add about 50 mL water, and dissolve by the dropwise addition of concentrated HCl (about 10 mL will be required). When dissolution is complete, dilute to 1000 mL in a volumetric flask. This solution is also readily available from laboratory supply houses.
- j. Potassium chloride solution, 4 molar: Dissolve 298 g reagent grade KCl in deionized water and dilute to 1 liter. This solution is used as an ionic strength adjustor (ISA).
- k. Buffer solution, pH 5.0: Dissolve 136 g sodium acetate trihydrate ($\text{NaC}_2\text{H}_3\text{O}_2 \times 3 \text{H}_2\text{O}$) in about 500 mL of deionized water. Add 57.5 mL of glacial acetic acid ($\text{HC}_2\text{H}_3\text{O}_2$) and dilute to 1 liter. This solution is used to adjust the pH of all standard and sample solutions to a pH of 4.8 – 5.2.
- l. Potassium chloride solution, 4 molar, saturated with silver: Transfer about 50 mL of the 4 M KCl (see 15.2j) to a 2-ounce plastic bottle and add silver nitrate solution dropwise until a cloudiness persists. This solution is used as the filling solution for the reference electrode (if desired, this solution is available from Orion, Catalog No. 90-00-1).

Note: Reference to the Orion electrodes is for information only and is not intended to exclude from use electrodes from other manufacturers. Other electrodes are acceptable so long as the reproducibility, resolution, and stability are equivalent to the Orion products. Not all commercially available electrodes have been evaluated, but the Orion products have consistently provided satisfactory performance. It is the responsibility of each analyst to verify the performance of his electrode system before continuing with any quantitative determination.

15.3 PROCEDURE-PREPARATION OF ELECTRODES

153.1 If the Orion 93-20 electrode is used, remove the sensing module from the vial and screw it firmly into the electrode body, taking care to ensure that the rubber washer is in place.

153.2 Shake down the electrode in the manner of a thermometer.

15.3.3 Soak the electrode in distilled or deionized water for at least 15 minutes, then store in a dilute calcium solution, containing 10 – 100 mg/L calcium, until ready for use.

15.3.4 By means of either an eye dropper or syringe, fill the single-junction reference electrode with the 4 molar potassium chloride (saturated with silver ion).

15.3.5 If other types of sensing or reference electrodes are used, follow the instructions of the manufacturer when preparing them for use.

15.4 PROCEDURE-OPERATIONAL CHECK OF ELECTRODE SYSTEM

15.4.1 The two properties of the electrode system which will have the greatest influence on accuracy and reproducibility are the slope and the stability. The slope is defined as the change in potential observed when the concentration changes by the factor of 10. The stability of the readings is an indication of the amount of noise (rapidly, continuously changing readings) and drift (readings slowly changing in one direction).

15.4.2 When performing the initial calibration with the standard solutions according to the instructions in either 15.5 or 15.6, record the actual measured potential for the 1 mg/L and the 10 mg/L standard solutions. For a new calcium electrode (or sensing module) the difference between these two readings should be between 25 and 29 millivolts. The slope will normally decrease with time. When the slope reaches a value of approximately 23 millivolts the module should be replaced.

15.4.3 If, when performing the initial calibration, there is an indication of noise or drift, refer to the troubleshooting section of the electrode manual. There are a number of possible causes for these effects with any electrode system, and the cause is normally found only by a process of elimination. If the analyst is unable to achieve a stability level which meets the manufacturer's specifications, the sensing electrode (or module) should be replaced.

15.5 PROCEDURE-MEASUREMENTS USING A METER WITH DIRECT CONCENTRATION READOUT CAPABILITY

15.5.1 Prepare a 10 mg/L calcium standard and a 1 mg/L calcium standard by serial dilution of the 1000 mg/L standard solution.

15.5.2 Add the ISA solution (4 molar potassium chloride) to each standard at the rate of 2 cm^3 of ISA per 100 cm^3 of standard. Add 1 cm^3 of the acetate buffer to each standard.

15.5.3 Transfer the standards to 150 cm^3 beakers. Place the 1 mg/L standard on the magnetic stirrer, place the electrodes in the standard solution, and stir at a moderate rate.

15.5.4 Wait for a stable reading (normally 1-2 minutes), then adjust the controls to display the value of the standard, following the procedure described in the meter instruction manual.

15.5.5 Remove the electrodes, rinse with distilled water, blot dry, and place in the 10 **mg/L** standard. Stir at a moderate rate.

15.5.6 Wait for a stable reading, then adjust the controls to display the **value** of the standard. The meter **and** electrode system are now calibrated.

15.5.7 Prepare a 1: 100 dilution of each mud filtrate sample.

15.5.8 Transfer 100 **cm³** of diluted sample to a 150 **cm³** beaker, add 1 **cm³** of the acetate buffer solution, add 2 **cm³** ISA, and stir at a moderate rate.

15.5.9 Rinse electrodes with distilled water, blot dry, immerse in sample and wait for a stable reading. The value displayed on the meter is the calcium concentration in the diluted sample. This value multiplied by the dilution factor (100 in this case) will give **the** calcium concentration in the original sample.

Note: Concentration values above or below the value of the standards used for calibration are still reliable, since the response of the calcium electrode is linear from about 0.2 **mg/L** to above 1000 **mg/L**.

15.6 PROCEDURE-MEASUREMENTS WITH INSTRUMENTS THAT PROVIDE EITHER DIGITAL OR ANALOG READOUT IN MILLIVOLTS

15.6.1 Prepare 100 **mg/L**, 10 **mg/L** and 1 **mg/L** calcium standards by serial dilution of the 1000 **mg/L** standard solution.

15.6.2 Add the ISA solution (4 molar potassium chloride) to each standard at the rate of 2 **cm³** of ISA per 100 **cm³** of standard. Add 1 **cm³** of the acetate buffer solution to each standard.

15.6.3 Transfer the standards to 150 **cm³** beakers. Place the 1 **mg/L** standard on the magnetic stirrer, place the electrodes in the standard solution and stir at a moderate rate.

15.6.4 Adjust the controls of the instrument so that the readout is in terms of millivolts, wait until the reading is stable, and record the reading.

15.6.5 Remove electrodes, rinse, blot dry and place in the 10 **mg/L** standard. When the reading is stable, record the reading.

15.6.6 Repeat Step **15.6.5** with the 100 **mg/L** standard.

15.6.7 Plot the millivolt readings on the linear axis and concentration in **mg/L** on the logarithmic axis of standard 3 or 4-cycle semilogarithmic paper.

15.6.8 Transfer 100 **cm³** of sample which has been diluted to 1:100 to a 150 **cm³** beaker, add 2 **cm³** of the ISA solution, add 1 **cm³** of the acetate buffer, place the electrodes in the solution, and stir at a moderate rate.

15.6.9 Record the reading when stable, and determine the concentration of the diluted sample by use of the calibration curve. This value multiplied **by** the dilution factor gives the calcium content of the original sample.

Note: For accurate measurements, all standards and samples should be at ambient temperature. Some magnetic stirrers generate considerable heat, and it may be necessary to place a layer of insulating material (plastic or cardboard) between the stirrer and beaker. Between measurements, rinse electrodes and blot **dry** with tissue. After immersion in sample or standard solutions, check end of electrode for air bubbles and remove if present.

Always read and carefully follow the manufacturer's instructions **regarding** the care, maintenance, storage, servicing and troubleshooting of the instrument and electrodes.

16 Density of Solids by the Stereopycnometer

16.1 DESCRIPTION

16.1.1 This method covers the determination of the true volume of dry powders and granular solids. When an accurately weighed sample of the solid is used, the density (mass per unit volume, normally expressed as **g/cm³**) can easily be calculated.

16.1.2 The method employs the **Archimedes** principle of fluid displacement for the volume measurement. The fluid in this case is actually a gas, preferably high-purity helium. The method is designed to be used primarily for the determination of the density of **barite and** other weighting materials, **but** it can also be used for determining the density of drill solids and commercial clays.

16.2 EQUIPMENT

The following equipment is needed:

- Oven: Regulated to 220 ± 5°F (105 ± 3°C).
- Desiccator.
- Balance: Precision off 0.05 g.
- Helium bottle.
- Stereopycnometer, such as Quantachrome **Stereopycnometer** model SPY-2, calibrated **and** operated according to manufacturer's instructions.

16.3 PROCEDURE

Note: The stereopycnometer method may be used as an alternative method for determining the specific gravity of **barite**. In case of dispute, the **Le Chatelier** Flask Method will prevail (See API Specification 13A).

16.3.1 Turn the instrument on and allow 10 - 15 minutes for the pressure transducer to warm up and stabilize.

16.3.2 Remove the sample cell by rotating the plastic cover on top of the sample cell holder clockwise, and insert the "U" shaped bayonet connector into the slots near the top of the cell to lift out the sample cell.

16.3.3 To the tared sample cell, add 150 ± 0.05 g barite which has been oven dried for at least two hours at $220 \pm 5^\circ\text{F}$ ($105 \pm 3^\circ\text{C}$) and cooled to room temperature in a desiccator.

16.3.4 Insert sample cell in compartment. Replace sample cell holder cover and rotate clockwise until the metal to metal contact is made with the cell holder.

16.3.5 **Purge** the system and sample with helium by closing the "FLOW" toggle valve and opening the "CELL VENT" toggle valve. Turn selector valve to "V_A IN".

16.3.6 Open "CELL, VENT CONTROL" fully counter-clockwise.

16.3.7 Close "FLOW CONTROL" needle valve fully clockwise. Open the "FLOW" toggle valve. The "FLOW CONTROL" needle valve should be adjusted to give a slow rate of gas bubbling through water in a beaker. Remove tubing from water.

16.3.8 After 20 minutes of flow, close both the "FLOW" toggle valve and the "FLOW CONTROL" needle valve. With the "CELL VENT" and "CELL VENT CONTROL" open, zero the digital display. Wait for a stable zero reading.

16.3.9 Set the selector valve to "VA OUT" and close "CELL VENT" valve.

Note: Close "CELL VENT" valve slowly; otherwise the reading on the digital display may deviate from zero.

16.3.10 Open "FLOW" toggle valve and pressurize to slightly less than 19.5 psig. Use the "FLOW CONTROL" needle valve to control the rate of pressurization. Stop flow with the "FLOW" toggle valve.

Note: Do not exceed 20 psig. If this happens, a flashing "20" will appear on the digital display and the measurement will have to be repeated.

16.3.11 Record display reading when stabilized.

16.3.12 **Turn** the selector valve to "V_A IN".

16.3.13 Record display reading when stabilized.

16.3.14 Vent pressure slowly to prevent blowing powder out of the cell by opening "CELL VENT" with the "CELL VENT CONTROL" cracked open.

16.4 CALCULATION

16.4.1 Calculate the volume of the barite using the equation supplied with the instrument.

16.4.2 Calculate the density of the barite using the following equation:

$$\text{Density, g/cm}^3 = \frac{\text{Mass of sample, g}}{\text{Sample volume, cm}^3} \quad (23)$$

17 Density of Solids by Air Pycnometer

17.1 DESCRIPTION

17.1.1 The density of drilling fluid weighting material is determined by the **Le** Chatelier Flask Method when the specification testing is being done.

17.1.2 The air comparison pycnometer may be used as an alternate method for determining density.

17.1.3 The air pycnometer compares the density relative to air, and is measured in g/cm^3 .

17.1.4 In case of dispute, the **Le** Chatelier Flask Method will prevail.

17.2 EQUIPMENT

The following equipment is needed:

- Oven: regulated $220 \pm 5^\circ\text{F}$ ($105 \pm 3^\circ\text{C}$).
- Desiccator.
- Balance:** sensitive to ± 0.01 g.
- Air comparison pycnometer-for example, Beckman Model 930 or equivalent, calibrated and operated according to manufacturer's instructions.

17.3 PROCEDURE-AIR PYCNOMETER METHOD

17.3.1 Open coupling valve and remove sample cup. Rotate both reference and measuring handwheels counter-clockwise to rest against stops.

17.3.2 Turn measuring handwheel clockwise until starting number for the instrument is set on counter.

17.3.3 To the sample cup, add $80 \text{ g} \pm 0.05$ of barite which has been oven dried for at least two hours and cooled to room temperature in a desiccator.

17.3.4 Insert cup in compartment. Lock sample cup in place by pressing handle down **firmly**.

17.3.5 Wait 15 seconds. **Close** coupling valve.

17.3.6 Turn both handwheels simultaneously or alternately until reference handwheel rests against stop. Keep pointer on scale during this process.

17.3.7 Wait 10 seconds. Bring point to null with measuring handwheel.

17.3.8 Open coupling valve. Record counter reading. Correct if necessary to give sample volume.

17.4 CALCULATION-AIR PYCNOMETER METHOD

$$\text{Specific gravity} = \frac{\text{weight sample}}{\text{sample volume}} \quad (24)$$

18 Sodium in Water-Based Drilling Fluids by the Ion-Selective Electrode (ISE) Method

18.1 DESCRIPTION

18.1.1 This procedure determines the amount of sodium in water-based drilling fluids. This procedure provides an accurate, quantitative value for the sodium content. At the present time there is no other approved field method for performing this analysis.

18.1.2 Sodium is present to some extent in all water-based fluid systems. Concentrations may range from less than 100 mg/L to over 100,000 mg/L in saturated salt systems.

18.1.3 This method is applicable to all concentrations. None of the other materials normally present in drilling fluids have been found to interfere under the conditions of this test. The combination of dilution and treatment with ionic strength adjuster (ISA) effectively eliminates interferences.

18.1.4 The sodium electrode, because of the glass membrane construction, is not as sturdy as the liquid membrane electrodes such as potassium and calcium. Strict adherence to the specific practices described in the instruction manuals is necessary in order to obtain reliable data.

18.1.5 The electrode should be stored in the designated storage solution only.

18.1.6 Never expose the glass sensing membrane of the electrode to fresh water—always rinse with the rinse solution designated in the applicable electrode manual.

18.1.7 NEVER wipe or blot electrodes dry with a cloth or tissue. Let the rinse solution drain, then shake off as much as possible of remainder.

18.1.8 Verify calibration at least every 2 hours during use, using a freshly-prepared standard for each recalibration.

18.1.9 Minimize the use of glass containers and apparatus, and when glass is used, ensure that it is a chemically-resistant glass. Store standards and reagents in plastic containers when possible.

18.1.10 Recondition the electrode when response reading is sluggish or a noticeable drift is detected. Be sure to follow

instructions in the applicable electrode manual when reconditioning.

18.2 EQUIPMENT

The following equipment is needed:

- a. Meter: a pH meter capable of providing a millivolt readout with a resolution of at least 0.1 millivolt, or a combination pH/ISE meter with provisions for direct concentration readout. Use of the combination instrument is recommended.
- b. Sodium ion-selective electrode: Orion Model 84-1 1, or equivalent.
- c. Reference electrode: Orion Model 80-03 reference electrode, or equivalent.
- d. Magnetic stirrer with plastic-coated stirring bars.
- e. Beakers: 150 cm³, PMP plastic or chemically-resistant glass (plastic is preferred).
- f. Volumetric pipettes, Class A: Various sizes for preparing dilutions of standards and samples. The use of microliter pipettes with disposable plastic tips is recommended for measuring and transferring small volumes (1.0 cm³ or less).
- g. Volumetric flasks, Class A: Various sizes.
- h. Graph paper: 3- or 4-cycle semilogarithmic paper for preparing calibration curves when using a meter which provides readout in millivolts only.
- i. Standard Sodium Solutions: Orion 1000 mg/L sodium standard, or equivalent (Orion 841108); Orion 100 mg/L sodium standard or equivalent (Orion 941107); Orion 10 mg/L sodium standard, or equivalent (Orion 94 1105); and 1 mg/L sodium standard, prepared immediately before use by serial dilution of the 10 mg/L standard or the 100 mg/L standard. These standards are prepared by dissolving sodium chloride, NaCl (CAS #7647-14-5) in deionized water.
- j. Sodium Ionic Strength Adjuster (ISA): A solution 4 M in both ammonium chloride, NH₄Cl (CAS #12125-02-9) and ammonium hydroxide, NH₄OH (CAS #1336-21-6), Orion 841111, or equivalent.
- k. Electrode rinse solution: Add 10 cm³ of ISA to water and dilute to 1 liter.
- l. Sodium electrode reconditioning solution: Ammonium bifluoride, NH₄HF₂ (CAS #1341-49-7), 0.1 M, Orion 841113, or equivalent.
- m. Sodium electrode storage solution: A solution which is 5 M in sodium chloride, NaCl (CAS #7647-14-5), 0.08 M in ammonium chloride, NH₄Cl (CAS #12125-02-9), and 0.08 M in ammonium hydroxide, NH₄OH (CAS #1336-21-6), Orion 841101, or equivalent.
- n. Reference electrode filling solution: 2 M ammonium chloride, NH₄Cl (CAS #12125-02-9)—use for filling the reference electrode. Do not use the KCl filling solutions normally supplied with the reference electrode.

Note: Reference to the Orion electrodes and supplies is for information only and is not intended to exclude other products. Not all available electrodes have been evaluated, but there are differences in

the performance characteristics of different products and each analyst should verify the satisfactory performance of any system which is used.

18.3 PROCEDURE-PREPARATION AND OPERATIONAL CHECK OF ELECTRODE SYSTEM

18.3.1 Remove sodium electrode from shipping container and rinse thoroughly with electrode rinse solution.

18.3.2 Soak the electrode for at least 2 hours, preferably overnight, in the sodium electrode storage solution.

18.3.3 Fill the reference electrode, using the 2 M ammonium chloride solution.

18.3.4 Connect electrodes to the meter, and rinse with the electrode rinse solution.

18.3.5 Transfer 100 cm³ of the 10 mg/L sodium standard to a 150 cm³ beaker, stir solution on the magnetic stirrer at a moderate rate, while adding 10 cm³ of the ISA, and then immerse the electrodes.

18.3.6 Operate the meter in the millivolt mode. When a stable reading is obtained, record the electrode potential in millivolts.

18.3.7 Remove electrodes and rinse with the electrode rinse solution. Do not wipe or blot electrode dry. Place 100 cm³ of the 100 mg/L sodium standard in a 150 cm³ beaker, add a stirring bar and stir at a moderate rate. Add 10 cm³ of the ISA to the standard sodium solution while stirring.

18.3.8 Immerse electrodes and record the potential, in millivolts, when a stable reading is obtained.

18.3.9 The difference between the two readings is the slope of the electrode. This difference should be in the range of 54–59 millivolts. If it is less than 54 millivolts, recondition the sodium electrode according to the procedure detailed in the electrode instruction manual.

18.4 PROCEDURE-MEASUREMENTS USING A METER WITH DIRECT CONCENTRATION READOUT CAPABILITY

18.4.1 Transfer 100 cm³ each of the 100 mg/L sodium standard and the 10 mg/L sodium standard to 150 cm³ beakers.

18.4.2 Add 10 cm³ of the ISA solution to each standard solution.

18.4.3 Place the 10 mg/L standard on the magnetic stirrer and stir at a moderate rate. Immerse electrodes and wait for a stable reading (normally 1–2 minutes), then adjust the controls on the instrument to display the value of the standard.

18.4.4 Remove electrodes, rinse, place in the 100 mg/L standard, and when a stable reading is obtained adjust controls to display the value of the standard.

18.4.5 Prepare either a 1:100 or a 1:1000 dilution of each filtrate sample, depending on the expected sodium concentration. The sodium concentration in the diluted sample in which the measurement is made should be between 10 and 100 mg/L.

18.4.6 Transfer 100 cm³ of diluted sample to a 150 cm³ beaker, add 10 cm³ of ISA, immerse electrodes and wait for a stable reading. The value displayed is the sodium concentration of the diluted sample. This value multiplied by the dilution factor will give the sodium concentration in the original filtrate.

Note: If the concentration of sodium in the diluted sample is a greater than 100 mg/L make an additional dilution in order to provide a concentration between 10 mg/L and 100 mg/L. If it is between 1 mg/L and 10 mg/L the value obtained should be reliable since the response of the electrodes are normally linear to values less than 1 mg/L.

18.5 PROCEDURE-MEASUREMENTS USING A METER WITH READOUT IN MILLIVOLTS

18.5.1 Transfer 100 cm³ each of the 100 mg/L, 10 mg/L, and 1 mg/L sodium standards to 150 cm³ beakers.

18.5.2 Add 10 cm³ of ISA to each standard.

18.5.3 Place the 1 mg/L standard on the magnetic stirrer and stir at a moderate rate. Immerse electrodes, and record reading in millivolts when stable.

18.5.4 Repeat 18.5.3 with the 10 mg/L standard, then the 100 mg/L standard. Be sure to rinse electrodes thoroughly when moving from one standard to another.

18.5.5 Plot the millivolt readings on the linear axis and the sodium concentration in mg/L on the logarithmic axis of 3- or 4-cycle semilogarithmic paper.

18.5.6 Transfer 100 cm³ of sample which has been diluted 1:100 or 1:1000 to a 150 cm³ beaker and add 10 ml of ISA.

18.5.7 Stir at a moderate rate, immerse electrodes and record reading when stable. Determine the sodium in the diluted sample by use of the calibration curve prepared in 18.5.5 above. This value multiplied by the dilution factor gives the sodium content of the original sample.

18.5.8 If the calculated concentration of sodium in the diluted sample is outside the 1–100 mg/L range, prepare another sample, using a dilution which will provide a concentration within this range.

19 Aging of Water-Based Drilling Fluids

19.1 DESCRIPTION

19.1.1 Drilling fluid aging is the process of allowing a drilling fluid sample, which contains all required ingredients and has been subject to a period of shear, to more fully develop its **rheological** and filtration properties through additional time for hydration, etc., of its components. The time period needed to more fully develop properties varies from as little as several hours (more commonly overnight, or 16 hours) or as much as several days. The aging can be done at either ambient or elevated temperatures, depending upon the system under investigation.

19.1.2 This section is not intended to cover aging technology appropriate to either oil-based drilling fluids or brines used for completion, packer, or **workover** fluids.

19.1.3 The purposes of this section are:

- a. To inform those unfamiliar with drilling fluid formulation and testing of the techniques and equipment commonly used, in a laboratory setting, to mix and age **drilling** fluid systems, and
- b. To remind those already familiar with drilling fluid formulation and testing of some basic guidelines and safety considerations.

Note: It is strongly recommended that this complete document be read before any elevated temperatures aging of drilling fluid samples is carried out.

19.1.4 Users of this section on aging must familiarize themselves with the types of test, testing methods, and properties measured found in API Recommended Practice 13B-1, and the methodologies used in API Recommended Practice 131, Sections 10 – 12 on (evaluating) Filtration Control Agents, Methylene Blue Test and Deflocculation Tests for Thinner Evaluations, respectively.

19.2 PROCEDURE-PRACTICES COMMON TO PREPARATION, HANDLING AND TESTING OVER ALL TEMPERATURE RANGES

19.2.1 Water-Based Drilling Fluids and Components

Most drilling fluid formulations contain a base liquid and additives which must be dissolved or mechanically dispersed into the liquid to form a homogenous fluid. The resulting fluid may contain one or more of the following: water-dispersible (soluble) polymers or resins, clays or other insoluble but dispersible fine solids, and soluble salts. The fluids are mixed or sheared for times appropriate to achieve a **homogeneous** mixture and are then set aside to “age.” Aging is done under conditions which vary from static to dynamic and from ambient to highly elevated temperatures.

19.2.2 Mixing/Blending/Shearing Devices

Drilling fluid formulations are commonly mixed with various shearing devices which may be either fixed speed or variable speed. The motors may turn mixing shafts with rounded “propellers,” sharp blades, wave-form shapes, or others. Single shaft or multiple shaft devices are used. Some examples of the more widely used mixer types are: Hamilton Beach® Model 936, or equivalent; **Dispersator**®, or equivalent high shear mixer, Waring **Blendor**® or equivalent; **MultiMixer**® Model 9B with 9B29 impeller, or equivalent; **Silverson**® 14R mixer, and **Oliani**®, or equivalent, mixer. Nozzle shear devices are also used to prepare some formulations.

These shearing devices vary widely in the amount of shear they impart. This means that long shearing times may be required for low shear devices to achieve complete dissolution/hydration of fluid components; high shear devices can produce nearly completely yielded drilling fluid blends in a few minutes. Aging of drilling fluid samples tends to minimize differences in properties which can result from shearing treatment.

19.2.3 Property Ranges of Drilling Fluids or Base Fluids, Emphasizing pH Values

Formulation and expected ranges of fluid properties for different types of systems used to evaluate chemical additives are found in API Recommended Practice 131, Section 10, “Filtration Control Agents.”

The **pH** of drilling fluid formulation containing bentonite clays usually never falls below a value of 8.5 unless acidic material are added to these based fluids. Except for some drilling fluid systems **viscosified** with certain water-soluble polymers, the **pH** of these formulations is usually raised above **pH** 8.5 with **alkalinity** control agents, such as sodium or potassium hydroxide (caustic soda or caustic potash), or calcium hydroxide (lime).

CAUTION: Wear eye protection whenever drilling fluids are formulated, handled or tested.

Fluid alkalinity is lowered by the reaction of hydroxide groups with aluminosilicates (clays), gradually at ambient temperature and rapidly at elevated temperature. Some Drilling Fluid additives require that alkalinities be maintained within certain narrow, but elevated, ranges in order to function at optimum levels. Therefore, **pH** levels are often raised after aging if there has been a substantial **pH** drop.

19.2.4 Drilling Fluid Sample Storage, Disposal, and Testing Methods

Drilling fluid formulations or base fluid (bentonite slurries) are kept until used up or until properties move outside acceptable ranges. Some laboratories do not routinely keep unused portion of test muds at ambient temperatures longer than

some arbitrary time, such as one week or one month. Prolonged storage is best done in refrigerated units with capability of storage up to one year at 40°F (4°C).

Drilling fluid samples which contain certain organic materials or polymer products subject to fermentation (i.e., starches, biopolymers, etc.) should either have a preservative added or should be discarded after a suitable time which precedes the expected onset of biological degradation.

Discard unused samples in an environmentally appropriate manner based upon known ingredients. For example, clays, most polymers derived from **celluloses** and starches, and **lignites** pose no special threat to **living** organisms and may be sent to normal landfill sites. Dispose of drilling fluids containing toxic materials as appropriate for chemical waste.

The recommended test equipment and methods for water-based drilling fluids may be found in API Recommended Practice 13B-1 and API Recommended Practice 131.

19.3 PROCEDURE-AMBIENT TEMPERATURE DRILLING FLUID SAMPLE PREPARATION AND AGING

19.3.1 Equipment

Drilling fluids may be mixed at ambient temperatures in a variety of open containers made from metal, plastic or glass. Little or no incompatibility exists between normal water-based drilling fluid and these materials under ambient temperature conditions.

19.3.1.1 Glass containers commonly used include the following:

- a. Crack-resistant glass, such as home canning jars, supplied by Mason®, Kerr®, or Ball®, or equivalent jars or bottles.
- b. Heat-resistant containers, such as Pyrex®, or equivalent materials.

19.3.1.2 Coverings/lids for glass vessels are commonly plastic or metal, often lined with plastic, rubber, enamel, or other relatively "inert" material. Plastic containers and lids may be made from polyethylene, polypropylene, or other suitably inert, mechanically strong, and durable materials.

A wide variety of metal containers may be used at ambient temperatures. Commonly used metal include various grades of stainless steel (303, 304 or 316) or mild carbon steel. More inert metal formulations may be used but these are expensive for ambient temperature use. Inexpensive metal container/cells, such as aluminum bronze, are not recommended for **any** drilling fluid formulations. Reactions between bronze aging cells and numerous drilling fluid systems or additives have been observed.

19.3.1.3 Mixer: as described in 19.2.2.

19.3.1.4 Balance: precision ± 0.01 g.

19.3.2 Procedure—Aging Drilling Fluids at Ambient Temperatures

19.3.2.1 After the initial shearing/blending stages of drilling fluid base or full sample preparation, most additional aging at ambient temperatures is done statically.

19.3.2.2 Prepared samples are left overnight (16 hours) or for days, if necessary, to reach stable or desirable properties.

19.3.2.3 Rolling or tumbling (rotating) the prepared samples may be used to combat settling of solid components or segregation of liquid layers, but this is seldom done at ambient temperatures.

19.3.2.4 Drilling fluids are routinely aged at ambient temperatures in metal, plastic or glass containers capped to prevent loss of moisture. The fluid properties may continue to change until the components are fully hydrated.

19.3.2.5 The properties may continue to change as some components react further in the aqueous suspension to degrade due to secondary reactions between various components or due to **bacterial** attack on susceptible materials. **Biocides** may be added to prolong shelf life of these susceptible formulations.

19.3.2.6 Drilling fluid formulations which are to **be** kept for extended periods are usually stored under refrigeration at 40°F (4°C). Drilling fluids stored at ambient temperatures are often discarded after several days and are seldom kept for longer than a month. Formulations are monitored to ensure that the properties remain within acceptable ranges.

19.4 PROCEDURE—MODERATE TEMPERATURE AGING TO 150°F (66°C)

19.4.1 Description

Drilling fluid samples to be aged at elevated **temperatures** are mixed at ambient temperatures as described in 19.2.2 and 19.3.1. Additional materials may be added to **slurries** which have already been aged at ambient or elevated **temperatures**.

19.4.2 Equipment

- a. Containers: Most of the acceptable glass, plastic or metal containers used for ambient aging (19.2.2 and 19.3.1) have been found to be acceptable for static or dynamic aging up to and including 150°F (66°C).
- b. Covers: Acceptable covers are ones which seal the container.
- c. Oven: Regulated to 50°F (66°C), or other suitable temperature.
- d. Mixer: As described in 19.2.2.

19.4.3 Procedure

Aging at elevated temperature is usually done for one of the following reasons:

- a. To hasten the equilibrium hydration level of clays and/or polymers in the fluid system, or
- b. To expose the fluid to thermal conditions similar to field conditions.

For exposure to elevated temperature, drilling fluid samples are placed in one of a wide variety of commercially available or custom-built ovens. These ovens range from being tabletop, portable models small enough to hold only a few one-pint samples, to large floor-standing units having very large capacities. These ovens also vary from static units to those equipped with rollers or pulley systems to roll the containers or to tumble (rotate) strapped-in cells.

Preliminary studies indicate that the methods of heating and cooling the drilling fluid samples (i.e., preheating the oven versus no preheating, cooling samples in open or closed ovens or in water), uniformity of temperature throughout the oven (due to the degree of adequate air circulation), and exact heating exposure time will affect the data values measured. To optimize data repeatability between test runs, use the same heating and cooling methods and heat exposure time for each sample in a series of comparative tests.

One artifact of aging at temperatures through 150°F (66°C) is that some glass and plastic vessels containing drilling fluids, especially those with elevated salinity (e.g., to sea water salinity of approximately 19,000 mg/L chlorides) develop modes pressures in the air overlying the liquid drilling fluid. After 15–30 minutes of heating these vessels are removed from the oven, the lids carefully loosened to allow excess pressure to escape (“burping”), the lids restored tightly and the containers replaced in the oven to continue heating to the desired temperature. Sometimes a second check for pressure buildup is advisable.

19.4.4 Maintaining Fluid Properties at Moderate Temperatures

As mentioned in 19.2.3, alkalinity levels drop with time when exposed to clay (or other drilling fluid additives which exhibit weakly acidic behavior). The reaction between clays and hydroxyl ions is accelerated with increasing temperature. Therefore, alkalinities need to be more closely monitored and more frequently adjusted, if desired, in drilling fluids which are exposed to elevated temperature.

Purging and blanketing samples with nitrogen gas appears to impart strong benefits in reducing oxidative degradation of polymer-treated samples. The use of nitrogen leads to better reproducibility in testing polymer drilling fluids.

19.4.5 Storage and Testing Practices for Samples Aged at Moderate Temperatures

Even though samples may have been aged at elevated temperature for time periods ranging from hours to days, prolonged storage of such samples, if desired, is usually done at ambient temperatures or under refrigeration, depending upon the length of time for anticipated storage.

In addition to the rheology and filtration measuring devices mentioned in API documents Specification 13A, Recommended Practice 13B-1, and Recommended Practice 131, there are ambient to elevated temperature dynamic filtration and elevated temperature rheometers available for use. Included among the devices for testing rheology are Fann® Model 50 Rheometer, Fann Consistometer, Huxley Bertram® Rheometer, Haake® Rotoviscometer.

19.5 PROCEDURE-AGING AT ELEVATED TEMPERATURES OVER 160°F (70°C)

19.51 Description

Drilling fluid samples to be aged at substantially elevated temperatures are also blended at ambient temperatures as described in 19.2.2 and 19.3.1. Additional quantities of pre-existing components or new materials designed to increase stability at elevated temperature may be added to slurries which have already been aged at ambient or elevated temperatures.

19.5.2 Equipment

Once the 150–160°F (66–71°C) temperature threshold is crossed, only aging cells constructed from suitable metals are used to expose drilling fluids to elevated temperatures.

Note: Aluminum bronze cells are not recommended for drilling fluids at any temperature.

19.5.2.1 Since aging temperatures are often selected to be near the estimated or anticipated bottom-hole temperatures, aging cells chosen must be designed and constructed in such a way as to meet (or exceed) the pressure and temperature requirements of the aging tests.

19.5.2.2 Beyond the issue of being able to meet the temperature and pressure requirements of specific aging conditions, the choice of the proper metal is complex. It depends both upon the ultimate temperature which the sample and cell will experience as well as the salinity of the fluid sample.

19.5.2.3 The largest percentage of aging between 150 and 350°F (66 and 177°C) is done in cells constructed from various grades of stainless steel (303, 304 or 316, but not 314). Sometimes high carbon-content steel cells are used if there is a strong desire to simulate field conditions wherein “mild steel” pipe is in use and to which drilling fluids are naturally exposed. These stainless or mild steel cells, when properly used, have never experienced catastrophic failure, i.e., they

have not exploded upon failure. When these cells have failed, they have simply sprung leaks.

19.5.2.4 For prolonged exposure to elevated salinity at high temperatures, e.g., 20,000 mg/L chlorides at 350°F (177°C), cells constructed from premium metals may be desirable. Examples of these premium metals are **Hastalloy®**, **Inconel® 600**, or **Incolloy® 825**. Cells made from these premium metals with the same wall thickness as standard stainless steel cells, however, suffer from lower strength ratings. Therefore, cells made from premium metals do not have the same pressure ratings as stainless steel and must be derated to lower pressure limits.

19.5.2.5 A key consideration in the use of metal aging cells at elevated temperatures is that care must be taken to ensure that the cells not be overfilled. When enclosed liquids expand with increasing temperature, an inadequate head space of gas (air) can lead to a piston effect as liquid hydraulically loads against the cell cap. Leaving gaps of 1.5 – 2 in. (3.8 – 5.1 cm) between the top of the liquid and the cell cap or not filling the cell over 85 – 90% of its volume appear to be adequate for safe operation during most aging conditions between 150 – 400°F (66 – 190°C).

19.5.3 Additional Safety Considerations

19.5.3.1 It needs to be emphasized that metal aging cells only be used to age drilling fluids containing the usual types or classes of mud additives. Do not use materials whose high temperature compatibility with drilling fluid chemical and minerals are in doubt. Test such compatibilities in equipment, such as autoclaves, capable of withstanding or designed for extreme pressure service. For protection, perform these compatibilities in specially designed enclosures.

19.5.3.2 The only known instances of catastrophic failures (explosions) of stainless steel aging cells resulted from either the cells being used inappropriately as chemical reaction autoclaves or being over-filled.

19.5.3.3 The types of failures of metal aging cells reported either from inadequate inspection and maintenance or from prolonged use for drilling fluids aging were simply leaks which developed in the cells. The drilling fluids leaked into the aging ovens but caused no danger to laboratory personnel.

19.5.3.4 Rupture disks may be placed in the cell caps if there is concern about cell failure. Since the use of rupture disks essentially derates the pressure rating of the metal aging cell, conduct tests at temperatures that keep the pressure lower.

19.5.4 Metal Aging Cell Maintenance

19.5.4.1 It is suggested that the cell bodies and cell caps have serial numbers recorded along with the cell contents whenever they are used. Further, it is suggested that separate

sets of metal aging cells be maintained for testing fresh water fluids, high chloride fluids, and oil-based fluids. The cells exposed to fresh water should have the longest useful life.

19.5.4.2 Stainless steel aging cells tend to experience chloride-stress corrosion cracking or pitting in environments where there has been prolonged exposure at high temperatures, e.g., over 220°F (105°C) to fluid with substantially elevated chloride levels (over several thousand mg/L). Such concerns are legitimate, but there are a number of mitigating circumstances which exist in drilling fluids aging technology. These tend to lessen the severity of the effects experienced.

19.5.4.3 One drilling fluid aging practice that mitigates accelerated corrosion is that metal aging cells are washed and visually inspected after each use to see if they have been affected by the exposure. The onset of pitting and cracking corrosion is often detected at such a time. Affected stainless steel cells are then usually sand blasted to remove corrosion spots. The cells can be returned to useful service.

19.5.4.4 Even when no obvious corrosion in a metal aging cell is detected, historical experience has shown that periodic sand blasting of the cells removes developing problem sites, keeps the surface passive, and significantly increases the useful life of the cell.

19.5.4.5 Other mitigating circumstances include:

- The cells are used intermittently and cleaned, and are not used in continuous process exposures.
- The cells used in drilling fluids testing are made from metal bar stock and are not welded. The absence of the stress point which would be present in welded or cast cell bodies mitigates against the likelihood or severity of corrosion which might be experienced under certain aggressive conditions of exposure.
- Drilling fluid formulations have high pH levels and typically low oxygen contents.

19.5.5 Aging Practices at Elevated Temperatures

19.5.5.1 If the correct match of cell metallurgy and drilling fluid is established for a given temperature regime, then the samples may be statically or dynamically aged in a suitable oven, as described in 19.4.3.

19.5.5.2 Dynamic aging should be performed in rolling or rotating ovens capable of achieving and safely maintaining temperatures of 150 – 400°F (66 – 190°C). A 16-hour minimum aging time is recommended for such exposures. Depending on the temperature stability of the product(s) being evaluated, the time and temperature may need to be adjusted.

19.5.5.3 For static aging, any qualified oven or the same ovens may be used but with their rollers/rotating mechanisms turned off. Static aging at elevated temperatures simulates

the conditions when a drilling fluid is left quiescent **downhole** during regular rig operations.

19.5.5.4 For high temperature wells over 300°F (135°C), the usual 16-hour aging interval is a reasonable simulation of the time a drilling fluid is left in the hole during a bit trip. For longer operations, such as extended electric well logging runs, 48 to 72-hour aging periods is appropriate. In choosing a test temperature, it should be noted that fluid left in the hole may take many hours to even approach the actual bottomhole temperature since wellbores are cooled by the circulating fluid. Therefore, a test temperature below the bottomhole temperature may be the most realistic simulation of **downhole** conditions for bit trip periods.

Note: A meaningful API test to use in evaluating long term gelation of heat-aged drilling fluids is the shear strength test found in API Recommended Practice 13B-1, Appendix entitled "Shear Strength Measurement Using Shearometer Tube."

19.6 CAUTIONS ABOUT INERTNESS AND CHEMICAL COMPATIBILITY IN HIGH TEMPERATURE AGING CELLS

19.6.1 Chemical Compatibility of Materials within Metal Aging Cells

19.6.1.1 As mentioned in 19.5.3, do not subject materials of unknown or suspicious reactivity to high temperature aging in cells containing drilling fluids.

19.6.1.2 Do not include materials which are known to produce or suspected of initiating or taking part in highly **exothermic** reactions in heat aged drilling fluid formulations.

19.6.1.3 Exclude materials that are known or suspected strong **redox** (oxidation-reduction) reagents from heat-aged drilling fluid situations.

19.6.2 Inertness of Metal Aging Cells to Chemicals

19.6.2.1 The greatest degree of inertness to potentially corrosive conditions is provided by the nickel-based alloys, such as the **Hastalloy®**, **Inconel® 600** and **Incolloy® 825** formulations.

19.6.2.2 Iron may be leached from the various stainless steels by high chloride fluids at high temperatures. This iron removal causes pits and cracks after stainless steel cells are exposed to severe temperature/chloride conditions.

19.6.2.3 Various materials can be used as more inert liners within standard stainless steel cells. These are listed in 19.6.3.

19.6.3 Use of Lining Materials in Metal Aging Cells

19.6.3.1 Some investigators have found that test results from high chloride-content drilling fluids containing **water-soluble** polymers may not have been reproducible when

stainless steel vessels are used. Iron appears to be leached from the cell bodies causing these anomalies.

19.6.3.2 If the more economical stainless steel cells are used under aggressive fluid aging conditions, lining materials may be inserted in the aging cells. Materials which have seen limited to widespread acceptance as liners include: Teflon®, glass, and ceramic materials.

19.6.3.3 It is generally agreed that Teflon liners may be safely used to at least **400°F (190°C)** and offer the **highest** degree of inertness to a wide variety of drilling fluid formulations.

19.6.3.4 Glass liners may be attacked at the high **pH** levels used in many drilling fluid formulations. These are also susceptible to attack by certain highly saline conditions at elevated temperatures.

19.6.4 Considerations Regarding Metal Plating to Enhance Contamination Resistance of Aging Cells

19.6.4.1 Plating of stainless steel cells with inert or noble metals, such as gold, has been suggested to avoid the much greater expense of fabricating new cells from premium metal alloys, such as **Inconel® 600**, etc.

19.6.4.2 This otherwise attractive consideration has one serious drawback: if the plated cell is scratched, the exposed steel will **experience** accelerated severe localized corrosion rates above what unplated steel would experience. Therefore, scratching of plated cells leads to an earlier likelihood of cell leaks and failures.

19.6.4.3 Since drilling fluid samples aged at elevated temperatures often become very viscous or even hard, it is often necessary to scrape solidified samples from the cells. Despite great care being taken, there is a risk that scratches can occur when removing solidified drilling fluids. Therefore, **gold-plated** cells are not recommended for aging drilling fluids at elevated temperatures.

19.6.5 Contrast Between Drilling Fluid Material Performance in Inert and Real Work Environments

19.6.5.1 Be cautious about interpreting results from laboratory aging studies using cells which contain inert metallurgy or inert material **liners**. Drilling fluids in field applications are exposed to the steel in drill pipe, **usually** made from mild carbon steel.

19.6.5.2 While the amount of steel surface exposure to drilling fluids is **much less than that** experienced in steel aging cells, it is still more than would be experienced within inert aging cells.

19.6.5.3 Therefore, field results can be at variance with the results obtained from aging tests conducted under more ideal inert conditions in the laboratory environment.

19.7 OBTAINING SUPPLIES AND SAFETY AND DESIGN ADVICE FOR THE AGING OF DRILLING FLUID SAMPLES

19.7.1 Equipment Suppliers

19.7.1.1 Glass and plastic jars and bottles are widely available from laboratory supply houses, container suppliers, and many retail and wholesale outlets.

19.7.1.2 Metal aging cells, ovens suitable for aging drilling fluids at elevated temperatures, and/or drilling fluid testing equipment and ancillary supplies may be obtained from oil-field testing equipment suppliers.

19.7.2 Metallurgical Consultants

For best advice on acquiring the proper metal aging cells required for severe service (elevated chlorides at high temperatures), contact a reputable consulting metallurgist or firm which specializes in metallurgical consulting.

19.7.3 Pressure Vessel Consultants

For advice on pressure vessel design for a situation not adequately addressed by commercially available or special order metal aging cells, contact a firm or individual with established credentials in pressure vessels design.

20 Aging of Oil-Based Drilling Fluids

26.1 PURPOSE AND DESCRIPTION OF THIS SUBCLAUSE

20.1.1 This subclause is intended:

- a. To inform those unfamiliar with formulating and testing drilling fluids about the techniques and equipment commonly used in a laboratory to mix and age oil-based drilling fluid systems.
- b. To serve as a reminder of the basic guidelines and safety considerations in working with aging practices.

20.1.2 This subclause is not intended to cover aging technology of water-based drilling fluids or brines used for completion, packer, or workover fluids.

20.1.3 Drilling fluid aging is the process in which a drilling fluid sample, previously extensively sheared, is allowed to more fully develop rheological and filtration properties. The time required for the sample to develop properties may be from 16 hours to as much as several days, at either ambient or elevated temperatures.

Note: It is strongly recommended that this complete document be read from beginning to end before any aging of drilling fluid samples at elevated temperatures is attempted.

Note 2: Users of this subclause should familiarize themselves with the types of tests, testing methods, and properties found in API Recommended Practice 13B-2, and the methodologies described in API Recommended Practice 131.

20.2 EQUIPMENT

20.2.1 Shearing Devices

20.2.1.1 Description

Shearing devices, which may be either fixed speed or variable speed, are used to blend drilling fluid formulations. The motors may turn mixing shafts with rounded propellers, corrugated impellers, sharp blades, or wave-form shapes, among others. Either single shaft or multiple shaft devices are used.

26.2.1.2 Equipment

Examples of widely used shearing devices are: the Hamilton Beach® Model 936 mixer, or equivalent; Dispersator®, or equivalent; MultiMixer® Model 9B with 9B29X impeller, or equivalent; Silverson® L4R mixer, or equivalent; and the Oliani® Mixer, or equivalent. Some users prefer nozzle shearing devices.

Note: The preceding shearing devices vary widely in the amount of shear they impart. Aging of drilling fluid samples tends to minimize differences in properties that may result from these samples being prepared by different shearing devices.

20.2.2 Mixing and Storage Containers

20.2.2.1 Glass Jars and Bottles

20.2.2.1.1 Glass containers commonly used to prepare batches up to one gallon (4 liters) in volume are crack-resistant glass jars such as those supplied for home canning purposes by Mason®, Kerr®, and Ball®, or equivalent types of jars/bottles that come with metal screw-on caps; commercially available straight-sided round jars; and wide-mouth round jars. The latter types are commonly used in rolling ovens up to about 150°F (65°C). All types may be securely fastened in tumbling ovens operating below 150°F (65°C).

20.2.2.1.2 Heat-resistant containers such as Pyrex®, or equivalent materials, are also used to temperatures of 150°F (65°C).

Note: Coverings/lids for glass vessels are plastic or metal. Inert seals or liners [e.g., those made of polytetrafluoroethylene (PTFE)] are recommended as the best for long-term use regardless of the composition of the lid.

20.2.2.2 Plastic Jars and Containers

Acceptable plastic containers and lids include polyethylene, polypropylene, or other suitably inert, mechanically strong, and durable materials. These materials may not be compatible with some oil-based fluids. Most oil-based drilling fluids may be mixed and stored in thick-walled polyethylene commercial buckets. Thin-walled plastic containers, such as milk, soft drink, or juice bottles, are not recommended.

20.2.2.3 Metal Containers

20.2.2.3.1 Variety

A wide variety of metal containers may be used at ambient temperatures. Most commonly used metals are various grades of stainless steel (303, 304, or 316) or mild carbon steel. More inert metal formulations may be used, but these are expensive for ambient temperature use.

Note: Aluminum bronze cells are not recommended for any drilling fluid formulations. Electrochemical reactions between bronze aging cells and numerous drilling fluids systems or additives have been observed.

20.2.2.3.2 Maintenance

It is suggested that the cell bodies and cell caps have serial numbers that are logged along with the cell contents whenever the cells are used.

20.2.2.3.3 Corrosion Cracking or Pitting

A source of concern is the tendency of stainless steel aging cells to develop chloride-stress corrosion cracking or pitting in environments where there has been prolonged high-temperature exposure — over 220°F (105°C) — to fluids with substantially elevated chloride levels (over several thousand milligrams per liter). This should not be a concern when testing oil-based muds in which the water-in-oil emulsion is maintained throughout the aging process.

20.2.2.3.3.1 Such concerns are legitimate, but there are a number of mitigating circumstances which exist in drilling fluids aging technology.

20.2.2.3.3.2 One drilling fluid aging practice that mitigates accelerated corrosion is the cleaning and visual inspection of metal aging cells after each use to see if they have been affected by fluid exposure. The onset of pitting and cracking corrosion is often detected at such a time.

20.2.2.3.3.3 Stainless steel cells on which the beginnings of corrosion are observed are usually sand blasted clean to

remove corrosion spots: the cells can then be returned for as many as 25 years of frequent service.

20.2.2.3.3.4 Even when no obvious corrosion in a metal aging cell is detected, experience has shown that periodic sand blasting of the cell removes developing anionic anode sites, keeps the surface passive, and increases the useful life of the cell.

20.2.2.3.3.5 Another circumstance that reduces the corrosion in well-maintained metal aging cells is the fact that those used in drilling fluids testing are made from metal bar stock and are not welded. The absence of welded stress points reduces the potential for corrosion initiation points under severe exposure conditions.

20.2.2.3.3.6 Another fact reducing corrosion is that many oil-based drilling fluids have high pH levels in the internal water phase and low oxygen contents due to the oil phase.

20.2.3 Liners for Metal Cells

20.2.3.1 When concerns exist about reactivity of a particular type of static metal aging cell with drilling fluid that will be exposed to elevated temperatures, liners of material believed to be more inert to such fluids may be used.

20.2.3.2 Example liner materials include: glass, ceramic, and polytetrafluoroethylene (e.g., Teflon, or equivalent).

20.2.3.3 Liners of each type may be commercially obtained.

20.2.4 Ovens

Many commercially available or custom-built oven types are used for aging drilling fluid formulations.

20.2.4.1 Static ovens range from portable (tabletop) models that can only hold a few sample jars or cells to larger (floor-standing) multishelf models that may hold dozens of samples.

20.2.4.2 Adequate air circulation within ovens to ensure uniform temperature distribution is essential to the validity of aging.

20.2.4.3 Ovens may be rated to operate at temperatures from 150°F (65°C) to 500°F (260°C).

20.2.4.4 Ovens may be static or equipped with rollers or pulley systems that provide rolling of jars or cells or tumbling (rotating) of strapped-in cells.

CAUTION: Ovens used with oil-based fluids shall not have exposed heating elements that could cause ignition of combustible materials in the event of sample leakage.

20.3 PROCEDURE: PRACTICES COMMON TO PREPARATION, HANDLING, AND TESTING OF OIL-BASED DRILLING FLUIDS-ALL TEMPERATURE RANGES

20.3.1 Oil-Based Drilling Fluids and Their Components

20.3.1.1 Oil-based drilling fluids contain one or more base liquids and additives that must be dissolved or mechanically dispersed into the liquid to form a homogenous fluid. The resulting fluid may contain one or more of the following: oil-dispersible, soluble polymers or resins; and organophilic clays or other insoluble but dispersible fine solids.

20.3.1.2 The oil-based muds may be all-oil muds, i.e., formulated without an aqueous phase, or invert oil-based emulsion where the oil is the continuous phase and the emulsified water phase is the dispersed phase. Typically the oil-based mud in the water phase contains sodium chloride (NaCl) or calcium chloride (CaCl₂).

20.3.1.3 Fluids are mixed or sheared to achieve a **homogeneous** mixture of the components and then set aside to age. Aging may be done under static or dynamic conditions and from ambient to elevated temperatures.

20.3.2 Mixing/Blending/Shearing Devices

20.3.2.1 The shearing devices listed in 20.2.1 vary widely in the amount of shear that they impart. Long shearing times may be required for low shear devices to achieve fairly complete dissolution or solvation of fluid components.

20.3.2.2 High shear devices, such as the Silverson LAR mixer, or equivalent, can produce nearly completely dispersed blends in a few minutes.

20.3.2.3 Aging of drilling fluids samples tends to minimize differences in properties that may result from using different shearing devices.

20.3.3 Property Ranges of Oil-Based Drilling Fluids or Their Base Fluids

20.3.3.1 The base oils vary widely in their inherent viscosities. These viscosities also vary with temperature, differently for **different** base fluids.

20.3.3.2 Therefore, the drilling fluid properties vary widely. These may also be **affected** by the presence of different emulsifier packages (or the absence of them for all-oil fluids), and/or organophilic clays and/or oil-soluble viscosifying polymers used to provide suspending characteristics.

20.3.3.3 Some emulsifiers or wetting agents are calcium soaps and require the presence of lime to be activated. Sometimes lime is added to control acid gases, such as hydrogen

sulfide or carbon dioxide, which may be encountered in drilling operations or result from high temperature breakdown of some chemical additives.

20.3.4 Sample Storage and Disposal

20.3.4.1 Oil-based drilling fluid samples are not generally subject to deterioration over time. However, it is considered good practice to limit testing reliance on samples that are over 1-month old, unless the samples have been refrigerated at 40°F (4°C).

20.3.4.2 Unused samples shall be discarded, based on their known ingredients, in accordance with local statutory regulations.

20.4 PROCEDURE: AMBIENT TEMPERATURE AGING

20.4.1 Preparation and Storage Containers

The drilling fluids may be mixed at ambient temperature in a variety of open containers made from metal, plastic, or glass, as described in 20.2.2. Incompatibility may exist between oil-based drilling fluids and some plastic materials. There should be no problems with metal or glass containers.

20.4.2 Aging

20.4.2.1 After the initial shearing/blending stages of drilling fluid base or full sample preparation, most additional aging at ambient temperatures is done statically.

20.4.2.2 **Prepared** samples are **left** overnight (16 hours) or for days, if necessary, to reach stable or desirable properties.

20.4.2.3 Rolling or tumbling (rotating) of the **prepared** samples may be used to combat settling of solid components or segregation of liquid layers, but this is seldom done at ambient temperatures.

20.4.2.4 Oil-based drilling fluids are routinely aged at ambient temperatures in metal or glass containers that are capped to prevent loss of organic vapors. The fluid properties should change very little from this point on.

20.4.3 Storage

20.4.3.1 Drilling fluids to be kept for extended periods are usually stored **under** refrigeration at 40°F (4°C).

20.4.3.2 Drilling fluids stored at ambient temperatures are often discarded after several days and are seldom kept for longer than a month.

20.4.3.3 Fluid samples are monitored to ensure that acceptable properties are maintained.

20.5 PROCEDURE: MODERATE TEMPERATURE AGING-AMBIENT TO BELOW 150°F (65°C)

20.5.1 Preparation and Storage Containers

Oil-based drilling fluid samples to be aged at elevated temperatures are still usually blended at ambient temperatures, as described in 20.3.2 and 20.4.

20.5.1.1 Additional materials may be added to slurries that have already been aged at ambient or elevated temperatures.

20.5.1.2 Most of the acceptable glass and metal containers used for ambient temperature aging (see 20.3.2 and 20.4) have been found to be acceptable for static or dynamic aging up to 150°F (65°C).

20.5.2 Aging

20.5.2.1 Study of drilling fluids at elevated temperature is usually done for one of the following reasons:

- To hasten the chemical equilibrium of the system.
- To expose the fluid to thermal conditions similar to what may occur in a field application.

20.5.2.2 For exposures to elevated temperatures, drilling fluid samples are placed in any one of a number of commercially available or custom-built ovens, as described in 20.2.4.

20.5.2.3 The methods of heating and cooling the samples (e.g., preheating the oven versus not preheating, cooling samples in open or closed ovens or in water), uniformity of temperature throughout the oven (due to the degree of adequate air circulation), and exact heating exposure time may affect the data measured.

20.5.2.4 To optimize data repeatability between test runs, the same heating and cooling methods and heat exposure times shall be employed for each in a series of comparative tests.

20.5.3 Maintaining Fluid Properties

Purging and blanketing samples with nitrogen gas appears to have the benefit of reducing oxidative degradation of polymer-treated samples. This use of nitrogen leads to better reproducibility.

20.5.4 Storage and Testing Practices

20.5.4.1 Storage of samples is usually done at ambient temperatures or under refrigeration, depending upon the length of time for storage, even though the samples have been aged at elevated temperatures for time periods ranging from hours to days.

20.5.4.2 In addition to the rheology and filtration measuring devices mentioned in API Specification 13A, Recommended Practice 13B-2, and Recommended Practice 131,

there are ambient to elevated temperature dynamic filtration and elevated temperature rheometers available for use.

20.5.4.3 Examples of the devices for testing rheology are the following: Fann Model 50C Viscometer, Fann Model 5STD Consistometer, Fann Model 70 Viscometer, EG&G Chandler Engineering Model 7400 Rheometer, and Haake RV20/D 100 Viscometer, or equivalent.

20.6 PROCEDURE: SUBSTANTIALLY ELEVATED TEMPERATURES AGING-OVER 150°F (65°C)

20.6.1 Preparation and Storage Containers

20.6.1.1 Drilling fluid samples to be aged at substantially elevated temperatures are still usually blended at ambient temperatures as described in 20.3.2 and 20.4.

20.6.1.2 Additional quantities of pre-existing components or new materials designed to increase stability at elevated temperatures may be added to slurries that have already been aged at ambient or elevated temperatures.

20.6.1.3 Once the 150°F (65°C) temperature threshold is crossed, only aging cells constructed from suitable metals are used, as stated in 20.2.2.3.

20.6.2 Aging

20.6.2.1 Aging temperatures are often selected to be near the anticipated bottom hole temperature and near the anticipated temperature for applications where specific systems or additives are used. Aging cells shall be constructed to meet or exceed the pressure and temperature requirements of the aging test parameters.

20.6.2.2 The choice of the proper metal is a complex issue and goes beyond the metal's ability to meet the temperature and pressure requirements. The choice depends on the ultimate temperature that the sample and cell will experience and the chloride salinity of the aqueous phase in the oil-based fluid sample.

20.6.2.3 By far the largest percentage of drilling fluids sample aging between 150°F and 400°F (65°C and 190°C) is done in cells constructed from various grades of stainless steel (303, 304, or 316, but not 314).

20.6.2.4 Sometimes, high carbon-content steel cells are used if there is a strong desire to simulate field conditions wherein mild steel pipe is in use and to which drilling fluids are naturally exposed. These stainless or mild steel cells, when properly used, have never experienced catastrophic failure, i.e., they have not exploded upon failure. When these cells have failed, they have simply sprung leaks.

20.6.2.5 For prolong exposure of aging cells to elevated salinity at high temperatures, cells constructed from premium metals may be desirable. Examples of premium metals are Hastalloy® C, Inconel® 600, or Incolloy® 825, or their equivalent.

20.6.2.5.1 Cells made from these premium metals that have the same wall thickness as the standard stainless steel cells do, however, suffer from lower strength ratings.

20.6.2.5.2 Cells made from premium metals do not have the same pressure ratings as stainless steel and must be derated to lower pressure limits. Consult the supplier before conducting tests with such cells.

20.6.2.6 A key consideration in the use of metal aging cells at elevated temperatures is that care must be taken not to overfill the cells.

20.6.2.6.1 When enclosed liquid expands with increasing temperature, an inadequate head space of gas (air) can lead to a piston effect as liquid hydraulically loads against the cell cap.

20.6.2.6.2 Leaving a gap of 1.5 – 2.0 in. (40 – 50 mm) between the liquid and the cell cap (or not filling over 85 – 90% of the cell's volume) appears to be adequate for safe operation under aging conditions between 150°F and 400°F (65°C and 190°C).

20.6.2.7 If the correct match of cell metallurgy and drilling fluid within has been established for a given elevated temperature, the sample may be statically or dynamically aged in an oven as described in 20.5.3.

20.6.2.8 Dynamic aging shall be performed in rolling or rotating ovens capable of achieving and safely maintaining temperatures of 150° – 400°F (65° – 190°C). A 16-hour minimum aging time is usually recommended. Depending upon the temperature stability of the product(s) being evaluated, the time and temperature may be adjusted.

20.6.2.9 For static aging the same ovens may be used if their rollers/rotating mechanisms are turned off. Static ovens are also available. Static aging at elevated temperatures simulates the conditions experienced in a drilling fluid left quiescent down hole.

20.6.2.10 For high temperature wells [>300°F (>135°C)] the usual 16-hour aging interval is a reasonable simulation for a bit trip. For longer operations, such as extended electric well logging runs, 48- to 72-hour aging periods are more appropriate.

20.6.2-1 1 It should be noted in choosing a test temperature that fluid left in the hole can take many hours to approach the actual bottom hole temperature. Wellbores are cooled by the circulating fluid. Therefore, a test temperature lower than the bottom hole temperature may be a more realistic simulation of down hole conditions for bit trip periods.

Note: A meaningful API test for evaluating long term gelation of heat-aged chilling fluids is the shear strength test found in API Recommended Practice 13B-2. This is especially useful for oil-based muds considered for use as packer fluids.

20.6.3 Safety Options

20.6.3.1 Metal aging cells shall be used only to age drilling fluids containing known, usual additives.

20.6.3.2 Experimental material whose high temperature compatibility with the usual chemicals/minerals is in doubt shall not be aged at elevated temperatures in metal cells. Such compatibility shall be tested in equipment such as autoclaves designed for extreme pressure service.

20.6.3.3 The only known catastrophic failures (explosions) of stainless steel aging cells have resulted either from the cells being used inappropriately as chemical reaction autoclaves (instead of drilling-fluid aging) or from the cells being overfilled.

20.6.3.4 Cells that failed because of inadequate inspection and maintenance simply leaked.

20.6.3.5 Rupture disks may be placed in the cell caps if there is concern about cell failure. This generally leads to using aging temperature conditions amenable to lower pressure buildup, because the use of rupture disks essentially lowers the pressure rating of the cell.

CAUTION: It is especially critical to use ovens with no exposed heating elements if cells with rupture disks are used (see 20.2.4.4).

20.7 PROCEDURE: CAUTIONS ABOUT INERTNESS AND CHEMICAL COMPATIBILITY IN HIGH TEMPERATURE AGING CELLS

20.7.1 Chemical Compatibility of Materials With Metal Aging Cells

Materials known to produce, or materials suspected of initiating or taking part in, highly exothermic reactions shall not be included in heat-aged drilling fluid formulations.

20.7.2 Inertness of Metal Aging Cells to Chemicals

20.7.2.1 The, greatest degree of inertness of metal aging cells to potentially corrosive environments is provided by the nickel-based alloys such as the Hastalloy®, Inconel® 600, and Incolloy® 8925, or equivalent, formulations.

20.7.2.2 Iron can be leached from the various stainless steels by high-chloride fluids at high temperatures. This removal of iron leads to the pits and cracks seen after exposing stainless steel cells to severe temperature/high-chloride level conditions.

20.7.2.3 Various materials have been proposed and used as more inert liners within standard stainless steel cells. These are listed in 20.7.3.

20.7.3 Considerations Regarding Metal Plating to Enhance Contamination Resistance of Aging Cells

20.7.3.1 Gold-plated cells are not recommended for aging drilling fluids at elevated temperatures. Plating stainless steel cells with inert or noble metals, such as gold, has been suggested as a means of avoiding the greater expense of fabricating new cells from the premium metal alloys such as Inconel® 600, or equivalents. This attractive consideration has one serious drawback: if the plated cell is scratched, the exposed steel is prone to accelerated, more severe localized corrosion rates than unplated steel would experience. Scratching of plated cells could lead to earlier cell leaks and failures.

20.7.3.2 Drilling fluid samples aged at elevated temperatures often become very viscous or even **hard** and often require scraping solidified samples from the cells. Despite the fact that great care is taken, it is risky to assume that scratches never result when cleaning solidified drilling fluids from aging cells.

20.7.4 Contrast Between Drilling Fluid Material Performance in Inert and Real World Environment

20.7.4.1 Caution should be used when interpreting the results of laboratory aging studies using cells made from inert metal or containing inert material liners. Field applications expose drilling fluids to the steel in drill pipe, which is usually made from mild carbon steel.

20.7.4.2 Field results may vary from laboratory results obtained under more ideal conditions. While the amount of steel surface exposure to drilling fluids is much less than that occurring in steel aging cells, it is still more than would occur in inert aging cells.

20.8 PROCEDURE: OBTAINING SUPPLIES AND SAFETY AND DESIGN ADVICE

20.8.1 Equipment Suppliers

20.8.1.1 Glass and plastic jars and bottles are widely available from laboratory supply houses, container suppliers, and many retail and wholesale outlets.

20.8.1.2 Metal aging cells, ovens suitable for aging drilling fluids at elevated temperatures, and/or drilling fluid testing equipment and ancillary supplies may be obtained from companies that specialize in drilling fluid testing equipment.

20.8.2 Metallurgical Consultants

For the best advice on acquiring the proper metal aging cells required for severe service (involving elevated chlorides at high temperatures), contact a reputable metallurgist or firm that specializes in metallurgical consulting.

20.8.3 Pressure Vessel Consultants

For advice on pressure vessel design, if one expects to experience a situation that commercially available or special order metal aging cells may not adequately address, contact a firm or individual with established credentials in pressure vessel design.

21 Shale Particle Disintegration Test by Hot Rolling

21.1 DESCRIPTION

21.1.1 This procedure involves grinding, sieving, and placing shale particles into a drilling fluid before aging the fluid, either statically or dynamically. Retrieving, cleaning, and reweighing of the particles will give an indication of the fluid's tendency to prevent shale dispersion. This test is only a relative measure and should be included as part of a comprehensive testing program.

21.1.2 A drilling fluid's ability to inhibit the disintegration of shale cuttings is important. The laboratory method presented here can be used for evaluating the inhibiting properties of drilling fluids with regard to shale particle disintegration. This test is intended as a comparison of various whole mud compositions.

21.1.3 Hot rolling, though commonly used, is not the only test indicating the inhibitive characteristics of drilling fluids. There are many laboratory techniques used to assess the degree to which a drilling fluid formulation may weaken and disintegrate a shale immersed in it. Hot rolling can usually give a wide variety of results. Strict control and reporting of the test shale condition and the rheology of the test fluid as well as the washing of shale are all necessary.

21.1.4 This procedure neither specifies nor limits shale selection and condition (e.g., water content), although they are extremely important. Properly preserved shale samples should be used. It is strongly recommended that the shale be as near to its in-situ moisture content as possible and to not have been air dried or oven dried prior. The way the shale has been preserved prior to its use in the test should be recorded with the results.

21.1.5 The drilling fluid rheology variable has proven difficult to control from test to test. As this can have an independent influence on the shale recovery, it is recommended that the rheological properties (e.g., the apparent viscosity) be

measured at the test temperature and be recorded with the results. This enables consideration of the effect of rheology among the variables. Modest changes in the rheology from one fluid to another can strongly influence shale dispersion final results.

21.1.6 It should be noted that the results do not necessarily imply **wellbore** stability. This test may not be appropriate for evaluation of inhibiting properties of certain additives.

21.2 EQUIPMENT

The following equipment with the following specifications should be used for this procedure:

- a. Balance: precision of ± 0.01 g.
- b. Roller oven: typically 17 to 35 revolutions per minute (rpm).

Note: For best results all tests shall be conducted with the same roller oven to ensure constant test conditions.

- c. Aging cells (not aluminum bronze).
- d. Clock or timer.
- e. Weighing boats.
- f. Spatula.
- g. Wash bath or sink, at least $\frac{1}{2}$ gallon (2 liters) in size.
- h. Inhibitive wash water that is either:
 - 1. A synthetic version of the mud base brine.
 - 2. A fluid known to be substantially inhibitive to the shale used, e.g., 1.5 lb per barrel potassium **chloride** (KCl).

Note: Wash water of salinity much greater than the mud base brine can cause sieve blocking due to mud coagulation.

- i. Inhibitive wash water supply at 0.53 gallon per minute, ± 0.05 gallon per minute (2 liters/minute, ± 0.2 liter per minute), with approximately a 0.30 in. (0.75 cm) ID outlet. See Figure 8 for wash system.

- j. Sieves:
 - 1. One 3.9 in. (10 cm) diameter sieve with 0.02 in. (0.5 mm) aperture, U.S. Sieve Number 35.
 - 2. One 3.9 in. (10 cm) diameter sieve with 0.157 in. (4 mm) aperture, U.S. Sieve Number 5.
 - 3. One 3.9 in. (10 cm) diameter sieve with 0.079 in. (2 mm) aperture, U.S. Sieve Number 10.
 - 4. Sieve base, 3.9 in. (10 cm) in diameter.

- k. Drying oven regulated to 220°F , $\pm 5^{\circ}\text{F}$ (105°C , $\pm 3^{\circ}\text{C}$).

- l. Desiccator.

21.3 PROCEDURE

The procedure for performing the shale particle disintegration test is as follows:

- a. Obtain shale pieces with as near to in-situ moisture content as possible. Just prior to use, screen shale pieces to size

less than 0.157 in. (4 mm) and greater than 0.079 in. (2 mm). The moisture content of bulk supply **shall** be established at the time of sampling by the method for API **Bentonite**, Section 4 of Specification **13A**, and recorded.

- b. **Prescreen** the drilling fluid through a 0.02 in. (0.5 mm) sieve to remove any oversize particles that could be incorrectly reported as recovered shale.

- c. Add 350 cm^3 of drilling fluid measured either by volume or by weight to each aging cell.

- d. Add 20.0 g of shale pieces (screened prior to use) to the fluid in each aging cell, seal, and quickly and gently agitate to wet and separate the particles.

Note: With high viscosity muds it may be advantageous to add mud and shale simultaneously to achieve separate wetting of particles or gently stir the mud with a spatula while adding shale.

- e. Place the aging cells in a roller oven preheated to the desired temperature and roll for a predetermined time period.

Note: The **rolling** time must be the same for **all** fluids being evaluated on a given shale. The time is shale dependent and some shales will require more rolling time than others before sufficient disintegration occurs to allow discrimination between fluids.

- f. After the rolling **period** is complete and the aging cells have cooled to a safe handling temperature, transfer them to a static position to cool further to a temperature constant for all sequential **recovery** operations (e.g., cold water bath to room temperature).

- g. The contents of each aging cell are in turn poured onto a numbered, preweighed 0.02 in. (0.5 mm) aperture sieve while wash water is applied to the screen to prevent viscous blocking. The residual contents of the aging cell are also displaced onto the sieve with wash water (use of a wash bottle is convenient).

Note: Viscous blocking, if it occurs, may be alleviated by applying a small pressure above the sieve with an apparatus such as that shown in Figure 9.

- h. The drilling fluid is washed from the remaining shale pieces by a wash water flow of 0.53 gallon Per minute, ± 0.05 gallon per minute (2 liters/minute, ± 0.2 liter/minute), through a hose with a circular outlet of approximately 0.39 in. (0.75 cm) ID. Water is systematically played across the **full** area of the screen many times for a period of approximately 1 minute or **until** the shale and screen are **visually** free of mud residue. See Figure 8.

- i. Transfer the sieve to a $\frac{1}{2}$ gallon (2 liter) bath containing **tapwater** and quickly but gently submerge the sieve (at an angle to avoid an air trap) and remove it so that the sieve and shale have been rinsed of wash water.

- j. Place the sieve onto a preweighed sieve base and dry in the drying oven to a constant weight. Cool in a desiccator and reweigh to 0.01 g. This is the **final** dried weight.

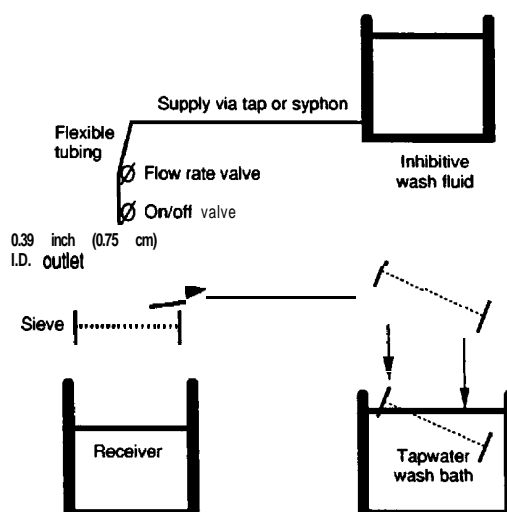


Figure 8—Wash System

21.4 CALCULATION

21.4.1 Results are expressed as:

$$\text{recovery (\%)} = \frac{\text{final dried weight} \times 100}{\text{initial dried weight}} = 100 \frac{a}{b} \quad (25)$$

where

$a = y - z$, the final dried shale weight,

$b = 20.0 (100 - c)/100$, the initial dried shale weight,

c = initial shale moisture content, %,

y = weight of sieve + base + dried recovered shale,

z = weight of sieve + base.

21.4.2 Record conditions used (shale preservation technique, history prior to test, initial moisture content, mud rheology, test temperature, and rolling time) and the percent shale recovery. Replication of the test is recommended. Reproducibility is typically within 4% relative standard.

22 High Viscosity (Regular) Polyanionic Cellulose

22.1 DESCRIPTION

22.1.1 High viscosity polyanionic cellulose, PAC-HV, is a water-soluble polymer produced only from cellulose chemically reacted with carboxy-methyl (anionic) groups and shall not contain any other polysaccharides such as starch, guar, or other naturally occurring polymers or their derivatives. The product is a free-flowing powder or granular powder.

22.1.2 PAC-HV is widely used in water-based drilling fluid for a variety of applications such as filtration control, viscosity, and inhibition. Although field use may vary, this procedure focuses on viscosity and filtration control characteristics.

22.1.3 The intention of this procedure is to present a simple and reproducible method for assessing the performance properties of PAC-HV. **Specification** parameters have not yet been developed for this material, but it is the intention of API Committee 3/Subcommittee 13 to adopt specifications based on data generated by users.

22.1.4 A synthetic sea water mud is used for determining the filtration control and apparent viscosity of PAC-HV.

22.2 MOISTURE-EQUIPMENT

The following equipment will be needed to measure the moisture content of PAC-HV:

- Oven: Regulated to 220°F, ± 5°F (105°C, ± 3°C).
- Balance: Precision of ± 0.1 gram.
- Evaporating dish.
- Spatula.
- Desiccator with calcium sulfate (CAS #7778-18-9) desiccant or equivalent.

22.3 MOISTURE-PROCEDURE

The procedure for measuring the moisture content of PAC-HV is as follows:

- Weigh 10.0 g, ± 0.1 g, PAC-HV sample into a tared evaporating dish. Record the weight added to the evaporating dish.
- Dry the sample in the oven for 4 hours.

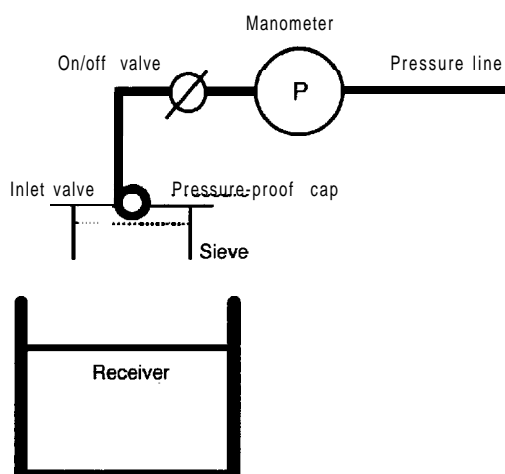


Figure 9—Pressure System

- c. Cool to room temperature in desiccator.
- d. Reweigh the evaporating dish containing the dried sample. Record the weight.

22.4 MOISTURE-CALCULATION

$$\text{moisture weight \%} = \quad (26)$$

$$\frac{100 \times [\text{original weight (g)} - \text{dried weight (g)}]}{\text{original weight (g)}}$$

22.5 FLUID PROPERTIES-EQUIPMENT

The following equipment will be needed to measure the fluid properties of the test fluid:

- a. Thermometer: 32 – 140°F, ± 2°F (CL WC, ± 1°C).
- b. Balance: Precision off 0.01 gram.
- c. Mixer (e.g., Multimixer Model 9B with 9B29X impellers, or equivalent, as shown in API Specification 13A, Figure 2.1). Each spindle will be fitted with a single sine-wave impeller approximately 1 in. (25 mm) in diameter mounted flash side up.
- d. Mixer container: Approximate dimensions-7 in. (180 mm) deep, 3¹³/₁₆ in. (97 mm) ID top, 2 ³/₄ in. (70 mm) ID bottom (e.g., Hamilton Beach Mixer Cup M110-D, or equivalent).
- e. Spatula.
- f. Container: Glass or plastic with stopper or lid for salt solutions.

g. Motor-driven direct indicating viscometer as referenced in API Recommended Practice 13B-1.

h. Timers: Two interval, mechanical or electrical, precision of ± 0.1 minute.

i. Filter press: As referenced in API Recommended Practice 13B-1.

j. Graduated cylinders: **One** 10 cm³, ± 0.1 cm³ (TC); one 500 cm³, ± 5 cm³ (TD).

k. Sea salt, ASTM D1141-52 (or latest revision), Formula A.

l. API **Standard** Base Evaluation Clay (see API Specification 13A).

m. Potassium chloride (CAS #7447-40-7).

n. Sodium bicarbonate (CAS #144-55-8).

o. Deionized or distilled water.

22.6 FILTRATE VOLUME-PROCEDURE

The procedure for measuring the filtrate volume of the test fluid is as follows:

- a. Add 42.0 grams, ± 0.01 g, sea salt to 1000 cm³, ± 2 cm³, de-ionized water.
- b. To 358 g of the sea salt solution, add 35.00 g, ± 0.01 g, potassium chloride.
- c. After stirring 3 minutes, ± 0.1 minute, add 1.00 g, ± 0.01 g, sodium bicarbonate.
- d. After stirring 3 minutes, ± 0.1 minute, add 28.00 g, ± 0.01 g, API Standard Base Evaluation Clay.
- e. After stirring 5 minutes, ± 0.1 minute, remove the container from the mixer and scrape its side with the spatula to dislodge any material adhering to the container wall. Be sure

all material clinging to the spatula is incorporated into the suspension.

f. Replace the container on the mixer and continue to stir an additional 5 minutes, ± 0.1 minute.

g. Weigh 1.00 g, ± 0.01 g, PAC-HV. Add the PAC-HV slowly at a uniform rate over a time interval of about 60 seconds while stirring the suspension on the mixer.

Note: The PAC-HV shall be added away from the impeller shaft but in the vortex to minimize dusting.

h. After stirring 5 minutes, ± 0.1 minute, remove container from the mixer and scrape its side with the spatula to dislodge any PAC-HV adhering to the container walls. Be sure all PAC-HV clinging to the spatula is incorporated into the suspension.

i. Replace the container on the mixer and continue to stir. **The container** may need to be removed from the mixer and the side scraped to dislodge any adhering PAC-HV after another 5 or 10 minutes. Total mixing time elapsed from the beginning of PAC-HV addition shall equal 20 minutes, ± 1 minute.

j. Age the suspension for 16 hours, ± 0.5 hour, in a sealed or **covered** container at 77°F , $\pm 2^{\circ}\text{F}$ (25°C , $\pm 1^{\circ}\text{C}$). Record storage temperature and storage duration.

k. After aging, stir the suspension on the mixer for 5 minutes, ± 0.1 minute.

l. Pour the PAC-HV suspension into a filter **press** cell. Before adding the suspension, be sure each part of the filter cell is dry and that **no** gaskets are distorted or worn. The temperature of the suspension **shall be** 77°F , $\pm 2^{\circ}\text{F}$ (25°C , $\pm 1^{\circ}\text{C}$). Pour the suspension to within $1/2$ in. (13 mm) of the top of the cell. Complete assembly of the filter press cell. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.

m. Set one timer for 7.5 minutes, ± 0.1 minute, and the second timer for 30 minutes, ± 0.1 minute. Start both timers and adjust pressure on the cell to 100 psi, ± 5 lb/psi (690 kPa, ± 35 kPa). Pressure shall be supplied by compressed air, nitrogen, or helium **and** applied within 15 seconds.

n. After 7.5 minutes, ± 0.1 minute, on the first timer, remove the container and any adhering liquid on the drain tube and discard. Place a dry 10 cm^3 graduated cylinder under the drain tube and continue collecting filtrate **to the end** of the second **timer** set at 30 minutes. Remove the graduated cylinder and record the volume of filtrate collected. This filtrate volume represents the volume collected in the **time interval** from 7.5 minutes to 30 minutes.

22.7 FILTRATE VOLUME-CALCULATION

$$\text{filtrate volume (cm}^3\text{)} = 2 \times [\text{filtrate volume (cm}^3\text{)}] \quad (27)$$

22.8 VISCOSITY-PROCEDURE

The procedure for measuring the apparent viscosity of PAC-HV is as follows:

a. Add 42.00 g, ± 0.01 g, of sea salt to 1000 cm^3 , ± 2 cm^3 , deionized water.

b. To 358 g of the sea salt solution, add 35.00 g, ± 0.01 g, potassium **chloride**.

c. Weigh 3.00 grams, ± 0.01 gram, PAC-HV. Add the PAC-HV slowly at a uniform rate over a time **interval** of **about 60** seconds while stirring on the mixer.

Note: The PAC-HV shall **be** added away from the impeller shaft but in the vortex to minimize dusting.

d. After stirring 5 minutes, ± 0.1 minute, remove the container from the mixer and scrape its side with the spatula to dislodge any material adhering to the container wall. Be sure all material clinging to the spatula is incorporated into the suspension.

e. **Replace the container on** the mixer and continue to stir. **The container may need** to be removed from the mixer and the side scraped to dislodge any adhering PAC-HV after another 5 or 10 minutes. Total mixing time elapsed from beginning of the PAC-HV addition shall equal 20 minutes, ± 1 minute.

f. Age the suspension for 16 hours, ± 0.5 hour, in a sealed or covered container at 77°F , $\pm 2^{\circ}\text{F}$ (25°C , $\pm 1^{\circ}\text{C}$). Record storage temperature and storage duration.

g. **After aging, stir the suspension on the** mixer for 5 minutes, ± 0.1 minute.

h. Pour the suspension into the viscometer cup provided with the direct indicating viscometer. The dial reading at the 600 rpm rotor speed setting of the viscometer shall be taken at a suspension test temperature of 77°F , $\pm 2^{\circ}\text{F}$ (25°C , $\pm 1^{\circ}\text{C}$). Record the 600 rpm dial reading.

22.9 VISCOSITY—CALCULATION

$$\text{apparent viscosity} = \frac{600 \text{ rpm dial reading}}{2} \quad (28)$$

23 Low Viscosity (LOWS) Polyanionic Cellulose

23.1 DESCRIPTION

23.1.1 Low viscosity polyanionic cellulose, PAC-LV, is a water-soluble polymer produced only from cellulose chemically reacted with anionic groups and should not contain any other polysaccharides such as starch, **guar** or other naturally occurring polymers or their derivatives. The product is a **free-flowing** powder or granular powder.

23.1.2 PAC-LV is widely used in water-based drilling fluids for a variety of applications such as filtration control, **vis-**

cosity, and inhibition. Although field use may vary, this procedure focuses on viscosity and filtration control characteristics.

23.1.3 The intention of this procedure is to present a simple and reproducible method for assessing the performance properties of PAC-LV. Specification parameters have not yet been developed for this material, but it is the intention of Subcommittee 13 to adopt specifications based on data generated by users.

23.1.4 A synthetic seawater mud is used for determining the viscosity and filtration control of PAC-LV.

23.2 MOISTURE-EQUIPMENT

The following equipment will be needed to measure the moisture content of PAC-LV:

- a. Oven: Regulated to 220°F, ± 5°F (105°C, ± 3°C).
- b. Balance: Precision off 0.1 g.
- c. Evaporating dish.
- d. Spatula.
- e. Desiccator with calcium sulfate (CAS #7778-18-9) desiccant, or equivalent.

23.3 MOISTURE-PROCEDURE

The procedure for measuring the moisture content of PAC-LV is as follows:

- a. Weigh 10.0 g, ± 0.1 g, PAC-LV sample into a tared evaporating dish. Record the weight added to evaporating dish.
- b. Dry sample in the oven for 4 hours.
- c. Cool to room temperature in desiccator.
- d. Reweigh the evaporating dish containing the dried sample. Record the weight.

23.4 MOISTURE-CALCULATION

moisture weight % = (29)

$$\frac{100 \times [\text{original weight (g)} - \text{dried weight (g)}]}{\text{original weight (g)}}$$

23.5 FLUID PROPERTIES-EQUIPMENT

The following equipment will be needed to measure the fluid properties of the test fluid:

- a. Thermometer: 32 – 220°F, ± 5°F (0 – 105°C, ± 3°C).
- b. Balance: Precision off 0.01 g.
- c. Mixer (e.g., Multimixer Model 9B with 9B29X impellers, or equivalent, as shown in Figure 3).
- d. Mixer container: Approximate dimensions-7 in. (180 mm) deep, 3¹³/₁₆ in. (97 mm) ID top 2³/₄ in. (70 mm) ID bot-

tom (e.g., Hamilton Beach Mixer Cup No. M110-D, or equivalent).

- e. Spatula.
- f. Container: Glass or plastic with stopper or lid for salt solutions.
- g. Motor-driven direct indicating viscometer as referenced in API Recommended Practice 13B-1.
- h. Timers: Two interval, mechanical or electrical, precision of ± 0.1 minute.
- i. Filter press: As referenced in API 13B-1, 3.2.
- j. Graduated cylinders: One 10 cm³, ± 0.1 cm³ (TC); one 500 cm³, ± 5 cm³ (TD).
- k. Sea salt, ASTM D1141-52 (or latest revision), Formula A.
- l. API Standard Base Evaluation Clay (see API Specification 13A).
- m. Potassium chloride (CAS #7447-40-7).
- n. Sodium bicarbonate (CAS #144-55-8).
- o. Deionized or distilled water.

23.6 FILTRATE VOLUME-PROCEDURE

The procedure for measuring the volume of the test fluid is as follows:

- a. Add 42.00 g, ± 0.01 g, of sea salt to 1000 cm³, ± 2 cm³, deionized water.
- b. To 358 g of the sea salt solution, add 35.00 g, ± 0.01 g, potassium chloride.
- c. After stirring 3 minutes, ± 0.1 minute, add 1.00 g, ± 0.01 g, sodium bicarbonate.
- d. After stirring 3 minutes, ± 0.1 minute, add 28.00 g, ± 0.01 g, API Standard Base Evaluation Clay.
- e. After stirring 5 minutes, ± 0.1 minute, remove the container from the mixer and scrape its sides with the spatula to dislodge any material adhering to the container wall. Be sure all material clinging to the spatula is incorporated into the suspension.
- f. Replace the container on the mixer and continue to stir an additional 5 minutes, ± 0.1 minute.
- g. Weigh 2.00 g, ± 0.01 g, PAC-LV. Add the PAC-LV slowly at a uniform rate over a time interval of about 60 seconds while stirring on the mixer.

Note: The PAC-LV shall be added away from the impeller shaft but in the vortex to minimize dusting.

h. After stirring 5 minutes, ± 0.1 minute, remove the container from the mixer and scrape its side with the spatula to dislodge any PAC-LV adhering to the container walls. Be sure all PAC-LV clinging to the spatula is incorporated into the suspension.

i. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the side scraped to dislodge any adhering PAC-LV after another 5 or 10 minutes. Total mixing time elapsed from the

beginning of PAC-LV addition shall equal 20 minutes, ± 1 minute.

j. Age the suspension for 16 hours, ± 0.5 hour, in a sealed or covered container at $77^{\circ}\text{F}, \pm 2^{\circ}\text{F}$ ($25^{\circ}\text{C}, \pm 1^{\circ}\text{C}$). Record storage temperature and storage duration.

k. After aging, stir the suspension on the mixer for 5 minutes, ± 0.1 minute.

l. Pour the PAC-LV suspension into a filter press cell. Before **adding** the suspension, be sure each part of the filter cell is dry and that no gaskets are distorted or worn. The temperature of the suspension shall be $77^{\circ}\text{F}, \pm 2^{\circ}\text{F}$ ($25^{\circ}\text{C}, \pm 1^{\circ}\text{C}$). Pour the suspension to within $\frac{1}{2}$ in. (13 mm) of the top of the cell. Complete assembly of the filter press cell. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.

m. Set one timer for 7.5 minutes, ± 0.1 minute, and the second timer for 30 minutes, ± 0.1 minute. Start both timers and adjust pressure on the cell to 100 psi, ± 5 psi (690 kPa, ± 35 kPa). Pressure shall be supplied by compressed air, nitrogen, or helium and applied within 15 seconds.

n. After 7.5 minutes, ± 0.1 minute, on the first timer, remove the container and any adhering liquid on the drain tube and discard. Place a dry 10 cm^3 graduated cylinder under the drain tube and continue collecting filtrate to the end of the second timer set at 30 minutes. Remove the graduated cylinder and record the volume of filtrate collected.

23.7 FILTRATE VOLUME-CALCULATION

(30)

$$\text{filtrate vol. (cm}^3\text{)} = 2 \times [\text{filtrate vol. in 30 min (cm}^3\text{)} - \text{filtrate vol. in 7.5 min (cm}^3\text{)}]$$

Record the calculated value.

23.8 VISCOSITY-PROCEDURE

The procedure for measuring the Apparent viscosity of PAC-LV is as follows:

- Add 42.0 g, ± 0.1 g, of sea salt to 1000 cm^3 , ± 2 cm^3 of deionized water.
- To 358 g of the sea salt solution, add 35.00 g, ± 0.01 g, potassium chloride.
- Weigh 5.00 g, ± 0.01 g, PAC-LV. Add the PAC-LV slowly at a uniform rate over a time interval of about 60 seconds while stirring on the mixer.

Note: The PAC-LV shall be added away from the impeller shaft but in the vortex to minimize dusting.

d. After stirring 5.0 minutes, ± 0.1 minute, remove the container from the mixer and scrape its side with the spatula to dislodge any material adhering to the container wall. Be sure all material clinging to the spatula is incorporated into the suspension.

e. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the side scraped to dislodge any adhering PAC-LV after another 5 or 10 minutes. Total mixing time elapsed from the beginning of PAC-LV addition shall equal 20 minutes, ± 1 minute.

f. Age the suspension for 16 hours, ± 0.5 hours, in a sealed or covered container at $77^{\circ}\text{F}, \pm 2^{\circ}\text{F}$ ($25^{\circ}\text{C}, \pm 1^{\circ}\text{C}$). Record storage temperature and storage duration.

g. After aging, stir the suspension on the mixer for 5 minutes, ± 0.1 minute.

h. Pour the suspension into the viscometer cup provided with the **direct** indicating viscometer. The dial reading at the 600 rpm rotor **speed** setting of the viscometer shall be taken at a suspension test temperature of $77^{\circ}\text{F}, \pm 2^{\circ}\text{F}$ ($25^{\circ}\text{C}, \pm 1^{\circ}\text{C}$). Record the reading at 600 rpm.

23.9 VISCOSITY-CALCULATION

$$\text{apparent viscosity} = \frac{600 \text{ rpm dial reading}}{2} \quad (31)$$

24 Preparation and Evaluation of Invert Emulsion Drilling Fluids

24.1 DESCRIPTION

24.1.1 Occasionally, evaluation and comparison of invert emulsion drilling fluids are required. The tests described are designed to provide a standardized method for preparing the slurries under the same conditions.

24.1.2 The test methods provide for specific mixing and heat aging procedures in an attempt to minimize the inter-laboratory variation.

24.1.3 Where possible, the test methods and equipment used follow API guidelines.

24.1.4 The test protocol is deliberately simple, designed only to provide a means of comparing the fluids response to heat aging and to the incorporation of reasonable levels of various contaminants that could be expected during drilling, such as drill solids, seawater and formation brines.

24.1.5 The test methods are not designed to be the only way of evaluating invert emulsion drilling fluids.

24.2 EQUIPMENT

The following equipment with the following specifications should be used for this procedure:

- a. Metal contact thermometer: 32 – 220°F (0 – 104°C).
- b. Balance: **Precision off 0.1 gram.**
- c. Mixer: Silverson **L2R** or **L4R** heavy-duty laboratory model or equivalent, with square hole-high shear impeller screen. The screen should be replaced when approximately 10% weight **loss** has occurred through wear. The **bushing** should be replaced when any appreciable play can be felt between it and the shaft. Similarly, the shaft should be replaced when the diameter at the bushing has decreased by ten-thousandths of an inch (1%).

Note: Silverson mixers are available through agents in Argentina, Australia, Belgium, Canada, Chile, Colombia, Denmark, the Dominican Republic, Finland, France, Germany, Greece, Israel, Italy, Korea, Malaysia, Malta, Mexico, the Netherlands, New Zealand, Norway, Peru, Singapore, South **Africa**, Spain, Sweden, Switzerland, Taiwan, Thailand, the USA and Zimbabwe. The main office is in the UK and further information can be obtained at the following address:

Silverson **Machines** Ltd., Waterside, Chesham,
Buckinghamshire, England, HP5 1 PQ

USA representatives can be found at the following address:

- 1. Silverson Machines, Inc., P.O. Box 589, 355 Chestnut Street, East Longmeadow, Massachusetts 01028.
- 2. Silverers Machines, Inc., P.O. Box 1167, Poway, California **92074-1167**.

- d. Mixer: **10,000 rpm**, ± 300 rpm, under load, with a single **sinewave** impeller system, approximately 1 in. (2.5 cm) in diameter (e.g., Hamilton Beach at the low gear/speed setting). The impeller should be replaced when approximately 11 to 13% weight loss has occurred through wear.
- e. Container: conical, stainless steel, approximately **2000 cm³** capacity, with base internal diameter of approximately 3 in. (7.6 cm) and top internal diameter **of** approximately 6 in. (15.2 cm).
- f. Container: Hamilton Beach Mixer Cup **M-110D**, or equivalent.
- g. Spatula.
- h. Tachometer: Optical, magnetic induction, or equivalent.
- i. Mud balance: As described in API Recommended Practice **13B-2**, Field Testing of Oil-Based Drilling Fluids.
- j. Direct indicating viscometer: As **described in API Recommended Practice 13B-2**.
- k. **Thermostatically controlled viscometer cup**: As described in API Recommended Practice **13B-2**.
 - 1. High temperature/high pressure filter press: as described in API Recommended Practice **13B-2**.
- m. Electrical stability meter: as described in API Recommended Practice **13B-2**.

n. Heat aging oven: capable of maintaining a stable **tempera-**ture and equipped with a roller system, or equivalent, to keep the aging cells in motion.

o. Heat aging cell: preferably stainless steel, capacity 350 – 500 **cm³**, with the capacity of pressurization by means of a valving system if necessary.

p. Water bath.

q. Sea salt: sea salt is obtained from local laboratory supply houses, e.g., Merck, **#33153 3P—Seawater** Corrosion Test Mixture, and mixed according to manufacturer's instructions. The standard seawater mix should conform to **BS3990/ASTM** standards.

r. Base evaluation clay: API Base Evaluation Clay can be obtained from the API Upstream Segment, 1220 L Street N.W., Washington, D.C. 20005, USA or as OCMA Base Evaluation Clay or Hymod Prima Clay from Whitfield Chemicals Ltd., 23 Albert Street, Newcastle-Under-Lyme, Staffordshire, ST5 **1JP**, Great Britain.

24.3 PROCEDURE-MIXING OF INITIAL DRILLING FLUID

The procedure for mixing initial drilling fluid is as follows:

a. Prepare four laboratory barrels (one laboratory barrel is 350 **cm³**) of fluid to the required formulation with the following parameters specified:

- 1. Mud weight.
- 2. **Oil:water** ratio.
- 3. Percent by weight brine phase.
- 4. HTHP filtrate volume (a range).
- 5. HTHP filtrate testing temperature.
- 6. Rheology requirements (a range).

Mix the fluid over a total of 60 minutes with a Silverson mixer, fitted with a high shear, square hole screen surrounding the impeller.

b. Starting at ambient temperature, the sample should be mixed in a 2 quart (2 **liter**) **conical mixing vessel and the shaft** speed during mixing should be controlled at **6000 rpm**, ± 300 **rpm** using the optical tachometer or magnetic **induction** tachometer to monitor rotational speed. The rotational speed should be measured under actual mixing load and monitored during the period of mixing. Adjustment should be made as necessary, particularly during and after the addition of weighting material.

c. The fluid temperature should be allowed to rise through shear to a maximum of 150°F (**66°C**), as measured by a metal contact thermometer. Thereafter, it should be controlled as necessary by means of a water bath to between 140°F and 150°F (60°C and **66°C**).

d. If the program of testing requires more than 4 laboratory barrels, any additional volume of mud should be mixed in **batches** of 4 laboratory barrels using the procedure outlined above.

e. Blend all batches together to ensure uniformity **before** commencing test regime. Blending should be carried out by

means of a large spatula or by pouring one container to another several times.

Note: Blending should not be done by means of a mechanical mixer. This pouring or gentle stirring technique is necessary to ensure that each laboratory barrel is subject to the same shear per unit time per unit volume.

24.4 PROCEDURE-TESTING OF INITIAL FLUID

The fluid should be tested after mixing using the techniques described in API Recommended Practice 13B-2. The procedure for testing of initial fluid is as follows:

- Density at ambient temperature, 70°F (21°C).
- Rheological** properties at speeds: 600 rpm, 300 rpm, 200 rpm, 100 rpm, 6 rpm and 3 rpm, **10-second** and 10-minute gels with temperature maintained at 120°F (49°C).
- Electrical stability at 120°F (49°C).
- HTHP filtrate volume at 250°F (121°C) temperature and 500 psi (3447 kPa) differential pressure, or other specified temperature and pressure. Consult API Recommended Practice 13B-2, for recommended temperature/pressure values.

Note: If there is a requirement for **HTHP** filtrate volume testing, especially above 250°F (121°C), note that there may be variance in results with different test equipment and test procedures. It is recommended that the test equipment and procedures be agreed upon between vendor and client prior to initiation of testing.

- One 350 cm³ (1 lab barrel) sample of initial mud should be submitted to hot rolling to be used as a blank in all further testing.

24.5 PROCEDURE—CONTAMINATION BY SEAWATER

The procedure for contamination by seawater is as follows:

- To one laboratory barrel of mud, add 10% by volume of standard synthetic seawater mix.
- Mix the mud and synthetic seawater under ambient conditions on a Hamilton Beach mixer. All seawater should be added over 1 minute and a further 4 minutes of mixing should be done.
- Hot roll the sample at the temperature agreed upon by the interested parties.

24.6 PROCEDURE-CONTAMINATION BY API BASE EVALUATION CLAY

The procedure for contamination by API Base Evaluation Clay is as follows:

- To one laboratory barrel of mud, add base evaluation clay on a Hamilton Beach mixer at low speed setting.
- Additions of base evaluation clay are as follows:

		Density		
	U.S.	S.I.	Base	Evaluation Clay
Below 15 lb/gal	Below 1797.4 kg/m ³		35 g	
Above 15 lb/gal	Above 1797.4 kg/m ³		20 g	

- Mix the mud and base evaluation clay under ambient conditions on a Hamilton Beach mixer. All the clay should be added over a 1-minute period and a further 4 minutes of mixing should be done.

- Hot roll the sample at the temperature agreed upon by the interested parties.

24.7 PROCEDURE-CONTAMINATION WITH MIXED-SALT BRINE

This is an additional test recommended for those wells where it is considered relevant, e.g., where Zechstein salt is expected. The brine type to be used in the test will already have been determined by discussion between the interested parties. The procedure for contamination by mixed-salt brine is as follows:

- To one laboratory barrel of mud, add 10% salt brine by volume on a Hamilton Beach mixer at a low speed setting. The brine should be added to the mud at a low speed setting, over a 1-minute period, with an additional 4 minutes mixing. The procedure should be carried out at ambient temperature.
- A typical mixed-salt brine is Camallite Brine, but any simulated brine can be used. To form a Camallite Brine, add to 100 g of water:
 - 125.0 g of magnesium chloride hexahydrate.
 - 14.5 g of potassium chloride.
 - 5.0 g of sodium chloride.

Mix the brine thoroughly on a Hamilton Beach and extract the volume to be used as a contaminant with a syringe during mixing. This ensures that any undissolved salts are incorporated into the contaminant.

- Hot roll the sample at the temperature agreed upon by the interested parties.

24.8 PROCEDURE—HOT ROLLING

The procedure for hot rolling is as follows:

- Samples of the initial formulation and of the contaminated muds should be hot rolled for 16 hours at 250°F (121°C) or other specified temperature. The hot rolling oven should be preheated.
- Pressurization of the hot roll cell with nitrogen should be at the discretion of the company formulating the mud.
- After 16 hours of hot rolling, the oven heat should be switched off and the door opened. The samples should be rolled during the cooling period for at least 30 minutes prior to being removed. Subsequent cooling in air should be for an additional 30 minutes at least. The cell should then be cooled to ambient temperature by partially immersing it in water.

24.9 PROCEDURE-STATIC AGING

This is an additional testing sequence recommended for those wells where it is considered relevant.

- a. Samples of the initial formulation and of the contaminated muds should be static aged for 16 hours at 250°F (121°C) or other specified temperature. The aging oven should be preheated.
- b. Pressurization of the hot roll cell with nitrogen should be at the discretion of the company formulating the mud.
- c. After 16 hours of aging, the oven heat should be switched off and the door opened. The samples should be cooled for at least 30 minutes prior to being removed. Subsequent cooling in air should be for an additional 30 minutes at least. The cell should then be cooled to ambient temperature by partially immersing it in water.
- d. All aged samples should be stirred on a Hamilton Beach mixer for 5 minutes immediately prior to testing.

24.10 PROCEDURE-TESTING AFTER HEAT AGING

The fluid should be tested after mixing as follows using the techniques described in API Recommended Practice 13B-2:

- a. Density at ambient temperature: 70°F (21°C).
- b. Rheological properties at speeds: 600 rpm, 300 rpm, 200 rpm, 100 rpm, 6 rpm and 3 rpm, 10-second and 10-minute gels with temperature maintained at 120°F (49°C).
- c. Electrical stability at 120°F (49°C).
- d. HTHP filtrate volume at 250°F (121 °C) temperature and 500 psi (3447 kPa) differential pressure, or other specified temperature and pressure. Consult API Recommended Practice 13B-2 for recommended temperature/pressure values.

Note: If there is a requirement for HTHP filtrate volume testing, especially above 250°F (121°C), note that there may be variance in results with different test equipment and test procedures. It is recommended that the test equipment and procedures be agreed upon between vendor and client prior to initiation of testing.

25 Laboratory Procedures for High-Temperature/High Pressure Filtration Testing Using the Permeability Plugging Apparatus and Cells Equipped with Set Screw Secured End Caps

25.1 DESCRIPTION

25.1.1 Measurements of the filtration behavior and wall building characteristics of drilling mud are fundamental to drilling fluid control and treatment, as are the characteristics of the filtrate itself, such as its oil, water, or emulsion content.

25.1.2 These characteristics are affected by the types and quantities of solids in the fluid, and by their physical and

chemical interactions. The Permeability Plugging Apparatus (PPA) is a modified high temperature, high pressure filter press used to evaluate these interactions through various types of filter media at pressures up to 2000 psi (13,800 kPa) and temperatures from ambient to as high as 500°F (260°C). Like the standard HTHP filter press, the PPA is suitable for use in either the field or the laboratory.

25.2 SAFETY CONSIDERATIONS

CAUTION: The pressure limitation in the use of the PPA depends upon the sample cell in use. There are two types of cells available: those with threaded end caps and those with set screw-secured end caps. Among them they have a total of five different pressure ratings. For safety's sake, it is imperative that the operator know the maximum operating pressure of his equipment with certainty, and that this pressure not be exceeded. If in doubt, contact the manufacturer, or use the lowest of the possible limits.

On all PPAs with a threaded end cap, manufacturers have modified the hydraulic pressurization system with a means of pressure relief. These might be available on the earlier models using a set screw secured end cap. This should relieve the hydraulic pressure should it approach the sample cell maximum pressure rating. The operator of the equipment should familiarize himself with this portion of the apparatus and ensure that it is working properly.

Safe operation of the PPA requires that the operator understand and practice the correct assembly and operation of the equipment. Improper assembly, incorrect operation, or the use of defective parts, create the possibility of cell leakage or failure, which could result in serious injury or equipment damage.

The sample cell is hot during operation. The operator should be aware of the hot areas and avoid contact with them. Burns can result from touching parts of the equipment during normal operation.

These instruments are electrically heated and, as with any electrical device, if the wiring is damaged or faulty, electrical shorts can occur and create the risk of fire, injury, and equipment damage. These devices should be used only on grounded circuits.

Following are suggestions that must be observed to ensure safe operation and maintenance of the PPA.

25.2.1 Safe Operation of the Hydraulic Pressurization System

25.2.1.1 Make sure the hydraulic pressure has been released and that the pressure gauge on the pump reads zero, before doing the following:

- a. Attempting to disconnect pressure hose from cell at quick coupler.
- b. Attempting to remove cell from heating jacket.

- c. Moving the PPA.
- d. Refilling the hydraulic pump.
- e. Performing any maintenance, including tightening leaking fittings on the hydraulic pump, hydraulic fittings, or cell assembly.

25.2.1.2 After refilling or repairing the hydraulic system, clean up any spilled oil. Oil left on **floors** is hazardous. Also, accumulations of spilled oil near the PPA are tire hazards.

25.2.1.3 Make sure, when assembling the cell, that the end cap set screws are properly aligned and tightened.

25.2.2 Safe Pneumatic Pressurization of Back Pressure Receiver

25.2.2.1 Always use either nitrogen or carbon dioxide to pressurize the back pressure receiver. With silicate fluids, use only nitrogen. Never use compressed air, oxygen, or other nonrecommended gas. If nitrogen is used it must be supplied in an approved nitrogen gas cylinder, or the nitrogen supply system must be built into the laboratory. Nitrogen cylinders must be secured to meet safety standards. **CO₂** is normally supplied in small, pressurized cartridges at about 900 psi (6206 kPa). They are primarily used for field operations. Do not allow these cartridges to be heated or exposed to **fire**. They can explode if overheated.

25.2.2.2 Maintain pressure regulators and gauges in good condition. Never use oil on pressure regulators.

25.2.2.3 Leaking pressurization systems, hydraulic or pneumatic, should be repaired or replaced. Gauges, fittings, and hoses should be kept in good condition, and leaks should be found and corrected. Periodically test the pressure relief valve on the hydraulic pump to verify that it will function properly should excessive pressure develop. Never plug or bypass this safety valve.

25.2.2.4 When pressurizing the back pressure assembly, always open the supply pressure first, then adjust the regulator. Do not attempt to operate the apparatus at pressures in excess of the equipment rating or relief valve settings. When relieving back pressure, shut off the supply pressure, bleed the pressure from the system, then back out the regulator T-screw.

25.2.3 Safe Heating

25.2.3.1 Caution must be exercised to avoid injury while operating the PPA. It becomes hot enough to cause serious burns. Never leave a hot or heating PPA unattended without posting a warning.

25.2.3.2 The practice of removing the cell and cooling it with water is dangerous and should be avoided. Serious injuries can be caused by the steam generated when a hot cell contacts water, by direct contact with the cell, or by accidentally dropping the cell.

25.2.4 Safe Electrical Operation

25.2.4.1 Make sure that the electrical source is fused and grounded. Verify that the power cord on the heating jacket is in good condition and that it is properly grounded.

25.2.4.2 Electrical problems in the wiring or heaters cannot always be detected by visual inspection. The first sign of trouble is often blown fuses, tripped breakers, lengthened heating time, or erratic thermostat performance. Never begin any electrical repairs without first disconnecting the unit from the power source.

25.2.5 Safe Test Cell Maintenance

The filtration cell is a pressure vessel and should be considered to be a source of potential danger. The safety precautions listed should be followed to ensure safe operation.

- a. Cell material should be compatible with the test samples.
- b. Never use cells that show signs of severe pitting or stress cracking.
- c. Never use cells, cell caps, or retainer rings that show any sign of deformation or damage. Inspect all threads carefully for signs of damage.
- d. Use only undamaged, hardened steel, set screws. Others are unsafe.

25.3 EQUIPMENT-PERMEABILITY PLUGGING APPARATUS WITH SET SCREW SECURED END CAPS

CAUTION: There are three different types of cells available that use set screw secured end caps. Those of current and recent manufacture are rated at either 2000 psi (13,800 kPa) or 1800 psi (12,420 kPa). There are still a number of older cells in use which are stamped "2500 psi." In 1996 the rating on these cells was reduced to 1800 psi (12,420 kPa). Unless the user is absolutely certain that the cell to be used is rated at 2000 psi, the 1800 psi (12,420 kPa) pressure limit must be observed.

It is imperative that the manufacturer's recommendations concerning maximum temperature, pressure, and sample size be followed. Failure to do so can lead to serious injury. The operating manual, or this procedure, should be attached to the apparatus and read by anyone who is unfamiliar with the equipment, prior to using it.

25.3.1 Permeability Plugging Apparatus

The PPA is designed to provide improved static filtration measurements. It can be operated at pressures and temperatures approximating those prevailing **downhole** and it permits the use of filtration media chosen to simulate exposed sands as closely as possible. The fluid cell is inverted with the pressure applied from the bottom of the cell, the filter medium on

top, and the filtrate collected from the top. The cell pressure is applied by a small hydraulic hand pump. Pressure is transferred to the drilling fluid sample through a floating piston within the cell. Hydraulic **oil/sample** contamination is prevented by redundant O-ring seals on the piston.

25.3.1.1 Test pressures are limited by the safety limits of the cell as specified by the manufacturer (See preceding Caution); usually either 1800 or 2000 psi (12,420 or 13,800 **kPa**) at some defined temperature. If back pressure is used in the test, the test pressure may have to be reduced to avoid exceeding the pressure limit of the cell.

Note: Cell damage caused by excessive pressure can be categorized as follows: end cap bending, end cap compression, cylinder shear, and cylinder stress. End cap bending can be detected visually or by measurement. End cap compression can be detected by the distortion of the set screw holes or seats, which become oval rather than round. Caps showing signs of damage must not be used and should be discarded. Cell bodies that show signs of stress cracking or serious pitting, or have damaged set screw holes, must not be used.

25.3.1.2 For temperatures above 200°F (93°C), the back pressure receiver must be pressurized to prevent boiling of the filtrate. The standard back pressure receiver uses a **CO₂** pressurizing source to provide the back pressure. A nitrogen pressure source and a nitrogen manifold may be substituted for the CO, when desired.

25.3.1.3 The PPA cell is encased in a thermostatically controlled aluminum heating chamber during heating and filtration. This chamber completely encloses the filtering area, permitting filtration at any desired temperature from ambient to 500°F (260°C). The cell temperature can be measured using a metal stem thermometer inserted into the thermometer into the well in the cell wall. The temperature is adjusted by means of a knob on the thermostat. The dial has a reference scale of 1 to 10. After the desired temperature is obtained once, it can be repeated by setting the thermostat knob to the same reference setting. The standard cells for the PPA filter press are made of stainless steel. Power consumption for the PPA heating jacket is 800 watts.

25.3.2 Filter Medium

25.3.2.1 The PPA can use any one of a number of filtration media, including porous ceramic or **sintered** metal disks, core samples, and beds of coated or uncoated sand. Ceramic disks are available with permeabilities ranging from 100 **mD** to 100 D. The use of media that simulate exposed sand faces, together with the use of relevant test pressures and temperatures, provide the user with a greatly improved picture of what is happening down-hole.

25.3.2.2 The filter medium can be any porous material such as ceramic, **sintered** metal, or resin-coated sand disks, graded sands, or core samples. Standard disk thickness is 0.25 in. (6.4 mm) but with adapters, thicker disks can be used.

A new disk is required for each test. Disks should be soaked in the base fluid for 5 – 10 minutes prior to use. Vacuum saturation should be used for filter media with low porosity and permeability.

25.3.2.3 Other disk types are available, including Berea Sand cores of different porosities and permeabilities. The user should note that these cores, too, have some variability in porosity and permeability, and that this can affect the repeatability of test results. Cores can be cut to fit the apparatus cylinder and are usually 0.25 in. (6.4 mm) thick. With **modification of the cylinder, 1.00 in. (25.4 mm) cores can also be used.**

25.3.2.4 Resin coated sand can be made into a solid disk, selecting the sand size to provide the desired permeability. The sand is heated at 300°F (149°C) for 1 – 3 hours in molds slightly larger than the normal disk size, and either 0.25 or 1.00 in. (6.4 or 25.4 mm) thick. The molds should be coated with silicone grease prior to heating. Resin coated sand disks can be manufactured to provide a **substantial** variation in pore throat size and permeability by varying the mesh sizes of the sands. Coarser sands can be used to provide a filter medium for testing lost circulation material to be used to control seepage losses to severe fluid loss environments.

25.3.2.5 Sintered metal disks or slotted metal disks can be used to simulate fractures or high permeability formations. In the evaluation of seepage loss material needed to seal off a specific formation, the disk pore throat size should be matched with that of the formation.

25.3.2.6 Sand beds can be used as a filtering medium if the PPA cell is oriented with the filter at the bottom of the cell. For greater repeatability in the height of the sand bed, first determine the desired height of the bed, and then weigh the amount of sand necessary to obtain that height. The sand bed should be saturated with the base fluid before the test. If the user desires to run the test in the standard manner, with the filter medium at the top of the cell, resin coated sand can be placed in the cell, heated for 1 – 3 hours at 300°F (149°C), cooled, and then inverted for the test.

25.3.2.7 There is unavoidable variability in the pore throat sizes of the ceramic disks normally used in these tests. Consequently, when running comparative tests, it is recommended that the disks be tested and classified to achieve as much uniformity as possible. The manufacturers run a quality control test for a disk classification and can, upon request, provide the user the mean pore throat diameter and an average porosity. The user can use a simple flow test with water to further classify the disk.

Note: Procedure for ceramic disk comparison—Install disk in a PPA cell and fill the cell with water. Using the air permeability equipment, with the upper cell valve closed, adjust the pressure on 30 psi test gauge to 4.0 – 4.5 psi. Open the valve on top of the cell and

adjust pressure to 2.0 psi \pm 0.1 psi. After opening valve at the bottom of the cell, readjust pressure with the upper valve to 2.0 psi \pm 0.1 psi. Measure time for 300 ml to pass through using a 500 ml graduated cylinder; timing from the 100 ml mark to the 400 ml mark. If the PPT (Permeability Plugging Test) is to be used for comparison purposes, run several disks, classify the disks, and use those of similar values.

253.3 Timer: 30-minute interval.

25.3.4 Thermometer: Up to 500°F (260°C).

253.5 Graduate cylinder (TC): 25 ml or 50 ml.

25.3.6 High speed mixer.

25.4 PROCEDURE—HIGH TEMPERATURE, HIGH PRESSURE

25.4.1 Preheating the Heating Jacket

Connect the power cord to the proper voltage as indicated on the nameplate. Turn the thermostat to the mid-scale and place a metal stem dial thermometer in the thermometer well of the heating jacket. The pilot light will turn on when the heating jacket temperature has reached the thermostat setting. Readjust the thermostat to 10°F (5.6°C) over the desired test temperature.

25.4.2 Loading the Filtration Cell

WARNING: The filtration cell is a pressure vessel. The following safety precautions should be followed to ensure safe operation: The cell material should be resistant to the test sample. Cell bodies that show signs of stress cracking or severe pitting, must not be used. Use only undamaged, hardened steel, set screws. The use of damaged, or common, mild steel, set screws is hazardous.

As received from the manufacturer, the PPA will be equipped with valves that are rated to 500°F (260°C). Should it become necessary to change any valves during the life of this equipment, it is imperative that the replacements be designed and rated for use at 500°F (260°C) or more.

25.4.2.1 Loosen the set screws securing the end caps, then remove the caps from the cell by pulling them straight out, using the nipples and connected hardware as handles. If the cap is stuck, and cannot be freed by rocking it gently, screw the cell cap removal tool into the seat for the nipple and press downward on the handle to remove it. Then unscrew the nipples from the caps and remove the piston from the cell.

25.4.2.2 Check the O-rings on the nipples, the floating piston, the cell body, and the end caps, and replace any that are damaged or brittle. [All O-rings should be replaced routinely after tests at temperatures above 300°F (149°C).] Apply a thin coating of stopcock grease completely around all of the O-rings being especially careful to ensure that those on the piston are well lubricated. Screw the floating piston onto the

T-bar wrench and install the piston into the bottom of the cell, working it up and down to ensure that it moves freely. (The bottom of the cell, the inlet end, has a shorter recess than the top.) Position the piston so that it is at or near the bottom end of the cell, then unscrew the wrench from the piston.

25.4.2.3 Fill the space above the piston with hydraulic oil to just above the end face.

25.4.2.4 Install the hydraulic end cap onto the bottom of the cell: Push in on the back pressure ball on the nipple of the end cap on the pressure inlet end of the cell to relieve the pressure and allow the cap to slide into the cell more easily. Install and tighten the set screws.

Note: Some oil will flow from the threaded hole in the end cap, indicating that no air is trapped between the piston and the end cap.

25.4.2.5 Connect the bottom nipple assembly to the pump hose, and pump enough hydraulic oil to expel all air from the nipple. Then, being careful not to allow any oil to spill from the nipple, connect the nipple assembly to the bottom cell cap and disconnect the pump hose.

Note: The steps that follow can be accomplished in the jacket that is being preheated, in an unheated jacket if one is available—or in a specially constructed stand.

Note: For improved consistency in test results, stir drilling fluid for five minutes immediately before loading the cell.

25.4.2.6 Turn the cell upright and fill with approximately 275 ml of drilling fluid. This allows for expansion while heating. Do not exceed this amount.

25.4.2.7 Reconnect the pump hose to the quick-connect coupling on the nipple at the bottom of the cell and close the pressure valve on the pump. Operate the pump to raise the level of the fluid sample to the O-ring recess.

25.4.2.8 Install the O-ring and set the selected ceramic disk, or other filtering medium, on top of it.

Note: The disk should be soaked in the base fluid, water or base-oil, for at least five minutes before use. Disks should never be reused.

25.4.2.9 Position the top end cap in the cell, tighten the set screws snugly, and close the valve on the top end cap

CAUTION: Thermal expansion of its contents, and of the hydraulic fluid, will cause cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump must be connected quickly to permit the release of hydraulic fluid to prevent over-pressurization. During heating, the pressure in the cell must be controlled by bleeding off the excess periodically.

25.4.2.10 Unless it was installed in 25.4.2.6 above, install the cell in the heating jacket. Make sure that the cell support has been pulled outward using the handle, then insert the cell assembly and rotate it so that the pin in the bottom of the

heating jacket seats into the hole in the bottom of the cell body. This prevents rotation of the **cell**.

25.4.3 Pressurizing the Cell

Note: Filtration at temperatures above the boiling point of the fluid sample requires the use of the back pressure receiver to prevent vaporization of the filtrate. It also requires that the sample be pressurized to prevent it from boiling.

WARNING: When the closed cell is placed in the hot heating jacket, the pressure in the cell will begin to rise rapidly due to thermal expansion of the sample and the hydraulic fluid. The pump must be connected quickly to allow release of hydraulic oil to prevent over-pressurization. During heating, the pressure in the cell must be reduced **periodically**.

25.4.3.1 Refer to Table 3 for the pressure corresponding to the test temperature, and use the hydraulic pump to apply this pressure to the cell. If a manually operated pump is used, it should always be operated at about one stroke per second.

25.4.3.2 While the **cell** is heating, use the following procedure to prepare the back pressure receiver.

a. Check to ensure that the regulator T-screw has been rotated counterclockwise enough to release all pressure. When the pressure has been released the screw will turn **freely**.

b. Open the pressure release valve to relieve **any** remaining pressure and remove the **CO₂** cartridge barrel from the pressure unit. Dispose of the empty **cartridge**, replace it with a new one, and tighten the barrel enough to puncture the cartridge. Do not adjust the regulator at this time.

c. Verify that the pressure release valve on the **CO₂** assembly and the filtrate drain valve are closed.

d. Set the back pressure assembly aside. It will be installed at 25.4.3.4.

25.4.3.3 Monitor the cell temperature with the thermometer in the well in the cell wall, not the well in the heating **jacket**. When the cell reaches the desired temperature, lower the thermostat to reduce the jacket temperature to the test temperature. Hold the cell at the desired temperature until thermal expansion is complete **and** the **cell** pressure stops increasing. This **can** take as long as an hour.

25.4.3.4 With the cell is at the desired temperature and cell pressure stabilized, mount the back pressure receiver on the upper valve adapter **and** secure it with a retaining pin, then install the **CO₂** pressurizing **unit** on top of the receiver and lock it in place with the other retaining pin.

25.4.3.5 If a drain hose is to be used for the filtrate, connect it from the drain valve to the graduated cylinder receiving the filtrate.

Note: To ensure accurate measurements, the space between the filtration medium and the back pressure receiver outlet, and the receiver valve, must **be** filled with the base fluid before starting the test. This will ensure that the fluid passing through the filter displaces an equal volume of fluid to the receiver. Failure to follow this corrective procedure can introduce significant error.

25.4.3.6 Refer to Table 3 to determine the appropriate pressure for the back pressure receiver, and apply it by turning the T-screw on the pressure regulator until the desired pressure is reached.

25.4.3.7 Actuate the pump to raise the cell pressure to the desired level, then open the valve between the cell **and** the back pressure receiver to start the test.

Note: The differential filtration pressure is the difference between the pressure applied to the cell and that maintained on the back pressure receiver.

25.4.4 Conducting the Filtration Test

25.4.4.1 A laboratory timer should be used **and** filtrate should be collected-and its volume recorded at 1, 7.5, and 30 minutes. Plotting this data against the square root of time provides a useful description of the spurt loss. If desired, samples can be taken more frequently, but none should be taken before one minute. Precisely recorded test times and volumes are necessary for accurate calculation of the filtration parameters.

Note: For improved definition of spurt loss, collect filtrate at 1, 5, 7.5, 15, 25, and 30 minutes, and plot cumulative filtrate volumes vs. the **square** root of time.

25.4.4.2 Begin filtration by opening the valve between the cell and the back pressure receiver. Verify that both the cell pressure, as indicated on the pump gauge, and the back pressure are at desired levels. Adjust them as necessary **throughout the test**.

25.4.4.3 Cell pressure can be expected to decrease slightly as filtration reduces the cell contents, and from any leakage at the pump. The pump should be used as necessary to maintain the pressure at the desired level. If a **manually** operated pump is in use, actuate it at about one stroke per second.

25.4.4.4 After each interval, the filtrate should be drained into the graduated cylinder from the back pressure receiver, and the **time** and cumulative volume should be recorded.

Note: It is recommended to recover the **filtrate** directly from the back pressure receiver, not from a drain hose attached to it. If a hose must be used, its length should be minimized to **reduce the** error caused by liquid retention on its internal surface.

25.4.4.5 After 30 minutes, close the filtrate valve and drain all of the filtrate from the back pressure receiver into the graduated cylinder. The total volume of the filtrate in the graduated cylinder should be recorded.

25.5 TEST CONCLUSION AND DISASSEMBLY

25.5.1 Disconnect the heating jacket from the power source.

CAUTION: The temperature of the sample in the cell must be reduced to below 100°F (37.8°C) before the cell can safely be opened.

25.5.2 The pressurized cell assembly should be allowed to cool in the heating jacket. (See above.) When these tests are run with sufficient frequency to justify it, a cooling stand, station, or bath can be provided to expedite the cooling process. There is a cell handling tool available which should be used any time a hot cell is to be handled.

Note: Extreme care must be exercised in cooling hot cells. This procedure, as recommended, makes it difficult to perform more than one test in an eight hour work day with a single PPA. In the interest of improving productivity, users may want to design their own cell cooling procedures and equipment. Safety must be the primary consideration in these designs.

25.5.3 Close the valve between the cell and the back pressure receiver.

25.5.4 Release the pressure on the pump and cell by opening the pump valve, then disconnect the quick coupler between the pump and the nipple adapter on the bottom of the cell.

25.5.5 Release the back pressure by turning the T-screw on the regulator counterclockwise until it turns freely.

25.5.6 Bleed the pressure from the back pressure receiver by opening the pressure relief valve on the CO₂ unit.

25.5.7 Open the drain valve on the back pressure receiver and collect the last few drops of filtrate in the graduated cylinder.

25.5.8 After removing the locking pin securing it, remove the CO₂ assembly from the top nipple adapter.

25.5.9 After removing its locking pin, remove the back pressure receiver.

25.5.10 The cell may be opened after its contents have been allowed to cool,

CAUTION: If it is suspected that pressure remains in the cell, and the lower end cap does not include a screen, the following procedure can be used to determine the position of the floating piston. Remove the quick-connect assembly from the bottom end cap of the cell and insert a small drill bit or wire through the end cap to determine whether the floating piston is at the bottom. If the piston is *not* at the bottom, there is no pressure. If the piston is at the bottom, there could be pressure remaining in the cell. Reconnect the hydraulic pump and pump several strokes to move the piston. If the cell is pressurized, it will be obvious from the force required to move the piston.

If there are indications are that the cell is pressurized, completely remove the filtration valve assembly from the cell and insert a small drill bit or wire into the cell cap to remove the obstruction. The drill or wire will stop when it contacts the filter disk. Make sure that gloves are worn and that the opening is pointed away from the operator when inserting the bit or wire.

The cell should be opened only when the user is confident that the contents are no longer under pressure.

Note: The cell may be raised in the heating jacket and placed on the cell support, or laid on a bench, while it is being opened.

25.5.11 Loosen the set screws securing the end cap and, using the valve and nipple adapter as a handle, pull the cap from the cell. If it is stuck, a rocking motion of the valve and nipple adapter may free it. If it cannot be freed in this manner, unscrew the valve and nipple adapter, install the cap removal tool in its place, and use it to remove the cap.

25.5.12 The end cap must be removed with the cell in the vertical position and the filtration end facing upwards.

25.5.13 Remove the filter disk. Use a small knife, small screwdriver, or similar thin-bladed tool to pry the edge of the disk up, then remove the disk and the filter cake. If required, wash the filter cake lightly with fresh water, or a suitable solvent if the sample is oil-based, then measure and record its thickness and remarks concerning its composition.

25.5.14 Empty the sample out of the cell. Wash the inside of the cell with fresh water or a suitable solvent if the sample is oil-based. It is usually not necessary to remove the floating piston and the bottom end cap unless the last test was run at 300°F (149°C) or higher.

Note: If testing was conducted at temperatures above 300°F (149°C), the O-rings must be replaced. Perform the following three steps to replace the O-rings on the floating piston and the bottom end cap.

25.5.15 Remove the bottom end cap using the procedure outlined in 25.5.11 and 25.5.12.

25.5.16 Remove the floating piston. Screw the T-bar wrench into the floating piston and push or pull to slide the piston out of either end of the cell. Note that the floating piston can be removed through the top end without removal of the bottom end cap. Remove and dispose of all of the O-rings on the piston and the cap.

25.5.17 Clean the parts for reuse.

25.6 DATA REPORTING

25.6.1 Filtrate Reporting

Report the actual cumulative filtrate volume, in ml, collected through each of the selected time periods.

25.6.2 Spurt Loss

The Spurt Loss is the volume of fluid that passes through the **filtration** medium before a filter cake is formed. It can be depicted by the intercept, on the y-axis, of the straight line representing the static filtration rate, when the square root of filtration time is plotted along the x-axis, and the filtrate volume (doubled to correct for filtration area when using 3.5 in.² filtration media) is plotted along the y-axis. Alternatively, an approximate value can be calculated using the equation which follows.

Note: To define the spurt loss more accurately, collect and record the filtrate more frequently and plot the data as described in the note following 25.4.4.1.

25.6.3 Calculations

PPT Value, ml = $2 \times EV_{30}$

Spurt Loss, ml = $2 \times [EV_{7.5} - (EV_{30} - EV_{7.5})]$

Static Filtration Rate, $R_S = 2 \times [(EV_{30} - EV_{7.5})] / 2.739$

where

EV = the effluent volume (filtrate) in ml using a 3.5 in.² disk,

$EV_{7.5}$ = the filtrate volume in ml after 7.5 minutes,

EV_{30} = the filtrate in ml after 30 minutes.

Note: Observe that all three of these parameters are calculated on the basis of filtrate volume corrected for filtration area. The filter media routinely used in these tests have half the filtration area of that used in the standard API low pressure filtration test. Doubling the filtrate volume compensates for this area difference. The constant, 2, can be modified as necessary to accommodate tests made utilizing other filtration areas.

25.6.4 Filter Cake Reporting

Measure and record the filter cake thickness to the nearest 1/32 in. (0.8 mm). Include a description such as hard, soft, tough, flexible, rubbery, firm, etc. **Although** these are necessarily subjective judgements, they can convey important information.

Note: Nitrogen manifolds having a 600 psi gauge for back-pressure need to be modified to attain the 700 psi. However, tests are possible within the limits of the manifolds.

26 Laboratory Procedures for High-Temperature/High Pressure Filtration Testing Using the Permeability Plugging Apparatus and Cells Equipped with Threaded End Caps

26.1 DESCRIPTION

26.1.1 Measurements of the filtration behavior and wall building characteristics of drilling mud are fundamental to

Table 3-Starting Cell Pressures and Back Pressures for Various Test Temperatures

Temperature Range, °F	Heating Pressure or Back Pressure, psi
Less than 2000	0
200-300	100
301-350	150
351-375	200
376-400	250
401-425	350
426-450	450
451-475	550
476-500	700

drilling fluid control and treatment, as are the characteristics of the filtrate itself, such as its oil, water, or emulsion content.

26.1.2 These characteristics are **affected** by the types and quantities of solids in the fluid, and by their physical and chemical interactions. The Permeability Plugging Apparatus (PPA) is a modified high temperature, high pressure filter press used to evaluate these interactions through various types of filter media at pressures up to 5000 psi (34,500 kPa) and temperatures from ambient to as high as 500°F (260°C). Like the standard HTHP filter press, the PPA is suitable for use in either the field or the laboratory.

26.2 SAFETY CONSIDERATIONS

CAUTION: The pressure limitation in the use of the PPA depends upon the sample cell in use. There are two types of cells available: those with threaded end caps and those with set screw-secured end caps. Among them they have a total of five different pressure ratings. For safety's sake, it is imperative that the operator know the maximum operating pressure of his equipment with certainty, and that this pressure not be exceeded. If in doubt, contact the manufacturer, or use the lowest of the possible limits.

On all PPAs with a threaded end cap, manufacturers have modified the hydraulic pressurization system with a means of **pressure** relief. These might be available on the earlier models using a set screw secured end cap. This should relieve the hydraulic pressure should it approach the sample cell maximum pressure rating. The operator of the equipment should familiarize himself with this portion of the apparatus and ensure that it is working properly.

Safe operation of the PPA requires that the operator understand and practice the correct assembly and operation of the equipment. Improper assembly, incorrect operation, or the use of defective **parts**, create the possibility of cell leakage or failure, which could result in serious injury or equipment damage.

The sample cell is hot during operation. The operator should be aware of the hot areas and avoid contact with them.

Burns can result from touching parts of the equipment during normal operation.

These instruments are electrically heated and, as with any electrical device, if the wiring is damaged or faulty, electrical shorts can occur **and** create the risk of fire, injury, and equipment damage. These devices should be used only on grounded circuits.

Following are suggestions that must be observed to ensure safe operation and maintenance of the PPA.

26.2.1 Safe Operation of the Hydraulic Pressurization System

26.2.1.1 Make sure the hydraulic pressure has been released and that the pressure gauge on the pump reads zero, before doing the following:

- a. Attempting to disconnect pressure hose from cell at quick coupler.
- b. Attempting to remove cell from heating jacket:
- c. Moving the PPA.
- d. Refilling the hydraulic pump.
- e. Performing any maintenance, including tightening leaking fittings on the hydraulic pump, hydraulic fittings, or cell assembly.

26.2.1.2 After refilling or repairing the hydraulic system, clean up any spilled oil. Oil left on floors is hazardous. Also, accumulations of spilled oil near the PPA are fire hazards.

26.2.1.3 When assembling the cell, make sure that the O-rings in the end caps are properly seated.

26.2.2 Safe Pneumatic Pressurization of Back Pressure Receiver

26.2.2.1 Always use either nitrogen or carbon dioxide to pressurize the back pressure receiver. With silicate fluids, use only nitrogen. Never use compressed air, oxygen, or other non-recommended gas. If nitrogen is used it must be supplied in an approved nitrogen gas cylinder, or the nitrogen supply system must be built into the laboratory. Nitrogen cylinders must be secured to meet safety standards. CO₂ is normally supplied in small, pressurized cartridges at about 900 psi (6206 kPa). They are primarily used for field operations. Do not allow these cartridges to be heated or exposed to fire. They can explode if overheated.

26.2.2.2 Maintain pressure regulators and gauges in good condition. Never use oil on pressure regulators.

26.2.2.3 Leaking pressurization systems, hydraulic or pneumatic, should be repaired or replaced. Gauges, fittings, and hoses should be kept in good condition, and leaks should be found and corrected. Periodically test the pressure relief valve on the hydraulic pump to verify that it **will** function

properly should excessive pressure develop. Never plug or bypass this safety valve.

26.2.2.4 When pressurizing the back pressure assembly, always open the supply pressure first, then adjust the regulator. Do not attempt to operate the apparatus at pressures in excess of the equipment rating or relief valve settings. When relieving back pressure, shut off the supply pressure, bleed the pressure from the system, then back out the regulator T-screw.

26.2.3 Safe Heating

26.2.3.1 Caution must be exercised to avoid injury while operating the PPA. It becomes hot enough to cause serious burns. Never leave a hot- or heating-PPA unattended without posting a warning.

26.2.3.2 The practice of removing the cell and cooling it with water is dangerous and should be avoided. Serious injuries can be caused by the steam generated when a hot cell contacts water, by direct contact with the cell, or by **accidentally** dropping the cell.

26.2.4 Safe Electrical Operation

26.2.4.1 Make sure that the electrical source is fused and grounded. Verify that the power cord on the heating jacket is in good condition and that it is properly grounded.

26.2.4.2 Electrical problems in the wiring or heaters cannot always be detected by visual inspection. The first sign of trouble is often blown fuses, tripped breakers, lengthened heating time, or erratic thermostat performance. Never begin any electrical repairs without first disconnecting the unit from the power source.

26.2.5 Safe Test Cell Maintenance

The filtration cell is a pressure vessel and should be considered to be a source of potential danger. The following safety precautions should be followed to ensure safe operation:

- a. Cell material should be compatible with the test samples.
- b. Never use cells that show signs of severe pitting or stress cracking.
- c. Never use cells, cell caps, or retainer rings that show any sign of deformation or damage. Inspect all threads carefully for signs of damage.

26.3 EQUIPMENT-PERMEABILITY PLUGGING APPARATUS WITH THREADED END CAPS

CAUTION: There are two manufacturers of PPAs. Both use threaded end caps for the cells used for tests run at pressures in excess of 2000 psi (13,800 kPa). There are threaded caps with three different pressure ratings available: 3000, 4000, and 5000 psi (20,700; 27,600; and 34,500 kPa respectively).

It is imperative that the manufacturers' recommendations concerning maximum temperature, pressure, and sample size be followed. Failure to do so can lead to serious injury. The operating manual, or this procedure, should be attached to the apparatus and read by anyone who is unfamiliar with the equipment before using it. If the user is unable to determine the operating limits with certainty, the lowest pressure limit must be assumed to be applicable.

As received from the manufacturer, the PPA will be equipped with valves that are rated to 500°F. Should it become necessary to change any valves during the life of this equipment, it is imperative that the replacements be designed and rated for use at 500°F or more.

26.3.1 Permeability Plugging Apparatus

The Permeability Plugging Apparatus is designed to provide improved static filtration measurements. It can be operated at pressures and temperatures approximating those prevailing **downhole** and it permits the use of filtration media chosen to simulate exposed sands as closely as possible. The fluid cell is inverted with the pressure applied from the bottom of the cell, the filter medium on top, and the filtrate collected from the top. The cell pressure is applied by a small hydraulic hand pump. Pressure is transferred to the drilling fluid sample through a floating piston within the cell. Hydraulic oil/sample contamination is prevented by redundant O-ring seals on the piston.

26.3.1.1 Test pressures are limited by the safety limits of the cell as specified by the manufacturer; usually either 3000, 4000 or 5000 psi (20,700, 27,600, or 34,500 **kPa**) at 500°F (260°C). The back pressure receiver may be used at pressures as high as 750 psi (5171 kPa). If back pressure is used in the test, the maximum test pressure may have to be reduced to avoid exceeding the pressure limit of the cell.

Note: **Cell** caps showing signs of damage must not be used and should be discarded. Cell bodies **that** show signs of stress cracking or serious pitting must not be used.

26.3.1.2 For temperatures above 200°F (93°C), the back pressure receiver must be pressurized to prevent boiling of the filtrate. The standard back pressure receiver uses a **CO₂** pressurizing source to provide the back pressure. A nitrogen pressure source and a nitrogen manifold may be substituted for the **CO₂** when desired.

26.3.1.3 The PPA cell is encased in a thermostatically controlled aluminum chamber during heating and filtration. This chamber completely encloses the filtering area permitting filtration at any desired temperature from ambient to 500°F (260°C). The cell temperature can be **measured** using a metal stem thermometer inserted into the well in the cell wall. The temperature is adjusted by means of a knob on the thermostat. The dial has a reference scale of 1 to 10. After the desired temperature is obtained once, it can be repeated by setting the

thermostat knob to the same reference setting. The standard cells for the PPA filter press are made of stainless steel. Power consumption for the PPA heating jacket is 800 watts.

The PPA can be used in the field or in the laboratory. A stainless steel carrying case with a fold-down work shelf is available for use in the field.

26.3.2 Filter Medium

26.3.2.1 The PPA can use any one of a number of filtration media, including porous ceramic or **sintered** metal disks, core samples, and beds of coated or uncoated sand. Ceramic disks are available with **permeabilities** ranging from 100 **mD** to 100 **D**. The use of media that simulate exposed sand faces, together with the use of relevant test pressures and temperatures, provide the user with a greatly improved picture of what is happening **down-hole**.

26.3.2.2 The filter medium can be any porous material such as ceramic, sintered metal, or resin-coated sand disks, graded sands, or core samples. Standard disk thickness is 0.25 in. (6.4 mm) but, with adapters, thicker disks can be used. A new disk is required for each test. Disks should be soaked in the base fluid for 5 – 10 minutes prior to use. Vacuum saturation should be used for filter media with low porosity and permeability.

26.3.2.3 Other disk types are available, including **Berea Sand** cores of different porosities and permeabilities. The user should note that these cores, too, have some **variability** in porosity and permeability, and that this can affect the repeatability of test results. Cores can be cut to fit the apparatus cylinder and are usually 0.25 in. (6.4 mm) thick. **With** modification of the cylinder, 1.00 in. (25.4 mm) cores can also be used.

26.3.2.4 Resin-coated sand can be made into a solid disk, selecting the sand size to provide the desired permeability. The sand is be heated at **300°F** (149°C) for 1 – 3 hours in molds slightly larger than the normal disk size, and either 0.25 or 1.00 in. (6.4 or 25.4 mm) thick. The molds should be coated with silicone grease prior to heating. Resin coated sand disks can be manufactured to provide a substantial variation in pore **throat** size and permeability by varying the mesh sizes of the sands. Coarser sands can be used to provide a filter medium for testing lost circulation material to be used to control seepage losses to severe fluid loss environments.

26.3.2.5 Sintered metal disks or slotted metal disks can be used to simulate fractures or high permeability formations. In the evaluation of seepage loss material **needed** to seal off a specific formation, the disk pore throat size should be matched with that **of** the formation.

26.3.2.6 Sand beds can be used as a filtering medium if the PPA cell is oriented with the filter at the bottom of the cell. For greater repeatability in the height of the sand bed, first

determine the desired height of the **bed**, and then weigh the amount of sand **necessary to obtain that height**. The sand bed should be saturated with the base fluid before the test. If the user desires to run the test in the standard manner, with the filter **medium** at the top of the cell, resin coated sand can be placed in the cell, heated for 1 – 3 hours at 300°F (149°C), cooled, and then inverted for the test.

26.3.2.7 There is unavoidable variability in the pore **throat** sizes of the ceramic disks normally used in these tests. Consequently, when **running** comparative tests, it is **recommended** that the disks be tested and classified to achieve as much uniformity as possible. The manufacturers run a quality control test for a disk classification and can, upon request, provide the user the mean pore throat diameter and an average porosity. The user can use a simple flow test with water to further classify the disk.

Note: Procedure for ceramic disk comparison—Install disk in a PPA cell and fill the cell with water. Using **the** air permeability equipment, **with the** upper cell valve closed, adjust **the** pressure on 30 psi test gauge to 4.0 to 4.5 psi. Open **the** valve on top of **the** cell and adjust pressure to 2.0 psi \pm 0.1 psi. After opening valve at the bottom of **the** cell, readjust pressure with the upper valve to 2.0 psi \pm 0.1 psi. Measure time for 300 ml to pass through using a 500 ml graduated cylinder; timing from **the** 100 ml **mark to the 400 ml** mark. If the PPT (Permeability Plugging Test) is to be used for comparison purposes, run several disks, classify the disks, and use **those** of similar values.

26.3.3 Timer: 30-minute interval.

26.3.4 Thermometer: Up to 500°F (260°C).

26.3.5 Graduated cylinder (TC): 25 ml or 50 ml.

26.3.6 High speed mixer

26.4 PROCEDURE—HIGH TEMPERATURE, HIGH PRESSURE

26.4.1 Preheating the Heating Jacket

Connect the power cord to the proper voltage as indicated on the nameplate. Turn the thermostat to the mid-scale and place a metal stem dial thermometer in the thermometer well of the heating jacket. The pilot light will turn on when the heating jacket temperature has reached the thermostat setting. Readjust the thermostat to 10°F (5.6°C) over the desired test temperature.

26.4.2 Loading the Filtration Cell

WARNING: The filtration cell is a pressure vessel. The following safety precautions should be followed to ensure safe operation: The cell material should be resistant to the test sample. Cell bodies that show signs of stress cracking or severe pitting, must not be used.

As received from the manufacturer, the PPA will be equipped with valves that are rated to 500°F (260°C). Should it become necessary to change any valves during the life of this equipment, it is imperative that the replacements be designed and rated for use at 500°F (260°C) or more.

26.4.2.1 Use the spanner wrench to remove the end caps. Then unscrew the nipples **from** the caps and remove the piston from the cell.

26.4.2.2 Check the O-rings on the nipples, the floating piston, the cell body, and the end caps, and replace any that are damaged or brittle. [All O-rings should be replaced routinely after tests at temperatures above 300°F (149°C).] Apply a thin coating of stopcock grease completely around all of **the** O-rings being especially careful to ensure that those on the piston are **well** lubricated. Screw the floating piston onto the T-bar wrench and install the piston into the bottom of the cell, working it up and down to ensure that it moves freely. (The bottom of the cell, the inlet end, has a **shorter recess than the** top.) Position the piston so that it is at or near the bottom end of the cell, then unscrew the **wrench from** the piston.

26.4.2.3 Lubricate the end face of the cell bore, the horizontal **area at the** end of the **bore, with** anti-seize compound and fill the space above the piston with hydraulic oil to just above the end face

26.4.2.4 Lubricate the threads with high temperature grease and then screw the end cap into place, tightening it moderately with the two-pin spanner wrench. Over-tightening will not improve the seal, and will make the cap difficult to remove.

26.4.2.5 Install the hydraulic end cap onto the bottom of the cell: Push in **on** the back pressure ball on the nipple of the end cap on the pressure inlet end of the cell to relieve the pressure and allow the cap to be screwed into the cell more easily.

Note: Some oil will flow from the threaded hole in the end cap, indicating **that** no air is trapped between the piston **and the** end cap.

26.4.2.6 Connect the bottom nipple assembly to the pump hose, and pump enough hydraulic oil to expel all air from the nipple. Then, being careful not to allow any oil to spill from the nipple, connect the nipple assembly to the bottom cell cap and disconnect the pump hose.

Note: The steps **that** follow can be accomplished in **the** jacket **that** is being preheated, in an unheated jacket-if one is available-r in a specially constructed stand.

Note 2: For improved consistency in test results, stir drilling fluid for 5 minutes immediately before loading **the** cell.

26.4.2.7 Turn the cell upright and fill with approximately 275 ml of drilling fluid. This allows for expansion while heating. Do not exceed this amount.

26.4.2.8 Reconnect the pump hose to the quick-connect coupling on the nipple at the bottom of the cell and close the pressure valve on the pump. Operate the pump to raise the level of the fluid sample to the O-ring recess.

26.4.2.9 Install the O-ring and set the selected ceramic disk, or other filtering medium, on top of it.

Note: The disk should be soaked in the base fluid, water or base-oil, for at least 5 minutes before use. Disks should never be reused.

26.4.2.10 Install the top end cap in the cell.

Note: The following step applies only to cells that utilize retainer rings for the top end caps.

26.4.2.11 Lubricate the threads and the bottom of the retainer ring, and screw the ring into the top of the cell. Tighten it, using the single pm spanner wrench if necessary, until the outer knurled flange of the retainer ring is flush against the top of the cell body. Attempting to tighten it further will not improve the seal and will make the cap more difficult to remove.

CAUTION: Thermal expansion of its contents, and of the hydraulic fluid, will cause cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump must be connected quickly to permit the release of hydraulic fluid to prevent over-pressurization. During heating, the pressure in the cell must be controlled by bleeding off the excess periodically.

26.4.2.12 Unless it was installed in 26.4.2.7. above, install the cell in the heating jacket. Make sure that the cell support has been pulled outward using the handle, then insert the cell assembly and rotate it so that the pin in the bottom of the heating jacket seats into the hole in the bottom of the cell body. This prevents rotation of the cell.

26.4.3 Pressurizing the Cell

Note: Filtration at temperatures above the boiling point of the fluid sample requires the use of the back pressure receiver to prevent vaporization of the filtrate. It also requires that the sample be pressurized to prevent it from boiling.

WARNING: When the closed cell is placed in the hot heating jacket, the pressure in the cell will begin to rise rapidly due to thermal expansion of the sample and the hydraulic fluid. The pump must be connected quickly to allow release of hydraulic oil to prevent over-pressurization. During heating, the pressure in the cell must be reduced periodically.

26.4.3.1 Refer to Table 3 for the pressure corresponding to the test temperature, and use the hydraulic pump to apply this pressure to the cell. If a manually operated pump is used, it should always be operated at about one stroke per second.

26.4.3.2 While the cell is heating, use the following procedure to prepare the back pressure receiver:

- a. Check to ensure that the regulator T-screw has rotated counterclockwise enough to release all pressure. When the pressure has been released the screw will turn freely.
- b. Open the pressure release valve to relieve any remaining pressure and remove the CO₂ cartridge barrel from the pressure unit. Dispose of the empty cartridge, replace it with a new one, and tighten the barrel enough to puncture the cartridge. Do not adjust the regulator at this time.
- c. Verify that the pressure release valve on the CO₂ assembly and the filtrate drain valve are closed.
- d. Set the back pressure assembly aside. It will be installed in 26.4.3.4.

26.4.3.3 Monitor the cell temperature with the thermometer in the well in the cell wall, not the well in the heating jacket. When the cell reaches the desired temperature, lower the thermostat to reduce the jacket temperature to the test temperature. Hold the cell at the desired temperature until thermal expansion is complete and the cell pressure stops increasing. This can take as long as an hour.

26.4.3.4 With the cell is at the desired temperature and cell pressure stabilized, mount the back pressure receiver on the upper valve adapter and secure it with a retaining pin, then install the CO₂ pressurizing unit on top of the receiver and lock it in place with the other retaining pin.

26.4.3.5 If a drain hose is to be used for the filtrate, connect it from the drain valve to the graduated cylinder receiving the filtrate.

Note: To ensure accurate measurements, the space between the filtration medium and the back pressure receiver outlet, and the receiver valve, must be filled with the base fluid before starting the test. This will ensure that the fluid passing through the filter displaces an equal volume of fluid to the receiver. Failure to follow this corrective procedure can introduce significant error.

26.4.3.6 Refer to Table 3 to determine the appropriate pressure for the back pressure receiver, and apply it by turning the T-screw on the pressure regulator until the desired pressure is reached.

26.4.3.7 Actuate the pump to raise the cell pressure to the desired level, then open the valve between the cell and the back pressure receiver to start the test.

Note: The differential filtration pressure is the difference between the pressure applied to the cell and that maintained on the back pressure receiver.

26.4.4 Conducting the Filtration Test

26.4.4.1 Verify that the back pressure as read on the pressure regulator gauge is correct. Adjust if required.

26.4.4.2 Set the timer for the desired filtration test times. Filtrate should be collected at 1, 7.5, and 30 minute intervals. Additional data can be collected if desired, however, the first sample should not be taken prior to **one** minute. Precisely recorded test times and filtrate measurements are necessary for accurate calculation of the filtration parameters.

Note: For improved definition of spurt loss, collect filtrate at 1, 5, 7.5, **15, 25**, and 30 minutes, and plot cumulative filtrate volumes vs. **the** square root of time.

26.4.4.3 **Open** the filtration valve to start the test. The cell pressure, as read on the pump gauge, will drop initially. Operate the pump **to** maintain it as close to test pressure as possible. If a manually actuated pump is used, it should be operated at about one stroke per second.

26.4.4.4 At the desired times, use the drain valve to bleed the filtrate **from the back** pressure receiver into the graduated cylinder, **and** record the cumulative amount received. After 30 minutes, record total filtrate collected.

26.4.4.5 The pressure may slowly decrease as the test continues due to the volume loss through filtration, additional pressure should be applied to the cell in order to maintain a constant pressure. Hold the desired pressure on the cell and on the back pressure receiver for the duration of the test.

26.4.4.6 After 30 minutes, close the filtrate valve and drain any remaining filtrate from the back pressure receiver into the graduated cylinder. The total volume of filtrate in the graduated cylinder should be recorded.

Note: It is recommended to recover the filtrate directly from the back pressure receiver, not through a drain hose attached to it. If a hose must be used, its length should be minimized to reduce the **error**-caused liquid retention on its internal surface.

26.5 TEST CONCLUSION AND DISASSEMBLY

26.5.1 Disconnect the heating jacket from the power source.

CAUTION: The temperature of the sample in the cell must be reduced to below 100°F (37.8°C) before the cell can safely be opened.

26.5.2 The pressurized cell assembly should be allowed to cool in the heating jacket (see above). When these tests are run with sufficient frequency to justify it, a cooling stand, station, or bath can be provided to expedite the cooling process. There is a cell handling tool available which should be used any time a hot cell is to be handled.

Note: Extreme care must **be** exercised in **cooling** hot cells. This procedure, as recommended, makes it difficult to perform **more than** one test in an eight hour work day **with** a single PPA. In the interest of improving productivity, users may want to design their own cell cooling **procedures** and equipment. Safety must be the primary consideration in these designs.

26.5.3 Isolate the back pressure assembly from its pressure source by turning the T-screw on the back pressure regulator counterclockwise until it turns freely.

26.5.4 Open the bleed valve on the **CO₂** unit to release the pressure in the back pressure receiver.

26.5.5 Remove its locking pin and **the** **CO₂** pressuring assembly from the top nipple adapter.

26.5.6 Remove its locking pin **and** the back pressure receiver.

26.5.7 Open the valve on the hydraulic pump to release cell pressure, then disconnect the hydraulic quick coupler.

26.5.8 Open the filtration valve. This will relieve any pressure remaining between the cell filter and the back pressure receiver.

CAUTION: If it is suspected that the cell may be pressured, **and the screened** end cap is not in the lower position, the following procedure can be used to verify the position of the floating piston. Remove the quick-connect assembly from the bottom end cap of the cell and insert a small drill bit or wire through the end cap to determine whether the floating piston is at the bottom. If the piston is **nor** at the bottom, there is no pressure. If the piston is at the bottom, there could be pressure remaining in the cell. **Reconnect** the hydraulic pump **and** pump several strokes to move the piston. If the cell is pressurized, it **will** be obvious from the force required to move the piston.

If there are indications that pressure remains in the cell, completely remove the filtration valve assembly from the cell and insert a small drill bit or wire into the cell cap to remove the obstruction. The drill or wire will stop when it contacts the filter disk. Make sure that gloves are worn and that the opening is pointed away from the operator when inserting the bit or wire.

The cell should be opened only when the operator is fully confident that the contents are no longer under pressure.

26.5.9 Raise or remove the cell assembly. If desired, the cell may be raised in the heating jacket either **by lifting** it by the filter valve assembly or using the optional cell handling tool. Attach this tool to the back pressure inlet nipple just above the filtrate valve where the back pressure receiver is normally attached. Secure using the valve **stem** locking pin. The cell can be supported on the cell support, or lifted out of the heating well and laid on a bench while the cell is being opened.

26.5.10 Threaded caps are removed using spanner wrenches. It may be necessary to tap on the wrench to get it started. Opening difficulty is an indication of insufficient lubrication, over-tightening, or insufficient cleaning. It may be necessary to use a suitable holding tool such as a soft jaw

vice, chain wrench, strap wrench, or another similar device to secure the cell while the cap is unscrewed.

26.511 Reposition the cell so that the filter end is up and unscrew the top cap.

26.512 Remove the filter disk. Use a small knife, small screw driver, or similar thin blade to pry the edge of the disk up, then remove the disk and the filter cake. If required, wash the filter cake lightly with fresh water, or the base oil if the sample is oil-based, then measure and record its thickness and remarks concerning its composition.

26.513 Pour the remaining fluid from the cell and wash the inside of the cell with fresh water, or a suitable solvent if the sample is oil-based. It is usually not necessary to remove the floating piston and the bottom end cap unless the last test was run at 300°F (149°C) or higher.

Note: If testing was conducted at temperatures above 300°F (149°C), the O-rings must be replaced. Perform the following three steps to replace the O-rings on the floating piston and the bottom end cap.

26.514 Remove the bottom end cap using the procedure outlined in 26.5.10 and 26.5.11 except that the cell position is reversed, and the two-pin spanner wrench is used.

26.5.15 Remove the floating piston. Screw the T-bar wrench into the floating piston and push or pull to slide the piston out of either end of the cell. Note that the floating piston can be removed through the top end without removal of the bottom end cap. Remove and dispose of all of the O-rings on the piston and the cap.

26.5.16 Clean the parts for re-use.

26.6 DATA REPORTING

26.6.1 Filtrate Reporting

Report the actual cumulative filtrate volume, in ml, collected through each of the selected time periods.

26.6.2 Spurt Loss

The Spurt Loss is the volume of fluid that passes through the filtration medium before a filter cake is formed. It can be

depicted by the intercept, on the y-axis, of the straight line representing the static filtration rate, when the square root of filtration time is plotted along the x-axis, and the filtrate volume (doubled to correct for filtration area when using 3.5 in.² filtration media) is plotted along the y-axis. Alternatively, an approximate value can be calculated using the equation which follows.

Note: To define the spurt loss more accurately, collect and record the filtrate more frequently and plot the data as described in the note following 26.4.4.1.

26.6.3 Calculations

PPT Value, ml = $2 \times EV_{30}$

Spurt Loss, ml = $2 \times [EV_{7.5} - (EV_{30} - EV_{7.5})]$

Static Filtration Rate, $R_s = 2 \times [(EV_{30} - EV_{7.5})]/2.739$

where

EV = the effluent volume (filtrate) in ml using a 3.5 in.² disk,

$EV_{7.5}$ = the filtrate volume in ml after 7.5 minutes,

EV_{30} = the filtrate in ml after 30 minutes.

Note: Observe that all three of these parameters are calculated on the basis of filtrate volume corrected for filtration area. The filter media routinely used in these tests have half the filtration area of that used in the standard API low pressure filtration test. Doubling the filtrate volume compensates for this area difference. The constant, 2, can be modified as necessary to accommodate tests made utilizing other filtration areas.

26.6.4 Filter Cake Reporting

Measure and record the filter cake thickness to the nearest 1/32 in. (0.8 mm). Include a description such as hard, soft, tough, flexible, rubbery, firm, etc. Although these are necessarily subjective judgements, they can convey important information.

APPENDIX A-METRIC “SI” UNIT CONVERSION TABLE

Table A-I -Metric “SI” Unit Conversion Table

Property	Traditional Unit	Recommended SI Unit	Symbol	Conversion Factor	Example
Mechanical Properties				Multiply By:	
Depth	feet	metre	m	0.3048	10,000 ft = 3048 m
Hole Diameter	inch	millimetre	mm	25.4	12 ¹ / ₄ in. = 311 mm
Pipe Diameter	inch	millimetre	mm	25.4	4 ¹ / ₂ in. = 114 mm
Bit Size	inch	millimetre	mm	25.4	12 ¹ / ₄ in. = 311 mm
Weight on Bit	pound	newton	N	4.4	20,000 lb = 88,000 N
Rotary Table Speed	rpm	*revolutions per minute	r/min	1	45 rpm = 45 r/min
Nozzle Size	¹ / ₃₂ inch	millimetre	mm	0.79	¹⁰ / ₃₂ in. = 7.9 mm
Nozzle Velocity	feet/sec	metre/sec	m/s	0.3048	400 ft/sec = 122 m/s
Drilling Rate	feet/hour	*metre/hour	m/h	0.3048	30 ft/h = 9 m/h
Volume	barrels	cubic metre	m³	0.159	3000 bbl = 447 m ³
Liner Size	inch	millimetre	mm	25.4	6 ¹ / ₂ in. = 165 mm
Rod Diameter	inch	millimetre	mm	25.4	2 ¹ / ₄ in. = 57 mm
Stroke length	inch	millimetre	mm	25.4	16 in. = 406 mm
Pump Output	barrel/minute	*cubic metre/min	m³/min	0.159	8.5 bbl/min = 1.35 m ³ /min
	gallon/minute	*cubic metre/min	m³/min	.00378	3.57 gal/min = 0.0135 m ³ /min
Pump Pressure	pound/sq inch	kilopascal	kPa	6.9	2500 psi = 17,300 kPa
Annular Velocity	feet/min	*metre/minute	m/min	0.3048	200 ft/min = 61 m/min
Slip Velocity	feet/min	*metre/minute	m/min	0.3048	20 ft/min = 6.1 m/min
Temperature	degree Fahrenheit	degree Celsius	°C	(°F - 32) / 1.8	80°F = 27°C
	degree Centigrade	degree Celsius	°C	1.0	
Funnel Viscosity	seconds/quart	seconds/litre	s/L	units cannot normally be converted	
	seconds/1000 cm³	seconds/litre	s/L	1.0	
Mud Weight	pounds/gallon	kilogram/cubic metre	kg/m³	120	10 lb/gal = 1200 kg/m ³
	pounds/ft³	kilogram/cubic metre	kg/m³	16	74.8 lb/ft ³ = 1200 kg/m ³
Pressure Gradient	psi/ft	kilopascal/metre	kPa/m	22.6	0.52 psi/ft = 11.8 kPa/m
Hydrostatic Head	psi	kilopascal	kPa	6.9	4000 psi = 27,600 kPa
Shear Stress	lbf/100 ft²	pascal	Pa	0.48	20 lb/100 ft ² = 960 Pa
	dynes/cm ²	pascal	Pa	0.100	10 dynes/cm ² = 1.0 Pa
Shear Rate	reciprocal second	reciprocal second	s⁻¹	1.0	
Apparent, Plastic & Effective Viscosity	centipoise	*centipoise	cP		
Yield Point	lbf/100 ft²	pascal	Pa	0.48	15 lb/100 ft ² = 7.2 Pa
Gel Strength	lbf/100 ft²	pascal	Pa	0.48	3 lb/100 ft ² = 1.44 Pa
Dial Reading	lbf/100 ft²	pascal	Pa	0.51	dial reading -10 = 5.1 Pa
Standard V-G Meter	1.065				
Power Law Constants					
n	dimensionless				
K	dynes s ⁿ /cm ²	millipascal seconds ⁿ /square centimetre	mPa x sⁿ/cm²	100	10 dynes s ⁿ /cm ² = 100 mPa(s ⁿ /cm ²)
	lbf sⁿ/100 ft²	millipascal seconds ⁿ /square centimetre	mPa x (sⁿ/cm²)	479	1.2 lb s ⁿ /100 ft ² = 575 mPa x s ⁿ /cm ²
API Filtrate	cubic centimetre/	*cubic centimetre/30 min	cm³/30 min		

Table A-I-Metric "SI" Unit Conversion Table

Property	Traditional unit	Recommended SI unit	Symbol	Conversion Factor	Example
Filter Cake Sand Content)	1/32 inch	30 min millimetre	mm	0.8	3/32 in. = 2.4 mm
Solids Content)	Volume percent	cubic metre/cubic metre	m ³ /m ³	0.01	10% = 0.1 m ³ /m ³
Oil Content)					
Water Content)					
Particle Size	micron	micrometre	µm	1.0	
Chemical Properties					
Ionic Concentration	grains/gallon	*milligram/litre parts/million	mg/L	17.1	500 grains/gal = 8600 mg/L
			*milligram/litre	mg/L × specific gravity	100,000 ppm of NaCl x 1.0707 = 107,070 mg/L
Alkalinity P _f , M _f , P ₁ , P ₂ MBT	cubic centimetre/ cubic centimetre	cubic centimetre/ cubic centimetre	cm ³ /cm ³		
Methylene Blue Content	cubic centimetre/ cubic centimetre	cubic centimetre/ cubic centimetre	cm ³ /cm ³		
Miscellaneous					
Additive Concentration	pound/barrel	kilogram/cubic metre	kg/m ³	2.85	10 lb/bbl = 28.5 kg/m ³
Corrosion Rate	lb/ft ² /yr	kilogram/square metre/ year	kg/m ² /yr	4.9	0.9 lb/ft ² /year = 4.3 kg/m ² /year
Bentonite Yield	mils/year	year millimetre/year	mm/a	.0254	200 mils/year = 5.08 mm/a
Hydraulic HP	barrels/US ton	*cubic met&metric tonne	m ³ /t	0.175	100 bbl/ton = 17.5 m ³ /t
Screen Size	horsepower	kilowatt	kw	0.746	600 hhp = 450 kw
Mesh	openings/inch	openings/centimetre	openings/cm	0.254	100 mesh = 25.4 openings/cm
Aperture	micron	micrometer	µm	1	
Open Area	area percent	square metre/square metre	m ² /m ²	0.01	30% = 0.3 m ² /m ²
Weight of Drill Pipe	pound/foot	kilogram/metre	kg/m	1.49	19 lb/ft = 29.1 kg/m
Oil Muds					
Oil Content)	volume percent	cubic metre/cubic metre	m ³ /m ³	0.01	10% = 0.1 m ³ /m ³
Water Content)					
Emulsion Stability	volts	volts	V		
Activity	Dimensionless				
Salinity	ppm	*milligrams/litre	mg/L	specific gravity	250,000 ppm of CaCl ₂ x 1.24 = 310,000 mg/L
Aniline Point	degree Fahrenheit	degree Celsius	°C	(°F-32)	150°F = 66°C

Note: * Denotes acceptable deviation from API 2564 preferred unit.

1.8

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